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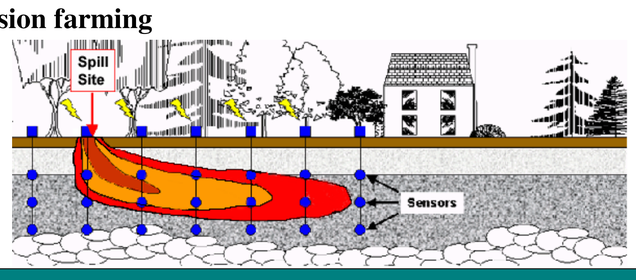
A High-Performance Micromachined Amperometric Nitrate Sensor for Environmental Monitoring

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Introduction: Why a Micromachined Amperometric Nitrate Sensor?

Why Nitrate (NO_3^-) Sensor Important?

- Nitrate is a major contaminant in ground water
- Nitrate-sensor applications
  - In-situ nitrate monitoring, environmental science/biology research, and precision farming



Electrochemical Methods

- Sensor requirements
  - Inexpensive, small, remotely operable, detection range (1 µM to 1 mM)
- Electrochemical techniques meet the requirements
  - Amperometry and Potentiometry
  - Simple design and operation
  - Easily minaturized
  - Low power & voltage
  - Sensitive



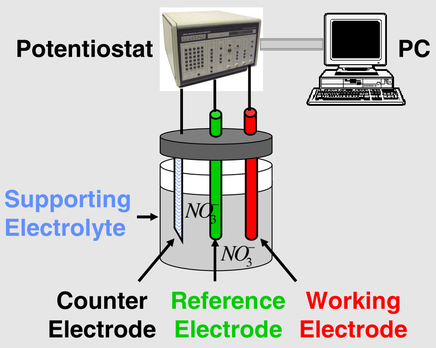
Glucose Sensor

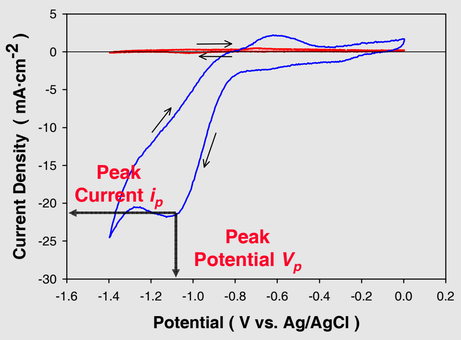


Ion-Selective Electrode

Working Principle: Electroanalytic Chemistry and Anion Permeable Membrane

Electrochemical Study





- Electrochemical reduction of nitrate: $NO_3^- + H_2O + 2e^- \rightarrow NO_2^- + 2OH^-$
- NaOH supporting electrolyte, working electrode (Ag), reference electrode (Ag/AgCl), counter electrode (Pt)

Removing Oxygen Interference

- Oxygen in electrolyte (≅ 0.26 mM) interferes with detection
- Nitrate reduction current = (nitrate + oxygen reduction current) – (oxygen reduction current)

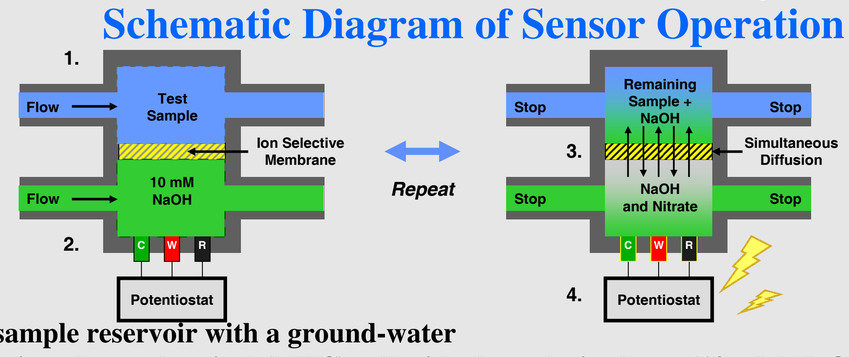
Anion Permeable Membrane

- Ground water contains many ionic/non-ionic species interfering nitrate measurement
- Selective measurement with an anion selective membrane
  - Anion-permeable membrane blocks all cations
  - Nitrate diffuse faster than the other interfering ions



TokuyamaACS Membrane

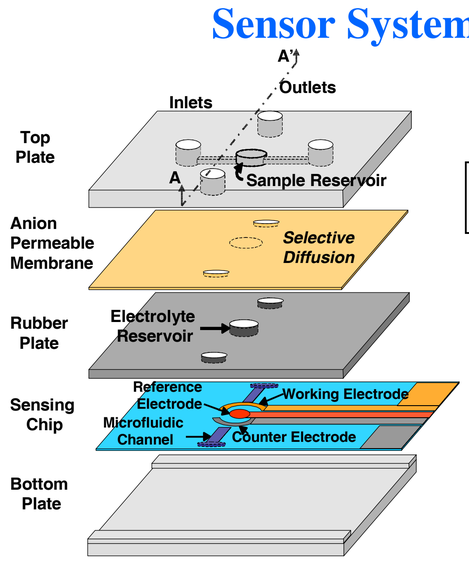
Schematic Diagram of Sensor Operation

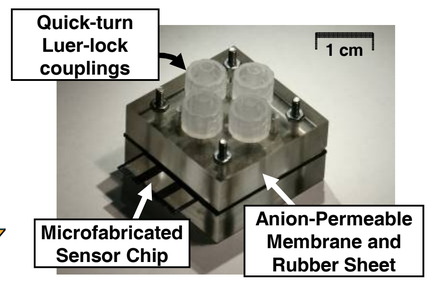


1. Fill sample reservoir with a ground-water
2. Fill microelectrochemical (MEC) cell with electrolytic eluent (10 mM NaOH)
3. OH^- and NO_3^- diffuse across anion-permeable membrane in opposite directions
4. Perform measurements when concentrations reach equilibrium

Design, Fabrication, Experimental Results, and Summary

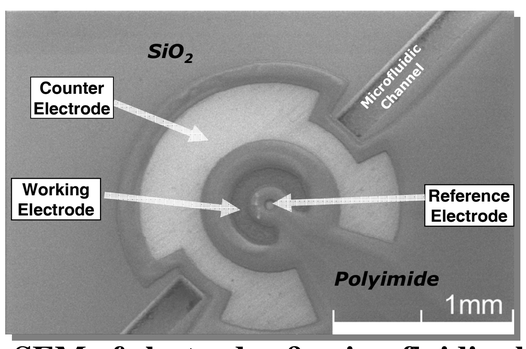
Sensor System Integration





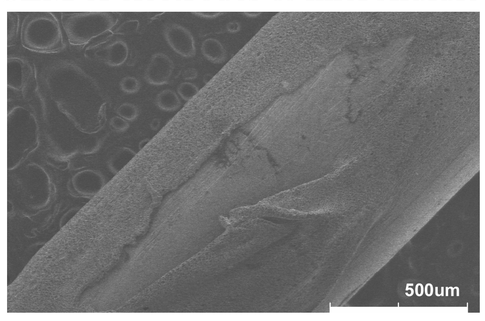
Assembled Sensor Unit

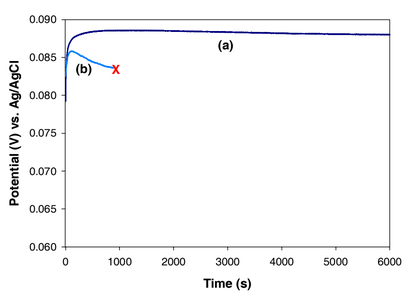
Micromachined Nitrate Sensing Chip



- Pt counter electrode (7.7x 10^{-3} cm^2)
- Ag working electrode (0.7×10^{-3} cm^2)
- Silver oxide reference electrode (0.038×10^{-3} cm^2)
- polyimide insulation layer

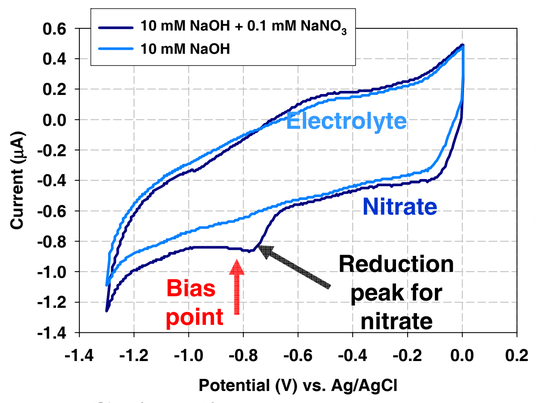
SEM of electrodes & microfluidic channel

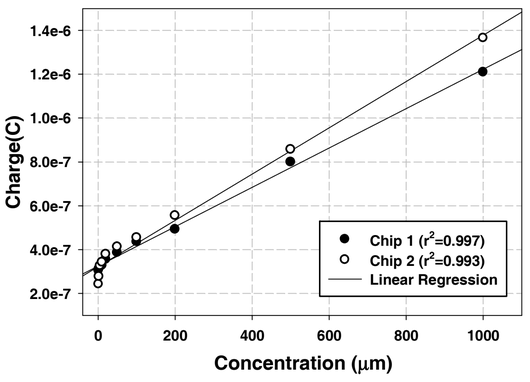




SEM of *polyurethane*-coated Ag/AgCl reference electrode and its potential drift (a) in comparison to silver oxide electrode (b). The minimal potential drift observed (3 mV for 27 hours)

Experiment: Calibration Curves and Sensor Selectivity





- Calibration curves
  - 1 to 1000 µM detection range
  - r^2=0.99 linearty
- Detection limit: ~1 µM
- Sensor selectivity to interfering ions
  - Sensor output to a mixture of 100-µM nitrate and interfering ions (100 µM each of PO_4^{2-}, SO_4^{3-}, F^-, Cl^-) is only 13.9% higher than the average response for the sample consisting of nitrate

Summary and Future Work

- Electrochemical study proves that amperometric nitrate sensors are feasible with Ag working electrode and NaOH supporting electrolyte
- Integrated micromachined nitrate sensor units have been fabricated
  - Micromachined electrodes and fluidic channels
  - Anion-permeable membrane
- Achieved a detection limit of ~1 µM and linear calibration curves
- Future work
  - Integration of PU-coated Ag/AgCl reference electrode
  - Long-term qualification test
  - Integrate into wireless sensor motes and network
  - Explore chip-scale and board-scale potentiostats for miniaturizaion
  - Field test