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Los Angeles

Toward Stable Cortisol Sensing with Indium Oxide Field-Effect Transistors

A thesis submitted in partial satisfaction
of the requirements for the degree Master of Science
in Bioengineering

by

Anica Tillu

2026

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2026

ABSTRACT OF THE THESIS

Toward Stable Cortisol Sensing with Indium Oxide Field-Effect Transistors

by

Anica Tillu

Master of Science in Bioengineering

University of California, Los Angeles, 2026

Professor Anne M. Andrews, Co-Chair

Professor Jun Chen, Co-Chair

This thesis asks a different question than most cortisol-sensing work. It asks not whether cortisol can be detected, but whether the field-effect transistors (FETs) used for detection remain electrically stable over anticipated monitoring periods. Previous work has demonstrated cortisol detection using aptamer-functionalized indium oxide field-effect transistors, but stability over prolonged intervals remains underexplored. Importantly, the surface-preparation steps required before aptamer attachment affect FET electrical stability, and it remains unclear whether this stability is preserved through these steps.

This thesis addresses these questions in three stages. The first stage involved a pilot experiment in which bare, untreated In_2O_3 thin-film FETs were exposed to phosphate-buffered saline (PBS), a simple surrogate body fluid. The devices exhibited distinct electrical failure patterns, including increased gate leakage, loss of output saturation, and progressive threshold shifts. This motivated the second stage of the thesis, which examined the surface-preparation workflow used before aptamer functionalization. The workflow began with bare devices, then added dodecanethiol to passivate the gold source and drain electrodes, and progressively incorporated additional pre-aptamer surface chemistry used in the Andrews group's sensing protocol.

The third stage evaluated how electrical states of the FETs changed over time after different stages of surface chemistry. The surface-condition groups were compared under matched air controls and PBS exposure. The results identify how successive surface-preparation steps and environmental exposure contribute to the electrical stability of In_2O_3 FETs, providing a basis for distinguishing sources of instability as the devices progress toward fully assembled aptamer-based sensors. One separate supplemental document (Supplemental Data S1–S3) provides complete device-level and raw-detail electrical records for the Chapter 3 protocol-development experiments, Chapter 4 surface-processing characterization, and Chapter 5 longitudinal environmental-exposure study.

This thesis of Anica Tillu is approved.

Timothy Deming

Jun Chen, Committee Co-Chair

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University of California, Los Angeles

2026

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List of Acronyms

AFM — Atomic force microscopy
APTES — 3-Aminopropyltriethoxysilane
BNC — Bayonet Neill–Concelman connector
CGM — Continuous glucose monitoring
CI — Confidence interval
DDT — 1-Dodecanethiol
DI — Deionized
DMSO — Dimethyl sulfoxide
DNA — Deoxyribonucleic acid
FET — Field-effect transistor
HC3 — Heteroscedasticity-consistent covariance estimator, type 3
HPA — Hypothalamic–pituitary–adrenal
IGZO — Indium gallium zinc oxide
ITO — Indium tin oxide
MBS — 3-Maleimidobenzoic acid N-hydroxysuccinimide ester
NHS — N-Hydroxysuccinimide
OEET — Organic electrochemical transistor
PBS — Phosphate-buffered saline
PDMS — Polydimethylsiloxane
PTFE — Polytetrafluoroethylene
PTMS — Trimethoxy(propyl)silane
QC — Quality control
REML — Restricted maximum likelihood
RF — Radio frequency
RH — Relative humidity
SAM — Self-assembled monolayer
SELEX — Systematic evolution of ligands by exponential enrichment
SMU — Source-measure unit
SS — Subthreshold swing
TFT — Thin-film transistor
UV — Ultraviolet
XPS — X-ray photoelectron spectroscopy

Supplementary Materials

One separate supplemental document accompanies this thesis and contains the following sections:

Supplemental Data S1. Chapter 3 complete device-level records. Complete longitudinal and PRE/POST electrical records for the five-day bare-device pilot, additional-wafer PBS replication, DDT compatibility study, thermal-conditioning study, and humidity-control study.

Supplemental Data S2. Chapter 4 complete PRE/POST surface-processing records and raw metric detail. Complete PRE/POST electrical characteristics and raw electrical-metric tables for the DDT, PTMS + DDT, and UV + PTMS + DDT processing conditions.

Supplemental Data S3. Chapter 5 complete device-level records. Complete standardized T0–12 h device records and separate exploratory Day 7 device records for the 48-FET cohort.

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Chapter 1. From cortisol detection to continuous monitoring: Why transducer stability matters

A cortisol measurement provides a cortisol level at a single point in time. However, cortisol is a dynamic biomarker influenced by ultradian pulses, the cortisol awakening response, and changes after acute stress. A single measurement cannot capture these biological phenomena (Russell & Lightman, 2019; Stalder et al., 2025; Upton et al., 2023). This is why continuous cortisol monitoring matters, as it tracks cortisol as it changes throughout the day. Beyond capturing within-day variation, continuous or high-frequency cortisol profiles can improve physiological and clinical interpretation because abnormalities may involve the timing and pattern of cortisol secretion, not only a single concentration. Twenty-four-hour profiles can reveal disturbances in circadian and ultradian regulation and help distinguish transient physiological fluctuations from sustained deviations that isolated measurements may miss (Lightman & Conway-Campbell, 2024).

Much of the field has focused on detecting these changes through improved recognition elements and sensing architectures (Vignesh et al., 2024; Wang et al., 2022). But cortisol is not the only thing that can change during monitoring. The FET can change too. If its electrical characteristics shift over time, it becomes difficult to tell what is actually changing. Is it cortisol, or is it the device beneath it?

This thesis begins with that distinction by examining the FET before the molecular recognition layer is added. First, the electrical behavior of the bare In_2O_3 transducer is monitored under phosphate-buffered saline (PBS) exposure and under matched ambient-air control conditions. The surface-preparation steps used before aptamer attachment are then examined to determine how they affect the FET's electrical stability.

1.1 Cortisol is a dynamic biomarker

Cortisol is the primary glucocorticoid produced by the adrenal cortex. The hypothalamic-pituitary-adrenal (HPA) axis regulates its release. Cortisol performs several functions, including supporting glucose and energy metabolism, modulating immune activity, influencing cardiovascular function, and participating in the physiological stress response (Raff et al., 2014; Russell & Lightman, 2019). Disrupted cortisol regulation is central to endocrine disorders such as Cushing syndrome and adrenal insufficiency (Raff et al., 2014). Researchers have also examined altered HPA-axis activity across psychiatric, cardiovascular, metabolic, and inflammatory conditions (Russell & Lightman, 2019; Ring, 2025). However, its many functions are only part of the challenge in measuring cortisol. Cortisol levels also vary across multiple timescales (Russell & Lightman, 2019; Vignesh et al., 2024).

Throughout the day, cortisol is released in repeated bursts called ultradian pulses (Russell & Lightman, 2019). These pulses occur within a larger circadian pattern, with cortisol levels generally higher in the morning and lower at night. Waking triggers an additional increase in cortisol, known as the cortisol awakening response (CAR), while acute stress can alter cortisol levels by activating the stress response (Russell & Lightman, 2019; Stalder et al., 2025). These processes are related but not the same. For example, a morning cortisol measurement may reflect circadian phase, the awakening response, sleep, recent stress, and the phase of an ultradian pulse simultaneously. The measurement may be technically precise, but it can still be physiologically incomplete when taken only once (Russell & Lightman, 2019).

Upton et al. (2023) made this limitation clear by measuring 24-hour profiles of free cortisol and related adrenal steroids in 214 healthy volunteers. Using portable automated microdialysis, they collected measurements at 20-minute intervals. The resulting profiles

captured overnight dynamics, ultradian pulsatility, and substantial between-individual variation. The study does not establish that every cortisol sensor should collect measurements every 20 minutes, nor does it define a universal clinical sampling interval. Instead, it shows that the biological signal is a trajectory rather than a single concentration.

This temporal structure explains why continuous or near-continuous monitoring can yield information that a single measurement cannot. A useful monitoring device must distinguish a transient pulse, a sustained shift, and a change in the daily cortisol pattern. A single measurement at one point in time cannot provide this information (Upton et al., 2023; Vignesh et al., 2024).

1.2 What “continuous” monitoring requires

The terms wearable, real-time, and continuous are often used together or interchangeably, even though they describe distinct aspects of sensor performance (Parlak, 2021; Ok et al., 2024; Vignesh et al., 2024). Wearable describes how the sensing system integrates with the body. Real-time describes how quickly the system can generate or communicate a measurement. Continuous monitoring means the system must keep producing measurements as cortisol changes over time (Ok et al., 2024; Vignesh et al., 2024). In other words, it is not enough for the sensor to be wearable or to produce a measurement quickly. It must also produce interpretable measurements over time. This creates a different set of challenges, including sustained sample access, fouling, power requirements, and device longevity (Vignesh et al., 2024).

Wearable, real-time cortisol sensing has already been demonstrated using aptamer-functionalized In_2O_3 FETs (Wang et al., 2022). The challenge relevant to this thesis is whether repeated measurements remain interpretable as both the biological environment and sensor components change over time. The relationship between cortisol concentration and electrical

output must remain sufficiently stable, or the drift must be characterized well enough to permit interpretation or correction (Yang & Gao, 2019; Flynn et al., 2023). Signal instability can arise from multiple sources, including receptor degradation, biofouling, sampling variability, and instability at the sensing interface (Watkins et al., 2024; Lai et al., 2026). Because these processes can occur simultaneously, drift in a measured signal may reflect several sources of change.

This thesis focuses on one aspect of that problem: the electrical stability of FETs as successive pre-aptamer fabrication and surface-preparation stages are built. First, it asks whether the electrical state of the bare device can be monitored over time under ambient-air and PBS-exposure conditions. It then asks how the surface-preparation steps used before aptamer attachment alter a FET's initial electrical state. From there, we followed sequential surface chemistries under the same longitudinal air-versus-PBS comparison to determine whether these modifications affect stability.

In an aptamer-functionalized FET, the sensor output depends on the electrical behavior of the transducer underlying the recognition chemistry. If the FET's electrical characteristics change during monitoring, that instability can become part of the measured signal (Kaisti, 2017; Nakatsuka et al., 2018; Wang et al., 2022).

1.3 Cortisol-sensing technologies: Detection is not the same as monitoring

Cortisol is a small steroid molecule, and because it is relatively electrochemically inactive, direct electrochemical detection is challenging (Karuppaiah et al., 2023). Unlike glucose, which can be measured using established glucose-oxidase-based electrochemical sensors, cortisol lacks an equivalent enzyme-catalyzed sensing pathway (Teymourian et al.,

2020; Karuppaiah et al., 2023). Therefore, cortisol sensors often rely on affinity recognition using antibodies, molecularly imprinted polymers, or aptamers. Once a binding event occurs, it can be converted into an electrical, optical, or mechanical signal (Weber et al., 2024; Naeem et al., 2024).

Although cortisol is difficult to detect directly, researchers have explored electrochemical methods for cortisol sensing (Zea et al., 2020; Karuppaiah et al., 2023; Li et al., 2025). These methods include amperometry, voltammetry, and impedance measurements to detect cortisol in buffer and biological samples (Zea et al., 2020; Karuppaiah et al., 2023). Transistor-based electrochemical platforms, including OECTs, have also been used for cortisol sensing (Janardhanan et al., 2022). These approaches are important because they demonstrate that electrochemical readouts can be miniaturized and integrated with wearable electronics (Zea et al., 2020; Wu et al., 2023; Vignesh et al., 2024).

However, detection does not equal sustained operation. Studies commonly report metrics such as limit of detection, response time, selectivity, reproducibility, flexibility, or performance during an on-body demonstration (Karuppaiah et al., 2023; Vignesh et al., 2024; Li et al., 2025). These metrics indicate whether the device can detect cortisol under the tested conditions. What happens after repeated exposure and measurement is a different question. A device can show strong analytical performance without necessarily demonstrating that the sensor remains interpretable over time, which is a separate requirement for long-term monitoring (Li et al., 2025).

Field-effect transistors (FETs) are particularly relevant here because the Andrews group and collaborators developed an ultrathin In_2O_3 aptamer-FET platform that was later adapted for wearable cortisol sensing (Kim et al., 2015; Nakatsuka et al., 2018; Wang et al., 2022). These

devices use a different transduction mechanism. In structure-switching aptamer-FETs, target binding changes the position of the charged aptamer near the semiconductor surface. This conformational change alters channel conductance and produces electrical signals (Nakatsuka et al., 2018; Wang et al., 2022). This thesis addresses a specific question rather than the performance of the complete sensor. It focuses on the electrical stability of In_2O_3 FETs underlying this sensing platform before biological functionalization.

1.4 Molecular recognition and the Debye-screening problem

Field-effect transistor (FET) biosensing becomes more challenging in physiological solutions because of a fundamental electrostatic limitation known as the Debye length limitation (Nakatsuka et al., 2018). Mobile ions in solution screen the electric field produced by a charged molecule (Nakatsuka et al., 2018; Chu et al., 2017). At physiological ionic strength, the Debye screening length is less than one nanometer, whereas conventional immunoglobulin G (IgG) antibodies are approximately 5-10 nm in size (Casal et al., 2012; Chu et al., 2017). A target can therefore bind to its molecular recognition element (receptor) while the binding event still occurs several nanometers away from the semiconductor, where it may have little measurable effect on channel conductance (Nakatsuka et al., 2018).

Nakatsuka and colleagues showed that target binding can alter aptamer conformations, shifting charged regions of the receptor closer to or farther from semiconductor surfaces. When aptamer backbone charges move due to target binding, particularly in stem regions near semiconductor surfaces, they modulate or gate channel conductance even in physiological-strength solutions (Nakatsuka et al., 2018). This enables aptamer-FETs to overcome the Debye-length limitation. However, ionic screening has not disappeared. Instead, the positions of charges

carried by the aptamer near the semiconductor surface shift, allowing the FET to respond even when the target is small and/or electrically neutral (Nakatsuka et al., 2018).

This mechanism is particularly relevant to cortisol sensing. Aptamers can be chemically synthesized and modified for surface attachment. Structure-switching aptamers can also change conformation upon target binding (Odeh et al., 2020; Eisen et al., 2024). Their practical performance still depends on the aptamer sequence, immobilization method, and the environment in which they are used (Lim et al., 2010; Qi et al., 2022).

Wang and colleagues functionalized ultrathin In_2O_3 FETs with a cortisol-specific DNA aptamer and integrated the sensing array into a smartwatch form factor (Wang et al., 2022). The platform detected cortisol over a broad concentration range and was compared with established analytical methods. The authors also used it in diurnal and acute-stress experiments (Wang et al., 2022). The study was an important demonstration of cortisol detection and wearable integration. However, the study did not establish the multi-day electrical stability of the underlying FETs or of the complete aptamer-functionalized sensing interface (Wang et al., 2022).

1.5 The Andrews group In_2O_3 platform

The wearable cortisol sensor was not the first In_2O_3 device developed by the Andrews group. It grew out of a longer line of work by Andrews, Weiss, and collaborating laboratories on ultrathin In_2O_3 FETs, aptamer-based small-molecule sensing, and, eventually, flexible device formats (Kim et al., 2015; Nakatsuka et al., 2018; Liu et al., 2020; Zhao et al., 2022). Early work fabricated ultrathin In_2O_3 FET arrays and demonstrated aptamer-based biosensing on rigid substrates, but only in dilute physiological buffer (Kim et al., 2015). Later studies extended the platform to other small molecules under physiologically relevant ionic conditions. These

included dopamine and serotonin in artificial cerebrospinal fluid, as well as phenylalanine in Ringer's solution and in serum samples (Nakatsuka et al., 2018; Cheung et al., 2019). Flexible formats have also been explored, including PET-based devices and polyimide probes for implantable sensing (Liu et al., 2020; Zhao et al., 2022).

One study from this line of work is particularly relevant to the present thesis. Liu and colleagues reported flexible, multiplexed In_2O_3 nanoribbon FETs, with transfer characteristics measured after immersion in undiluted PBS for up to seven days (Liu et al., 2020). These devices are related to those studied here but are not direct replicas. They used RF-sputtered In_2O_3 nanoribbons on PET and liquid-gated the devices in PBS via a common Au gate electrode (Liu et al., 2020). The devices studied in this thesis are instead solution-processed, back-gated Si/SiO₂ TFTs. Stability was also evaluated differently. Liu and colleagues evaluated longitudinal stability by comparing transfer characteristics measured immediately after fabrication (Day 0) and after 1, 2, 3, 4, 5, and 7 days of immersion in undiluted PBS. Based on the transfer curves presented on logarithmic and linear scales, they reported no significant changes in FET performance over the seven-day period. The authors did not report a quantitative acceptance or failure threshold for longitudinal stability (Liu et al., 2020).

Those differences matter when interpreting the seven-day results. Liu and colleagues reported stable transfer characteristics over one week of immersion in PBS (Liu et al., 2020). The relevant question for the present work is not simply whether an unmodified FET can remain stable during prolonged exposure, but how the successive fabrication and surface-preparation stages used to construct the sensing platform contribute to its long-term electrical behavior.

The previous work establishes what successive generations of the Andrews group platform have already demonstrated: small-molecule sensing at physiological ionic strength and

target concentrations, flexible device formats, and ultimately wearable cortisol sensing (Nakatsuka et al., 2018; Liu et al., 2020; Zhao et al., 2022; Wang et al., 2022). More specifically, what remains unresolved is how the particular FET architecture studied here behaves during longitudinal environmental exposure; how threshold behavior, current, hysteresis, leakage, and functional survival evolve during such exposure; and whether the surface-preparation steps that precede aptamer attachment alter that stability (Liu et al., 2020; Wang et al., 2022).

1.6 Operational stability: The missing layer between detection and deployment

A device can be considered stable for several reasons. It may retain its response after storage, tolerate bending, produce reproducible measurements across devices, resist biofouling, or be reused without performance loss (Kusov et al., 2023; Liu et al., 2020; Li et al., 2025). All of these are useful measures of sensor performance. However, none of them alone indicates whether FET electrical states remain interpretable as devices are exposed and measured over time. For the sensing platform studied here, the relevant question is how those electrical states change as successive fabrication and surface-preparation steps are added, and what each stage contributes to the long-term stability of the progressively assembled device.

For this thesis, operational electrical stability refers to how well FETs maintain an interpretable electrical state across different exposure and measurement intervals. This was evaluated by tracking the following FET variables over time: threshold voltage (V_{th}), drain-to-source current (I_{DS}), subthreshold swing (SS), hysteresis, and gate leakage current (I_G). I_{DS} is the current measured between the source and drain electrodes. I_G is the current measured at the gate electrode and represents unintended current through the gate path. Hysteresis is measured as the difference in V_{th} between forward and reverse gate-to-source voltage (V_{GS}) sweeps. These

parameters describe complementary aspects of FET switching behavior, channel conduction, gate control, and electrical leakage and are established measures of TFT electrical performance and reliability (Abdullah et al., 2021; Bak et al., 2015; Kumaran et al., 2024).

These measurements do not always tell the same story. FETs can still carry substantial drain-to-source current even as their threshold behavior or subthreshold characteristics degrade, indicating that channel conduction can persist despite changes in switching behavior (Bak et al., 2015). Likewise, measurable gate modulation does not rule out gate leakage that is large enough to interfere with measured FET responses (Bak et al., 2015; Lee et al., 2018). This is why simply observing current flow or gate modulation alone is insufficient to establish that FET transfer behavior remains interpretable for a longitudinal duration.

This thesis focuses both on how long reliable thresholds can be extracted and how long FETs remain functionally usable. Recognizable transfer behavior does not always guarantee that V_{th} can still be reliably defined, so threshold extractability was tracked across individual FET trajectories (Yoon, 2023). This thesis also examines functional survival, defined here as the length of time the device continues to meet the prespecified criteria for interpretable FET operation. Together with the electrical parameters above, these measures indicate whether the device remains functionally usable over time in ambient air vs. under PBS exposure.

Instability does not necessarily develop the same way in every device. Some stress-induced shifts can recover, while other degradation processes involve persistent changes in the device or its interfaces (Abdullah et al., 2021; Bak et al., 2015). Looking only at the beginning and end could hide that difference. For that reason, cohort-level summaries are considered alongside each device's trajectory.

One more distinction matters here: device stability is not the same as signal stability. In a functional cortisol (or other analyte) sensor, the observed electrical response includes contributions from the transistor, electrolyte, recognition layer, sample matrix, reference system, and readout electronics (Leung et al., 2021; Wang et al., 2022; Lai et al., 2026). The question is whether a signal begins to drift and, if so, which part of the system is changing. This thesis cannot answer that for the complete sensor. Instead, it isolates FET-level contributions by studying bare and surface-prepared devices under controlled ambient-air and PBS exposure before aptamer attachment. This establishes the device-level behavior that future sensor-level drift measurements would need to distinguish from changes in the recognition layer or sensing environment.

1.7 What the existing literature does and does not establish

A structured audit of the literature summarized in Appendix A shows that, across cortisol-sensing reviews and frameworks, continuous, real-time, wearable monitoring are important goals, and stability is repeatedly identified as a challenge to practical use (Parlak, 2021; Vignesh et al., 2024; Li et al., 2025; Lai et al., 2026). Within these papers, stability does not always mean the same thing. Depending on the study, it may refer to storage or long-term retention, repeatability, reuse, recognition-layer durability, fouling, calibration, reproducibility, or general device robustness (Karuppaiah et al., 2023; Vignesh et al., 2024; Li et al., 2025; Lai et al., 2026). The audit found no shared transistor-level acceptance framework for threshold-voltage

drift, drain-to-source current change, hysteresis, gate leakage, or loss of functional operation across the sources included (Appendix A).

This does not mean that researchers have ignored device-level stability. For example, Sheibani et al. (2021) evaluated drift in an aptamer-functionalized extended-gate FET configuration and found no systematic shift in the transfer response across three consecutive 30-minute buffer incubations. Other studies have examined the same problem under longer or more demanding aqueous conditions. Son et al. (2020) evaluated the reliability of bilayer IGZO electrolyte-gated FETs through drift measurements in $1\times$, $0.1\times$, and $0.01\times$ PBS, and through cyclic pH-hysteresis testing. Hideshima et al. (2022) tracked coated silicon FET biosensors over seven days of immersion in $1\times$ PBS at 37°C and assessed stability by monitoring changes in transconductance. Albarghouthi et al. (2022) treated electrical stability as a device-level issue in solution-gated carbon-nanotube bioFETs. They compared passivation strategies using leakage current, on-current stability, hysteresis, subthreshold swing, and performance during repeated PBS cycling.

Zhu et al. (2021) provide a more direct comparison of gate geometry with the FETs studied in this thesis. Their In_2O_3 nanoline FETs were also back-gated. With increasing exposure time, the transfer curves and V_{th} shifted toward more positive gate voltages (Zhu et al., 2021). However, the architecture differed from the devices studied here. Their Ti/Pt electrodes were covered by a patterned $5\text{-}\mu\text{m}$ SU-8 layer, while a PDMS well confined the solution to the exposed In_2O_3 line region (Zhu et al., 2021). As mentioned previously, Liu et al. (2020) also evaluated In_2O_3 FET stability during prolonged PBS immersion, measuring transfer characteristics through seven days in undiluted PBS. Together, these studies demonstrate that longitudinal electrical behavior of In_2O_3 FETs during physiological-buffer exposure has been

investigated as a device-stability problem, although the device architectures, exposure configurations, and criteria used to evaluate stability differ from those used in this thesis.

A separate framework for this question comes from the oxide-TFT reliability literature. Bias stress can cause threshold-voltage shifts in oxide TFTs via charge-trapping processes (Lee et al., 2008; Na et al., 2024). In solution-processed In_2O_3 , residual hydroxyl groups can affect electrical stability (Yao et al., 2021). Repeated operation in ambient air has also been linked to increased hysteresis in solution-processed In_2O_3 TFTs (H. Lee et al., 2019). More recently, Lin et al. (2026) found that adsorbed oxygen at In_2O_3 surfaces can alter channel conductivity and contribute to threshold-voltage drift. These studies were conducted as FET-reliability or materials experiments, not as cortisol biosensor experiments.

This thesis uses the oxide-TFT literature as a framework for interpreting the electrical behavior and changes observed here. Additionally, the present experiments differ from the FET studies above in device architecture, gating configuration, exposure conditions, surface treatment, and stability endpoints.

1.8 Thesis objective and scope

This thesis investigates how the electrical state of solution-processed In_2O_3 FETs changes under matched air and PBS exposure and what successive pre-aptamer surface preparation stages contribute to that long-term electrical stability.

The first part of this work was a preliminary pilot using bare devices. The pilot characterized electrical failure during PBS exposure and established the exposure and measurement protocol for the primary experiment. The second part focused on surface preparation. We compared four conditions: bare devices; devices with dodecanethiol (DDT)

electrode passivation; devices with PTMS (trimethoxy(propyl)silane) treatment and DDT electrode passivation; and devices with UV treatment followed by PTMS treatment and DDT electrode passivation. We measured devices before and after processing to determine how the added preparation steps altered the FET electrical state before environmental exposure began. The third part evaluated the four surface conditions under matched ambient-air and PBS exposure. Condition-specific measurements collected immediately before exposure served as the baseline (T_0) for each device. From there, we evaluated threshold behavior, drain-to-source current, hysteresis, subthreshold swing, gate leakage, threshold extractability, and functional survival over time.

Our experiments have clear limits on what they are designed to address. No aptamer is attached, and no cortisol response is measured at any stage. The surface treatments are limited to the progressive pre-aptamer conditions examined in this thesis, and no separate structural encapsulation layer is introduced.

We collected electrical measurements periodically after environmental exposure using the common Si/SiO₂ back gate rather than continuously through an electrolyte gate. Keeping the same electrical measurement geometry for the air and PBS groups allows PBS to serve as a comparable exposure condition rather than becoming a separate gating-configuration variable. Other aqueous-stability studies have used different approaches. Liu et al. (2020) used solution-gated In₂O₃ nanoribbon FETs. Zhu et al. (2021) used a back-gated In₂O₃ architecture, but protected the electrodes with patterned SU-8 and confined PBS to the sensing region with a PDMS well. Neither study reported a matched air-exposure control group (Liu et al., 2020; Zhu et al., 2021).

The PBS condition used here is a standardized exposure applied across all four surface conditions. It is not intended to replicate complete microfluidic or wearable sensing environments. The findings are limited to the electrical behavior of the underlying FETs under the surface-preparation and exposure conditions tested here. They do not establish aptamer stability, cortisol specificity, continuous operation, or wearable-format performance.

1.9 Chapter roadmap

Chapter 2 describes the back-gated In_2O_3 TFT platform and the four surface conditions examined in this thesis. It also outlines the matched ambient-air and PBS experimental design, the exposure and handling workflow, the electrical measurements, and the analysis plan. Chapter 3 presents the preliminary pilot study using bare devices, along with the protocol-development experiments that informed how the primary study would be conducted. Chapter 4 examines what each added surface-preparation step contributed to FET electrical states before environmental exposure began. Chapter 5 tracked the four surface conditions over time under matched ambient-air and PBS exposure. Finally, Chapter 6 returns to continuous monitoring and interprets the findings in relation to previous aqueous FET studies. It explains what the results establish and what remains unanswered for future longitudinal evaluation of fully assembled aptamer-functionalized cortisol sensors.

Chapter 2. Device platform and longitudinal experimental design

Chapter 2 describes the device platform and the primary experimental design used in this thesis. The experiment was not intended to reproduce a complete wearable cortisol sensor. Instead, it examined how successive pre-aptamer surface-preparation stages affected the electrical state of the In_2O_3 FETs and how these changes evolved over time under ambient-air vs. PBS exposure. We studied four surface conditions: Bare FETs, which received no study-specific surface treatment after common post-fabrication conditioning; DDT, which introduced dodecanethiol passivation of the Au source and drain electrodes; PTMS + DDT, which added PTMS treatment before the same dodecanethiol passivation; and UV + PTMS + DDT, which added UV pretreatment before the PTMS and dodecanethiol sequence. We evaluated each surface condition under parallel ambient-air and PBS conditions, using the common Si/SiO₂ back gate for electrical characterization.

The experimental workflow incorporated the handling, thermal conditioning, and humidification procedures established in the preliminary experiments described in Chapter 3. The formal design used three parent wafers, with each parent contributing one wafer piece to every Surface $\times$ Exposure condition. This allowed the parent wafer to be retained as a fabrication block in the analysis so that the between-wafer fabrication variability could be accounted for separately from the assigned surface and exposure conditions.

2.1 Device architecture and fabrication

The devices studied in this thesis were solution-processed In_2O_3 FETs fabricated on Si/SiO₂ substrates (Kim et al., 2015; Rim et al., 2015). We performed device fabrication, including post-metallization processing, in the UCLA NanoLab cleanroom. After fabrication, all

devices underwent a common post-fabrication conditioning in the laboratory before entering either the preliminary or formal experiments.

We fabricated the devices on 100-mm-diameter, 500-550- μm -thick, boron-doped p⁺⁺ Si wafers from Silicon Valley Microelectronics (Santa Clara, CA). The wafers had a {100} crystal orientation, a resistivity of 0.001-0.005 $\Omega\cdot\text{cm}$, and a 100-nm-thick thermally grown SiO₂ layer. During electrical characterization, the heavily doped Si substrate served as the common back gate, and the thermally grown SiO₂ served as the gate dielectric. Electrical access to the back gate was achieved by mechanically scratching the backside of each wafer with a Thorlabs S90C carbide scribe. No conductive paint or separately deposited backside electrode was added.

We prepared the In₂O₃ precursor as a 0.1 M solution of indium(III) nitrate hydrate (99.999% trace-metals basis, Sigma-Aldrich, product no. 254215) in deionized water. The solution was sonicated for 5 min and then filtered through a 0.2- μm polyethersulfone (PES) syringe filter (Thermo Scientific, cat. no. 720-1320) before deposition. Before In₂O₃ deposition, the Si/SiO₂ substrates were plasma-treated using the Plasma Etch PE-100 system. The recipe was run twice, with each cycle lasting 60 s at 140 W RF power. Gas 1 was set to zero flow, while the active Gas 2 line supplied oxygen during treatment. The precursor was then spin-coated onto the substrates. The coating process began with a 500-rpm spread step for 5 s at a ramp of 200 rpm/s, followed by a 3000-rpm spin for 40 s at a ramp of 500 rpm/s. After coating, the wafers were prebaked at 100 °C for 5 min and annealed on a hotplate at 250 °C for 2 h. All semiconductor deposition and fabrication steps were completed in the cleanroom environment.

We did not directly measure the In₂O₃ thickness on the wafers used in this study. Previous work on similar solution-processed In₂O₃ platforms reported film thicknesses of approximately 3–4 nm, with approximately 4 nm on rigid substrates and approximately 3 nm on

flexible platforms (Kim et al., 2015; Zhao et al., 2022). These literature values provide platform context but were not treated as measurements for the wafers in the present study.

After In_2O_3 deposition and annealing, we applied MEGAPOSIT SPR 700-1.2 positive photoresist (Rohm and Haas Electronic Materials) using the SVG 8800 wafer track. The programmed coating sequence began with a spin at 1.20 krpm for 3 s. We then dispensed photoresist at 2.00 krpm for 30 s, followed by a final spin at 5.00 krpm for 30 s. The wafers were soft-baked at 95 °C for 90 s. The resist was exposed to our photomask for 6 s using the Karl Suss MA6 Aligner and developed in MF-26A (Rohm and Haas Electronic Materials, cat. no. 10016574) for 75 s. After development, the patterned wafers were rinsed in DI water, dried under N_2 , and post-development dehydrated at 90 °C for 30 s. We deposited metal using a CHA Mark 40 electron-beam evaporator. A 10-nm Ti adhesion layer was deposited at 1 Å/s, followed by 50 nm of Au at 2 Å/s. We performed lift-off in acetone, after which the wafers underwent a 150 °C dehydration bake for 3 min.

The interdigitated Ti/Au source and drain electrodes had a drain-to-source channel length (L) of approximately 25 μm . Each device contained six parallel channel segments, each with an overlapping finger length of approximately 149 μm . This configuration yielded an effective channel width of approximately 894 μm and a (W/L) ratio of ~36. Individual metal fingers were ~30 μm wide.

After cleanroom fabrication, the wafers were transferred to the laboratory in cleanroom storage boxes and allowed to equilibrate to the laboratory environment for approximately 24 h. Laboratory relative humidity was typically 67-70%. Before subdividing the wafers and performing electrical screening, the wafers underwent two 10 min thermal bakes at 100 °C on a laboratory hotplate, with a 10 min cool between each bake.

Post-fabrication conditioning was completed before assigning the FETs to the experimental surface conditions and was distinct from the post-exposure thermal-conditioning procedure developed during the preliminary PBS experiments in Chapter 3. At this stage, the FETs contained no aptamer or other biological recognition layer. The Bare condition refers to the fabricated FET after post-fabrication conditioning but without any study-specific surface treatments described in Section 2.3.

2.2 Study design

The primary experiment included four study arms representing progressively treated surfaces: Bare, DDT, PTMS + DDT, and UV + PTMS + DDT. Each surface condition was tested under two environmental conditions, ambient-air control or PBS exposure, yielding eight experimental conditions.

Table 2.1. Surface $\times$ Exposure design of the primary experiment.

Exposure	Bare	DDT	PTMS + DDT	UV + PTMS + DDT
Ambient air	No surface treatment	Dodecanethiol Au-electrode passivation	PTMS treatment and dodecanethiol Au-electrode passivation	UV pretreatment, PTMS treatment, and dodecanethiol Au-electrode passivation
PBS	No surface treatment	Dodecanethiol Au-electrode passivation	PTMS treatment and dodecanethiol Au-electrode passivation	UV pretreatment, PTMS treatment, and dodecanethiol Au-electrode passivation

Note. Each Surface $\times$ Exposure condition contained three wafer pieces, one from each parent wafer, with two FETs measured per piece. Each condition contained three treatment units and six tested FETs. Detailed treatment procedures are described in Section 2.3, and the chemical basis of the surface-preparation states is discussed in Chapter 4.

The primary experiment used three parent wafers. All three parent wafers were fabricated in the same cleanroom batch. Steps completed separately for each wafer, such as spin coating, were performed sequentially using the same protocol. We carried out other steps across the wafers together when possible. We retained the parent wafer as a fabrication block in the analysis.

We divided each parent wafer into eight pieces. Within each parent wafer, one piece was assigned to each of the eight Surface $\times$ Exposure conditions. Each condition therefore contained three wafer pieces, one from each parent wafer, and these were randomized to the study arms (Figure 2.1). We selected two FETs on each wafer piece, yielding six FETs per Surface $\times$ Exposure condition and 48 FETs across the full cohort. We assigned surface preparation and environmental exposure at the wafer-piece level. We treated each wafer piece as the independent treatment unit and the two FETs measured on the same piece as device-level subsamples.

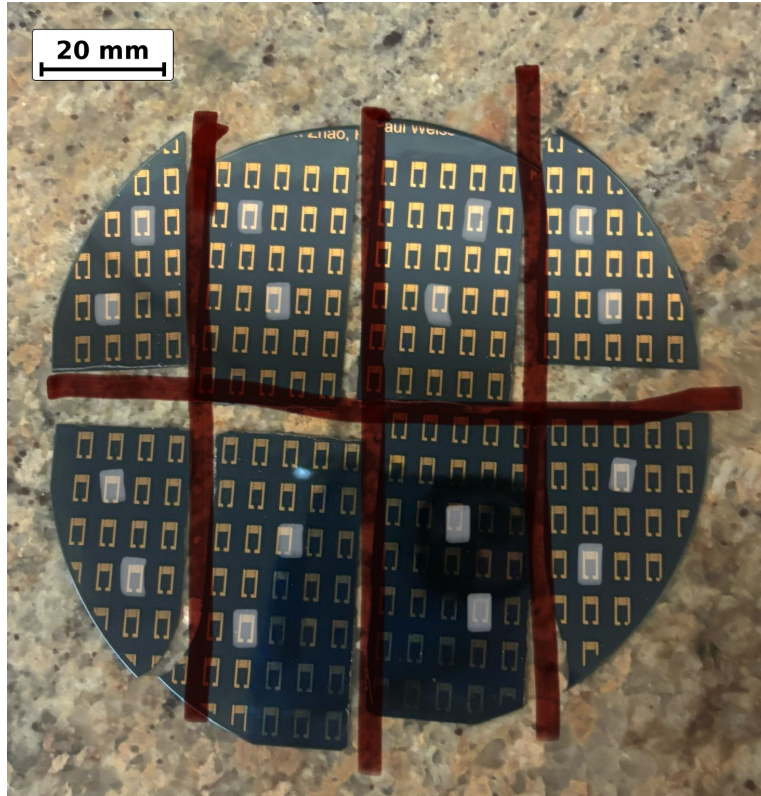


Figure 2.1. Wafer subdivision and exposure allocation for the primary experiment. We divided each of three parent wafers into eight pieces. We selected two FETs (highlighted in white) at corresponding device positions on each wafer piece. Within each study arm, one piece from each parent wafer was assigned to the ambient-air condition, and one piece from each parent wafer was assigned to the PBS condition.

We selected corresponding FET locations across parent wafers so that the selected FETs occupied comparable positions within their parent wafers. Before entering the cohort, we electrically screened the selected FETs on each piece to confirm interpretable FET behavior. We did not rank FETs in the cohort by baseline performance, and we did not substitute reserve devices after T0. If an FET deteriorated after entering the formal experiment, we retained the deterioration as an experimental outcome.

For the three surface-prepared conditions, we collected electrical measurements immediately before processing and immediately after completing the assigned treatment sequence. These paired measurements formed the PRE/POST comparison used in the Chapter 4

surface-processing analysis. We collected a separate T0 prior to environmental exposure. Bare FETs did not undergo a study-specific surface treatment; we collected their T0 after the common conditioning described in Section 2.1 and before environmental exposure.

2.3 Surface-preparation conditions

We divided the FETs into four study arms representing successive stages of the pre-aptamer surface-preparation protocol: Bare, DDT, PTMS + DDT, and UV + PTMS + DDT. These conditions were derived from the surface-chemistry steps our group previously used to fabricate In_2O_3 aptamer-FETs, although the present study stopped before biological functionalization (Wang et al., 2022; Zhao et al., 2022). Therefore, these conditions represent experimental steps in the pre-aptamer workflow, not complete biosensor functionalization states.

In the published wearable cortisol platform, the In_2O_3 FETs were first treated with a 1:9 APTES/PTMS mixture via vapor deposition for 1 h at 40 °C, then heated at 80 °C for 10 min (Wang et al., 2022). The Au source and drain electrodes were subsequently passivated by incubation in 1 mM DDT for 1 h. A 3-maleimidobenzoic acid N-hydroxysuccinimide ester (MBS) and the thiolated aptamer were then introduced to construct the molecular-recognition interface (Wang et al., 2022). Zhao *et al.* (2022) used the same general sequence of mixed-silane treatment and dodecanethiol passivation for flexible In_2O_3 aptamer-FETs on a polyimide substrate.

Here, we omitted APTES from the In_2O_3 treatment conditions. The terminal amine groups of APTES are highly reactive and participate in the downstream coupling chemistry of the complete aptamer interface. For example, in the Wang *et al.* cortisol-sensing devices, MBS served as the crosslinker between the amino-silanized (APTES) In_2O_3 surfaces and the thiolated

cortisol aptamer (Wang et al., 2022). In the fully functionalized format, the APTES amino groups are no longer free; they are incorporated into the linker chemistry. Using only PTMS allowed us to study silane treatment of In_2O_3 without introducing the reactive amine functionality used for downstream coupling. The chemical basis and intended interfacial roles of these surface-preparation states are discussed in detail in Chapter 4.

The order of the remaining treatments was informed by observations from the bare-device pilot. As described in Chapter 3, PBS exposure increased gate leakage and degraded the interpretable output behavior of FETs. These findings did not suggest that unpassivated Au electrodes caused the failures. Nonetheless, they provided a rationale for systematically testing electrode passivation. Zhu *et al.* (2021) used a different type of passivation in their back-gated In_2O_3 aqueous-stability device, employing patterned SU-8 to protect the metal electrodes. However, our group most often uses dodecanethiol self-assembled monolayers to passivate Au source and drain electrodes (Wang et al., 2022; Zhao et al., 2022). We used dodecanethiol (1-Dodecanethiol; Sigma-Aldrich, product no. 471364) to test how an Au-electrode-passivation step affected the electrical state of the FETs and their subsequent stability during PBS exposure. We immersed devices in 1 mM dodecanethiol in ethanol for 1 h. We then rinsed the devices with ethanol and dried them under N_2 . The concentration and incubation time matched the Au-passivation step used in Wang *et al.* (2022).

The next condition incorporated PTMS (trimethoxy(propyl)silane; TCI America, product no. T1801) before dodecanethiol treatment. A 450- μL aliquot of PTMS was placed in a vapor chamber, matching the PTMS content of the 500- μL , 1:9 APTES/PTMS formulation in the complete protocol, with the 50- μL APTES component omitted. The chamber was held in a 40 °C water bath and evacuated for 1 min. Vapor treatment then continued for 1 h. Afterward, the

devices were removed and heated on a hotplate at 80 °C for 10 min before receiving the 1 mM dodecanethiol treatment described above. The 40 °C, 1 h vapor exposure and 80 °C post-silane bake followed the conditions reported for the Wang *et al.* cortisol platform.

The last condition began with 10 min of UV pretreatment, followed by sequential PTMS and dodecanethiol treatment. We included UV treatment because it is the upstream activation step used before silanization in the standard workflow. The UV-related interface modification can also affect the electrical behavior of solution-processed In₂O₃ TFTs. For example, Kim *et al.* used UV-based surface-energy modification during device fabrication and reported effects on active-layer patterning and gate leakage. Their UV treatment modified the substrate/interface during formation of the In₂O₃ active layer, while the present study applied UV to already fabricated FETs before PTMS treatment (Kim et al., 2017). While their study provides evidence that UV-related interface processing can affect electrical characteristics, it does not establish a mechanism or electrical effects of the UV treatment used here.

We characterized devices in all three surface-prepared conditions immediately before and after their assigned treatment sequence. We established a separate baseline before evaluating the effects of PBS exposure. We applied no MBS, aptamer, or subsequent biological functionalization at any stage of this experimental design. This will be the subject of future investigations.

2.4 Environmental exposure conditions

We evaluated each of the four study arms in two environments: an ambient-air control environment and a PBS exposure condition. The PBS exposure provided a controlled, high-ionic-strength aqueous environment that did not replicate all of the proteins, metabolites, and

other constituents of a complete biofluid. Full-strength PBS is relevant to this work because the In_2O_3 aptamer-FET platform was developed for operation under physiological ionic-strength conditions. Related In_2O_3 devices have also been evaluated during multi-day exposure to undiluted PBS (Liu et al., 2020; Zhu et al., 2021).

At the start of the longitudinal study, we prepared $1\times$ PBS by diluting Thermo Scientific/Affymetrix-USB $10\times$ PBS, Molecular Biology Grade (J75889 series), tenfold with DI water. The $10\times$ stock was $0.2\text{ }\mu\text{m}$ -filtered and contained 80.6 mM sodium phosphate, 19.4 mM potassium phosphate, 27 mM KCl, and 1.37 M NaCl at pH 7.4. During the experiment, we kept the prepared $1\times$ working solution tightly closed at laboratory temperature and checked its pH throughout the study with a pH strip to confirm it remained approximately 7.4.

For each exposure, we centered a $6\text{ }\mu\text{L}$ PBS droplet over the active sensing region of each device. Because two devices were tested on each wafer piece, each piece received two droplets. The droplets remained free on the surface without a PDMS well or other patterned liquid-confinement structure. Placement was restricted to the sensing region and did not intentionally wet the probe pads, the back-gate contact, or the cut edges of the piece.

The PBS-exposed pieces were kept in humidified chambers ($150\times 15\text{ mm}$ PYREX borosilicate-glass Petri dishes; Corning 3160-150). We placed five unused PTFE caps around the perimeter of each dish. Each contained approximately 1 mL of DI water and served as an open reservoir for humidification. The reservoirs did not contact the wafer pieces or PBS droplets, and we covered each dish with its fitted glass lid. The water reservoirs limited evaporation of the free PBS droplets. We included them based on the protocol-development experiment described in Chapter 3 rather than on an assumed chamber relative-humidity target. Relative humidity inside the dishes was not measured. We described the chambers operationally as humidified.

We distributed the 12 PBS-exposed wafer pieces across four humidified chambers, with three pieces per chamber. The 12 ambient-air pieces were distributed in the same way across four identical sterile plastic Petri dishes, with three pieces per dish. These chambers contained neither PBS droplets nor supplemental water reservoirs. The air condition served as an ambient-air control and was not a humidity-matched control for the PBS chambers. Each chamber contained the three parent-wafer pieces assigned to a single Surface $\times$ Exposure condition.

At each scheduled measurement timepoint, PBS exposure was interrupted to perform the handling and electrical-characterization sequence described in Section 2.5. After measurements, we applied a fresh 6 μ L PBS droplet and returned the wafer pieces to their assigned chambers. Air-control pieces underwent the same handling sequence before being returned to their dry chambers.

2.5 Exposure, rinse, drying, and measurement workflow

At each scheduled electrical characterization timepoint, the PBS droplets were first aspirated with a micropipette without touching the underlying FETs. Each wafer piece was held at a $\sim 45^\circ$ angle, and each FET was rinsed with DI water for approximately 10 s. Each wafer piece was then dried under N₂ until no visible liquid remained.

After rinsing and N₂ drying, the PBS-exposed pieces were heated at 70 °C for 10 min. This standardized thermal-drying step was established in the protocol-development experiment described in Chapter 3. The air-control pieces received the same thermal treatment. Because these pieces had not undergone aqueous exposure, they were not rinsed with DI water or dried under N₂. After heating, wafer pieces were cooled to laboratory temperature for 2 min before electrical characterization. We used the same cooling interval at every time point.

We performed electrical characterization after exposure and drying, with PBS absent from device surfaces during measurement. After measurements were complete, we added fresh PBS droplets to the PBS-exposed devices and returned the pieces to their humidified chambers. Air-control pieces returned directly to their dry chambers.

2.6 Electrical characterization

Electrical characterization was performed using a Keithley 4200A-SCS semiconductor parameter analyzer with Signatone S-926 XYZ micropositioners and Signatone probe tips. The p++ Si substrate served as the common back gate. We connected source-measurement unit-2 (SMU2) to the back gate, SMU1 to one Ti/Au source/drain electrode, and the opposing Ti/Au electrode to the ground unit (GNDU) at 0 V. The GNDU-connected electrode served as the source reference and the SMU1-connected electrode as the drain. Once assigned, we maintained the same physical source/drain orientation for each device throughout the experiment.

This thesis denotes the current through the drain-to-source conduction path as I_{DS} , consistent with the notation used in the aptamer-FET literature. Gate leakage current is denoted I_G . The I_G represents the current measured at the back-gate electrode through the gate path toward the transistor structure; the measurement configuration did not separately resolve gate-to-source or gate-to-drain leakage components. We recorded I_{DS} and I_G simultaneously during transfer-curve characterization to interpret drain-to-source conduction together with the corresponding gate leakage.

We collected all electrical measurements after the exposure, rinsing, drying, thermal conditioning, and cooling sequence described in Section 2.5, with PBS absent from the device surface during characterization. We measured the devices using the Si/SiO₂ back gate rather than

an electrolyte gate. In the Andrews-group cortisol platform, the In_2O_3 FET is gated through an Ag/AgCl reference electrode in solution during sensing (Wang et al., 2022). The measurements here were used to determine the electrical stability of FETs after environmental exposure.

We collected transfer characteristics at each electrical-characterization timepoint. We collected output characteristics at selected timepoints to assess gate-dependent drain-to-source conduction and whether the output behavior supported operation in the saturation regime. Transfer curves described I_{DS} as a function of gate-source voltage (V_{GS}) at a fixed drain-to-source voltage (V_{DS}). We used these curves to evaluate gate modulation and extract the electrical metrics described in Section 2.7. We used the output curves to determine I_{DS} as a function of V_{DS} at several fixed V_{GS} values. Thus, the output curves showed whether drain-to-source conduction remained gate dependent and whether recognizable linear and saturation-like behaviors were retained.

In a conventional FET output characteristic, I_{DS} initially increases with V_{DS} in the approximately linear regime. As the channel approaches pinch-off near the drain, I_{DS} becomes progressively less dependent on further increases in V_{DS} and approaches current saturation (Joo et al., 2019; He et al., 2021). For this thesis, an output characteristic was classified as saturation-qualified when qualitative review of the gate-dependent output family showed recognizable saturation-like output behavior at or near $V_{\text{DS}} = 5$ V over the gate-bias range relevant to the corresponding transfer measurement. Saturation qualification was a study-specific qualitative eligibility criterion and not a separately fitted electrical parameter. It was used only to determine whether the saturation-regime square-root I_{DS} method could be used as a supporting threshold-voltage extraction. Related studies of solution-processed In_2O_3 use both transfer and output characteristics to establish FET behavior (Kim et al., 2015; He et al., 2021).

We recorded gate current (I_G) during every transfer measurement because abnormal I_G can interfere with the source/drain response being interpreted as channel conduction. Lee *et al.* demonstrated this effect directly in oxide TFTs, where abnormal I_G altered the measured source and drain-to-source currents and could progress to gate-to-source/drain short-like behavior (Lee et al., 2018).

We collected transfer characteristics at $V_{DS} = 5$ V while sweeping V_{GS} from -30 to $+30$ V in 0.5 V increments. We selected a 5 V drain bias during preliminary characterization because lower V_{DS} values did not consistently produce enough current to meet a 10 μ A constant-current threshold. The 5 V V_{DS} produced a reproducible gate-modulated response across the devices used in this study. Increasing V_{DS} further increased current but did not produce a comparably large displacement of the transfer response in the preliminary measurements. Published studies have used different drain biases depending on device architecture and measurement purpose. For example, He *et al.* characterized undoped In_2O_3 TFTs at $V_{DS} = 1.1$ or 15.1 V, sweeping V_{GS} from -30 to $+30$ V (He et al., 2021). Zhu *et al.* used a lower drain bias in their back-gated aqueous devices (Zhu et al., 2021). We treated 5 V as the fixed drain bias appropriate for the present devices and analysis.

The fixed -30 to $+30$ -V gate range was retained from the preliminary pilot study because it captured the transfer response while limiting repeated gate-voltage excursion. The same range was used at every formal timepoint. Keeping the gate range and sweep settings fixed was necessary because measured hysteresis depends on the gate-sweep waveform and timing, and prolonged gate bias can itself shift oxide-TFT electrical characteristics (Chen & Tai, 2015; Yao et al., 2021). A wider gate range was not introduced into the formal measurement solely to provide additional headroom.

When the transfer transition moved outside the formal -30 to $+30$ V window, a wider diagnostic sweep was collected separately to determine whether gate modulation remained present. Diagnostic sweeps were retained as qualitative evidence of strongly shifted but continuing FET behavior. They were not substituted for the standardized transfer sweep when calculating quantities that depend on the measurement window, including hysteresis and on/off ratio.

Output characteristics were collected with V_{GS} stepped from 0 to 30 V in 5-V increments while V_{DS} was swept from 0 to 15 V in 0.5 V increments. The output family was used to assess whether increasing positive gate bias continued to modulate drain-to-source conduction and whether the output behavior showed saturation-like behavior. Related solution-processed In_2O_3 studies have used comparable gate-voltage stepping for output characterization, although the drain-voltage range depends on device architecture (He et al., 2021).

At each timepoint, the forward transfer sweep was collected first, immediately followed by the reverse sweep. The output curve was taken at selected timepoints. No intentional dwell was introduced between forward and reverse sweeps. Transfer measurements used a programmed source-to-measure delay of 0.02 s per gate-voltage step. The gate-voltage range, 0.5 V step size, sweep order, and programmed delay were held constant across measurements because oxide-TFT hysteresis is sensitive to sweep timing (Chen & Tai, 2015).

During transfer measurements, current compliance was set to 0.001 A on SMU1 and 0.011 A on SMU2. During output measurements, compliance was set to 0.07 A on both active SMUs. If a formal trace reached compliance, the compliance-limited trace was retained and flagged rather than replaced by a repeat acquired under different settings.

Measurements were performed at laboratory temperature, approximately 25 °C, and a typical laboratory relative humidity of approximately 67-70%. Devices were measured under ordinary room illumination without directed task lighting. Illumination was kept as consistent as practical because the electrical behavior of solution-processed In_2O_3 TFTs can depend on illumination and bias history (He et al., 2021).

2.7 Electrical metrics and extraction

Electrical response was evaluated using threshold voltage (V_{th}), threshold-voltage change (ΔV_{th}), signed transfer hysteresis (ΔV_{H}), fixed-bias drain-to-source current magnitude ($|I_{\text{DS}}|$), subthreshold swing (SS), on/off current ratio ($I_{\text{on}}/I_{\text{off}}$), and gate leakage current (I_{G}). These metrics describe different aspects of FET operation and are interpreted together rather than as interchangeable measures of device stability. V_{th} , SS, hysteresis, and $I_{\text{on}}/I_{\text{off}}$ are established parameters in solution-processed In_2O_3 TFT characterization and reliability studies (Kyndiah et al., 2018; Yao et al., 2021).

The primary threshold metric was a constant-current threshold extracted separately from the forward and reverse transfer sweeps. $V_{\text{th},10\mu\text{A}}$ was defined as the value of V_{GS} at which $|I_{\text{DS}}| = 10 \mu\text{A}$. Constant-current threshold extraction defines V_{th} from the gate voltage corresponding to a specified I_{DS} criterion; the general method is established in transistor characterization, although the 10 μA criterion used here was selected specifically for the current range of the present FETs (Ortiz-Conde et al., 2002).

Because V_{GS} was sampled in 0.5 V increments, $V_{\text{th},10\mu\text{A}}$ was calculated by linear interpolation between the two adjacent measured points bracketing the 10 μA crossing. A value was reported only when a unique and interpretable crossing was present within the off-to-on

transition. Threshold voltage was recorded as unextractable when the formal sweep did not cross 10 μA , when multiple crossings prevented assignment of a unique value, or when the curve shape prevented the crossing from being interpreted as a valid drain-to-source channel response. No alternative current criterion, sweep-boundary value, or imputed threshold was substituted.

Gate leakage was evaluated locally at the threshold crossing. $V_{\text{th},10\mu\text{A}}$ was retained when $|I_G| < |I_{\text{DS}}|$ at both measured points used to bracket the 10 μA crossing. This criterion prevented a nominal drain-to-source current crossing from being treated as a valid channel threshold when gate leakage was equal to or larger than the current being interpreted.

Threshold-voltage shift was calculated independently for the forward and reverse sweeps relative to the condition-specific pre-exposure baseline:

$$\Delta V_{\text{th}}(t) = V_{\text{th}}(t) - V_{\text{th}}(T_0)$$

For the Chapter 4 surface-processing analysis, the corresponding response was calculated relative to the electrical state measured immediately before the assigned processing sequence:

$$\Delta V_{\text{th,proc}} = V_{\text{th,POST}} - V_{\text{th,PRE}}$$

Threshold-voltage displacement is widely used in oxide-TFT reliability studies because a transfer curve can remain measurable even after its gate-voltage position has shifted substantially (Yao et al., 2021).

A secondary threshold estimate was obtained using square-root extrapolation when the transfer and output measurements supported the saturation approximation. Under the conventional square-law form:

$$|I_{\text{DS}}| = [(W\mu\text{Cox})/2L](V_{\text{GS}} - V_{\text{th}})^2$$

Under this approximation, V_{th} was estimated from the V_{GS} -axis intercept of a linear fit to $\sqrt{|I_{\text{DS}}|}$ versus V_{GS} (Ortiz-Conde et al., 2002). Candidate fitting regions began at or above the valid

forward 10 μA threshold and did not extend beyond the highest V_{GS} for which the corresponding output characteristic supported saturation behavior near $V_{\text{DS}} = 5 \text{ V}$. A candidate region required at least five consecutive points and $R^2 \geq 0.95$. When more than one region satisfied these criteria, the longest qualifying region was selected; R^2 was used as the tie-breaker when regions had the same length. If the transfer-fit or output-behavior requirement was not met, square-root V_{th} was not reported. Because $V_{\text{DS}} = 5 \text{ V}$ did not consistently support the saturation approximation for all FETs, square-root V_{th} was treated as a secondary threshold metric rather than as the primary longitudinal endpoint. Ortiz-Conde et al. supports the general saturation-region $\sqrt{|I_{\text{DS}}|}$ extrapolation method; the specific eligibility rules above are study-specific.

Transfer hysteresis was calculated from the forward and reverse constant-current thresholds:

$$\Delta V_{\text{H}} = V_{\text{th,reverse}} - V_{\text{th,forward}}$$

The signed value was retained so that both the magnitude and direction of the forward-to-reverse displacement remain visible. Because measured hysteresis depends on gate-sweep waveform and timing, the formal gate range, step size, forward/reverse order, and programmed measurement delay were held constant across measurements (Chen & Tai, 2015). Hysteresis calculated from wider diagnostic sweeps was not pooled with the formal measurement.

Fixed-bias $|I_{\text{DS}}|$ was evaluated as $|I_{\text{DS}}|$ at $V_{\text{GS}} = +30 \text{ V}$ and $V_{\text{DS}} = 5 \text{ V}$. This quantity was treated as a fixed-bias $|I_{\text{DS}}|$ rather than as a guaranteed fully on-state current because a shift in the transfer characteristic could place a FET in a different operating region at the same V_{GS} . A fixed-bias $|I_{\text{DS}}|$ value was retained for channel-response analysis only when $|I_{\text{G}}| < |I_{\text{DS}}|$ at $+30 \text{ V}$.

Subthreshold swing was calculated from the inverse slope of the logarithmic transfer response:

$$SS = [(\partial \log_{10}|I_{DS}|) / \partial V_{GS}]^{-1}$$

SS was extracted from the steepest approximately linear region of $\log_{10}|I_{DS}|$ versus V_{GS} within the subthreshold region. This approach is consistent with conventional TFT definitions of subthreshold swing (Shang et al., 2019). For this study, a valid fit required at least five consecutive measured points, a span of at least one decade in $|I_{DS}|$, and $R^2 \geq 0.95$. Among qualifying windows, the window with the steepest slope was selected. If no window satisfied these criteria, SS was recorded as unextractable.

Gate leakage within the selected SS window was evaluated as a separate quality-control variable rather than as a reason to remove an otherwise valid numerical SS value. An SS measurement was leakage-flagged when $|I_G| \geq |I_{DS}|$ at any point within the selected fitting window. The numerical SS value was retained, while the flag indicated that gate current could contribute to interpretation of the fitted subthreshold response.

The on/off current ratio was calculated from the maximum and minimum I_{DS} magnitudes measured within the fixed formal transfer sweep:

$$I_{on}/I_{off} = \max|I_{DS}| / \min|I_{DS}|$$

Using the maximum-to-minimum current ratio is a common TFT convention for extracting I_{on}/I_{off} from a transfer characteristic (Petti et al., 2016). It was used here instead of assigning fixed nominal “on” and “off” gate voltages because the transfer response could shift substantially along the gate-voltage axis during processing or environmental exposure. Because off-state current was frequently noisy, the maximum and minimum were determined from three-point-averaged current values rather than individual raw extrema. Values from wider diagnostic sweeps were not substituted into the formal I_{on}/I_{off} calculation.

The numerical $I_{\text{on}}/I_{\text{off}}$ ratio was retained whenever the formal transfer measurement was available. A separate leakage flag identified cases in which $|I_G| \geq |I_{\text{DS}}|$ at a point contributing to the three-point-averaged maximum or minimum used for the ratio. This distinction was necessary because $|I_{\text{DS}}|$ can become very small in the off-state region, causing the relative gate-current contribution to become large even when absolute I_G remains modest. The numerical ratio was preserved, and the flag was treated as interpretive caution regarding possible leakage contribution to the measured current range.

Gate current was recorded throughout every transfer characteristic. Absolute $|I_G|$, maximum forward-sweep $|I_G|$, and the relative gate-current ratio

$$R_{\text{IG}} = |I_G| / |I_{\text{DS}}|$$

were retained as separate electrical and quality-control quantities. No universal R_{IG} cutoff from another transistor architecture was imposed as a general device-failure criterion. For study-specific interpretation, $R_{\text{IG}} \leq 0.10$ was treated as below warning level, $0.10 < R_{\text{IG}} < 1$ as elevated relative gate current, and $R_{\text{IG}} \geq 1$ as a gate-current-dominated measurement region. These boundaries were not treated as universal FET acceptance criteria.

Both absolute I_G and its magnitude relative to I_{DS} were retained because a developing gate-current pathway can interfere with the interpretation of measured I_{DS} (Lee et al., 2018).

2.8 Operational-state and failure definitions

Measurable current alone was not sufficient to determine whether FET electrical behavior remained interpretable because different components of FET operation could deteriorate at different times. Oxide TFTs may continue to carry measurable current despite abnormal transfer characteristics, reduced field-effect modulation, increased gate leakage, or abnormal drain-to-

source behavior (Sowade et al., 2016; Lee et al., 2018). Aqueous FET studies have similarly treated leakage, shorting, and loss of gate modulation as distinct operational-stability problems rather than defining stability solely by the presence or absence of measurable current (Albarghouthi et al., 2022).

The present study followed several electrical-state features separately: transfer modulation, constant-current threshold extractability, square-root threshold eligibility, output behavior, gate leakage, and the availability of individual numerical metrics. A FET could deteriorate in one feature while remaining informative in another. For example, a FET could have a reproducible gate-dependent transfer response after losing saturation-like output, or could retain a numerical $I_{\text{on}}/I_{\text{off}}$ value while the off-state region was leakage-flagged.

Transfer behavior was considered electrically interpretable when the formal sweep retained a recognizable and reproducible gate-dependent response and the measured currents could still be meaningfully evaluated together with the simultaneously recorded gate current. Loss of a constant-current threshold alone was not treated as equivalent to loss of gate control. A threshold could become unextractable because the transition shifted outside the fixed -30 to $+30$ V formal window, because a unique $10\text{-}\mu\text{A}$ crossing was absent, or because the local crossing was leakage-dominated. When needed, a wider diagnostic sweep could be used to distinguish an out-of-window shift from complete loss of gate modulation, but diagnostic values were not substituted into the formal metric analysis.

Output behavior was evaluated separately from transfer behavior. Output characteristics were classified according to whether drain-to-source conduction remained gate-dependent and whether the recognizable saturation-like behavior was present. Loss of saturation-qualified output behavior prevented use of the square-root threshold method when its saturation

assumption was not supported, but did not automatically invalidate the formal constant-current threshold or end longitudinal measurement.

Gate leakage was also treated as a separate operational dimension. Constant-current V_{th} required $R_{IG} < 1$ at the points bracketing the 10- μ A crossing, and fixed-bias $|I_{DS}|$ required $R_{IG} < 1$ at $V_{GS} = +30$ V. In contrast, numerical SS and I_{on}/I_{off} values were retained when their numerical extraction criteria were satisfied even if the relevant region was leakage-flagged. For SS, the flag indicated that $|I_G| \geq |I_{DS}|$ occurred somewhere within the selected fitting window. For I_{on}/I_{off} , the flag indicated that this condition occurred at a point contributing to the smoothed maximum or minimum used for the ratio. These flags identified possible leakage influence without converting every locally leakage-dominated off-state measurement into a missing numerical value.

The primary operational failure endpoint was loss of reproducible off-to-on gate modulation across the formal transfer range after an obvious probe-contact problem had been checked. Loss of gate-dependent output behavior, failure to meet the saturation-qualified criterion, failure of square-root threshold eligibility, loss of a unique 10- μ A threshold, increased gate current, or leakage flags on SS or I_{on}/I_{off} could occur before this endpoint and were recorded separately. These states represent different forms of electrical deterioration rather than a single binary definition of failure.

The first observed loss of an electrically interpretable FET response and the first observed loss of reproducible gate modulation were recorded separately. If a device showed an abnormal state at one timepoint but later recovered a previously absent electrical behavior, both observations were retained. Missing, unextractable, or otherwise invalid metric values were not replaced with sweep-boundary values, earlier measurements, or interpolated estimates.

2.9 Planned study schedule and completed observation interval

The PRE/POST surface-preparation measurements described in Sections 2.2 and 2.3 were completed before environmental exposure began and were analyzed separately in Chapter 4. For surface-prepared FETs, PRE and POST measurements were collected before a separate pre-exposure T0 measurement. Bare FETs did not undergo a study-specific surface treatment. Therefore, their T0 was the corresponding pre-exposure measurement collected after the common conditioning described in Section 2.1.

The primary experiment was designed to evaluate the electrical response of the four surface conditions under ambient-air and PBS exposure from T0 through Day 14. Transfer characteristics were planned at T0, 6 h, 12 h, 24 h, 48 h, Day 3, Day 5, Day 7, Day 10, and Day 14. The early measurement schedule was intentionally denser because the preliminary bare-device experiments showed substantial electrical change during the first one to two days of PBS exposure. The later schedule extended the planned observation period beyond the seven-day PBS exposure reported for related In_2O_3 nanoribbon FETs by Liu et al. (2020) and the Day 3, Day 6, and Day 9 measurements reported for the back-gated In_2O_3 FETs of Zhu et al. (2021).

Output characteristics were scheduled less frequently than transfer measurements in the primary experiment. Routine output measurements were planned at 24 h, 48 h, Day 7, Day 10, and Day 14, with an additional output measurement to be collected if a transfer response suggested possible operational failure before one of those scheduled timepoints.

Valid measurements using the original probe-station back-gate configuration were obtained at T0, 6 h, and 12 h. During the scheduled 24 h characterization, the measurement system began producing flat transfer responses. Previously functioning FETs outside the formal cohort were tested using the same configuration and produced the same behavior, indicating

failure of the measurement path rather than simultaneous loss of gate modulation across the experimental cohort.

The 24 h handling cycle had already begun when the measurement-system problem was identified. Because thermal conditioning had already been applied to part of the cohort, the 70 °C, 10 min treatment was completed across the remaining study arms to preserve the scheduled handling history at that timepoint. Environmental exposure then continued during the interruption in electrical characterization. At the 48 h, Day 3, and Day 5 maintenance points, the PBS workflow continued, including aspiration, rinsing, drying, and PBS renewal, but measurement-associated electrical sweeps and thermal conditioning were not performed.

At Day 7, electrical characterization resumed using a temporary alternative physical connection to the same scratched p⁺⁺ Si back-gate node. The source/drain connections, programmed transfer sweep, PBS handling, and thermal-conditioning procedure otherwise remained the same. However, the physical back-gate contact method had changed, and the gate-current signal obtained under the alternative configuration was not considered reliable for leakage analysis. Day 7 was treated as a separate exploratory observation rather than as a quantitatively continuous extension of the standardized T0-to-12 h trajectory.

The temporary gate connection was attempted again at Day 10. Although isolated measurements initially showed recognizable transfer behavior, flat or inconsistent responses subsequently recurred across experimental and previously functioning comparison devices. Day 10 did not provide an analyzable formal experimental timepoint.

The completed dataset was consequently divided into a standardized early phase and an exploratory later observation. T0, 6 h, and 12 h constitute the quantitative environmental-exposure phase obtained using the original back-gate configuration. Day 7 provides a separate

exploratory view of the electrical state after continued exposure under a different physical gate-contact configuration. No values were imputed for the unobserved 24 h through Day 5 interval or for the failed Day 10 measurement, and no continuous quantitative trajectory was fitted across the change in measurement configuration.

2.10 Statistical analysis

Statistical analyses were structured around the randomized complete-block design of the formal experiment and the nesting of individual FETs within wafer pieces. Three parent wafers were included, and each parent wafer contributed one wafer piece to every Surface $\times$ Exposure condition. Within parent wafer, surface and exposure assignments were made at the wafer-piece level. Parent wafer was treated as the fabrication block, and wafer piece was the independent treatment unit. Two FETs were followed on each wafer piece. These FETs were retained as device-level subsamples because they provided information about within-piece variability, but they were not treated as independent repetitions of the assigned surface or exposure condition. Treating both FETs on the same piece as independent treatment replicates would constitute pseudoreplication (Hurlbert, 1984; Lazic, 2010).

Parent wafer was included as a fixed blocking factor because each of the three fabrication wafers contributed material to every formal experimental condition. FET-level mixed-effects models included a random intercept for wafer piece to account for correlation between the two FETs sharing the same independently treated piece. Linear mixed-effects models were fitted by restricted maximum likelihood (REML), and Kenward-Roger small-sample inference was used for fixed-effect tests and model-based contrasts (Kenward & Roger, 1997).

2.10.1 Surface-processing analysis

The electrical response to surface processing was analyzed separately from the subsequent environmental-exposure experiment. The formal processing analysis contained the three treated sequences: DDT, PTMS + DDT, and UV + PTMS + DDT. Each processing sequence contained six independently processed wafer pieces: two pieces from each of the three parent wafers. Two FETs were measured on each piece, producing 12 FET observations per processing sequence and 36 FET observations across 18 independently processed pieces. The future Air and PBS assignments were retained in the identifiers for traceability but were not treated as an exposure factor because environmental exposure had not begun at either the PRE or POST processing measurement. Bare FETs were not included in this analysis because they did not undergo a corresponding study-specific processing event.

For each treated FET, forward and reverse threshold responses were calculated separately as: $\Delta V_{th,proc} = V_{th,POST} - V_{th,PRE}$,

and signed hysteresis response was calculated as: $\Delta V_{H,proc} = \Delta V_{H,POST} - \Delta V_{H,PRE}$.

For fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$, processing response was analyzed as:

$$\log_{10}(Y_{POST} / Y_{PRE})$$

when both values required for the numerical response were available and strictly positive. No arbitrary constant was added to accommodate unavailable, zero, nonpositive, or otherwise invalid values. Log-scale summaries and model estimates were back-transformed to POST/PRE ratios where useful for interpretation.

Metric validity and leakage quality control followed Section 2.7. Constant-current V_{th} required a valid threshold crossing and $R_{IG} < 1$ at the bracketing points. Hysteresis required valid forward and reverse thresholds. Fixed-bias $|I_{DS}|$ required $R_{IG} < 1$ at $V_{GS} = +30$ V. Numerical SS

and $I_{\text{on}}/I_{\text{off}}$ were retained when their numerical extraction criteria were satisfied, while the corresponding leakage flags were recorded separately. Square-root V_{th} was treated as a secondary outcome and was retained only when both the transfer-fit and output-behavior criteria were satisfied.

FET-level processing responses were analyzed using: Processing response $\sim$ Processing sequence + Parent wafer + (1 | Wafer piece).

Three pairwise comparisons among the treated processing sequences were included in the final analysis:

PTMS + DDT – DDT

UV + PTMS + DDT – PTMS + DDT

UV + PTMS + DDT – DDT.

The first two comparisons describe differences between adjacent stages of the progressive processing sequence, while the third directly compares the two endpoints of the treated sequence. Holm adjustment was applied across all three pairwise comparisons separately within each electrical response metric (Holm, 1979). Confidence intervals for the model-estimated contrasts are pointwise 95% confidence intervals and were not multiplicity-adjusted. The comparisons were interpreted as differences among complete realized processing histories rather than as chemically isolated effects of PTMS or UV.

Continuous processing responses were supplemented by descriptive operational and quality-control outcomes, including forward and reverse constant-current threshold validity, fixed-bias $|I_{\text{DS}}|$ validity, numerical SS availability, SS leakage flags, numerical $I_{\text{on}}/I_{\text{off}}$ availability, $I_{\text{on}}/I_{\text{off}}$ leakage flags, R_{IG} at +30 V, gate-dependent output behavior, saturation-qualified output status, and square-root V_{th} reportability. These outcomes were summarized descriptively because

the number of independently processed wafer pieces per sequence was too small to support stable binary-outcome mixed models.

2.10.2 Environmental-exposure analysis

The environmental analysis used the FET-specific pre-exposure T0 measurement as the reference state. T0, 6 h, and 12 h measurements obtained under the original back-gate configuration were retained as the standardized quantitative phase. Responses at 6 h and 12 h were analyzed separately rather than by fitting a continuous time slope.

The primary environmental outcome was forward-sweep constant-current threshold-voltage change at 12 h: $\Delta V_{th,F}(12\text{ h}) = V_{th,F}(12\text{ h}) - V_{th,F}(T0)$.

Forward threshold response at 6 h and the remaining electrical outcomes were treated as supporting analyses. Reverse threshold voltage was analyzed separately from the forward threshold, and signed hysteresis remained a derived response rather than treating the two sweep directions as replicate observations.

For voltage outcomes, environmental response was calculated as arithmetic change from T0. For valid positive fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ values, environmental response was calculated as: $\log_{10}(Y_t / Y_{T0})$.

Numerical SS and I_{on}/I_{off} remained available when their numerical extraction criteria were satisfied even when their corresponding leakage flags were present. Leakage status was analyzed and reported separately.

For each continuous outcome, 6 h and 12 h were modeled separately using FET-level linear mixed-effects models of the form: Response $\sim$ Surface + Exposure + Surface $\times$ Exposure + Parent wafer + (1 | Wafer piece).

Models were fitted by REML, and Kenward-Roger small-sample inference was used for fixed-effect tests, confidence intervals, and model-based contrasts.

Chamber was not included as an independent model factor because chamber identity was completely associated with the corresponding Surface $\times$ Exposure condition in the realized design. Chamber-to-chamber variation could not be estimated separately from the implemented experimental condition.

The three primary 12 h Surface $\times$ Exposure comparisons followed the progressive surface sequence:

[(PBS – Air)DDT – (PBS – Air)Bare]

[(PBS – Air)PTMS+DDT – (PBS – Air)DDT]

[(PBS – Air)UV+PTMS+DDT – (PBS – Air)PTMS+DDT].

These difference-in-differences contrasts tested whether each successive surface state changed the PBS-versus-Air response relative to the preceding state. Holm adjustment was applied across the three primary 12 h contrasts. PBS-versus-Air contrasts within individual surface states and the corresponding 6 h contrasts were treated as supporting comparisons. Inferential analyses of reverse V_{th} , hysteresis, fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ were supporting rather than additional members of the primary Holm-adjusted hypothesis family.

The PBS-versus-Air comparison represents the complete environmental workflows implemented in the study. PBS-exposed FETs experienced PBS exposure together with the corresponding aqueous aspiration, rinsing, N₂ drying, humidified-chamber storage, and PBS-renewal procedures. Air controls experienced the corresponding nonaqueous handling and measurement-associated thermal-conditioning workflow but did not undergo PBS-specific

aqueous handling. The experiment does not separately identify the causal contributions of PBS chemistry, chamber humidity, and PBS-specific handling.

Gate leakage was analyzed both as a continuous electrical response and as an operational quality-control dimension. Maximum forward-sweep $|I_G|$ and R_{IG} at $V_{GS} = +30$ V were retained where valid during the standardized T0-to-12 h phase. The complete R_{IG} -versus- V_{GS} response was retained for device-level interpretation. $R_{IG} \leq 0.10$, $0.10 < R_{IG} < 1$, and $R_{IG} \geq 1$ were interpreted using the study-specific categories defined in Section 2.7.

Output characteristics were not treated as uniformly sampled longitudinal outcomes because routine output measurements were collected only at selected timepoints and additional output curves could be triggered by questionable transfer behavior. Output measurements were used as operational and diagnostic evidence rather than entered as a regularly sampled repeated outcome. Square-root V_{th} was likewise treated as a conditional supporting metric because its reportability required a corresponding saturation-qualified output characteristic.

Unextractable, unavailable, or otherwise invalid values were not assigned sweep-boundary values, carried forward, interpolated, or imputed. Mixed-effects analyses retained any other valid FET observation from the same wafer piece. Reasons for metric unavailability were retained separately, including absence of a unique threshold crossing, local leakage domination of a threshold or fixed-bias $|I_{DS}|$ measurement, failure of the SS fitting criterion, loss of gate modulation, lack of square-root eligibility, or documented measurement-system failure.

2.10.3 Day 7 exploratory analysis

Day 7 was analyzed separately from the standardized T0-to-12 h phase because the physical gate-contact configuration had changed and all analyses for this section are considered

exploratory. For descriptive context, absolute Day 7 metrics were also displayed alongside the standardized early-phase summaries. These visual comparisons were exploratory only: Day 7 values were not analyzed as change from T0 or treated as a continuation of the T0–12 h trajectory.

Absolute Day 7 electrical metrics and operational-state counts were summarized descriptively. Where sufficient valid data were available, exploratory FET-level mixed-effects models used Surface, Exposure, Surface $\times$ Exposure, and parent wafer as fixed effects with wafer piece as a random intercept. The three sequential Surface $\times$ Exposure contrasts were examined for the exploratory Day 7 forward-threshold analysis with Holm adjustment across those three comparisons. These analyses were treated as exploratory and were not interpreted as a second confirmatory longitudinal test. Gate-current and R_{IG} outcomes were not analyzed at Day 7 because the gate-current channel under the temporary connection was not considered reliable for leakage interpretation.

Day 10 did not yield a valid formal experimental dataset and was not entered into a longitudinal or cross-sectional model. No Day 7-to-Day 10 change score was calculated.

2.10.4 Sensitivity analyses

Robustness of the primary 12 h forward-threshold result was evaluated using four sensitivity analyses. First, valid FET responses were averaged within each independently treated wafer piece, and the resulting piece-level responses were analyzed using a blocked Surface $\times$ Exposure model with parent wafer included as a fixed factor. HC3 heteroscedasticity-consistent confidence intervals were used for this treatment-unit-level sensitivity analysis (Long & Ervin, 2000).

Second, the primary mixed model was refitted after omitting one parent wafer at a time. These leave-one-wafer-out models were used as influence analyses to determine whether the estimated primary contrast depended disproportionately on a single fabrication wafer.

Third, the 12 h forward V_{th} value was modeled with the corresponding T0 forward V_{th} included as a baseline covariate rather than using the change score as the response. This analysis evaluated sensitivity to baseline imbalance and regression to the mean (Vickers & Altman, 2001).

Fourth, when a wafer piece contained only one FET with a valid primary 12 h response, the primary model was repeated using only wafer pieces for which both tracked FETs produced valid primary-outcome values.

Model diagnostics included residual-versus-fitted plots, normal Q-Q plots, convergence assessment, and evaluation of the wafer-piece random-effect fit. Observations were not removed solely because they were statistically extreme. Measurements that failed a predefined physical, extraction, or documented measurement-validity criterion were handled according to the rules described above. Any additional exclusion involving investigator judgment was reported explicitly and accompanied by a sensitivity analysis where appropriate.

Results were interpreted from the model-estimated effects and confidence intervals together with the direction and consistency of responses across parent wafers and the underlying FET- and wafer-piece-level data. Exact P values were reported as inferential quantities but were not used alone to determine scientific importance. A nonsignificant result was not interpreted as evidence that two conditions were equivalent.

The primary environmental conclusions are restricted to the standardized T0-to-12 h phase obtained using the original probe-station back-gate configuration. Day 7 provides a

separate exploratory assessment after continued exposure under the temporary gate-contact configuration. The missing intermediate measurements and unsuccessful Day 10 characterization are not used to imply an uninterrupted quantitative multi-day stability trajectory.

Chapter 3. Preliminary bare-device stability and protocol development

The study was originally designed as a comparison of bare In_2O_3 FETs under ambient-air and PBS exposure. While it was intended to be a longitudinal study, the PBS-exposed FETs developed substantial electrical changes over the first several days, and some measurements no longer supported an interpretable FET response. This study became a starting point for protocol development and eventual redesign of the main study.

Therefore, this chapter covers the bare-device pilot and then follows the protocol-development experiments that were motivated by it. First, electrical behavior of bare devices was examined under matched air and PBS exposure. The same exposure was then repeated on devices from additional wafers from different fabrication batches to determine whether the observed degradation was limited to the original pilot wafers. The results were considered alongside previous aqueous FET studies to identify differences in device architecture and surface protection that could matter for the present platform. Lastly, targeted protocol experiments evaluated two problems that emerged directly from the pilot: recovery of a usable electrical state after PBS exposure and evaporation of the PBS droplet. These experiments establish the thermal-conditioning and humidity-control procedures that were carried forward into the main study.

3.1 Preliminary bare-device study design and analysis

The preliminary study was designed to follow untreated In_2O_3 FETs under ambient-air and PBS exposure before any surface-preparation chemistry was introduced. Two parent wafers from the same fabrication batch were used. Each parent wafer was divided into four pieces, with two pieces assigned to ambient air and two pieces assigned to PBS exposure (see Figure 3.1).

Two FETs were tracked on each wafer piece. Each exposure condition contained eight devices distributed across four wafer pieces from two parent wafers.

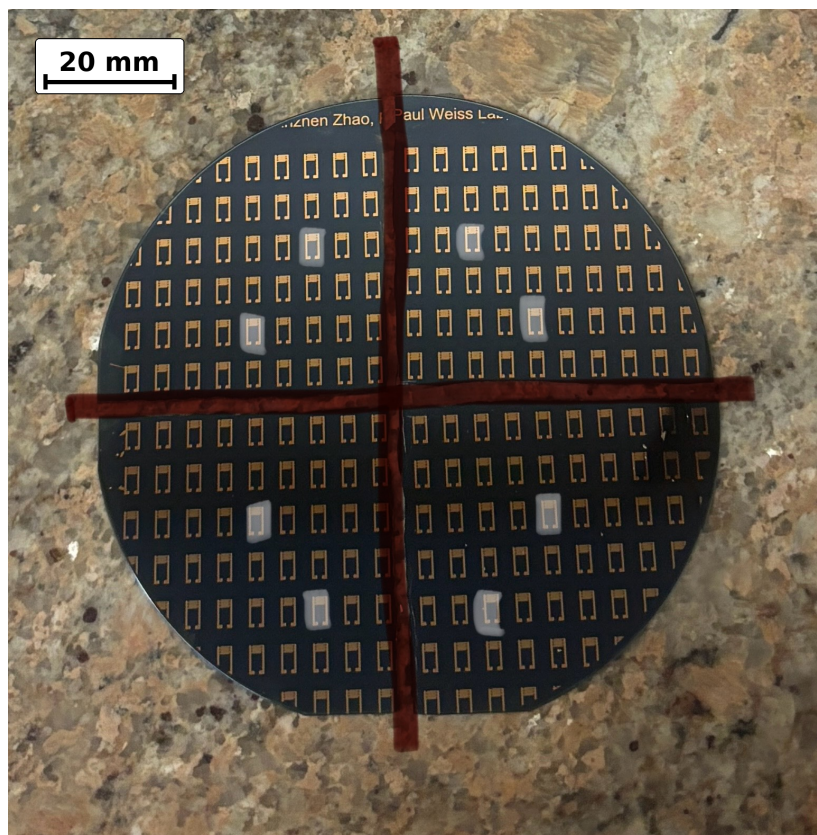


Figure 3.1. Wafer subdivision and exposure allocation for the preliminary bare-device pilot. Two parent wafers were each divided into four pieces. Two FETs were tracked on each piece, as highlighted in white. Two pieces from each parent wafer were assigned to ambient air and two to PBS exposure, resulting in four independently exposed wafer pieces and eight tracked devices per exposure condition.

Electrical characterization was performed at T0, 3 h, 6 h, 12 h, 24 h, 48 h, Day 3, and Day 5. The T0 measurements were collected before the later post-exposure thermal-conditioning had been developed. For PBS exposure, 6 μ L of PBS was applied to the FET sensing region. The rinse, N₂-drying, and fresh-PBS application procedure described in Chapter 2 was used throughout the pilot. The humidified exposure chamber used in the primary experiment had not yet been introduced.

At the first post-exposure measurement (3 h), rinsing and N₂ drying alone did not consistently return the PBS-exposed devices to an electrically readable dry state. A 70 °C, 10 min hotplate treatment was introduced before electrical characterization and retained as a provisional drying step for the remainder of the pilot for devices in both conditions. This condition was adopted during the pilot experiment. Because it was not a previously validated recovery protocol for the present In₂O₃ devices, the need to determine whether this thermal step was necessary emerged. The question of whether a lower temperature could provide the same recovery more consistently is what motivated the thermal-conditioning experiment described later in this chapter.

The pilot was evaluated using electrical metrics and validity rules defined in Chapter 2. Threshold voltage, threshold-voltage shift, signed hysteresis, fixed-bias $|I_{DS}|$, subthreshold swing, on/off ratio, and gate leakage were retained when their extraction criteria were satisfied. Transfer modulation, output behavior, threshold extractability, and leakage status were also followed because these features did not necessarily deteriorate at the same time. The first loss of an electrically interpretable FET response and the first loss of reproducible gate modulation were recorded separately. If a FET later recovered a previously lost electrical behavior, both observations were retained.

3.2 Electrical behavior during the five-day bare-device pilot

All 16 FETs selected for the preliminary study were electrically interpretable at T₀, and all 16 also met the saturation-qualified output criterion. The two exposure groups began to separate as the experiment continued, although the changes were not monotonic and did not occur as a single binary failure event. At 3 h, 7/8 devices in air and 6/8 devices in PBS groups

remained electrically interpretable. The air group returned to 8/8 electrically interpretable devices at 6 h and 12 h, then retained 7/8 through Day 5. In the PBS group, 6/8 devices were electrically interpretable at 6 and 12 h, decreasing to 5/8 at 24 h and 4/8 at 48 h. Five of eight PBS devices were electrically interpretable at Day 3 and Day 5. However, 4/8 PBS devices had saturation-qualified output behavior at 24 h, 3/8 at 48 h and Day 3, and 0/8 at Day 5, compared with 6/8 air devices at Day 5 (Table 3.1).

Continuous electrical metrics changed alongside these operational states. Among PBS-exposed FETs with valid paired measurements, mean forward threshold-voltage shift became increasingly negative after the first day, changing by -7.79 ± 7.42 V at 24 h, -11.69 ± 9.18 V at 48 h, -15.78 ± 10.2 V at Day 3, and -17.48 ± 11.0 V at Day 5. Corresponding air-device shifts were smaller overall, including -1.62 ± 0.72 V at 24 h, -0.57 ± 1.94 V at 48 h, and -1.42 ± 1.41 V at Day 3. Hysteresis also increased in the PBS cohort among devices for which both directional thresholds remained extractable. At 24 h, the mean change in signed hysteresis was $+7.60 \pm 4.35$ V in PBS compared with $+0.34 \pm 1.20$ V in air; at 48 h, the corresponding changes were $+11.83 \pm 7.16$ V and -0.04 ± 0.71 V. As electrical degradation progressed, fewer PBS-exposed FETs retained valid reverse-threshold and hysteresis measurements, so the later metric summaries describe the subset of devices for which those quantities remained extractable rather than the complete original cohort (Table 3.1).

Table 3.1. Electrical-state retention and paired threshold changes during the original five-day bare-device pilot.

Timepoint	Exposure	Electrically interpretable, <i>N</i> /8	Saturation-qualified, <i>N</i> /8	Δ forward V_{th} (V)	Δ signed hysteresis (V)
T0	Air	8/8	8/8	0.00 ± 0.00 (n=8)	0.00 ± 0.00 (n=8)
T0	PBS	8/8	8/8	0.00 ± 0.00 (n=8)	0.00 ± 0.00 (n=8)
3 h	Air	7/8	7/8	-0.45 ± 0.88 (n=7)	-0.02 ± 0.64 (n=7)
3 h	PBS	6/8	6/8	-1.76 ± 3.22 (n=6)	$+5.26 \pm 4.33$ (n=6)
6 h	Air	8/8	8/8	-1.14 ± 0.95 (n=7)	$+0.01 \pm 0.25$ (n=7)
6 h	PBS	6/8	6/8	-0.50 ± 4.85 (n=6)	$+3.33 \pm 3.51$ (n=6)
12 h	Air	8/8	8/8	-1.02 ± 0.66 (n=7)	-0.01 ± 0.67 (n=7)
12 h	PBS	6/8	6/8	-2.06 ± 6.14 (n=6)	$+5.57 \pm 5.11$ (n=6)
24 h	Air	7/8	7/8	-1.62 ± 0.72 (n=7)	$+0.34 \pm 1.20$ (n=7)
24 h	PBS	5/8	4/8	-7.79 ± 7.42 (n=5)	$+7.60 \pm 4.35$ (n=5)
48 h	Air	7/8	7/8	-0.57 ± 1.94 (n=7)	-0.04 ± 0.71 (n=7)
48 h	PBS	4/8	3/8	-11.69 ± 9.18 (n=4)	$+11.83 \pm 7.16$ (n=4)
Day 3	Air	7/8	7/8	-1.42 ± 1.41 (n=7)	-0.28 ± 0.77 (n=7)
Day 3	PBS	5/8	3/8	-15.78 ± 10.20 (n=5)	$+5.06 \pm 13.10$ (n=5)
Day 5	Air	7/8	6/8	-0.89 ± 5.50 (n=7)	-0.80 ± 0.79 (n=7)
Day 5	PBS	5/8	0/8	-17.48 ± 11.00 (n=5)	$+10.38 \pm 6.35$ (n=2)

Note. Electrically interpretable and saturation-qualified FET counts are reported separately. Threshold-voltage and hysteresis changes were calculated within each FET relative to its own T0 measurement before cohort summarization and are reported as mean $\pm$ SD. The metric-specific number of valid paired measurements is shown in parentheses. “Electrically interpretable” indicates that the available transfer and leakage behavior remained sufficient for device-level electrical interpretation under the criteria defined in Chapter 2. “Saturation-qualified” indicates that the output behavior supported the study-specific saturation QC criterion. Missing or unextractable values were not imputed.

Supporting metrics showed the same broader loss of electrically interpretable FET behavior. The $I_{\text{on}}/I_{\text{off}}$ ratio decreased substantially among the remaining interpretable PBS devices, while subthreshold swing generally increased. In contrast, neither maximum gate-current magnitude nor $|I_{\text{G}}| / |I_{\text{DS}}|$ at $V_{\text{GS}} = +30$ V showed a corresponding monotonic cohort-wide increase. The degradation could not be reduced to a single gate-leakage trajectory. Complete device-level metrics are reported in Appendix Table B.1.1. Additional pilot metrics, quality-control records, and longitudinal summary figures are retained in Appendix B.1.

The raw transfer characteristics show this progression directly. Air Device 8 retained a recognizable gate-modulated transfer response across the five-day observation period, with comparatively limited displacement in its forward and reverse characteristics (Figure 3.2A-C). PBS Device 5 began with a similarly interpretable FET response but developed progressively larger changes in transfer position and shape (Figure 3.2D-F). By Day 5, gate modulation remained detectable, but the FET no longer retained the output behavior required for the saturation-qualified state. This distinction between continued gate modulation and loss of saturation-like output behavior was also present in the cohort-level classification: five PBS FETs remained electrically interpretable at Day 5, while none remained saturation-qualified. Complete longitudinal device records are provided in Supplemental Data S1.

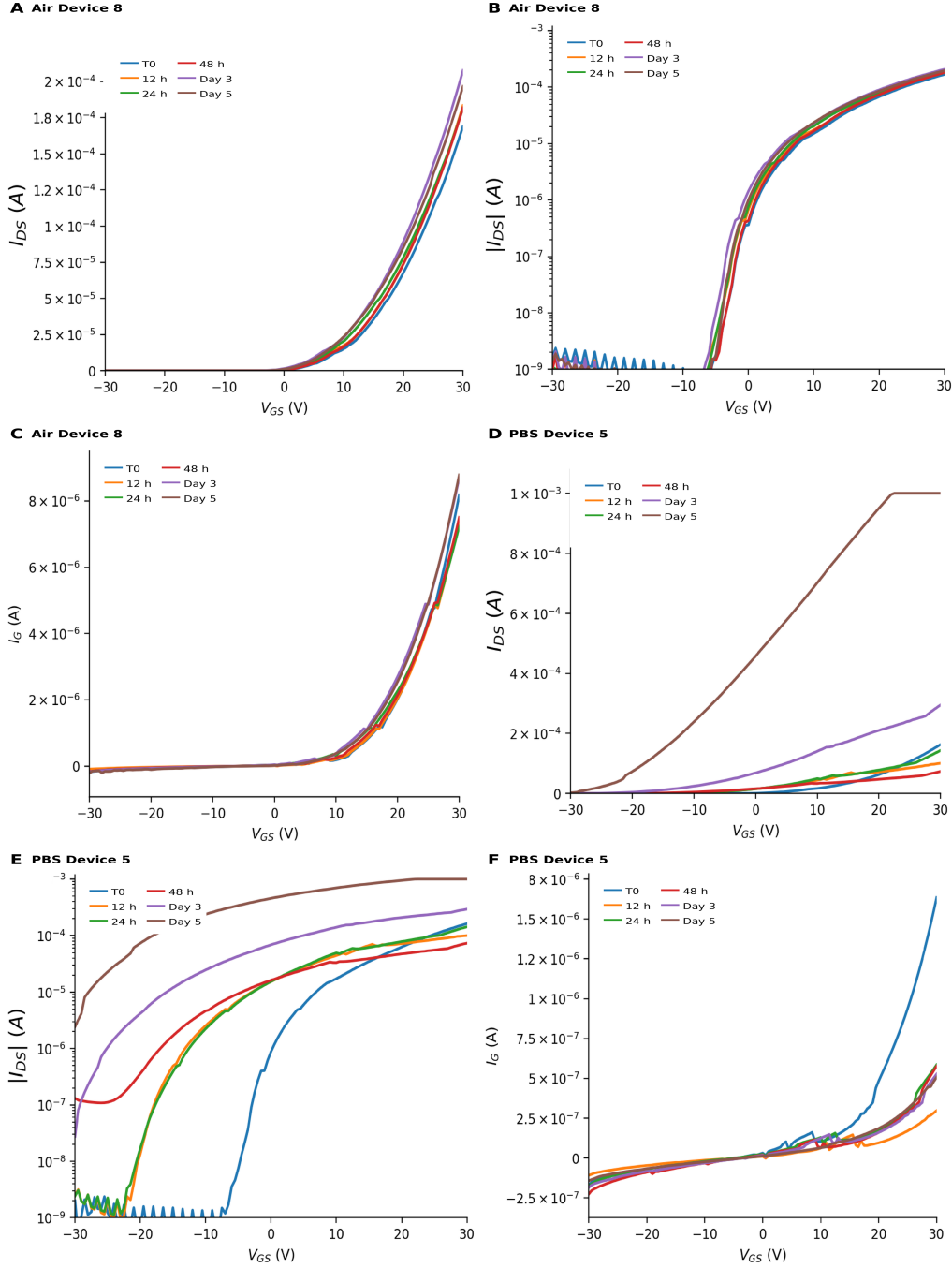


Figure 3.2. Example longitudinal transfer characteristics of bare In_2O_3 TFTs under air and PBS exposure. Example transfer measurements are shown for Air Device 8 (A-C) and PBS Device 5 (D-F) over the five-day pilot. (A,D) Drain-to-source current (I_{DS}) as a function of gate-source voltage (V_{GS}) on a linear current scale. (B,E) Corresponding $|I_{DS}|$ characteristics on a logarithmic scale. (C,F) Gate current I_G recorded during the same transfer measurements. Transfer characteristics were collected at $V_{DS} = 5$ V using the standardized gate-voltage sweep. Air Device 8 retained recognizable gate modulation throughout the observation interval, whereas PBS Device 5 developed substantial displacement and distortion of its transfer response. Complete longitudinal curves for all pilot devices are provided in Supplemental Data S1. Colors identify measurement time consistently across panels: T0 is blue, 12 h orange, 24 h green, 48 h red, Day 3 purple, and Day 5 brown.

A more abrupt electrical transition was observed within individual measurement sessions. For PBS Device 2 at 3 h, the first output measurement showed a recognizable gate-dependent output family. The transfer measurement collected immediately afterward was strongly abnormal, with I_{DS} and I_G becoming comparable over portions of the sweep. When the output curve was repeated, the original behavior was no longer retained (Figure 3.3). Similar output-transfer-output sequences were observed for PBS Device 1 at 48 h and PBS Device 4 at Day 3 (Appendix Figure B.1.1). In the latter sequences, the transfer measurements also contained large, sign-changing currents and the repeated output characteristics were substantially different from the initial output families.

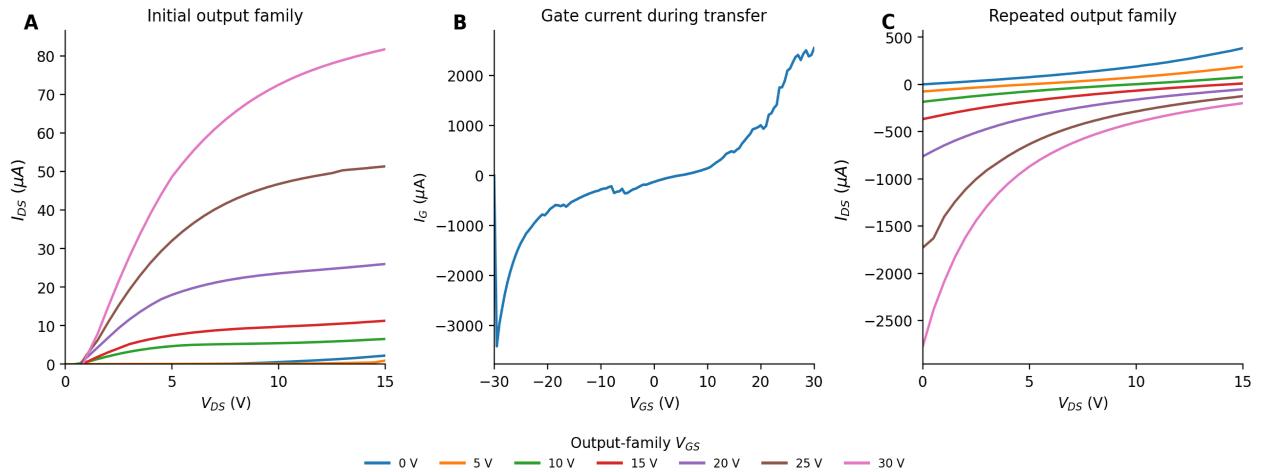


Figure 3.3. Rapid change in electrical behavior during sequential characterization of PBS Device 2 at 3 h. (A) The initial output measurement showed a recognizable gate-dependent output family. (B) The transfer measurement collected immediately afterward was strongly abnormal, with drain-to-source and gate currents becoming comparable over portions of the gate-voltage sweep. (C) A repeated output measurement collected after the transfer sweep no longer retained the behavior observed in the initial output family. The sequence documents rapid within-session change in electrical behavior but does not establish the physical origin of the abnormal current pathway. In the output families (A,C), colors denote gate-source voltage: 0 V blue, 5 V orange, 10 V green, 15 V red, 20 V purple, 25 V brown, and 30 V pink. In the transfer measurement (B), the blue solid trace is drain-to-source current and the orange dashed trace is gate current.

These measurements are described as short-like electrical behavior, but not as confirmed physical short circuits. Lee et al. (2018) showed in a different oxide-TFT architecture that sufficiently large gate-current stress could produce SiO₂ dielectric breakdown and an unintended gate-to-electrode conduction pathway (Lee et al., 2018). The present FETs showed features consistent with an unintended current pathway, including I_G approaching I_{DS} and substantial changes in repeated output families, but the electrical measurements do not identify the location or physical origin of that pathway. The behavior is interpreted operationally rather than assigned to a specific gate-to-drain, gate-to-source, contact, dielectric, or channel mechanism.

The pilot also showed why a single abnormal curve could not automatically be classified as irreversible FET failure. At 6 h, PBS Device 4 produced an abnormal transfer response despite visible probe contact. The next saved transfer measurement at 12 h remained gate modulated but it did not reach the 10- μ A threshold criterion, and by Day 3 the FET again showed a strongly abnormal transfer response (Figure 3.4). This sequence motivated the later distinction between an obvious contact problem, loss of an electrically interpretable FET response, abnormal FET operation, and loss of gate modulation.

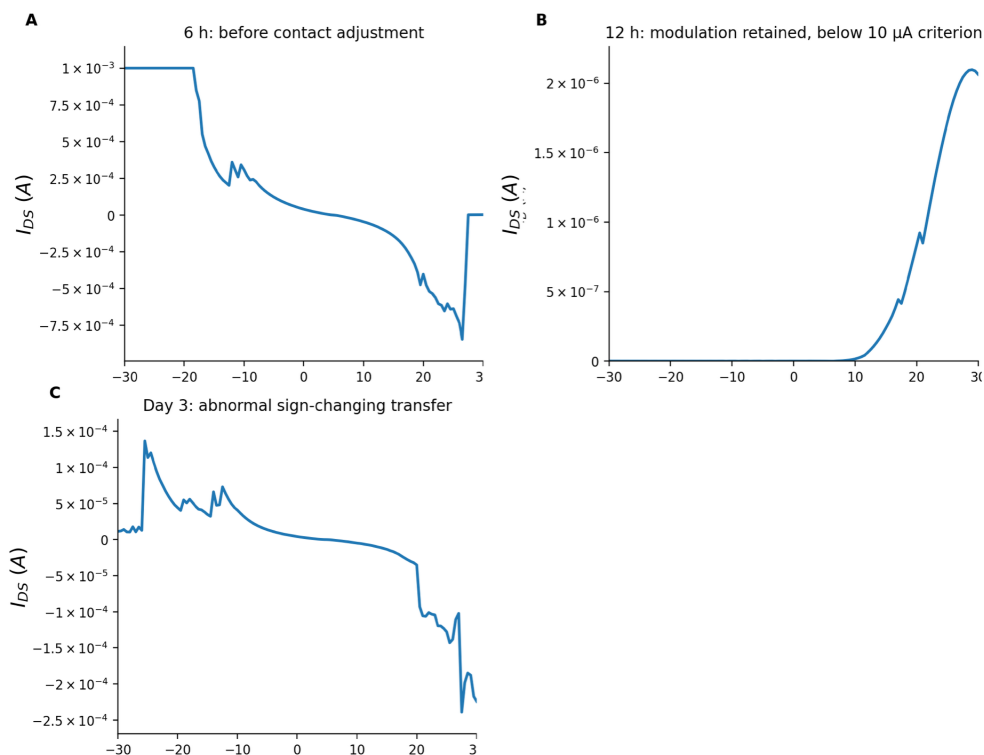


Figure 3.4. Contact troubleshooting and subsequent electrical degradation in PBS Device 4. (A) The 6-h forward-transfer measurement collected before adjustment of the back-gate contact showed an abnormal response. Adjustment of the contact restored gate-dependent behavior during the same measurement session, but the immediate post-adjustment transfer run was not retained in the exported dataset. (B) The next saved transfer measurement at 12 h had gate modulation but it did not reach the 10- μ A threshold criterion within the standardized sweep. (C) By Day 3, the FET produced a strongly abnormal transfer characteristic. The sequence illustrates why contact-related measurement problems were distinguished from later loss of interpretable FET behavior.

The endpoint comparison reinforces the same point. Air Device 8 retained recognizable forward and reverse transfer characteristics between T0 and Day 5, while PBS Device 5 showed substantial directional and current-response changes over the same interval (Figure 3.5). The PBS FET still responded to gate voltage at Day 5. What had been lost was electrically interpretable FET behavior. The bare-device pilot showed that aqueous degradation in this platform could not be represented by a single metric or by a simple working/failed label. Threshold position, hysteresis, transfer shape, output behavior, leakage, and gate modulation could change at different times and, in some cases, could again produce an interpretable response at a later measurement even if a previous measurement was not interpretable.

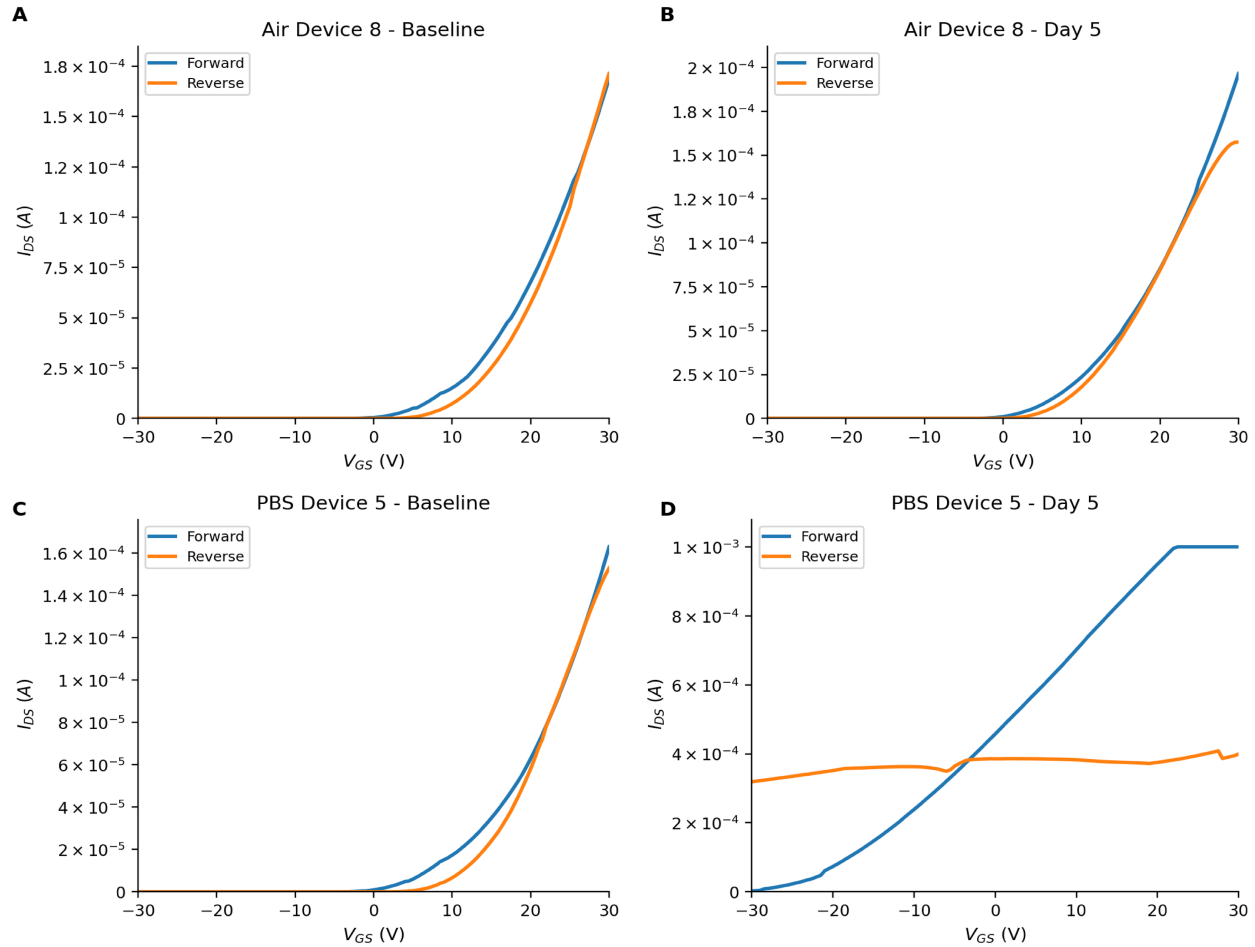


Figure 3.5. Forward and reverse transfer characteristics at baseline and Day 5 for representative air- and PBS-exposed FETs. (A,B) Air Device 8 at T0 and Day 5, respectively. (C,D) PBS Device 5 at T0 and Day 5, respectively. Air Device 8 retained recognizable forward and reverse gate modulation across the observation interval. PBS Device 5 also retained detectable gate dependence at Day 5 but showed pronounced changes in transfer position, directional behavior, and current response. Any flattening caused by the programmed current-compliance limit is interpreted as an instrument limit rather than saturation. Blue and orange curves denote forward and reverse gate sweeps, respectively.

3.3 Replication of PBS-associated electrical degradation on additional wafers

The electrical changes observed during the original five-day pilot raised the question of whether the result was specific to the two parent wafers used in that experiment. Bare In_2O_3 FETs from three additional parent wafers were evaluated under PBS exposure. Four FETs were tested from each wafer. Tests 1-4 were taken from Wafer A and Tests 5-8 from Wafer B; both

sets were characterized at T0, 24 h, and 48 h. Tests 9-12 were taken from Wafer C and were characterized at T0 and 24 h. The purpose of this experiment was not to generate a second independently powered longitudinal comparison, but to determine whether the broad electrical failure pattern observed in the first pilot recurred on separately fabricated wafers.

All 12 devices showed interpretable FET behavior at T0. At 24 h, electrical degradation was present on all three wafers, although threshold extractability, transfer modulation, and output behavior did not change together. On Wafer A, 3/4 FETs retained an interpretable transfer response and 2/4 retained a 10- μ A threshold crossing within the standardized -30 to $+30$ V sweep. Three of four devices retained gate-dependent output behavior, and 2/4 remained saturation qualified. Wafer B showed a similar separation: 3/4 devices retained interpretable transfer behavior and a 10- μ A crossing within the formal sweep, while all four output measurements remained gate dependent but none retained saturation-qualified output behavior. On Wafer C, 3/4 devices retained interpretable transfer behavior at 24 h, but none crossed the 10- μ A threshold criterion within the standardized sweep and none remained saturation qualified, despite gate-dependent output behavior being present in all four measured devices (Table 3.2).

The 48-h measurements from Wafers A and B showed that this separation persisted. On Wafer A, 3/4 devices remained transfer interpretable and 3/4 retained a 10- μ A crossing within the formal sweep. Output characteristics were available for three devices, and all three remained gate dependent, but none were saturation qualified. On Wafer B, 3/4 devices again retained interpretable transfer behavior at 48 h, although none retained a 10- μ A crossing within the standardized gate-voltage range. All four measured output families remained gate dependent, while saturation-qualified behavior remained absent. The additional-wafer tests reproduced a

central feature of the first pilot: loss of threshold extractability or recognizable saturation behavior could occur before complete loss of gate modulation.

Table 3.2. Operational electrical outcomes during additional-wafer PBS replication.

Wafer	Time	Electrically interpretable transfer	10- μ A crossing within formal sweep	Output measured	Gate-dependent output	Saturation-qualified output
A	Baseline	4/4	4/4	4/4	4/4	4/4
A	24 h	3/4	2/4	4/4	3/4	2/4
A	48 h	3/4	3/4	3/4	3/4	0/4
B	Baseline	4/4	4/4	4/4	4/4	4/4
B	24 h	3/4	3/4	4/4	4/4	0/4
B	48 h	3/4	0/4	4/4	4/4	0/4
C	Baseline	4/4	4/4	4/4	4/4	4/4
C	24 h	3/4	0/4	4/4	4/4	0/4

Note. Counts are reported relative to the four tested devices from each parent wafer. Tests 1–4 correspond to Wafer A, Tests 5–8 to Wafer B, and Tests 9–12 to Wafer C. Wafers A and B were measured at baseline, 24 h, and 48 h; Wafer C was measured at baseline and 24 h. Transfer interpretability, presence of a 10- μ A threshold crossing within the standardized -30 to $+30$ V sweep, gate-dependent output behavior, and saturation qualification are reported separately because these electrical properties did not necessarily change at the same time. Missing output measurements were retained as unmeasured rather than classified as output failure. Wider diagnostic gate-voltage sweeps were used only to determine whether gate modulation remained present and were not substituted into standardized longitudinal metric calculations.

The replication tests also showed that the post-PBS electrical state could depend on the drying procedure used before characterization. In Test 3, the FET showed a substantially altered electrical response after PBS exposure and N_2 drying. After the provisional 70°C , 10 min thermal treatment, the transfer response became more clearly gate dependent and the output characteristics became more interpretable relative to the pre-heat measurement (Figure 3.6). This observation did not establish that 70°C was the appropriate thermal condition for the platform, nor did it identify the mechanism responsible for the change. It showed that an abnormal electrical state measured immediately after PBS exposure and N_2 drying was not necessarily

irreversible. This result motivated the controlled temperature experiment described in Section 3.6.

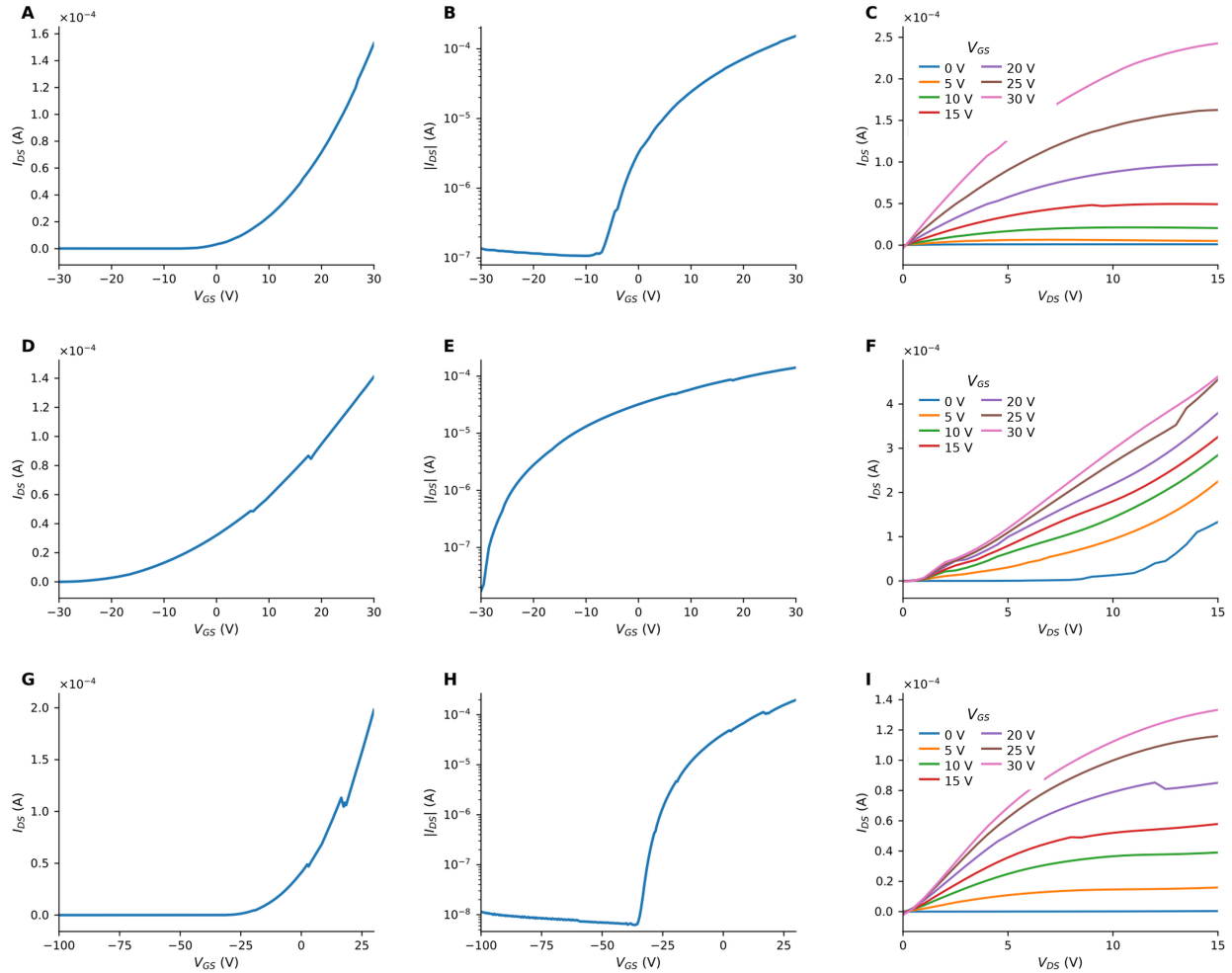


Figure 3.6. Change in electrical behavior before and after provisional thermal drying during additional-wafer PBS testing. Representative electrical characteristics from Test 3 are shown at baseline (A–C), after 24 h PBS exposure before thermal drying (D–F), and after the 70 °C, 10 min thermal treatment (G–I). Linear transfer characteristics are shown in A, D, and G; the corresponding logarithmic $|I_{DS}|$ transfer characteristics are shown in B, E, and H; and output characteristics are shown in C, F, and I. The post-exposure pre-heat measurement showed substantial alteration relative to baseline, while the post-heat measurement recovered a more interpretable gate-dependent response in the curves. This observation motivated the dedicated thermal-conditioning experiment and is not interpreted as establishing an optimized recovery temperature or a specific recovery mechanism. Output-family colors in panels C, F, and I follow Figure 3.3 and represent gate-source voltages from 0 to 30 V in 5 V increments.

Representative longitudinal measurements show the range of behavior underlying the operational counts (Figure 3.7). Some FETs retained clear gate modulation while their transfer

response progressively shifted outside the standardized -30 to $+30$ V window. Wider diagnostic sweeps were collected in selected cases to determine whether a transition remained present at more negative gate voltage. These wider sweeps were used only to distinguish a shifted but still gate-responsive device from one that had lost modulation altogether. They were not substituted into the formal $10\text{-}\mu\text{A}$ threshold extraction or the standardized longitudinal metric calculations. Forward and reverse sweeps also became increasingly separated in some FETs, and the accompanying gate-current traces changed independently of the drain-to-source current response.

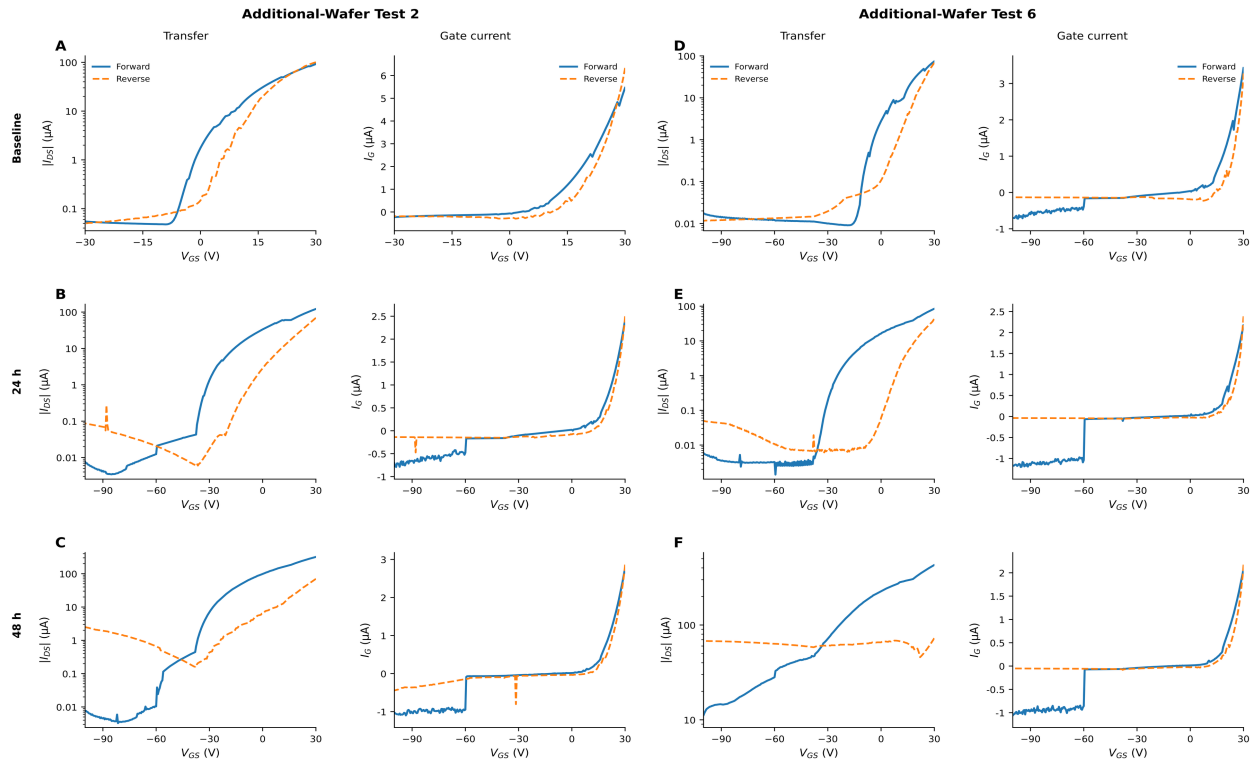


Figure 3.7. Representative longitudinal transfer and gate-current behavior during additional-wafer PBS replication. Two representative devices followed through 48 h are shown across baseline, 24 h, and 48 h. Panels A–C show the first representative device and panels D–F the second. Each panel contains forward and reverse $|I_{DS}|$ transfer characteristics together with the corresponding gate-current response. Wider diagnostic gate-voltage sweeps were collected where the standardized -30 to $+30$ V sweep no longer captured the transfer transition. These sweeps were used to determine whether gate modulation remained present but were not substituted into the standardized threshold extraction or longitudinal metric calculations. Blue solid and orange dashed curves denote forward and reverse gate sweeps, respectively, for both drain-to-source and gate-current traces.

The abrupt within-session electrical behavior observed in the original pilot also recurred in the additional-wafer testing. In Test 4, the initial output family remained gate dependent, although it did not show fully developed behavior. The transfer measurement collected immediately afterward became largely flat as a transfer response and was accompanied by a large I_G . When the output curve was repeated, its behavior had changed substantially from the initial output measurement (Figure 3.8). This sequence is described here as a short-like electrical phenotype, not a confirmed physical short circuit. Abnormal gate-current pathways can alter measured drain-to-source behavior in oxide TFTs, but the present measurements do not establish where an unintended current pathway formed or what caused it (Lee et al., 2018). Detailed replication metrics and selected supporting figures are retained in Appendix B.2; complete device-level replication records are provided in Supplemental Data S1.

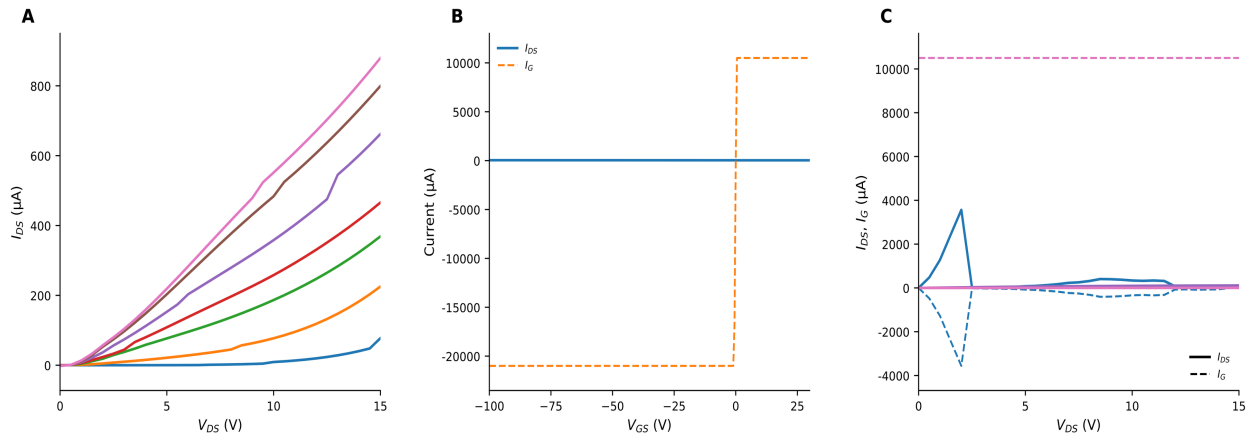


Figure 3.8. Recurrence of short-like electrical behavior during additional-wafer PBS testing. Sequential electrical measurements from Test 4 are shown in acquisition order. (A) The initial output family retained gate-dependent drain-to-source behavior, although saturation-like behavior was not fully developed. (B) The subsequently collected transfer characteristic was strongly abnormal, with little interpretable transfer modulation and a large accompanying gate-current response. (C) A repeated output measurement collected after the abnormal transfer no longer retained the behavior of the initial output family. The sequence is described as a short-like electrical phenotype but does not establish the physical location or mechanism of an unintended conduction pathway. Output-family colors in panels A and C follow Figure 3.3 and represent gate-source voltages from 0 to 30 V in 5 V increments. In panel B, the blue solid trace is drain-to-source current and the orange dashed trace is gate current; in panel C, solid and dashed line styles distinguish drain-to-source and gate-current traces for the corresponding gate-voltage color.

Overall, the additional-wafer tests showed that similar PBS-associated degradation observed in the original pilot could be reproduced on FETs from other fabrication batches. The replicated features included loss of threshold extractability, loss of saturation-like output behavior, large transfer shifts, and short-like electrical behavior while some degree of gate modulation remained. These findings shifted the next question from whether the original pilot was specific to those wafers toward why the bare FET architecture changed so rapidly under the PBS-exposure workflow. This prompted a review of prior aqueous In_2O_3 FET studies and, ultimately, subsequent redesign of the longitudinal experiment.

3.4 Literature context and redesign of the longitudinal study

The repeated degradation of bare FETs raised a second question: why had other In_2O_3 FET studies reported multi-day operation in aqueous environments when the devices used here changed substantially within the first 24–48 h? The closest studies differed from the present experiment in architecture, gating configuration, or surface protection. Liu et al. (2020) evaluated flexible In_2O_3 nanoribbon FETs during multi-day PBS exposure. These results showed that an In_2O_3 FET platform could remain electrically measurable during prolonged PBS exposure. However, they did not establish the expected stability of the architecture studied here because the reported devices were fabricated on flexible PET and operated in solution using an integrated Au common-gate electrode, rather than the dry Si/SiO_2 back-gated configuration used in this thesis (Liu et al., 2020).

Zhu et al. (2021) provide a closer comparison because the In_2O_3 nanoline FETs were also back gated through a Si substrate. Transfer characteristics were obtained after 3, 6, and 9 days of PBS exposure and shifted as exposure continued. Their architecture protected regions that

remained exposed in the present bare-device pilot. The Ti/Pt electrodes were covered with a patterned 5- μm SU-8 layer, and a PDMS well confined liquid to the exposed In_2O_3 region (Zhu et al., 2021). The multi-day measurements reported by Zhu et al. did not establish that the unprotected architecture used here should exhibit the same behavior.

Studies on other FET platforms point to the same broader issue: how the device is protected from an ionic environment can substantially change its electrical stability. Albarghouthi et al. (2022) compared unpassivated and passivated carbon-nanotube BioFETs during prolonged PBS operation and found that passivation affected leakage current, device yield, hysteresis, subthreshold swing, on/off ratio, and long-term current stability. Hideshima et al. (2022) similarly found that adding surface coatings to a silicon FET improved retention of electrical characteristics during extended $1\times$ PBS exposure. These studies use different transistor materials and gating configurations, so their specific passivation strategies are not directly transferable to the present In_2O_3 devices. They do support treating the exposed device interfaces as part of the aqueous-stability problem rather than assuming that stability is determined by the semiconductor channel alone (Albarghouthi et al., 2022; Hideshima et al., 2022).

This literature changed the direction of the study. Switching the PBS-exposed FETs to an electrolyte gated architecture would have made the electrical measurement itself different from the matched air controls, while adding a new patterned SU-8/PDMS encapsulation scheme would have introduced a device architecture that was not part of the Andrews-laboratory cortisol platform being developed here. The back-gated measurement geometry was retained for both exposure groups. Therefore, the study shifted toward surface treatments already relevant to preparation of the In_2O_3 aptamer-FET platform.

The published Andrews-laboratory cortisol FET provides the lineage for that decision. Wang et al. (2022) used vapor-deposited (3-aminopropyl)triethoxysilane (APTES)/PTMS on the In_2O_3 surface followed by 1 mM dodecanethiol treatment of the Au source and drain electrodes before aptamer functionalization. FETs taken through the silanization and dodecanethiol steps were also used as pH FETs without the aptamer chemistry, showing that the FET remained operational after those pre-aptamer surface treatments (Wang et al., 2022). The conditions decided on in this thesis were not a direct reproduction of that published APTES/PTMS protocol. Instead, the current laboratory preparation sequence was separated into progressive pre-aptamer states: Bare, DDT, PTMS + DDT, and UV + PTMS + DDT. These arms were designed as such to observe whether adding successive stages of surface preparation changed the electrical state and longitudinal stability of the underlying device.

Before committing a cohort to these conditions, three protocol questions still had to be resolved. First, it was necessary to confirm that dodecanethiol treated devices retained interpretable back-gated operation before carrying the electrode-passivation step into the main study. Second, the pilot had shown that the electrical state measured after PBS exposure depended on the drying procedure used before characterization, requiring a standardized thermal-conditioning procedure. Third, the PBS droplet visibly evaporated during exposure, which meant that the PBS exposure could not be assumed to remain compositionally unchanged over time. The remaining experiments described in this chapter addressed these three questions.

3.5 Electrical compatibility of dodecanethiol electrode passivation

Dodecanethiol was the first surface-treatment step added to the bare-device architecture. In the Andrews-laboratory functionalization workflow, dodecanethiol is used to passivate the

exposed Au source and drain electrodes before subsequent sensing-surface chemistry (Wang et al., 2022). The published pH FETs remained functional after silane treatment and dodecanethiol passivation, but those measurements used a liquid gate rather than the Si/SiO₂ back gate used in the present study. A preliminary experiment was performed to determine whether dodecanethiol treated FETs retained interpretable back-gated electrical behavior before this treatment was incorporated into the longitudinal study.

Two wafer pieces were used, with four FETs tested on each piece. Each FET was measured immediately before treatment and immediately after immersion in 1 mM dodecanethiol in ethanol for 1 h, followed by an ethanol rinse and N₂ drying. The eight FETs were retained as paired device-level observations nested within the two treated wafer pieces and were not treated as eight independent treatment replicates.

All eight devices had gate-modulated transfer behavior and gate-dependent output characteristics after dodecanethiol treatment. However, their electrical state was changed after treatment. Forward constant-current threshold voltage shifted by an average of -4.92 ± 2.68 V across all eight paired measurements. Reverse threshold voltage shifted by -6.51 ± 3.30 V across seven paired measurements. Reverse V_{th} used $N = 7$ because the post-treatment reverse sweep of P2D3 crossed the 10- μ A criterion three times. Because the Chapter 2 definition requires a unique interpretable crossing, no reverse threshold was assigned for that measurement. Signed hysteresis also used the same seven-device subset and decreased by 2.30 ± 2.46 V on average.

Table 3.3. Immediate electrical response to dodecanethiol treatment.

Metric	<i>N</i>	Before treatment mean $\pm$ SD	After treatment mean $\pm$ SD	Paired response mean $\pm$ SD
Forward $V_{th,10\ \mu A}$ (V)	8	5.24 ± 4.90	0.32 ± 5.55	-4.917 ± 2.683 V
Reverse $V_{th,10\ \mu A}$ (V)	7	13.27 ± 6.50	6.77 ± 8.33	-6.505 ± 3.304 V
Hysteresis, ΔV_H (V)	7	8.82 ± 2.73	6.51 ± 3.07	-2.304 ± 2.461 V
I_{DS} at $V_{GS} = +30$ V (μA)	8	183.25 ± 120.83	262.11 ± 185.35	0.112 ± 0.213 (1.29 $\times$ geometric mean)
Subthreshold swing (V/dec)	8	7.70 ± 4.96	5.21 ± 1.38	-0.088 ± 0.311 (0.82 $\times$ geometric mean)
I_{on}/I_{off} (measured)	8	$3.24 \times 10^4 \pm 6.64 \times 10^4$	$1.04 \times 10^4 \pm 9.13 \times 10^3$	0.160 ± 1.056 (1.45 $\times$ geometric mean)
Maximum forward $ I_G $ (μA)	8	18.21 ± 8.51	17.41 ± 3.99	0.004 ± 0.097 (1.01 $\times$ geometric mean)
R_{IG} at $V_{GS} = +30$ V	8	0.15 ± 0.09	0.13 ± 0.13	-0.014 ± 0.095

Note. Values are descriptive summaries from eight FETs on two treated wafer pieces. The reported *n* is the number of devices with a paired measurement for that metric. Threshold voltage and hysteresis are reported as after-minus-before changes. Drain-to-source current, subthreshold swing, I_{on}/I_{off} , and maximum gate current are reported as $\log_{10}(\text{after}/\text{before})$ changes, with geometric-mean ratios included for interpretation. Numerical SS values were retained whenever the Chapter 2 fitting criteria were met; gate current was evaluated separately from the numerical fit. Numerical I_{on}/I_{off} values were also retained. Six of eight FETs before treatment and all eight FETs after treatment had $|I_G| \geq |I_{DS}|$ within the low-current region used to determine I_{off} ; this was treated as a QC flag rather than as a reason to remove the measured ratio. Reverse V_{th} and hysteresis use $N = 7$ because P2D3 had three 10- μA crossings in the post-treatment reverse sweep, so a unique reverse threshold could not be assigned. Complete transfer and gate-current measurements are shown in Supplemental Data S1.

Current-related metrics also changed after dodecanethiol treatment. Fixed-bias $|I_{DS}|$ at $V_{GS} = +30$ V increased overall, with a geometric-mean after/before ratio of 1.29. SS decreased overall, with a geometric-mean after/before ratio of 0.82. SS was calculated for all eight devices because each met the Chapter 2 fitting criteria; gate leakage within the fitted region was evaluated separately rather than used to remove the numerical SS value.

The I_{on}/I_{off} ratio showed substantial FET-to-FET variability. The mean measured I_{on}/I_{off} was 3.24×10^4 before treatment and 1.04×10^4 after treatment. However, when the change was calculated separately within each device before summarization, the geometric-mean after/before

ratio was 1.45. The difference between these summaries reflects the wide variation among FETs: some increased substantially while others decreased, so the treatment did not produce a consistent directional change in $I_{\text{on}}/I_{\text{off}}$. Numerical $I_{\text{on}}/I_{\text{off}}$ values were retained for all eight FETs. I_G exceeded I_{DS} within the low-current I_{off} region in six devices before treatment and all eight devices after treatment, so those measurements were flagged for possible leakage contribution to the off-state current.

Gate-current measurements changed very little overall. Maximum forward gate current had a geometric-mean after/before ratio of 1.01, indicating essentially no overall change. The ratio of $|I_G| / |I_{\text{DS}}|$ at $V_{\text{GS}} = +30$ V also changed little, decreasing by 0.014 ± 0.095 on average. The electrical changes observed after dodecanethiol treatment were not accompanied by a corresponding overall increase in gate leakage. Because the eight FETs were nested within only two treated wafer pieces, these results are interpreted as descriptive evidence of electrical compatibility and FET-level response and not as an independently replicated estimate of the treatment effect.

Representative devices show that FET operation was retained after treatment, although the electrical response changed. P1D1 retained clear forward and reverse gate modulation and gate-dependent output behavior before and after dodecanethiol treatment, with a shift in the transfer curve toward more negative gate voltage (Figure 3.9). P1D2 showed a larger change in its transfer and output characteristics but also remained clearly gate responsive after treatment (Figure 3.10). Complete paired measurements for all eight FETs are shown in Supplemental Data S1.

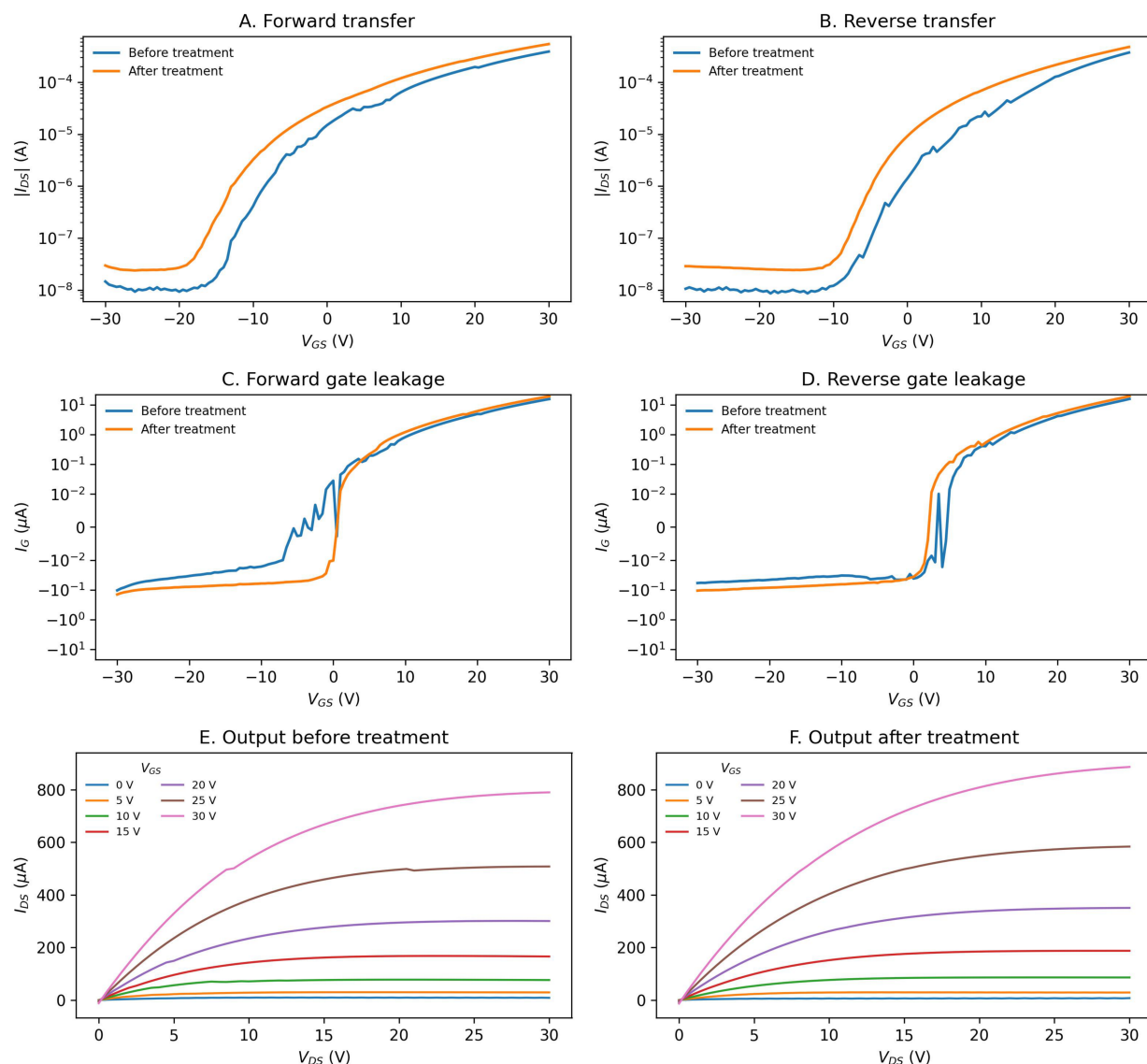


Figure 3.9. Representative back-gated electrical response before and after dodecanethiol treatment of P1D1. (A,B) Forward and reverse $|I_{DS}|$ transfer characteristics before and after treatment. (C,D) Corresponding forward and reverse gate-current responses. (E,F) Output characteristics before and after treatment. The FET retained gate-modulated transfer behavior and gate-dependent output characteristics after dodecanethiol treatment, while the transfer response shifted toward more negative gate voltage. Blue and orange denote PRE and POST processing, respectively, in panels A-D. In the output families (E,F), colors denote gate-source voltage: 0 V blue, 5 V orange, 10 V green, 15 V red, 20 V purple, 25 V brown, and 30 V pink.

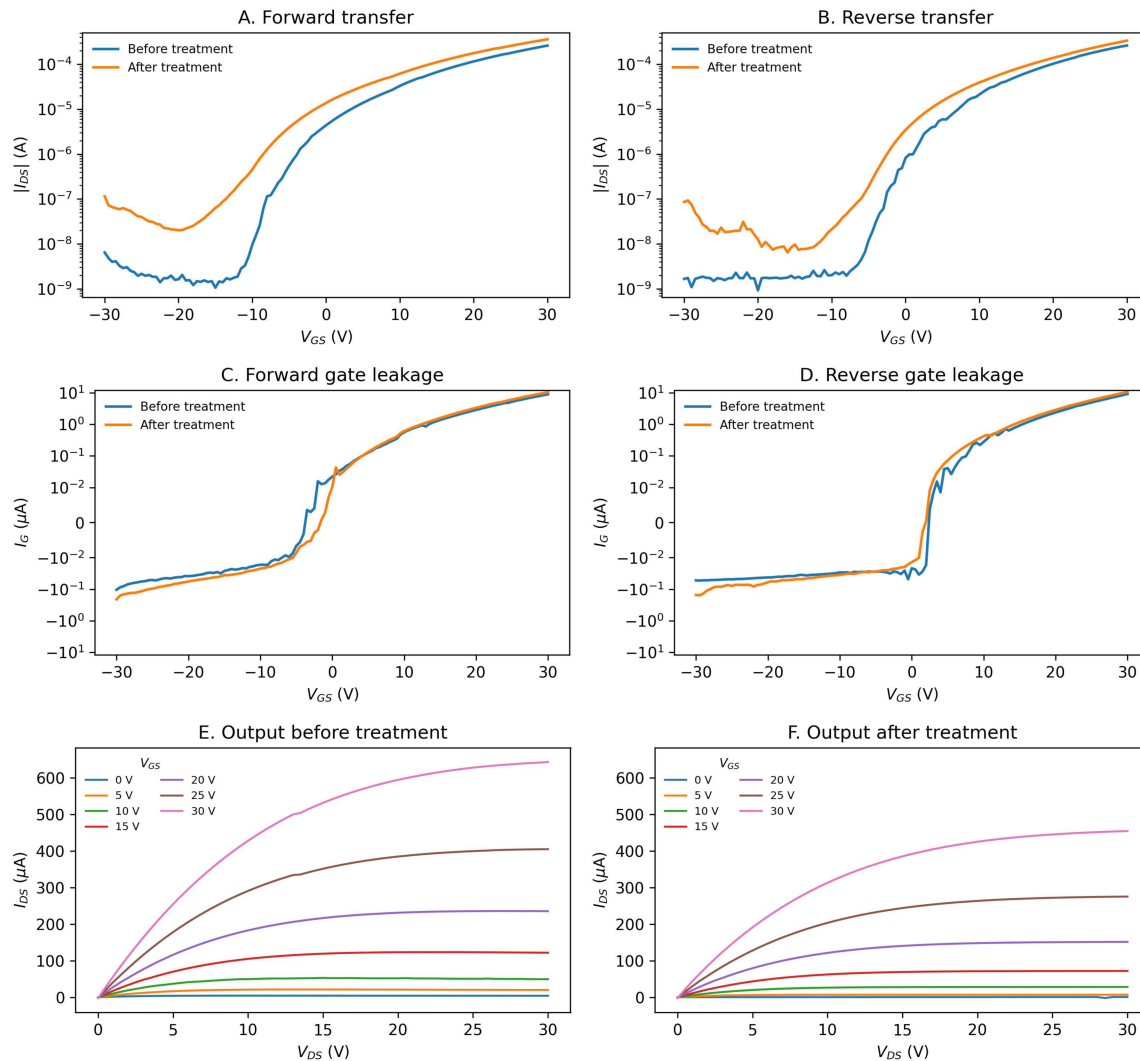


Figure 3.10. Representative back-gated electrical response before and after dodecanethiol treatment of P1D2. (A,B) Forward and reverse $|I_{DS}|$ transfer characteristics before and after treatment. (C,D) Corresponding forward and reverse gate-current responses. (E,F) Output characteristics before and after treatment. This FET showed a larger change in transfer and output behavior than the device in Figure 3.9, but retained interpretable back-gated operation after treatment. Blue and orange denote PRE and POST processing, respectively, in panels A-D. Output-family colors in panels E and F follow Figure 3.9 and represent gate-source voltages from 0 to 30 V in 5 V increments.

The FETs remained electrically interpretable with the back-gated FET characterization after dodecanethiol treatment. However, the treatment was not electrically neutral. Several FET metrics changed after processing and the magnitude and direction of those changes varied among FETs. For that reason, the primary experiment measured each FET immediately before and after its assigned surface-treatment sequence instead of assuming that processing steps left the

electrical state unchanged. Detailed DDT compatibility metrics are retained in Appendix B.3; complete paired device records are provided in Supplemental Data S1.

3.6 Selection of the post-exposure thermal-conditioning temperature

The bare-device pilot showed that rinsing and N₂ drying alone did not consistently return PBS-exposed devices to an electrically interpretable dry state. Environmental moisture is a plausible procedural variable because relative humidity has been shown to change threshold voltage, current, and other electrical characteristics of solution-processed In₂O₃ TFTs (Ding et al., 2022). Ding et al. also showed that at least part of the humidity-associated electrical response could reverse after transfer to dry air, although their experiment did not involve PBS exposure or the thermal-drying procedure used here. The temperature screen in the present study was treated as an empirical protocol-development experiment. The purpose of this experiment was to identify a practical post-exposure treatment that produced a sufficiently reproducible electrical state for subsequent dry back-gated characterization.

Four temperatures were evaluated: 40, 50, 60, and 70 °C. At each temperature, matched air- and PBS-exposed wafer pieces were characterized at baseline, after 24 h of exposure before heating, and again after 10 min of heating followed by a 2 min cooling period. Two wafer pieces were used for each exposure condition, with two FETs measured on each piece. Each temperature and exposure condition contained four selected FETs that were distributed across two wafer pieces. Device-level measurements were treated as descriptive subsamples.

The effect of heating was evaluated in both the PBS and air groups, as the thermal step was intended to become part of the measurement procedure applied to both conditions. A

suitable condition had to improve post-exposure measurability without assuming that heating itself was electrically neutral.

The lower temperatures did not provide a sufficiently consistent post-exposure state. At 40 °C, only one of four PBS devices had an extractable constant-current threshold after heating, while none of the four air devices did. At 50 °C, none of the four PBS devices retained an extractable post-heat threshold, although all four air devices did. The 50 °C PBS devices were inconsistent in recovery even with evidence of recovery in some other transfer metrics (Table 3.4).

The clearest improvement occurred at 60 and 70 °C. At 60 °C, the number of PBS-exposed FETs with an extractable threshold increased from 2/4 before heating to 3/4 afterward, while the air group increased from 1/4 to 4/4. At 70 °C, the PBS group increased from 0/4 before heating to 2/3 devices with an extractable threshold after heating, with one additional device unavailable for quantitative POST analysis because of handling damage. Numerical SS was extractable in all four PBS devices after 60 and all three viable devices after 70 °C treatment, with mean values of 3.12 ± 1.75 V/dec (n=4) and 2.62 ± 1.32 V/dec (n=3), respectively (Table 3.4).

Table 3.4. Thermal-conditioning temperature screen.

Temp	PBS threshold extractable PRE → POST	Air threshold extractable PRE → POST	PBS POST SS (V/dec)	SS leakage-flagged	PBS POST I_{on}/I_{off} mean $\pm$ SD [range]	I_{on}/I_{off} leakage-flagged	R_{IG} recovery PRE/POST mean $\pm$ SD	QC
40 °C	1/4 → 1/4	1/4 → 0/4	8.92 (n=1)	1/1	$1.14e+02 \pm 2.22e+02$ [1.00e+00–4.47e+02]	4/4	$0.99 \pm 0.27\times$ (2/4 improved)	Poor recovery; few extractable post-heat transfer metrics.
50 °C	1/4 → 0/4	2/4 → 4/4	5.19 ± 3.93 (n=3)	3/3	$5.79e+04 \pm 6.70e+04$ [1.54e+01–1.25e+05]	4/4	$2.43 \pm 2.66\times$ (2/4 improved)	Mixed recovery; PBS remains inconsistent.
60 °C	2/4 → 3/4	1/4 → 4/4	3.12 ± 1.75 (n=4)	2/4	$2.86e+05 \pm 4.34e+05$ [5.18e+03–9.30e+05]	4/4	$13.68 \pm 18.28\times$ (4/4 improved)	Usable PBS recovery with cleaner leakage behavior; broad drain-current recovery range.
70 °C	0/4 → 2/3 evaluable†	0/4 → 4/4	2.62 ± 1.32 (n=3)	3/3	$2.96e+05 \pm 2.47e+05$ [2.60e+04–5.09e+05]	3/3	$3.55 \pm 3.03\times$ (2/3 improved)	More uniform PBS drain-current recovery among the three evaluable POST devices. One evaluable device was strongly gate-current dominated POST.

Note. Threshold-extractability counts are shown before and after the 10-min thermal treatment for matched PBS-exposed and air-control FETs. Post-heat subthreshold swing, I_{on}/I_{off} , and leakage behavior are summarized for the PBS-exposed devices, together with the change in relative gate current after heating. Numerical SS and I_{on}/I_{off} values were retained when leakage-flagged under the revised Chapter 2 definitions. SS was flagged when $|I_G| \geq |I_{DS}|$ anywhere within the selected SS fitting window. I_{on}/I_{off} was flagged when $|I_G| \geq |I_{DS}|$ at any point contributing to the three-point-averaged I_{on} or I_{off} extrema. Leakage-ratio recovery was calculated as R_{IG} PRE / R_{IG} POST, where $R_{IG} = |I_G|/|I_{DS}|$ at $V_{GS} = +30$ V; values greater than 1 indicate lower relative gate current after heating. Leakage flags indicate interpretive caution and do not remove the numerical metric. FETs are subsamples nested within wafer pieces.

Heating also changed the air-exposed FETs substantially. At 60 °C, mean R_{IG} at $V_{GS} = +30$ V decreased from 1.075 ± 0.649 before heating to 0.129 ± 0.047 afterward, while the number of devices with an extractable threshold increased from 1/4 to 4/4. At 70 °C, mean R_{IG} decreased from 1.833 ± 0.766 to 0.101 ± 0.046 and the number of FETs with an extractable threshold increased from 0/4 to 4/4. The thermal treatment changed the measured electrical state of both exposure groups and was treated as part of the standardized characterization workflow rather than as an electrically neutral drying step.

Example 60 °C measurements show this behavior directly (Figure 3.11). In both the air and PBS-exposed FET examples, the post-heat transfer response differed substantially from the corresponding 24 h pre-heat measurement while retaining recognizable gate modulation. Similar recovery was observed in the representative 70 °C measurements (Figure 3.12). Complete measurements from all devices and temperatures are provided in Supplemental Data S1.

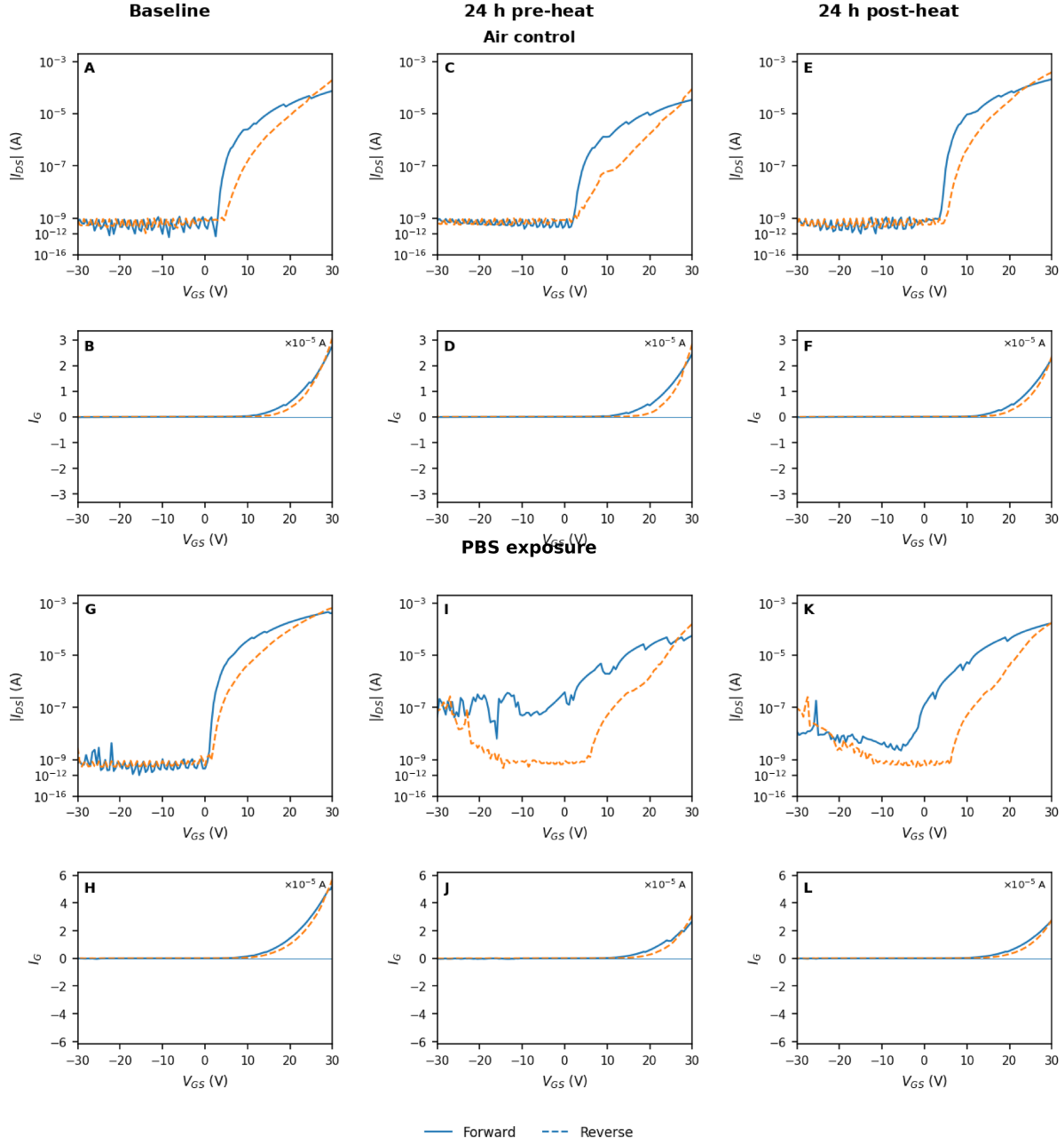


Figure 3.11. Electrical response to 60 °C thermal conditioning after 24 h air and PBS exposure. Device 2 measurements from the air-control and PBS-exposure groups are shown at baseline, after 24 h of exposure before heating, and after the 60 °C thermal-conditioning step. For the air-control FET, panels A-B show baseline transfer and gate-current characteristics, C-D show the 24 h pre-heat measurement, and E-F show the 24 h post-heat measurement. For the PBS-exposed FET, panels G-H show baseline characteristics, I-J show the 24 h pre-heat measurement, and K-L show the 24 h post-heat measurement. Post-heat measurements were collected after 10 min at 60 °C followed by 2 min of cooling. Complete characterization of all devices tested at 60 °C is provided in Supplemental Data S1. Blue solid and orange dashed curves denote forward and reverse gate sweeps, respectively.

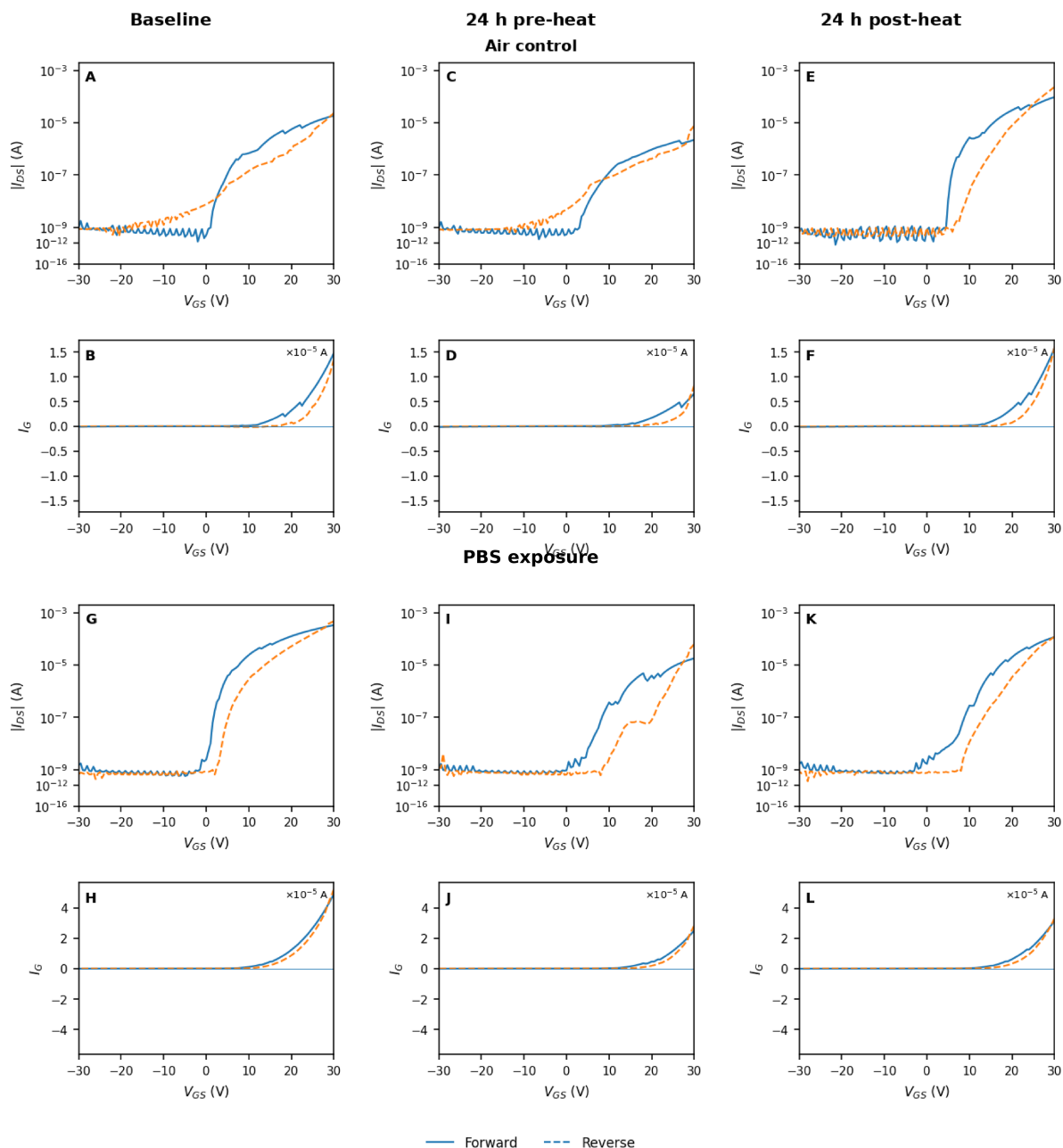


Figure 3.12. Electrical response to 70 °C thermal conditioning after 24 h air and PBS exposure. Device 2 measurements from the air-control and PBS-exposure groups are shown at baseline, after 24 h of exposure before heating, and after the 70 °C thermal-conditioning step. For the air-control FET, panels A-B show baseline transfer and gate-current characteristics, C-D show the 24 h pre-heat measurement, and E-F show the 24 h post-heat measurement. For the PBS-exposed FET, panels G-H show baseline characteristics, I-J show the 24 h pre-heat measurement, and K-L show the 24 h post-heat measurement. Post-heat measurements were collected after 10 min at 70 °C followed by 2 min of cooling. Complete characterization of all devices tested at 70 °C is provided in Supplemental Data S1. Blue solid and orange dashed curves denote forward and reverse gate sweeps, respectively.

The 60 and 70 °C conditions were not equivalent in the consistency of the PBS response. At 60 °C, all four PBS devices showed increased fixed-bias $|I_{DS}|$ magnitude after heating, but the magnitude of the increase varied widely, from 2.43× to 31.54×. Two FETs increased by only 2.43× and 3.04×, while the other two increased by 10.38× and 31.54×. At 70 °C, the recovery was more tightly grouped in comparison, ranging from 6.57–7.82× across the three viable FETs, with a mean of $7.00 \pm 0.71\times$. The coefficient of variation of the recovery factor was 1.15 at 60 °C and 0.10 at 70 °C. The tighter distribution of fixed-bias $|I_{DS}|$ recovery among the evaluable 70 °C FETs was the primary reason that 70 °C was selected for the formal study.

The 70 °C condition was not uniformly superior across all electrical metrics. All four 60 °C PBS-exposed FETs showed improvement in the fixed-bias R_{IG} after heating, compared with two of three at 70 °C. Mean post-heat R_{IG} was 0.215 ± 0.046 at 60 °C, while the 70 °C mean was strongly influenced by one device with a post-heat R_{IG} of 8.72. One 70 °C PBS-exposed FET was physically damaged during handling and could not provide a reliable post-heat measurement. POST sweeps collected after the damage were retained for traceability but excluded from quantitative POST summaries.

While both 60 and 70 °C restored substantially more interpretable transfer behavior than the lower-temperature conditions, the 70 °C, 10 min treatment was selected as the standardized condition for the main study because it produced more uniform gross fixed-bias $|I_{DS}|$ recovery across the PBS-exposed FETs tested. Since heating also changed the air-exposed FETs, the final longitudinal protocol applied the same 70 °C treatment and 2 min cooling interval to both air and PBS cohorts at every measurement timepoint. This maintained a matched thermal history between the exposure groups while providing a consistent post-exposure state for electrical

characterization. Detailed thermal-conditioning summaries are retained in Appendix B.4; complete device-level thermal-conditioning records are provided in Supplemental Data S1.

3.7 Humidity control and PBS evaporation

The preliminary pilot also showed visible loss of the PBS droplet during extended exposure. Evaporation of saline droplets removes water while retaining nonvolatile salts, increasing salt concentration as evaporation proceeds and eventually permitting crystallization (Bunio et al., 2022). Evaporation rate also depends on environmental humidity. Qu et al. (2020) showed substantial effects of humidity on brine-droplet evaporation and crystallization. These studies used simpler saline solutions rather than PBS and are used only to establish the physical consequence of solvent loss, not to predict the exact composition of the PBS droplet used here.

A humidified chamber was evaluated as a means of reducing evaporation and maintaining a more consistent aqueous exposure over the 24 h interval.

The humidified chamber used the same covered PYREX petri dish format planned for the main experiment. Small PTFE caps were placed around the perimeter of the chamber and used as reservoirs for DI water (Figure 3.13). PTFE caps were selected because they fit within the chamber and allowed the water reservoirs to remain physically separate from the wafer pieces during handling and transport. Each cap contained approximately 1 mL of DI water. No chamber relative-humidity measurement was collected, so “humidified” is used here as an operational description of the chamber condition.

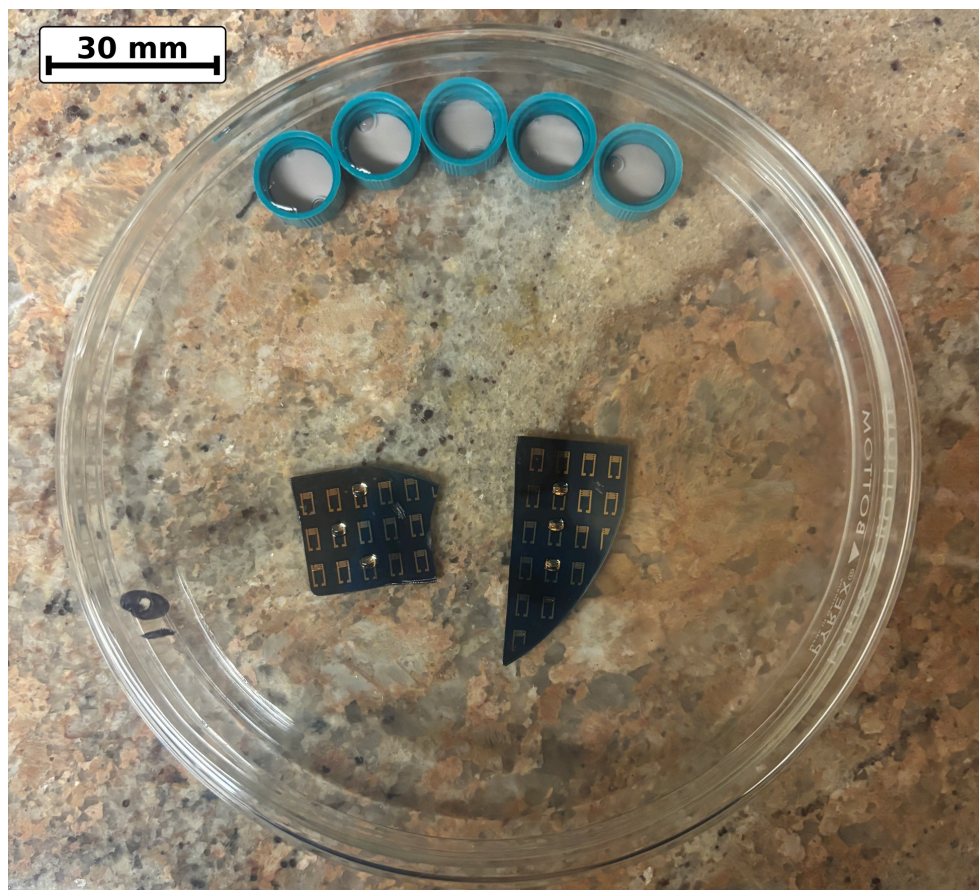


Figure 3.13. Humidified chamber configuration used for PBS exposure. Wafer pieces were placed within a covered chamber containing separate PTFE caps used as DI-water reservoirs around the chamber perimeter. The reservoirs were physically separated from the wafer pieces and were used to reduce evaporation of the PBS droplets during extended exposure. Chamber relative humidity was not measured; “humidified” refers to the operational chamber configuration rather than a quantified RH condition.

Two independently exposed wafer pieces were used for each chamber condition, with three FETs tested on each piece. Each FET received the same 6 μ L of PBS exposure used in the study. Electrical characteristics were measured at baseline and again after 24 h. The wafer pieces were also weighed before PBS application, immediately after PBS application, and after the 24 h exposure. After the 24 h mass was taken, residual PBS was aspirated, pieces were rinsed, dried under N₂, heated at 70 °C for 10 min, cooled for 2 min before electrical characterization, using the thermal-conditioning procedure selected in Section 3.6. The primary purpose of the chamber comparison was to determine whether humidification preserved the applied aqueous phase. The

electrical measurements were taken to determine whether the resulting exposure affected electrical behavior of the FETs.

The initial PBS-associated mass was similar across all four pieces, ranging from 17.9 to 18.2 mg (Table 3.5). The retained mass differed substantially between the chamber conditions. The two humidified pieces retained 72.0% and 71.5% of their initial PBS-associated mass, corresponding to a piece-level mean of $71.7 \pm 0.3\%$. In contrast, the two non-humidified pieces retained only 4.5% and 6.6%, with a mean of $5.5 \pm 1.5\%$. Thus, nearly all of the initially applied liquid-associated mass was lost from the non-humidified pieces over 24 h, while approximately 72% remained under the humidified condition.

Table 3.5. Apparent PBS-associated mass retention after 24 h of humidified and non-humidified chamber exposure.

Condition	Piece	Dry mass (g)	After PBS (g)	24 h mass (g)	Initial PBS-associated mass (g)	PBS-associated mass at 24 h (g)	Apparent retention (%)
Humidified	1	0.7052	0.7234	0.7183	0.0182	0.0131	72.0
Humidified	2	0.8593	0.8772	0.8721	0.0179	0.0128	71.5
Non-humidified	1	0.6985	0.7164	0.6993	0.0179	0.0008	4.5
Non-humidified	2	0.9698	0.9880	0.9710	0.0182	0.0012	6.6
Humidified, mean $\pm$ SD	—	—	—	—	—	—	71.7 ± 0.3
Non-humidified, mean $\pm$ SD	—	—	—	—	—	—	5.5 ± 1.5

Note. Piece mass was measured before PBS application, immediately after PBS application, and after the 24 h exposure. Apparent retention was calculated relative to the mass associated with the initial PBS application.

Apparent PBS-associated mass retention was calculated as $100 \times (\text{mass at 24 h} - \text{mass dry})/(\text{mass after PBS} - \text{mass dry})$. Values are piece-level measurements from two independently

exposed wafer pieces per chamber condition. Because retained mass may include adsorbed or condensed water, the calculation is interpreted as apparent PBS-associated mass retention rather than an exact measurement of remaining PBS volume. No chamber RH measurement was collected.

Electrical degradation remained evident in both chamber conditions after 24 h. Both groups showed reductions in fixed-bias $|I_{DS}|$ and I_{on}/I_{off} , increases in subthreshold swing, and changes in threshold voltage and hysteresis after 24 h (Table 3.6). The geometric mean fixed-bias $|I_{DS}|$ retention was similar in magnitude between conditions, corresponding to approximately 14% of baseline in the humidified group and 16% in the non-humidified group. Forward and reverse SS also increased by similar factors in the two conditions. The mean $\log_{10}$ forward SS ratio was 0.341 ± 0.285 for humidified devices and 0.381 ± 0.375 for non-humidified devices, while the corresponding reverse values were 0.303 ± 0.118 and 0.321 ± 0.230 .

Table 3.6. Paired electrical response after 24 h of PBS exposure with and without humidification.**Part A.** Threshold, hysteresis, fixed-bias current, and SS responses

Condition	Unique forward 10 μ A crossing (N/6)	Unique reverse 10 μ A crossing (N/6)	Δ forward V_{th} (V)	Δ reverse V_{th} (V)	Δ signed hysteresis (V)	$\log_{10} I_{DS} $ (+30 V) ratio	$\log_{10}$ forward SS ratio	$\log_{10}$ reverse SS ratio
Humidified	5/6	6/6	9.742 ± 3.88 (N=5)	15.8 ± 3.89 (N=6)	5.248 ± 2.06 (N=5)	-0.8618 ± 0.321 (N=6)	0.3409 ± 0.285 (N=6)	0.303 ± 0.118 (N=6)
Non-humidified	5/6	5/6	-1.883 ± 6.99 (N=5)	8.636 ± 2.58 (N=5)	10.52 ± 4.83 (N=5)	-0.7907 ± 0.462 (N=6)	0.381 ± 0.375 (N=6)	0.3213 ± 0.23 (N=6)

Part B. Leakage/QC, Ion/Ioff, relative gate current, and output behavior

Condition	10 μ A F leakage-flagged (N/unique)	10 μ A R leakage-flagged (N/unique)	SS F leakage-flagged (N/6)	SS R leakage-flagged (N/6)	$\log_{10}$ forward I_{on}/I_{off} ratio	$\log_{10}$ reverse I_{on}/I_{off} ratio	R_{IG} at +30 V, 24 h	Saturation-like output at 24 h (N/6)
Humidified	2/5	4/6	6/6	5/6	-1.058 ± 0.637 (N=6)	-1.106 ± 0.843 (N=6)	0.6821 ± 0.47 (N=6)	3/6
Non-humidified	0/5	0/5	6/6	4/6	-0.8968 ± 0.827 (N=6)	-0.8961 ± 0.227 (N=6)	0.338 ± 0.551 (N=6)	4/6

Note. Electrical responses were calculated within each FET relative to its own baseline measurement before chamber-condition summaries were generated. Device observations are nested within two independently exposed wafer pieces per chamber condition, with three FETs measured on each piece. Threshold voltage and signed hysteresis are reported as arithmetic 24 h-minus-baseline changes. Fixed-bias $|I_{DS}|$, subthreshold swing, and I_{on}/I_{off} are reported as $\log_{10}(24 \text{ h}/\text{baseline})$ responses. The reported N is the number of devices with a paired numerical value for the corresponding metric and does not represent the number of independent treatment replicates. A numerical 10- μ A threshold was retained when a unique interpretable crossing was present; leakage was recorded separately as a QC flag. Numerical SS and I_{on}/I_{off} values were retained when calculable under the Chapter 2 definitions, with leakage flags reported separately.

Threshold behavior differed more clearly between the two chamber conditions. Among FETs with a unique numerical crossing at both measurements, forward threshold voltage shifted positively by 9.74 ± 3.88 V in the humidified group, whereas the non-humidified group showed a mean change of -1.88 ± 6.99 V. Reverse threshold voltage shifted positively in both conditions but more strongly under humidification, by 15.80 ± 3.89 V compared with 8.64 ± 2.58 V. Signed hysteresis increased in both groups, although the increase was smaller among the humidified devices with valid paired hysteresis measurements: 5.25 ± 2.06 V in the humidified group and 10.52 ± 4.83 V in the non-humidified group. Because these summaries include only devices for

which the corresponding thresholds remained uniquely extractable, they were treated as descriptive.

$I_{\text{on}}/I_{\text{off}}$ decreased in both chamber conditions. The mean paired $\log_{10}$ forward $I_{\text{on}}/I_{\text{off}}$ ratio was -1.058 ± 0.637 in the humidified devices and -0.897 ± 0.827 in the non-humidified devices. Reverse $I_{\text{on}}/I_{\text{off}}$ showed a similar pattern, with mean $\log_{10}$ ratios of -1.106 ± 0.843 and -0.896 ± 0.227 , respectively. Electrical response remained heterogeneous in both groups despite the much larger difference in liquid-mass retention. Numerical $I_{\text{on}}/I_{\text{off}}$ and SS values were retained under the Chapter 2 definitions even when the associated leakage QC flag was present.

Similarly, output characteristics were not uniformly improved by the humidified chamber. Only three of six humidified devices and four of six non-humidified devices had saturation-like output behavior at 24 h. Other devices were still gate-dependent but had abnormal or largely unsaturated output behavior. Complete transfer, gate-current, and output measurements for all twelve devices are provided in the humidity-control subsection of Supplemental Data S1. Device-level summary metrics are retained in Appendix B.5.

The chamber experiment supported humidification as a method for reducing evaporation of the free PBS droplet, but not as a treatment that improved bare-device electrical stability. The much greater mass retention under humidified conditions indicated that the PBS-exposed FETs remained in contact with an aqueous phase for a larger fraction of the 24-h interval. Because of the small sample size, the evolving solute concentration, exact droplet volume, and transition to residual dried salts were not measured directly, no definitive quantitative claim can be made about PBS concentration during the exposure.

3.8 Final protocol decisions carried forward

The experiments covered in this chapter changed the design of the original bare-device air-versus-PBS study into the formal surface-preparation experiment described in Chapter 2. The bare-device pilot and additional-wafer tests showed that electrical degradation could occur within the first several days of PBS exposure.

Four progressive surface-preparation conditions were chosen as study arms for the primary study: Bare, DDT, PTMS + DDT, and UV + PTMS + DDT. The preliminary dodecanethiol experiment showed that the Au-passivation step remained compatible with back-gated characterization but changed the device's electrical state. For this reason, FETs in each surface-prepared condition were characterized immediately before and after their assigned surface-preparation sequence. These changes were analyzed in Chapter 4. A separate T0 measurement was then used as the reference immediately before environmental exposure.

Each surface condition was evaluated under matched ambient-air and PBS exposure. PBS was applied as a 6 μ L droplet over each tested device. The humidity-control experiment showed that free PBS droplets lost nearly all of their associated liquid mass over 24 h without supplemental humidification, while approximately 72% remained in the humidified chamber. PBS-exposed pieces were maintained in covered PYREX petri dishes containing separate DI-water reservoirs to reduce evaporation. This chamber configuration was selected to better preserve and standardize the intended aqueous exposure, not because humidification improved the electrical stability of the bare FETs. Air-control pieces also remained in dry, sterile chambers and were not humidity matched to the PBS condition.

The post-exposure measurement protocol was also standardized. At each electrical-characterization timepoint, PBS was aspirated from the exposed FETs, followed by a DI-water

rinse and N₂ drying. PBS-exposed and air-control pieces then received the same 70 °C thermal treatment for 10 min and were cooled for 2 min before electrical characterization. As the drying step occurs in a different lab, air-control pieces underwent the same transport and thermal history but did not receive the aqueous rinse or N₂ drying. The 70 °C condition was selected because fixed bias I_{DS} recovery among the PBS-exposed FETs was more tightly grouped than at the lower-temperature conditions. Gate-leakage behavior was comparable to the 60 °C condition in two of the three evaluable FETs, although one 70 °C FET developed marked post-heat gate leakage.

Electrical characterization was performed with the dry device surface using the common Si/SiO₂ back gate. Immediately after measurement, fresh 6 µL PBS droplets were reapplied to the PBS-exposed devices and the pieces were returned to their assigned humidified chambers, while air controls were returned to their dry chambers. FETs that deteriorated after entering the experiment were retained as experimental outcomes and not replaced.

These protocol-development experiments established the exposure, conditioning, and measurement procedures used in Chapters 4 and 5. They also motivated the shift from a bare-device stability experiment to a study of how successive pre-aptamer surface-preparation states contributed to FET electrical behavior before and during environmental exposure.

Chapter 4. Immediate electrical effects of progressive surface preparation

Chapter 3 established why the primary study moved beyond the bare-device pilot and motivated the evaluation of stability for the progressive pre-aptamer surface conditions. However, before determining whether those surface states alter the stability during air or PBS exposure, immediate electrical effects of surface processing had to be separated from later environmental effects. This chapter compares each treated FET immediately before and after its assigned surface-preparation sequence. The analysis first places the experimental surface states within the broader In_2O_3 aptamer-FET functionalization pathway and then evaluates how DDT, PTMS + DDT, and UV + PTMS + DDT processing changed the electrical state of the FETs before environmental exposure began. These processing responses are analyzed separately from the longitudinal air-versus-PBS responses in Chapter 5.

4.1 Surface preparation within the complete aptamer-FET interface

The surface-treatment conditions used in this thesis are considered within the broader functionalization pathway of the In_2O_3 aptamer-FET platform. In the published cortisol-sensing platform, Wang et al. fabricated flexible In_2O_3 FETs on polyimide and used an Ag/AgCl solution gate for sensing. Their surface functionalization began with vapor-phase deposition of APTES/PTMS at a 1:9 volume ratio. The devices and silane solution were held at 40 °C for 1 h, followed by a 10 min bake at 80 °C. The exposed Au source and drain electrodes were then incubated for 1 h in 1 mM dodecanethiol in ethanol. Devices used for pH sensing were taken through these silanization and Au-passivation steps without subsequent aptamer functionalization. For the complete cortisol aptamer-FET, the silanized devices were subsequently exposed to 1 mM 3-maleimidobenzoic acid N-hydroxysuccinimide ester (MBS) in

a 1:9 DMSO:PBS mixture for 30 min, followed by overnight immobilization of a 1 μ M thiolated cortisol aptamer. Before immobilization, the aptamer was heated to 95 °C for 5 min, rapidly cooled in an ice bath, and returned to room temperature (Wang et al., 2022).

The first of these surface-processing stages modifies the oxide surface with organosilanes. Trialkoxysilanes can hydrolyze to form silanol groups and subsequently undergo condensation with hydroxyl-containing oxide surfaces or with neighboring silanols. The resulting layer can contain surface-linked siloxane bonds as well as condensed silane structures. Studies of APTES on oxide surfaces have shown that the resulting layer depends strongly on reaction time, solvent, temperature, and curing conditions rather than forming a single invariant monolayer structure (Vandenberg et al., 1991; Acres et al., 2012). Trimethoxy(propyl)silane (PTMS) belongs to the same trialkoxysilane class but is propyl-terminated and does not provide the primary amine used for downstream MBS coupling. The molecular structures shown in Figure 4.1 are idealized representations of the intended surface chemistry rather than measurements of the molecular organization present on the FETs.

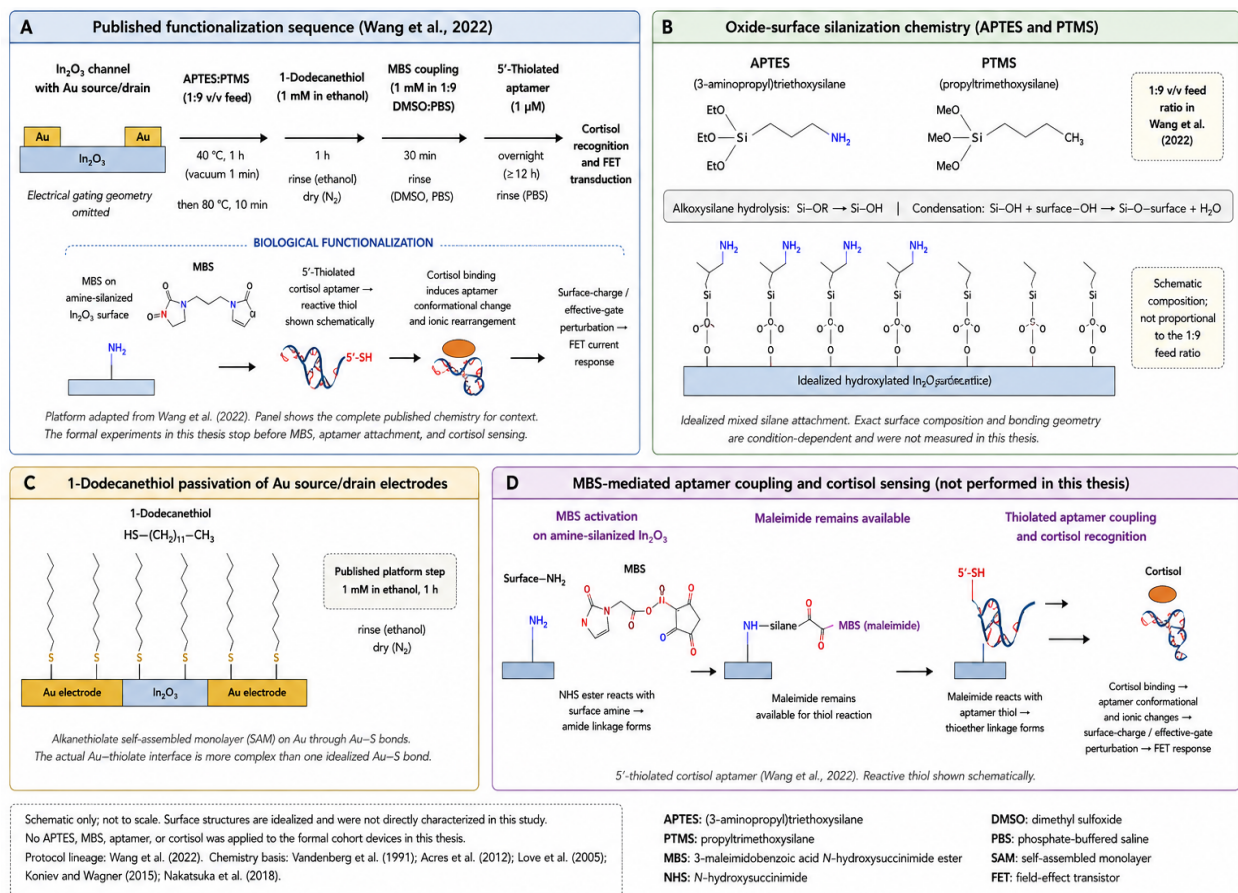


Figure 4.1. Functionalization chemistry of the complete In_2O_3 aptamer-FET platform and relationship to the present study. (A) Published cortisol-sensing sequence showing the complete workflow from mixed APTES/PTMS silanization through 1-dodecanethiol passivation, MBS coupling, thiolated-aptamer attachment, and cortisol recognition. (B) Oxide-surface silanization chemistry of APTES and PTMS, shown schematically as hydrolysis and condensation with a hydroxylated In_2O_3 surface; the idealized mixed attachment and feed ratio are included for context and were not measured here. (C) 1-Dodecanethiol passivation of the exposed Au source and drain electrodes through Au-S bonding. (D) MBS-mediated coupling of a thiolated aptamer to the amine-silanized surface and subsequent cortisol recognition, shown for context. The experiments in this thesis stop before MBS, aptamer attachment, and cortisol sensing and instead evaluate selected pre-aptamer surface-preparation states. Surface structures are schematic and were not directly characterized in the present study.

Dodecanethiol (DDT) modifies a different interface from the organosilane treatment.

Long-chain alkanethiols adsorb onto Au and can form organized alkanethiolate self-assembled layers with hydrocarbon-terminated surfaces (Love et al., 2005). In the Wang functionalization sequence, dodecanethiol was used to passivate the exposed Au source and drain electrodes rather than to functionalize the In_2O_3 recognition surface (Wang et al., 2022). The dodecanethiol

condition in the present study altered the exposed metal interface but did not add a structural encapsulation layer over the complete FET.

The later MBS and aptamer steps convert the prepared surface into a molecular-recognition interface. The MBS crosslinker contains an N-hydroxysuccinimide (NHS) ester and a maleimide group. Activated NHS esters can react with primary amines to form amide linkages. This allows the amine functionality supplied by APTES to anchor the crosslinker while leaving the maleimide available for subsequent thiol coupling. Maleimides undergo addition with sulfhydryl groups to form thioether-linked succinimide products, providing the connection to the thiolated DNA aptamer (Koniev and Wagner, 2015). In the Wang platform, the cortisol aptamer carried a 5' thiol modification and was covalently immobilized on the amino-silanized In_2O_3 surface through MBS (Wang et al., 2022).

Once attached to the surface, the aptamer provides molecular recognition and couples target binding to the electrical response of the FET. Binding-induced changes in aptamer conformation can reposition charged regions of the DNA receptor and associated ions relative to the semiconductor surface. Changes in this local electrostatic environment can modulate conductance and allow small-molecule detection under physiological ionic-strength conditions (Nakatsuka et al., 2018; Wang et al., 2022). For the intended continuous-monitoring application, the completed aptamer-FET must do more than generate a cortisol-dependent signal at a single timepoint; the electrical state of the device and the resulting sensing response must remain interpretable as measurements are repeated over time. The present study examines the electrical stability of the FET and the pre-aptamer surface states that precede that later sensing layer. Stability after aptamer attachment and during repeated cortisol sensing remains outside the scope of this thesis.

The present study stopped before MBS attachment, aptamer immobilization, and cortisol sensing. It also did not reproduce the complete published APTES/PTMS silanization state. The preparation workflow was separated into four experimental arms: bare, DDT, PTMS + DDT, and UV + PTMS + DDT. The surface conditions did not include APTES. Therefore, the PTMS-containing conditions should be interpreted as experimental decomposition states within the broader preparation pathway rather than replicas of the published mixed APTES/PTMS interface. Chapter 4 evaluates how those processing states changed the electrical behavior of the underlying FETs before environmental exposure.

4.2 Chemical basis of the surface-preparation states

Figure 4.2 shows the four pre-aptamer surface states evaluated experimentally: Bare, DDT, PTMS + DDT, and UV + PTMS + DDT. None of these conditions contained APTES, MBS, aptamer, or cortisol. The treated conditions were separately assigned processing sequences rather than successive measurements of the same wafer piece. Their purpose was to test whether progressively different pre-aptamer processing histories changed the electrical state of the FET before environmental exposure.

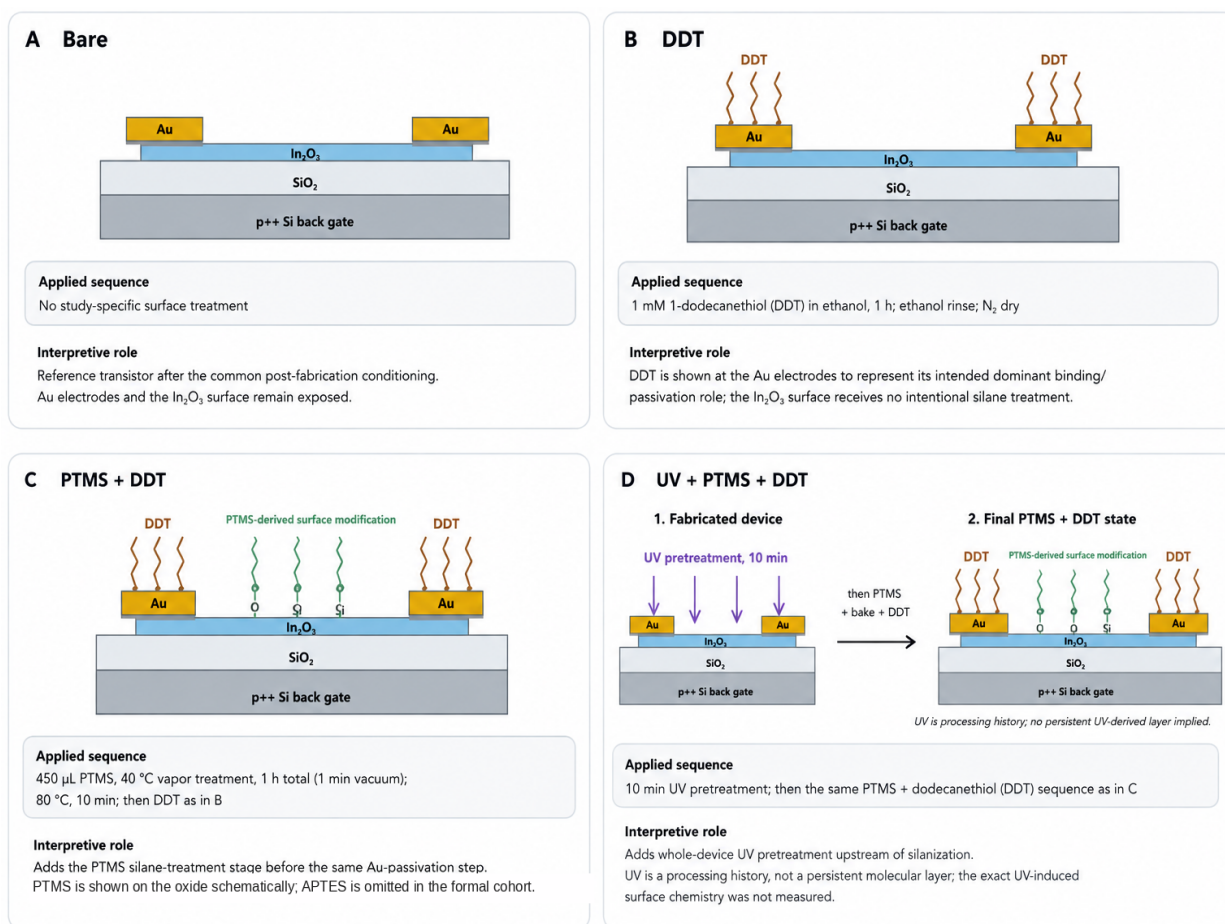


Figure 4.2. Pre-aptamer surface-preparation states evaluated in the study. (A) Bare FETs received no study-specific surface treatment after the common post-fabrication conditioning. (B) The DDT condition introduced Au-electrode passivation using 1 mM dodecanethiol in ethanol for 1 h, followed by an ethanol rinse and N_2 drying. (C) The PTMS + DDT condition added vapor-phase PTMS treatment and post-silane thermal conditioning before the same DDT treatment. (D) The UV + PTMS + DDT condition added a 10 min whole-FET UV pretreatment before the PTMS and DDT sequence used in C. The four panels represent separately assigned experimental conditions rather than sequential treatment of a single FET. Surface structures represent intended interfacial roles and are not measurements of molecular coverage, exclusive binding location, or bonding geometry.

The first treated condition was the DDT group. The DDT condition introduced electrode passivation without silane treatment of the exposed oxide surface. Long-chain alkanethiols adsorb strongly to Au and form alkanethiolate self-assembled layers, providing the chemical basis for dodecanethiol treatment of the Au source and drain electrodes (Love et al., 2005). In the

study protocol, FETs were immersed in 1 mM dodecanethiol in ethanol for 1 h, rinsed with ethanol, and dried under N₂.

The PTMS + DDT condition added organosilane processing before the same electrode-passivation step. Trimethoxy(propyl)silane (PTMS) is a trialkoxysilane containing hydrolyzable methoxy groups and a terminal propyl group. Hydrolysis and condensation reactions can support attachment and polymerization at hydroxyl-containing oxide interfaces. Quinton and Dastoor (1999) showed that PTMS interactions with aluminum- and iron-oxide surfaces included both adsorbed monomeric and condensed species and depended on the adsorption state of the surface. Their metal-oxide system does not reproduce the vapor-phase treatment or In₂O₃ surface used here, but it provides evidence that PTMS-derived oxide interfaces cannot be assumed to have a single fixed molecular organization (Quinton and Dastoor, 1999).

In the primary study, 450 µL of PTMS was placed in the vapor chamber. This amount corresponded to the PTMS quantity used in the laboratory's mixed APTES/PTMS preparation while omitting APTES. The FETs underwent 1 h of vapor treatment at 40 °C, including the initial 1 min vacuum step, followed by a 10 min bake at 80 °C and the same dodecanethiol treatment used in the dodecanethiol condition. The temperature and duration were derived from the broader Andrews-group platform workflow. The PTMS-only silane treatment was an experimental modification introduced for this thesis.

The final treated condition added a 10 min UV pretreatment before the same PTMS + DDT sequence. Ultraviolet treatment (UV) can alter oxide-surface properties relevant to subsequent silanization, although the outcome depends on the material and processing conditions. Zong et al. (2009) reported increased APTS silanization on UV-irradiated TiO₂ relative to nonirradiated regions. Their TiO₂ substrate, UV procedure, and solution-based

silanization differ from the present experiment, so this result is used only as an oxide-surface precedent and not as evidence that the same chemical change occurred on the In_2O_3 FETs studied here.

The UV treatment was also not assumed to be electrically neutral. Kim et al. (2017) used UV-related surface-energy modification during fabrication of solution-processed In_2O_3 TFTs and demonstrated that changes in interfacial surface condition were associated with changes in final FET characteristics. In that study, UV was used in a different location and at a different stage of device fabrication than in the present experiment. Therefore, it does not establish the mechanism or electrical effect of UV exposure on the completed In_2O_3 TFTs studied here. It does support treating UV as a potentially consequential processing step whose electrical effect should be measured rather than assumed to be negligible (Kim et al., 2017).

No X-ray photoelectron spectroscopy, contact-angle measurement, ellipsometry, or other direct surface-chemistry characterization was performed on the study devices. Figure 4.2 represents the intended dominant interfacial roles of the treatments schematically. The electrical analyses in this chapter compare each treated device immediately before and after its complete assigned processing sequence. Differences among the treated conditions reflect complete processing sequences and are not interpreted as isolated measurements of any single surface reaction.

4.3 Surface-processing cohort and analysis

The surface-processing analysis evaluated the immediate electrical response of the FETs in the three treated processing sequences before environmental exposure began. The analyzed conditions were DDT, PTMS + DDT, and UV + PTMS + DDT. Bare FETs were not included in

the PRE-to-POST processing analysis because they did not undergo a study-specific surface-treatment event.

Each treated processing sequence contained six wafer pieces distributed across the three parent wafers, with two pieces contributed by each parent wafer. Two FETs were tested on each piece, giving a total of 12 FET observations per processing sequence and 36 FETs across 18 treated wafer pieces. The wafer piece was the independent processing unit because both FETs on a piece underwent the same surface-treatment sequence. The two FETs were retained as device-level subsamples rather than treated as independent treatment replicates. Parent wafer was retained as a fabrication block because pieces originating from the same wafer shared their earlier fabrication history.

Each processed piece had also been assigned to a future air or PBS condition for the experiment described in Chapter 5. Those assignments were used as device identifiers for traceability. Exposure was not included as a factor in the present analysis because all PRE and POST processing measurements were collected before the environmental exposure began.

For each FET, processing response was calculated from the electrical measurement obtained immediately before its assigned complete processing sequence and the measurement obtained immediately afterward. Forward and reverse constant-current threshold-voltage responses were calculated separately as $\Delta V_{th,proc} = V_{th,POST} - V_{th,PRE}$ and signed hysteresis response was calculated as: $\Delta V_{H,proc} = \Delta V_{H,POST} - \Delta V_{H,PRE}$.

Positive values indicated a shift toward more positive voltage after processing and negative values indicated a shift toward more negative voltage. For strictly positive metrics, including fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$, processing response was calculated as: $\log_{10}(Y_{POST} / Y_{PRE})$.

A value of zero indicates no PRE-to-POST change. Positive values indicate an increase after processing and negative values indicate a decrease. Model estimates and descriptive summaries on the log scale were back-transformed where useful for interpretation and reported as POST/PRE ratios. No arbitrary constant was added when a value was zero, nonpositive, unavailable, or invalid for logarithmic transformation.

Metric extraction and leakage QC followed the criteria defined in Chapter 2. Forward and reverse constant-current V_{th} were retained only when their local threshold-crossing criteria were satisfied. Signed hysteresis required valid thresholds in both sweep directions. Fixed-bias $|I_{DS}|$ at $V_{GS} = +30$ V was retained for channel-response analysis only when $|I_G| < |I_{DS}|$ at that bias. Numerical SS and I_{on}/I_{off} values were still retained when their numerical extraction criteria were satisfied, and leakage flags were recorded separately. Square-root V_{th} was retained only when both the transfer-fit and output-behavior criteria defined in Chapter 2 were satisfied and was treated as a secondary threshold metric.

The primary between-sequence analysis used FET-level processing responses in linear mixed-effects models. Processing sequence and parent wafer were included as fixed effects, and wafer piece was included as a random intercept to account for the two FET measurements obtained from the same processed piece. This model retained the FET-level information while accounting for the nested experimental structure.

Since the PTMS vapor chamber could accommodate only three wafer pieces at a time, each PTMS-containing processing sequence was completed in two three-piece processing loads. Once assigned to a load, the same wafer pieces remained grouped through the subsequent applicable processing steps. For PTMS + DDT, the pieces remained grouped through PTMS treatment, post-silane thermal conditioning, and DDT treatment. For UV + PTMS + DDT, the

same grouping was maintained through UV treatment, PTMS treatment, post-silane thermal conditioning, and DDT treatment. Batch A contained the W1-air, W2-PBS, and W3-air pieces, whereas Batch B contained the W1-PBS, W2-air, and W3-PBS pieces. Each processing load contained one piece from each parent wafer.

Processing batch was treated as a composite load factor rather than as an isolated measure of PTMS-chamber effects. A retrospective sensitivity analysis was performed to determine whether the observed processing responses were strongly associated with load structure. Within each PTMS-containing condition, independently processed wafer-piece responses were analyzed using processing batch and parent wafer as predictors. A second sensitivity analysis compared PTMS + DDT with UV + PTMS + DDT while including processing batch and parent wafer, and a Processing sequence $\times$ Processing batch interaction was examined to determine whether the between-sequence response depended strongly on load. Because each treatment contained only three independent wafer pieces per processing batch, these analyses were considered exploratory robustness checks and did not replace the primary mixed-effects analysis.

Three pairwise comparisons were evaluated for each response metric:

PTMS + DDT - DDT

UV + PTMS + DDT - PTMS + DDT

UV + PTMS + DDT - DDT.

The first comparison evaluated the difference between DDT treatment alone and the sequence containing PTMS before DDT. The second evaluated the difference associated with the complete UV-containing sequence relative to PTMS + DDT. The third directly compared the least and most extensively processed treated conditions. Since each condition represents a

complete realized processing history, these contrasts were interpreted as comparisons among processing sequences, not as chemically isolated effects of PTMS or UV.

Models were fit by restricted maximum likelihood (REML). Small-sample inference for fixed effects and model-based contrasts used the Kenward-Roger approximation (Kenward & Roger, 1997). Holm correction was applied across the three pairwise processing-sequence comparisons separately within each electrical response metric (Holm, 1979). Model-estimated contrasts were reported with pointwise 95% confidence intervals and Holm-adjusted P values.

Continuous electrical responses were supplemented by descriptive operational outcomes because processing could affect whether a metric remained extractable without producing a meaningful continuous value. These outcomes included forward and reverse 10- μ A threshold validity, fixed-bias $|I_{DS}|$ validity, numerical SS availability, SS leakage flags, numerical I_{on}/I_{off} availability, I_{on}/I_{off} leakage flags, gate-dependent output behavior, **saturation-qualified output status**, fixed-bias R_{IG} status, and square-root V_{th} reportability. Operational outcomes were summarized descriptively because the number of independent wafer pieces per processing sequence was too small for stable binary-outcome inference.

4.4 Immediate electrical response to surface preparation

4.4.1 DDT processing

Dodecanethiol was the least extensive of the three treated processing sequences and served as the first treated condition in the progressive surface-preparation analysis. Twelve FETs distributed across six wafer pieces had electrical measurements collected immediately before and after treatment. Two pieces were represented from each parent wafer, with two FETs measured

on each piece. The air and PBS device identifiers indicated only the condition assigned for the later longitudinal experiment; environmental exposure had not begun at either measurement.

Processing did show changes in the devices. Complete PRE and POST transfer, gate-current, and output characteristics for all 12 DDT-treated FETs are provided in Supplemental Data S2. An example FET is shown in Figure 4.3. This device retained clear forward and reverse transfer gate modulation and saturation-like output behavior after treatment, but the transfer characteristics shifted between the PRE and POST measurements.

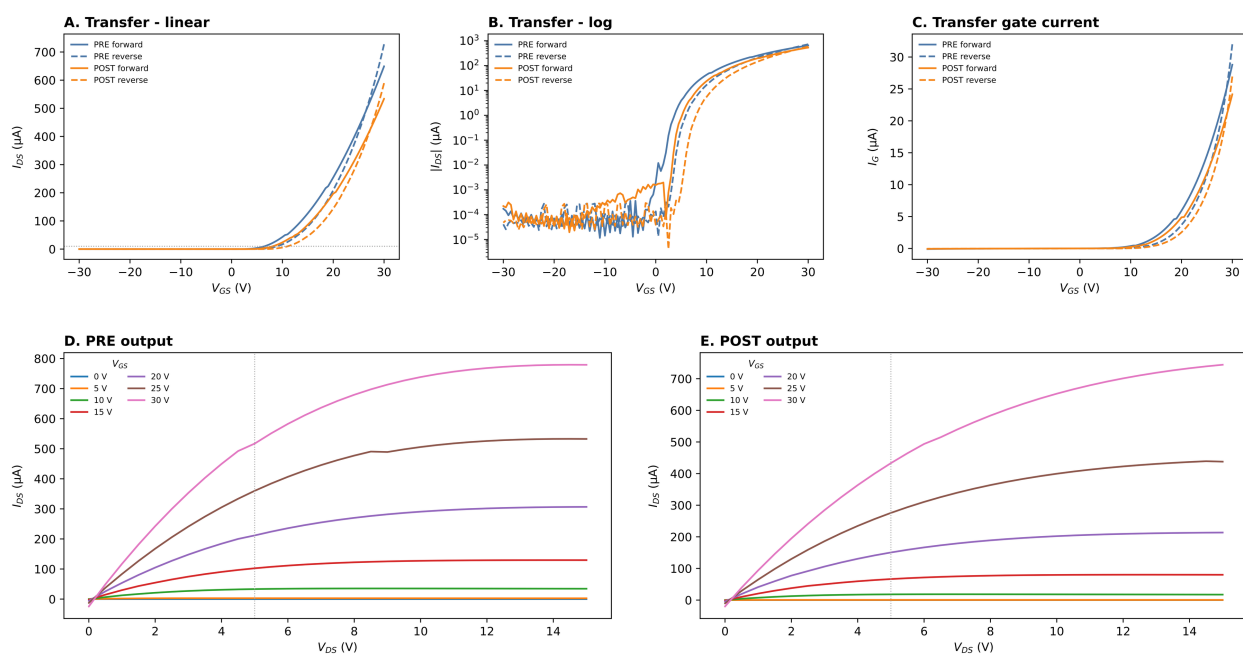


Figure 4.3. Electrical response before and after DDT processing. Forward and reverse transfer characteristics are shown on linear and logarithmic $|I_{DS}|$ scales together with the corresponding gate-current response and PRE and POST output characteristics for DDT-treated device air4. The device retained gate-dependent transfer and output characteristics after processing while exhibiting a measurable shift in its transfer response. The air identifier denotes the environmental condition assigned for the subsequent longitudinal experiment; environmental exposure had not begun at either measurement. Panels A-C show the linear transfer, logarithmic transfer, and transfer gate-current responses, respectively; panels D and E show the PRE and POST output families. In A-C, blue denotes PRE and orange POST, with solid and dashed curves denoting forward and reverse sweeps. In D-E, output-family colors denote gate-source voltage: 0 V blue, 5 V orange, 10 V green, 15 V red, 20 V purple, 25 V brown, and 30 V pink.

The paired threshold responses were heterogeneous across the 12 FETs (Table 4.1). Mean forward constant-current V_{th} changed by $+0.22 \pm 2.44$ V, while reverse V_{th} changed by -0.70 ± 2.93 V. Signed hysteresis changed by -0.93 ± 2.86 V. These small cohort-average changes occurred alongside substantially larger FET-level variation. Forward V_{th} responses ranged from -2.44 to $+5.91$ V, reverse V_{th} responses ranged from -4.64 to $+5.19$ V, and signed-hysteresis responses ranged from -4.31 to $+6.20$ V. The direction of the response was not uniform across the DDT-treated FETs.

The same heterogeneity was visible when the responses were summarized by parent wafer. Mean forward V_{th} changed by $+0.98$ V on W1, -0.01 V on W2, and -0.30 V on W3. Mean reverse V_{th} changed by -1.12 V on W1, $+0.94$ V on W2, and -1.93 V on W3. Signed hysteresis decreased on W1 and W3 but increased slightly on W2. The small overall threshold and hysteresis responses were not the result of a common directional shift across all three fabrication wafers. These findings are summarized in Appendix C.

Table 4.1. Immediate paired electrical response to DDT processing.

Metric	PRE mean $\pm$ SD	POST mean $\pm$ SD	Paired change mean $\pm$ SD	Equivalent POST/PRE
Forward V_{th}	7.60 $\pm$ 2.87 V	7.83 $\pm$ 2.85 V	+0.22 $\pm$ 2.44 V	—
Reverse V_{th}	12.69 $\pm$ 3.03 V	11.99 $\pm$ 2.61 V	−0.70 $\pm$ 2.93 V	—
Signed hysteresis	5.09 $\pm$ 3.35 V	4.17 $\pm$ 1.31 V	−0.93 $\pm$ 2.86 V	—
Fixed-bias $ I_{DS} $	487.3 $\pm$ 162.7 μ A	514.2 $\pm$ 150.7 μ A	+0.026 $\pm$ 0.132 $\log_{10}$ ratio	1.06 $\times$ (+6.1%)
SS	1.08 $\pm$ 0.50 V/dec	1.81 $\pm$ 2.02 V/dec	+0.098 $\pm$ 0.407 $\log_{10}$ ratio	1.25 $\times$ (+25.3%)
I_{on}/I_{off}	1.06E+07 $\pm$ 5.10E+06	1.05E+07 $\pm$ 9.33E+06	−0.471 $\pm$ 1.128 $\log_{10}$ ratio	0.34 $\times$ (−66.2%)
Maximum $ I_G $	25.6 $\pm$ 12.1 μ A	27.7 $\pm$ 12.0 μ A	+0.043 $\pm$ 0.131 $\log_{10}$ ratio	1.10 $\times$ (+10.5%)

Note. Values are descriptive FET-level summaries from 12 FETs nested within six independently treated wafer pieces. Threshold voltage and signed hysteresis are reported as arithmetic POST – PRE changes. Fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ are summarized as $\log_{10}(\text{POST/PRE})$ responses, with geometric-mean POST/PRE ratios shown for interpretation. Numerical SS and I_{on}/I_{off} values were retained separately from their local leakage flags as defined in Chapter 2. The FET-level summaries are descriptive; between-sequence inference accounts for parent wafer and FET nesting within wafer pieces in Section 4.5.

The current-related metrics showed comparatively small average changes after DDT treatment. Fixed-bias $|I_{DS}|$ had a mean paired log response of $+0.026 \pm 0.132$, corresponding to a geometric-mean POST/PRE ratio of 1.06. Parent-wafer ratios were similarly close to one at 1.10 $\times$ for W1, 1.02 $\times$ for W2, and 1.06 $\times$ for W3. Maximum $|I_G|$ also changed little overall, with a geometric-mean POST/PRE ratio of 1.10. No DDT-treated FET exceeded the study-specific $R_{IG} = 0.10$ warning level at $V_{GS} = +30$ V either before or after processing.

There was greater variability in the lower-current transfer metrics. Numerical SS had a geometric-mean POST/PRE ratio of 1.25, but the parent-wafer responses differed substantially: 2.67 $\times$ on W1, 0.96 $\times$ on W2, and 0.77 $\times$ on W3. The SS fitting region was leakage-flagged in 10/12 PRE measurements and 7/12 POST measurements. I_{on}/I_{off} had a cohort geometric-mean

POST/PRE ratio of 0.34 but showed extreme FET-level variability, including both large decreases and increases. Its extraction region was leakage-flagged in 12/12 PRE measurements and 11/12 POST measurements. These low-current metrics were retained descriptively but were interpreted cautiously because changes in the measured low-current floor could not be attributed to channel current alone.

Operationally, all 12 DDT-treated FETs retained valid forward and reverse 10- μ A thresholds, valid fixed-bias $|I_{DS}|$ measurements, gate-dependent output behavior, and saturation-like output characteristics after processing. Square-root V_{th} was reportable in 11/12 FETs both before and after treatment, with paired PRE/POST values available for 10/12 FETs. The paired square-root threshold change was $+0.50 \pm 2.35$ V and did not indicate a large common threshold shift.

DDT processing preserved back-gated operation across the cohort but produced heterogeneous electrical changes among the individual FETs and parent wafers. The most stable cohort-level response was observed in fixed-bias $|I_{DS}|$, while threshold, hysteresis, SS, and especially I_{on}/I_{off} varied more strongly among FETs. The DDT condition served as the treated reference sequence for the between-processing comparison in Section 4.5.

4.4.2 PTMS + DDT processing

The second treated arm added PTMS processing before the same DDT electrode-passivation step. Twelve FETs distributed across six independently treated wafer pieces were included in the PTMS + DDT cohort. Two pieces were represented from each parent wafer, with two FETs measured on each piece. Within each parent wafer, one piece was processed in Batch A and the other in Batch B. The air and PBS identifiers reflected only the environmental

condition assigned for the later longitudinal experiment; environmental exposure had not begun at either PRE or POST measurement.

Similar to the DDT cohort, processing did show changes in the devices. Complete PRE and POST transfer, gate-current, and output characteristics for all 12 PTMS + DDT treated FETs are provided in Supplemental Data S2. An example device is shown in Figure 4.4. The example PTMS + DDT device shows the pattern that predominated across this cohort: a shift of the transfer response toward more negative gate voltage with preserved gate modulation and output behavior.

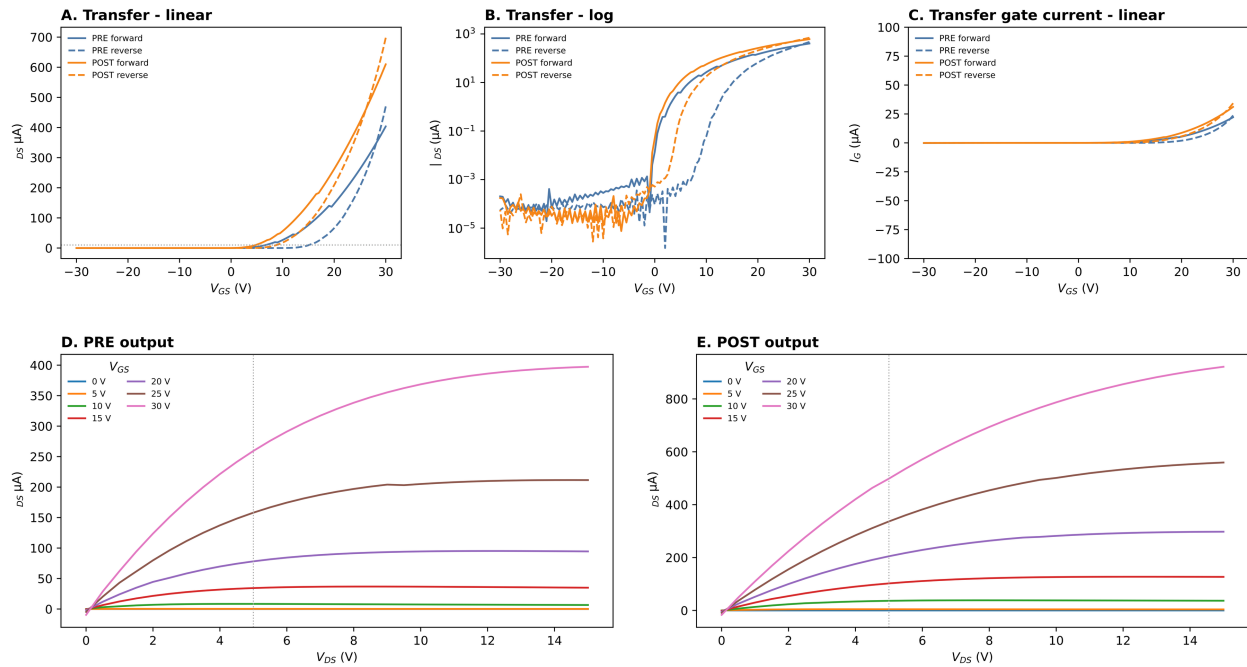


Figure 4.4. Electrical response before and after PTMS + DDT processing. Forward and reverse transfer characteristics are shown on linear and logarithmic $|I_{DS}|$ scales together with the corresponding gate-current response and PRE and POST output characteristics for PTMS + DDT treated device air3. The device retained gate-dependent transfer and output characteristics after processing while both forward and reverse transfer responses shifted toward more negative gate voltage. The air identifier denotes the environmental condition assigned for the subsequent longitudinal experiment; environmental exposure had not begun at either measurement. Panels A-C show the linear transfer, logarithmic transfer, and transfer gate-current responses, respectively; panels D and E show the PRE and POST output families. In A-C, blue denotes PRE and orange POST, with solid and dashed curves denoting forward and reverse sweeps. Output-family colors in D-E follow Figure 4.3.

The full cohort showed a predominantly negative threshold response (Table 4.2). Forward constant-current V_{th} changed by -2.15 ± 2.10 V, with 10/12 FETs shifting toward more negative gate voltage. Reverse V_{th} changed by -4.37 ± 2.89 V, again with 10/12 FETs shifting negatively. Signed hysteresis decreased by -2.22 ± 2.19 V and decreased in 10/12 FETs. The individual responses were not identical. Forward V_{th} changes ranged from -6.37 to $+0.34$ V, reverse V_{th} changes ranged from -7.58 to $+1.06$ V, and signed-hysteresis changes ranged from -5.53 to $+1.15$ V. However, unlike the DDT condition, most PTMS + DDT FETs changed in the same direction for each of these threshold-related measures.

The parent-wafer summaries showed the same broad pattern. Mean forward V_{th} changed by -3.18 V on W1, -1.66 V on W2, and -1.62 V on W3. Mean reverse V_{th} changed by -6.04 , -4.28 , and -2.80 V, respectively, while signed hysteresis decreased by -2.86 , -2.62 , and -1.17 V. Therefore, the negative threshold and hysteresis responses were not confined to one parent wafer. Since each parent wafer contributed one piece to each processing load (Batch A and Batch B), each parent-wafer summary averaged across both processing batches. Processing-load sensitivity was evaluated in Section 4.5.

Table 4.2. Immediate paired electrical response to PTMS + DDT processing.

Metric	PRE mean $\pm$ SD	POST mean $\pm$ SD	Paired change mean $\pm$ SD	Equivalent POST/PRE
Forward V_{th}	8.59 $\pm$ 2.71 V	6.44 $\pm$ 1.82 V	-2.15 $\pm$ 2.10 V	—
Reverse V_{th}	14.64 $\pm$ 3.22 V	10.27 $\pm$ 1.89 V	-4.37 $\pm$ 2.89 V	—
Signed hysteresis	6.05 $\pm$ 2.28 V	3.83 $\pm$ 1.00 V	-2.22 $\pm$ 2.19 V	—
Fixed-bias $ I_{DS} $	392.9 $\pm$ 184.3 μ A	571.3 $\pm$ 159.0 μ A	+0.182 $\pm$ 0.132 $\log_{10}$ ratio	1.52 $\times$ (+52.1%)
SS	1.28 $\pm$ 0.80 V/dec	0.84 $\pm$ 0.19 V/dec	-0.138 $\pm$ 0.216 $\log_{10}$ ratio	0.73 $\times$ (-27.2%)
I_{on}/I_{off}	5.85E+06 $\pm$ 6.38E+06	1.24E+07 $\pm$ 8.70E+06	+0.383 $\pm$ 0.753 $\log_{10}$ ratio	2.42 $\times$ (+141.8%)
Maximum $ I_G $	22.7 $\pm$ 18.3 μ A	950.3 $\pm$ 3164.2 μ A	+0.473 $\pm$ 0.778 $\log_{10}$ ratio	2.97 $\times$ (+197.4%)

Note. Values are descriptive FET-level summaries from 12 FETs nested within six independently treated wafer pieces. Threshold voltage and signed hysteresis are reported as arithmetic POST – PRE changes. Fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ are summarized as $\log_{10}(\text{POST/PRE})$ responses, with geometric-mean POST/PRE ratios shown for interpretation. Numerical SS and I_{on}/I_{off} values were retained separately from their local leakage flags as defined in Chapter 2. The POST maximum- $|I_G|$ summary includes one isolated extreme gate-current point in FET air2 at $V_{GS} = -30$ V; this point was retained in the prespecified metric and documented separately in the QC record. FET-level summaries are descriptive; between-sequence inference accounts for parent wafer and FET nesting within wafer pieces in Section 4.5.

The negative threshold shift was accompanied by an increase in drain-to-source current at the common fixed gate bias. Fixed-bias $|I_{DS}|$ had a mean paired log response of $+0.182 \pm 0.132$, corresponding to a geometric-mean POST/PRE ratio of 1.52. Ten of 12 FETs showed increased fixed-bias $|I_{DS}|$ after processing. This direction was also present at the parent-wafer level: geometric-mean POST/PRE ratios were 1.87 $\times$ for W1, 1.42 $\times$ for W2, and 1.32 $\times$ for W3. The increase in fixed-bias $|I_{DS}|$ occurred across all three fabrication wafers rather than being driven by a single wafer.

Numerical SS decreased overall, with a mean paired $\log_{10}$ response of -0.138 ± 0.216 and a geometric-mean POST/PRE ratio of 0.73. Nine of 12 FETs had a lower numerical SS after processing. Parent-wafer ratios were similarly consistent at 0.74 $\times$ for W1, 0.72 $\times$ for W2, and

0.72× for W3. However, the selected SS fitting region was leakage-flagged in 9/12 PRE measurements and all 12 POST measurements. The numerical SS values were retained according to the Chapter 2 extraction rules, but the extensive local leakage flags limit interpretation of the fitted low-current region as an uncontaminated channel response.

I_{on}/I_{off} increased on average but was much less directionally consistent. The cohort geometric-mean POST/PRE ratio was 2.42, while individual FET ratios ranged from approximately 0.04× to 46.85×. Parent-wafer ratios also differed substantially, from 0.75× on W1 to 5.77× on W2 and 3.28× on W3. In addition, the I_{on}/I_{off} extraction region was leakage-flagged in 11/12 PRE measurements and all 12 POST measurements. I_{on}/I_{off} was retained as a description of the measured transfer-current range but was not used as evidence for a uniform change in channel switching behavior.

Maximum $|I_G|$ also required separate qualification. The geometric-mean POST/PRE ratio was 2.97, but this summary was strongly influenced by a single isolated gate-current excursion in the POST sweep of FET air2 at $V_{GS} = -30$ V. The point was retained because maximum $|I_G|$ was a prespecified metric rather than removed after inspection. Importantly, the same measurement did not show elevated leakage at the fixed $V_{GS} = +30$ V analysis point: air2 had $R_{IG} = 0.056$ POST, and all 12 PTMS + DDT FETs remained at or below the study-specific $R_{IG} = 0.10$ warning level at +30 V. The isolated maximum- $|I_G|$ excursion did not invalidate the fixed-bias $|I_{DS}|$ measurement.

Operationally, all 12 PTMS + DDT FETs retained valid forward and reverse 10-μA thresholds, valid fixed-bias $|I_{DS}|$ measurements, gate-dependent output behavior, and saturation-like output characteristics after processing. Square-root V_{th} was reportable in all 12 FETs before

and after processing. Its paired change was -1.67 ± 2.21 V, providing a secondary threshold measure in the same overall direction as the primary constant-current threshold response.

The PTMS + DDT sequence produced a more directionally consistent electrical response than DDT alone while preserving the measurements required for subsequent longitudinal characterization. Negative threshold responses, reduced signed hysteresis, increased fixed-bias $|I_{DS}|$, and lower numerical SS were observed across all three parent wafers, although individual FET responses remained variable and the low-current metrics were frequently leakage-flagged. Whether the PTMS + DDT response differed from DDT after accounting for parent wafer and FET nesting within wafer pieces is evaluated formally in Section 4.5. The possible contribution of the two sequential processing loads is also examined there as a sensitivity analysis.

4.4.3 UV + PTMS + DDT processing

The final treated condition added a 10 min UV pretreatment before the same PTMS + DDT processing sequence. Twelve FETs distributed across six independently treated wafer pieces were included in the UV + PTMS + DDT cohort. Two pieces were represented from each parent wafer, with two FETs measured on each piece. Within each parent wafer, one piece was processed in Batch A and the other in Batch B. The air and PBS identifiers reflected only the environmental condition assigned for the later longitudinal experiment; environmental exposure had not begun at either PRE or POST measurement.

As in the above conditions, processing did show changes in the devices. Complete PRE and POST transfer, gate-current, and output characteristics for all 12 UV + PTMS + DDT treated FETs are provided in Supplemental Data S2. An example device is shown in Figure 4.5. The

device retained forward and reverse gate modulation and recognizable gate-dependent output behavior after processing.

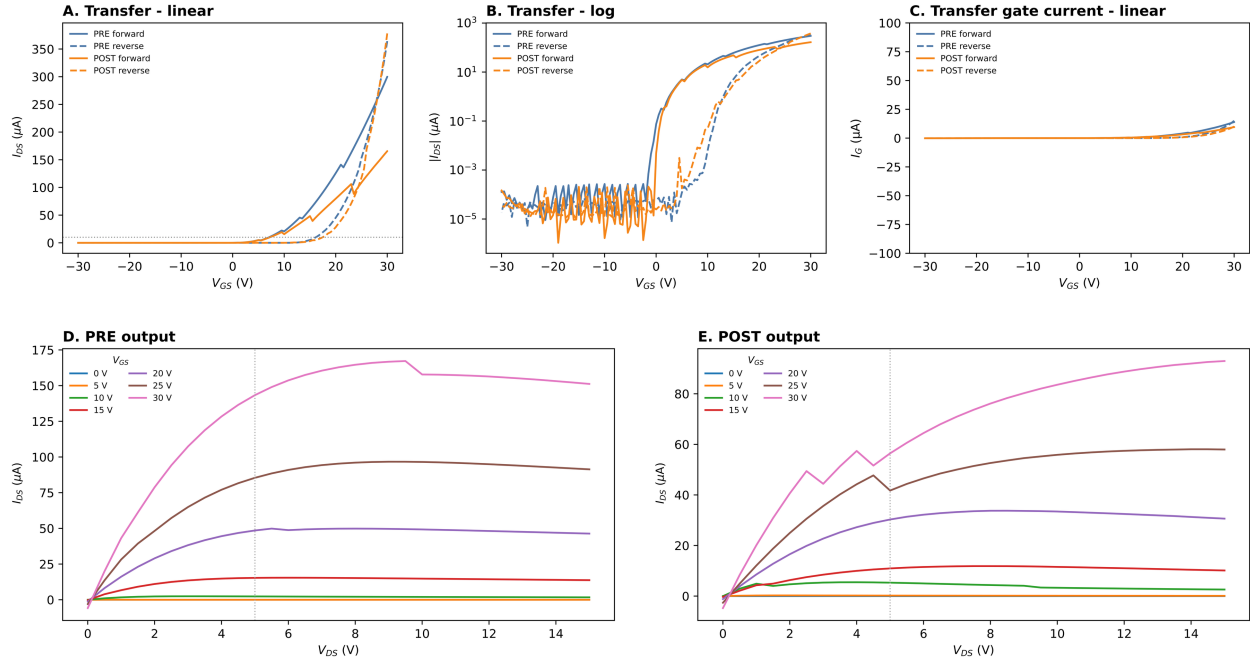


Figure 4.5. Electrical response before and after UV + PTMS + DDT processing. Forward and reverse transfer characteristics are shown on linear and logarithmic $|I_{DS}|$ scales together with the corresponding gate-current response and PRE and POST output characteristics for UV + PTMS + DDT-treated device air1. The device retained gate-dependent transfer and output characteristics after processing while fixed-bias I_{DS} decreased and the transfer response changed relative to PRE. The air identifier denotes the environmental condition assigned for the subsequent longitudinal experiment; environmental exposure had not begun at either measurement. Panels A-C show the linear transfer, logarithmic transfer, and transfer gate-current responses, respectively; panels D and E show the PRE and POST output families. In A-C, blue denotes PRE and orange POST, with solid and dashed curves denoting forward and reverse sweeps. Output-family colors in D-E follow Figure 4.3.

The paired responses for the full UV + PTMS + DDT cohort are summarized in Table 4.3. The threshold response was not uniform. Mean forward constant-current V_{th} changed by $+1.13 \pm 3.66$ V and reverse V_{th} by $+2.33 \pm 5.67$ V. Seven of 12 FETs shifted toward more positive gate voltage in each sweep direction, while the remaining FETs shifted negatively. Signed hysteresis increased by $+1.20 \pm 2.83$ V on average and increased in 8/12 FETs. The FET-level range was wide: forward V_{th} responses ranged from -5.43 to $+7.18$ V, reverse V_{th} responses

ranged from -3.90 to $+12.75$ V, and signed-hysteresis responses ranged from -2.74 to $+5.64$ V.

The positive cohort means did not represent a uniform positive shift across the processed FETs.

Table 4.3. Immediate paired electrical response to UV + PTMS + DDT processing.

Metric	PRE mean $\pm$ SD	POST mean $\pm$ SD	Paired change mean $\pm$ SD	Equivalent POST/PRE
Forward V_{th}	6.46 ± 2.64 V	7.59 ± 2.47 V	$+1.13 \pm 3.66$ V	—
Reverse V_{th}	12.00 ± 3.40 V	14.33 ± 3.70 V	$+2.33 \pm 5.67$ V	—
Signed hysteresis	5.54 ± 2.18 V	6.73 ± 2.25 V	$+1.20 \pm 2.83$ V	—
Fixed-bias $ I_{DS} $	490.4 ± 213.1 μ A	316.3 ± 180.4 μ A	-0.220 ± 0.339 $\log_{10}$ ratio	$0.60\times$ (-39.8%)
SS	0.91 ± 0.52 V/dec	1.72 ± 0.98 V/dec	$+0.270 \pm 0.325$ $\log_{10}$ ratio	$1.86\times$ ($+86.1\%$)
I_{on}/I_{off}	$8.36E+06 \pm 9.42E+06$	$8.03E+06 \pm 1.19E+07$	$+0.011 \pm 0.631$ $\log_{10}$ ratio	$1.02\times$ ($+2.5\%$)
Maximum $ I_G $	28.2 ± 18.1 μ A	18.9 ± 7.2 μ A	-0.126 ± 0.283 $\log_{10}$ ratio	$0.75\times$ (-25.2%)

Note. Values are FET-level descriptive summaries from 12 FETs nested within six treated wafer pieces. The 12 FETs are not independent treatment replicates; treatment-level inference is performed using the mixed-effects analysis in Section 4.5. Threshold voltage and signed hysteresis are reported as arithmetic POST - PRE changes. Fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ are summarized as $\log_{10}(\text{POST/PRE})$ responses, with the corresponding geometric-mean ratio shown for interpretation. Numerical SS and I_{on}/I_{off} values are retained separately from their local leakage flags as defined in Chapter 2.

The parent-wafer summaries showed additional heterogeneity. Mean forward V_{th} changed by $+2.18$ V on W1 and $+1.51$ V on W2 but by -0.30 V on W3. Mean reverse V_{th} changed by $+4.21$ V on W1, $+3.31$ V on W2, and -0.54 V on W3. Signed hysteresis likewise increased by $+2.03$ V on W1 and $+1.80$ V on W2 but changed by only -0.24 V on W3. Thus, the cohort-average positive threshold and hysteresis responses were more evident on W1 and W2 than on W3.

The two independently treated pieces within each parent wafer also did not necessarily respond in the same direction. On W1, the two piece-level mean forward V_{th} responses were -0.40 and $+4.75$ V, with corresponding reverse responses of $+0.51$ and $+7.91$ V. On W2, the two pieces changed by -1.73 and $+4.75$ V in forward V_{th} and by -3.74 and $+10.36$ V in reverse V_{th} . On W3, the corresponding piece means were $+3.00$ and -3.60 V for forward V_{th} and $+2.23$ and -3.31 V for reverse V_{th} . Because the direction of the difference between the two pieces reversed across parent wafers, the heterogeneity did not follow a simple common A-versus-B processing-load pattern.

Fixed-bias $|I_{DS}|$ decreased overall after UV + PTMS + DDT processing. The mean paired $\log_{10}$ response was -0.220 ± 0.339 , corresponding to a geometric-mean POST/PRE ratio of 0.60. Nine of 12 FETs showed lower fixed-bias $|I_{DS}|$ after processing. However, the parent-wafer responses were again not uniform. The geometric-mean POST/PRE ratio was $0.38\times$ on W1 and $0.57\times$ on W2, whereas W3 remained close to its PRE value at $1.02\times$. At the piece level, fixed-bias-current responses ranged from $0.23\times$ to $1.43\times$. The cohort decrease reflected strong reductions on several pieces rather than a common reduction of the same magnitude across the full cohort.

Numerical SS increased overall, with a mean paired $\log_{10}$ response of $+0.270 \pm 0.325$ and a geometric-mean POST/PRE ratio of 1.86. Nine of 12 FETs showed increased numerical SS. The parent-wafer responses nevertheless differed: the geometric-mean ratio was $3.22\times$ on W1, $1.04\times$ on W2, and $1.91\times$ on W3. Unlike the DDT and PTMS + DDT conditions, the frequency of local SS leakage flags decreased after the UV-containing processing sequence, from 10/12 PRE measurements to 4/12 POST measurements. The increase in numerical SS cannot be explained simply by an increased prevalence of leakage-flagged fitting windows.

$I_{\text{on}}/I_{\text{off}}$ showed little cohort-average change but very large FET-level variation. The mean paired $\log_{10}$ response was $+0.011 \pm 0.631$, corresponding to a geometric-mean POST/PRE ratio of 1.02. Individual FET ratios ranged from $0.10\times$ to $7.33\times$, and parent-wafer ratios ranged from $0.44\times$ on W1 to $3.15\times$ on W2 and $0.77\times$ on W3. The $I_{\text{on}}/I_{\text{off}}$ extraction region was leakage-flagged in all 12 FETs both before and after processing. The near-unity cohort ratio should not be interpreted as evidence that individual FET switching behavior was unchanged.

Maximum $|I_G|$ decreased on average, with a paired log response of -0.126 ± 0.283 and a geometric-mean POST/PRE ratio of 0.75. High-bias leakage remained comparatively small. One of 12 FETs exceeded the study-specific $R_{\text{IG}} > 0.10$ warning boundary at $V_{\text{GS}} = +30$ V before processing and one of 12 exceeded it afterward; no retained FET reached $R_{\text{IG}} \geq 1$ at this fixed-bias point. Fixed-bias $|I_{\text{DS}}|$ measurements remained valid in all 12 devices under the Chapter 2 criterion.

Operationally, all 12 UV + PTMS + DDT FETs retained valid forward and reverse 10- μA thresholds, valid fixed-bias $|I_{\text{DS}}|$ measurements, numerical SS and $I_{\text{on}}/I_{\text{off}}$ values, gate-dependent output behavior, and saturation-like output characteristics after processing. Device pbs6 was retained despite a comparatively adverse POST response because the prespecified primary metrics remained extractable, gate modulation and output behavior were preserved, and R_{IG} at +30 V remained below the study-specific 0.10 warning level. These operational outcomes are compared across all three treated processing sequences in Section 4.6.

The secondary square-root threshold analysis did not show the same cohort-average direction as the constant-current thresholds. Square-root V_{th} was reportable in all 12 FETs both before and after processing, with a paired PRE-to-POST change of -0.14 ± 3.95 V. The constant-current and square-root summaries did not provide the same cohort-average threshold

direction. The near-zero square-root response and its large dispersion further support treating the UV-containing threshold response as heterogeneous rather than as evidence for a uniform positive or negative threshold shift.

Because the six pieces were divided between two sequential processing loads, the UV + PTMS + DDT response was also examined for sensitivity to Batch A versus Batch B. The wafer-adjusted A-versus-B comparisons were not statistically resolved for any of the five primary electrical responses. For example, the estimated Batch A – Batch B difference was +2.65 V for forward V_{th} ($P = 0.567$) and +4.08 V for reverse V_{th} ($P = 0.581$), while the corresponding fixed-bias $|I_{DS}|$ and SS response ratios were $0.75\times$ ($P = 0.744$) and $0.74\times$ ($P = 0.381$). These analyses contained only three independently treated wafer pieces per processing load and were treated as exploratory. Tables for this analysis can be found in Appendix C.

The UV + PTMS + DDT sequence preserved the primary measurements required for continued back-gated characterization but produced a heterogeneous electrical state across FETs, wafer pieces, and parent wafers. Fixed-bias $|I_{DS}|$ decreased and numerical SS increased on average, whereas threshold voltage, hysteresis, I_{on}/I_{off} , and individual current responses varied substantially across the cohort. These patterns could not be reduced to a simple common processing-load effect. Whether the complete UV + PTMS + DDT processing response differed from PTMS + DDT or DDT after accounting for parent wafer and FET nesting within wafer pieces is evaluated formally in Section 4.5.

4.5 Comparison among surface-processing sequences

The preceding sections described the PRE-to-POST electrical response within each treated processing sequence. The mixed-effects analysis next tested whether those responses differed among DDT, PTMS + DDT, and UV + PTMS + DDT after accounting for parent wafer and the nesting of the two FETs measured on each independently treated wafer piece. Figure 4.6 shows the six observed wafer-piece responses for each sequence together with the model-adjusted response. Complete FET-level responses are retained in Appendix C so that variability between the two FETs measured on the same piece remains available for inspection.

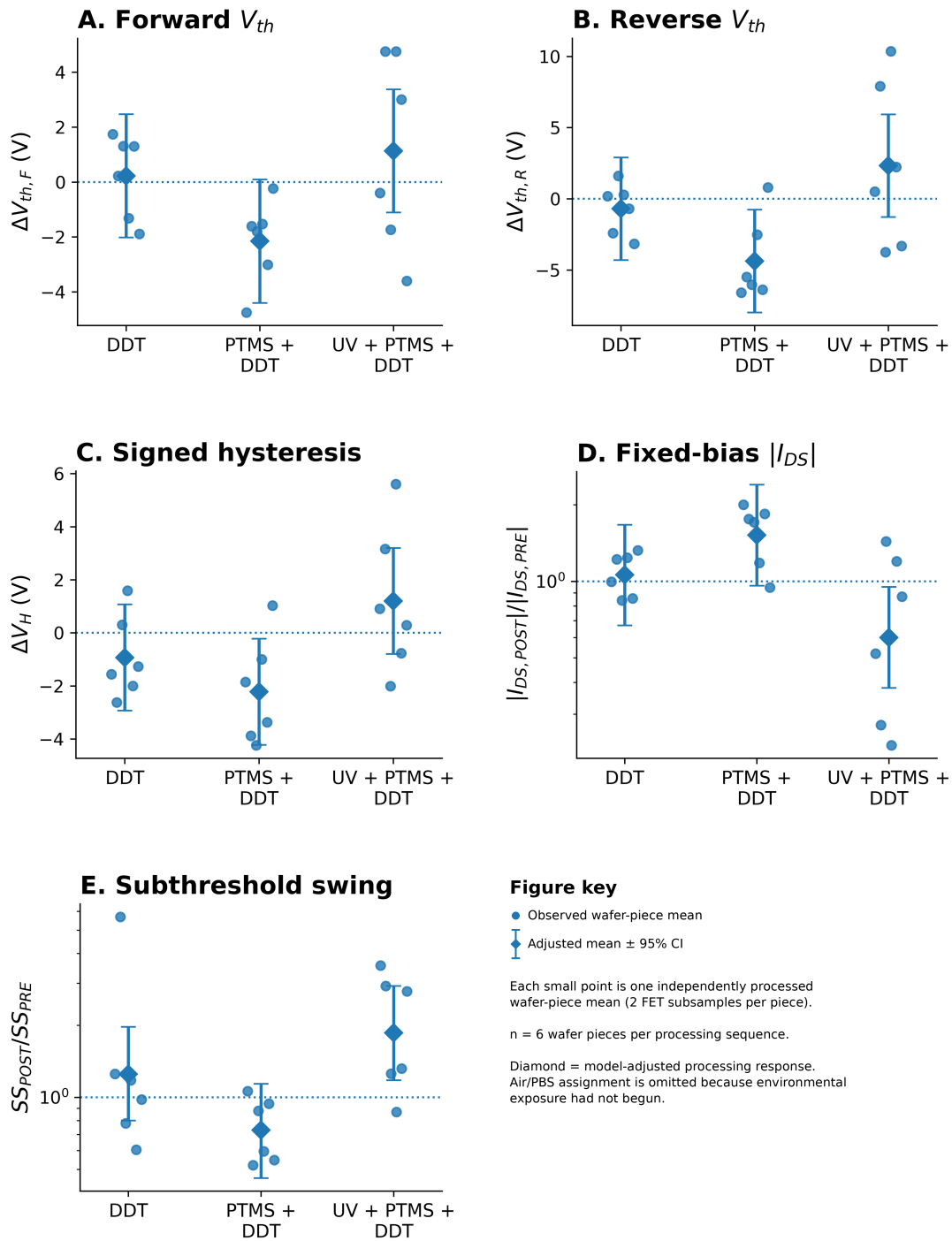


Figure 4.6. Immediate electrical response by processing sequence. Small points show the six independently treated wafer-piece means for each sequence, with each piece mean averaging the two FET subsamples measured on that piece. Diamonds show model-adjusted responses with pointwise 95% confidence intervals. Panels A–C show POST – PRE changes in forward V_{th} , reverse V_{th} , and signed hysteresis. Panels D–E show back-transformed POST/PRE responses for fixed-bias $|I_{DS}|$ and SS. The reference value is 0 V for voltage changes and 1 for ratios. Future air/PBS assignment is omitted because environmental exposure had not begun.

The parent-wafer summaries further show why the processing responses should not be interpreted from cohort averages alone (Table 4.4). DDT produced small overall threshold responses, but the direction differed among parent wafers. PTMS + DDT showed a more consistent pattern across W1, W2, and W3, with negative forward and reverse threshold responses, lower signed hysteresis, increased fixed-bias $|I_{DS}|$, and lower numerical SS on all three wafers. In contrast, UV + PTMS + DDT showed stronger positive threshold responses and reduced fixed-bias $|I_{DS}|$ on W1 and W2, while W3 remained close to zero in threshold response and near unity in fixed-bias $|I_{DS}|$. The cross-wafer summaries support the FET- and piece-level observations in Section 4.4 that the processing responses differed both in magnitude and in directional consistency among the three sequences.

Table 4.4. Parent-wafer descriptive summaries of immediate electrical response to surface processing.

Processing sequence	Parent wafer	Mean $\Delta V_{th,F}$ (V)	Mean $\Delta V_{th,R}$ (V)	Mean ΔV_H (V)	Geo. mean $ I_{DS} $ ratio	Geo. mean SS ratio
DDT	W1	+0.98	-1.12	-2.09	1.10×	2.67×
DDT	W2	-0.01	+0.94	+0.94	1.02×	0.96×
DDT	W3	-0.30	-1.93	-1.63	1.06×	0.77×
PTMS + DDT	W1	-3.18	-6.04	-2.86	1.87×	0.74×
PTMS + DDT	W2	-1.66	-4.28	-2.62	1.42×	0.72×
PTMS + DDT	W3	-1.62	-2.80	-1.17	1.32×	0.72×
UV + PTMS + DDT	W1	+2.18	+4.21	+2.03	0.38×	3.22×
UV + PTMS + DDT	W2	+1.51	+3.31	+1.80	0.57×	1.04×
UV + PTMS + DDT	W3	-0.30	-0.54	-0.24	1.02×	1.91×

Note. Values summarize PRE-to-POST processing responses within each parent wafer. Each parent-wafer row contains four FET observations from two independently treated wafer pieces. Threshold voltage and signed hysteresis are shown as mean POST – PRE changes. Fixed-bias $|I_{DS}|$ and SS are shown as geometric-mean POST/PRE ratios. Parent-wafer summaries are descriptive and are not treated as independent treatment-level inference; formal between-sequence comparisons account for parent wafer and FET nesting within wafer pieces using the mixed-effects analysis described in Section 4.3.

These parent-wafer averages still compress substantial variability among individual FETs and wafer pieces. Complete FET-level PRE-to-POST responses, wafer-piece summaries, and the underlying PRE and POST electrical metrics for each processing sequence are provided in Supplemental Data S2.

Across the three processing sequences, the omnibus processing-sequence term met the 0.05 threshold for reverse V_{th} ($P = 0.043$), fixed-bias $|I_{DS}|$ ($P = 0.026$), and SS ($P = 0.024$). The corresponding omnibus tests did not meet the 0.05 threshold for forward V_{th} ($P = 0.107$), signed hysteresis ($P = 0.062$), or I_{on}/I_{off} ($P = 0.098$). Maximum $|I_G|$ also differed among processing sequences in the omnibus model ($P = 0.036$), but this metric was retained as a supporting outcome because the PTMS + DDT cohort contained the isolated high-current gate-current point described in Section 4.4.2.

The model-adjusted responses reflected the same broad patterns observed in the descriptive FET-, piece-, and parent-wafer summaries. The DDT sequence produced small average threshold responses, with adjusted changes of +0.22 V in forward V_{th} and -0.70 V in reverse V_{th} . The PTMS + DDT sequence produced adjusted changes of -2.15 V and -4.37 V, respectively, together with lower signed hysteresis. In contrast, the UV + PTMS + DDT sequence produced adjusted forward and reverse V_{th} responses of +1.13 V and +2.33 V, respectively, with higher signed hysteresis on average. The ratio-based outcomes also differed in direction between the latter two sequences: the adjusted fixed-bias $|I_{DS}|$ response was 1.52 $\times$ after PTMS + DDT and 0.60 $\times$ after UV + PTMS + DDT, while the corresponding SS responses were 0.73 $\times$ and 1.86 $\times$.

The three pairwise processing-sequence comparisons are shown in Figure 4.7 and summarized in Table 4.5. Holm correction was applied across the three comparisons separately

within each response metric. Confidence intervals in Figure 4.7 and Table 4.5 are model-based pointwise 95% confidence intervals and the reported p -values are Holm-adjusted. A pointwise confidence interval can exclude the null even when the corresponding multiplicity-adjusted P value does not meet the 0.05 threshold.

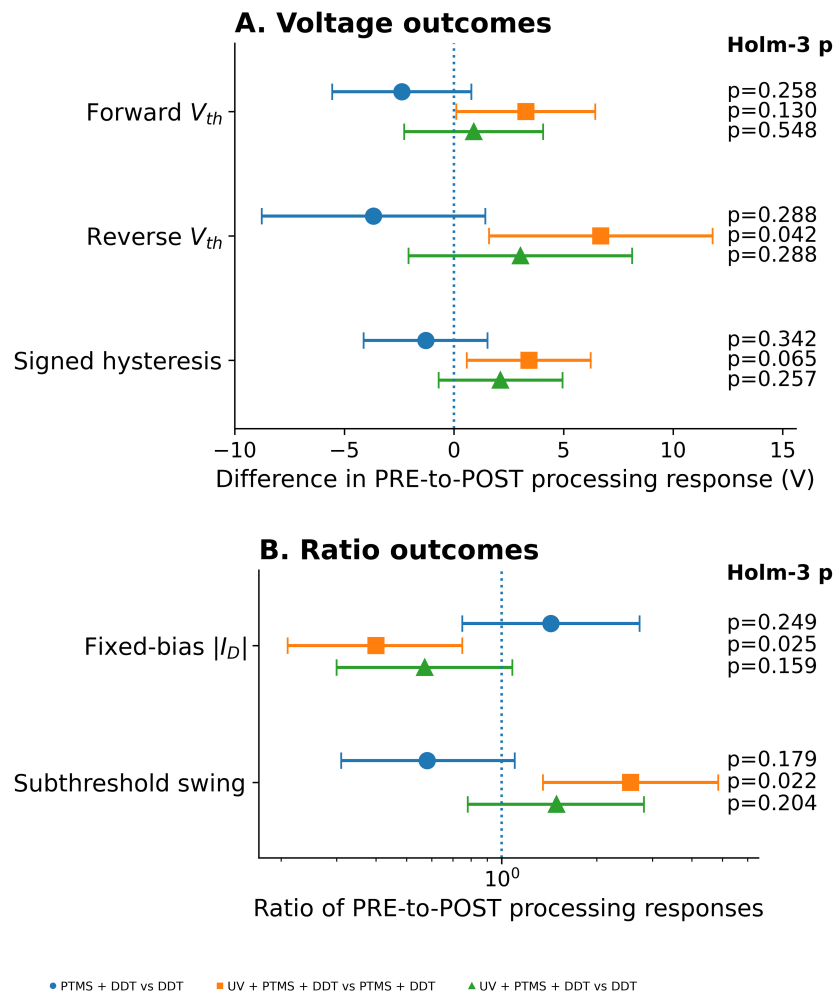


Figure 4.7. Pairwise comparisons of immediate electrical processing response. Model-estimated contrasts are shown with pointwise 95% confidence intervals for the three prespecified processing-sequence comparisons. Voltage-response contrasts are shown relative to 0 V and ratio outcomes relative to 1. P values were Holm-adjusted across the three pairwise comparisons within each response metric; confidence intervals were not multiplicity-adjusted. Blue circles denote PTMS + DDT versus DDT, orange squares denote UV + PTMS + DDT versus PTMS + DDT, and green triangles denote UV + PTMS + DDT versus DDT. Dotted vertical reference lines mark the null value for each outcome scale.

Table 4.5. Pairwise comparisons of immediate electrical processing response.

Outcome	PTMS + DDT vs DDT	UV + PTMS + DDT vs PTMS + DDT	UV + PTMS + DDT vs DDT
Forward V_{th}	-2.38 V [-5.55, +0.79]; Holm $P=0.258$	+3.28 V [+0.11, +6.45]; Holm $P=0.130$	+0.90 V [-2.27, +4.07]; Holm $P=0.548$
Reverse V_{th}	-3.67 V [-8.77, +1.43]; Holm $P=0.288$	+6.70 V [+1.60, +11.80]; Holm $P=0.042$	+3.03 V [-2.07, +8.13]; Holm $P=0.288$
Signed hysteresis	-1.29 V [-4.12, +1.53]; Holm $P=0.342$	+3.42 V [+0.59, +6.24]; Holm $P=0.065$	+2.12 V [-0.70, +4.95]; Holm $P=0.257$
Fixed-bias $ I_{DS} $	1.43× [0.75, 2.73]; Holm $P=0.249$	0.40× [0.21, 0.75]; Holm $P=0.025$	0.57× [0.30, 1.08]; Holm $P=0.159$
SS	0.58× [0.31, 1.10]; Holm $P=0.179$	2.56× [1.35, 4.85]; Holm $P=0.022$	1.49× [0.78, 2.82]; Holm $P=0.204$

Note. All three prespecified pairwise processing-sequence comparisons are included. Confidence intervals are model-based pointwise 95% confidence intervals. P values are Holm-adjusted across the three pairwise comparisons separately within each response metric. Voltage outcomes represent differences in POST – PRE response; ratio outcomes represent ratios of POST/PRE geometric-mean responses.

No comparison between PTMS + DDT and DDT remained significant after Holm correction. Forward V_{th} differed by -2.38 V between the two sequences (95% CI: -5.55 to +0.79 V; Holm-adjusted $P = 0.258$), while reverse V_{th} differed by -3.67 V (95% CI: -8.77 to +1.43 V; $P = 0.288$). The fixed-bias $|I_{DS}|$ processing response was 1.43× the DDT response (95% CI: 0.75× to 2.73×; $P = 0.249$), and the SS response was 0.58× the DDT response (95% CI: 0.31× to 1.10×; $P = 0.179$). Although the PTMS + DDT cohort showed more directionally consistent negative threshold responses, increased fixed-bias $|I_{DS}|$, and lower numerical SS descriptively, those responses were not distinguishable from DDT at the prespecified multiplicity-adjusted threshold.

The strongest separation occurred between PTMS + DDT and UV + PTMS + DDT. Relative to PTMS + DDT, the UV-containing sequence produced a +6.70 V difference in reverse V_{th} response (95% CI: +1.60 to +11.80 V; Holm-adjusted $P = 0.042$). Its fixed-bias $|I_{DS}|$

processing response was $0.40\times$ that of PTMS + DDT (95% CI: $0.21\times$ to $0.75\times$; $P = 0.025$), while its SS processing response was $2.56\times$ as large (95% CI: $1.35\times$ to $4.85\times$; $P = 0.022$).

Signed hysteresis differed by +3.42 V and forward V_{th} by +3.28 V. Their pointwise 95% confidence intervals excluded zero, but neither comparison remained significant after Holm adjustment across the three processing-sequence comparisons ($P = 0.065$ and $P = 0.130$, respectively).

Direct comparison of UV + PTMS + DDT with DDT did not identify a Holm-adjusted difference in any of the five primary electrical responses shown in Figure 4.7. The forward V_{th} response differed by +0.90 V ($P = 0.548$), reverse V_{th} by +3.03 V ($P = 0.288$), and signed hysteresis by +2.12 V ($P = 0.257$). The fixed-bias $|I_{DS}|$ response was $0.57\times$ the DDT response ($P = 0.159$), and the SS response was $1.49\times$ the DDT response ($P = 0.204$).

The between-sequence analysis did not support a monotonic electrical response to progressively more extensive processing. The strongest separation occurred between PTMS + DDT and UV + PTMS + DDT processing sequences, for which reverse V_{th} , fixed-bias $|I_{DS}|$, and SS responses differed after Holm correction. In contrast, neither PTMS + DDT versus DDT nor UV + PTMS + DDT versus DDT met the same multiplicity-adjusted threshold for the primary outcomes. The differences in reverse V_{th} , fixed-bias $|I_{DS}|$, and SS are consistent with the complete processing sequences differing across more than one feature of the measured transfer response rather than only one extracted parameter. The underlying physical origin of that difference cannot be determined from the electrical measurements alone and is considered in Chapter 6.

The PTMS-containing sequences were each completed in two three-piece sequential processing loads. The PTMS + DDT versus UV + PTMS + DDT comparison was examined for sensitivity to the A/B workflow grouping. The same pieces remained grouped through all

processing steps, so processing batch represents a composite workflow-load factor rather than an isolated PTMS- or UV-specific effect.

No primary electrical response showed a statistically resolved A versus B difference within either PTMS-containing sequence. Additionally, the direction of the UV + PTMS + DDT versus PTMS + DDT contrast was preserved within both processing loads for all five primary outcomes. No Processing sequence $\times$ Processing batch interaction met the 0.05 threshold for forward V_{th} ($P = 0.251$), reverse V_{th} ($P = 0.403$), signed hysteresis ($P = 0.754$), fixed-bias $|I_{DS}|$ ($P = 0.614$), or SS ($P = 0.291$). Including processing batch and parent wafer in the independent-piece sensitivity analysis also left the central UV versus PTMS response estimates essentially unchanged. However, the confidence intervals widened when only the six independent wafer-piece means per treatment were analyzed.

Each treatment contained only three independent pieces per processing load. Therefore, these analyses were low-power. Additionally, the same pieces remained grouped through successive processing steps, so the batch factor cannot identify whether any session difference arose from UV treatment, PTMS processing, thermal handling, timing, ambient conditions, DDT treatment, or another correlated feature of the workflow. Nevertheless, the sensitivity analysis showed that the principal PTMS + DDT versus UV + PTMS + DDT separation was present in both loads and was not attributable solely to one A or B processing session. Tables for this analysis can be found in Appendix C.

I_{on}/I_{off} and maximum $|I_G|$ were retained as supporting outcomes and were not used to define the principal between-sequence interpretation. I_{on}/I_{off} showed substantial FET-level dispersion and was associated with extensive leakage flags at the low-current extrema used for extraction. Maximum $|I_G|$ was sensitive to the isolated PTMS + DDT air2 POST gate-current

excursion described previously. Complete pairwise results for these supporting metrics, with model diagnostics and Kenward–Roger verification, are provided in Appendix C.

4.6 Post-processing operational compatibility

The continuous electrical responses in Sections 4.4 and 4.5 describe how the measured FET characteristics changed after surface processing. A separate question was whether those changes prevented the FETs from remaining suitable for the intended longitudinal study. Post-processing metric validity and operational behavior were evaluated separately from the magnitude of the continuous PRE-to-POST responses.

All 36 treated FETs retained valid forward and reverse 10- μ A constant-current threshold measurements after processing. Fixed-bias $|I_{DS}|$ at $V_{GS} = +30$ V also remained valid in all 36 FETs. All FETs retained gate-dependent output behavior and met the saturation-qualified output criterion. Thus, all 18 independently processed wafer pieces retained both selected FETs for the primary electrical measurements after surface preparation.

High-bias gate leakage remained limited at the fixed $V_{GS} = +30$ V analysis point. No DDT or PTMS + DDT FET exceeded the study-specific $R_{IG} > 0.10$ warning level after processing. One UV + PTMS + DDT FET exceeded that warning level. But no device in any processing sequence reached $R_{IG} \geq 1$. The fixed-bias $|I_{DS}|$ measurements remained valid under the Chapter 2 criterion across the complete treated cohort.

The low-current regions required greater caution. Numerical SS remained available for all 36 FETs, but the POST SS fitting region was leakage-flagged in 7/12 DDT, 12/12 PTMS + DDT, and 4/12 UV + PTMS + DDT FETs. Numerical I_{on}/I_{off} was also available for all 36 FETs, the I_{on}/I_{off} extraction region was leakage-flagged in 11/12, 12/12, and 12/12 FETs, respectively.

These flags did not remove the numerical metrics under the Chapter 2 rules, but they limit interpretation of the corresponding low-current regions as uncontaminated channel responses. Square-root V_{th} remained reportable after processing in 11/12 DDT FETs and all 12 FETs in each PTMS-containing sequence. Table 4.6 describes these outcomes.

Individual atypical responses were retained when the prespecified validity criteria remained satisfied. For example, the isolated maximum- $|I_G|$ excursion described for PTMS + DDT air2 did not invalidate the fixed-bias measurement. FET-level validity and QC summaries are provided in Appendix C, with complete device-level processing records provided in Supplemental Data S2.

Surface processing changed the electrical state of the FETs without eliminating the measurements required for the longitudinal experiment. The POST measurement characterized the electrical state immediately after surface processing. A separate T0 measurement was collected before environmental exposure and served as the device-specific reference for the analysis in Chapter 5.

Table 4.6. Post-processing operational compatibility across treated surface-preparation sequences.

Outcome	DDT POST	PTMS + DDT POST	UV + PTMS + DDT POST
Forward 10- μ A V_{th} valid	12/12	12/12	12/12
Reverse 10- μ A V_{th} valid	12/12	12/12	12/12
Fixed-bias $ I_{DS} $ valid	12/12	12/12	12/12
Gate-dependent output	12/12	12/12	12/12
Recognizable saturation-like output	12/12	12/12	12/12
$R_{IG} > 0.10$ at +30 V	0/12	0/12	1/12
$R_{IG} \geq 1$ at +30 V	0/12	0/12	0/12
Numerical SS available	12/12	12/12	12/12
SS leakage-flagged	7/12	12/12	4/12
Numerical I_{on}/I_{off} available	12/12	12/12	12/12
I_{on}/I_{off} leakage-flagged	11/12	12/12	12/12
Square-root V_{th} reportable	11/12	12/12	12/12

Note. Counts are FET-level POST outcomes from 12 FETs nested within six independently treated wafer pieces per processing sequence. Numerical SS and I_{on}/I_{off} availability are reported separately from their local leakage flags according to the criteria defined in Chapter 2. PRE and POST FET-level validity and QC summaries are provided in Appendix C, with complete device-level processing records provided in Supplemental Data S2.

4.7 Summary and transition to environmental exposure

Chapter 4 separated the immediate electrical consequences of surface preparation from the later effects of environmental exposure. All three treated sequences: DDT, PTMS + DDT, and UV + PTMS + DDT preserved back-gated operation across the treated cohort, but they did not leave devices in the same electrical state. DDT produced relatively small cohort-average

changes in threshold voltage and fixed-bias $|I_{DS}|$, but these averages occurred alongside substantial variation among individual FETs, wafer pieces, and parent wafers. PTMS + DDT produced a more directionally consistent response: most FETs shifted toward more negative threshold voltage, signed hysteresis decreased, fixed-bias $|I_{DS}|$ increased, and numerical SS decreased. These directions were also present across all three parent wafers, although the magnitude of the response remained variable among individual FETs and pieces. UV + PTMS + DDT produced a more heterogeneous response. Fixed-bias $|I_{DS}|$ decreased and numerical SS increased on average, while threshold voltage and hysteresis differed substantially among FETs, wafer pieces, and parent wafers. Complete FET-level and piece-level responses underlying these summaries are provided in Appendix C.

The mixed-effects analysis confirmed that the strongest between-sequence distinction occurred between PTMS + DDT and UV + PTMS + DDT. After Holm adjustment across all three pairwise comparisons within each response metric, the two sequences differed in reverse V_{th} response, fixed-bias $|I_{DS}|$ response, and SS response. Neither PTMS + DDT versus DDT nor UV + PTMS + DDT versus DDT met the same multiplicity-adjusted threshold for the primary outcomes. The progression from DDT to PTMS + DDT to UV + PTMS + DDT did not produce a simple monotonic electrical response as additional processing stages were introduced.

The processing-load sensitivity analysis did not resolve a common A versus B response within the PTMS-containing sequences, and the direction of the UV + PTMS + DDT versus PTMS + DDT contrast was preserved in both processing loads for all primary outcomes. Because each processing load contained only three wafer pieces per sequence and the same pieces remained grouped through successive processing steps, the analyses cannot exclude session-related variability or attribute variability to an individual processing step. However, they

do show that the principal PTMS + DDT versus UV + PTMS + DDT separation was not attributable solely to one processing load.

These electrical differences are interpreted at the level of the complete processing sequence. The experiment was not designed to isolate the molecular contribution of PTMS, DDT, or UV, and no direct surface-chemistry characterization was performed. The electrical measurements establish that the completed pre-aptamer processing histories changed the state of the FETs before environmental exposure, while the physical and chemical mechanisms underlying those changes cannot be determined from these measurements alone. Despite these changes and the substantial variability among individual FETs, all 36 treated FETs retained the primary threshold, fixed-bias-current, and output measurements required for continued longitudinal characterization. The POST measurements document the immediate electrical consequences of surface preparation. A separate T0 measurement was subsequently collected before environmental exposure and served as the device-specific reference for Chapter 5. Longitudinal air- and PBS-associated changes were evaluated relative to T0 after completion of the assigned surface-processing sequence.

Chapter 5. Longitudinal electrical response under ambient-air and PBS exposure

Chapter 4 showed that the surface-processing sequences changed the electrical state of the devices before environmental exposure began. Chapter 5 asks whether those completed surface states changed the subsequent electrical response during ambient-air or PBS exposure.

5.1 Study objective and realized longitudinal measurement window

This longitudinal study was designed to evaluate the longitudinal changes of the FETs from the four study arms (bare, DDT, PTMS + DDT, and UV + PTMS + DDT) under matched air and PBS exposure conditions. Electrical characterization was originally planned at T0, 6 h, 12 h, 24 h, 48 h, Day 3, Day 5, Day 7, Day 10, and Day 14. T0 was collected after completion of the assigned surface-processing sequence and immediately before environmental exposure, and it served as the device-specific reference for subsequent electrical changes.

The planned measurement schedule could not be completed under a single electrical configuration. T0, 6 h, and 12 h measurements were successfully acquired using the original probe-station back-gate connection. During the scheduled 24 h characterization, the back-gate probe assembly failed, including the holder-to-BNC measurement path, and required replacement. The failure produced flat transfer responses during testing. Previously functioning comparison devices outside the formal cohort showed the same loss of gate modulation under the failed configuration, supporting the conclusion that the flat responses reflected the measurement-path failure rather than simultaneous electrical failure of the experimental cohort. Quantitative analysis under the original configuration was therefore restricted to the standardized T0, 6 h, and 12 h measurements.

The 24 h handling cycle had already begun when the equipment problem was recognized and thermal conditioning had already been applied to part of the cohort. The corresponding 70 °C, 10 min treatment was completed across the remaining experimental arms to maintain a consistent scheduled handling history at that timepoint. PBS aspiration, rinse, drying, and exposure reapplication were continued at the later maintenance timepoints, but measurement-associated thermal conditioning and electrical sweeps were omitted when electrical characterization was not performed.

At Day 7, exploratory electrical characterization resumed using a temporary alternative back-gate connection. The source and drain contacts, programmed electrical sweeps, and exposure-handling procedures were otherwise maintained, but the physical gate-contact configuration had changed and the recorded gate-current channel was not reliable for leakage analysis. The two gate-contact methods were not directly validated against one another. Day 7 was therefore treated as a separate exploratory measurement phase rather than as a quantitative continuation of the T0–12 h trajectory.

The temporary setup was attempted at Day 10, but flat transfer responses recurred in both the experimental cohort and previously functioning comparison devices. Therefore, Day 10 did not yield an analyzable electrical dataset.

5.2 Experimental cohort and data availability

The standardized environmental analysis included 48 tracked FETs distributed across eight Surface $\times$ Exposure arms formed by four study arms and two exposure conditions. Each condition had three wafer pieces, one from each of the three parent wafers, with two FETs tested on each piece. The wafer piece represented the independent treatment unit, while the two FETs

on each piece were retained as device-level subsamples. Parent wafer remained identifiable throughout the analysis so that responses from the three fabrication wafers could be distinguished.

Three FETs in the UV + PTMS + DDT condition failed to produce gate-modulated transfer behavior at T0 and were replaced before the longitudinal cohort was finalized. These were the positions subsequently labeled air5, pbs5, and pbs6. For each, an alternate FET on the corresponding wafer piece was selected from a nearby device position and assigned the study identifier at T0. The replacement FETs entered the longitudinal dataset at baseline, before environmental response was evaluated. No FET was replaced after T0, regardless of subsequent electrical deterioration or metric loss.

Electrical transfer measurements were collected at T0, 6 h, and 12 h. This produced 144 scheduled device-timepoint records across the 48-device cohort. T0 was collected immediately before environmental exposure and served as the device-specific reference for change over time.

Metric availability was determined separately for each electrical outcome using the extraction and validity criteria defined in Chapter 2. A device could retain measurable gate modulation, fixed-bias $|I_{DS}|$, SS, or I_{on}/I_{off} while no longer providing a valid 10- μ A threshold crossing within the formal transfer sweep. Signed hysteresis required valid forward and reverse constant-current thresholds and therefore became unavailable whenever either directional threshold could not be extracted. Missing or invalid values were not assigned boundary values, carried forward, or imputed.

The resulting denominators differed among metrics and timepoints without changing the 48-device cohort established at T0. FET-level paired responses, wafer-piece summaries, condition-level descriptive summaries, and metric-specific quality-control classifications for the

standardized T0–12 h phase are provided in Appendix D. Complete device-level transfer characteristics and extracted electrical records are provided in Supplemental Data S3. Model outputs and sensitivity analyses are provided in Appendix E. The exploratory Day 7 analysis is provided separately in Appendix F.

5.3 Observed electrical response during the standardized T0–12 h phase

The T0, 6 h, and 12 h measurements showed different early electrical responses across the four surface conditions and between the air and PBS workflows. Figure 5.1 shows forward constant-current threshold-voltage change relative to each device’s own T0 measurement. Individual FET responses are shown together with the mean response of the two FETs measured on each independently treated wafer piece.

The clearest descriptive separation was observed in the Bare condition. Bare Air devices remained comparatively close to their T0 forward-threshold values, whereas Bare PBS devices shifted toward more positive forward threshold voltage. At 6 h, the three Bare PBS wafer-piece means were +11.36, +1.11, and +6.96 V for W1, W2, and W3, respectively, compared with –2.74, +4.89, and –0.29 V for the corresponding Bare Air pieces. By 12 h, all three Bare Air piece means remained near or below T0 (–2.40, –0.02, and –2.97 V). Among the Bare PBS pieces, valid forward-threshold values remained available for W2 and W3, with piece-level changes of +15.61 and +6.62 V, whereas a valid 12 h forward threshold was no longer available for the W1 piece.

The DDT-containing surface conditions did not show the same consistent positive forward-threshold displacement under PBS. Instead, responses varied in both direction and magnitude among wafer pieces. At 12 h, DDT PBS piece-level forward-threshold changes were

−8.87, +3.03, and +2.65 V across W1, W2, and W3. The corresponding PTMS + DDT PBS values were +2.19, +3.83, and −3.52 V. In the UV + PTMS + DDT PBS condition, the available W1 and W3 piece means were −5.12 and +1.50 V; a valid piece-level forward-threshold mean was not available for W2. Thus, the processed PBS conditions remained heterogeneous, but they did not reproduce the broadly positive Bare PBS response observed over the same interval.

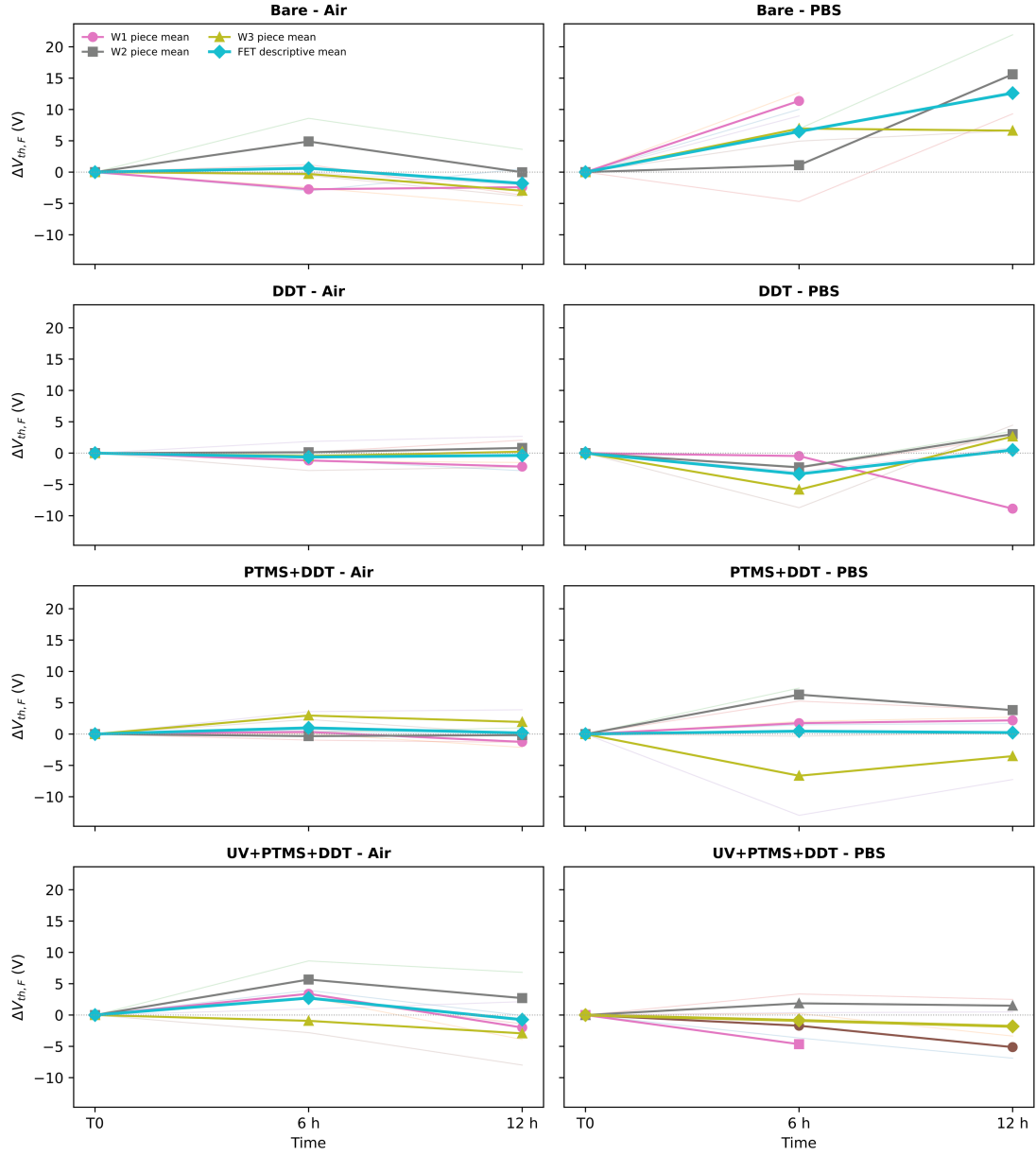


Figure 5.1. Forward constant-current threshold-voltage response during the standardized T0–12 h environmental-exposure phase. Forward-sweep threshold-voltage change is shown relative to each device’s T0 measurement for Bare, DDT, PTMS + DDT, and UV + PTMS + DDT devices maintained under ambient-air or PBS exposure. Individual FET trajectories are shown together with W1, W2, and W3 wafer-piece means, where each piece mean represents the valid FET subsamples measured on that independently treated piece. A condition-level FET mean is included as a descriptive summary. Lines connect the observed T0, 6 h, and 12 h measurements for visualization only and do not represent a fitted continuous time trend. Missing points indicate that a valid forward 10- μ A threshold could not be extracted under the prespecified criteria. The legend encodes the descriptive summaries as follows: W1 piece means are pink circles, W2 piece means gray squares, W3 piece means yellow-green triangles, and the FET descriptive mean cyan diamonds; faint thin lines show individual FET trajectories.

The device-level trajectories showed that the response was not identical across the three parent-wafer pieces or between the two FETs measured on the same piece. Figure 5.1 retains the individual FET trajectories while emphasizing the W1, W2, and W3 piece-level means. However, devices measured on the same wafer piece did not always change by the same magnitude, and responses differed among the W1, W2, and W3 pieces within a condition. Some devices remained gate modulated while a particular derived metric became unavailable, including cases in which a valid 10- μ A threshold could no longer be extracted within the formal sweep window. Metric availability and leakage-related validity are therefore considered separately in Section 5.6. Complete FET-level paired responses and wafer-piece summaries are provided in Appendix D.

Logarithmic transfer characteristics provide a complementary view of these changes (Figure 5.2). For each surface and exposure condition, the T0 baseline and 12 h transfer curves are shown for the same FETs, with the forward and reverse sweeps overlaid. The logarithmic $|I_{DS}|$ scale makes changes in the transfer transition and lower-current region more visible, while the separate forward and reverse sweeps preserve sweep-direction-dependent behavior. These examples are included to illustrate the observed electrical changes rather than to substitute for the complete cohort. Transfer and gate-current characteristics for all 48 devices are provided in Supplemental Data S3.

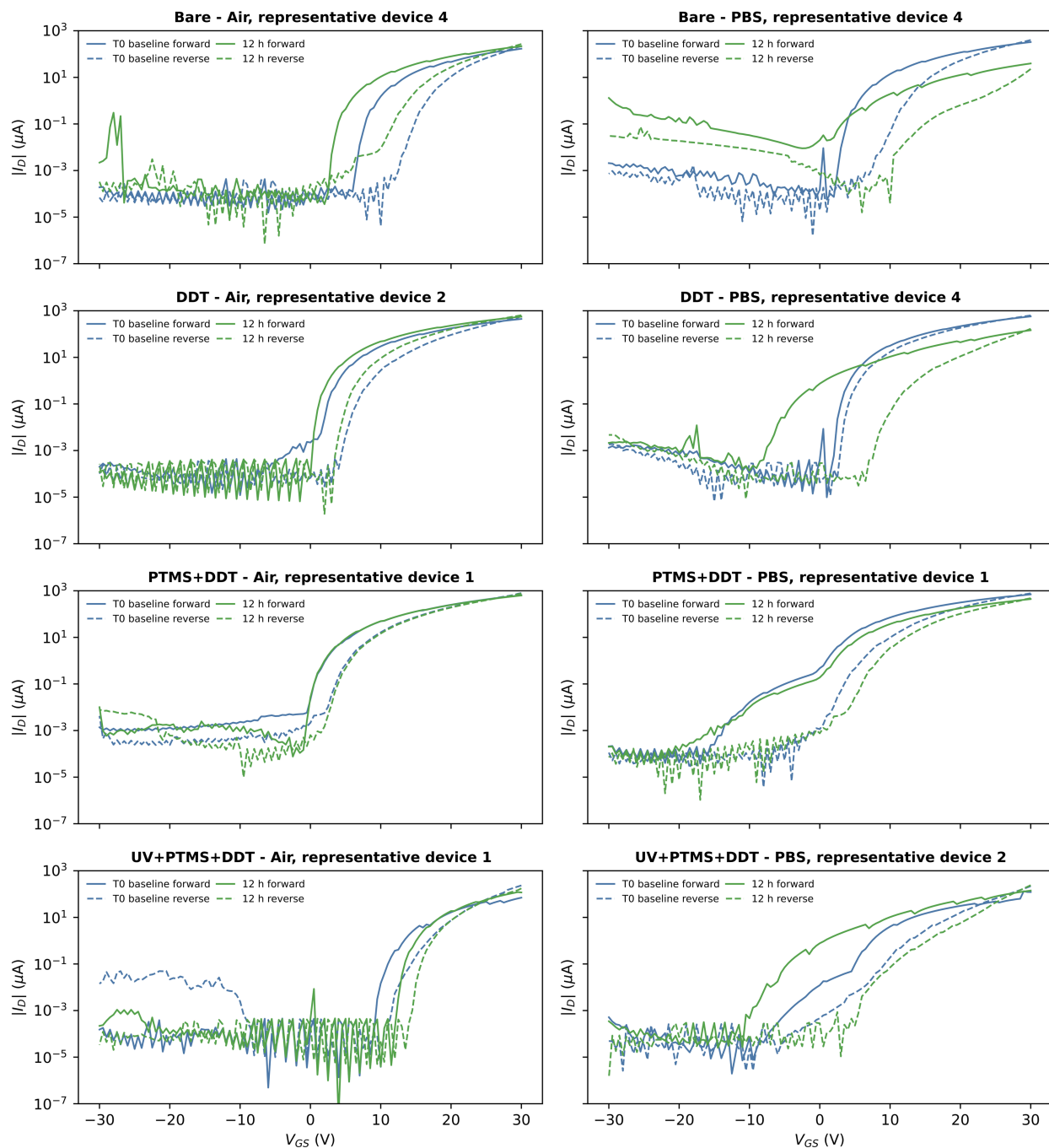


Figure 5.2. Example logarithmic transfer characteristics at T0 and 12 h. Example air- and PBS-exposed devices are shown for the Bare, DDT, PTMS + DDT, and UV + PTMS + DDT surface conditions. Within each panel, the T0 and 12 h measurements were obtained from the same tracked device. Drain-to-source current magnitude, $|I_{DS}|$, is shown on a logarithmic scale as a function of V_{GS} , with forward and reverse sweeps shown separately. The examples are provided for visualization of the observed electrical response and were not used in place of the complete cohort for statistical analysis. Complete device-level records are provided in Supplemental Data S3. Blue curves denote T0 and green curves denote 12 h; solid and dashed curves denote forward and reverse gate sweeps, respectively.

Condition-level paired descriptive summaries were calculated separately for each outcome at 6 h and 12 h and are provided in Appendix D. For each paired summary, T0 and follow-up values were calculated using the same contributing devices, so the denominator differed when a metric became unavailable. Because the two FETs measured on each wafer piece were subsamples rather than independent treatment replicates, these descriptive means were not used as independent replicates for inference. Formal Surface $\times$ Exposure comparisons are presented in Section 5.4.

5.4 Primary 12 h forward-threshold response

The prespecified primary environmental outcome was the change in forward-sweep constant-current threshold voltage at 12 h relative to each FET's T0 value: $\Delta V_{th,F}(12\text{ h}) = V_{th,F}(12\text{ h}) - V_{th,F}(T0)$.

The primary 12 h model included 41 valid FET responses from 22 independently treated wafer pieces. Surface condition, exposure condition, the Surface $\times$ Exposure interaction, and parent wafer were included as fixed effects, with wafer piece included as a random intercept to account for the two FET subsamples measured on each piece. Models were estimated by restricted maximum likelihood, with Kenward–Roger small-sample adjustment used for fixed-effect inference and 95% confidence intervals. Chamber was not entered as a separate model effect because each Surface $\times$ Exposure condition was maintained in its own dedicated chamber, making chamber identity inseparable from experimental condition in the realized design.

The forward-threshold response at 12 h depended on the combination of surface condition and environmental workflow. The overall Surface $\times$ Exposure interaction was significant, $F(3, 11.79) = 4.738$, $P = 0.0215$. The estimated PBS-minus-Air difference was

largest for Bare devices: PBS exposure was associated with an additional +12.96 V forward-threshold displacement relative to Air (95% CI, +6.56 to +19.37 V; $P = 0.00073$). In contrast, the estimated PBS-minus-Air differences were +0.45 V for DDT, +0.52 V for PTMS + DDT, and +0.08 V for UV + PTMS + DDT. The 95% confidence intervals for all three DDT-containing surface conditions spanned zero (Figure 5.3A; Table 5.1).

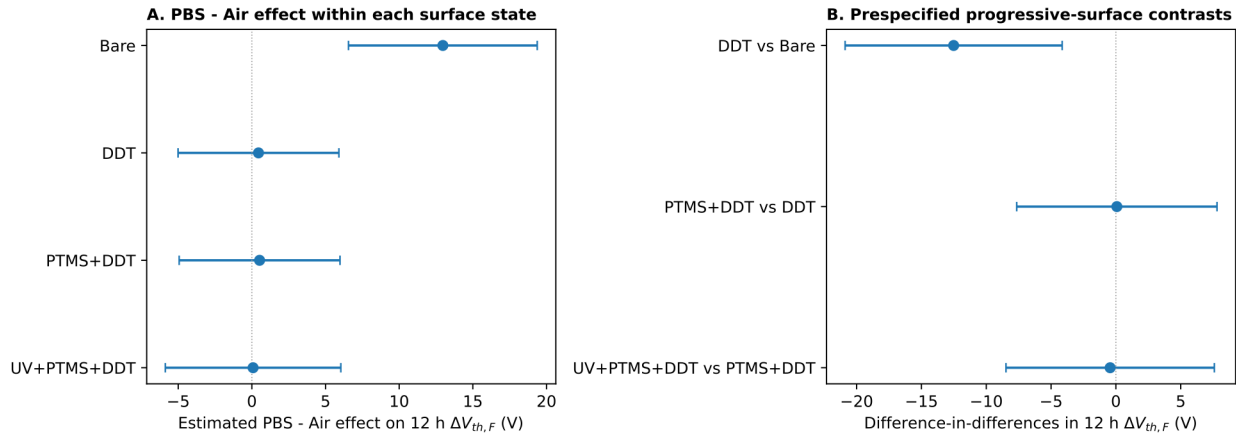


Figure 5.3. Primary Surface $\times$ Exposure effects on 12 h forward constant-current threshold-voltage change. (A) Model-estimated PBS-minus-air effects on forward ΔV_{th} at 12 h within the Bare, DDT, PTMS + DDT, and UV + PTMS + DDT surface conditions. (B) Prespecified sequential difference-in-differences contrasts comparing the PBS–air response across successive surface-preparation states. Points show model-estimated effects and horizontal error bars show 95% Kenward–Roger confidence intervals. The vertical reference line at zero represents no PBS–air difference in Panel A and no change in the PBS–air effect between successive surface states in Panel B. Holm adjustment was applied across the three primary sequential contrasts. The model included surface condition, exposure condition, the Surface $\times$ Exposure interaction, and parent wafer as fixed effects, with wafer piece included as a random intercept.

The three prespecified sequential Surface $\times$ Exposure contrasts were used to determine where the largest change in the PBS–Air response occurred along the progressive surface-preparation sequence. The DDT-versus-Bare difference-in-differences, [(PBS – Air)DDT – (PBS – Air)Bare], was –12.52 V (95% CI, –20.89 to –4.14 V; $P = 0.0067$). This contrast remained significant after Holm adjustment across the three primary 12 h comparisons (Holm-

adjusted $P = 0.0202$). The result indicates that the large PBS–Air forward-threshold separation observed in Bare devices was substantially attenuated in the DDT condition (Figure 5.3B; Table 5.1).

The subsequent progressive contrasts were much smaller. The PTMS + DDT-versus-DDT difference-in-differences was $+0.07$ V (95% CI, -7.65 to $+7.80$ V; Holm-adjusted $P = 1.000$), and the UV + PTMS + DDT-versus-PTMS + DDT contrast was -0.44 V (95% CI, -8.48 to $+7.59$ V; Holm-adjusted $P = 1.000$). Neither comparison provided evidence of an additional change in the PBS–air forward-threshold response comparable in magnitude to the Bare-to-DDT transition.

The primary analysis located the largest change in the 12 h forward-threshold response between the bare and DDT-containing surface states. The comparison reflects the complete surface-processing and exposure workflows, and chamber identity, humidification, PBS application and renewal, and other condition-specific handling were not independently separable within the realized design. The result also does not imply that the DDT-containing devices were electrically stable across every measured outcome. Reverse threshold voltage, fixed-bias $|I_{DS}|$, hysteresis, SS, I_{on}/I_{off} , gate-current behavior, and metric availability are evaluated separately in Sections 5.5 and 5.6. Complete model output, supporting contrasts, primary-model diagnostics, and sensitivity analyses are provided in Appendix E.

Table 5.1. Primary mixed-effects analysis of 12 h forward constant-current threshold-voltage change.

Comparison	Contrast	Estimate (V)	95% KR CI	KR df	KR P	Holm P
PBS–Air within surface	Bare	12.96	[6.56, 19.37]	13.35	<0.001	—
PBS–Air within surface	DDT	0.45	[−5.01, 5.91]	11.74	0.860	—
PBS–Air within surface	PTMS + DDT	0.52	[−4.94, 5.98]	11.75	0.838	—
PBS–Air within surface	UV + PTMS + DDT	0.08	[−5.88, 6.04]	10.38	0.976	—
Sequential DoD	DDT vs Bare	−12.52	[−20.89, −4.14]	12.44	0.007	0.020
Sequential DoD	PTMS + DDT vs DDT	0.07	[−7.65, 7.80]	11.84	0.984	1.000
Sequential DoD	UV + PTMS + DDT vs PTMS + DDT	−0.44	[−8.48, 7.59]	10.78	0.906	1.000

Note. The primary model included 41 valid FET responses from 22 independently treated wafer pieces. PBS-minus-air effects are reported separately within each surface condition, followed by the three prespecified sequential Surface × Exposure difference-in-differences contrasts. Estimates are reported in volts with 95% Kenward–Roger confidence intervals and corresponding *P* values. Holm-adjusted *P* values are reported for the three primary sequential contrasts. Positive within-surface estimates indicate a more positive 12 h forward-threshold response under the PBS workflow than under air. For the sequential contrasts, a negative estimate indicates attenuation of the PBS–air difference relative to the preceding surface state. Parent wafer was included as a fixed blocking factor and wafer piece as a random intercept.

5.5 Supporting electrical responses

The supporting analyses were used to determine whether the Surface × Exposure pattern observed for the primary 12 h forward-threshold outcome was also present at 6 h, in the reverse sweep, or in other electrical characteristics. Forward and reverse threshold voltage and signed hysteresis were analyzed as arithmetic changes from T0. Fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ were analyzed as $\log_{10}(\text{follow-up}/T0)$ responses. The same blocked mixed-effects structure and Kenward–Roger inference used for the primary model were applied separately to

each outcome and timepoint. These analyses were supporting rather than additional primary hypotheses, so their P values were not included in the Holm adjustment applied to the three primary forward-threshold contrasts.

At 6 h, forward threshold voltage did not yet show the Surface $\times$ Exposure separation observed at 12 h. The interaction for forward ΔV_{th} was $F(3, 13.84) = 1.729$, $P = 0.207$. The estimated PBS-minus-air differences were +5.85 V for Bare, -2.56 V for DDT, -0.53 V for PTMS + DDT, and -3.82 V for UV + PTMS + DDT. The DDT-versus-Bare difference-in-differences was -8.41 V (95% CI, -18.48 to +1.65 V; $P = 0.095$). Although this estimate was in the same direction as the primary 12 h contrast, the 6 h confidence interval included zero.

Other electrical responses showed surface-dependent differences by 6 h. Reverse threshold voltage had a Surface $\times$ Exposure interaction, $F(3, 13.64) = 6.963$, $P = 0.004$. The estimated PBS-minus-air reverse-threshold responses were +10.02 V for Bare, +7.89 V for DDT, +4.21 V for PTMS + DDT, and +1.71 V for UV + PTMS + DDT. Fixed-bias $|I_{DS}|$ also showed a Surface $\times$ Exposure interaction at 6 h, $F(3, 13.90) = 6.883$, $P = 0.005$. The estimated PBS-minus-air effects on $\log_{10}(\text{follow-up}/T0) |I_{DS}|$ were -0.69 for Bare, -0.40 for DDT, -0.27 for PTMS + DDT, and -0.04 for UV + PTMS + DDT. The Surface $\times$ Exposure interactions for signed hysteresis, SS, I_{on}/I_{off} , and maximum $|I_G|$ did not meet the 0.05 threshold at 6 h. Complete 6 h model estimates and contrasts are provided in Appendix E.

By 12 h, reverse threshold voltage remained dependent on Surface $\times$ Exposure condition, $F(3, 14.30) = 7.239$, $P = 0.003$. Its pattern differed from the primary forward-threshold result, however. A positive PBS-minus-air reverse-threshold response remained present in all four surface conditions: +12.30 V for Bare, +11.70 V for DDT, +7.18 V for PTMS + DDT, and +3.67 V for UV + PTMS + DDT (Table 5.2A). The DDT-versus-Bare difference-in-differences was

only -0.61 V (95% CI, -5.59 to $+4.38$ V; $P = 0.801$), so the Bare-to-DDT change observed for forward threshold voltage was not reproduced in the reverse sweep. The PTMS + DDT-versus-DDT contrast was -4.52 V (95% CI, -8.94 to -0.09 V; $P = 0.046$), while the UV + PTMS + DDT-versus-PTMS + DDT contrast was -3.51 V (95% CI, -7.85 to $+0.83$ V; $P = 0.104$). Because these were supporting contrasts without multiplicity adjustment, the individual P values are interpreted as descriptive evidence rather than additional confirmatory findings.

Table 5.2A. Supporting 12 h PBS–Air effects within each surface condition.

Outcome	Bare	DDT	PTMS + DDT	UV + PTMS + DDT
$\Delta V_{th,R}$ (V)	12.30 [8.37, 16.24]; P<0.001	11.70 [8.57, 14.83]; P<0.001	7.18 [4.06, 10.31]; P<0.001	3.67 [0.66, 6.69]; P=0.021
ΔV_H (signed hysteresis)	-2.78 [-10.49 , 4.93]; P=0.453	12.00 [5.37, 18.63]; P=0.002	6.80 [0.17, 13.44]; P=0.045	4.70 [-2.76 , 12.15]; P=0.194
$\log_{10}$ fixed-bias $ I_{DS} $ ratio	-1.22 [-1.48 , -0.95]; P<0.001	-0.62 [-0.89 , -0.36]; P<0.001	-0.31 [-0.58 , -0.05]; P=0.025	-0.23 [-0.48 , 0.03]; P=0.079
$\log_{10}$ SS ratio	0.24 [-0.20 , 0.69]; P=0.255	-0.21 [-0.65 , 0.24]; P=0.330	0.13 [-0.33 , 0.58]; P=0.568	0.13 [-0.31 , 0.57]; P=0.537
$\log_{10}$ I_{on}/I_{off} ratio	-2.43 [-4.18 , -0.68]; P=0.010	-0.87 [-2.62 , 0.89]; P=0.307	-1.58 [-3.33 , 0.18]; P=0.074	-0.80 [-2.55 , 0.96]; P=0.346
$\log_{10}$ maximum $ I_G $ ratio	-0.40 [-1.03 , 0.24]; P=0.200	0.20 [-0.43 , 0.83]; P=0.511	0.14 [-0.49 , 0.77]; P=0.647	0.12 [-0.51 , 0.75]; P=0.687

Note. Mixed-model estimates are shown for reverse-sweep threshold-voltage change, signed hysteresis change, fixed-bias $|I_{DS}|$ response, SS, I_{on}/I_{off} , and maximum $|I_G|$. Threshold and hysteresis outcomes are arithmetic changes from T0. Fixed-bias $|I_{DS}|$, SS, I_{on}/I_{off} , and maximum $|I_G|$ are expressed as $\log_{10}(12 \text{ h}/T0)$ responses. Values are estimated PBS-minus-air effects with 95% Kenward–Roger confidence intervals and corresponding P values. Positive voltage estimates indicate a more positive change under the PBS workflow than under air. For logarithmic outcomes, positive estimates indicate a larger follow-up-to-T0 ratio under PBS and negative estimates indicate a smaller ratio. These analyses were supporting outcomes and were not included in the Holm-adjusted primary contrast family.

Fixed-bias $|I_{DS}|$ showed the clearest supporting pattern corresponding to the primary forward-threshold result. The 12 h Surface $\times$ Exposure interaction was $F(3, 13.40) = 13.277, P < 0.001$. The estimated PBS-minus-air effects on $\log_{10}(\text{follow-up}/T_0) |I_{DS}|$ were -1.22 for Bare, -0.62 for DDT, -0.31 for PTMS + DDT, and -0.23 for UV + PTMS + DDT. The DDT-versus-Bare difference-in-differences was $+0.59 \log_{10}$ units (95% CI, $+0.22$ to $+0.97$; $P = 0.005$). Thus, the large negative PBS-associated fixed-bias-current response observed in the Bare condition was substantially smaller in the DDT condition. The subsequent PTMS + DDT-versus-DDT and UV + PTMS + DDT-versus-PTMS + DDT contrasts were smaller and their confidence intervals included zero. Among the supporting outcomes, fixed-bias $|I_{DS}|$ most clearly placed the largest change in PBS-versus-air response at the same Bare-to-DDT transition identified by the primary forward-threshold analysis.

Signed hysteresis provided a less consistent pattern. The overall 12 h Surface $\times$ Exposure interaction approached, but did not meet, the conventional significance threshold, $F(3, 12.30) = 3.369, P = 0.054$. The supporting DDT-versus-Bare difference-in-differences was $+14.77 \text{ V}$ (95% CI, $+4.63$ to $+24.92 \text{ V}$; $P = 0.008$), but this individual contrast was not part of the multiplicity-adjusted primary hypothesis family and occurred in the context of an omnibus interaction that did not meet the 0.05 threshold. Hysteresis was retained as evidence of sweep-direction-dependent differences rather than interpreted as independent confirmation of the primary result.

SS did not show evidence of either an overall exposure effect or a Surface $\times$ Exposure interaction at 12 h (interaction $P = 0.467$). I_{on}/I_{off} showed an overall exposure effect, $F(1, 14.00) = 8.854, P = 0.010$, but no Surface $\times$ Exposure interaction, $F(3, 14.00) = 0.869, P = 0.480$.

$I_{\text{on}}/I_{\text{off}}$ differed between the air and PBS workflows without evidence that the magnitude of that difference varied systematically among the four surface states. Maximum $|I_G|$ showed no evidence of a 12 h Surface $\times$ Exposure interaction ($P = 0.425$). Gate-current magnitude alone did not reproduce the surface-dependent pattern observed for forward threshold voltage and fixed-bias $|I_{\text{DS}}|$; leakage-related validity and operational behavior are considered separately in Section 5.6.

Table 5.2B. Supporting 12 h progressive Surface $\times$ Exposure contrasts.

Outcome	DDT vs Bare	PTMS + DDT vs DDT	UV + PTMS + DDT vs PTMS + DDT
$\Delta V_{\text{th,R}}$ (V)	−0.61 [−5.59, 4.38]; P=0.801	−4.52 [−8.94, −0.09]; P=0.046	−3.51 [−7.85, 0.83]; P=0.104
ΔV_{H} (signed hysteresis)	14.77 [4.63, 24.92]; P=0.008	−5.19 [−14.57, 4.19]; P=0.251	−2.11 [−12.07, 7.86]; P=0.652
$\log_{10}$ fixed-bias $ I_{\text{DS}} $ ratio	0.59 [0.22, 0.97]; P=0.005	0.31 [−0.07, 0.69]; P=0.100	0.09 [−0.28, 0.46]; P=0.617
$\log_{10}$ SS ratio	−0.45 [−1.08, 0.17]; P=0.143	0.33 [−0.30, 0.97]; P=0.281	0.01 [−0.63, 0.64]; P=0.987
$\log_{10}$ $I_{\text{on}}/I_{\text{off}}$ ratio	1.56 [−0.91, 4.04]; P=0.197	−0.71 [−3.19, 1.77]; P=0.549	0.78 [−1.70, 3.26]; P=0.510
$\log_{10}$ maximum $ I_G $ ratio	0.60 [−0.30, 1.49]; P=0.175	−0.06 [−0.96, 0.83]; P=0.886	−0.02 [−0.91, 0.88]; P=0.969

Note. Sequential difference-in-differences estimates compare the PBS-minus-air response between DDT and Bare, PTMS + DDT and DDT, and UV + PTMS + DDT and PTMS + DDT for each supporting electrical outcome. Estimates are reported with 95% Kenward–Roger confidence intervals and corresponding P values. Voltage outcomes are expressed as arithmetic changes from T0. Fixed-bias $|I_{\text{DS}}|$, SS, $I_{\text{on}}/I_{\text{off}}$, and maximum $|I_G|$ are expressed as $\log_{10}(12 \text{ h}/T0)$ responses. These were supporting analyses, and their P values were not included in the Holm adjustment applied to the primary forward-threshold contrasts.

The supporting outcomes show that the effect of surface preparation was metric dependent. Reverse threshold voltage and fixed-bias $|I_{\text{DS}}|$ both showed Surface $\times$ Exposure interactions at 6 h and 12 h, but only fixed-bias $|I_{\text{DS}}|$ reproduced the pronounced Bare-to-DDT change observed for the primary 12 h forward-threshold outcome. Reverse threshold voltage

retained a positive PBS-minus-air response across all four surface conditions, while hysteresis, SS, $I_{\text{on}}/I_{\text{off}}$, and maximum $|I_G|$ did not provide the same pattern. The primary forward-threshold result should be interpreted as one component of a multidimensional electrical response rather than as evidence that any surface condition was uniformly stable.

5.6 Leakage, metric validity, and operational-state behavior

Gate leakage and metric availability were evaluated separately from the magnitude of the continuous electrical responses. A change in one electrical metric did not necessarily mean that the complete device response had been lost. As defined in Chapter 2, the relative gate-current ratio, $R_{\text{IG}} = |I_G|/|I_{\text{DS}}|$, was used to identify regions in which gate current became large relative to drain-to-source current. $R_{\text{IG}} \leq 0.10$ was below the study-specific warning level, $0.10 < R_{\text{IG}} < 1$ indicated elevated relative gate current, and $R_{\text{IG}} \geq 1$ identified a gate-current-dominated measurement region. These criteria were applied to the portion of the transfer characteristic used for the affected metric rather than as a single criterion for whole-device failure.

The clearest exposure-associated change in relative gate current occurred at the fixed +30 V gate bias. At 6 h, none of the 24 Air FETs exceeded $R_{\text{IG}} = 0.10$ at +30 V, while 16 of 24 PBS-exposed FETs exceeded this level. No FET in either exposure condition reached $R_{\text{IG}} \geq 1$ at +30 V at 6 h. At 12 h, all 24 Air FETs remained below $R_{\text{IG}} = 0.10$, while 15 of 24 PBS-exposed FETs exceeded 0.10 and three reached or exceeded $R_{\text{IG}} = 1$. The latter consisted of one FET each in the Bare, DDT, and PTMS + DDT PBS conditions; no UV + PTMS + DDT PBS FET reached the leakage-dominated boundary at +30 V (Table 5.3).

Table 5.3. Metric availability and leakage-related QC from T0–12 h.

Surface	Exposure	Time	SS leakage flag	I_{on}/I_{off} leakage flag	$R_{IG} > 0.10$ at +30 V	$R_{IG} \geq 1$ at +30 V
Bare	Air	T0	6/6	6/6	0/6	0/6
Bare	Air	6 h	5/6	6/6	0/6	0/6
Bare	Air	12 h	4/6	6/6	0/6	0/6
Bare	PBS	T0	6/6	6/6	0/6	0/6
Bare	PBS	6 h	5/6	6/6	5/6	0/6
Bare	PBS	12 h	4/6	5/6	6/6	1/6
DDT	Air	T0	5/6	6/6	0/6	0/6
DDT	Air	6 h	3/6	6/6	0/6	0/6
DDT	Air	12 h	3/6	6/6	0/6	0/6
DDT	PBS	T0	2/6	6/6	0/6	0/6
DDT	PBS	6 h	5/6	5/6	2/6	0/6
DDT	PBS	12 h	6/6	6/6	4/6	1/6
PTMS + DDT	Air	T0	6/6	6/6	0/6	0/6
PTMS + DDT	Air	6 h	3/6	6/6	0/6	0/6
PTMS + DDT	Air	12 h	4/6	6/6	0/6	0/6
PTMS + DDT	PBS	T0	5/6	6/6	0/6	0/6
PTMS + DDT	PBS	6 h	4/6	6/6	4/6	0/6
PTMS + DDT	PBS	12 h	4/6	6/6	5/6	1/6
UV + PTMS + DDT	Air	T0	3/6	6/6	1/6	0/6
UV + PTMS + DDT	Air	6 h	5/6	6/6	0/6	0/6
UV + PTMS + DDT	Air	12 h	4/6	6/6	0/6	0/6
UV + PTMS + DDT	PBS	T0	1/6	6/6	3/6	0/6
UV + PTMS + DDT	PBS	6 h	5/6	6/6	5/6	0/6
UV + PTMS + DDT	PBS	12 h	6/6	6/6	0/6	0/6

Note. Counts are descriptive FET-level outcomes; the wafer piece remained the independent treatment unit. R_{IG} was evaluated at $V_{GS} = +30$ V. Numerical SS and I_{on}/I_{off} availability are reported separately from their corresponding leakage flags. A leakage flag identifies possible gate-current contribution within the metric-specific extraction region and does not automatically make the numerical metric unavailable. T0, 6 h, and 12 h metric-availability and leakage/QC summaries are provided in Appendix D.

The relative-leakage response was not the same across either surface condition or time. For example, five of six UV + PTMS + DDT PBS devices exceeded $R_{IG} = 0.10$ at 6 h, while none exceeded the warning level at 12 h. Relative gate current did not follow a simple progressive relationship with the extent of surface processing.

This pattern also differed from maximum $|I_G|$. As shown in Section 5.5, maximum forward-sweep $|I_G|$ did not show a 12 h Surface $\times$ Exposure interaction. R_{IG} depends on both $|I_G|$ and $|I_{DS}|$, so it can increase when $|I_{DS}|$ decreases even if absolute gate-current magnitude changes comparatively little. Maximum $|I_G|$ and R_{IG} were therefore retained as separate measures rather than interpreted interchangeably.

Metric availability also differed among the surface and exposure conditions. At 12 h, all six Bare Air devices retained valid forward and reverse 10- μ A thresholds, whereas only 3/6 Bare PBS devices retained a valid forward threshold and 2/6 retained a valid reverse threshold. Threshold availability was higher in the processed PBS conditions. Five of six DDT PBS devices and five of six PTMS + DDT PBS devices retained valid thresholds in both sweep directions. Five of six UV + PTMS + DDT PBS devices retained a valid forward threshold, and all six retained a valid reverse threshold. The larger loss of threshold extractability in Bare PBS occurred alongside the pronounced threshold displacement described in Sections 5.3–5.5, but loss of a valid threshold did not by itself indicate complete loss of device operation.

Other electrical metrics frequently remained available when a constant-current threshold could not be extracted. At 12 h, fixed-bias $|I_{DS}|$ remained valid in 5/6 Bare PBS, 5/6 DDT PBS, 5/6 PTMS + DDT PBS, and 6/6 UV + PTMS + DDT PBS devices. Numerical SS remained available in 6/6, 6/6, 5/6, and 6/6 devices, respectively. Numerical I_{on}/I_{off} remained calculable for all six devices in every Surface $\times$ Exposure condition at T0, 6 h, and 12 h. An unavailable 10- μ A

crossing therefore did not necessarily mean that the remainder of the transfer characteristic was unusable.

Leakage flags on SS and $I_{\text{on}}/I_{\text{off}}$ were also kept separate from numerical metric availability. $I_{\text{on}}/I_{\text{off}}$ leakage flags were common throughout the cohort, including at T0, because the low-current end of the transfer characteristic could approach gate-current magnitude even when absolute $|I_G|$ remained comparatively small. Numerical $I_{\text{on}}/I_{\text{off}}$ was therefore retained when its extraction criteria were satisfied, while the leakage flag separately identified possible gate-current contribution to the measured current range. The same approach was used for SS: a numerical value was retained when the fitting criteria were met, while a separate flag recorded whether gate current equaled or exceeded drain-to-source current within the selected fitting region.

Square-root V_{th} was not treated as a complete longitudinal endpoint. Numerical square-root fits could be obtained from the transfer characteristics, but the Chapter 2 reportability criterion also required corresponding output behavior to support the saturation approximation. Routine output characteristics were not collected at 6 h or 12 h, so this criterion could not be evaluated at those follow-up timepoints. Square-root V_{th} was therefore not used for the standardized T0–12 h longitudinal comparison.

The standardized early response consequently included both changes in the magnitude of extractable electrical metrics and changes in whether individual metrics remained valid or interpretable. Bare PBS showed the greatest loss of constant-current threshold extractability by 12 h, while elevated relative gate current occurred across several PBS-exposed surface conditions. Other measurements often remained available in the same devices. The longitudinal

response therefore cannot be reduced to a single binary classification of operational versus failed devices.

5.7 Robustness of the primary 12 h forward-threshold result

Four sensitivity analyses evaluated whether the primary DDT-versus-Bare difference-in-differences depended strongly on the treatment of the two FET subsamples, one parent wafer, the change-score formulation, or wafer pieces represented by only one valid FET at 12 h.

First, valid FET responses were averaged within each independently treated wafer piece and analyzed at the treatment-unit level using a blocked Surface $\times$ Exposure model with HC3 heteroscedasticity-consistent confidence intervals. The DDT-versus-Bare estimate was -12.45 V, nearly identical to the primary mixed-model estimate of -12.52 V. The 95% confidence interval widened substantially, however, from the primary interval of -20.89 to -4.14 V to -25.00 to $+0.09$ V ($P = 0.052$). The similarity of the point estimates indicates that the estimated contrast was not strongly dependent on retaining the two FET subsamples at the FET level. The wider interval reflects the limited precision available when inference is based directly on the 22 independently treated wafer pieces.

Leave-one-parent-wafer-out analyses were used as influence checks. The estimated DDT-versus-Bare contrast remained negative when W1, W2, or W3 was omitted: -11.31 V, -9.59 V, and -15.84 V, respectively. The corresponding 95% confidence intervals were -22.35 to -0.27 V, -24.53 to $+5.36$ V, and -27.70 to -3.97 V. The direction of the contrast was therefore preserved in all three reduced datasets, although its precision changed substantially and the interval crossed zero when W2 was omitted. Because each reduced analysis contained only two

parent wafers, these checks are interpreted as influence analyses rather than evidence of reproducibility across a broader wafer population.

The primary contrast also remained similar when baseline threshold voltage was handled differently. Modeling absolute 12 h forward V_{th} with the corresponding T0 forward V_{th} included as a covariate produced a DDT-versus-Bare estimate of -12.05 V (95% CI, -20.64 to -3.47 V; $P = 0.010$). Restricting the analysis to wafer pieces for which both tracked FETs retained valid 12 h forward-threshold measurements produced an estimate of -13.41 V (95% CI, -22.52 to -4.30 V; $P = 0.009$). These estimates were close to the primary -12.52 V change-score result.

Across the sensitivity analyses, the DDT-versus-Bare estimate ranged from -9.59 to -15.84 V and remained negative under every specification. The two later sequential surface contrasts remained smaller and did not show a consistent additional effect of comparable magnitude (Table 5.4). The sensitivity analyses therefore support the direction and approximate magnitude of the principal Bare-to-DDT change while also showing that precision is limited by the small number of independent wafer pieces.

Table 5.4. Sensitivity analyses for the primary 12 h forward-threshold result.

Analysis	Variant	Contrast	Inference	Estimate (V)	95% CI	<i>P</i>
Piece-level HC3	All pieces	DDT vs Bare	HC3	-12.45	[-25.00, +0.09]	0.052
Piece-level HC3	All pieces	PTMS + DDT vs DDT	HC3	+1.35	[-9.86, +12.57]	0.813
Piece-level HC3	All pieces	UV + PTMS + DDT vs PTMS + DDT	HC3	-0.39	[-11.82, +11.05]	0.947
Leave-one-wafer-out	Omit W1	DDT vs Bare	KR	-11.31	[-22.35, -0.27]	0.046
Leave-one-wafer-out	Omit W1	PTMS + DDT vs DDT	KR	-3.79	[-14.83, +7.25]	0.428
Leave-one-wafer-out	Omit W1	UV + PTMS + DDT vs PTMS + DDT	KR	+4.52	[-7.90, +16.95]	0.398
Leave-one-wafer-out	Omit W2	DDT vs Bare	KR	-9.59	[-24.53, +5.36]	0.177
Leave-one-wafer-out	Omit W2	PTMS + DDT vs DDT	KR	+0.33	[-12.70, +13.36]	0.953
Leave-one-wafer-out	Omit W2	UV + PTMS + DDT vs PTMS + DDT	KR	+1.66	[-11.29, +14.61]	0.762
Leave-one-wafer-out	Omit W3	DDT vs Bare	KR	-15.84	[-27.70, -3.97]	0.020
Leave-one-wafer-out	Omit W3	PTMS + DDT vs DDT	KR	+4.97	[-5.62, +15.57]	0.284
Leave-one-wafer-out	Omit W3	UV + PTMS + DDT vs PTMS + DDT	KR	-7.65	[-19.52, +4.21]	0.151
Baseline-covariate	12 h $V_{th,F} \sim T0 V_{th,F}$	DDT vs Bare	KR	-12.05	[-20.64, -3.47]	0.010
Baseline-covariate	12 h $V_{th,F} \sim T0 V_{th,F}$	PTMS + DDT vs DDT	KR	-0.15	[-8.10, +7.80]	0.968
Baseline-covariate	12 h $V_{th,F} \sim T0 V_{th,F}$	UV + PTMS + DDT vs PTMS + DDT	KR	-0.50	[-8.84, +7.83]	0.896
Both-FET-valid pieces	Both FETs valid	DDT vs Bare	KR	-13.41	[-22.52, -4.30]	0.009
Both-FET-valid pieces	Both FETs valid	PTMS + DDT vs DDT	KR	-2.79	[-10.86, +5.27]	0.454
Both-FET-valid pieces	Both FETs valid	UV + PTMS + DDT vs PTMS + DDT	KR	-0.23	[-8.06, +7.59]	0.948

Note. Estimates are sequential Surface $\times$ Exposure difference-in-differences with 95% confidence intervals. The piece-level HC3 analysis averaged valid FET responses within each independently treated wafer piece before fitting the blocked Surface $\times$ Exposure model with parent wafer included as a fixed effect. The leave-one-parent-wafer-out analyses are influence checks and are not interpreted as estimates of population-wide wafer reproducibility. The T0-covariate and both-FET-valid-piece analyses used the same blocked mixed-model structure as the primary analysis where applicable. Sensitivity-analysis *P* values were not included in the Holm-adjusted primary hypothesis family. Complete sensitivity-model output is provided in Appendix E.

5.8 Exploratory Day 7 electrical state under the alternative back-gate contact

Environmental exposure continued during the measurement interruption, and the devices were characterized again at Day 7 using the temporary alternative connection to the same back-gate node. Because the physical gate-contact configuration differed from the configuration used at T0, 6 h, and 12 h, Day 7 was treated as an exploratory timepoint. For descriptive context, absolute Day 7 condition means were displayed alongside the standardized early-phase summaries, but they were not interpreted as Day 7 change scores or as a continuation of the T0–12 h trajectory. Additional visual comparisons are provided in Appendix F.

The descriptive Day 7 forward-threshold values differed among the surface and exposure conditions. Mean forward V_{th} was 8.73 ± 2.85 V for Bare Air and 15.4 ± 15.0 V for Bare PBS, although only two Bare PBS devices retained an extractable forward threshold. The corresponding means were 5.36 ± 0.80 V and 5.65 ± 3.86 V for DDT Air and PBS, 7.83 ± 1.77 V and 14.3 ± 5.93 V for PTMS + DDT Air and PBS, and 7.45 ± 2.00 V and 6.54 ± 3.16 V for UV + PTMS + DDT Air and PBS.

The operational state at Day 7 differed substantially among the surface and exposure conditions. All six Bare air devices remained electrically interpretable, while only 1/6 Bare PBS devices retained an interpretable transfer response. The DDT condition retained interpretable responses in 6/6 air and 5/6 PBS devices, while PTMS + DDT retained interpretable responses in 5/6 devices under both air and PBS. The UV + PTMS + DDT condition retained 6/6 interpretable air devices but only 2/6 interpretable PBS devices. Forward constant-current threshold remained extractable in 2/6 Bare PBS, 5/6 DDT PBS, 5/6 PTMS + DDT PBS, and 2/6 UV + PTMS + DDT PBS devices. These counts are reported in Appendix F rather than as a main-text operational-count figure.

An exploratory mixed model was fitted to absolute Day 7 forward threshold voltage for devices in which that metric remained extractable (Figure 5.4). The model included 37 FET observations from 20 independently represented wafer pieces. The estimated PBS-minus-Air difference was +9.00 V for Bare (95% CI, +2.18 to +15.81 V; $P = 0.0097$), +0.55 V for DDT (95% CI, -4.49 to +5.60 V; $P = 0.830$), +7.12 V for PTMS + DDT (95% CI, +2.01 to +12.24 V; $P = 0.0063$), and -2.34 V for UV + PTMS + DDT (95% CI, -9.22 to +4.54 V; $P = 0.505$).

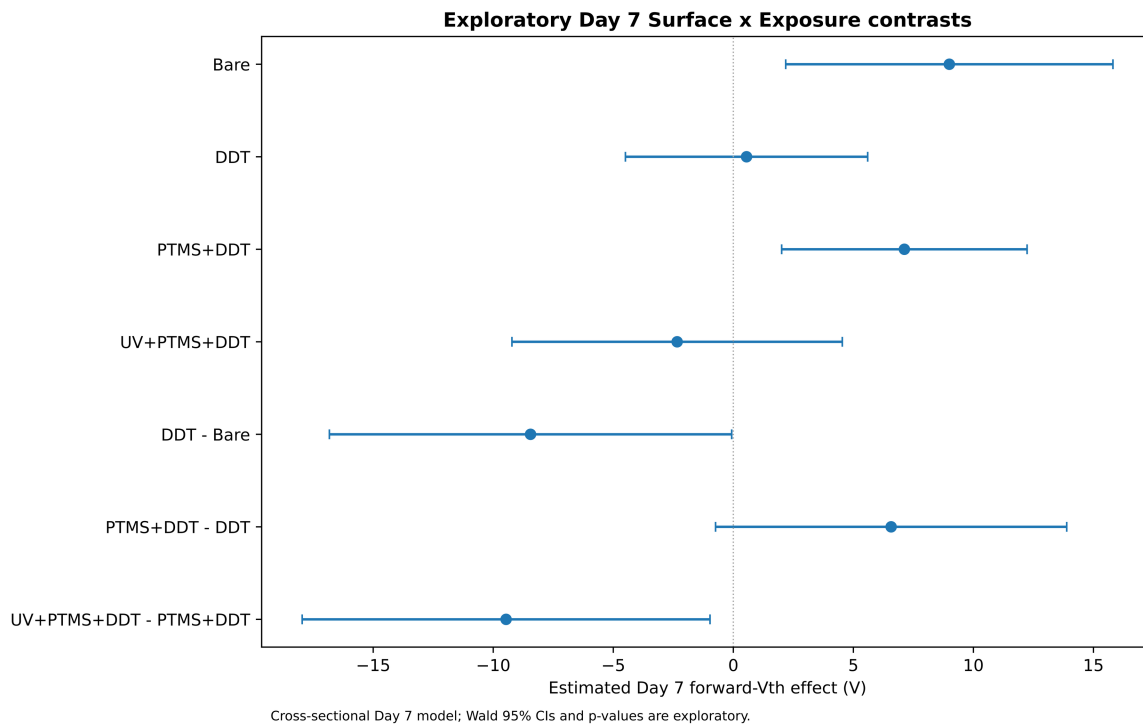


Figure 5.4. Exploratory Day 7 mixed-model Surface $\times$ Exposure contrasts for forward threshold voltage under the alternative back-gate contact configuration. Points show model-estimated Day 7 forward-threshold effects and horizontal error bars show 95% confidence intervals from the exploratory cross-sectional mixed-effects model. The upper four rows show estimated PBS-minus-Air differences within the Bare, DDT, PTMS + DDT, and UV + PTMS + DDT surface conditions. Positive values indicate a more positive Day 7 forward threshold under PBS than under Air. The lower three rows show sequential Surface $\times$ Exposure difference-in-differences comparing DDT with Bare, PTMS + DDT with DDT, and UV + PTMS + DDT with PTMS + DDT. Negative sequential estimates indicate a reduction in the PBS-minus-Air difference relative to the preceding surface state. The vertical dotted line at 0 V represents no estimated PBS-minus-Air effect or no change between successive surface states. The model included surface condition, exposure condition, their interaction, and parent wafer, with wafer piece included as a random intercept. The analysis included 37 valid FET observations from 20 wafer pieces. P values are exploratory; Holm adjustment was applied across the three sequential contrasts.

The three exploratory sequential Surface $\times$ Exposure contrasts were -8.45 V for DDT versus Bare, $+6.57$ V for PTMS + DDT versus DDT, and -9.46 V for UV + PTMS + DDT versus PTMS + DDT. The DDT-versus-Bare and UV + PTMS + DDT-versus-PTMS + DDT contrasts had unadjusted P values below 0.05, but none of the three sequential contrasts remained below the 0.05 threshold after Holm adjustment. The corresponding Holm-adjusted P values were 0.0963, 0.0963, and 0.0869. The Day 7 forward-threshold model showed surface-dependent descriptive differences but did not produce a multiplicity-adjusted sequential contrast that met the prespecified 0.05 threshold.

Supporting Day 7 outcomes also differed among electrical metrics. PBS-exposed devices generally showed more positive reverse threshold voltage and hysteresis and lower fixed-bias $|I_{DS}|$ than the corresponding air-exposed devices, but the magnitude and ordering of these differences varied among surface conditions. The fixed-bias $|I_{DS}|$ and SS models also produced singular-matrix warnings. Numerical I_{on}/I_{off} was lower under PBS in several conditions, while no sequential supporting contrast established a simple progressive surface-state hierarchy across all metrics. Gate-current magnitude and R_{IG} were not analyzed because the gate-current channel under the temporary connection was not reliable. Complete exploratory Day 7 model estimates are provided in Appendix F.

The temporary back-gate connection was attempted again at Day 10, but flat transfer responses recurred in both the experimental cohort and previously functioning comparison devices. The Day 10 attempt therefore did not produce a reliable analyzable electrical dataset, and no Day 10 values were imputed.

The Day 7 measurements provide evidence about the electrical state that remained observable after continued environmental exposure, but they do not establish quantitative seven-

day drift from the original T0 measurement. Complete Day 7 condition summaries, operational classifications, forward- and reverse-threshold models, hysteresis, fixed-bias $|I_{DS}|$, SS, and I_{on}/I_{off} analyses are provided in Appendix F. Complete device-level Day 7 records for all 48 FETs are provided in Supplemental Data S3.

5.9 Chapter summary and transition to broader implications

The standardized T0–12 h experiment showed that the early electrical response to the environmental workflow depended on the surface state present before exposure. The clearest separation occurred in forward constant-current threshold voltage. Bare PBS devices developed a substantially more positive 12 h response than Bare air devices, whereas the PBS-minus-air differences were small in the three DDT-containing conditions. The prespecified DDT-versus-Bare difference-in-differences remained below the Holm-adjusted 0.05 threshold, while the subsequent PTMS + DDT-versus-DDT and UV + PTMS + DDT-versus-PTMS + DDT contrasts did not identify additional changes of comparable magnitude.

However, the DDT-containing devices were not uniformly stable. Fixed-bias $|I_{DS}|$ showed the clearest supporting Bare-to-DDT change, while reverse threshold voltage retained a positive PBS-minus-air response across all four surface conditions. Hysteresis, SS, I_{on}/I_{off} , absolute gate-current magnitude, relative gate current, and metric availability followed different patterns. Electrical stability therefore depended on which part of the FET response was examined and could not be represented by threshold voltage or a single operational classification alone.

The sensitivity analyses preserved the direction and approximate magnitude of the primary Bare-to-DDT contrast, although uncertainty increased when inference was reduced directly to the independently treated wafer pieces. The Day 7 measurements provided a later

exploratory view but did not establish a quantitative seven-day trajectory because the physical back-gate contact had changed. They also did not support a simple progression in which each additional surface-processing stage produced greater retained stability.

Chapter 4 established that surface preparation itself changed the electrical state of the FETs before environmental exposure. Chapter 5 shows that the resulting surface state also changed how the devices responded after exposure began. These two sources of electrical variation could become superimposed in a fully assembled sensor: changes introduced during device preparation and changes that develop during subsequent operation. The present study stops before aptamer attachment and cortisol sensing, so it does not establish long-term stability of the completed sensor. Chapter 6

considers what these stage-specific electrical changes mean for interpreting a future aptamer-mediated cortisol response and what must be controlled before continuous sensing can be evaluated.

Chapter 6. Implications for continuous cortisol monitoring and conclusions

6.1 What does this thesis establish?

Wearable, real-time cortisol sensing with aptamer-functionalized In_2O_3 FETs has already been demonstrated (Wang et al., 2022). The question in this thesis is the stability of the FET underneath the recognition chemistry while the sensing interface is being assembled and while the device is exposed over time.

That question matters because a completed aptamer-FET does not have only one source of electrical change. The desired signal comes from target-dependent changes at the recognition layer. The underlying FET can also move electrically because of its surface state, its contacts, aqueous exposure, charge trapping, leakage, handling, or other device-level processes. Once an aptamer is added, the recognition layer introduces additional possible sources of drift. If these contributions occur at the same time, a changing electrical output cannot automatically be assigned to changing cortisol concentration.

The experiments in this thesis separate several of those contributions before aptamer attachment. The bare-device pilot first showed that the present FET architecture was sensitive to repeated PBS exposure and that deterioration was not a single event. Threshold position, hysteresis, drain-to-source current, output behavior, gate leakage, and metric extractability did not change together. The protocol-development experiments then showed that the physical exposure and measurement history also mattered. Droplet evaporation changed the nature of the intended PBS exposure, and thermal conditioning changed the electrical state recovered before measurement. Those observations informed the primary study design.

The next source of variation entered before environmental exposure began. Chapter 4 showed that DDT, PTMS + DDT, and UV + PTMS + DDT did not leave the FETs in equivalent

electrical states. The processing sequence itself became part of the electrical history of each device. Chapter 5 then showed that those pre-exposure states were related to what happened after the environmental workflow began. The largest early difference in the primary forward-threshold response occurred between Bare and DDT, but the other electrical outcomes did not collapse into the same simple ordering.

The main conclusion is that the device preparation and environmental exposure produced separable electrical effects, and the effect of environmental exposure depended on the state created during preparation. Stability was also metric dependent. A device could have a small forward-threshold change while developing increased leakage, a different reverse-threshold response, or loss of another metric. That distinction becomes important when these FETs are eventually interpreted as sensors rather than as FETs being characterized in isolation.

6.2 What the pilot experiments changed

The bare-device pilot established the practical problem. Bare FETs remained substantially more interpretable in air than during repeated PBS exposure. By Day 5, none of the PBS devices retained the study-defined saturation-qualified output behavior, even though several devices still showed enough transfer modulation to remain electrically interpretable. The experiment did not produce a clean time at which every device changed from functional to failed. Different parts of the electrical response deteriorated at different times.

The pilot also showed why “PBS exposure” was too broad a description of the experiment. At the first post-exposure measurements, rinsing and N₂ drying did not reliably return the PBS devices to a dry state that could be measured reproducibly. Thermal conditioning was introduced during the experiment, and the subsequent protocol-development work showed

that the selected temperature affected the recovered electrical response. The humidity experiment separately showed that an unhumidified 6 μL droplet largely ceased to represent sustained liquid exposure as it evaporated. The primary study used a standardized post-exposure thermal history and a humidified PBS chamber specifically to preserve the intended aqueous exposure.

This helps explain why the threshold response in the original bare pilot should not be forced into the same directional trajectory as the main bare cohort. The pilot later showed a negative forward-threshold displacement, whereas the standardized formal experiment produced a large positive bare PBS response relative to bare air by 12 h. However, it should be noted that the experiments did not begin from the same electrical reference state and did not use the same exposure or measurement history. The pilot T0 preceded standardization of the thermal-conditioning step, and the humidified chamber had not yet been introduced.

The broader In_2O_3 literature also shows that prolonged operation in PBS is possible, which argues against interpreting the pilot as evidence that In_2O_3 itself is intrinsically incompatible with aqueous exposure. Liu et al. reported little change in representative In_2O_3 nanoribbon transfer characteristics after immersion in undiluted PBS for as long as seven days. Zhu et al. reported operation for more than nine days in PBS in a different back-gated In_2O_3 nanoline architecture, although the transfer response shifted positively with longer exposure time (Liu et al., 2020; Zhu et al., 2021).

Those architectures were not equivalent to the devices studied here. In the Zhu platform, a patterned approximately 5 μm SU-8 layer protected the electrodes and a PDMS well confined the solution to the exposed In_2O_3 sensing region. The present study did not use that type of structural encapsulation. The exposed metal, channel region, and free-droplet geometry created a different set of aqueous interfaces (Zhu et al., 2021). More generally, passivation studies in other

BioFET architectures have shown that which electronic interfaces are exposed to ionic solution can strongly affect leakage, drift, functional yield, hysteresis, and current stability (Albarghouthi et al., 2022).

In the present platform, the pilot identified that the specific architecture and measurement workflow being used for this thesis could not be treated as electrically stable by default. It also identified the experimental variables that had to be controlled before the surface-preparation study could be interpreted.

6.3 Why did the three processing sequences behave differently?

Chapter 4 provided the first direct evidence that the pre-aptamer processing sequence itself changed the FET. All 36 treated FETs remained usable for the principal measurements immediately after processing, but the three sequences produced very different patterns. The DDT condition produced relatively small cohort-average changes with substantial variation among FETs and parent wafers. The PTMS + DDT condition produced a more directionally consistent negative threshold response, increased fixed-bias $|I_{DS}|$, and lower numerical SS. The UV + PTMS + DDT produced lower fixed-bias $|I_{DS}|$ and higher numerical SS on average, while threshold and hysteresis responses were more heterogeneous. The important question is why.

6.3.1 Why was the immediate DDT response so variable?

The location of the dodecanethiol treatment is important. In the intended surface chemistry, dodecanethiol primarily modifies the exposed Au source and drain electrodes rather than the In_2O_3 channel. Long-chain alkanethiols adsorb onto Au and organize into alkanethiolate

self-assembled layers, which is why dodecanethiol is used as the Au-passivation step in the published cortisol-FET workflow (Love et al., 2005; Wang et al., 2022).

A self-assembled alkanethiol layer on Au should not be assumed to be identical at every local electrode surface. Studies of alkanethiol SAM formation show sensitivity to the structure of the underlying Au. Godin et al. specifically found that dodecanethiol produced a much more complete organized layer on large, flat Au grains than on small-grained Au surfaces. Other microscopy studies of alkanethiols on sputtered or crystalline Au describe domains, domain boundaries, pinholes, and substrate-associated defects within SAMs (Godin et al., 2004; O'Dwyer et al., 2004; Vericat et al., 2005).

That literature does not demonstrate that the Au electrodes in this thesis had those exact defects, because Au grain structure and dodecanethiol coverage were not measured. It does make one explanation for the electrical variability plausible: dodecanethiol acts at the source/drain interface, and drain-to-source current must pass through those local contacts. If the starting Au surface, local contact geometry, or resulting dodecanethiol layer differed among FETs, the electrical consequence of treatment could differ even when the nominal chemical protocol was identical. A local contact-level effect would also be consistent with relatively small cohort-average threshold changes accompanied by larger device-to-device variation.

There are other possibilities. The dodecanethiol treatment included complete-device immersion in ethanol-based solution, rinsing, drying, and handling. The electrical experiment did not isolate molecular dodecanethiol adsorption from every correlated processing step. Contact resistance was not measured independently, and the Au surface was not characterized before or after treatment. The Chapter 4 result cannot identify the source of the variability. What it does

show is that the dodecanethiol step was not electrically neutral, even though its average effect was smaller than the response of the later sequences.

This also explains why the immediate dodecanethiol variability and the later Bare-to-DDT environmental result are not contradictory. The electrical offset introduced during processing and the protection provided during later aqueous exposure are different questions. A molecular layer could produce variable local changes when it forms and still reduce direct interaction between PBS and exposed Au during subsequent exposure. Studies in other BioFET architectures support the broader engineering principle that protecting exposed electronic interfaces can reduce solution-associated leakage and drift, although the materials and gating geometries differ from the present devices (Albarghouthi et al., 2022).

6.3.2 Why was PTMS + DDT more directionally consistent?

The PTMS + DDT condition behaved differently. Most FETs shifted toward more negative threshold voltage, fixed-bias $|I_{DS}|$ increased in most devices, and numerical SS decreased. The same broad directions were present across W1, W2, and W3. This was a more directionally consistent electrical response than dodecanethiol alone.

It would be too strong to conclude from that result that PTMS formed a uniform molecular layer. Organosilane films on oxides can be structurally complex. Quinton and Dastoor showed that PTMS adsorption on aluminum oxide depended strongly on processing conditions, while work on other trialkoxysilanes has shown that oxide-bound layers can include condensed or partial multilayer structures rather than an ideal single monolayer (Vandenberg et al., 1991; Acres et al., 2012). Those substrates and deposition conditions differ from the present vapor-

treated In_2O_3 FETs, so they establish chemical plausibility rather than the actual structure present here.

The electrical consistency can be considered in terms of which interface was being modified. Dodecanethiol primarily targeted the Au contacts. PTMS added a treatment of the exposed oxide surface before the same dodecanethiol step. In_2O_3 electrical behavior is known to be sensitive to its surface environment. Repeated operation in air, adsorption-related processes, and perturbation of surface oxygen can change mobility, conductivity, hysteresis, and threshold voltage in In_2O_3 FETs (Lee et al., 2019; Lin et al., 2026).

One plausible interpretation is that the PTMS-containing workflow changed a larger portion of the channel-facing surface than dodecanethiol alone. A surface-wide electrostatic or adsorption-related change could produce a common directional response that dominates some of the more local contact variability seen after dodecanethiol. That is a hypothesis, not a measured mechanism.

PTMS can also change oxide-surface properties relevant to later environmental interaction. In a substantially different ITO system, PTMS deposition increased contact angle and changed surface work function as deposition conditions were varied (Baek et al., 2022). This does not show that the present In_2O_3 surfaces reached the same coverage or contact angle, especially because the temperatures and substrate were different. It does establish that a propyl-terminated silane layer can alter both wetting and electronic surface properties on an indium-containing oxide (Baek et al., 2022).

The processing-load analysis also matters here. The directionally consistent PTMS + DDT response was not confined to one A or B workflow load, and no primary electrical response showed a statistically resolved A-versus-B effect within this condition. That does not prove that

the treatment was uniform, because only three independent pieces were present in each load. It does make a single anomalous processing session an insufficient explanation for the entire PTMS + DDT pattern.

The distinction between changing the electrical baseline and improving environmental stability is especially important. PTMS + DDT changed the FET more consistently during processing than DDT did, but it did not produce an additional primary 12 h PBS-versus-air improvement beyond the DDT condition. A processing step can have a large electrical effect without necessarily adding a large stability benefit over the next exposure interval.

6.3.3 Why was the UV + PTMS + DDT response different?

The UV-containing sequence is the condition that most clearly argues against treating additional processing as automatically beneficial. Immediately after processing, fixed-bias $|I_{DS}|$ decreased on average, numerical SS increased, and threshold and hysteresis responses were heterogeneous. The strongest multiplicity-adjusted Chapter 4 differences occurred between PTMS + DDT and UV + PTMS + DDT for reverse V_{th} , fixed-bias $|I_{DS}|$, and SS.

There are at least two physically plausible ways UV pretreatment could contribute to such a difference. First, UV can act directly on In_2O_3 . Lin et al. (2026) showed that UV exposure can perturb surface-oxygen interactions in ultrathin In_2O_3 , producing substantial V_{th} shifts followed by time-dependent recovery over days. The devices and UV conditions in those studies differ from the present FETs, so their mechanisms cannot simply be assigned to this experiment. They demonstrate that completed In_2O_3 should not be assumed electrically indifferent to UV exposure.

Second, UV can change the oxide surface that the silane sees next. Kim et al. showed that UV-related interface-energy modification affected the electrical characteristics of solution-processed In_2O_3 TFTs, although UV was used at a different fabrication stage. In a TiO_2 system, Zong et al. found greater subsequent APTS silanization on UV-irradiated regions than on nonirradiated regions (Kim et al., 2017; Zong et al., 2009). The latter result is not evidence that UV increased PTMS coverage on the present In_2O_3 surface. It shows why UV pretreatment could alter both the semiconductor state and the chemistry of the silanization step that follows.

The present experiment cannot separate those possibilities. The electrical measurements were collected before and after the complete UV + PTMS + DDT sequence. There was no UV-only electrical endpoint immediately before PTMS, no PTMS surface characterization, and no direct measurement of oxide adsorption or composition. The difference from PTMS + DDT could reflect direct UV modification of In_2O_3 , altered PTMS deposition, altered adsorption during the later workflow, or a combination of these effects.

The processing-batch sensitivity analysis makes one alternative explanation less compelling. The direction of the UV + PTMS + DDT-versus-PTMS + DDT difference was preserved in both processing loads across the five primary outcomes, and no Processing sequence $\times$ Processing batch interaction was resolved. With only three pieces per processing load, this analysis had little power to exclude session effects. It does show that the principal UV-versus-PTMS separation was not produced solely by one A or B load.

The three flat T0 FET positions add another reason to take the UV sequence seriously. All 12 UV + PTMS + DDT FETs used in Chapter 4 were electrically valid immediately after processing. Before the longitudinal cohort was finalized, however, three intended UV positions—the positions ultimately represented by air5, pbs5, and pbs6—produced flat transfer

responses at T0 and had to be replaced. A gate-modulated FET two device positions away on the same wafer piece was substituted in each case and received the study identifier at T0. No device was replaced after T0.

That pattern does not demonstrate that UV caused delayed failure. A flat transfer curve is a nonspecific outcome. It could result from a local channel defect, contact problem, device-to-device fabrication variability, a delayed surface change, or another local electrical problem. The fact that a nearby FET on the same processed piece remained usable also argues against interpreting these events as complete failure of the whole processed wafer piece.

Still, the fact that all three pre-cohort substitutions occurred in the UV-containing condition should not be ignored. Combined with the heterogeneous Chapter 4 response, it suggests that the UV-containing workflow may have produced a less reproducible or more locally susceptible electrical state in this experiment. The time-dependent post-UV electrical evolution reported for other In_2O_3 devices make delayed surface evolution physically plausible, but those literature results do not explain flat transfer behavior here (Lin et al., 2026).

This also affects interpretation of Chapter 5. The UV longitudinal cohort was defined only after the three unusable T0 positions were replaced. Its later continuous response metrics describe the FETs that entered environmental exposure with interpretable T0 behavior. The three original positions remain part of the processing/yield story, not the longitudinal exposure outcome. For the next study, baseline process compatibility and subsequent environmental stability should be treated as two separate endpoints.

6.4 What changed during the early environmental exposure?

The primary 12 h result identified the largest early Surface $\times$ Exposure change between Bare and DDT. Bare PBS devices shifted substantially more positively in forward V_{th} than Bare Air devices. The estimated PBS-minus-air difference was +12.96 V. The corresponding differences in the three DDT-containing states were all close to zero, and the DDT-versus-Bare difference-in-differences was -12.52 V after accounting for parent wafer and FET nesting within wafer piece.

Fixed-bias $|I_{DS}|$ provided the strongest supporting version of the same pattern. Bare PBS devices showed the largest relative reduction in fixed-bias $|I_{DS}|$, while that PBS-associated reduction was smaller in the dodecanethiol state. Because a positive threshold displacement in an n-type FET reduces gate overdrive at a fixed V_{GS} , the correspondence between the forward-threshold and fixed-current results is electrically consistent. It gives more confidence that the primary result represented a broader change in the transfer response rather than movement of one extracted number alone.

The chemical role of dodecanethiol provides one plausible explanation. The published cortisol workflow uses dodecanethiol to passivate the exposed Au source and drain electrodes (Wang et al., 2022). More generally, BioFET studies have shown that exposed electronic interfaces can become important sources of leakage and drift in ionic solutions and that electrode/passivation strategies can substantially alter aqueous stability (Albarghouthi et al., 2022). The Bare-to-DDT difference is consistent with reduced sensitivity of the exposed metal/contact region to the PBS workflow after the dodecanethiol-containing step.

That mechanism was not measured directly. The comparison also includes the complete dodecanethiol treatment and associated solvent/handling history. No contact-resistance,

electrochemical, or surface-coverage measurements identify how the Au interface changed. The appropriate claim is that entry into the dodecanethiol-treated state changed the early environmental response, with Au passivation as a plausible mechanism.

The absence of an additional primary change after PTMS is also informative. The PTMS + DDT sequence changed the immediate electrical baseline strongly in Chapter 4, but the primary forward-threshold PBS-minus-air difference at 12 h was almost the same as for DDT. One possible explanation is that the dominant early pathway captured by the forward-threshold endpoint involved an interface that was already altered by DDT. In that case, additional modification of the exposed oxide by PTMS could change the starting electrical state without producing a similarly large additional change in the first 12 h environmental response.

That interpretation remains incomplete because reverse V_{th} told a different story. The estimated 12 h PBS-minus-air reverse-threshold response remained +12.30 V in Bare and +11.70 V after DDT, then decreased to +7.18 V after PTMS + DDT and +3.67 V after UV + PTMS + DDT. The abrupt Bare-to-DDT change seen in forward V_{th} did not occur in reverse V_{th} . Signed hysteresis also behaved differently, and elevated relative gate current remained in several PBS-exposed treated conditions.

The DDT result should consequently not be summarized as “dodecanethiol stabilized the devices.” It substantially changed two important early outcomes, forward threshold and fixed-bias $|I_{DS}|$, relative to Bare. Other electrical dimensions remained sensitive to the PBS workflow.

6.5 What does the Day 7 experiment add?

Day 7 cannot be treated as the next quantitative point on the T0–12 h trajectory. The physical back-gate contact had changed, no bridging experiment established metric equivalence

between the two configurations, and the recorded gate-current channel could not be used for leakage analysis. The Day 7 measurements answer a narrower question: what electrical state was still observable after seven days of continued exposure?

The Bare PBS result was not surprising in light of the pilot and early formal data. Only one of six Bare PBS FETs remained electrically interpretable at Day 7, compared with all six Bare Air FETs. The earlier bare-device study had already shown progressive loss of interpretable and saturation-qualified behavior under repeated PBS exposure. The Day 7 observation extends the same engineering concern, although it cannot quantify seven-day drift from the original T0.

The DDT and PTMS + DDT conditions looked very different operationally. Five of six PBS FETs remained interpretable in each condition. That is consistent with the possibility that modifying the exposed Au interface reduced one important route of aqueous deterioration. In the PTMS + DDT condition, modification of the oxide surface could also have changed how water, ions, or ambient adsorbates interacted with the channel. PTMS has increased hydrophobicity on ITO in other processing systems, although the temperatures, substrate, and deposition protocol were different from those used here (Baek et al., 2022). The Day 7 data make altered interfacial wetting or adsorption plausible, not established.

The UV + PTMS + DDT condition is more difficult to interpret. All six UV Air FETs were electrically interpretable at Day 7, but only two of six UV PBS FETs were. A simple explanation that “UV processing destroyed the FETs” is inadequate. If UV treatment alone had rendered the complete cohort nonfunctional, the air devices should not have shown full operational retention. The poor PBS result instead suggests that whatever state existed after the UV-containing sequence was more vulnerable under the subsequent exposure history in at least some devices.

That interpretation is consistent with, but not proven by, two earlier observations: the heterogeneous immediate Chapter 4 response and the three UV T0 substitutions. Together, these results raise the possibility that the UV-containing sequence produced a wider distribution of local electrical states or susceptibilities. Repeated aqueous exposure, rinsing, drying, and thermal conditioning may then have amplified that heterogeneity. Surface-oxygen sensitivity after UV exposure and UV-dependent changes in oxide surface chemistry provide plausible physical routes, but the present experiment cannot select among them (Kim et al., 2017; Lin et al., 2026; Zong et al., 2009).

The Day 7 results also expose a limitation of using only the mean change among FETs that still have a valid metric. At 12 h, the UV + PTMS + DDT condition showed an approximately zero PBS-minus-air forward-threshold difference among the valid measurements. That result could look reassuring if considered alone. By Day 7, however, only two of six UV PBS FETs retained an interpretable state. A small conditional means among the FETs that remain measurable is not the same thing as high operational retention.

The reverse example occurred in PTMS + DDT. Five of six PTMS + DDT PBS FETs remained interpretable at Day 7, but the exploratory absolute forward-threshold PBS-minus-air difference was still positive. Operational yield and threshold drift describe different properties.

For that reason, Day 7 does not establish that DDT or PTMS + DDT is a fully stable long-duration condition. It does provide a useful prioritization signal. Among the experimental states studied here, DDT and PTMS + DDT retained substantially more interpretable PBS-exposed FETs than Bare or UV + PTMS + DDT. The UV-containing sequence did not provide evidence that adding another surface-processing step necessarily improved long-duration

robustness. That result is strong enough to justify a targeted repeat; it is not strong enough to assign a UV-specific failure mechanism.

6.6 What does “electrical stability” mean for this platform?

The combined experiments show why stability cannot be reduced to one V_{th} value. Forward and reverse constant-current V_{th} were intentionally retained separately. If every exposure-induced change were simply a static lateral translation of an otherwise unchanged transfer curve, the two sweep directions would be expected to provide much more similar information. Instead, the formal experiment produced a pronounced Bare-to-DDT change in forward V_{th} while the positive PBS-minus-air reverse response persisted after dodecanethiol.

Oxide-FET literature provides a reason to take that distinction seriously. Chen and Tai showed that hysteresis in amorphous oxide TFTs depends on gate-sweep timing because the response times of traps or donor-like states can overlap the measurement waveform. Lee et al. similarly reported increased hysteresis during repeated operation of solution-processed In_2O_3 FETs in air and related the behavior to trapping and interactions with polar water molecules (Chen & Tai, 2015; Lee et al., 2019). These studies do not identify the traps or kinetics in the present FETs. They show why forward and reverse sweeps can contain information about dynamic electrical state rather than being interchangeable measurements of one fixed threshold.

Fixed-bias $|I_{DS}|$ adds another dimension. Its agreement with the primary forward-threshold pattern supports the existence of a meaningful transfer-response change, but it does not make the two metrics redundant. Current at a fixed gate bias also depends on transconductance, contact behavior, channel conductance, and where the threshold sits relative to that bias. SS and I_{on}/I_{off} probe still different parts of the transfer characteristic.

Gate leakage requires another distinction. Maximum $|I_G|$ did not show the same 12 h Surface $\times$ Exposure interaction as forward V_{th} or fixed-bias $|I_{DS}|$. In contrast, the local ratio $R_{IG} = |I_G|/|I_{DS}|$ became elevated in many PBS-exposed FETs. This is possible because R_{IG} can increase when $|I_{DS}|$ falls even if absolute gate current changes relatively little. Absolute gate-current magnitude and gate current relative to the measured drain-to-source current answer different questions.

Metric availability is a stability outcome too. Bare PBS lost forward and reverse threshold extractability more often than the other conditions by 12 h, yet some of those devices retained measurable current or other numerical metrics. Conversely, a numerical SS or I_{on}/I_{off} value could remain calculable while the corresponding low-current region carried a leakage warning. A device should not be classified as completely failed merely because one extraction became unavailable, but neither should a numerical value be treated as fully interpretable merely because software can calculate it.

These distinctions become even more important at longer times because of selection. If only the devices that remain measurable contribute to a mean threshold change, worsening operational yield can disappear from that mean. The UV Day 7 result is the clearest example in this thesis.

For the next stage of this platform, electrical stability needs to include at least the retention of interpretable gate modulation, threshold extractability and drift in both sweep directions, fixed-bias $|I_{DS}|$ retention, hysteresis, gate current relative to drain-to-source current, and functional yield. Output behavior should also be retained at planned intervals so that secondary threshold methods and changes in output characteristics can be evaluated over time. There is no reason to expect one universal cutoff for all of these metrics.

The eventual criterion should instead be tied to the sensing task. A 2 V threshold shift may be irrelevant if the cortisol-associated signal is large and the change is predictable, or unacceptable if it is comparable to the signal generated by a physiologically important change in cortisol. The relevant question is not whether the FET changed at all. It is whether its change is small enough, reproducible enough, or characterizable enough that the molecular signal remains recoverable.

6.7 What does this mean for a completed cortisol aptamer-FET?

The completed cortisol sensor adds another layer of interpretation. In the Wang platform, cortisol binding changes aptamer conformation and the arrangement of associated charge near the In_2O_3 surface, which changes effective gating and drain-to-source current (Wang et al., 2022). Those changes are the signal of interest.

The experiments in this thesis contained no aptamer and no cortisol. Any electrical changes observed here occurred before target recognition could contribute. That makes these data useful as a lower-level control. They show how much electrical change can occur in the device and pre-aptamer surface state even in the absence of cortisol.

The fully functionalized sensor will add its own sources of drift. Work on electrochemical aptamer-based sensors, although based on a different transduction architecture, has shown that receptor/SAM loss and fouling can produce signal drift that is unrelated to target concentration (Leung et al., 2021). A future cortisol aptamer-FET will have to distinguish at least device-level electrical drift from recognition-layer drift and actual target-dependent response.

The actual functionalization sequence also matters. The PTMS + DDT surface studied here is **not** the final Wang silane surface. The published cortisol platform uses mixed

APTES/PTMS vapor deposition at a 1:9 ratio, followed by dodecanethiol passivation, MBS crosslinking, and thiolated aptamer immobilization (Wang et al., 2022). APTES introduces the amine functionality needed for the later MBS coupling, so the completed interface cannot be assumed to behave electrically like the PTMS decomposition condition used in this thesis.

The next completed-sensor experiment should preserve the staged logic developed here rather than jumping directly from bare FET to cortisol response. Electrical characterization should be collected after the actual APTES/PTMS treatment, after dodecanethiol, after MBS, and after aptamer immobilization. A no-cortisol aging period can then establish the baseline behavior of the fully functionalized sensor before repeated cortisol challenges are introduced. In that design, a change that appears after MBS or aptamer attachment can be separated from a change that already existed in the FET or silane-treated surface.

Controls become especially important at that stage. Aptamer-free FETs or FETs containing a nonbinding or scrambled receptor can be aged beside active cortisol sensors. If all devices drift together, the origin is more likely to be shared transducer or environmental behavior. If only active recognition layers change, a receptor-related mechanism becomes more plausible. Aptamer-FET studies have already used scrambled and receptor-free controls to distinguish receptor-dependent sensing responses from nonspecific FET responses (Liu et al., 2020).

Repeated known cortisol concentrations are then required. The fully functionalized study should determine whether baseline, sensitivity, calibration slope, recovery, hysteresis, and concentration discrimination remain usable as the sensor ages. The size of the device-level drift can then be compared directly with the electrical response produced by physiologically relevant cortisol changes.

If the repeated bare/pre-apptamer study continues to show instability at exposed nonsensing interfaces, structural protection can also be evaluated as a later engineering step. Other platforms have improved aqueous stability by protecting electrode or dielectric regions while leaving the sensing interface accessible. Zhu et al. used SU-8 protection around the electrodes with a PDMS sensing well in an In_2O_3 architecture; Albarghouthi et al. showed substantial improvements in solution stability after passivating exposed regions of CNT BioFETs; Hideshima et al. improved aqueous FET stability using additional surface coatings (Zhu et al., 2021; Albarghouthi et al., 2022; Hideshima et al., 2022). These solutions cannot simply be copied onto the cortisol platform because the receptor must remain sufficiently close to the semiconductor for FET transduction, but they show that the nonsensing parts of the device do not have to remain exposed simply because the sensing surface does.

The larger point is that continuous cortisol monitoring is a systems-level stability problem. Aptamer recognition, In_2O_3 electrical behavior, electrode interfaces, environmental exposure, sample composition, reference measurements, and readout hardware all contribute to whether a repeated electrical value can still be interpreted as cortisol. This thesis isolates the transducer and pre-apptamer part of that problem and shows that its contribution is large enough that it should be measured, not assumed away.

6.8 What remains unresolved, and what should be done next?

6.8.1 Limits of the present study

The first limitation is duration. The formal experiment was designed to continue through Day 14, but physical failure of the original back-gate probe assembly and holder-to-BNC measurement path at the scheduled 24 h measurement restricted the standardized quantitative

dataset to T0, 6 h, and 12 h. Day 7 provides useful cross-sectional information, but the changed physical gate contact and unreliable gate-current channel prevent it from extending the original trajectory quantitatively.

The second limitation is independent replication. Each Surface $\times$ Exposure condition contained three independently treated wafer pieces, one from each parent wafer, with two FET subsamples per piece. The mixed model preserved the nested structure, but three pieces per condition still leaves substantial treatment-level uncertainty. The piece-level HC3 analysis makes this visible: the DDT-versus-Bare point estimate remained almost unchanged, while the confidence interval widened across zero.

The environmental comparison also represents the realized PBS workflow, not an isolated chemical effect of phosphate-buffered saline. PBS pieces received liquid exposure in humidified chambers, while air pieces remained dry. Each Surface $\times$ Exposure cell also occupied its own dedicated chamber. Humidity, chamber identity, liquid contact, ion exposure, and associated handling cannot all be separated statistically from one another. This was necessary to preserve the PBS droplet, but the resulting effect should not be labeled as an isolated ionic-PBS mechanism.

The measurement itself was not continuous wet sensing. PBS was removed, the FETs were rinsed and dried, both exposure groups received the standardized 70 °C treatment, and measurements were collected dry through the Si/SiO₂ back gate. This design provided a consistent electrical geometry, but it measures the state left behind after exposure and conditioning. It does not measure the instantaneous electrical behavior of a solution-gated FET while immersed in liquid. The pilot directly showed that thermal conditioning could change the

measured electrical response, so the heating step cannot be assumed to be merely a neutral preparation step.

The surface-processing experiment was also electrical rather than chemical. No XPS, contact-angle measurements, ellipsometry, AFM, or equivalent measurements established dodecanethiol coverage, PTMS organization, or UV-induced surface changes. Contact resistance was not separated from channel behavior. The mechanistic explanations in this discussion are hypotheses constrained by the electrical data and external literature, not direct measurements of the interfaces.

The progressive conditions were composite workflows rather than a factorial chemistry experiment. PTMS was followed by DDT, and UV was followed by PTMS and DDT. The contrasts ask what changed when the workflow progressed from one state to the next. They cannot separately estimate every UV, PTMS, solvent, thermal, and handling contribution.

The three UV T0 substitutions add a related selection limitation. The replacements were made before environmental exposure was evaluated, and no FET was replaced after T0, so longitudinal outcomes were not selected on the basis of later performance. However, the UV cohort entering Chapter 5 was conditioned on having interpretable T0 behavior. The original three flat positions need to remain visible as a pre-exposure yield/compatibility result rather than disappearing from the interpretation.

Finally, none of these experiments included the APTES/PTMS mixture, MBS, aptamer, cortisol, or biological sweat. The thesis cannot determine aptamer durability, calibration drift, fouling, cortisol sensitivity after aging, or wearable long-duration performance.

6.8.2 The next study should proceed in stages

The immediate next experiment should be the study that was originally intended: a complete longitudinal repeat of the four pre-aptamer surface conditions under air and PBS using one validated electrical measurement configuration from T0 through the final timepoint. The replacement gate-probe assembly should be stress-tested through repeated connection cycles before experimental exposure begins. If a backup gate-contact method is going to exist, a bridging experiment should quantify its agreement with the primary configuration before it is ever needed.

The repeat should retain the two-FET-per-piece structure but increase the number of independently treated pieces and, if feasible, the number of parent fabrication wafers. More independent pieces are more valuable for treatment-level precision than simply increasing the number of FETs measured on the same piece.

The environmental design should also separate variables that were combined here. Replicate chambers within a Surface $\times$ Exposure condition would allow chamber-to-chamber variation to be estimated. A humidified-air control would help separate elevated humidity from direct liquid PBS contact. Recording chamber humidity and droplet mass or volume over time would document the physical exposure rather than assuming it from chamber design.

The UV-containing sequence deserves an additional measurement between processing and exposure. Because all Chapter 4 UV FETs were valid immediately POST but three intended positions were flat at T0, repeated electrical measurements during the POST-to-T0 interval could determine whether some devices evolve after processing even before PBS is applied. That would directly test the possibility of delayed post-processing instability without using environmental exposure as a confound.

A separate mechanistic study should then ask why the individual processing steps behaved differently. Dodecanethiol-treated and untreated Au can be compared using surface characterization and contact-resistance measurements. PTMS can be examined with and without UV under otherwise matched conditions before DDT is added. XPS, contact-angle measurements, AFM or ellipsometry, where appropriate, would make it possible to connect the electrical response to actual changes in surface chemistry or wetting rather than inferring those changes from current-voltage curves. The literature provides strong reasons to test Au morphology/SAM quality and UV-sensitive In_2O_3 surface states, but it does not tell us which mechanism occurred in these FETs (Godin et al., 2004; Kim et al., 2017; Lin et al., 2026).

Only after the underlying and pre-aptamer FET behavior can be followed reproducibly for the intended duration should the same design be extended through the fully functionalized sensor. The next level should use the actual mixed APTES/PTMS interface, dodecanethiol passivation, MBS, and the thiolated cortisol aptamer. Electrical characterization after each stage would reveal whether a new source of drift enters with the recognition chemistry itself (Wang et al., 2022).

The fully functionalized sensors should first be aged without cortisol challenge so that the no-target baseline is known. Repeated cortisol exposures at controlled concentrations can then be added. PBS can serve as the first controlled medium, followed by artificial sweat and then carefully controlled biological samples. Active aptamer sensors should be run in parallel with receptor-free or nonbinding controls.

At that point, stability can finally be defined in terms of the intended application. Instead of assigning an arbitrary universal allowable V_{th} shift, the experiment can determine how much threshold drift, current loss, hysteresis, leakage, or loss of functional yield can occur before

cortisol concentration can no longer be recovered with the required accuracy. That directly connects FET stability to sensing performance.

6.9 Conclusions

This thesis began with a question underneath the completed cortisol sensor: what happens electrically to the In_2O_3 FET before an aptamer-mediated signal is ever measured?

The bare-device pilot showed that the answer depended on more than elapsed time. PBS exposure, evaporation, drying, thermal conditioning, and the electrical metric being examined all affected what was observed. The pilot became protocol development rather than a simple lifetime measurement.

The processing study then showed that surface preparation had its own electrical consequences. The DDT condition produced relatively modest averages with substantial device-level variability. The PTMS + DDT produced a more directionally consistent electrical shift. The UV-containing sequence produced a different and more heterogeneous state, and the later need to substitute three UV FET positions at T0 raises an additional question about pre-exposure process compatibility that should be tested directly.

After environmental exposure began, the largest early change in the primary forward-threshold response occurred between Bare and DDT. Fixed-bias $|I_{\text{DS}}|$ supported the same broad result. That did not translate into uniform stability across reverse threshold voltage, hysteresis, leakage, or every later device. At Day 7, DDT and PTMS + DDT retained substantially more interpretable PBS-exposed devices than Bare or UV + PTMS + DDT, while the UV condition showed that a small early threshold difference among surviving devices did not guarantee later operational retention.

The study does not identify one surface treatment that can simply be labeled stable. It shows something more useful for the next experiment: the FET has an electrical history before sensing begins, and different parts of that history can change both the baseline and the way the device responds later. Processing-induced change, exposure-induced change, leakage, metric loss, and eventual recognition-layer drift need to be separated rather than allowed to appear as one sensor signal.

That separation is the next requirement for the cortisol platform. The complete pre-apramer experiment now needs to be repeated for the intended duration under one reliable measurement configuration. The same approach can then be carried through the full APTES/PTMS–DDT–MBS–apramer sequence and finally into repeated cortisol measurements. Only then can device drift be compared directly with the cortisol-dependent signal and an application-relevant stability limit be defined.

Appendix A. Review audit supporting Section 1.7

This appendix presents a structured, representative audit of review and framework literature used to support Section 1.7. It is not a systematic review. The audit was assembled to span the major literature categories relevant to the Section 1.7 question: cortisol-specific sensing reviews, wearable and biofluid monitoring frameworks, continuous molecular-monitoring frameworks, and comparator literatures on mature continuous glucose monitoring (CGM), BioFETs, and organic electrochemical transistor (OECT) operational stability. The audit looks at how continuous monitoring is framed, the role of FETs, what “stability” or sustained operation means, whether a quantitative transistor-level acceptance criterion is identified, and how each source is used or bounded in the thesis. Because the corpus is curated rather than systematic, it supports qualitative synthesis and gap framing rather than prevalence estimates across the entire field. Updated through August 2026.

Table A.1. Review audit supporting Section 1.7.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Kaushik, A., Vasudev, A., Arya, S. K., Pasha, S. K., & Bhansali, S. (2014). Recent advances in cortisol sensing technologies for point-of-care application. <i>Biosensors and Bioelectronics</i> , 53, 499–512. https://doi.org/10.1016/j.bios.2013.09.060	Point-of-care cortisol sensing technologies.	Frames wearable/online cortisol monitoring as a future point-of-care objective rather than an established capability.	Absent to peripheral.	Sustained use is discussed through sensor lifetime, portability, analytical performance, and translation rather than longitudinal transistor drift.	Not identified in the reviewed source.	Early benchmark for the gap between cortisol detection and deployable point-of-care sensing.	Does not define a transistor-level drift or failure threshold for prolonged cortisol monitoring.
Zainol Abidin, A. S., Rahim, R. A., Md Arshad, M. K., et al. (2017). Current and potential developments of cortisol aptasensing towards point-of-care diagnostics (POTC). <i>Sensors</i> , 17(5), 1180. https://doi.org/10.3390/s17051180	Cortisol aptasensing and point-of-care diagnostic strategies.	Continuous monitoring is not the central framework; emphasis is on aptamer-based cortisol detection and possible point-of-care formats.	Absent.	Stability is relevant mainly through recognition-layer properties and practical assay/sensor performance rather than transistor electrical drift.	Not identified in the reviewed source.	Historical aptamer-specific review; useful for recognition-layer context, not TFT reliability.	Does not provide an operational transistor-stability framework for cortisol aptasensing.
Bariya, M., Nyein, H. Y. Y., & Javey, A. (2018). Wearable sweat sensors. <i>Nature Electronics</i> , 1, 160–171. https://doi.org/10.1038/s41928-018-0043-y	Wearable sweat sensing across analytes and platforms.	Continuous/real-time wearable biochemical monitoring is a major goal.	Peripheral.	Sustained operation includes sampling, calibration, contamination, device reliability, and mechanical robustness at the wearable-system level.	Not identified in the reviewed source.	Adjacent wearable framework for what continuous deployment requires beyond analyte detection.	Does not establish a cortisol-specific electrical acceptance standard for FET operation.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Steckl, A. J., & Ray, P. (2018). Stress biomarkers in biological fluids and their point-of-use detection. <i>ACS Sensors</i> , 3(10), 2025–2044. https://doi.org/10.1021/acssensors.8b00726	Stress biomarkers and point-of-use detection in biofluids.	Continuous monitoring is framed mainly as a motivation for stress assessment rather than an established capability.	Absent to peripheral.	Sustained use is discussed through matrix effects, sampling variability, portability, and sensor robustness.	Not identified in the reviewed source.	Broad stress-biomarker context for cortisol sensing and sample-matrix constraints.	Does not provide a framework for longitudinal transistor drift or failure.
Acciaroli, G., Vettoretti, M., Facchinetti, A., & Sparacino, G. (2018). Calibration of minimally invasive continuous glucose monitoring sensors: State-of-the-art and current perspectives. <i>Biosensors</i> , 8(1), 24. https://doi.org/10.3390/bios8010024	Calibration of mature minimally invasive continuous glucose monitoring systems.	Multi-day continuous monitoring is an established use case; the review focuses on maintaining accuracy as sensor sensitivity and baseline vary over time.	Absent.	Operational stability is handled through time-varying sensitivity/baseline, calibration frequency, sensor warm-up, and accuracy across the wear period.	Not applicable at transistor level; quantitative CGM calibration and accuracy metrics are reported.	Mature continuous-monitoring comparator showing that sustained operation requires explicit treatment of time-varying sensor behavior.	CGM calibration standards and numerical accuracy criteria are not directly transferable to cortisol FETs.
Hogenelst, K., Soeter, M., & Kallen, V. (2019). Ambulatory measurement of cortisol: Where do we stand, and which way to follow? <i>Sensing and Bio-Sensing Research</i> , 22, 100249. https://doi.org/10.1016/j.sbsr.2018.100249	Ambulatory cortisol measurement and point-of-care technologies.	Ambulatory measurement is the central translational goal; many technologies are described as remaining at proof-of-concept stages.	Peripheral.	Practical durability and ambulatory feasibility are discussed at the assay/device level rather than as longitudinal TFT electrical drift.	Not identified in the reviewed source.	Historical benchmark for the gap between laboratory cortisol assays and ambulatory sensing.	Does not define when a cortisol-sensing transistor should be considered electrically unstable.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Kim, J., Campbell, A. S., de Ávila, B. E.-F., & Wang, J. (2019). Wearable biosensors for healthcare monitoring. <i>Nature Biotechnology</i> , 37, 389–406. https://doi.org/10.1038/s41587-019-0045-y	Wearable biosensors for health monitoring.	Continuous, real-time biochemical monitoring is a core premise.	Peripheral.	Sustained operation is framed through biofouling, calibration, power, signal drift, material stability, and wearable integration.	Not identified in the reviewed source.	Adjacent systems-level benchmark for wearable biochemical monitoring requirements.	Does not provide a cortisol-specific TFT drift/failure standard.
Heikenfeld, J., Jajack, A., Feldman, B., Granger, S. W., Gaitonde, S., Begtrup, G., & Katchman, B. A. (2019). Accessing analytes in biofluids for peripheral biochemical monitoring. <i>Nature Biotechnology</i> , 37, 407–419. https://doi.org/10.1038/s41587-019-0040-3	Peripheral biofluids and biochemical monitoring.	Continuous monitoring is discussed through comparison with established CGM and the limitations of peripheral-biofluid sensing.	Peripheral.	Sustained operation includes analyte transport, sampling, calibration, correlation to blood, fouling, and long-term measurement reliability.	Not identified in the reviewed source.	Important systems-level boundary: reliable transistor operation does not by itself guarantee interpretable wearable biomarker measurement.	Does not establish a cortisol-specific electrical drift standard comparable to mature CGM performance frameworks.
Yang, Y., & Gao, W. (2019). Wearable and flexible electronics for continuous molecular monitoring. <i>Chemical Society Reviews</i> , 48(6), 1465–1491. https://doi.org/10.1039/C7CS00730B	Wearable and flexible platforms for continuous molecular analysis across biofluids.	Continuous, real-time molecular monitoring is the organizing goal.	One of several transducer classes.	Sustained monitoring is treated as a system problem involving sampling, calibration, selectivity, wearable integration, and long-term operation.	Not identified in the reviewed source.	Core continuous-monitoring framework used to separate repeated molecular measurement from one-time analytical detection.	Does not translate continuous-monitoring requirements into a cortisol-specific transistor pass/fail rule.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Zea, M., Bellagambi, F. G., Ben Halima, H., Zine, N., Jaffrezic-Renault, N., Villa, R., Gabriel, G., & Errachid, A. (2020). Electrochemical sensors for cortisol detections: Almost there. <i>TrAC Trends in Analytical Chemistry</i> , 132, 116058. https://doi.org/10.1016/j.trac.2020.116058	Electrochemical cortisol-sensor landscape.	Real-time and continuous cortisol monitoring is an important translational direction.	Limited.	Stability is discussed through sensor-level properties such as storage, repeatability/reusability, fouling, and robustness rather than one operational electrical definition.	Not identified in the reviewed source.	Shows that analytical performance and continuous-monitoring goals can coexist without a common transistor-level stability framework.	No quantitative FET drift or functional-failure threshold is identified.
Samson, C., & Koh, A. (2020). Stress monitoring and recent advancements in wearable biosensors. <i>Frontiers in Bioengineering and Biotechnology</i> , 8, 1037. https://doi.org/10.3389/fbioe.2020.01037	Wearable biosensors for physiological and biochemical stress monitoring.	Continuous/wearable stress monitoring is a major goal.	Peripheral.	Sustained operation includes bioreceptor stability, reproducibility, sampling, contamination, and wearable reliability.	Not identified in the reviewed source.	Adjacent wearable-stress review reinforcing the gap between sensing feasibility and sustained operation.	Does not define a transistor-specific longitudinal stability framework.
Ghaffari, R., Yang, D. S., Kim, J., et al. (2021). State of sweat: Emerging wearable systems for real-time, noninvasive sweat sensing and analytics. <i>ACS Sensors</i> , 6(8), 2787–2801. https://doi.org/10.1021/acssensors.1c01133	Wearable sweat sensing and analytics.	Real-time and continuous sweat sensing is a central systems-level goal.	Peripheral.	Sustained operation includes sampling reliability, calibration, contamination, signal interpretation, and system integration.	Not identified in the reviewed source.	Adjacent sweat-sensing framework for continuous sampling and signal-reliability challenges.	Does not define transistor-level drift limits for cortisol FETs.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Parlak, O. (2021). Portable and wearable real-time stress monitoring: A critical review. <i>Sensors and Actuators Reports</i> , 3, 100036. https://doi.org/10.1016/j.snr.2021.100036	Portable and wearable real-time stress monitoring.	Real-time and continuous cortisol monitoring is a central framing and future direction.	Limited.	Sustained operation is discussed through stability, handling, regeneration/reuse, and practical wearable demands.	Not identified in the reviewed source.	Supports the argument that continuous monitoring introduces requirements beyond one-time analyte detection.	Recognizes stability as a challenge without converting it into an electrical transistor acceptance rule.
Iqbal, T., Elahi, A., Wijns, W., & Shahzad, A. (2023). Cortisol detection methods for stress monitoring in connected health. <i>Health Sciences Review</i> , 6, 100079. https://doi.org/10.1016/j.hsr.2023.100079	Cortisol detection for connected-health and ambulatory use.	Ambulatory/real-time cortisol assessment is framed as unresolved and requiring accurate, rapid, repeatable measurement.	Absent to peripheral.	Sustained performance is framed mainly through repeatability, deployment, and reliability of ambulatory measurement.	Not identified in the reviewed source.	Translational framing for why laboratory detection is not equivalent to a deployable monitoring system.	Does not define TFT drift, leakage, hysteresis, or failure limits.
Karachaliou, C.-E., Koukouvinos, G., Goustouridis, D., et al. (2023). Cortisol immunosensors: A literature review. <i>Biosensors</i> , 13(2), 285. https://doi.org/10.3390/bios13020285	Literature review of cortisol immunosensors.	Real-time point-of-care and wearable continuous cortisol measurement are presented as important directions.	Absent to peripheral.	Performance is discussed through recognition chemistry, matrix use, repeatability/reproducibility, and practical sensor reliability rather than transistor drift.	Not identified in the reviewed source.	Recognition-specific review for comparing sensor-level stability concepts.	Does not convert operational performance in real matrices into a transistor-level acceptance framework.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Karuppaiah, G., Lee, M.-H., Bhansali, S., & Manickam, P. (2023). Electrochemical sensors for cortisol detection: Principles, designs, fabrication, and characterisation. <i>Biosensors and Bioelectronics</i> , 239, 115600. https://doi.org/10.1016/j.bios.2023.115600	Electrochemical cortisol sensing principles, devices, and performance.	Wearable and real-world cortisol sensing are important translational directions.	Partial.	The review explicitly identifies biofouling, flexibility, stability, sample preparation, and recognition-layer performance as challenges.	Not identified in the reviewed source.	Technology-landscape review for electrochemical cortisol sensing and recognition/transduction background.	Does not provide quantified transistor drift rates, failure criteria, or a long-term electrical acceptance standard.
Kaur, S., Gupta, N., & Malhotra, B. D. (2023). Recent developments in wearable & non-wearable point-of-care cortisol biosensors. <i>Expert Review of Molecular Diagnostics</i> , 23(3), 217–230. https://doi.org/10.1080/14737159.2023.2184260	Wearable and non-wearable point-of-care cortisol biosensors.	Continuous cortisol monitoring is a central translational objective.	Partial.	Deployment challenges include calibration, circadian and inter-individual variability, endocrine interference, and practical repeatable measurement.	Not identified in the reviewed source.	Direct cortisol-specific review for deployment and calibration challenges.	Does not define a transistor-specific quantitative stability threshold.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Kusov, P. A., Kotelevtsev, Y. V., & Drachev, V. P. (2023). Cortisol monitoring devices toward implementation for clinically relevant biosensing in vivo. <i>Molecules</i> , 28(5), 2353. https://doi.org/10.3390/molecules28052353	Clinically relevant in-vivo cortisol monitoring.	Continuous real-time cortisol monitoring is an explicit unmet goal.	Substantial.	Sustained operation is discussed through reversibility, receptor behavior, fouling, kinetics, and the ability to follow changing cortisol over time.	No cortisol-specific transistor pass/fail criterion identified in the reviewed source.	Bridge between sensor-platform design and requirements of true continuous cortisol monitoring.	Identifies barriers to continuous sensing without allowable V_{th} , current, hysteresis, or leakage drift.
Flynn, C. D., Chang, D., Mahmud, A., et al. (2023). Biomolecular sensors for advanced physiological monitoring. <i>Nature Reviews Bioengineering</i> , 1, 560–575. https://doi.org/10.1038/s44222-023-00067-z	Body-based wearable, implantable, and consumable biomolecular sensing.	Sequential real-time and continuous physiological monitoring are central goals; CGM is treated as the mature benchmark.	Thin-film transistors are one of several highlighted sensing approaches.	Sustained performance is framed through reagentless operation, selectivity, sensitivity, physiological matrix constraints, transport, and interface limitations.	Not identified in the reviewed source.	Broad bioengineering framework connecting continuous molecular monitoring to the requirements of the complete sensing system.	Does not define a cortisol-specific transistor drift or functional-failure threshold.
Naeem, A. N., Guldin, S., & Ghoreishizadeh, S. S. (2024). Electrochemical sensors for cortisol: A review. <i>IEEE Sensors Journal</i> , 24(5), 5746–5758. https://doi.org/10.1109/JSEN.2023.3349293	Electrochemical cortisol sensors in human biofluids.	Real-time and continuous monitoring are explicit motivations.	Partial.	Stability is discussed mainly as a sensor/bioreceptor property together with reusability, reversibility, sensitivity, selectivity, and reproducibility.	Not identified in the reviewed source.	Supports the distinction between recognition-layer/sensor stability and transistor-level operational electrical stability.	Does not provide a quantitative transistor drift or functional-failure criterion.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Weber, C. J., Clay, O. M., Lycan, R. E., Anderson, G. K., & Simoska, O. (2024). Advances in electrochemical biosensor design for the detection of the stress biomarker cortisol. <i>Analytical and Bioanalytical Chemistry</i> , 416(1), 87–106. https://doi.org/10.1007/s00216-023-05047-1	Electrochemical cortisol biosensor design in human biofluids.	Rapid, real-time and continuous cortisol monitoring are presented as motivations for electrochemical biosensor development.	Peripheral to partial.	Translation is discussed through recognition elements, reproducibility, matrix effects, wearability, and large-scale production rather than a bare-transistor stability definition.	Not identified in the reviewed source.	Supports the broader sensor-performance discussion while remaining distinct from TFT reliability.	Does not define a standardized transistor-level operational stability criterion.
Ok, J., Park, S., Jung, Y. H., & Kim, T.-I. (2024). Wearable and implantable cortisol-sensing electronics for stress monitoring. <i>Advanced Materials</i> , 36(1), 2211595. https://doi.org/10.1002/adma.202211595	Wearable and implantable cortisol-sensing electronics.	Continuous cortisol measurement is a central application goal.	Central to part of the reviewed landscape.	Sustained operation is treated primarily as a system-level challenge involving device integration and long-term wearable/implantable use.	Not identified in the reviewed source.	Shows that wearable integration and continuous-monitoring goals do not themselves establish transistor-level electrical stability.	Does not define a standardized electrical pass/fail criterion for the transistor during prolonged cortisol sensing.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Wankhade, U. A., Thakare, Y. N., Hardas, B. M., & Pande, R. S. (2024). Cortisol detection methods for stress monitoring: Current insight and future prospect: A review. <i>IEEE Sensors Journal</i> , 24(15), 23389–23400. https://doi.org/10.1109/JSEN.2024.3404399	Broad cortisol detection methods across biofluids, including wearable and transistor-based examples.	Real-time, continuous and long-term cortisol monitoring are explicit future goals.	Partial; FET and OECT examples are included among several sensor classes.	Reliability is framed through real-time accuracy/dependability, calibration, biofluid choice, and wearable integration rather than a defined longitudinal transistor criterion.	Not identified in the reviewed source.	Recent broad cortisol review that helps test whether the stability gap persists across multiple sensing architectures.	Does not define allowable V_{th} , leakage, hysteresis, or functional-survival limits for cortisol transistors.
Vignesh, V., Castro-Dominguez, B., James, T. D., Gamble-Turner, J. M., Lightman, S., & Reis, N. M. (2024). Advancements in cortisol detection: From conventional methods to next-generation technologies for enhanced hormone monitoring. <i>ACS Sensors</i> , 9(4), 1666–1681. https://doi.org/10.1021/acssensors.3c01912	Broad cortisol-detection review spanning physiology, matrices, conventional assays, and next-generation sensors.	Rapid and continuous monitoring are framed as major directions.	Partial.	Sustained operation is discussed through longevity, fouling, reproducibility, sampling, and practical device limitations rather than a single electrical definition.	Not identified in the reviewed source.	Broad organizing review for the chapter and for the distinction between detection performance and sustained monitoring.	Does not define a transistor-level acceptance threshold for drift or functional loss.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Watkins, Z., McHenry, A., & Heikenfeld, J. (2024). Wearing the lab: Advances and challenges in skin-interfaced systems for continuous biochemical sensing. <i>Advances in Biochemical Engineering/Biotechnology</i> , 187, 223–282. https://doi.org/10.1007/10_2023_238	Skin-interfaced systems for continuous biochemical sensing.	Continuous, contextual biochemical monitoring is the core premise, with CGM as the mature comparison.	One of several transduction approaches.	Sustained operation is framed through biorecognition, transduction, sampling, materials, integration, calibration, and practical wear challenges.	Not identified in the reviewed source.	Adjacent continuous-monitoring framework for system-level requirements beyond recognition chemistry alone.	Does not establish a cortisol-specific quantitative TFT acceptance standard.
Davis, N., Heikenfeld, J., Milla, C., & Javey, A. (2024). The challenges and promise of sweat sensing. <i>Nature Biotechnology</i> , 42, 860–871. https://doi.org/10.1038/s41587-023-02059-1	Critical perspective on wearable sweat sensing and physiological interpretation.	Real-time, continuous molecular analysis is an important application, but the review emphasizes unresolved sampling and physiological-validity constraints.	Peripheral.	Sustained measurement is constrained by sweat access/secretion, sample collection, analyte validity, and interpretation rather than transistor drift alone.	Not identified in the reviewed source.	Modern sweat-sensing boundary for separating transducer stability from sample-access and physiological-validity problems.	Does not provide a cortisol-FET electrical drift or failure criterion.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Li, Z., Luo, D., Zhang, Y., Niu, X., & Liu, H. (2025). Smart health monitoring: Review of electrochemical biosensors for cortisol monitoring. <i>Advanced Healthcare Materials</i> , 14, 2404454. https://doi.org/10.1002/adhm.202404454	Electrochemical cortisol biosensors and translation toward smart health monitoring.	Explicitly discusses real-time, continuous, and long-term cortisol monitoring.	Partial.	Long-term stability is considered alongside reusability, reproducibility, continuous-monitoring capability, and response time at the sensor level.	Not identified in the reviewed source.	Direct support that long-term stability is a distinct translational requirement beyond analytical detection performance.	Does not provide a quantitative transistor-level drift or failure threshold.
Balasamy, S., Atchudan, R., Arya, S., & Sundramoorthy, A. K. (2025). Emerging trends in wearable and non-invasive cortisol sensing technologies – a review. <i>Journal of Materials Chemistry B</i> , 13, 13154–13183. https://doi.org/10.1039/D5TB01519G	Wearable and non-invasive cortisol sensing technologies.	Continuous wearable cortisol monitoring and wireless data transmission are central translational directions.	Partial; FETs are one of several reviewed technologies.	Long-term deployment is framed through wearable integration, power, communication, fabrication, calibration, and practical device performance.	Not identified in the reviewed source.	Recent field update spanning electrochemical, optical, FET, microfluidic, and wearable cortisol platforms.	Does not establish a standardized transistor-level operational acceptance criterion.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Pandey, M., Bhaiyya, M., Rewatkar, P., Zalke, J. B., Narkhede, N. P., & Haick, H. (2025). Advanced materials for biological field-effect transistors (Bio-FETs) in precision healthcare and biosensing. <i>Advanced Healthcare Materials</i> , 14(13), e2500400. https://doi.org/10.1002/adhm.202500400	Material and device challenges in BioFET biosensing across applications.	Real-time biosensing and wearable integration are important application contexts rather than cortisol-specific continuous monitoring.	Central.	Operational durability, long-term stability, reproducibility, material degradation, passivation, and performance drift are treated as distinct BioFET translation problems.	No universal quantitative BioFET pass/fail threshold identified in the reviewed source.	BioFET comparator showing that device-level operational durability is a recognized engineering problem outside cortisol-specific reviews.	The review is not cortisol-specific and does not provide acceptance limits for the In ₂ O ₃ architecture studied in this thesis.
Mukhin, N., Dietzel, A., Issakov, V., & Bakhchova, L. (2025). Balancing performance and stability characteristics in organic electrochemical transistor. <i>Biosensors and Bioelectronics</i> , 281, 117476. https://doi.org/10.1016/j.bios.2025.117476	Review of performance and long-term stability in organic electrochemical transistors (OECTs).	Long-term operation is a central device-engineering concern in OECT bioelectronics and biosensing.	OECT is the central transistor platform; comparator rather than In ₂ O ₃ FET literature.	Stability is treated through degradation mechanisms, electrochemical/cycling stability, reversible versus irreversible processes, performance-stability trade-offs, and device lifespan.	No universal cortisol-FET acceptance threshold; stability metrics are platform-specific.	Adjacent transistor benchmark showing that operational stability can be treated as a device-level problem independent of analyte-detection performance.	OECT degradation mechanisms and numerical criteria are not directly transferable to back-gated In ₂ O ₃ TFTs.

Source	Scope	Continuous-monitoring framing	FET role	What “stability” / sustained operation means	Quantitative transistor-level acceptance criterion identified?	Thesis use / boundary	Key gap
Lai, Q., Dong, Y., & Liu, G. (2026). Continuous molecular monitoring to advance biosensing: Strategies and challenges. Chemical Engineering Journal, 179383. https://doi.org/10.1016/j.cej.2026.179383	Broad continuous molecular monitoring framework across transducer classes.	Continuous molecular monitoring is the central premise and is defined by reagentless operation, reversible response, batch-free workflow, and continuous readout.	FETs are one of several transducer technologies considered.	Sustained operation is organized around interacting failure modes including signal drift, biofouling, receptor degradation, reference-electrode instability, transport, and engineering constraints.	No cortisol-specific transistor acceptance criterion identified in the reviewed source.	Strong systems-level framework for distinguishing device-level electrical behavior from complete-sensor drift.	Failure modes are organized at the continuous-sensor level rather than as allowable V_{th} /current/hysteresis/leakage limits for this In_2O_3 platform.

Note. “Not identified in the reviewed source” means that the source did not provide a quantitative transistor-level acceptance criterion relevant to the Section 1.7 question in the current audit, not a claim that quantitative electrical-stability metrics are absent from the broader BioFET or biosensor literature. Primary BioFET studies can report device-level stability quantitatively; for example, Albarghouthi et al. (2022) evaluated leakage current, on-current retention, on/off ratio, hysteresis, subthreshold swing, device yield, and long-duration cycling in PBS when comparing passivation strategies. That study is included here as a boundary condition rather than as a review/framework row: the gap addressed in Section 1.7 is not that transistor stability metrics do not exist, but that cortisol-monitoring reviews and systems do not share a standardized transistor-level operational acceptance framework. This appendix is a curated field audit rather than a systematic review.

APPENDIX B

Chapter 3 Supporting Electrical Characterization and Protocol-Development Data

B.1 Five-Day Bare-Device Pilot Electrical Characterization

This section retains the numerical metrics, quality-control and troubleshooting records, and unique longitudinal summary figures supporting the five-day bare-device pilot in Chapter 3.

Tables B.1.1 through B.1.19 and Figures B.1.1–B.1.2 are retained here. Complete longitudinal electrical records for all pilot FETs are provided in Supplemental Data S1.

Table B.1.1: Threshold, hysteresis, I_{DS} , and leakage metrics (T0-3 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Forward V_{th} (V)	Reverse V_{th} (V)	Hysteresis (V)	I_{DS} at +30 V (A)	I_G at +30 V (A)	$ I_G / I_{DS} $ at +30 V
AIR1	Air	T0	Yes	Yes	11.048	14.824	3.776	4.251e-04	2.025e-05	0.048
AIR2	Air	T0	Yes	Yes	15.664	18.993	3.329	2.230e-04	7.643e-06	0.034
AIR3	Air	T0	Yes	Yes	13.642	16.772	3.130	3.356e-04	1.571e-05	0.047
AIR4	Air	T0	Yes	Yes	6.211	10.478	4.267	2.254e-04	5.836e-06	0.026
AIR5	Air	T0	Yes	Yes	6.507	8.994	2.486	2.756e-04	8.218e-06	0.030
AIR6	Air	T0	Yes	Yes	6.955	10.697	3.741	2.371e-04	4.619e-06	0.019
AIR7	Air	T0	Yes	Yes	6.809	10.761	3.951	1.901e-04	7.500e-06	0.039
AIR8	Air	T0	Yes	Yes	7.635	11.054	3.419	1.690e-04	8.188e-06	0.048
PBS1	PBS	T0	Yes	Yes	11.870	15.586	3.716	3.141e-04	1.571e-05	0.050
PBS2	PBS	T0	Yes	Yes	20.671	19.708	-0.963	2.863e-05	6.911e-07	0.024
PBS3	PBS	T0	Yes	Yes	15.587	18.621	3.034	2.346e-04	1.074e-05	0.046
PBS4	PBS	T0	Yes	Yes	17.156	20.315	3.158	1.236e-04	6.190e-06	0.050
PBS5	PBS	T0	Yes	Yes	6.880	11.331	4.451	1.629e-04	1.635e-06	0.010
PBS6	PBS	T0	Yes	Yes	6.954	8.818	1.864	1.761e-04	8.883e-06	0.050
PBS7	PBS	T0	Yes	Yes	7.350	11.758	4.408	1.798e-04	5.442e-06	0.030
PBS8	PBS	T0	Yes	Yes	6.522	9.311	2.789	1.970e-04	7.896e-06	0.040
AIR1	Air	3 h	Yes	Yes	9.725	13.980	4.255	4.774e-04	2.211e-05	0.046
AIR2	Air	3 h	No	No	—	17.846	—	2.860e-04	2.088e-03	7.300
AIR3	Air	3 h	Yes	Yes	12.991	15.973	2.981	3.739e-04	1.802e-05	0.048
AIR4	Air	3 h	Yes	Yes	7.473	10.585	3.112	2.156e-04	6.455e-06	0.030
AIR5	Air	3 h	Yes	Yes	5.262	8.349	3.087	3.007e-04	8.735e-06	0.029
AIR6	Air	3 h	Yes	Yes	6.923	10.127	3.204	2.465e-04	6.322e-06	0.026
AIR7	Air	3 h	Yes	Yes	6.420	10.637	4.217	1.978e-04	7.341e-06	0.037
AIR8	Air	3 h	Yes	Yes	6.890	10.677	3.786	1.817e-04	8.342e-06	0.046
PBS1	PBS	3 h	Yes	Yes	10.029	24.651	14.622	2.162e-04	1.204e-05	0.056
PBS2	PBS	3 h	No	No	—	—	—	1.468e-04	1.370e-04	0.933
PBS3	PBS	3 h	Yes	Yes	9.849	19.862	10.013	1.838e-04	8.454e-06	0.046
PBS4	PBS	3 h	No	No	20.252	25.126	4.874	4.176e-05	5.787e-06	0.139
PBS5	PBS	3 h	Yes	Yes	3.123	16.498	13.376	1.035e-04	6.607e-07	0.006
PBS6	PBS	3 h	Yes	Yes	10.738	12.402	1.664	1.180e-04	8.176e-06	0.069
PBS7	PBS	3 h	Yes	Yes	6.565	13.091	6.526	1.829e-04	6.160e-06	0.034
PBS8	PBS	3 h	Yes	Yes	4.296	9.899	5.603	1.882e-04	4.456e-06	0.024

Table B.1.2: Threshold, hysteresis, I_{DS} , and leakage metrics (6-12 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Forward V_{th} (V)	Reverse V_{th} (V)	Hysteresis (V)	I_{DS} at +30 V (A)	I_G at +30 V (A)	$ I_G / I_{DS} $ at +30 V
AIR1	Air	6 h	Yes	Yes	9.402	13.249	3.847	4.186e-04	1.800e-05	0.043
AIR2	Air	6 h	Yes	Yes	—	—	—	3.981e-04	2.312e-04	0.581
AIR3	Air	6 h	Yes	Yes	10.611	13.740	3.128	3.025e-04	1.140e-05	0.038
AIR4	Air	6 h	Yes	Yes	5.151	9.607	4.456	2.472e-04	7.134e-06	0.029
AIR5	Air	6 h	Yes	Yes	5.790	8.281	2.491	2.928e-04	1.021e-05	0.035
AIR6	Air	6 h	Yes	Yes	6.332	9.560	3.228	2.584e-04	6.576e-06	0.025
AIR7	Air	6 h	Yes	Yes	6.636	10.689	4.053	1.970e-04	6.210e-06	0.032
AIR8	Air	6 h	Yes	Yes	6.936	10.578	3.642	1.814e-04	7.514e-06	0.041
PBS1	PBS	6 h	Yes	Yes	14.501	22.157	7.657	1.317e-04	1.170e-05	0.089
PBS2	PBS	6 h	No	No	—	—	—	2.724e-05	2.813e-06	0.103
PBS3	PBS	6 h	Yes	Yes	18.994	23.649	4.655	7.714e-05	1.240e-05	0.161
PBS4	PBS	6 h	No	No	—	—	—	2.906e-06	8.125e-06	2.796
PBS5	PBS	6 h	Yes	Yes	-0.462	13.821	14.283	1.278e-04	6.516e-07	0.005
PBS6	PBS	6 h	Yes	Yes	11.616	13.012	1.396	1.121e-04	9.043e-06	0.081
PBS7	PBS	6 h	Yes	Yes	2.605	9.965	7.360	1.991e-04	2.181e-06	0.011
PBS8	PBS	6 h	Yes	Yes	4.901	9.798	4.897	1.775e-04	3.119e-06	0.018
AIR1	Air	12 h	Yes	Yes	9.261	12.543	3.282	4.890e-04	1.660e-05	0.034
AIR2	Air	12 h	Yes	Yes	—	—	—	3.610e-04	1.600e-04	0.443
AIR3	Air	12 h	Yes	Yes	11.642	13.780	2.138	4.101e-04	1.890e-05	0.046
AIR4	Air	12 h	Yes	Yes	4.991	10.119	5.127	2.318e-04	4.847e-06	0.021
AIR5	Air	12 h	Yes	Yes	5.934	8.524	2.590	2.843e-04	8.934e-06	0.031
AIR6	Air	12 h	Yes	Yes	6.560	9.792	3.232	2.370e-04	4.356e-06	0.018
AIR7	Air	12 h	Yes	Yes	6.381	10.737	4.356	1.973e-04	4.843e-06	0.025
AIR8	Air	12 h	Yes	Yes	6.889	10.832	3.943	1.834e-04	7.359e-06	0.040
PBS1	PBS	12 h	Yes	Yes	9.845	21.108	11.263	1.779e-04	1.702e-05	0.096
PBS2	PBS	12 h	No	No	—	—	—	-1.000e-03	3.912e-03	3.911
PBS3	PBS	12 h	Yes	Yes	21.040	25.482	4.442	4.010e-05	4.656e-06	0.116
PBS4	PBS	12 h	No	No	—	—	—	2.063e-06	4.475e-06	2.169
PBS5	PBS	12 h	Yes	Yes	-2.924	15.661	18.585	1.004e-04	2.980e-07	0.003
PBS6	PBS	12 h	Yes	Yes	10.450	12.400	1.949	1.288e-04	7.732e-06	0.060
PBS7	PBS	12 h	Yes	Yes	-1.052	10.153	11.206	2.031e-04	1.616e-06	0.008
PBS8	PBS	12 h	Yes	Yes	5.464	11.715	6.251	1.566e-04	2.855e-06	0.018

Table B.1.3: Threshold, hysteresis, I_{DS} , and leakage metrics (24-48 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Forward V_{th} (V)	Reverse V_{th} (V)	Hysteresis (V)	I_{DS} at +30 V (A)	I_G at +30 V (A)	$ I_G / I_{DS} $ at +30 V
AIR1	Air	24 h	Yes	Yes	9.203	12.505	3.302	5.178e-04	2.137e-05	0.041
AIR2	Air	24 h	No	No	—	—	—	-4.791e-05	1.240e-03	25.885
AIR3	Air	24 h	Yes	Yes	10.706	13.151	2.445	4.839e-04	2.172e-05	0.045
AIR4	Air	24 h	Yes	Yes	4.286	11.162	6.875	1.944e-04	4.835e-06	0.025
AIR5	Air	24 h	Yes	Yes	4.927	8.444	3.517	2.899e-04	9.673e-06	0.033
AIR6	Air	24 h	Yes	Yes	6.280	9.246	2.966	2.478e-04	4.760e-06	0.019
AIR7	Air	24 h	Yes	Yes	5.576	9.922	4.345	1.954e-04	4.814e-06	0.025
AIR8	Air	24 h	Yes	Yes	6.470	10.194	3.724	1.816e-04	7.242e-06	0.040
PBS1	PBS	24 h	No	No	9.452	21.815	12.363	1.402e-04	1.589e-05	0.113
PBS2	PBS	24 h	No	No	—	—	—	2.214e-05	1.050e-02	474.285
PBS3	PBS	24 h	Yes	Yes	4.760	17.895	13.135	1.666e-04	9.121e-06	0.055
PBS4	PBS	24 h	No	No	—	—	—	3.686e-06	6.093e-06	1.653
PBS5	PBS	24 h	Yes	Yes	-2.783	12.364	15.147	1.433e-04	5.857e-07	0.004
PBS6	PBS	24 h	Yes	Yes	10.788	13.054	2.265	1.251e-04	8.494e-06	0.068
PBS7	PBS	24 h	Yes	No	-8.802	5.839	14.642	2.645e-04	1.821e-06	0.007
PBS8	PBS	24 h	Yes	Yes	0.367	9.743	9.376	2.122e-04	2.708e-06	0.013
AIR1	Air	48 h	Yes	Yes	8.484	12.071	3.587	5.310e-04	2.275e-05	0.043
AIR2	Air	48 h	No	No	—	—	—	2.342e-04	1.350e-04	0.576
AIR3	Air	48 h	Yes	Yes	10.080	12.796	2.716	4.126e-04	2.371e-05	0.057
AIR4	Air	48 h	Yes	Yes	8.457	13.655	5.198	1.432e-04	4.817e-06	0.034
AIR5	Air	48 h	Yes	Yes	6.697	9.593	2.896	2.570e-04	8.287e-06	0.032
AIR6	Air	48 h	Yes	Yes	7.366	9.773	2.407	2.322e-04	4.120e-06	0.018
AIR7	Air	48 h	Yes	Yes	6.640	10.774	4.134	1.922e-04	4.765e-06	0.025
AIR8	Air	48 h	Yes	Yes	7.129	10.666	3.536	1.818e-04	7.510e-06	0.041
PBS1	PBS	48 h	No	No	—	—	—	-2.248e-03	2.544e-03	1.132
PBS2	PBS	48 h	No	No	—	—	—	-1.048e-02	1.050e-02	1.002
PBS3	PBS	48 h	Yes	Yes	-0.367	17.379	17.747	1.937e-04	9.766e-06	0.050
PBS4	PBS	48 h	No	No	—	—	—	2.684e-06	4.583e-06	1.707
PBS5	PBS	48 h	No	No	-4.498	14.386	18.884	7.358e-05	5.768e-07	0.008
PBS6	PBS	48 h	Yes	Yes	8.224	12.684	4.460	1.291e-04	7.803e-06	0.060
PBS7	PBS	48 h	Yes	No	-12.489	11.399	23.887	1.991e-04	1.480e-06	0.007
PBS8	PBS	48 h	Yes	Yes	-5.731	7.592	13.323	2.247e-04	1.905e-06	0.008

Table B.1.4: Threshold, hysteresis, I_{DS} , and leakage metrics (Day 3-Day 5).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Forward V_{th} (V)	Reverse V_{th} (V)	Hysteresis (V)	I_{DS} at +30 V (A)	I_G at +30 V (A)	$ I_G / I_{DS} $ at +30 V
AIR1	Air	Day 3	Yes	Yes	11.125	13.659	2.534	4.100e-04	1.774e-05	0.043
AIR2	Air	Day 3	No	No	—	—	—	2.842e-06	5.522e-04	194.316
AIR3	Air	Day 3	Yes	Yes	9.760	12.575	2.815	2.126e-04	2.833e-05	0.133
AIR4	Air	Day 3	Yes	Yes	6.070	10.329	4.259	2.061e-04	4.823e-06	0.023
AIR5	Air	Day 3	Yes	Yes	5.601	8.179	2.578	2.901e-04	8.340e-06	0.029
AIR6	Air	Day 3	Yes	Yes	6.254	8.595	2.342	2.573e-04	4.441e-06	0.017
AIR7	Air	Day 3	Yes	Yes	4.691	9.272	4.581	2.204e-04	5.701e-06	0.026
AIR8	Air	Day 3	Yes	Yes	5.355	9.041	3.686	2.076e-04	8.711e-06	0.042
PBS1	PBS	Day 3	No	No	—	—	—	2.369e-05	9.048e-04	38.185
PBS2	PBS	Day 3	No	No	—	—	—	1.532e-05	1.050e-02	685.578
PBS3	PBS	Day 3	Yes	Yes	0.947	17.342	16.395	1.737e-04	1.112e-05	0.064
PBS4	PBS	Day 3	No	No	—	—	—	-2.244e-04	2.355e-04	1.049
PBS5	PBS	Day 3	Yes	No	-15.987	-28.531	-12.543	2.947e-04	5.273e-07	0.002
PBS6	PBS	Day 3	Yes	Yes	5.661	11.350	5.688	1.471e-04	7.547e-06	0.051
PBS7	PBS	Day 3	Yes	No	-20.354	-0.496	19.858	4.316e-04	1.669e-06	0.004
PBS8	PBS	Day 3	Yes	Yes	-5.867	6.567	12.434	2.404e-04	2.095e-06	0.009
AIR1	Air	Day 5	Yes	Yes	8.502	11.014	2.511	5.341e-04	2.375e-05	0.044
AIR2	Air	Day 5	No	No	—	—	—	—	—	—
AIR3	Air	Day 5	Yes	Yes	5.893	8.970	3.077	5.406e-04	3.190e-05	0.059
AIR4	Air	Day 5	Yes	Yes	5.121	8.644	3.523	2.429e-04	5.336e-06	0.022
AIR5	Air	Day 5	Yes	Yes	4.655	6.713	2.058	3.104e-04	8.736e-06	0.028
AIR6	Air	Day 5	Yes	Yes	5.315	7.157	1.842	2.294e-04	3.172e-06	0.014
AIR7	Air	Day 5	Yes	No	17.291	21.521	4.230	1.665e-05	7.578e-06	0.455
AIR8	Air	Day 5	Yes	Yes	5.831	7.791	1.959	1.965e-04	8.794e-06	0.045
PBS1	PBS	Day 5	No	No	—	—	—	—	—	—
PBS2	PBS	Day 5	No	No	—	—	—	—	—	—
PBS3	PBS	Day 5	Yes	No	0.242	—	—	2.329e-05	4.314e-10	0.000
PBS4	PBS	Day 5	No	No	—	—	—	—	—	—
PBS5	PBS	Day 5	Yes	No	-27.918	—	—	1.000e-03	5.071e-07	0.001
PBS6	PBS	Day 5	Yes	No	0.385	8.140	7.755	1.706e-04	5.750e-06	0.034
PBS7	PBS	Day 5	Yes	No	-3.065	—	—	2.149e-04	4.033e-07	0.002
PBS8	PBS	Day 5	Yes	No	-13.763	3.903	17.666	2.884e-04	1.961e-06	0.007

Table B.1.5: Subthreshold, switching, leakage, and output metrics (T0-3 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturated on-qualified	SS (V/dec)	SS leakage flag	I_{on}/I_{off}	I_{on}/I_{off} leakage flag	Max $ I_G $ (A)	Output QC state	Measurement status
AIR1	Air	T0	Yes	Yes	0.621	Yes	2.733e+06	Yes	2.025e-05	Interpretable	measured
AIR2	Air	T0	Yes	Yes	0.807	Yes	1.597e+06	Yes	7.643e-06	Interpretable	measured
AIR3	Air	T0	Yes	Yes	0.530	Yes	2.184e+06	Yes	1.571e-05	Interpretable	measured
AIR4	Air	T0	Yes	Yes	1.529	No	3.753e+05	Yes	5.836e-06	Interpretable	measured
AIR5	Air	T0	Yes	Yes	1.338	No	2.713e+05	Yes	8.218e-06	Interpretable	measured
AIR6	Air	T0	Yes	Yes	1.692	No	4.060e+05	Yes	4.840e-06	Interpretable	measured
AIR7	Air	T0	Yes	Yes	1.548	No	3.910e+05	Yes	7.500e-06	Interpretable	measured
AIR8	Air	T0	Yes	Yes	1.667	No	3.376e+05	Yes	8.188e-06	Interpretable	measured
PBS1	PBS	T0	Yes	Yes	0.550	Yes	1.978e+06	Yes	1.571e-05	Interpretable	measured
PBS2	PBS	T0	Yes	Yes	1.132	Yes	1.559e+05	Yes	6.911e-07	Interpretable	measured
PBS3	PBS	T0	Yes	Yes	2.178	Yes	1.012e+06	Yes	1.074e-05	Interpretable	measured
PBS4	PBS	T0	Yes	Yes	3.894	No	5.322e+05	Yes	6.190e-06	Interpretable	measured
PBS5	PBS	T0	Yes	Yes	1.785	No	1.938e+05	Yes	1.635e-06	Interpretable	measured
PBS6	PBS	T0	Yes	Yes	1.414	Yes	6.570e+05	Yes	8.883e-06	Interpretable	measured
PBS7	PBS	T0	Yes	Yes	1.699	No	3.833e+05	Yes	5.442e-06	Interpretable	measured
PBS8	PBS	T0	Yes	Yes	1.586	Yes	7.377e+05	Yes	7.896e-06	Interpretable	measured
AIR1	Air	3 h	Yes	Yes	0.495	Yes	4.919e+06	Yes	2.211e-05	Interpretable	measured
AIR2	Air	3 h	No	No	0.898	Yes	6.962e+07	Yes	2.816e-03	Interpretable	measured
AIR3	Air	3 h	Yes	Yes	1.129	Yes	1.995e+06	Yes	1.802e-05	Interpretable	measured
AIR4	Air	3 h	Yes	Yes	1.493	No	8.681e+04	Yes	6.455e-06	Interpretable	measured
AIR5	Air	3 h	Yes	Yes	1.290	No	7.072e+05	Yes	8.735e-06	Interpretable	measured
AIR6	Air	3 h	Yes	Yes	1.555	No	1.031e+05	Yes	6.322e-06	Interpretable	measured
AIR7	Air	3 h	Yes	Yes	1.509	No	2.346e+05	Yes	7.341e-06	Interpretable	measured
AIR8	Air	3 h	Yes	Yes	1.664	Yes	3.721e+05	Yes	8.342e-06	Interpretable	measured
PBS1	PBS	3 h	Yes	Yes	4.001	No	2.332e+04	Yes	1.204e-05	Interpretable	measured
PBS2	PBS	3 h	No	No	3.537	Yes	4.944e+02	Yes	1.919e-04	Interpretable	measured
PBS3	PBS	3 h	Yes	Yes	2.618	Yes	2.059e+06	Yes	8.454e-06	Interpretable	measured
PBS4	PBS	3 h	No	No	2.977	Yes	6.703e+05	Yes	5.787e-06	Abnormal	measured
PBS5	PBS	3 h	Yes	Yes	2.269	Yes	1.196e+05	Yes	6.607e-07	Interpretable	measured
PBS6	PBS	3 h	Yes	Yes	1.224	Yes	4.732e+05	Yes	8.176e-06	Interpretable	measured
PBS7	PBS	3 h	Yes	Yes	1.598	No	2.373e+05	Yes	6.160e-06	Interpretable	measured
PBS8	PBS	3 h	Yes	Yes	2.438	Yes	5.637e+05	Yes	4.456e-06	Interpretable	measured

Table B.1.6: Subthreshold, switching, leakage, and output metrics (6-12 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturations-qualified	SS (V/dec)	SS leakage flag	I_{on}/I_{off}	I_{on}/I_{off} leakage flag	Max $ I_G $ (A)	Output QC state	Measurement status
AIR1	Air	6 h	Yes	Yes	0.500	Yes	9.875e+06	Yes	1.800e-05	Interpretable	measured
AIR2	Air	6 h	Yes	Yes	0.729	Yes	3.000e+07	Yes	2.684e-04	Interpretable	measured
AIR3	Air	6 h	Yes	Yes	0.548	Yes	2.376e+07	Yes	1.140e-05	Interpretable	measured
AIR4	Air	6 h	Yes	Yes	1.355	No	4.289e+05	Yes	7.134e-06	Interpretable	measured
AIR5	Air	6 h	Yes	Yes	1.613	No	9.128e+04	Yes	1.021e-05	Interpretable	measured
AIR6	Air	6 h	Yes	Yes	1.487	No	1.919e+05	Yes	6.576e-06	Interpretable	measured
AIR7	Air	6 h	Yes	Yes	1.681	No	2.367e+05	Yes	6.210e-06	Interpretable	measured
AIR8	Air	6 h	Yes	Yes	1.634	No	3.772e+05	Yes	7.514e-06	Interpretable	measured
PBS1	PBS	6 h	Yes	Yes	3.078	No	7.901e+04	Yes	1.170e-05	Interpretable	measured
PBS2	PBS	6 h	No	No	—	No	1.848e+00	No	3.554e-06	Abnormal	measured
PBS3	PBS	6 h	Yes	Yes	3.636	No	9.019e+03	Yes	1.240e-05	Interpretable	measured
PBS4	PBS	6 h	No	No	7.404	Yes	7.613e+02	Yes	1.000e-03	Abnormal	measured
PBS5	PBS	6 h	Yes	Yes	2.248	Yes	9.201e+04	Yes	6.516e-07	Interpretable	measured
PBS6	PBS	6 h	Yes	Yes	1.610	Yes	8.821e+04	Yes	9.043e-06	Interpretable	measured
PBS7	PBS	6 h	Yes	Yes	5.827	No	3.884e+04	Yes	2.181e-06	Interpretable	measured
PBS8	PBS	6 h	Yes	Yes	2.192	Yes	5.379e+05	Yes	3.119e-06	Interpretable	measured
AIR1	Air	12 h	Yes	Yes	0.638	Yes	4.091e+06	Yes	1.660e-05	Interpretable	measured
AIR2	Air	12 h	Yes	Yes	2.137	Yes	4.528e+06	Yes	1.809e-04	Interpretable	measured
AIR3	Air	12 h	Yes	Yes	0.438	Yes	4.617e+06	Yes	1.890e-05	Interpretable	measured
AIR4	Air	12 h	Yes	Yes	1.611	No	4.738e+05	Yes	4.849e-06	Interpretable	measured
AIR5	Air	12 h	Yes	Yes	1.326	No	3.480e+05	Yes	8.934e-06	Interpretable	measured
AIR6	Air	12 h	Yes	Yes	10.469	No	2.890e+03	Yes	4.356e-06	Interpretable	measured
AIR7	Air	12 h	Yes	Yes	1.582	No	4.352e+05	Yes	4.843e-06	Interpretable	measured
AIR8	Air	12 h	Yes	Yes	1.711	No	4.216e+05	Yes	7.359e-06	Interpretable	measured
PBS1	PBS	12 h	Yes	Yes	3.207	No	6.158e+05	Yes	1.702e-05	Interpretable	measured
PBS2	PBS	12 h	No	No	—	No	1.444e+00	Yes	3.912e-03	Abnormal	measured
PBS3	PBS	12 h	Yes	Yes	1.620	Yes	2.481e+05	Yes	4.844e-06	Interpretable	measured
PBS4	PBS	12 h	No	No	1.858	Yes	8.342e+04	Yes	4.475e-06	Abnormal	measured
PBS5	PBS	12 h	Yes	Yes	1.910	Yes	7.613e+04	Yes	2.980e-07	Interpretable	measured
PBS6	PBS	12 h	Yes	Yes	1.588	Yes	4.819e+05	Yes	7.732e-06	Interpretable	measured
PBS7	PBS	12 h	Yes	Yes	2.318	Yes	3.847e+05	Yes	1.616e-06	Interpretable	measured
PBS8	PBS	12 h	Yes	Yes	1.944	Yes	6.096e+05	Yes	2.855e-06	Interpretable	measured

Table B.1.7: Subthreshold, switching, leakage, and output metrics (24-48 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturations-qualified	SS (V/dec)	SS leakage flag	I_{on}/I_{off}	I_{on}/I_{off} leakage flag	Max $ I_G $ (A)	Output QC state	Measurement status
AIR1	Air	24 h	Yes	Yes	0.501	Yes	3.929e+06	Yes	2.137e-05	Interpretable	measured
AIR2	Air	24 h	No	No	—	No	6.190e+02	Yes	1.621e-03	Abnormal	measured
AIR3	Air	24 h	Yes	Yes	2.318	No	2.054e+06	Yes	2.172e-05	Interpretable	measured
AIR4	Air	24 h	Yes	Yes	1.608	No	5.940e+05	Yes	4.887e-06	Interpretable	measured
AIR5	Air	24 h	Yes	Yes	1.169	Yes	6.007e+05	Yes	9.673e-06	Interpretable	measured
AIR6	Air	24 h	Yes	Yes	4.253	Yes	2.430e+04	Yes	4.760e-06	Interpretable	measured
AIR7	Air	24 h	Yes	Yes	1.598	No	4.956e+05	Yes	4.823e-06	Interpretable	measured
AIR8	Air	24 h	Yes	Yes	1.697	No	5.473e+05	Yes	7.242e-06	Interpretable	measured
PBS1	PBS	24 h	No	No	9.989	No	4.674e+02	Yes	1.589e-05	Abnormal	measured
PBS2	PBS	24 h	No	No	—	No	1.081e+00	Yes	1.100e-02	Abnormal	measured
PBS3	PBS	24 h	Yes	Yes	3.299	Yes	2.503e+04	Yes	9.121e-06	Interpretable	measured
PBS4	PBS	24 h	No	No	2.608	Yes	1.135e+04	Yes	3.803e-04	Abnormal	measured
PBS5	PBS	24 h	Yes	Yes	2.452	Yes	9.290e+04	Yes	5.857e-07	Interpretable	measured
PBS6	PBS	24 h	Yes	Yes	2.523	Yes	3.210e+05	Yes	8.494e-06	Interpretable	measured
PBS7	PBS	24 h	Yes	No	2.091	Yes	9.721e+04	Yes	1.821e-06	Interpretable	measured
PBS8	PBS	24 h	Yes	Yes	1.933	Yes	5.680e+05	Yes	2.708e-06	Interpretable	measured
AIR1	Air	48 h	Yes	Yes	0.426	Yes	3.824e+06	Yes	2.275e-05	Interpretable	measured
AIR2	Air	48 h	No	No	2.226	Yes	5.073e+02	Yes	5.215e-04	Abnormal	measured
AIR3	Air	48 h	Yes	Yes	0.780	Yes	1.955e+06	Yes	2.371e-05	Interpretable	measured
AIR4	Air	48 h	Yes	Yes	6.167	No	6.120e+03	Yes	4.873e-06	Interpretable	measured
AIR5	Air	48 h	Yes	Yes	3.352	No	7.311e+04	Yes	8.287e-06	Interpretable	measured
AIR6	Air	48 h	Yes	Yes	7.999	No	6.029e+03	Yes	4.120e-06	Interpretable	measured
AIR7	Air	48 h	Yes	Yes	1.628	No	4.178e+05	Yes	4.765e-06	Interpretable	measured
AIR8	Air	48 h	Yes	Yes	1.584	No	4.243e+05	Yes	7.510e-06	Interpretable	measured
PBS1	PBS	48 h	No	No	5.313	Yes	4.706e+02	Yes	3.420e-03	Interpretable	measured
PBS2	PBS	48 h	No	No	—	No	1.608e+01	Yes	1.100e-02	Abnormal	measured
PBS3	PBS	48 h	Yes	Yes	2.924	Yes	1.917e+05	Yes	9.766e-06	Interpretable	measured
PBS4	PBS	48 h	No	No	1.318	Yes	2.623e+04	Yes	4.998e-06	Abnormal	measured
PBS5	PBS	48 h	No	No	7.129	No	6.546e+02	Yes	5.768e-07	Abnormal	measured
PBS6	PBS	48 h	Yes	Yes	2.942	Yes	5.913e+04	Yes	7.803e-06	Interpretable	measured
PBS7	PBS	48 h	Yes	No	6.606	Yes	4.855e+03	Yes	1.480e-06	Interpretable	measured
PBS8	PBS	48 h	Yes	Yes	2.273	Yes	1.773e+05	Yes	1.905e-06	Interpretable	measured

Table B.1.8: Subthreshold, switching, leakage, and output metrics (Day 3-Day 5).

Device	Exposure	Timepoint	Electrically interpretable	Saturations-qualified	SS (V/dec)	SS leakage flag	I_{on}/I_{off}	I_{on}/I_{off} leakage flag	Max $ I_G $ (A)	Output QC state	Measurement status
AIR1	Air	Day 3	Yes	Yes	0.458	Yes	2.553e+06	Yes	1.774e-05	Interpretable	measured
AIR2	Air	Day 3	No	No	1.457	Yes	2.965e+06	Yes	9.357e-04	Abnormal	measured
AIR3	Air	Day 3	Yes	Yes	0.459	Yes	1.437e+07	Yes	2.833e-05	Interpretable	measured
AIR4	Air	Day 3	Yes	Yes	4.679	Yes	2.658e+04	Yes	4.823e-06	Interpretable	measured
AIR5	Air	Day 3	Yes	Yes	1.922	No	2.230e+05	Yes	8.340e-06	Interpretable	measured
AIR6	Air	Day 3	Yes	Yes	8.416	No	4.316e+03	Yes	4.441e-06	Interpretable	measured
AIR7	Air	Day 3	Yes	Yes	2.445	No	3.213e+04	Yes	5.701e-06	Interpretable	measured
AIR8	Air	Day 3	Yes	Yes	1.570	No	4.749e+05	Yes	8.711e-06	Interpretable	measured
PBS1	PBS	Day 3	No	No	10.622	Yes	4.789e+01	Yes	9.152e-04	Abnormal	measured
PBS2	PBS	Day 3	No	No	—	No	1.054e+00	Yes	1.100e-02	Abnormal	measured
PBS3	PBS	Day 3	Yes	Yes	3.200	Yes	1.170e+05	Yes	1.112e-05	Interpretable	measured
PBS4	PBS	Day 3	No	No	7.960	Yes	2.314e+03	Yes	2.467e-04	Abnormal	measured
PBS5	PBS	Day 3	Yes	No	3.567	Yes	3.746e+03	Yes	5.273e-07	Interpretable	measured
PBS6	PBS	Day 3	Yes	Yes	2.069	Yes	2.442e+05	Yes	7.547e-06	Interpretable	measured
PBS7	PBS	Day 3	Yes	No	2.430	Yes	1.608e+05	Yes	1.669e-06	Interpretable	measured
PBS8	PBS	Day 3	Yes	Yes	2.118	Yes	2.183e+05	Yes	2.095e-06	Interpretable	measured
AIR1	Air	Day 5	Yes	Yes	0.486	Yes	9.706e+06	Yes	2.375e-05	Interpretable	measured
AIR2	Air	Day 5	No	No	—	No	—	No	—	not measured	not continued after Day 3 operational failure
AIR3	Air	Day 5	Yes	Yes	0.418	Yes	3.519e+07	Yes	3.190e-05	Interpretable	measured
AIR4	Air	Day 5	Yes	Yes	1.394	No	2.293e+05	Yes	5.336e-06	Interpretable	measured
AIR5	Air	Day 5	Yes	Yes	1.283	No	2.333e+05	Yes	8.736e-06	Interpretable	measured
AIR6	Air	Day 5	Yes	Yes	1.328	Yes	3.919e+05	Yes	3.172e-06	Interpretable	measured
AIR7	Air	Day 5	Yes	No	1.344	Yes	1.861e+04	Yes	7.578e-06	Interpretable	measured
AIR8	Air	Day 5	Yes	Yes	1.286	Yes	2.698e+05	Yes	8.794e-06	Interpretable	measured
PBS1	PBS	Day 5	No	No	—	No	—	No	—	not measured	not continued after Day 3 operational failure
PBS2	PBS	Day 5	No	No	—	No	—	No	—	not measured	not continued after Day 3 operational failure
PBS3	PBS	Day 5	Yes	No	17.041	No	5.162e+01	No	5.172e-10	Interpretable	measured
PBS4	PBS	Day 5	No	No	—	No	—	No	—	not measured	not continued after Day 3 operational failure
PBS5	PBS	Day 5	Yes	No	7.981	No	3.061e+02	No	5.071e-07	Interpretable	measured
PBS6	PBS	Day 5	Yes	No	1.887	Yes	1.035e+05	Yes	5.750e-06	Interpretable	measured
PBS7	PBS	Day 5	Yes	No	2.911	Yes	1.792e+05	Yes	4.033e-07	Interpretable	measured
PBS8	PBS	Day 5	Yes	No	1.772	Yes	7.681e+04	Yes	1.961e-06	Interpretable	measured

Table B.1.9: Quality-control and metric-validity records (T0).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{ds}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR3	Air	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR7	Air	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS1	PBS	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS2	PBS	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS3	PBS	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS4	PBS	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS5	PBS	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS6	PBS	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	T0	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS8	PBS	T0	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.10: Quality-control and metric-validity records (3 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{ds}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	3 h	No	No	measured	No	leakage - limited at 10- μ A crossing	Yes	Yes	Yes	Yes	review/indeterminate	Interpretable	—	No
AIR3	Air	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	3 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	3 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	3 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR7	Air	3 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS1	PBS	3 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS2	PBS	3 h	No	No	measured	No	leakage - limited at 10- μ A crossing	Yes	No	Yes	Yes	review/indeterminate	Interpretable	—	No
PBS3	PBS	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS4	PBS	3 h	No	No	measured	Yes	—	No	No	Yes	Yes	present	Abnormal	—	No
PBS5	PBS	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS6	PBS	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	3 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS8	PBS	3 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.11: Quality-control and metric-validity records (6 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{ds}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	6 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	6 h	Yes	Yes	measured	No	leakage - limited at 10- μ A crossing	Yes	No	Yes	Yes	—	Interpretable	—	No
AIR3	Air	6 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR7	Air	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS1	PBS	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS2	PBS	6 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	No	No	No	absent	Abnormal	—	No
PBS3	PBS	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS4	PBS	6 h	No	No	measured	No	leakage - limited at 10- μ A crossing	Yes	Yes	Yes	Yes	review/indeterminate	Abnormal	—	No
PBS5	PBS	6 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS6	PBS	6 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	6 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS8	PBS	6 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.12: Quality-control and metric-validity records (12 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{ds}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	12 h	Yes	Yes	measured	No	leakage-limited at 10- μA crossing	Yes	No	Yes	Yes	—	Interpretable	—	No
AIR3	Air	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	12 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	12 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	12 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR7	Air	12 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	12 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS1	PBS	12 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS2	PBS	12 h	No	No	measured	No	no unique interpretable 10- μA rising crossing	No	Yes	No	Yes	—	Abnormal	—	No
PBS3	PBS	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS4	PBS	12 h	No	No	measured	No	no unique interpretable 10- μA rising crossing	No	Yes	Yes	Yes	—	Abnormal	—	No
PBS5	PBS	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS6	PBS	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS8	PBS	12 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.13: Quality-control and metric-validity records (24 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{ds}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	24 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	No	Yes	review/indeterminate	Abnormal	—	No
AIR3	Air	24 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR4	Air	24 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR6	Air	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR7	Air	24 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	24 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS1	PBS	24 h	No	No	measured	Yes	—	No	No	No	Yes	present	Abnormal	—	No
PBS2	PBS	24 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	No	Yes	absent	Abnormal	—	No
PBS3	PBS	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS4	PBS	24 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	Yes	Yes	review/indeterminate	Abnormal	—	No
PBS5	PBS	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS6	PBS	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	24 h	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS8	PBS	24 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.14: Quality-control and metric-validity records (48 h).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{DS}(+30\text{ V})$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	48 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	48 h	No	No	measured	No	leakage-limited at 10- μ A crossing	Yes	No	Yes	Yes	review/in determinate	Abnormal	—	No
AIR3	Air	48 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	48 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	48 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	48 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR7	Air	48 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	48 h	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS1	PBS	48 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	Yes	Yes	review/in determinate	Interpretable	—	No
PBS2	PBS	48 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	No	Yes	review/in determinate	Abnormal	—	No
PBS3	PBS	48 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS4	PBS	48 h	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	Yes	Yes	review/in determinate	Abnormal	—	No
PBS5	PBS	48 h	No	No	measured	Yes	—	No	No	No	Yes	present	Abnormal	—	No
PBS6	PBS	48 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	48 h	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS8	PBS	48 h	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.15: Quality-control and metric-validity records (Day 3).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{DS}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	Day 3	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	Yes	Yes	review/indeterminate	Abnormal	—	Yes
AIR3	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR5	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR7	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR8	Air	Day 3	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
PBS1	PBS	Day 3	No	No	measured	No	leakage-limited at 10- μ A crossing	Yes	Yes	Yes	Yes	review/indeterminate	Abnormal	—	Yes
PBS2	PBS	Day 3	No	No	measured	No	no unique interpretable 10- μ A rising crossing	No	Yes	No	Yes	absent	Abnormal	—	Yes
PBS3	PBS	Day 3	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS4	PBS	Day 3	No	No	measured	No	leakage-limited at 10- μ A crossing	Yes	Yes	Yes	Yes	review/indeterminate	Abnormal	—	Yes
PBS5	PBS	Day 3	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS6	PBS	Day 3	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	Day 3	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS8	PBS	Day 3	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.16: Quality-control and metric-validity records (Day 5).

Device	Exposure	Timepoint	Electrically interpretable	Saturation-qualified	Measurement status	Forward V_{th} extractable	Forward V_{th} invalid reason	V_{th} leakage-limited	$I_{DS}(+30V)$ leakage-limited	SS leakage-limited	On/off leakage-limited	Transfer modulation review	Output QC state	Output note	Stopped after Day 3 failure
AIR1	Air	Day 5	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR2	Air	Day 5	No	No	not continued after Day 3 operational failure	No	—	No	No	No	No	not measured	not measured	—	Yes
AIR3	Air	Day 5	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR4	Air	Day 5	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR5	Air	Day 5	Yes	Yes	measured	Yes	—	No	No	No	Yes	present	Interpretable	—	No
AIR6	Air	Day 5	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR7	Air	Day 5	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
AIR8	Air	Day 5	Yes	Yes	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS1	PBS	Day 5	No	No	not continued after Day 3 operational failure	No	—	No	No	No	No	not measured	not measured	—	Yes
PBS2	PBS	Day 5	No	No	not continued after Day 3 operational failure	No	—	No	No	No	No	not measured	not measured	—	Yes
PBS3	PBS	Day 5	Yes	No	measured	Yes	—	No	No	No	No	present	Interpretable	—	No
PBS4	PBS	Day 5	No	No	not continued after Day 3 operational failure	No	—	No	No	No	No	not measured	not measured	—	Yes
PBS5	PBS	Day 5	Yes	No	measured	Yes	—	No	No	No	No	present	Interpretable	—	No
PBS6	PBS	Day 5	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS7	PBS	Day 5	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No
PBS8	PBS	Day 5	Yes	No	measured	Yes	—	No	No	Yes	Yes	present	Interpretable	—	No

Table B.1.17: Repeat measurements and troubleshooting events.

Timepoint	Device	Event type	Note
3 h	PBS2	Repeat after unexpected/Abnormal behavior	failure-sequence repeat
6 h	PBS4	Contact troubleshooting	contact adjustment
48 h	PBS1	Repeat after unexpected/Abnormal behavior	failure-sequence repeat
48 h	PBS5	Repeat after unexpected/Abnormal behavior	output repeat
Day 3	PBS4	Repeat after unexpected/Abnormal behavior	failure-sequence repeat
48 h	PBS7	Wider diagnostic sweep	to -50 V
48 h	PBS7	Wider diagnostic sweep	to -80 V
Day 3	PBS5	Wider diagnostic sweep	to -80 V
Day 3	PBS7	Wider diagnostic sweep	to -50 V

Table B.1.18: Secondary paired threshold, I_{DS} , and subthreshold metrics.

Timepoint	Exposure	Electrically interpretable, n/8	Saturation-qualified, n/8	Δ reverse V_{th} (V)	$\log_{10} I_{DS}(+30 \text{ V}) $ ratio	$\log_{10}$ SS ratio
T0	Air	8/8	8/8	0 ± 0 (n=8)	0 ± 0 (n=8)	0 ± 0 (n=8)
T0	PBS	8/8	8/8	0 ± 0 (n=8)	0 ± 0 (n=8)	0 ± 0 (n=8)
3 h	Air	7/8	7/8	-0.46 ± 0.35 (n=7)	0.026 ± 0.024 (n=7)	0.025 ± 0.139 (n=7)
3 h	PBS	6/8	6/8	3.496 ± 3.23 (n=6)	-0.1086 ± 0.0853 (n=6)	0.1905 ± 0.341 (n=6)
6 h	Air	8/8	8/8	-1.125 ± 0.967 (n=7)	0.04373 ± 0.0887 (n=8)	-0.01537 ± 0.0572 (n=8)
6 h	PBS	6/8	6/8	2.829 ± 3.09 (n=6)	-0.1939 ± 0.202 (n=6)	0.3004 ± 0.278 (n=6)
12 h	Air	8/8	8/8	-1.036 ± 1.14 (n=7)	0.05427 ± 0.069 (n=8)	0.1479 ± 0.302 (n=8)
12 h	PBS	6/8	6/8	3.516 ± 2.94 (n=6)	-0.2344 ± 0.281 (n=6)	0.1567 ± 0.311 (n=6)
24 h	Air	7/8	7/8	-1.279 ± 1.38 (n=7)	0.03781 ± 0.0691 (n=7)	0.1333 ± 0.277 (n=7)
24 h	PBS	5/8	4/8	-0.189 ± 3.69 (n=5)	-0.03055 ± 0.134 (n=5)	0.1491 ± 0.0689 (n=5)
48 h	Air	7/8	7/8	-0.6074 ± 2.32 (n=7)	-0.001926 ± 0.0985 (n=7)	0.2404 ± 0.325 (n=7)
48 h	PBS	4/8	3/8	0.1364 ± 2.55 (n=4)	-0.02915 ± 0.0947 (n=4)	0.298 ± 0.212 (n=4)
Day 3	Air	7/8	7/8	-1.704 ± 1.29 (n=7)	-0.005935 ± 0.0955 (n=7)	0.1883 ± 0.305 (n=7)
Day 3	PBS	5/8	3/8	-10.72 ± 17.2 (n=5)	0.1031 ± 0.217 (n=5)	0.1828 ± 0.0679 (n=5)
Day 5	Air	7/8	6/8	-1.681 ± 5.82 (n=7)	-0.08799 ± 0.433 (n=7)	-0.07826 ± 0.038 (n=7)
Day 5	PBS	5/8	0/8	-3.043 ± 3.34 (n=2)	0.002777 ± 0.644 (n=5)	0.3902 ± 0.365 (n=5)

Table B.1.19: Secondary paired switching and leakage metrics.

Timepoint	Exposure	Electrically interpretable, n/8	Saturation-qualified, n/8	SS leakage-flagged / numeric	$\log_{10} I_{on}/I_{off}$ ratio	I_{on}/I_{off} leakage-flagged / numeric	$\log_{10} \max I_G $ ratio	$ I_G / I_{DS} $ at +30 V
T0	Air	8/8	8/8	3/8	0 ± 0 (n=8)	8/8	0 ± 0 (n=8)	0.03648 ± 0.0109 (n=8)
T0	PBS	8/8	8/8	5/8	0 ± 0 (n=8)	8/8	0 ± 0 (n=8)	0.03761 ± 0.0148 (n=8)
3 h	Air	7/8	7/8	3/7	0.679 ± 2.193 (n=7)	7/7	0.04581 ± 0.0414 (n=6)	0.03605 ± 0.00947 (n=6)
3 h	PBS	6/8	6/8	4/6	-0.3828 ± 0.782 (n=6)	6/6	-0.1406 ± 0.159 (n=6)	0.03912 ± 0.0227 (n=6)
6 h	Air	8/8	8/8	3/8	0.2447 ± 0.645 (n=8)	8/8	0.1938 ± 0.555 (n=8)	0.1029 ± 0.193 (n=8)
6 h	PBS	6/8	6/8	3/6	-0.9626 ± 0.703 (n=6)	6/6	-0.2096 ± 0.218 (n=6)	0.06065 ± 0.0611 (n=6)
12 h	Air	8/8	8/8	3/8	-0.1053 ± 0.836 (n=8)	8/8	0.1302 ± 0.509 (n=8)	0.08233 ± 0.146 (n=8)
12 h	PBS	6/8	6/8	5/6	-0.2898 ± 0.251 (n=6)	6/6	-0.3466 ± 0.291 (n=6)	0.05016 ± 0.0481 (n=6)
24 h	Air	7/8	7/8	3/7	-0.03347 ± 0.536 (n=7)	7/7	-0.01351 ± 0.108 (n=7)	0.03259 ± 0.00985 (n=7)
24 h	PBS	5/8	4/8	5/5	-0.5893 ± 0.594 (n=5)	5/5	-0.2953 ± 0.229 (n=5)	0.02927 ± 0.0298 (n=5)
48 h	Air	7/8	7/8	2/7	-0.5656 ± 0.881 (n=7)	7/7	-0.02142 ± 0.117 (n=7)	0.03572 ± 0.013 (n=7)
48 h	PBS	4/8	3/8	4/4	-1.071 ± 0.58 (n=4)	4/4	-0.3201 ± 0.314 (n=4)	0.03169 ± 0.0277 (n=4)
Day 3	Air	7/8	7/8	3/7	-0.4795 ± 0.956 (n=7)	7/7	-0.001039 ± 0.124 (n=7)	0.04483 ± 0.0401 (n=7)
Day 3	PBS	5/8	3/8	5/5	-0.7973 ± 0.558 (n=5)	5/5	-0.3273 ± 0.277 (n=5)	0.02594 ± 0.0294 (n=5)
Day 5	Air	7/8	6/8	5/7	0.006134 ± 0.771 (n=7)	7/7	0.03092 ± 0.147 (n=7)	0.09534 ± 0.159 (n=7)
Day 5	PBS	5/8	0/8	3/5	-1.842 ± 1.66 (n=5)	3/5	-1.35 ± 1.69 (n=5)	0.008582 ± 0.0143 (n=5)

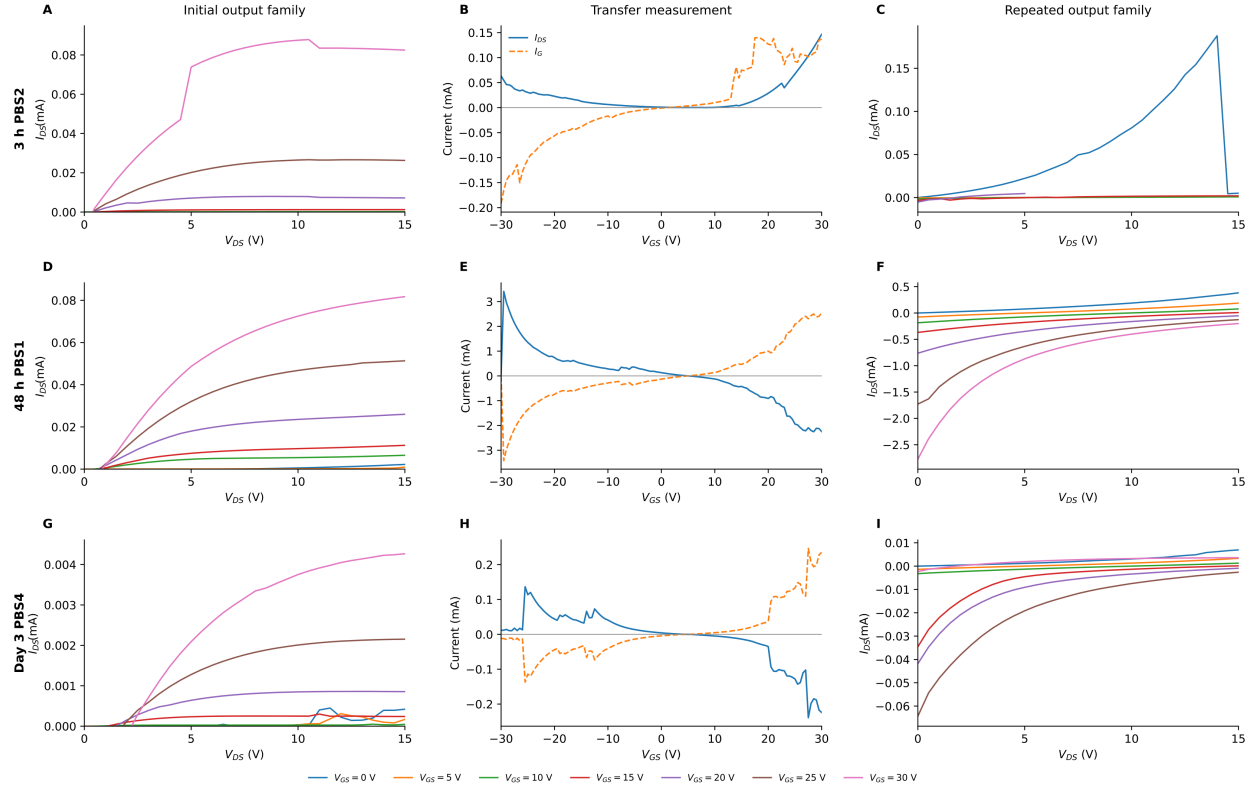


Figure B.1.1. PBS output-transfer-output failure sequences. Rows show three representative within-session sequences: 3 h PBS2 (A-C), 48 h PBS1 (D-F), and Day 3 PBS4 (G-I). In each row, the first panel is the initial output family, the center panel is the subsequently acquired transfer measurement, and the third panel is the repeated output family. Output-family colors denote gate-source voltage: 0 V blue, 5 V orange, 10 V green, 15 V red, 20 V purple, 25 V brown, and 30 V pink. In transfer panels B, E, and H, the solid blue trace is drain-to-source current and the dashed orange trace is gate current. The sequences document rapid within-session changes associated with the short-like electrical phenotype and do not identify the physical location or mechanism of the abnormal current pathway.

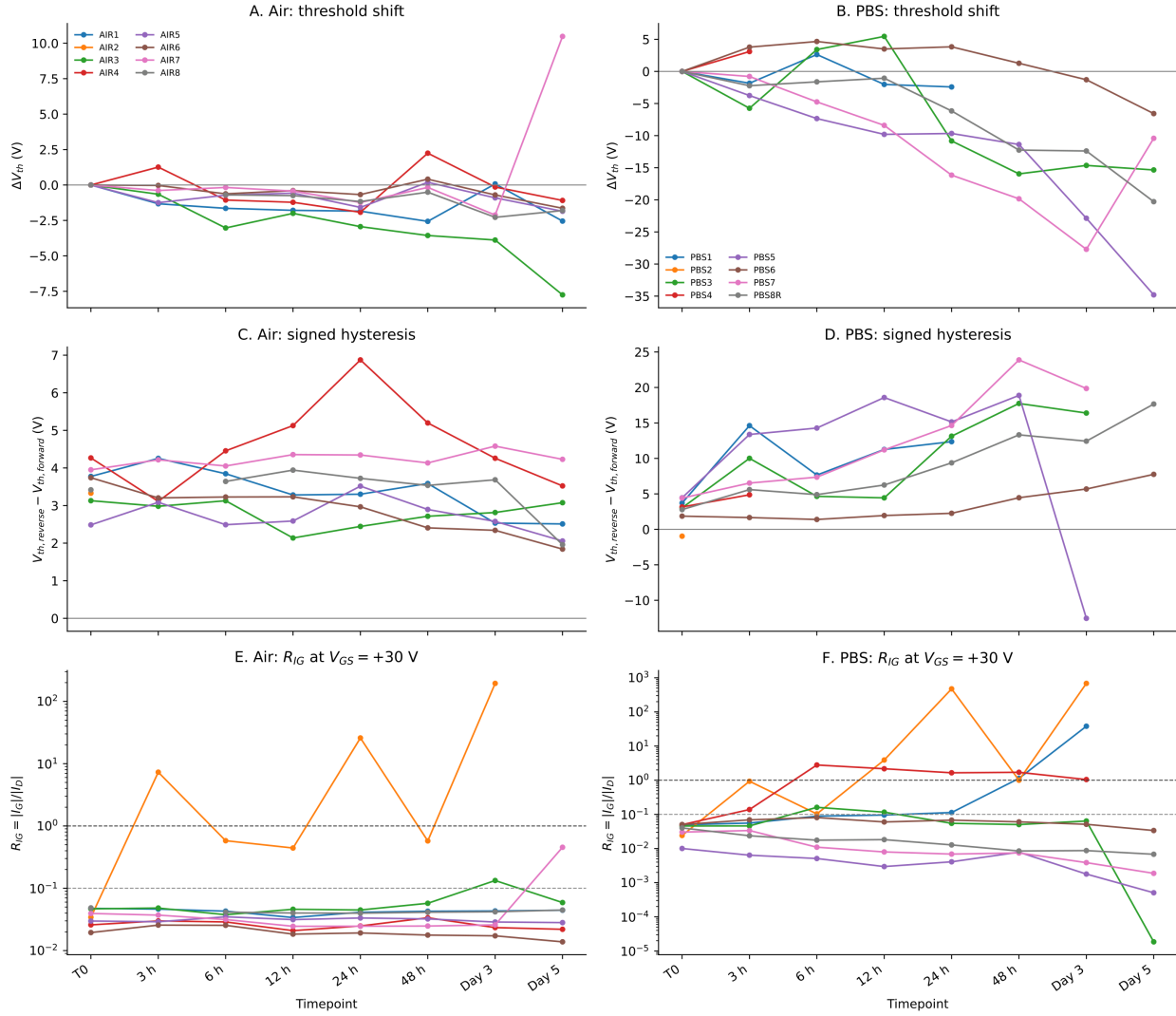


Figure B.1.2. Threshold shift, hysteresis, and leakage trajectories. Panels A and B show within-device forward threshold-voltage change from T0 for air and PBS devices, respectively; panels C and D show signed hysteresis; and panels E and F show the relative gate-current ratio at gate-source voltage +30 V on a logarithmic scale. Colors identify individual FETs within the air and PBS groups as listed in the panel legends, and lines connect observed timepoints only. Horizontal zero lines in A-D mark no threshold change or zero signed hysteresis, and dotted reference lines in E-F mark relative gate-current ratios of 0.1 and 1.

B.2 Additional-Wafer PBS Replication Test Data

This section retains the numerical metrics and unique across-test trajectory summaries for the additional-wafer PBS replication tests described in Section 3.3. Tables B.2.1 through B.2.4 and Figures B.2.1–B.2.2 are retained here. Complete electrical records for Tests 1–12 are provided in Supplemental Data S1.

Table B.2.1. Threshold, hysteresis, I_{DS} , and leakage metrics (Tests 1-6).

Test	Wafer	Time	10- μ A V_{th} within -30 to +30 V (V)	10- μ A crossing, full sweep (V)	Hysteresis (V)	I_{DS} at +30 V (μ A)	$ I_G / I_{DS} $ at +30 V
1	A	Baseline	4.31	4.31	5.07	170.8	0.0374
1	A	24 h	—	—	—	5.885	0.257
1	A	48 h	—	—	—	6.387	1.64e+03
2	A	Baseline	8.83	8.83	4.50	92.31	0.0592
2	A	24 h	-16.02	-16.02	26.49	121.9	0.0199
2	A	48 h	-27.95	-27.95	35.77	319.6	0.0087
3	A	Baseline	4.78	4.78	4.35	152.9	0.0406
3	A	24 h	-14.45	-14.45	25.12	198.1	0.014
3	A	48 h	-19.99	-19.99	33.16	172.5	0.0196
4	A	Baseline	5.08	5.08	2.18	191.9	0.0426
4	A	24 h	-19.22	-19.22	25.55	322	0.0119
4	A	48 h	-26.61	-26.61	29.85	247.1	0.0167
5	B	Baseline	11.67	11.67	9.25	112.8	0.0442
5	B	24 h	-13.92	-13.92	—	149.8	0.0272
5	B	48 h	—	—	—	312.3	0.0109
6	B	Baseline	12.75	12.75	6.42	74.35	0.0461

Table B.2.2. Threshold, hysteresis, I_{DS} , and leakage metrics (Tests 6-12).

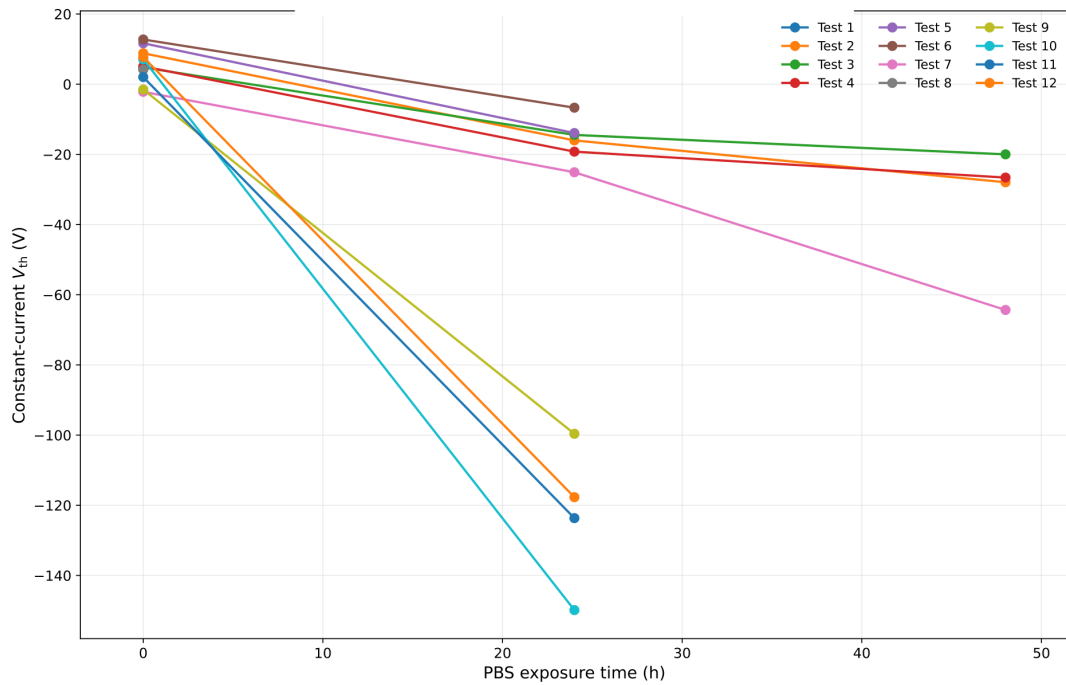
Test	Wafer	Time	10- μ A V_{th} within -30 to +30 V (V)	10- μ A crossing, full sweep (V)	Hysteresis (V)	I_{DS} at +30 V (μ A)	$ I_G / I_{DS} $ at +30 V
6	B	24 h	-6.69	-6.69	25.81	84.41	0.0258
6	B	48 h	—	—	—	429.9	0.00485
7	B	Baseline	-2.17	-2.17	10.95	175.2	0.0238
7	B	24 h	-25.09	-25.09	26.53	276.2	0.0111
7	B	48 h	—	-64.33	—	360.7	0.00985
8	B	Baseline	4.15	4.15	8.25	123.3	0.0472
8	B	24 h	—	—	—	324.3	0.0466
8	B	48 h	—	—	—	—	—
9	C	Baseline	-1.51	-1.51	14.16	168.6	0.0272
9	C	24 h	—	-99.63	—	841.1	0.00604
10	C	Baseline	7.11	7.11	6.99	110.5	0.0434
10	C	24 h	—	-149.87	—	538.8	0.00902
11	C	Baseline	2.03	2.03	7.31	164.5	0.0305
11	C	24 h	—	-123.66	—	253.6	0.022
12	C	Baseline	7.73	7.73	6.23	109.4	0.0396
12	C	24 h	—	-117.69	—	593.8	0.00757

Table B.2.3. Modulation, subthreshold, and output metrics (Tests 1-6).

Test	Wafer	Time	I_{on}/I_{off}	SS (V/dec)	SS valid	Output gate- separation ratio	Output traces with $ I_G / I_{DS} \geq 1$
1	A	Baseline	7234	3.71	No	737.8	2
1	A	24 h	53.47	18.74	No	10.84	0
1	A	48 h	2.31	—	No	—	—
2	A	Baseline	1868	4.25	No	286.2	0
2	A	24 h	78.5	18.32	Yes	225.7	0
2	A	48 h	40.79	24.54	Yes	20.76	0
3	A	Baseline	1383	4.54	No	240.1	0
3	A	24 h	692.8	6.88	Yes	377.1	0
3	A	48 h	76.94	18.59	Yes	7.522	0
4	A	Baseline	1767	4.18	Yes	785.3	0
4	A	24 h	361.8	9.12	Yes	156.3	2
4	A	48 h	31.58	33.86	Yes	3.096	0
5	B	Baseline	5759	4.29	No	1323	0
5	B	24 h	205.9	12.03	Yes	28.25	0
5	B	48 h	17.48	43.61	Yes	1.902	0
6	B	Baseline	7863	3.34	No	32.94	0

Table B.2.4. Modulation, subthreshold, and output metrics (Tests 6-12).

Test	Wafer	Time	I_{on}/I_{off}	SS (V/dec)	SS valid	Output gate-separation ratio	Output traces with $ I_G / I_{DS} \geq 1$
6	B	24 h	346.6	9.05	Yes	17.6	0
6	B	48 h	5.758	—	No	1.482	0
7	B	Baseline	4891	4.70	No	893.1	0
7	B	24 h	58.02	20.16	Yes	31.69	0
7	B	48 h	7.658	—	No	1.363	0
8	B	Baseline	2101	5.29	Yes	476.4	0
8	B	24 h	16.78	44.19	Yes	51.63	0
8	B	48 h	—	—	—	4.123	0
9	C	Baseline	1.793e+04	4.50	No	823.1	0
9	C	24 h	1.845	—	No	1.928	0
10	C	Baseline	1.405e+04	2.73	No	784.5	0
10	C	24 h	1.523	—	No	2.22	0
11	C	Baseline	1.315e+04	3.11	No	1118	0
11	C	24 h	3.177	—	No	1.885	0
12	C	Baseline	1.908e+04	3.13	No	1905	0
12	C	24 h	2.893	—	No	1.126	0

**Figure B.2.1.** Threshold-voltage trajectories during PBS exposure. Each colored line represents one of Tests 1-12, with test identifiers given in the legend. Points show the available constant-current threshold values at the measured PBS-exposure times, and lines connect observed measurements only; absent later points indicate that no value is plotted at that timepoint under the recorded test schedule or validity criteria.

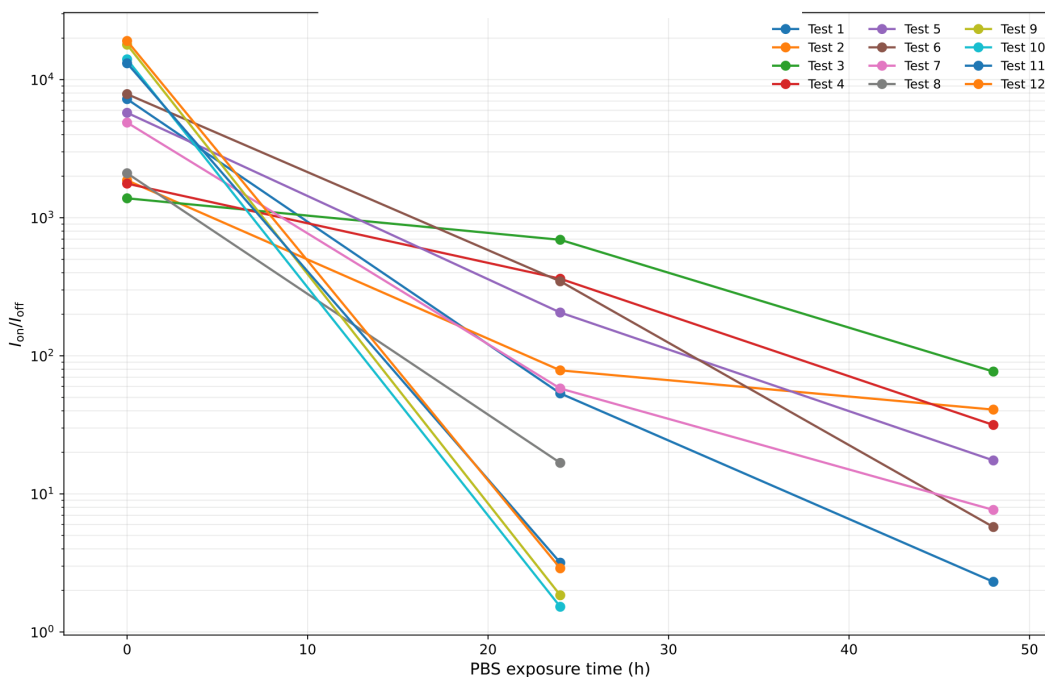


Figure B.2.2. On/off-ratio trajectories during PBS exposure. Each colored line represents one of Tests 1-12 using the same test-color mapping as Figure B.2.1. The on/off ratio is plotted on a logarithmic scale; points show available measurements and lines connect observed timepoints only.

B.3 Dodecanethiol Electrode Passivation Electrical Compatibility Data

This section retains Table B.3.1, which reports paired device-level electrical responses from the dodecanethiol (DDT) electrode-passivation compatibility experiment described in Section 3.5.

Complete before- and after-treatment transfer, gate-current, and output characteristics for all eight FETs are provided in Supplemental Data S1.

Table B.3.1. Individual-device paired electrical responses to DDT treatment.

Device	Δ forward V_{th} (V)	Δ reverse V_{th} (V)	Δ signed hysteresis (V)	$\log_{10} I_{Ds}(+30 \text{ V}) $ ratio	$\log_{10}$ SS ratio	$\log_{10}$ max $ I_G $ ratio	ΔR_{IG} at +30 V	$\log_{10} I_{on}/I_{off}$ ratio
P1D1	-4.942	-6.123	-1.180	0.144	0.068	0.092	-0.005	-0.178
P1D2	-4.863	-3.834	1.029	0.140	0.428	0.066	-0.005	-1.054
P1D3	-4.339	-10.633	-6.294	0.179	-0.446	0.096	-0.010	1.577
P1D4	-6.147	-10.994	-4.846	0.227	0.006	-0.183	-0.095	0.102
P2D1	-5.588	-6.895	-1.307	0.252	-0.289	-0.059	-0.129	0.444
P2D2	-0.531	-2.272	-1.741	-0.179	0.164	0.015	0.102	-0.113
P2D3	-9.932	—	—	0.375	-0.463	0.059	-0.103	1.674
P2D4	-2.996	-4.785	-1.788	-0.239	-0.169	-0.051	0.136	-1.168

B.4 Thermal-Conditioning Protocol Development Data

This section retains the tabulated supporting data for the thermal-conditioning temperature-selection experiment described in Section 3.6. Tables B.4.1 through B.4.3 summarize the 60 and 70 °C candidate conditions and device-level PBS recovery, switching, leakage, and quality-control metrics. Complete device-level electrical characterization across all four tested temperatures is provided in Supplemental Data S1.

Table B.4.1. 60 and 70 °C PBS recovery comparison.

Metric	60 °C PBS	70 °C PBS
POST threshold extractable	3/4	2/3 evaluable†
POST SS (V/dec)	3.12 ± 1.75 (n=4)	2.62 ± 1.32 (n=3)
SS leakage-flagged	2/4	3/3
POST I_{on}/I_{off} ratio, mean ± SD	2.86e+05 ± 4.34e+05	2.96e+05 ± 2.47e+05
I_{on}/I_{off} leakage-flagged	4/4	3/3
Fixed-bias $ I_{DS} $ recovery, POST/PRE	11.85 ± 13.62×	7.00 ± 0.71×
Fixed-bias $ I_{DS} $ recovery range	2.43–31.54×	6.57–7.82×
Fixed-bias $ I_{DS} $ recovery CV	1.15	0.10
Devices with improved R_{IG}	4/4	2/3
R_{IG} recovery factor, mean ± SD	13.68 ± 18.28×	3.55 ± 3.03×
R_{IG} recovery factor, range	2.443–40.79×	0.057–5.37×
POST R_{IG}	0.215 ± 0.046	3.019 ± 4.935
Maximum $ I_G $ POST/PRE	1.21 ± 0.56×	66.31 ± 112.51×
Handling-affected/non-interpretable POST response	0/4	1/4 measured device (D1; excluded)

Note. Condition-level continuous metrics are mean ± SD. †The 70 °C D1 device was physically damaged during handling before POST characterization. Its POST sweeps are retained as raw QC records but excluded from quantitative POST summaries. The remaining three evaluable 70 °C devices had a tighter fixed-bias $|I_{DS}|$ recovery distribution, while one remained strongly gate-current-dominated POST.

Table B.4.2. PBS I_{DS} recovery and switching metrics.

Temp	Device	Piece	POST SS (V/dec)	SS leakage-flagged	POST I_{on}/I_{off}	I_{on}/I_{off} leakage-flagged	I_{DS} recovery POST/PRE
60 °C	1	1	1.73	Yes	9.30e+05	Yes	2.43×
60 °C	2	1	1.83	No	5.93e+04	Yes	3.04×
60 °C	3	2	3.45	No	1.50e+05	Yes	31.54×
60 °C	4	2	5.46	Yes	5.18e+03	Yes	10.38×
70 °C	1	1	Not used†	—	Not used†	—	Not used†
70 °C	2	1	2.31	Yes	3.54e+05	Yes	6.57×
70 °C	3	2	1.47	Yes	5.09e+05	Yes	6.60×
70 °C	4	2	4.07	Yes	2.60e+04	Yes	7.82×

Table B.4.3. PBS leakage and quality-control metrics.

Temp	Device	Piece	R_{IG} PRE	R_{IG} POST	R_{IG} recovery PRE/POST	Max $ I_G $ POST/PRE	QC
60 °C	1	1	0.594	0.243	2.44×	0.99×	—
60 °C	2	1	0.479	0.162	2.96×	1.03×	—
60 °C	3	2	10.730	0.263	40.79×	0.77×	—
60 °C	4	2	1.630	0.191	8.52×	2.04×	—
70 °C	1	1	6.004	Not used†	Not used†	Not used†	70 °C D1 was physically damaged during handling before POST characterization. POST sweeps acquired from the damaged device were retained as raw QC records but excluded from quantitative POST summaries.
70 °C	2	1	1.388	0.266	5.22×	1.26×	Same piece; no comparable handling damage was observed.
70 °C	3	2	0.495	8.717	0.06×	196.23×	Strongly leakage-dominated POST.
70 °C	4	2	0.405	0.075	5.37×	1.46×	—

B.5 Humidity-Control and PBS-Evaporation Pilot Data

This section retains the tabulated electrical summaries supporting the humidity-control and PBS-evaporation experiment described in Section 3.7. Tables B.5.1 through B.5.3 report extracted electrical metrics and quality-control classifications. Complete baseline and 24 h device-level electrical characterization under humidified and nonhumid conditions is provided in Supplemental Data S1.

Table B.5.1. Threshold voltage, hysteresis, and fixed-bias current metrics.

Condition	Piece	Device	$V_{th,F}$ BL	$V_{th,F}$ 24 h	ΔF V_{th}	F 24 h leakage flag	$V_{th,R}$ BL	$V_{th,R}$ 24 h	ΔR V_{th}	R 24 h leakage flag	Hysteresis BL	Hysteresis 24 h	Δ hysteresis	1 μ A F 24 h	1 μ A R 24 h	$I_{DS}(+30\text{ V})$ BL (μ A)	$I_{DS}(+30\text{ V})$ 24 h (μ A)
Humidified	HUMP 1	HUMP 1D1	5.969	—	—	No	9.809	29.686	19.878	Yes	3.840	—	—	—	—	591.289	35.223
Humidified	HUMP 1	HUMP 1D2	8.883	16.517	7.634	No	11.870	23.086	11.215	No	2.988	6.569	3.581	11.434	18.109	405.442	85.066
Humidified	HUMP 1	HUMP 1D3	7.591	16.076	8.486	Yes	10.829	23.416	12.588	Yes	3.238	7.340	4.102	9.687	15.946	424.581	98.415
Humidified	HUMP 2	HUMP 2D1	4.800	10.179	5.380	No	8.254	21.407	13.153	No	3.454	11.228	7.774	4.092	14.789	597.524	211.967
Humidified	HUMP 2	HUMP 2D2	5.405	17.391	11.986	Yes	7.798	26.978	19.180	Yes	2.393	9.587	7.194	—	16.642	703.971	72.622
Humidified	HUMP 2	HUMP 2D3	5.990	21.213	15.223	No	9.643	28.456	18.813	Yes	3.653	7.243	3.590	10.874	19.019	601.844	38.315
Non-humidified	NONHUMP1	NONHUMP1 D1	6.059	8.851	2.792	No	15.511	24.026	8.515	No	9.452	15.175	5.723	-4.030	17.581	386.816	145.829
Non-humidified	NONHUMP1	NONHUMP1 D2	8.865	4.366	-4.499	No	13.294	21.288	7.994	No	4.429	16.922	12.493	-5.246	15.630	397.195	172.674
Non-humidified	NONHUMP1	NONHUMP1 D3	10.864	—	—	No	16.110	—	—	No	5.246	—	—	16.506	23.357	239.696	5.774
Non-humidified	NONHUMP2	NONHUMP2 D1	7.922	1.498	-6.424	No	13.078	21.346	8.268	No	5.156	19.848	14.693	-12.450	15.266	384.821	90.792
Non-humidified	NONHUMP2	NONHUMP2 D2	9.374	17.197	7.823	No	12.251	25.023	12.772	No	2.877	7.826	4.949	-6.150	18.006	418.636	44.490
Non-humidified	NONHUMP2	NONHUMP2 D3	11.201	2.095	-9.107	No	17.153	22.785	5.632	No	5.951	20.691	14.739	-10.809	17.497	177.521	32.305

Table B.5.2. Subthreshold swing and on/off ratio metrics.

Condit ion	Piece	Device	SS F BL	SS F 24 h	log ₁₀ SS F ratio	SS F 24 h leakag e flag	SS R BL	SS R 24 h	log ₁₀ SS R ratio	SS R 24 h leakag e flag	I_{on}/I_{off} F BL	I_{on}/I_{off} F 24 h	log ₁₀ F ratio	F 24 h leakag e flag	I_{on}/I_{off} R BL	I_{on}/I_{off} R 24 h	log ₁₀ R ratio
Humidi fied	HUMP 1	HUMP 1D1	1.016	1.634	0.206	Yes	1.149	1.967	0.234	Yes	2.72e+ 07	1.57e+ 06	-1.239	Yes	9.52e+ 06	37079. 803	-2.409
Humidi fied	HUMP 1	HUMP 1D2	1.136	1.285	0.054	Yes	1.176	1.604	0.135	Yes	1.05e+ 06	5.93e+ 05	-0.249	Yes	1.36e+ 06	1.62e+ 06	0.074
Humidi fied	HUMP 1	HUMP 1D3	1.457	1.554	0.028	Yes	1.040	1.850	0.250	Yes	1.81e+ 07	1.40e+ 06	-1.113	Yes	3.42e+ 06	2.67e+ 05	-1.107
Humidi fied	HUMP 2	HUMP 2D1	0.453	1.286	0.453	Yes	1.416	3.468	0.389	No	4.73e+ 06	1.76e+ 06	-0.429	Yes	2.72e+ 06	8.40e+ 05	-0.510
Humidi fied	HUMP 2	HUMP 2D2	0.586	2.578	0.644	Yes	1.358	3.088	0.357	Yes	2.71e+ 06	27441. 316	-1.995	Yes	1.01e+ 06	44271. 696	-1.358
Humidi fied	HUMP 2	HUMP 2D3	0.715	3.269	0.660	Yes	0.838	2.383	0.454	Yes	4.60e+ 06	2.20e+ 05	-1.320	Yes	3.82e+ 06	1.81e+ 05	-1.323
Non- humidi fied	NONH UMP1	NONH UMP1 D1	0.538	2.037	0.578	Yes	1.929	2.601	0.130	Yes	1.95e+ 06	2.87e+ 05	-0.833	Yes	1.76e+ 06	3.06e+ 05	-0.761
Non- humidi fied	NONH UMP1	NONH UMP1 D2	0.787	2.019	0.409	Yes	1.339	1.362	0.008	Yes	2.55e+ 06	6.33e+ 05	-0.605	Yes	2.13e+ 06	6.04e+ 05	-0.546
Non- humidi fied	NONH UMP1	NONH UMP1 D3	4.159	2.078	-0.301	Yes	2.147	3.637	0.229	Yes	61762. 002	2.37e+ 05	0.584	Yes	3.35e+ 05	24054. 607	-1.143
Non- humidi fied	NONH UMP2	NONH UMP2 D1	0.479	2.187	0.660	Yes	1.248	3.737	0.476	No	2.61e+ 06	1.00e+ 05	-1.416	Yes	1.71e+ 06	2.00e+ 05	-0.933
Non- humidi fied	NONH UMP2	NONH UMP2 D2	0.629	3.144	0.699	Yes	1.000	3.530	0.548	Yes	2.38e+ 06	71691. 177	-1.522	Yes	1.92e+ 06	1.44e+ 05	-1.126
Non- humidi fied	NONH UMP2	NONH UMP2 D3	1.651	2.880	0.242	Yes	0.942	3.243	0.537	No	1.39e+ 06	35772. 285	-1.590	Yes	1.14e+ 06	1.55e+ 05	-0.867

Table B.5.3. Gate leakage and output quality-control metrics.

Condition	Piece	Device	$ I_G / I_{DS} $ +30 V BL	$ I_G / I_{DS} $ +30 V 24 h	max $ I_G $ BL (A)	max $ I_G $ 24 h (A)	Output class at 24 h	Forward 10 μ A status	Reverse 10 μ A status	QC note
Humidified	HUMP1	HUMP1D1	0.096	0.823	3.85e-04	2.97e-05	Abnormal / unstable	3 crossings	unique	Run1686 retained as repeat; Run1684 is primary forward run.
Humidified	HUMP1	HUMP1D2	0.067	0.257	2.71e-05	2.19e-05	Saturation-like	unique	unique	—
Humidified	HUMP1	HUMP1D3	1.116	0.628	4.88e-04	0.002	Saturation-like	unique	unique	—
Humidified	HUMP2	HUMP2D1	0.069	0.168	4.12e-05	3.56e-05	Saturation-like	unique	unique	—
Humidified	HUMP2	HUMP2D2	0.210	1.480	2.24e-04	1.07e-04	Gate- dependent; largely unsaturated/abn ormal	unique	unique	—
Humidified	HUMP2	HUMP2D3	0.073	0.737	4.39e-05	2.83e-05	Gate- dependent; largely unsaturated/abn ormal	unique	unique	—
Non- humidified	NONHUM P1	NONHUMP 1D1	0.115	0.162	4.46e-05	2.36e-05	Saturation-like	unique	unique	—
Non- humidified	NONHUM P1	NONHUMP 1D2	0.067	0.081	2.67e-05	1.40e-05	Saturation-like	unique	unique	—
Non- humidified	NONHUM P1	NONHUMP 1D3	0.046	1.448	1.10e-05	8.36e-06	Saturation-like	no crossing	no crossing	—
Non- humidified	NONHUM P2	NONHUMP 2D1	0.087	0.063	3.34e-05	5.68e-06	Saturation-like	unique	unique	—
Non- humidified	NONHUM P2	NONHUMP 2D2	0.049	0.266	2.05e-05	1.18e-05	Nonmonotonic / abnormal	unique	unique	—
Non- humidified	NONHUM P2	NONHUMP 2D3	0.019	0.008	3.42e-06	2.62e-07	Gate- dependent; no normal saturation-like family	unique	unique	—

APPENDIX C

PRE/POST Surface-Processing Electrical Characterization

This appendix retains the derived PRE/POST processing responses, wafer-piece and parent-wafer summaries, quality-control records, between-sequence analyses, model verification, diagnostics, and processing-load sensitivity analyses supporting Chapter 4. Complete raw electrical-metric tables and complete device-level PRE/POST electrical characteristics are provided in Supplemental Data S2.

C.1 DDT Processing

Air and PBS identifiers denote the later environmental-assignment groups; these PRE/POST measurements characterize the DDT processing step itself. Derived response, wafer-piece, parent-wafer, quality-control, and square-root-threshold summaries are retained here; complete raw metrics and complete device-level PRE/POST characteristics are provided in Supplemental Data S2.

Table C.1.1. DDT FET-level paired responses.

Device	$\Delta V_{th,F}$ (V)	$\Delta V_{th,R}$ (V)	Δ hysteresis (V)	$ I_{DS} $ POST/PRE	SS POST/PRE	I_{on}/I_{off} POST/PRE	max $ I_G $ POST/PRE
air1	+5.91	+2.09	-3.83	0.60×	1.06×	0.54×	0.78×
air2	-2.44	-1.73	+0.71	1.66×	1.48×	1.22×	1.70×
air3	+0.99	+1.09	+0.10	0.86×	1.04×	0.07×	0.91×
air4	+1.62	+2.11	+0.50	0.82×	0.59×	0.82×	0.84×
air5	-0.63	-1.04	-0.41	0.81×	0.27×	6.10×	0.66×
air6	+3.22	-0.37	-3.59	0.91×	1.34×	0.52×	0.92×
pbs1	+0.28	-4.03	-4.31	1.40×	4.11×	0.02×	1.67×
pbs2	+0.16	-0.78	-0.94	1.06×	7.86×	$4.69 \times 10^{-4} \times$	1.18×
pbs3	-1.61	-4.64	-3.02	1.42×	1.25×	0.71×	1.20×
pbs4	-1.02	+5.19	+6.20	1.08×	1.11×	0.86×	1.18×
pbs5	-1.75	-3.92	-2.17	1.45×	2.48×	1.07×	1.30×
pbs6	-2.03	-2.40	-0.37	1.21×	0.39×	3.56×	1.46×

Table C.1.2. DDT wafer-piece responses.

Wafer	Piece	FETs	Mean $\Delta V_{th,F}$	Mean $\Delta V_{th,R}$	Mean Δ hyst.	$ I_{DS} $ ratio	SS ratio	I_{on}/I_{off} ratio	max $ I_G $ ratio
W1	W1-Air	air1, air2	+1.73	+0.18	-1.56	1.00×	1.25×	0.81×	1.15×
W1	W1-PBS	pbs1, pbs2	+0.22	-2.41	-2.63	1.22×	5.68×	$2.83 \times 10^{-3} \times$	1.41×
W2	W2-Air	air3, air4	+1.30	+1.60	+0.30	0.84×	0.78×	0.24×	0.88×
W2	W2-PBS	pbs3, pbs4	-1.31	+0.28	+1.59	1.24×	1.18×	0.79×	1.19×
W3	W3-Air	air5, air6	+1.30	-0.70	-2.00	0.86×	0.60×	1.79×	0.78×
W3	W3-PBS	pbs5, pbs6	-1.89	-3.16	-1.27	1.32×	0.98×	1.95×	1.38×

Table C.1.3. DDT parent-wafer responses.

Wafer	FETs	Mean $\Delta V_{th,F}$	Mean $\Delta V_{th,R}$	Mean Δ hyst.	$ I_{DS} $ ratio	SS ratio	I_{on}/I_{off} ratio	max $ I_G $ ratio
W1	air1, air2, pbs1, pbs2	+0.98	-1.12	-2.09	1.10×	2.67×	0.05×	1.27×
W2	air3, air4, pbs3, pbs4	-0.01	+0.94	+0.94	1.02×	0.96×	0.43×	1.02×
W3	air5, air6, pbs5, pbs6	-0.30	-1.93	-1.63	1.06×	0.77×	1.87×	1.04×

Table C.1.4. DDT operational and leakage QC.

Device	Stage	F V_{th} valid	R V_{th} valid	$I_{DS} +30$ valid	SS leak flag	I_{on}/I_{off} leak flag	$R_{IG} +30$	Gate output	Sat.-like output	sqrt V_{th}
air1	PRE	Yes	Yes	Yes	Yes	Yes	0.040	Yes	Yes	Reportable
air1	POST	Yes	Yes	Yes	Yes	Yes	0.053	Yes	Yes	Reportable
air2	PRE	Yes	Yes	Yes	Yes	Yes	0.049	Yes	Yes	Reportable
air2	POST	Yes	Yes	Yes	Yes	Yes	0.050	Yes	Yes	Reportable
air3	PRE	Yes	Yes	Yes	No	Yes	0.063	Yes	Yes	Reportable
air3	POST	Yes	Yes	Yes	No	Yes	0.067	Yes	Yes	Reportable
air4	PRE	Yes	Yes	Yes	Yes	Yes	0.044	Yes	Yes	Reportable
air4	POST	Yes	Yes	Yes	Yes	Yes	0.045	Yes	Yes	Not reportable
air5	PRE	Yes	Yes	Yes	Yes	Yes	0.062	Yes	Yes	Reportable
air5	POST	Yes	Yes	Yes	Yes	Yes	0.051	Yes	Yes	Reportable
air6	PRE	Yes	Yes	Yes	Yes	Yes	0.049	Yes	Yes	Reportable
air6	POST	Yes	Yes	Yes	No	Yes	0.050	Yes	Yes	Reportable
pbs1	PRE	Yes	Yes	Yes	Yes	Yes	0.048	Yes	Yes	Reportable
pbs1	POST	Yes	Yes	Yes	No	Yes	0.057	Yes	Yes	Reportable
pbs2	PRE	Yes	Yes	Yes	Yes	Yes	0.039	Yes	Yes	Reportable
pbs2	POST	Yes	Yes	Yes	No	No	0.044	Yes	Yes	Reportable
pbs3	PRE	Yes	Yes	Yes	Yes	Yes	0.046	Yes	Yes	Reportable
pbs3	POST	Yes	Yes	Yes	Yes	Yes	0.039	Yes	Yes	Reportable
pbs4	PRE	Yes	Yes	Yes	Yes	Yes	0.061	Yes	Yes	Reportable
pbs4	POST	Yes	Yes	Yes	Yes	Yes	0.067	Yes	Yes	Reportable
pbs5	PRE	Yes	Yes	Yes	Yes	Yes	0.077	Yes	Yes	Reportable
pbs5	POST	Yes	Yes	Yes	No	Yes	0.069	Yes	Yes	Reportable
pbs6	PRE	Yes	Yes	Yes	No	Yes	0.035	Yes	Yes	Not reportable
pbs6	POST	Yes	Yes	Yes	Yes	Yes	0.042	Yes	Yes	Reportable

Table C.1.5. DDT square-root threshold-voltage summary.

Square-root V_{th} result	n	Mean $\pm$ SD
PRE reportable	11/12	3.05 ± 2.31 V
POST reportable	11/12	3.88 ± 2.97 V
Paired PRE $\rightarrow$ POST	10/12	$+0.50 \pm 2.35$ V

C.2 PTMS + DDT Processing

This section contains derived PRE/POST processing-response analyses for the PTMS + DDT processing sequence. Air and PBS identifiers denote the later environmental-assignment groups; environmental exposure had not begun. Complete raw metrics and complete device-level PRE/POST characteristics are provided in Supplemental Data S2.

Table C.2.1. PTMS + DDT FET-level paired responses.

Device	$\Delta V_{th,F}$ (V)	$\Delta V_{th,R}$ (V)	Δ hysteresis (V)	$ I_{DS} $ POST/PRE	SS POST/PRE	I_{on}/I_{off} POST/PRE	max $ I_G $ POST/PRE
air1	-3.89	-6.53	-2.64	2.03×	0.72×	2.19×	2.14×
air2	-5.61	-6.66	-1.06	1.97×	1.56×	0.04×	699.67×
air3	-1.94	-6.33	-4.39	1.51×	0.61×	3.80×	1.41×
air4	-1.67	-5.75	-4.08	1.93×	1.25×	1.50×	3.12×
air5	+0.34	-5.19	-5.53	1.60×	1.00×	1.92×	1.68×
air6	-6.37	-7.58	-1.22	2.12×	0.89×	3.49×	3.63×
pbs1	-1.80	-6.52	-4.73	2.11×	1.05×	0.41×	4.75×
pbs2	-1.42	-4.45	-3.03	1.46×	0.26×	8.98×	1.39×
pbs3	-1.18	-2.54	-1.36	1.13×	0.74×	4.13×	1.28×
pbs4	-1.87	-2.51	-0.64	1.24×	0.48×	46.85×	1.41×
pbs5	-0.62	+0.52	+1.15	0.99×	0.72×	8.05×	1.07×
pbs6	+0.15	+1.06	+0.91	0.90×	0.41×	2.14×	0.94×

Table C.2.2. PTMS + DDT wafer-piece responses.

Wafer	Piece	Future arm	FETs	Mean $\Delta V_{th,F}$	Mean $\Delta V_{th,R}$	Mean Δ hysteresis	$ I_{DS} $ ratio	SS ratio	I_{on}/I_{off} ratio	max $ I_G $ ratio
W1	W1-Air	Air	air1, air2	-4.75	-6.60	-1.85	2.00×	1.06×	0.29×	38.74×
W1	W1-PBS	PBS	pbs1, pbs2	-1.61	-5.48	-3.88	1.76×	0.52×	1.92×	2.57×
W2	W2-Air	Air	air3, air4	-1.80	-6.04	-4.24	1.71×	0.88×	2.39×	2.09×
W2	W2-PBS	PBS	pbs3, pbs4	-1.52	-2.52	-1.00	1.18×	0.60×	13.91×	1.34×
W3	W3-Air	Air	air5, air6	-3.01	-6.39	-3.37	1.84×	0.94×	2.59×	2.47×
W3	W3-PBS	PBS	pbs5, pbs6	-0.24	+0.79	+1.03	0.95×	0.55×	4.15×	1.00×

Table C.2.3. PTMS + DDT parent-wafer responses.

Wafer	FETs	Mean $\Delta V_{th,F}$	Mean $\Delta V_{th,R}$	Mean Δ hyst.	$ I_{DS} $ ratio	SS ratio	I_{on}/I_{off} ratio	max $ I_G $ ratio
W1	air1, air2, pbs1, pbs2	-3.18	-6.04	-2.86	1.87×	0.74×	0.75×	9.97×
W2	air3, air4, pbs3, pbs4	-1.66	-4.28	-2.62	1.42×	0.72×	5.77×	1.68×
W3	air5, air6, pbs5, pbs6	-1.62	-2.80	-1.17	1.32×	0.72×	3.28×	1.57×

Table C.2.4. PTMS + DDT square-root threshold-voltage summary.

Square-root V_{th} result	n	Mean $\pm$ SD
PRE reportable	12/12	4.40 $\pm$ 2.64 V
POST reportable	12/12	2.73 $\pm$ 1.65 V
Paired PRE→POST	12/12	-1.67 $\pm$ 2.21 V

C.3 UV + PTMS + DDT Processing

Air and PBS identifiers denote the later environmental-assignment groups; these PRE/POST measurements characterize the UV + PTMS + DDT processing sequence before environmental exposure. Derived response, wafer-piece, parent-wafer, quality-control, and square-root-threshold summaries are retained here; complete raw metrics and complete device-level PRE/POST characteristics are provided in Supplemental Data S2.

Table C.3.1. UV + PTMS + DDT FET-level paired responses.

Device	$\Delta V_{th,F}$ (V)	$\Delta V_{th,R}$ (V)	Δ hysteresis (V)	$ I_{DS} $ POST/PRE	SS POST/PRE	I_{on}/I_{off} POST/PRE	max $ I_G $ POST/PRE
air1	+0.18	+1.33	+1.15	0.55×	3.53×	0.62×	0.69×
air2	-0.98	-0.31	+0.67	0.49×	3.59×	0.97×	1.01×
air3	-0.85	-3.59	-2.74	1.69×	2.73×	1.61×	1.83×
air4	-2.62	-3.90	-1.27	1.21×	0.58×	4.08×	1.00×
air5	+3.39	+3.68	+0.28	0.81 ×	2.32×	0.18×	0.74×
air6	+2.61	+0.79	-1.82	1.78×	0.75×	2.69×	0.62×
pbs1	+5.49	+8.69	+3.20	0.22×	3.93×	0.29×	0.71×
pbs2	+4.01	+7.12	+3.11	0.33×	2.17×	0.22×	0.62×
pbs3	+2.32	+7.96	+5.64	0.33×	1.12×	4.00×	0.30×
pbs4	+7.18	+12.75	+5.57	0.15×	0.68×	3.76×	0.19×
pbs5	-1.77	-3.51	-1.74	0.88×	1.51×	7.33×	1.37×
pbs6	-5.43	-3.11	+2.32	0.86×	5.11×	0.10×	1.53×

Table C.3.2. UV + PTMS + DDT wafer-piece responses.

Wafer	Piece	Future arm	FETs	Mean $\Delta V_{th,F}$	Mean $\Delta V_{th,R}$	Mean Δ hysteresis	$ I_{DS} $ ratio	SS ratio	I_{on}/I_{off} ratio	max $ I_G $ ratio
W1	W1-Air	Air	air1, air2	-0.40	+0.51	+0.91	0.52×	3.56×	0.78×	0.83×
W1	W1-PBS	PBS	pbs1, pbs2	+4.75	+7.91	+3.16	0.27×	2.92×	0.25×	0.66×
W2	W2-Air	Air	air3, air4	-1.73	-3.74	-2.01	1.43×	1.26×	2.56×	1.35×
W2	W2-PBS	PBS	pbs3, pbs4	+4.75	+10.36	+5.60	0.23×	0.87×	3.88×	0.24×
W3	W3-Air	Air	air5, air6	+3.00	+2.23	-0.77	1.20×	1.32×	0.69×	0.68×
W3	W3-PBS	PBS	pbs5, pbs6	-3.60	-3.31	+0.29	0.87×	2.78×	0.85×	1.45×

Table C.3.3. UV + PTMS + DDT parent-wafer responses.

Wafer	FETs	Mean $\Delta V_{th,F}$	Mean $\Delta V_{th,R}$	Mean Δ hyst.	$ I_{DS} $ ratio	SS ratio	I_{on}/I_{off} ratio	max $ I_G $ ratio
W1	air1, air2, pbs1, pbs2	+2.18	+4.21	+2.03	0.38×	3.22×	0.44×	0.74×
W2	air3, air4, pbs3, pbs4	+1.51	+3.31	+1.80	0.57×	1.04×	3.15×	0.57×
W3	air5, air6, pbs5, pbs6	-0.30	-0.54	-0.24	1.02×	1.91×	0.77×	0.99×

Table C.3.4. UV + PTMS + DDT operational and leakage QC.

Device	Stage	$V_{th,F}$ valid	$V_{th,R}$ valid	Fixed- I_{DS} valid	SS available	SS leak flag	I_{on}/I_{off} avail.	I_{on}/I_{off} leak flag	$R_{IG}@30$	Gate-dependent output	Sat.-like output	sqrt V_{th}	QC note
air1	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.047	Yes	Yes	Yes	—
air1	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.058	Yes	Yes	Yes	—
air2	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.038	Yes	Yes	Yes	—
air2	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.079	Yes	Yes	Yes	—
air3	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.051	Yes	Yes	Yes	—
air3	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.055	Yes	Yes	Yes	—
air4	PRE	Yes	Yes	Yes	Yes	No	Yes	Yes	0.043	Yes	Yes	Yes	—
air4	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.036	Yes	Yes	Yes	—
air5	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.071	Yes	Yes	Yes	—
air5	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.065	Yes	Yes	Yes	—
air6	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.119	Yes	Yes	Yes	—
air6	POST	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.041	Yes	Yes	Yes	—
pbs1	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.047	Yes	Yes	Yes	—
pbs1	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.148	Yes	Yes	Yes	—
pbs2	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.050	Yes	Yes	Yes	—
pbs2	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.094	Yes	Yes	Yes	—
pbs3	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.082	Yes	Yes	Yes	—
pbs3	POST	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.075	Yes	Yes	Yes	—
pbs4	PRE	Yes	Yes	Yes	Yes	No	Yes	Yes	0.064	Yes	Yes	Yes	—
pbs4	POST	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.078	Yes	Yes	Yes	—
pbs5	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.038	Yes	Yes	Yes	—
pbs5	POST	Yes	Yes	Yes	Yes	No	Yes	Yes	0.060	Yes	Yes	Yes	—
pbs6	PRE	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.027	Yes	Yes	Yes	—
pbs6	POST	Yes	Yes	Yes	Yes	Yes	Yes	Yes	0.048	Yes	Yes	Yes	—

Table C.3.5. UV + PTMS + DDT square-root threshold-voltage summary.

Square-root V_{th} result	n	Mean $\pm$ SD
PRE reportable	12/12	2.13 ± 3.06 V
POST reportable	12/12	1.99 ± 2.06 V
Paired PRE $\rightarrow$ POST	12/12	-0.14 ± 3.95 V

C.4 Between-Sequence Analysis

Table C.4.1. Pairwise processing-sequence comparisons.

Outcome	PTMS + DDT vs DDT	UV + PTMS + DDT vs PTMS + DDT	UV + PTMS + DDT vs DDT
Forward V_{th}	-2.38 V [-5.55, +0.79]; Holm-3 $p=0.258$	+3.28 V [+0.11, +6.45]; Holm-3 $p=0.130$	+0.90 V [-2.27, +4.07]; Holm-3 $p=0.548$
Reverse V_{th}	-3.67 V [-8.77, +1.43]; Holm-3 $p=0.288$	+6.70 V [+1.60, +11.80]; Holm-3 $p=0.042$	+3.03 V [-2.07, +8.13]; Holm-3 $p=0.288$
Signed hysteresis	-1.29 V [-4.12, +1.53]; Holm-3 $p=0.342$	+3.42 V [+0.59, +6.24]; Holm-3 $p=0.065$	+2.12 V [-0.70, +4.95]; Holm-3 $p=0.257$
Fixed-bias ID	$1.43 \times [0.75, 2.73]$; Holm-3 $p=0.249$	$0.40 \times [0.21, 0.75]$; Holm-3 $p=0.025$	$0.57 \times [0.30, 1.08]$; Holm-3 $p=0.159$
SS	$0.58 \times [0.31, 1.10]$; Holm-3 $p=0.179$	$2.56 \times [1.35, 4.85]$; Holm-3 $p=0.022$	$1.49 \times [0.78, 2.82]$; Holm-3 $p=0.204$

Table C.4.2. Operational compatibility and leakage quality control.

Operational outcome	DDT PRE	DDT POST	PTMS + DDT PRE	PTMS + DDT POST	UV + PTMS + DDT PRE	UV + PTMS + DDT POST
Forward 10- μ A V_{th} valid	12/12	12/12	12/12	12/12	12/12	12/12
Reverse 10- μ A V_{th} valid	12/12	12/12	12/12	12/12	12/12	12/12
Fixed-bias ID valid	12/12	12/12	12/12	12/12	12/12	12/12
Gate-dependent output	12/12	12/12	12/12	12/12	12/12	12/12
Recognizable saturation-like output	12/12	12/12	12/12	12/12	12/12	12/12
$R_{IG} > 0.10$ at +30 V	0/12	0/12	0/12	0/12	1/12	1/12
$R_{IG} \geq 1$ at +30 V	0/12	0/12	0/12	0/12	0/12	0/12
Numerical SS available	12/12	12/12	12/12	12/12	12/12	12/12
SS leakage-flagged	10/12	7/12	9/12	12/12	10/12	4/12
Numerical I_{on}/I_{off} available	12/12	12/12	12/12	12/12	12/12	12/12
I_{on}/I_{off} leakage-flagged	12/12	11/12	11/12	12/12	12/12	12/12
Square-root V_{th} reportable	11/12	11/12	12/12	12/12	12/12	12/12

Table C.4.3. Kenward-Roger mixed-model verification.

Metric	KR denominator df	KR F scaling	Treatment KR F	Treatment KR p	max KR cov adj - REML cov	Pairwise verification
Forward V_{th}	13.0	1.000	2.6710	0.1067	<1e-14	3 contrasts + Holm-3 verified
Reverse V_{th}	13.0	1.000	4.0359	0.0433	<1e-14	3 contrasts + Holm-3 verified
Signed hysteresis	13.0	1.000	3.4767	0.0617	<1e-14	3 contrasts + Holm-3 verified
Fixed-bias ID	13.0	1.000	4.9203	0.0256	<1e-14	3 contrasts + Holm-3 verified
SS	13.0	1.000	5.0696	0.0236	<1e-14	3 contrasts + Holm-3 verified
I_{on}/I_{off}	13.0	1.000	2.7893	0.0982	<1e-14	3 contrasts + Holm-3 verified
max $ I_G $	13.0	1.000	4.3376	0.0360	<1e-14	3 contrasts + Holm-3 verified

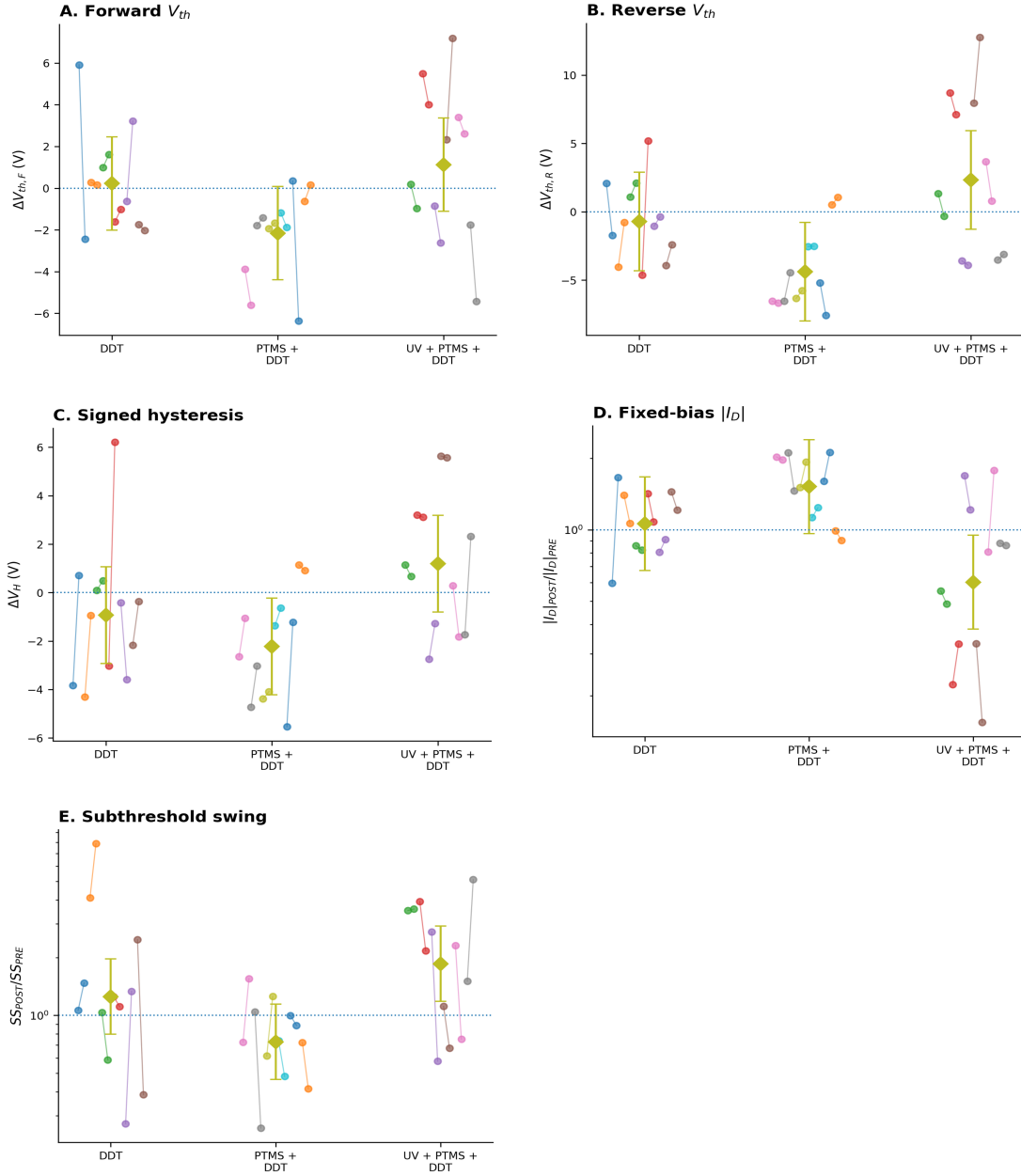


Figure C.4.1. Device-level core processing responses. (A) Change in forward threshold voltage, $\Delta V_{th,F}$. (B) Change in reverse threshold voltage, $\Delta V_{th,R}$. (C) Change in signed hysteresis. (D) POST/PRE ratio of fixed-bias $|I_{DS}|$. (E) POST/PRE ratio of subthreshold swing (SS). Colored connected pairs show the two FET subsamples measured on each independently processed wafer piece; diamonds show model-adjusted treatment responses with 95% confidence intervals. Horizontal dotted lines indicate no change (0 for voltage-difference metrics and 1 for ratio metrics). Colors distinguish independently processed wafer pieces within each processing sequence; the paired points joined by each colored line are the two FET subsamples from that piece. Color is used only as a within-sequence identifier and does not represent an additional experimental factor.

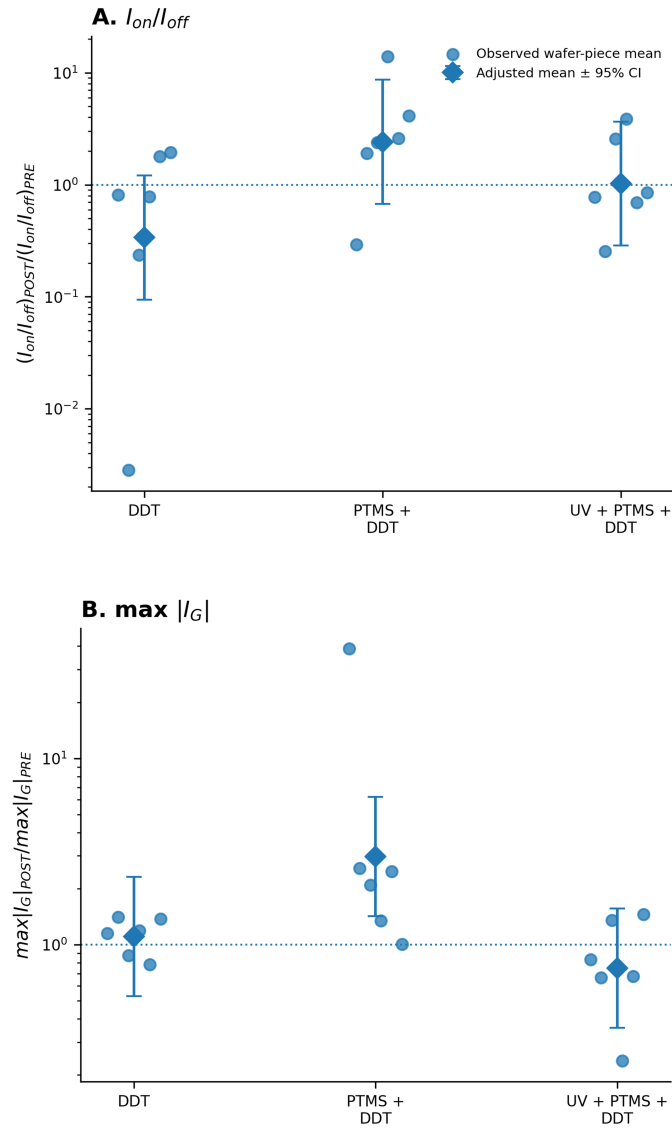


Figure C.4.2. Supporting processing-response metrics. (A) POST/PRE ratio of I_{on}/I_{off} . (B) POST/PRE ratio of maximum $|I_G|$. Points show observed wafer-piece means, diamonds show adjusted means, and error bars show 95% confidence intervals. The horizontal dotted line at 1 indicates no change from PRE to POST. These metrics are retained as supporting outcomes because of the QC limitations described in the accompanying text.

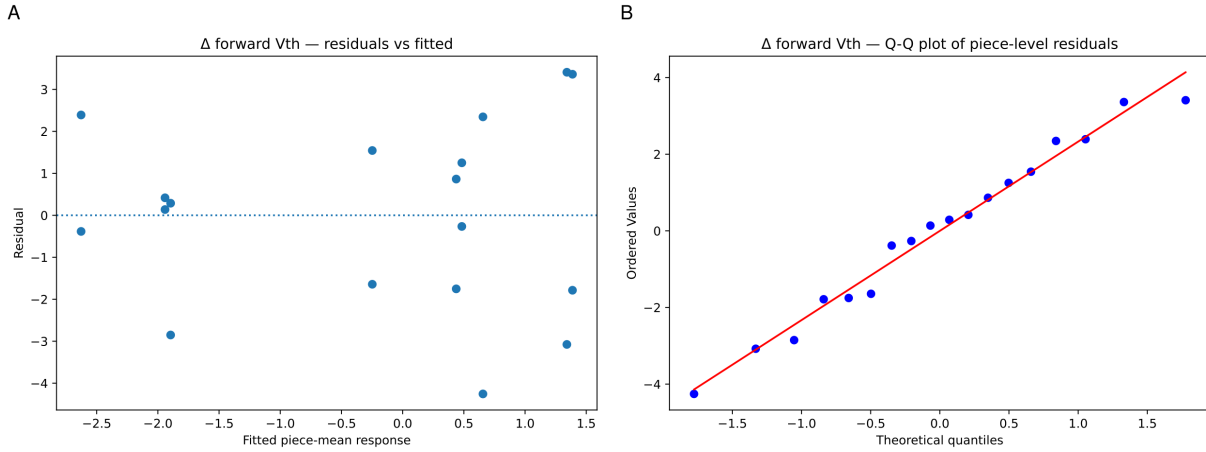


Figure C.4.3. Forward V_{th} model diagnostics. (A) Residuals from the piece-level forward-threshold model plotted against fitted piece-mean responses. (B) Q-Q plot of piece-level residuals used to assess approximate residual normality. In panel A, blue points are piece-level residuals and the horizontal dotted line at zero marks no residual deviation. In panel B, blue points are ordered residuals and the red diagonal line is the theoretical normal-reference line; closer agreement with that line indicates closer agreement with the normality assumption.

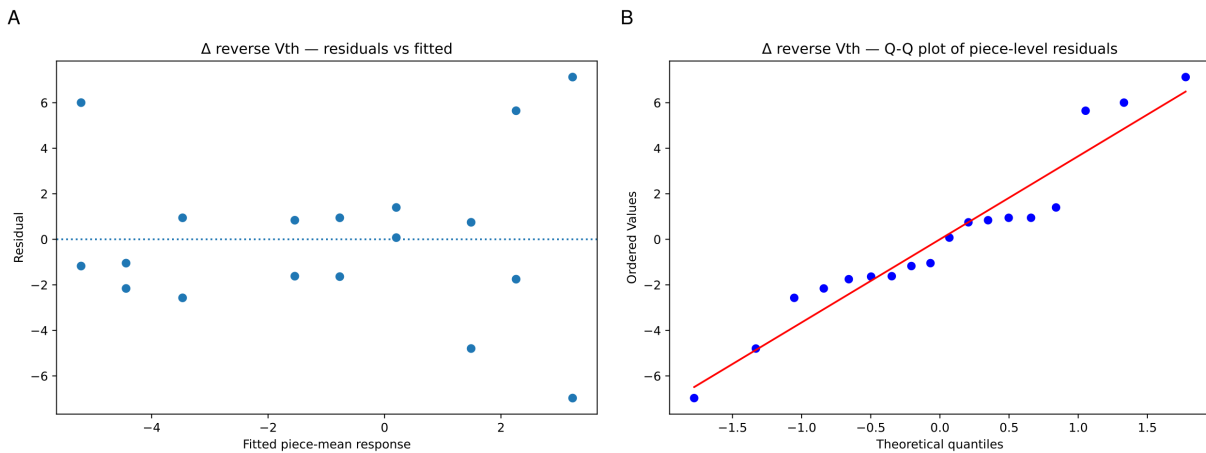


Figure C.4.4. Reverse V_{th} model diagnostics. (A) Residuals from the piece-level reverse-threshold model plotted against fitted piece-mean responses. (B) Q-Q plot of piece-level residuals used to assess approximate residual normality. Point and reference-line conventions are the same as Figure C.4.3: panel A shows blue residuals relative to the zero-residual dotted line, and panel B compares ordered residuals with the red theoretical normal-reference line.

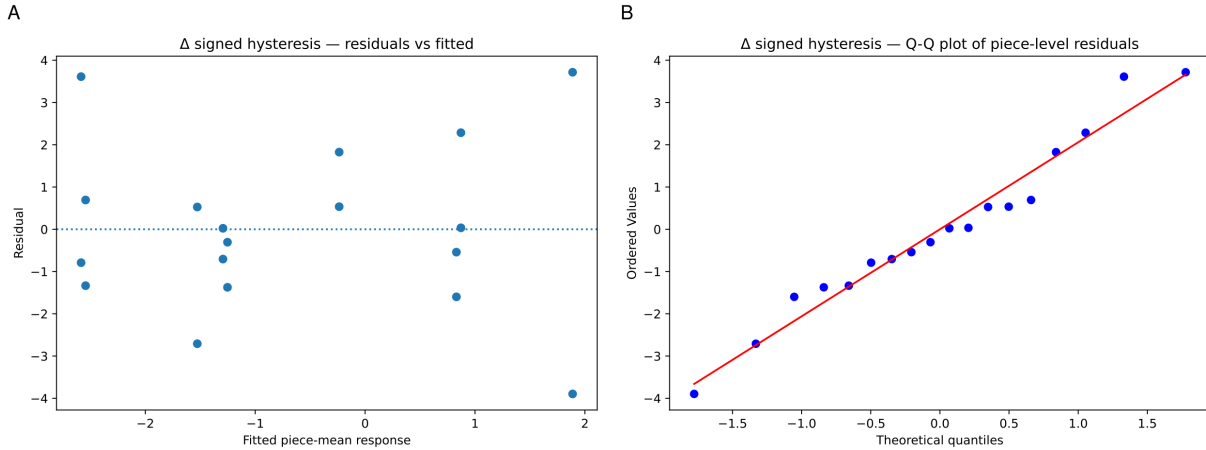


Figure C.4.5. Signed-hysteresis model diagnostics. (A) Residuals from the piece-level signed-hysteresis model plotted against fitted piece-mean responses. (B) Q–Q plot of piece-level residuals used to assess approximate residual normality. Point and reference-line conventions are the same as Figure C.4.3: panel A shows blue residuals relative to the zero-residual dotted line, and panel B compares ordered residuals with the red theoretical normal-reference line.

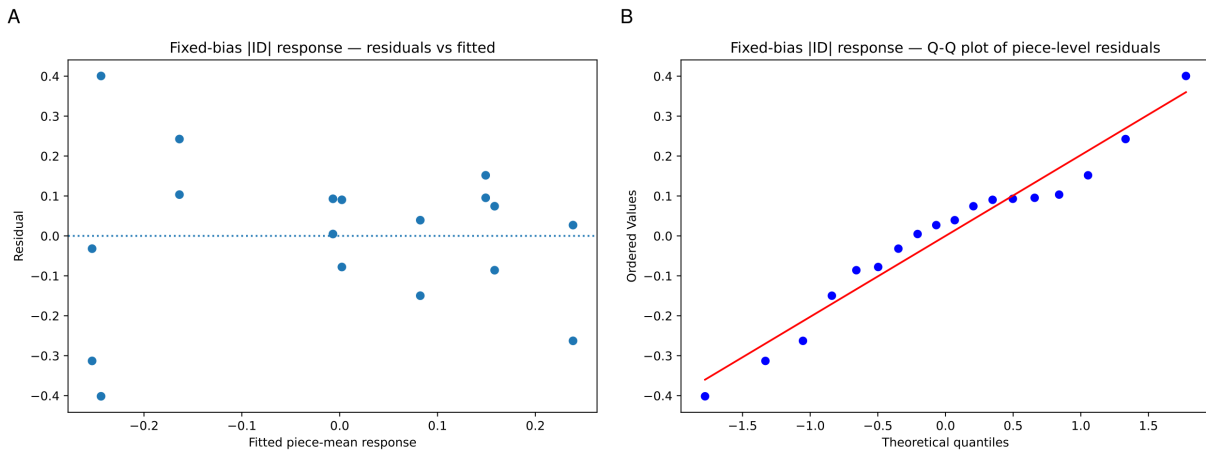


Figure C.4.6. Fixed-bias $|ID|$ response model diagnostics. (A) Residuals from the piece-level fixed-bias $|ID|$ response model plotted against fitted piece-mean responses. (B) Q–Q plot of piece-level residuals used to assess approximate residual normality. Point and reference-line conventions are the same as Figure C.4.3: panel A shows blue residuals relative to the zero-residual dotted line, and panel B compares ordered residuals with the red theoretical normal-reference line.

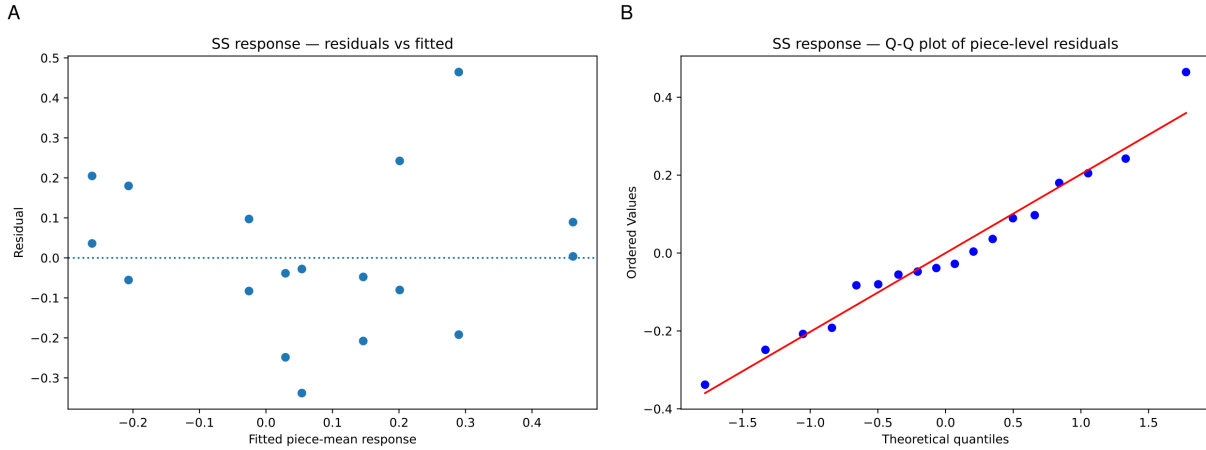


Figure C.4.7. Subthreshold-swing model diagnostics. (A) Residuals from the piece-level subthreshold-swing response model plotted against fitted piece-mean responses. (B) Q–Q plot of piece-level residuals used to assess approximate residual normality. Point and reference-line conventions are the same as Figure C.4.3: panel A shows blue residuals relative to the zero-residual dotted line, and panel B compares ordered residuals with the red theoretical normal-reference line.

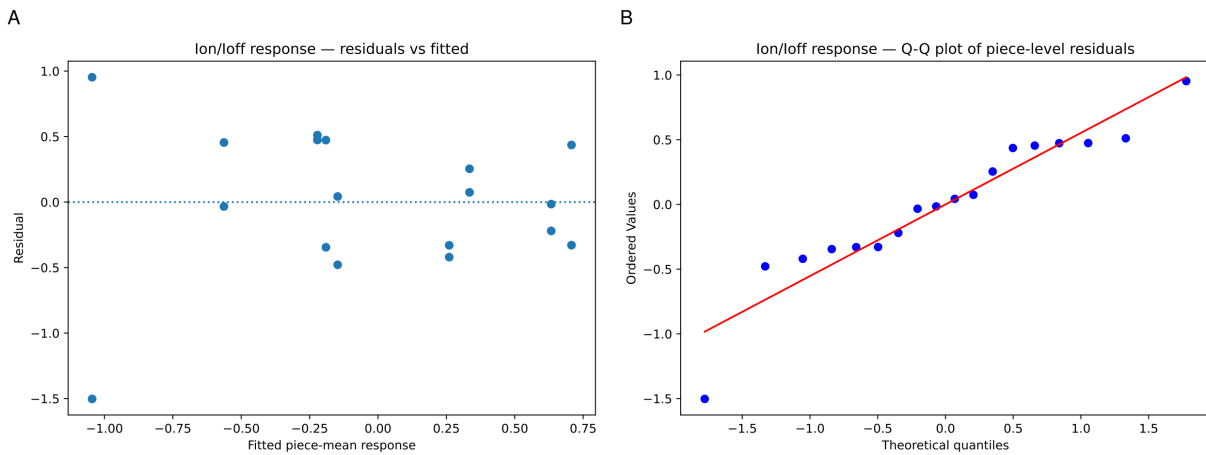


Figure C.4.8. I_{on}/I_{off} model diagnostics. (A) Residuals from the piece-level I_{on}/I_{off} response model plotted against fitted piece-mean responses. (B) Q–Q plot of piece-level residuals used to assess approximate residual normality. Point and reference-line conventions are the same as Figure C.4.3: panel A shows blue residuals relative to the zero-residual dotted line, and panel B compares ordered residuals with the red theoretical normal-reference line.

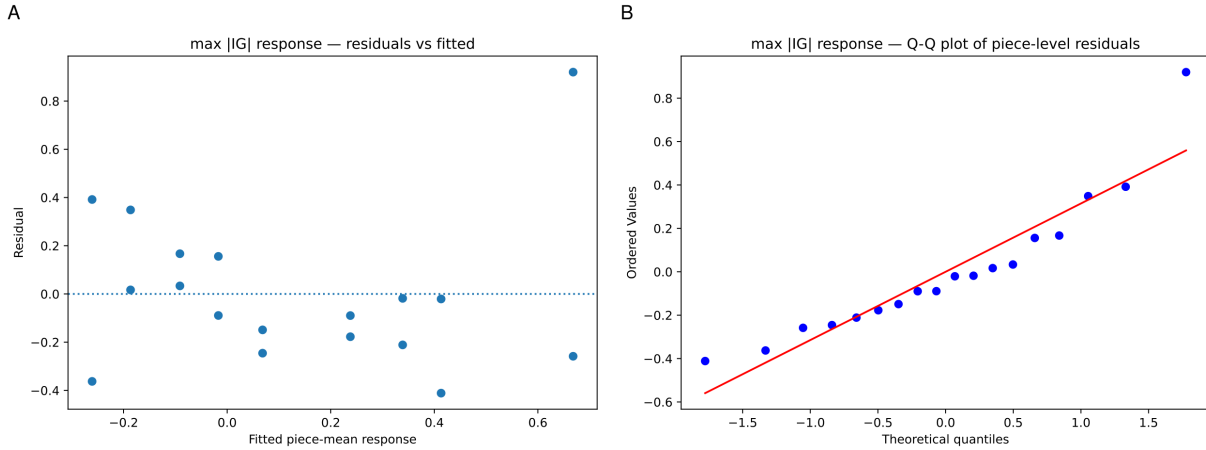


Figure C.4.9. Maximum $|I_G|$ model diagnostics. (A) Residuals from the piece-level maximum $|I_G|$ response model plotted against fitted piece-mean responses. (B) Q–Q plot of piece-level residuals used to assess approximate residual normality. Point and reference-line conventions are the same as Figure C.4.3: panel A shows blue residuals relative to the zero-residual dotted line, and panel B compares ordered residuals with the red theoretical normal-reference line.

C.5 Processing-Load Sensitivity

Table C.5.1. Primary processing-load sensitivity analysis.

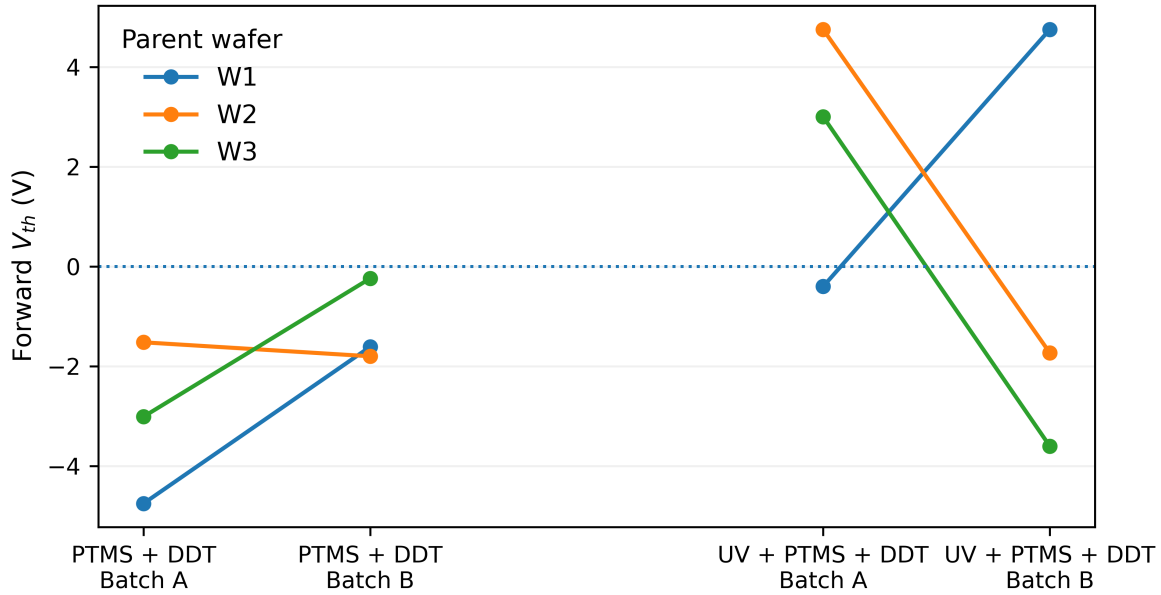
Metric	PTMS A	PTMS B	PTMS A-B effect [95% CI]	p	UV A	UV B	UV A-B effect [95% CI]	p
Delta forward V_{th}	-3.09 V	-1.22 V	-1.88 V [-6.54, +2.79]	0.225	+2.45 V	-0.20 V	+2.65 V [-14.12, +19.41]	0.567
Delta reverse V_{th}	-5.17 V	-3.58 V	-1.59 V [-14.91, +11.73]	0.658	+4.37 V	+0.28 V	+4.08 V [-22.80, +30.97]	0.581
Delta signed hysteresis	-2.07 V	-2.36 V	+0.29 V [-9.91, +10.49]	0.915	+1.91 V	+0.48 V	+1.43 V [-11.94, +14.81]	0.690
$ I_{DS} $ POST/PRE	1.63×	1.42×	1.15× [0.32, 4.18]	0.680	0.52×	0.70×	0.75× [0.03, 21.64]	0.744
SS POST/PRE	0.84×	0.63×	1.34× [0.31, 5.83]	0.487	1.60×	2.17×	0.74× [0.23, 2.39]	0.381

Table C.5.2. UV versus PTMS processing-load robustness.

Metric	Primary KR UV-PTMS [95% CI]	Holm p	UV-PTMS in A	UV-PTMS in B	Batch- adjusted sensitivity [95% CI]	p	Treatment $\times$ batch p
Delta forward V_{th}	+3.28 V [+0.11, +6.45]	0.130	+5.55 V	+1.02 V	+3.28 V [-1.11, +7.68]	0.121	0.251
Delta reverse V_{th}	+6.70 V [+1.60, +11.80]	0.042	+9.54 V	+3.86 V	+6.70 V [-0.66, +14.05]	0.068	0.403
Delta signed hysteresis	+3.42 V [+0.59, +6.24]	0.065	+3.99 V	+2.84 V	+3.42 V [-0.45, +7.28]	0.075	0.754
$ I_{DS} $ POST/PRE	0.40 $\times$ [0.21, 0.75]	0.025	0.32 $\times$	0.49 $\times$	0.40 $\times$ [0.16, 0.99]	0.048	0.614
SS POST/PRE	2.56 $\times$ [1.35, 4.85]	0.022	1.90 $\times$	3.44 $\times$	2.56 $\times$ [1.37, 4.76]	0.009	0.291

Table C.5.3. Supporting processing-load sensitivity metrics.

Metric	PTMS A	PTMS B	PTMS A-B [95% CI]	p	UV A	UV B	UV A-B [95% CI]	p
I_{on}/I_{off} POST/PRE	2.19 $\times$	2.67 $\times$	0.82 $\times$ [0.01, 78.85]	0.869	1.28 $\times$	0.82 $\times$	1.56 $\times$ [0.30, 8.15]	0.368
max $ I_G $ POST/PRE	5.04 $\times$	1.75 $\times$	2.88 $\times$ [0.06, 147.39]	0.368	0.51 $\times$	1.09 $\times$	0.47 $\times$ [0.04, 5.34]	0.312

**Figure C.5.1.** Forward V_{th} by processing load and parent wafer. Points are wafer-piece mean PRE-to-POST responses for the two processing-load batches. Blue denotes W1, orange W2, and green W3; lines connect Batch A and Batch B for the same parent wafer within each processing sequence. The horizontal dotted line at 0 V denotes no processing-induced forward-threshold change.

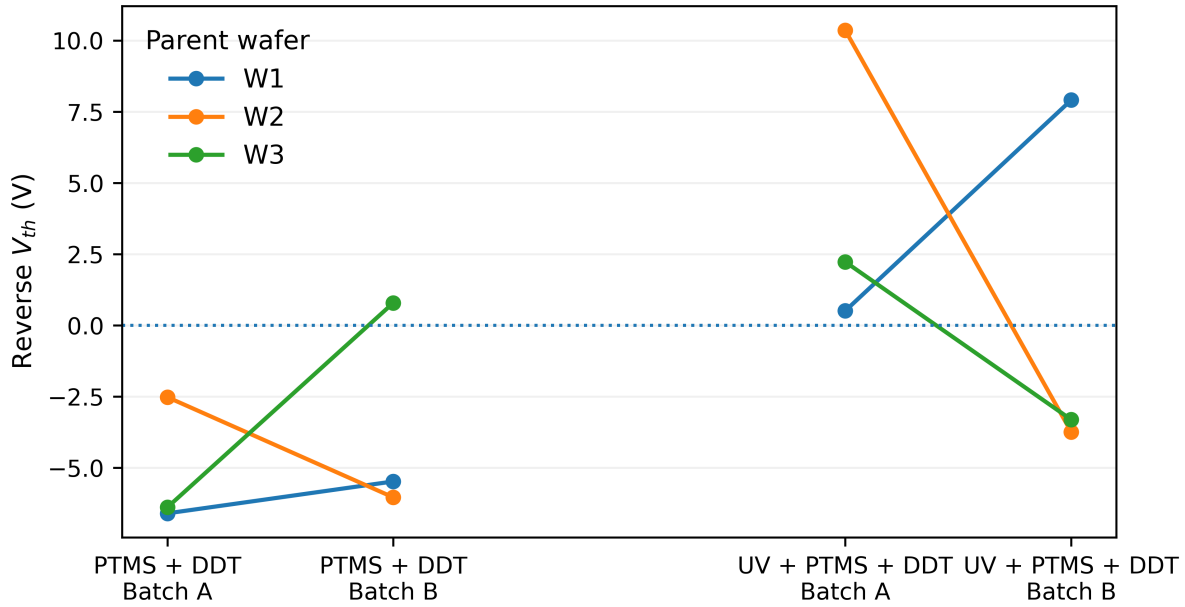


Figure C.5.2. Reverse V_{th} by processing load and parent wafer. Blue denotes W1, orange W2, and green W3; lines connect Batch A and Batch B for the same parent wafer within each processing sequence. The horizontal dotted line at 0 V denotes no processing-induced reverse-threshold change.

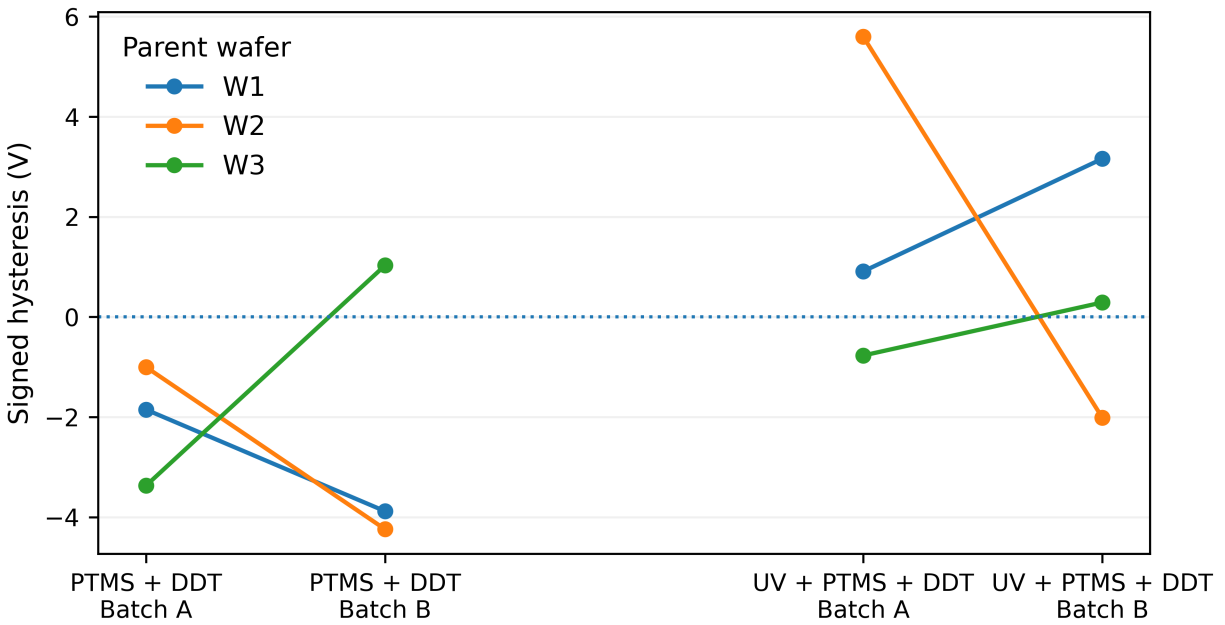


Figure C.5.3. Signed hysteresis by processing load and parent wafer. Blue denotes W1, orange W2, and green W3; lines connect Batch A and Batch B for the same parent wafer within each processing sequence. The horizontal dotted line at 0 V denotes no processing-induced change in signed hysteresis.

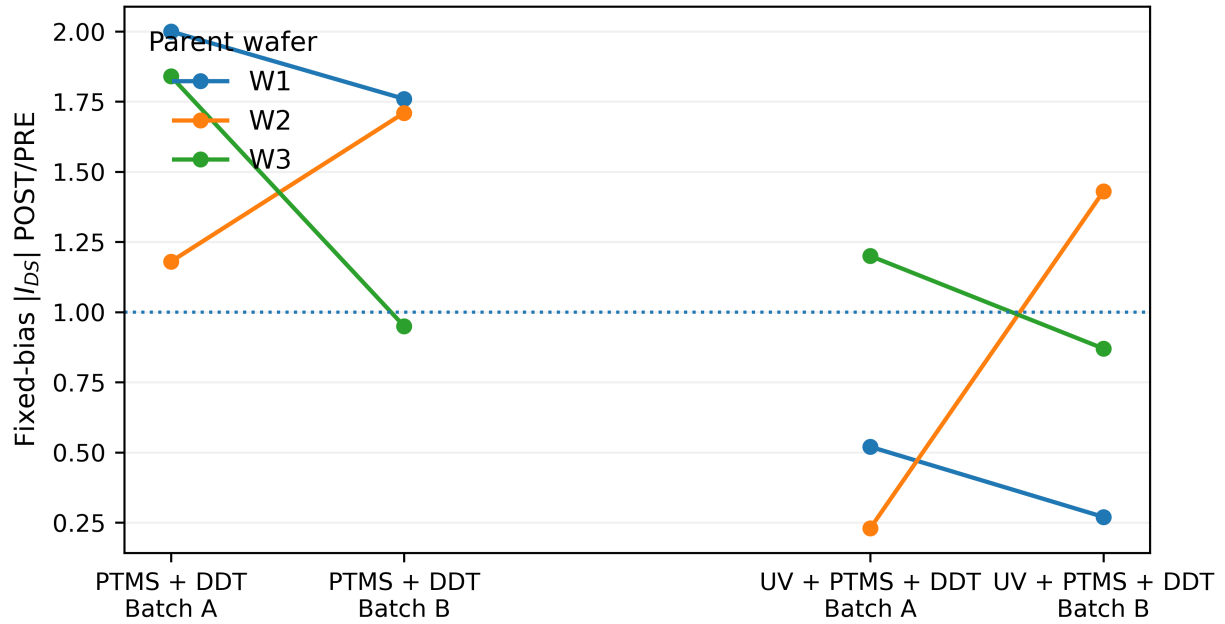


Figure C.5.4. Fixed-bias $|I_{DS}|$ by processing load and parent wafer. Blue denotes W1, orange W2, and green W3; lines connect Batch A and Batch B for the same parent wafer within each processing sequence. The horizontal dotted line at a POST/PRE ratio of 1 denotes no processing-induced change in fixed-bias drain-to-source current; values below and above 1 indicate decreases and increases, respectively.

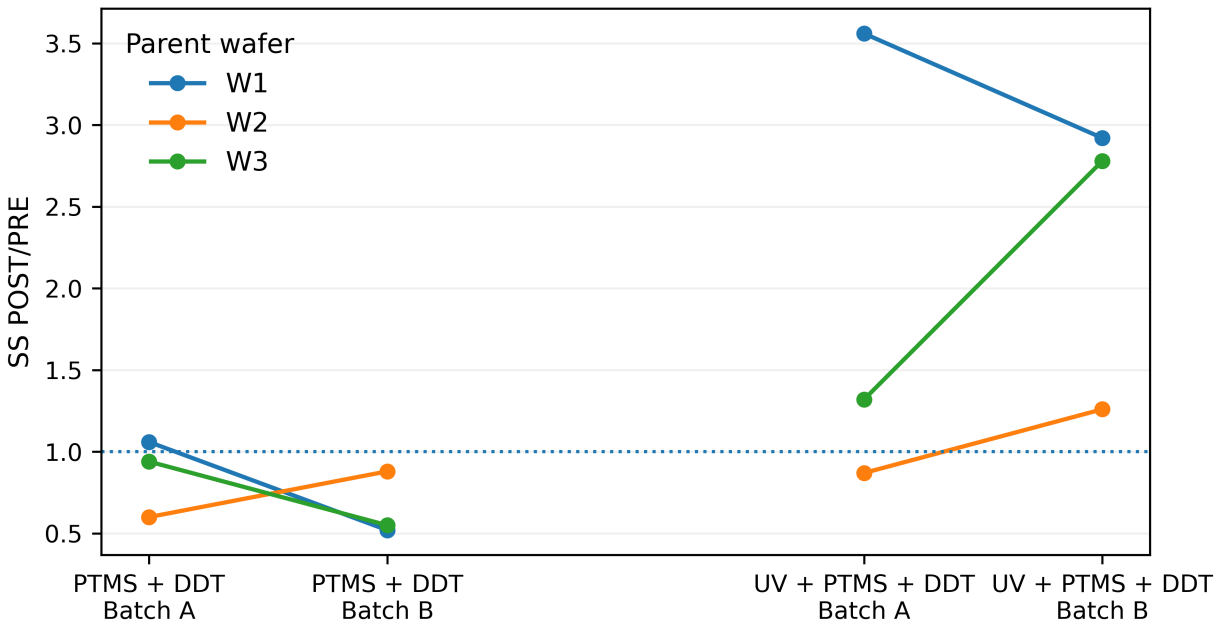


Figure C.5.5. Subthreshold swing by processing load and parent wafer. Blue denotes W1, orange W2, and green W3; lines connect Batch A and Batch B for the same parent wafer within each processing sequence. The horizontal dotted line at a POST/PRE ratio of 1 denotes no processing-induced change in subthreshold swing; values below and above 1 indicate decreases and increases, respectively.

APPENDIX D

Standardized T0–12 h Derived FET-Level and Descriptive Summaries

This appendix contains derived FET-level paired responses, condition-level paired summaries, and wafer-piece-level paired summaries from the standardized T0, 6 h, and 12 h phase obtained with the original back-gate configuration. Complete per-device transfer and gate-current characteristics, extracted electrical metrics, validity fields, and device-level quality-control records are provided in Supplemental Data S3. Day 7 results are kept separate in Appendix F.

D.1 Individual-FET paired voltage changes

Table D.1.1. Bare: individual-FET paired voltage changes.

Device	$dV_{th,F}$ 6 h	$dV_{th,F}$ 12 h	$dV_{th,R}$ 6 h	$dV_{th,R}$ 12 h	Δ hysteresis 6 h	Δ hysteresis 12 h
Air1	-3.006	0.533	-1.109	0.867	1.897	0.335
Air2	-2.469	-5.336	-1.202	-2.863	1.267	2.473
Air3	8.587	3.636	3.667	1.610	-4.921	-2.026
Air4	1.200	-3.675	-0.308	-2.460	-1.508	1.215
Air5	-0.059	-2.066	-5.754	-6.948	-5.695	-4.882
Air6	-0.513	-3.874	-0.249	-1.508	0.263	2.365
PBS1	10.026	—	8.029	—	-1.998	—
PBS2	12.697	—	—	—	—	—
PBS3	6.909	21.923	9.233	11.911	2.324	-10.012
PBS4	-4.685	9.300	7.273	—	11.959	—
PBS5	8.966	—	12.727	—	3.761	—
PBS6	4.949	6.619	8.939	9.725	3.990	3.106

Note. Changes are within-FET relative to the same device T0. Values are descriptive FET-level subsample responses; an em dash indicates that the paired metric was unavailable.

Table D.1.2. DDT: individual-FET paired voltage changes.

Device	$dV_{th,F}$ 6 h	$dV_{th,F}$ 12 h	$dV_{th,R}$ 6 h	$dV_{th,R}$ 12 h	Δ hysteresis 6 h	Δ hysteresis 12 h
Air1	-1.089	-2.807	-1.545	-2.679	-0.456	0.127
Air2	-1.259	-1.497	-2.217	-2.538	-0.959	-1.041
Air3	—	-0.412	-0.874	0.497	—	0.909
Air4	0.135	2.058	-0.481	2.277	-0.616	0.219
Air5	1.844	2.677	-1.255	-1.570	-3.098	-4.247
Air6	-2.782	-2.228	-3.141	-1.698	-0.359	0.529
PBS1	—	—	—	—	—	—
PBS2	-0.469	-8.868	7.592	9.519	8.061	18.387
PBS3	-2.160	3.498	6.317	11.552	8.476	8.055
PBS4	-2.393	2.558	5.587	10.809	7.980	8.251
PBS5	-2.948	0.859	6.644	10.443	9.592	9.583
PBS6	-8.715	4.446	5.519	12.238	14.234	7.792

Note. Changes are within-FET relative to the same device T0. Values are descriptive FET-level subsample responses; an em dash indicates that the paired metric was unavailable.

Table D.1.3. PTMS + DDT: individual-FET paired voltage changes.

Device	$dV_{th,F}$ 6 h	$dV_{th,F}$ 12 h	$dV_{th,R}$ 6 h	$dV_{th,R}$ 12 h	Δ hysteresis 6 h	Δ hysteresis 12 h
Air1	0.594	-0.362	0.322	0.276	-0.272	0.637
Air2	0.081	-2.125	-0.098	-1.441	-0.179	0.684
Air3	0.302	0.993	0.223	-0.110	-0.079	-1.103
Air4	-0.958	-1.329	-0.917	-1.003	0.041	0.326
Air5	3.590	3.879	1.955	2.377	-1.635	-1.502
Air6	2.334	-0.022	1.386	0.245	-0.948	0.267
PBS1	1.478	1.684	2.062	2.190	0.584	0.506
PBS2	2.003	2.686	4.437	6.738	2.433	4.052
PBS3	7.334	—	7.490	—	0.157	—
PBS4	5.260	3.828	8.045	10.586	2.785	6.759
PBS5	-12.969	-7.256	-2.595	7.050	10.373	14.307
PBS6	-0.339	0.209	8.714	8.518	9.053	8.309

Note. Changes are within-FET relative to the same device T0. Values are descriptive FET-level subsample responses; an em dash indicates that the paired metric was unavailable.

Table D.1.4. UV + PTMS + DDT: individual-FET paired voltage changes.

Device	$dV_{th,F}$ 6 h	$dV_{th,F}$ 12 h	$dV_{th,R}$ 6 h	$dV_{th,R}$ 12 h	Δ hysteresis 6 h	Δ hysteresis 12 h
Air1	3.932	-0.074	2.159	-0.024	-1.773	0.050
Air2	2.852	-3.912	0.627	-3.803	-2.225	0.109
Air3	8.638	6.809	3.569	1.494	-5.070	-5.316
Air4	2.696	-1.400	1.998	-1.536	-0.697	-0.136
Air5	0.964	2.134	1.143	0.867	0.179	-1.267
Air6	-2.810	-7.986	-2.724	-6.862	0.086	1.124
PBS1	-3.653	-6.885	2.931	1.069	6.584	7.954
PBS2	0.242	-3.356	4.347	2.832	4.105	6.188
PBS3	-4.653	—	1.122	0.382	5.775	—
PBS4	—	—	2.208	0.750	—	—
PBS5	3.363	2.495	3.836	3.249	0.473	0.754
PBS6	0.375	0.494	2.572	3.888	2.197	3.394

Note. Changes are within-FET relative to the same device T0. Values are descriptive FET-level subsample responses; an em dash indicates that the paired metric was unavailable.

D.2 Individual-FET paired supporting responses

Table D.2.1. Bare: individual-FET paired supporting responses.

Device	$\log_{10} I_{DS} $ ratio 6 h	$\log_{10} I_{DS} $ ratio 12 h	$\log_{10} SS$ ratio 6 h	$\log_{10} SS$ ratio 12 h	$\log_{10} I_{on}/I_{off}$ ratio 6 h	$\log_{10} I_{on}/I_{off}$ ratio 12 h	$\log_{10} \max I_G $ ratio 6 h	$\log_{10} \max I_G $ ratio 12 h
Air1	0.121	-0.068	-0.090	-0.010	-0.044	0.276	0.246	0.166
Air2	0.118	0.142	-0.405	-0.414	0.503	0.692	0.244	0.276
Air3	-0.299	-0.097	0.049	0.123	-0.122	-0.079	-0.453	-0.113
Air4	-0.005	0.106	0.130	-0.082	0.470	0.016	-0.153	0.321
Air5	0.258	0.308	0.476	0.291	0.099	0.187	0.187	0.273
Air6	-0.039	0.116	0.254	0.469	0.017	0.067	0.145	0.291
PBS1	-0.806	-1.557	-0.069	-0.272	-0.871	-1.557	-0.503	-0.615
PBS2	-0.853	—	0.896	0.900	-4.069	-4.726	-0.344	-0.036
PBS3	-0.556	-1.411	0.406	0.643	-2.000	-3.488	-0.165	-0.366
PBS4	-0.521	-0.919	—	0.639	-4.114	-2.999	-0.077	-0.097
PBS5	-0.687	-1.036	0.539	0.188	-0.168	-0.322	-0.082	-0.085
PBS6	-0.569	-0.789	-0.375	-0.255	-0.142	-0.331	0.084	0.032

Note. Values are $\log_{10}(\text{follow-up}/T0)$ responses calculated within the same FET. Numerical availability follows the metric-specific validity rules in Chapter 2; an em dash indicates an unavailable paired response.

Table D.2.2. DDT: individual-FET paired supporting responses.

Device	$\log_{10} I_{DS} $ ratio 6 h	$\log_{10} I_{DS} $ ratio 12 h	$\log_{10} SS$ ratio 6 h	$\log_{10} SS$ ratio 12 h	$\log_{10} I_{on}/I_{off}$ ratio 6 h	$\log_{10} I_{on}/I_{off}$ ratio 12 h	$\log_{10} \max I_G $ ratio 6 h	$\log_{10} \max I_G $ ratio 12 h
Air1	0.078	0.164	0.241	0.353	0.156	0.124	0.150	0.196
Air2	0.072	0.098	0.303	0.178	0.016	0.108	-0.096	-0.081
Air3	—	-0.003	—	0.052	-7.186	-0.014	-2.404	0.080
Air4	0.006	-0.080	0.412	-0.112	-0.035	-0.016	0.002	-0.019
Air5	0.059	0.052	-0.488	0.162	-0.276	-0.930	-0.069	-0.113
Air6	0.095	0.030	0.288	0.570	-0.210	-8.41e-04	0.215	-0.033
PBS1	-0.535	—	0.067	-0.355	-2.993	-4.098	0.029	2.365
PBS2	-0.398	-0.613	0.551	0.276	-1.674	-1.902	-0.246	-0.334
PBS3	-0.336	-0.699	0.489	0.294	-0.694	-0.595	-0.257	-0.232
PBS4	-0.350	-0.593	0.610	0.462	-1.526	-1.084	-0.116	-0.249
PBS5	-0.262	-0.438	-0.020	-0.082	-0.644	-0.068	-0.132	-0.109
PBS6	-0.160	-0.613	-0.596	-0.638	1.689	1.818	-0.066	-0.219

Note. Values are $\log_{10}(\text{follow-up}/T_0)$ responses calculated within the same FET. Numerical availability follows the metric-specific validity rules in Chapter 2; an em dash indicates an unavailable paired response.

Table D.2.3. PTMS + DDT: individual-FET paired supporting responses.

Device	$\log_{10} I_{DS} $ ratio 6 h	$\log_{10} I_{DS} $ ratio 12 h	$\log_{10} SS$ ratio 6 h	$\log_{10} SS$ ratio 12 h	$\log_{10} I_{on}/I_{off}$ ratio 6 h	$\log_{10} I_{on}/I_{off}$ ratio 12 h	$\log_{10} \max I_G $ ratio 6 h	$\log_{10} \max I_G $ ratio 12 h
Air1	-0.029	-0.012	0.220	-0.227	-0.690	0.913	-0.032	-0.003
Air2	9.86e-04	0.057	0.098	0.076	-0.299	0.024	0.024	0.086
Air3	0.050	-0.303	0.588	-0.053	-0.155	0.132	0.108	-0.442
Air4	-0.014	0.050	0.337	0.024	-1.165	0.259	-0.029	0.032
Air5	-0.091	-0.105	0.070	0.629	0.109	-0.124	0.054	-0.084
Air6	-0.071	-0.015	-0.097	0.034	0.206	0.033	-0.072	0.076
PBS1	-0.159	-0.201	0.132	0.103	-0.573	-0.146	-0.141	-0.163
PBS2	-0.642	-0.416	0.206	0.089	-0.097	-1.426	-0.648	-0.204
PBS3	-0.455	—	0.338	—	-0.876	-4.329	-0.038	1.287
PBS4	-0.308	-0.487	0.387	0.180	-0.591	-0.902	0.035	-0.093
PBS5	0.058	-0.230	0.106	-0.008	-0.697	-0.206	-0.158	-0.152
PBS6	-0.280	-0.411	—	0.601	-4.955	-1.213	-0.109	-0.183

Note. Values are $\log_{10}(\text{follow-up}/T_0)$ responses calculated within the same FET. Numerical availability follows the metric-specific validity rules in Chapter 2; an em dash indicates an unavailable paired response.

Table D.2.4. UV + PTMS + DDT: individual-FET paired supporting responses.

Device	$\log_{10} I_{DS} $ ratio 6 h	$\log_{10} I_{DS} $ ratio 12 h	$\log_{10} SS$ ratio 6 h	$\log_{10} SS$ ratio 12 h	$\log_{10} I_{on}/I_{off}$ ratio 6 h	$\log_{10} I_{on}/I_{off}$ ratio 12 h	$\log_{10} \max I_G $ ratio 6 h	$\log_{10} \max I_G $ ratio 12 h
Air1	-0.102	0.224	0.014	-0.088	-0.215	0.125	-0.273	-0.177
Air2	-0.203	0.455	-0.005	-0.415	0.398	0.479	-0.263	0.057
Air3	-0.301	-0.110	0.056	-0.011	0.430	0.529	-0.915	-0.841
Air4	-0.069	0.104	0.093	-0.196	-0.087	0.180	-0.070	0.072
Air5	-0.066	-0.076	0.148	0.020	0.275	0.242	-0.076	-0.114
Air6	0.273	0.641	0.060	-0.393	0.836	1.037	-0.173	0.060
PBS1	0.106	0.519	-0.138	-0.125	0.142	0.420	0.080	0.026
PBS2	-0.155	0.064	-0.237	-0.084	-0.482	-0.262	-0.070	-0.185
PBS3	0.035	0.021	0.147	0.411	0.100	-0.496	-0.016	0.356
PBS4	-0.200	-0.287	0.211	-0.058	-0.201	-0.099	-0.030	-0.324
PBS5	-0.324	-0.230	-0.028	-0.252	-2.329	-1.525	-0.070	-0.096
PBS6	-0.164	-0.204	-0.155	-0.193	-0.132	-0.223	0.022	0.008

Note. Values are $\log_{10}(\text{follow-up}/T_0)$ responses calculated within the same FET. Numerical availability follows the metric-specific validity rules in Chapter 2; an em dash indicates an unavailable paired response.

D.3 Condition-level paired means

Table D.3.1. Bare: condition-level paired means.

Metric	Time	Air mean $\pm$ SD	Air n	PBS mean $\pm$ SD	PBS n
$\Delta V_{th,F}$	6 h	0.624 ± 4.201	6	6.477 ± 6.076	6
$\Delta V_{th,F}$	12 h	-1.797 ± 3.328	6	12.614 ± 8.173	3
$\Delta V_{th,R}$	6 h	-0.826 ± 3.010	6	9.240 ± 2.097	5
$\Delta V_{th,R}$	12 h	-1.884 ± 3.061	6	10.818 ± 1.546	2
$\Delta hysteresis$	6 h	-1.449 ± 3.212	6	4.007 ± 5.054	5
$\Delta hysteresis$	12 h	-0.087 ± 2.870	6	-3.453 ± 9.276	2
$\log_{10} I_{DS} $	6 h	0.025 ± 0.191	6	-0.666 ± 0.139	6
$\log_{10} I_{DS} $	12 h	0.085 ± 0.149	6	-1.143 ± 0.328	5
$\log SS$	6 h	0.069 ± 0.301	6	0.279 ± 0.503	5
$\log SS$	12 h	0.063 ± 0.308	6	0.307 ± 0.498	6
$\log I_{on}/I_{off}$	6 h	0.154 ± 0.268	6	-1.894 ± 1.831	6
$\log I_{on}/I_{off}$	12 h	0.193 ± 0.275	6	-2.237 ± 1.795	6
$\log \max I_G$	6 h	0.036 ± 0.282	6	-0.181 ± 0.210	6
$\log \max I_G$	12 h	0.202 ± 0.163	6	-0.195 ± 0.247	6

Note. Condition summaries use the same contributing FETs within each paired comparison. Denominators vary when a metric is unavailable. These are descriptive FET-level summaries, not independent treatment replicates.

Table D.3.2. DDT: condition-level paired means.

Metric	Time	Air mean $\pm$ SD	Air n	PBS mean $\pm$ SD	PBS n
$\Delta V_{th,F}$	6 h	-0.630 ± 1.728	5	-3.337 ± 3.146	5
$\Delta V_{th,F}$	12 h	-0.368 ± 2.273	6	0.499 ± 5.401	5
$\Delta V_{th,R}$	6 h	-1.586 ± 0.965	6	6.332 ± 0.852	5
$\Delta V_{th,R}$	12 h	-0.952 ± 1.948	6	10.912 ± 1.042	5
$\Delta hysteresis$	6 h	-1.098 ± 1.141	5	9.669 ± 2.632	5
$\Delta hysteresis$	12 h	-0.584 ± 1.910	6	10.413 ± 4.510	5
$\log_{10} I_{DS} $	6 h	0.062 ± 0.034	5	-0.340 ± 0.127	6
$\log_{10} I_{DS} $	12 h	0.043 ± 0.084	6	-0.591 ± 0.095	5
$\log SS$	6 h	0.151 ± 0.363	5	0.184 ± 0.463	6
$\log SS$	12 h	0.200 ± 0.237	6	-0.007 ± 0.428	6
$\log I_{on}/I_{off}$	6 h	-1.256 ± 2.909	6	-0.974 ± 1.559	6
$\log I_{on}/I_{off}$	12 h	-0.121 ± 0.401	6	-0.988 ± 1.969	6
$\log \max I_G$	6 h	-0.367 ± 1.006	6	-0.132 ± 0.109	6
$\log \max I_G$	12 h	0.005 ± 0.114	6	0.204 ± 1.061	6

Note. Condition summaries use the same contributing FETs within each paired comparison. Denominators vary when a metric is unavailable. These are descriptive FET-level summaries, not independent treatment replicates.

Table D.3.3. PTMS + DDT: condition-level paired means.

Metric	Time	Air mean $\pm$ SD	Air n	PBS mean $\pm$ SD	PBS n
$\Delta V_{th,F}$	6 h	0.991 ± 1.662	6	0.461 ± 7.137	6
$\Delta V_{th,F}$	12 h	0.172 ± 2.110	6	0.230 ± 4.391	5
$\Delta V_{th,R}$	6 h	0.479 ± 1.037	6	4.692 ± 4.367	6
$\Delta V_{th,R}$	12 h	0.057 ± 1.331	6	7.017 ± 3.097	5
$\Delta hysteresis$	6 h	-0.512 ± 0.650	6	4.231 ± 4.386	6
$\Delta hysteresis$	12 h	-0.115 ± 0.943	6	6.786 ± 5.143	5
$\log_{10} I_{DS} $	6 h	-0.026 ± 0.051	6	-0.298 ± 0.241	6
$\log_{10} I_{DS} $	12 h	-0.055 ± 0.135	6	-0.349 ± 0.126	5
$\log SS$	6 h	0.202 ± 0.239	6	0.234 ± 0.124	5
$\log SS$	12 h	0.081 ± 0.289	6	0.193 ± 0.238	5
$\log I_{on}/I_{off}$	6 h	-0.332 ± 0.517	6	-1.298 ± 1.810	6
$\log I_{on}/I_{off}$	12 h	0.206 ± 0.369	6	-1.370 ± 1.540	6
$\log \max I_G$	6 h	0.009 ± 0.066	6	-0.176 ± 0.242	6
$\log \max I_G$	12 h	-0.056 ± 0.199	6	0.082 ± 0.592	6

Note. Condition summaries use the same contributing FETs within each paired comparison. Denominators vary when a metric is unavailable. These are descriptive FET-level summaries, not independent treatment replicates.

Table D.3.4. UV + PTMS + DDT: condition-level paired means.

Metric	Time	Air mean $\pm$ SD	Air n	PBS mean $\pm$ SD	PBS n
$\Delta V_{th,F}$	6 h	2.712 ± 3.746	6	-0.865 ± 3.270	5
$\Delta V_{th,F}$	12 h	-0.738 ± 5.073	6	-1.813 ± 4.163	4
$\Delta V_{th,R}$	6 h	1.129 ± 2.138	6	2.836 ± 1.157	6
$\Delta V_{th,R}$	12 h	-1.644 ± 3.184	6	2.028 ± 1.474	6
$\Delta hysteresis$	6 h	-1.583 ± 1.964	6	3.827 ± 2.519	5
$\Delta hysteresis$	12 h	-0.906 ± 2.291	6	4.573 ± 3.163	4
$\log_{10} I_{DS} $	6 h	-0.078 ± 0.194	6	-0.117 ± 0.159	6
$\log_{10} I_{DS} $	12 h	0.206 ± 0.297	6	-0.019 ± 0.300	6
$\log SS$	6 h	0.061 ± 0.055	6	-0.033 ± 0.179	6
$\log SS$	12 h	-0.181 ± 0.189	6	-0.050 ± 0.237	6
$\log I_{on}/I_{off}$	6 h	0.273 ± 0.381	6	-0.484 ± 0.932	6
$\log I_{on}/I_{off}$	12 h	0.432 ± 0.338	6	-0.364 ± 0.645	6
$\log \max I_G$	6 h	-0.295 ± 0.316	6	-0.014 ± 0.058	6
$\log \max I_G$	12 h	-0.157 ± 0.351	6	-0.036 ± 0.232	6

Note. Condition summaries use the same contributing FETs within each paired comparison. Denominators vary when a metric is unavailable. These are descriptive FET-level summaries, not independent treatment replicates.

D.4 Wafer-piece-level paired responses

Table D.4.1. Bare: wafer-piece-level paired responses.

Metric	Time	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
$\Delta V_{th,F}$	6 h	0.624	-2.737	4.894	-0.286	6.477	11.362	1.112	6.957
$\Delta V_{th,F}$	12 h	-1.797	-2.401	-0.019	-2.970	11.115	—	15.612	6.619
$\Delta V_{th,R}$	6 h	-0.826	-1.156	1.679	-3.001	9.038	8.029	8.253	10.833
$\Delta V_{th,R}$	12 h	-1.884	-0.998	-0.425	-4.228	10.818	—	11.911	9.725
$\Delta hysteresis$	6 h	-1.449	1.582	-3.214	-2.716	3.006	-1.998	7.141	3.876
$\Delta hysteresis$	12 h	-0.087	1.404	-0.406	-1.259	-3.453	—	-10.012	3.106
$\log_{10} I_{DS} $	6 h	0.025	0.119	-0.152	0.109	-0.666	-0.830	-0.539	-0.628
$\log_{10} I_{DS} $	12 h	0.085	0.037	0.004	0.212	-1.212	-1.557	-1.165	-0.913
$\log SS$	6 h	0.069	-0.248	0.089	0.365	0.300	0.414	0.406	0.082
$\log SS$	12 h	0.063	-0.212	0.021	0.380	0.307	0.314	0.641	-0.033
$\log I_{on}/I_{off}$	6 h	0.154	0.230	0.174	0.058	-1.894	-2.470	-3.057	-0.155
$\log I_{on}/I_{off}$	12 h	0.193	0.484	-0.032	0.127	-2.237	-3.142	-3.244	-0.327
$\log \max I_G$	6 h	0.036	0.245	-0.303	0.166	-0.181	-0.424	-0.121	0.001
$\log \max I_G$	12 h	0.202	0.221	0.104	0.282	-0.195	-0.326	-0.232	-0.026

Note. Piece-level values average the available FET subsamples on each independently treated wafer piece. W1–W3 identify the parent-wafer blocks.

Table D.4.2. DDT: wafer-piece-level paired responses.

Metric	Time	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
$\Delta V_{th,F}$	6 h	-0.503	-1.174	0.135	-0.469	-2.859	-0.469	-2.276	-5.832
$\Delta V_{th,F}$	12 h	-0.368	-2.152	0.823	0.225	-1.062	-8.868	3.028	2.653
$\Delta V_{th,R}$	6 h	-1.586	-1.881	-0.678	-2.198	6.542	7.592	5.952	6.081
$\Delta V_{th,R}$	12 h	-0.952	-2.609	1.387	-1.634	10.680	9.519	11.181	11.340
$\Delta hysteresis$	6 h	-1.017	-0.707	-0.616	-1.729	9.401	8.061	8.228	11.913
$\Delta hysteresis$	12 h	-0.584	-0.457	0.564	-1.859	11.742	18.387	8.153	8.687
$\log_{10} I_{DS} $	6 h	0.053	0.075	0.006	0.077	-0.340	-0.467	-0.343	-0.211
$\log_{10} I_{DS} $	12 h	0.043	0.131	-0.042	0.041	-0.595	-0.613	-0.646	-0.526
$\log SS$	6 h	0.194	0.272	0.412	-0.100	0.184	0.309	0.549	-0.308
$\log SS$	12 h	0.200	0.265	-0.030	0.366	-0.007	-0.039	0.378	-0.360
$\log I_{on}/I_{off}$	6 h	-1.256	0.086	-3.610	-0.243	-0.974	-2.333	-1.110	0.523
$\log I_{on}/I_{off}$	12 h	-0.121	0.116	-0.015	-0.465	-0.988	-3.000	-0.839	0.875
$\log \max I_G$	6 h	-0.367	0.027	-1.201	0.073	-0.132	-0.109	-0.187	-0.099
$\log \max I_G$	12 h	0.005	0.057	0.030	-0.073	0.204	1.016	-0.241	-0.164

Note. Piece-level values average the available FET subsamples on each independently treated wafer piece. W1–W3 identify the parent-wafer blocks.

Table D.4.3. PTMS + DDT: wafer-piece-level paired responses.

Metric	Time	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
$\Delta V_{th,F}$	6 h	0.991	0.338	-0.328	2.962	0.461	1.741	6.297	-6.654
$\Delta V_{th,F}$	12 h	0.172	-1.243	-0.168	1.929	0.830	2.185	3.828	-3.524
$\Delta V_{th,R}$	6 h	0.479	0.112	-0.347	1.671	4.692	3.250	7.768	3.059
$\Delta V_{th,R}$	12 h	0.057	-0.583	-0.557	1.311	7.612	4.464	10.586	7.784
$\Delta hysteresis$	6 h	-0.512	-0.226	-0.019	-1.291	4.231	1.509	1.471	9.713
$\Delta hysteresis$	12 h	-0.115	0.661	-0.388	-0.618	6.782	2.279	6.759	11.308
$\log_{10} I_{DS} $	6 h	-0.026	-0.014	0.018	-0.081	-0.298	-0.401	-0.382	-0.111
$\log_{10} I_{DS} $	12 h	-0.055	0.022	-0.127	-0.060	-0.372	-0.309	-0.487	-0.320
$\log SS$	6 h	0.202	0.159	0.462	-0.014	0.212	0.169	0.362	0.106
$\log SS$	12 h	0.081	-0.076	-0.014	0.331	0.191	0.096	0.180	0.297
$\log I_{on}/I_{off}$	6 h	-0.332	-0.495	-0.660	0.157	-1.298	-0.335	-0.733	-2.826
$\log I_{on}/I_{off}$	12 h	0.206	0.469	0.195	-0.045	-1.370	-0.786	-2.615	-0.709
$\log \max I_G$	6 h	0.009	-0.004	0.040	-0.009	-0.176	-0.394	-0.001	-0.133
$\log \max I_G$	12 h	-0.056	0.041	-0.205	-0.004	0.082	-0.183	0.597	-0.168

Note. Piece-level values average the available FET subsamples on each independently treated wafer piece. W1–W3 identify the parent-wafer blocks.

Table D.4.4. UV + PTMS + DDT: wafer-piece-level paired responses.

Metric	Time	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
$\Delta V_{th,F}$	6 h	2.712	3.392	5.667	-0.923	-1.496	-1.706	-4.653	1.869
$\Delta V_{th,F}$	12 h	-0.738	-1.993	2.705	-2.926	-1.813	-5.121	—	1.495
$\Delta V_{th,R}$	6 h	1.129	1.393	2.784	-0.790	2.836	3.639	1.665	3.204
$\Delta V_{th,R}$	12 h	-1.644	-1.913	-0.021	-2.997	2.028	1.950	0.566	3.569
$\Delta hysteresis$	6 h	-1.583	-1.999	-2.883	0.133	4.151	5.344	5.775	1.335
$\Delta hysteresis$	12 h	-0.906	0.080	-2.726	-0.071	4.573	7.071	—	2.074
$\log_{10} I_{DS} $	6 h	-0.078	-0.152	-0.185	0.103	-0.117	-0.025	-0.082	-0.244
$\log_{10} I_{DS} $	12 h	0.206	0.339	-0.003	0.283	-0.019	0.292	-0.133	-0.217
$\log SS$	6 h	0.061	0.004	0.074	0.104	-0.033	-0.187	0.179	-0.091
$\log SS$	12 h	-0.181	-0.251	-0.104	-0.187	-0.050	-0.104	0.176	-0.222
$\log I_{on}/I_{off}$	6 h	0.273	0.091	0.172	0.556	-0.484	-0.170	-0.050	-1.231
$\log I_{on}/I_{off}$	12 h	0.432	0.302	0.355	0.640	-0.364	0.079	-0.297	-0.874
$\log \max I_G$	6 h	-0.295	-0.268	-0.492	-0.124	-0.014	0.005	-0.023	-0.024
$\log \max I_G$	12 h	-0.157	-0.060	-0.384	-0.027	-0.036	-0.079	0.016	-0.044

Note. Piece-level values average the available FET subsamples on each independently treated wafer piece. W1–W3 identify the parent-wafer blocks.

APPENDIX E

Standardized T0–12 h Statistical Analyses

This appendix contains supporting Kenward–Roger model-fit information and contrasts for the standardized T0–12 h phase, together with sensitivity analyses for the primary 12 h forward-threshold result. The primary 12 h results already presented in Chapter 5 (Table 5.1 and Figure 5.3) are not duplicated here. The sensitivity table uses the final HC3/Kenward–Roger inference corresponding to the Chapter 5 prose rather than the earlier provisional Wald-output table.

Table E.1. Supporting T0–12 h mixed-model availability and fit status.

Outcome	FET N	Piece N	Piece variance	Residual variance	Fit status
6 h forward threshold change	45	24	9.6651	11.9910	Converged after optimizer fallback
6 h reverse threshold change	46	24	0.2425	5.1406	Converged after optimizer fallback
12 h reverse threshold change	42	23	0.4501	4.7512	Converged after optimizer fallback
6 h signed hysteresis change	44	24	5.7541	5.2816	Converged
12 h signed hysteresis change	40	22	10.6808	5.5385	Converged
6 h $\log_{10}$ fixed-bias I_{DS} ratio	47	24	0.0061	0.0199	Converged after optimizer fallback
12 h $\log_{10}$ fixed-bias I_{DS} ratio	45	24	4.419e-12	0.0415	Converged; boundary piece variance
6 h $\log_{10}$ SS ratio	45	24	0.0117	0.0746	Converged
12 h $\log_{10}$ SS ratio	47	24	0.0191	0.0888	Converged after optimizer fallback
6 h $\log_{10}$ I_{on}/I_{off} ratio	48	24	0.3875	2.0890	Converged after optimizer fallback
12 h $\log_{10}$ I_{on}/I_{off} ratio	48	24	0.6010	0.7997	Converged after optimizer fallback
6 h $\log_{10}$ max $ I_G $ ratio	48	24	0.0087	0.1508	Converged after optimizer fallback
12 h $\log_{10}$ max $ I_G $ ratio	48	24	3.212e-33	0.2606	Converged; boundary piece variance

Note. FET n and piece n vary by outcome according to the prespecified metric-validity rules. Fit-status comments are retained as model diagnostics/availability information from the final Chapter 5 analysis package.

Table E.2. Sensitivity analyses for the primary 12 h forward-threshold result.

Analysis	Variant	Contrast	Inference	Estimate (V)	95% CI	<i>P</i>
Piece-level HC3	All pieces	DDT vs Bare	HC3	−12.45	[−25.00, +0.09]	0.052
Piece-level HC3	All pieces	PTMS + DDT vs DDT	HC3	+1.35	[−9.86, +12.57]	0.813
Piece-level HC3	All pieces	UV + PTMS + DDT vs PTMS + DDT	HC3	−0.39	[−11.82, +11.05]	0.947
Leave-one-wafer-out	Omit W1	DDT vs Bare	KR	−11.31	[−22.35, −0.27]	0.046
Leave-one-wafer-out	Omit W1	PTMS + DDT vs DDT	KR	−3.79	[−14.83, +7.25]	0.428
Leave-one-wafer-out	Omit W1	UV + PTMS + DDT vs PTMS + DDT	KR	+4.52	[−7.90, +16.95]	0.398
Leave-one-wafer-out	Omit W2	DDT vs Bare	KR	−9.59	[−24.53, +5.36]	0.177
Leave-one-wafer-out	Omit W2	PTMS + DDT vs DDT	KR	+0.33	[−12.70, +13.36]	0.953
Leave-one-wafer-out	Omit W2	UV + PTMS + DDT vs PTMS + DDT	KR	+1.66	[−11.29, +14.61]	0.762
Leave-one-wafer-out	Omit W3	DDT vs Bare	KR	−15.84	[−27.70, −3.97]	0.020
Leave-one-wafer-out	Omit W3	PTMS + DDT vs DDT	KR	+4.97	[−5.62, +15.57]	0.284
Leave-one-wafer-out	Omit W3	UV + PTMS + DDT vs PTMS + DDT	KR	−7.65	[−19.52, +4.21]	0.151
Baseline-covariate	12 h $V_{th,F} \sim T0 V_{th,F}$	DDT vs Bare	KR	−12.05	[−20.64, −3.47]	0.010
Baseline-covariate	12 h $V_{th,F} \sim T0 V_{th,F}$	PTMS + DDT vs DDT	KR	−0.15	[−8.10, +7.80]	0.968
Baseline-covariate	12 h $V_{th,F} \sim T0 V_{th,F}$	UV + PTMS + DDT vs PTMS + DDT	KR	−0.50	[−8.84, +7.83]	0.896
Both-FET-valid pieces	Both FETs valid	DDT vs Bare	KR	−13.41	[−22.52, −4.30]	0.009
Both-FET-valid pieces	Both FETs valid	PTMS + DDT vs DDT	KR	−2.79	[−10.86, +5.27]	0.454
Both-FET-valid pieces	Both FETs valid	UV + PTMS + DDT vs PTMS + DDT	KR	−0.23	[−8.06, +7.59]	0.948

Note. Seq1 = DDT vs Bare; Seq2 = PTMS + DDT vs DDT; Seq3 = UV + PTMS + DDT vs PTMS + DDT. Piece-level inference uses HC3 heteroscedasticity-consistent covariance; mixed-model sensitivity variants use Kenward–Roger small-sample inference. These sensitivity *P* values are not members of the primary Holm-adjusted hypothesis family.

Table E.3.1. 6 h forward threshold change: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	5.854	3.231	13.00	-1.127 to 12.834	0.0932
DDT PBS-Air	-2.558	3.409	15.32	-9.812 to 4.695	0.4644
PTMS + DDT PBS-Air	-0.529	3.231	13.00	-7.510 to 6.451	0.8724
UV+PTMS + DDT PBS-Air	-3.819	3.321	14.13	-10.934 to 3.297	0.2692
Sequential DoD: DDT - Bare	-8.412	4.697	14.16	-18.476 to 1.652	0.0947
Sequential DoD: PTMS + DDT - DDT	2.029	4.697	14.16	-8.035 to 12.093	0.6723
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	-3.289	4.633	13.57	-13.256 to 6.678	0.4898

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.2. 6 h reverse threshold change: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	10.020	1.443	14.52	6.935 to 13.105	5.61e-06
DDT PBS-Air	7.893	1.443	14.52	4.808 to 10.979	7.25e-05
PTMS + DDT PBS-Air	4.214	1.369	12.85	1.252 to 7.176	0.0089
UV+PTMS + DDT PBS-Air	1.707	1.369	12.85	-1.255 to 4.669	0.2347
Sequential DoD: DDT - Bare	-2.127	2.037	14.35	-6.484 to 2.231	0.3136
Sequential DoD: PTMS + DDT - DDT	-3.680	1.990	13.70	-7.956 to 0.596	0.0861
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	-2.506	1.937	12.85	-6.695 to 1.682	0.2184

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.3. 12 h reverse threshold change: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	12.304	1.901	22.58	8.367 to 16.242	1.45e-06
DDT PBS-Air	11.698	1.444	12.69	8.570 to 14.825	2.29e-06
PTMS + DDT PBS-Air	7.182	1.444	12.68	4.055 to 10.309	2.74e-04
UV+PTMS + DDT PBS-Air	3.672	1.373	11.22	0.659 to 6.686	0.0212
Sequential DoD: DDT - Bare	-0.607	2.375	18.24	-5.592 to 4.379	0.8013
Sequential DoD: PTMS + DDT - DDT	-4.515	2.044	12.77	-8.940 to -0.091	0.0461
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	-3.510	1.992	11.96	-7.852 to 0.832	0.1036

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.4. 6 h signed hysteresis change: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	4.852	2.421	13.84	-0.347 to 10.050	0.0651
DDT PBS-Air	10.544	2.476	14.88	5.263 to 15.826	6.99e-04
PTMS + DDT PBS-Air	4.743	2.366	12.85	-0.374 to 9.860	0.0665
UV+PTMS + DDT PBS-Air	5.581	2.421	13.84	0.382 to 10.779	0.0372
Sequential DoD: DDT - Bare	5.693	3.461	14.29	-1.717 to 13.102	0.1219
Sequential DoD: PTMS + DDT - DDT	-5.801	3.425	13.86	-13.153 to 1.550	0.1126
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.838	3.385	13.34	-6.456 to 8.132	0.8082

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.5. 12 h signed hysteresis change: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-2.778	3.596	14.15	-10.485 to 4.928	0.4525
DDT PBS-Air	11.996	3.043	11.96	5.365 to 18.628	0.0020
PTMS + DDT PBS-Air	6.804	3.043	11.96	0.173 to 13.436	0.0452
UV+PTMS + DDT PBS-Air	4.698	3.400	11.35	-2.756 to 12.153	0.1936
Sequential DoD: DDT - Bare	14.775	4.702	13.10	4.625 to 24.924	0.0077
Sequential DoD: PTMS + DDT - DDT	-5.192	4.303	11.97	-14.571 to 4.187	0.2509
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	-2.106	4.553	11.53	-12.071 to 7.858	0.6522

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.6. 6 h $\log_{10}$ fixed-bias $|I_{DS}|$ ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-0.691	0.104	13.54	-0.914 to -0.468	1.25e-05
DDT PBS-Air	-0.396	0.108	15.04	-0.625 to -0.166	0.0022
PTMS + DDT PBS-Air	-0.272	0.104	13.54	-0.495 to -0.049	0.0204
UV+PTMS + DDT PBS-Air	-0.039	0.104	13.54	-0.262 to 0.184	0.7123
Sequential DoD: DDT - Bare	0.295	0.149	14.30	-0.025 to 0.615	0.0679
Sequential DoD: PTMS + DDT - DDT	0.124	0.149	14.30	-0.196 to 0.444	0.4208
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.233	0.146	13.54	-0.082 to 0.548	0.1347

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.7. 12 h $\log_{10}$ fixed-bias $|I_{DS}|$ ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-1.216	0.124	13.84	-1.483 to -0.949	1.37e-07
DDT PBS-Air	-0.624	0.124	13.84	-0.891 to -0.356	1.97e-04
PTMS + DDT PBS-Air	-0.314	0.124	13.83	-0.581 to -0.046	0.0247
UV+PTMS + DDT PBS-Air	-0.226	0.118	12.18	-0.482 to 0.030	0.0786
Sequential DoD: DDT - Bare	0.592	0.175	13.66	0.215 to 0.969	0.0047
Sequential DoD: PTMS + DDT - DDT	0.310	0.176	13.93	-0.068 to 0.688	0.1004
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.088	0.171	13.02	-0.282 to 0.457	0.6172

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.8. 6 h $\log_{10}$ SS ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	0.242	0.189	13.98	-0.164 to 0.648	0.2215
DDT PBS-Air	-0.003	0.189	13.98	-0.409 to 0.403	0.9872
PTMS + DDT PBS-Air	0.006	0.189	13.97	-0.401 to 0.412	0.9772
UV+PTMS + DDT PBS-Air	-0.094	0.181	12.43	-0.486 to 0.298	0.6108
Sequential DoD: DDT - Bare	-0.245	0.268	14.12	-0.820 to 0.329	0.3756
Sequential DoD: PTMS + DDT - DDT	0.009	0.267	13.90	-0.565 to 0.583	0.9748
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	-0.100	0.262	13.20	-0.664 to 0.465	0.7089

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.9. 12 h $\log_{10}$ SS ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	0.244	0.206	13.50	-0.198 to 0.687	0.2553
DDT PBS-Air	-0.208	0.206	13.50	-0.650 to 0.235	0.3304
PTMS + DDT PBS-Air	0.125	0.215	15.08	-0.332 to 0.583	0.5678
UV+PTMS + DDT PBS-Air	0.130	0.206	13.50	-0.312 to 0.573	0.5369
Sequential DoD: DDT - Bare	-0.452	0.291	13.50	-1.078 to 0.174	0.1433
Sequential DoD: PTMS + DDT - DDT	0.333	0.297	14.30	-0.304 to 0.970	0.2811
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.005	0.297	14.30	-0.632 to 0.642	0.9871

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.10. 6 h $\log_{10} I_{\text{on}}/I_{\text{off}}$ ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-2.048	0.977	14.00	-4.143 to 0.048	0.0548
DDT PBS-Air	0.282	0.977	14.00	-1.813 to 2.378	0.7770
PTMS + DDT PBS-Air	-0.966	0.977	14.00	-3.061 to 1.130	0.3397
UV+PTMS + DDT PBS-Air	-0.757	0.977	14.00	-2.852 to 1.339	0.4517
Sequential DoD: DDT - Bare	2.330	1.382	14.00	-0.634 to 5.294	0.1139
Sequential DoD: PTMS + DDT - DDT	-1.248	1.382	14.00	-4.212 to 1.716	0.3817
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.209	1.382	14.00	-2.754 to 3.173	0.8817

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.11. 12 h $\log_{10} I_{\text{on}}/I_{\text{off}}$ ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-2.430	0.817	14.00	-4.182 to -0.678	0.0100
DDT PBS-Air	-0.867	0.817	14.00	-2.619 to 0.885	0.3066
PTMS + DDT PBS-Air	-1.577	0.817	14.00	-3.328 to 0.175	0.0741
UV+PTMS + DDT PBS-Air	-0.796	0.817	14.00	-2.548 to 0.956	0.3463
Sequential DoD: DDT - Bare	1.564	1.155	14.00	-0.914 to 4.041	0.1973
Sequential DoD: PTMS + DDT - DDT	-0.710	1.155	14.00	-3.187 to 1.768	0.5488
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.780	1.155	14.00	-1.697 to 3.258	0.5104

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.12. 6 h $\log_{10} \max |I_G|$ ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-0.217	0.237	14.00	-0.725 to 0.291	0.3746
DDT PBS-Air	0.236	0.237	14.00	-0.272 to 0.744	0.3365
PTMS + DDT PBS-Air	-0.185	0.237	14.00	-0.693 to 0.323	0.4468
UV+PTMS + DDT PBS-Air	0.281	0.237	14.00	-0.227 to 0.789	0.2551
Sequential DoD: DDT - Bare	0.453	0.335	14.00	-0.265 to 1.171	0.1977
Sequential DoD: PTMS + DDT - DDT	-0.421	0.335	14.00	-1.139 to 0.297	0.2292
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	0.466	0.335	14.00	-0.252 to 1.185	0.1855

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

Table E.3.13. 12 h $\log_{10}$ max $|I_G|$ ratio: Kenward–Roger supporting contrasts.

Comparison	Estimate	KR SE	KR ddf	95% KR CI	KR <i>P</i>
Bare PBS-Air	-0.397	0.295	14.00	-1.029 to 0.235	0.1995
DDT PBS-Air	0.199	0.295	14.00	-0.433 to 0.831	0.5112
PTMS + DDT PBS-Air	0.138	0.295	14.00	-0.494 to 0.770	0.6472
UV+PTMS + DDT PBS-Air	0.121	0.295	14.00	-0.511 to 0.753	0.6868
Sequential DoD: DDT - Bare	0.595	0.417	14.00	-0.298 to 1.489	0.1750
Sequential DoD: PTMS + DDT - DDT	-0.061	0.417	14.00	-0.955 to 0.833	0.8860
Sequential DoD: UV+PTMS + DDT - PTMS + DDT	-0.016	0.417	14.00	-0.910 to 0.877	0.9690

Note. Supporting model contrasts. PBS–Air rows compare exposure workflows within each surface state; sequential rows compare the PBS–Air effect across successive surface states. These supporting contrasts are interpreted separately from the prespecified primary 12 h Holm-adjusted contrast family unless otherwise stated in Chapter 5.

APPENDIX F

Exploratory Day 7 Electrical State

This appendix contains the separate exploratory Day 7 analysis obtained after the physical back-gate contact configuration changed. Day 7 values are absolute electrical states and are not treated as change from T0 or as a continuation of the standardized T0–12 h trajectory. Complete per-device Day 7 records for all 48 FETs are provided in Supplemental Data S3. Main-text Figure 5.4 is not repeated here; the tables below retain its numerical forward-threshold contrasts and add the supporting Day 7 outcomes.

F.1 Day 7 wafer-piece-level absolute summaries

Table F.1.1. Bare: Day 7 wafer-piece-level absolute means.

Day 7 metric	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
Forward V_{th} (V)	8.73 ± 2.85 (n=6)	11.4	7.76	7.05	15.4 ± 15 (n=2)	—	—	15.4
Reverse V_{th} (V)	13.4 ± 2.69 (n=6)	16	13.7	10.7	21.2 (n=1)	—	—	21.2
Hysteresis (V)	4.72 ± 1.8 (n=6)	4.59	5.95	3.62	16.4 (n=1)	—	—	16.4
$ I_{DS} $ at +30 V (μ A)	294 ± 97.3 (n=6)	225	255	403	20.4 ± 25.3 (n=6)	3.09	21.6	36.4
SS (V/dec)	0.862 ± 0.329 (n=6)	1.03	0.658	0.896	3.45 ± 2 (n=3)	3.63	—	3.36
I_{on}/I_{off}	$1.43e+07 \pm 9.90e+06$ (n=6)	9.25e+06	1.72e+07	1.64e+07	$7.38e+04 \pm 1.20e+05$ (n=6)	7.47e+03	8.82e+01	2.14e+05

Note. Day 7 values are absolute metrics under the alternative back-gate contact. Piece-level entries average the available FET subsamples on each represented wafer piece; they are descriptive only.

Table F.1.2. DDT: Day 7 wafer-piece-level absolute means.

Day 7 metric	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
Forward V_{th} (V)	5.36 ± 0.798 (n=6)	5.49	5.61	4.96	5.65 ± 3.86 (n=5)	3.92	8.53	3.63
Reverse V_{th} (V)	8.66 ± 1.04 (n=6)	9.73	7.83	8.43	23.3 ± 2.38 (n=5)	24.1	24.5	21.8
Hysteresis (V)	3.31 ± 1.09 (n=6)	4.24	2.22	3.47	17.7 ± 2.54 (n=5)	20.2	16	18.2
$ I_{DS} $ at +30 V (μA)	458 ± 48.8 (n=6)	449	502	422	89.2 ± 81 (n=6)	147	46.6	73.9
SS (V/dec)	0.981 ± 0.353 (n=6)	1.03	0.837	1.08	2.44 ± 0.753 (n=5)	1.69	2.9	2.36
I_{on}/I_{off}	$2.76e+07 \pm 2.11e+07$ (n=6)	2.41e+07	2.89e+07	2.98e+07	$1.29e+05 \pm 1.35e+05$ (n=6)	1.20e+05	3.76e+04	2.29e+05

Note. Day 7 values are absolute metrics under the alternative back-gate contact. Piece-level entries average the available FET subsamples on each represented wafer piece; they are descriptive only.

Table F.1.3. PTMS + DDT: Day 7 wafer-piece-level absolute means.

Day 7 metric	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
Forward V_{th} (V)	7.83 ± 1.77 (n=5)	8.56	6.86	8.29	14.3 ± 5.93 (n=5)	17.7	20.3	7.92
Reverse V_{th} (V)	14.5 ± 6.08 (n=6)	11.9	11.3	20.2	25.2 ± 2.06 (n=5)	25.9	27.9	23.2
Hysteresis (V)	4.19 ± 1.28 (n=5)	3.35	4.41	5.42	10.9 ± 4.06 (n=5)	8.24	7.65	15.3
$ I_{DS} $ at +30 V (μA)	240 ± 153 (n=6)	308	362	50	37.5 ± 28.7 (n=6)	24.7	17.6	70.1
SS (V/dec)	0.891 ± 0.266 (n=6)	1.09	0.949	0.629	2.38 ± 0.663 (n=6)	2.1	3.08	1.96
I_{on}/I_{off}	$7.72e+06 \pm 8.96e+06$ (n=6)	1.59e+07	6.70e+06	6.04e+05	$2.02e+05 \pm 2.95e+05$ (n=6)	4.44e+05	1.22e+04	1.51e+05

Note. Day 7 values are absolute metrics under the alternative back-gate contact. Piece-level entries average the available FET subsamples on each represented wafer piece; they are descriptive only.

Table F.1.4. UV + PTMS + DDT: Day 7 wafer-piece-level absolute means.

Day 7 metric	Air mean	Air W1	Air W2	Air W3	PBS mean	PBS W1	PBS W2	PBS W3
Forward V_{th} (V)	7.45 ± 2 (n=6)	9.21	8.02	5.13	6.54 ± 3.16 (n=2)	6.54	—	—
Reverse V_{th} (V)	10.6 ± 2.08 (n=6)	13	10.2	8.6	13.6 ± 2.29 (n=2)	13.6	—	—
Hysteresis (V)	3.14 ± 1.37 (n=6)	3.81	2.13	3.47	7.07 ± 0.872 (n=2)	7.07	—	—
$ I_{DS} $ at +30 V (μ A)	503 ± 93.8 (n=6)	413	481	614	288 ± 367 (n=6)	240	112	513
SS (V/dec)	1.78 ± 0.451 (n=6)	1.41	1.78	2.16	3.65 ± 4.15 (n=3)	1.25	8.44	—
I_{on}/I_{off}	$1.20e+07 \pm 9.35e+06$ (n=6)	$1.57e+07$	$1.95e+07$	$6.16e+05$	$2.23e+06 \pm 3.55e+06$ (n=6)	$6.69e+06$	$2.67e+02$	$1.61e+02$

Note. Day 7 values are absolute metrics under the alternative back-gate contact. Piece-level entries average the available FET subsamples on each represented wafer piece; they are descriptive only.

Table F.2. Day 7 operational compatibility and metric availability.

Surface	Exposure	FET n	Forward V_{th} extractable	Reverse V_{th} extractable	Interpretable	Saturation-like output	sqrt V_{th} reportable
Bare	Air	6	6/6	6/6	6/6	6/6	6/6
Bare	PBS	6	2/6	1/6	1/6	4/6	3/6
DDT	Air	6	6/6	6/6	6/6	6/6	6/6
DDT	PBS	6	5/6	5/6	5/6	4/6	4/6
PTMS + DDT	Air	6	5/6	6/6	5/6	6/6	4/6
PTMS + DDT	PBS	6	5/6	5/6	5/6	5/6	4/6
UV+PTMS + DDT	Air	6	6/6	6/6	6/6	5/6	5/6
UV+PTMS + DDT	PBS	6	2/6	2/6	2/6	4/6	3/6

Note. Counts are descriptive FET-level Day 7 outcomes under the alternative contact. Gate-current and R_{IG} outcomes are not included because the Day 7 gate-current channel was not considered reliable for leakage interpretation.

Table F.3. Day 7 condition means overview.

Surface	Metric	Day 7 Air	Day 7 PBS
Bare	Forward V_{th} (V)	8.73 ± 2.85 (n=6)	15.4 ± 15 (n=2)
Bare	Reverse V_{th} (V)	13.4 ± 2.69 (n=6)	21.2 (n=1)
Bare	Hysteresis (V)	4.72 ± 1.8 (n=6)	16.4 (n=1)
Bare	$ I_{DS} $ at +30 V (μA)	294 ± 97.3 (n=6)	20.4 ± 25.3 (n=6)
Bare	SS (V/dec)	0.862 ± 0.329 (n=6)	3.45 ± 2 (n=3)
Bare	I_{on}/I_{off}	$1.43e+07 \pm 9.90e+06$ (n=6)	$7.38e+04 \pm 1.20e+05$ (n=6)
DDT	Forward V_{th} (V)	5.36 ± 0.798 (n=6)	5.65 ± 3.86 (n=5)
DDT	Reverse V_{th} (V)	8.66 ± 1.04 (n=6)	23.3 ± 2.38 (n=5)
DDT	Hysteresis (V)	3.31 ± 1.09 (n=6)	17.7 ± 2.54 (n=5)
DDT	$ I_{DS} $ at +30 V (μA)	458 ± 48.8 (n=6)	89.2 ± 81 (n=6)
DDT	SS (V/dec)	0.981 ± 0.353 (n=6)	2.44 ± 0.753 (n=5)
DDT	I_{on}/I_{off}	$2.76e+07 \pm 2.11e+07$ (n=6)	$1.29e+05 \pm 1.35e+05$ (n=6)
PTMS + DDT	Forward V_{th} (V)	7.83 ± 1.77 (n=5)	14.3 ± 5.93 (n=5)
PTMS + DDT	Reverse V_{th} (V)	14.5 ± 6.08 (n=6)	25.2 ± 2.06 (n=5)
PTMS + DDT	Hysteresis (V)	4.19 ± 1.28 (n=5)	10.9 ± 4.06 (n=5)
PTMS + DDT	$ I_{DS} $ at +30 V (μA)	240 ± 153 (n=6)	37.5 ± 28.7 (n=6)
PTMS + DDT	SS (V/dec)	0.891 ± 0.266 (n=6)	2.38 ± 0.663 (n=6)
PTMS + DDT	I_{on}/I_{off}	$7.72e+06 \pm 8.96e+06$ (n=6)	$2.02e+05 \pm 2.95e+05$ (n=6)
UV+PTMS + DDT	Forward V_{th} (V)	7.45 ± 2 (n=6)	6.54 ± 3.16 (n=2)
UV+PTMS + DDT	Reverse V_{th} (V)	10.6 ± 2.08 (n=6)	13.6 ± 2.29 (n=2)
UV+PTMS + DDT	Hysteresis (V)	3.14 ± 1.37 (n=6)	7.07 ± 0.872 (n=2)
UV+PTMS + DDT	$ I_{DS} $ at +30 V (μA)	503 ± 93.8 (n=6)	288 ± 367 (n=6)
UV+PTMS + DDT	SS (V/dec)	1.78 ± 0.451 (n=6)	3.65 ± 4.15 (n=3)
UV+PTMS + DDT	I_{on}/I_{off}	$1.20e+07 \pm 9.35e+06$ (n=6)	$2.23e+06 \pm 3.55e+06$ (n=6)

Note. Values are absolute Day 7 condition means $\pm$ SD with available n shown. They are presented for exploratory cross-sectional context only and are not changes from T0.

Table F.4. Day 7 exploratory model availability.

Outcome	FET n	Piece n	Converged	Model note
Forward V_{th} (V)	37	20	Yes	—
Reverse V_{th} (V)	37	20	Yes	—
Hysteresis (V)	36	20	Yes	—
$\log_{10} I_{DS} $ at +30 V	48	24	Yes	Singular matrix
$\log_{10}$ SS	41	22	Yes	Singular matrix
$\log_{10} I_{on}/I_{off}$	48	24	Yes	—

Note. Model availability reflects the metric-specific valid Day 7 observations. Singular or unstable fits are retained as analysis-status information and limit interpretation of affected supporting outcomes.

Table F.5. Day 7 forward-threshold exploratory contrasts.

Contrast type	Contrast	Estimate (V)	SE	95% CI	<i>p</i>	Holm <i>p</i> *
PBS-Air within surface	Bare	9.00	3.48	[2.18, 15.81]	0.009663	—
PBS-Air within surface	DDT	0.55	2.57	[-4.49, 5.60]	0.8304	—
PBS-Air within surface	PTMS + DDT	7.12	2.61	[2.01, 12.24]	0.006298	—
PBS-Air within surface	UV+PTMS + DDT	-2.34	3.51	[-9.22, 4.54]	0.505	—
Sequential DoD	DDT - Bare	-8.45	4.27	[-16.82, -0.07]	0.04814	0.09629
Sequential DoD	PTMS + DDT - DDT	6.57	3.73	[-0.74, 13.89]	0.07812	0.09629
Sequential DoD	UV+PTMS + DDT - PTMS + DDT	-9.46	4.33	[-17.96, -0.97]	0.02896	0.08687

Note. Exploratory forward-threshold model. Holm adjustment applies across the three sequential Surface × Exposure contrasts only; the analysis is not a second confirmatory longitudinal test.

Table F.6. Day 7 reverse-threshold exploratory contrasts.

Contrast type	Contrast	Estimate	SE	95% CI	<i>p</i>
PBS-Air within surface	Bare	8.26	3.97	[0.475, 16.1]	0.0376
PBS-Air within surface	DDT	14.8	2.45	[10, 19.6]	1.53e-09
PBS-Air within surface	PTMS + DDT	11	2.46	[6.14, 15.8]	8.14e-06
PBS-Air within surface	UV+PTMS + DDT	2.24	3.52	[-4.66, 9.14]	0.524
Sequential difference-in-differences	DDT - Bare	6.55	4.66	[-2.58, 15.7]	0.16
Sequential difference-in-differences	PTMS + DDT - DDT	-3.86	3.47	[-10.7, 2.95]	0.266
Sequential difference-in-differences	UV+PTMS + DDT - PTMS + DDT	-8.71	4.28	[-17.1, -0.324]	0.0418

Note. Supporting exploratory Day 7 contrasts. These are cross-sectional absolute-metric models under the alternative back-gate contact and are not interpreted as longitudinal T0-to-Day 7 change.

Table F.7. Day 7 signed-hysteresis exploratory contrasts.

Contrast type	Contrast	Estimate	SE	95% CI	<i>p</i>
PBS-Air within surface	Bare	10.8	2.69	[5.49, 16]	6.37e-05
PBS-Air within surface	DDT	14.6	1.7	[11.3, 18]	8.33e-18
PBS-Air within surface	PTMS + DDT	6.16	1.74	[2.76, 9.56]	0.00039
PBS-Air within surface	UV+PTMS + DDT	3.95	2.45	[-0.86, 8.76]	0.107
Sequential difference-in-differences	DDT - Bare	3.86	3.18	[-2.37, 10.1]	0.224
Sequential difference-in-differences	PTMS + DDT - DDT	-8.47	2.43	[-13.2, -3.7]	0.000504
Sequential difference-in-differences	UV+PTMS + DDT - PTMS + DDT	-2.21	3.01	[-8.1, 3.68]	0.463

Note. Supporting exploratory Day 7 contrasts. These are cross-sectional absolute-metric models under the alternative back-gate contact and are not interpreted as longitudinal T0-to-Day 7 change.

Table F.8. Day 7 log₁₀ fixed-bias $|I_{DS}|$ exploratory contrasts.

Contrast type	Contrast	Estimate	SE	95% CI	<i>p</i>
PBS-Air within surface	Bare	-1.68	0.418	[-2.5, -0.859]	5.88e-05
PBS-Air within surface	DDT	-0.821	0.418	[-1.64, -0.00263]	0.0493
PBS-Air within surface	PTMS + DDT	-0.673	0.418	[-1.49, 0.145]	0.107
PBS-Air within surface	UV+PTMS + DDT	-1.02	0.418	[-1.84, -0.205]	0.0142
Sequential difference-in-differences	DDT - Bare	0.857	0.591	[-0.301, 2.01]	0.147
Sequential difference-in-differences	PTMS + DDT - DDT	0.148	0.591	[-1.01, 1.31]	0.802
Sequential difference-in-differences	UV+PTMS + DDT - PTMS + DDT	-0.351	0.591	[-1.51, 0.807]	0.553

Note. Supporting exploratory Day 7 contrasts. These are cross-sectional absolute-metric models under the alternative back-gate contact and are not interpreted as longitudinal T0-to-Day 7 change.

Table F.9. Day 7 log₁₀ SS exploratory contrasts.

Contrast type	Contrast	Estimate	SE	95% CI	<i>p</i>
PBS-Air within surface	Bare	0.601	0.166	[0.276, 0.925]	0.000288
PBS-Air within surface	DDT	0.397	0.142	[0.119, 0.675]	0.00516
PBS-Air within surface	PTMS + DDT	0.43	0.137	[0.161, 0.698]	0.00173
PBS-Air within surface	UV+PTMS + DDT	0.186	0.17	[-0.148, 0.519]	0.275
Sequential difference-in-differences	DDT - Bare	-0.204	0.219	[-0.632, 0.225]	0.351
Sequential difference-in-differences	PTMS + DDT - DDT	0.0327	0.197	[-0.354, 0.419]	0.869
Sequential difference-in-differences	UV+PTMS + DDT - PTMS + DDT	-0.244	0.219	[-0.672, 0.184]	0.265

Note. Supporting exploratory Day 7 contrasts. These are cross-sectional absolute-metric models under the alternative back-gate contact and are not interpreted as longitudinal T0-to-Day 7 change.

Table F.10. Day 7 $\log_{10} I_{\text{on}}/I_{\text{off}}$ exploratory contrasts.

Contrast type	Contrast	Estimate	SE	95% CI	<i>p</i>
PBS-Air within surface	Bare	-3.8	1.14	[-6.04, -1.56]	0.000899
PBS-Air within surface	DDT	-2.38	1.14	[-4.62, -0.138]	0.0375
PBS-Air within surface	PTMS + DDT	-1.8	1.14	[-4.04, 0.441]	0.115
PBS-Air within surface	UV+PTMS + DDT	-3.25	1.14	[-5.5, -1.01]	0.00447
Sequential difference-in-differences	DDT - Bare	1.42	1.62	[-1.75, 4.59]	0.381
Sequential difference-in-differences	PTMS + DDT - DDT	0.579	1.62	[-2.59, 3.75]	0.721
Sequential difference-in-differences	UV+PTMS + DDT - PTMS + DDT	-1.45	1.62	[-4.62, 1.72]	0.37

Note. Supporting exploratory Day 7 contrasts. These are cross-sectional absolute-metric models under the alternative back-gate contact and are not interpreted as longitudinal T0-to-Day 7 change.

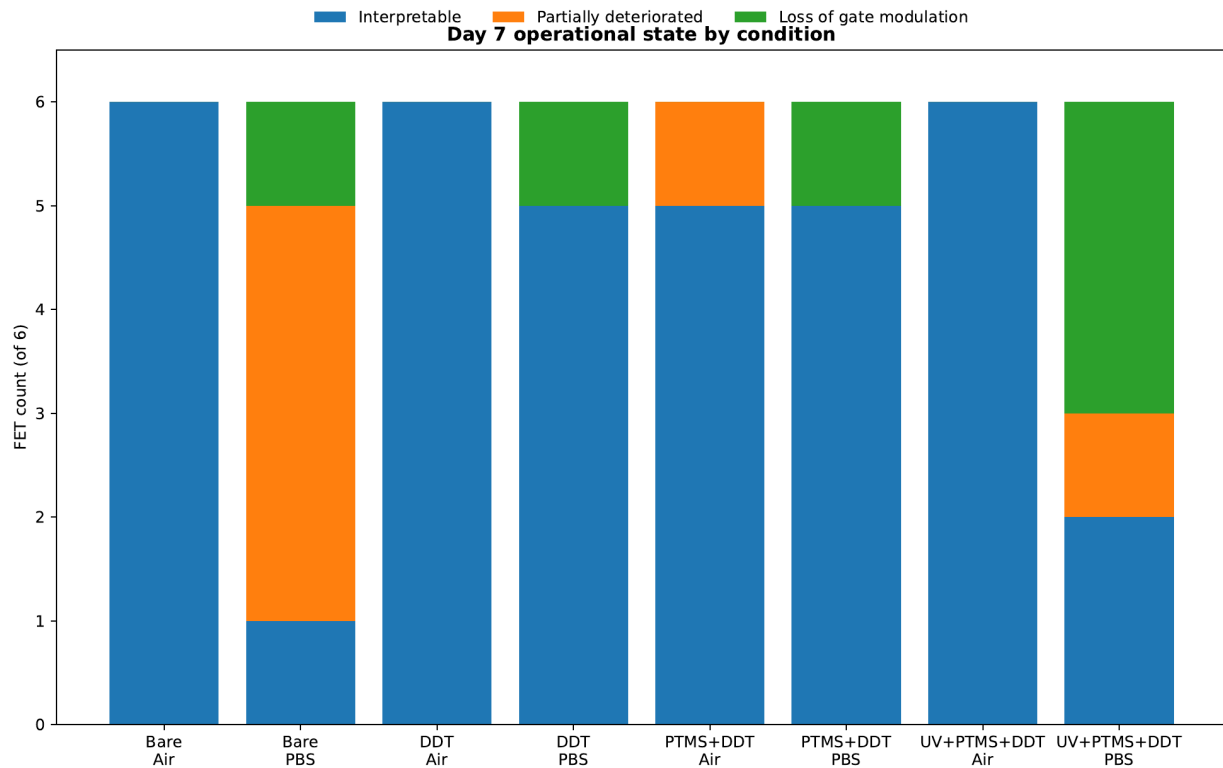


Figure F.1. Day 7 operational state by condition. Stacked counts summarize interpretable, partially deteriorated, and loss-of-gate-modulation states across the eight Surface $\times$ Exposure conditions at Day 7. Blue denotes interpretable FETs, orange partially deteriorated FETs, and green FETs with loss of gate modulation; bar heights are device counts out of six within each Surface $\times$ Exposure condition.

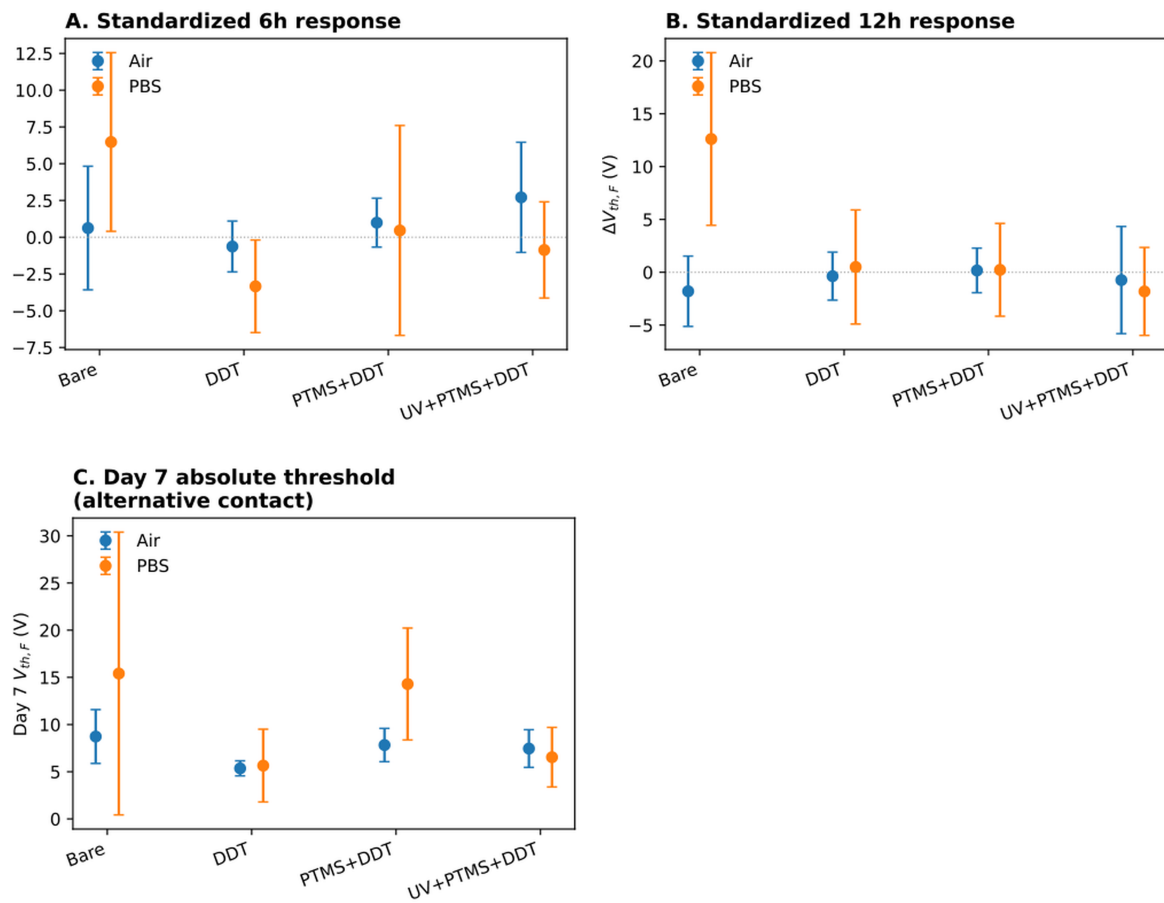


Figure F.2. Condition means across the standardized early phase and separate Day 7 exploratory phase. (A) Standardized 6 h forward-threshold response by surface-treatment condition under Air and PBS exposure. (B) Standardized 12 h forward-threshold response for the same conditions. (C) Absolute Day 7 forward threshold under the alternative physical back-gate contact. Panels A and B show within-FET change from T0. Panel C is an absolute Day 7 metric under a different physical back-gate contact and is shown for visual context only; it is not connected as a longitudinal trajectory. Blue points denote Air and orange points denote PBS. Points are descriptive condition means and vertical error bars show ± 1 SD using the available FETs for each condition and timepoint.

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