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Data Article

Dataset for SO₂, SO₃, H₂SO₄ and H₂O infrared absorption spectra at 300° C and 350° C temperatures



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ABSTRACT

The data presented here consists of library spectra obtained for use with a laser absorption spectroscopy gas sensor. The spectra include absorbance data for SO₂, SO₃, H₂O and H₂SO₄ at 300° C and 350° C temperatures in two wavelength bands, 7–8 μm and 8–9 μm. Datasets were collected in a heated multi-pass absorption Herriott cell using two tunable external cavity quantum cascade laser sources, with the resulting transmission signal measured using a thermoelectrically cooled MCT detector. The absorbance was calculated from measurements taken with and without gas samples and scaled for the length of the multi-pass cell. The data will be useful for scientists and engineers building SO₃ and H₂SO₄ gas sensing equipment for emission monitoring, process control, and other applications.

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Specifications Table

Subject	Energy Engineering and Power Technology
Specific subject area	The work presented here is in an area of gas detection and emissions monitoring for power generation and heavy industry.
Type of data	Table Graph
How the data were acquired	Data sets [1] were acquired utilizing a direct absorption spectroscopy method. A specially built apparatus was used for the experimental data collection, which consisted of catalyst, heated multi-pass absorption cell, controlled condensation system, flow control and two custom tunable external cavity quantum cascade lasers (EC-QCLs). The two EC-QCLs were operated in two different spectral regions: 7–8 μm and 8–9 μm . The design and implementation of the gas delivery system and absorption cell apparatus were performed by UCI [2]. The laser systems were designed and build by OKSI [3]. All software for data collection and data processing was produced by OKSI.
Data format	Raw
Description of data collection	Absorption spectra at 300 °C cell temperature were acquired for SO ₂ , H ₂ O and H ₂ SO ₄ species in the 8–9 μm laser tuning range. Spectra for 350 °C temperature were acquired for SO ₂ , SO ₃ and H ₂ O species in the 7–8 μm laser tuning range. All data collections were done at ambient pressure. Absorbance for each species was calculated based on the absorption path length and the estimated species concentrations.
Data source location	<ul style="list-style-type: none"> • Institution: University of California, Irvine (UCI) • City/Town/Region: Irvine, CA • Country: USA
Data accessibility	Repository name: EDX NETL's Data eXchange (EDX) Data identification number: 10.18141/1899775 Direct URL to data: https://edx.netl.doe.gov/dataset/infrared-absorption-spectra-of-h2so4-so3-so2-and-h2o-at-300c-and-350c

Value of the Data

- The data sets presented are library spectra for chemicals present in a flue gas of coal-fired power plants, carbon black facilities, and other facilities in which sulfur is present in high temperature processes. Monitoring of these chemicals will allow cleaner operation, lower costs and longer life of facility components [4].
- Researchers and companies developing flue gas monitoring systems for heavy industry including coal-fired power plants and carbon black process will benefit from the data. The spectra presented were acquired under conditions relevant to combustion and other high temperature applications.
- The data sets provide SO₃ and H₂SO₄ spectra under conditions which are not present in any spectra data bases known to us. Knowledge of absorbance of those elements as well as accompanying H₂O and SO₂ spectra can enable development of process control instrumentation as well as environmental monitoring systems for a range of industries including but not limited to coal-fired power production.

1. Objective

The main objective for generating the datasets was to provide library spectra that would enable quantification of SO₃ and H₂SO₄. Curve fitting algorithms for broad absorption spectra require accurate library spectra to produce high accuracy fits and to calculate concentrations of the gas constituents in a mixture. For that reason, the experimental conditions for the dataset were defined based on targeted conditions in coal-fired power plant flue gas including composition, pressure and temperature. The described dataset was successfully used for the simultaneous real-time detection of H₂SO₄, SO₂ and H₂O, in-situ at a operating coal fired power plant. Over-

all, the datasets provide the libraries for simultaneous multi-species monitoring for a variety of high temperature applications.

2. Data Description

Data presented below represents measured infrared spectra of SO_3 (Fig. 1), SO_2 (Fig. 2 and Fig. 5), H_2SO_4 (Fig. 4) and H_2O (Fig. 3 and Fig. 6). Data for H_2SO_4 , SO_2 and H_2O for 300°C temperature was acquired in the $\lambda=8\text{--}8.8\ \mu\text{m}$ wavelength range. Also, data for SO_3 , SO_2 and H_2O for 350°C temperature was acquired in the $\lambda=7\text{--}7.7\ \mu\text{m}$ wavelength range. SO_3 and H_2SO_4 in flue gas exist in a state of equilibrium. Lower temperature and higher humidity levels favour H_2SO_4 while higher temperature favours SO_3 . For that reason we are reporting spectra for SO_3 at higher temperature than H_2SO_4 .

The absorbance was calculated from the measurements performed with and without the gas sample inside the absorption cell according to the Beer-Lambert law. Each figure below has a description of the spectra and a list of corresponding files uploaded to the EDX repository.

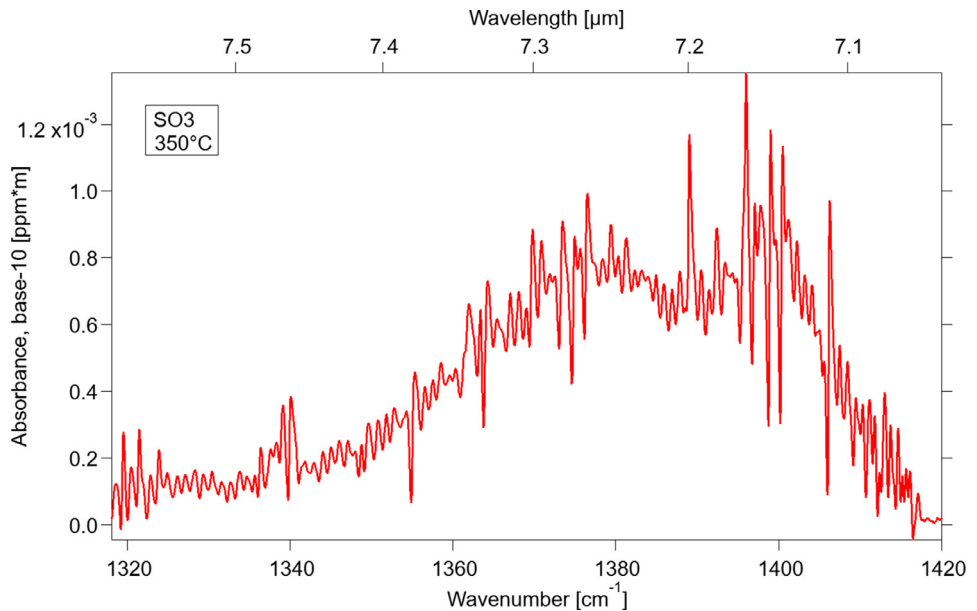


Fig. 1. Infrared absorption spectrum of Sulfur Trioxide (SO_3) taken at a temperature of 350°C in the wavelength range of 7 to 7.6 microns ($1318\text{--}1420\ \text{cm}^{-1}$). Image file: `so3_350c_7 um.pdf`; data file: `so3_350c_7 um.txt`.

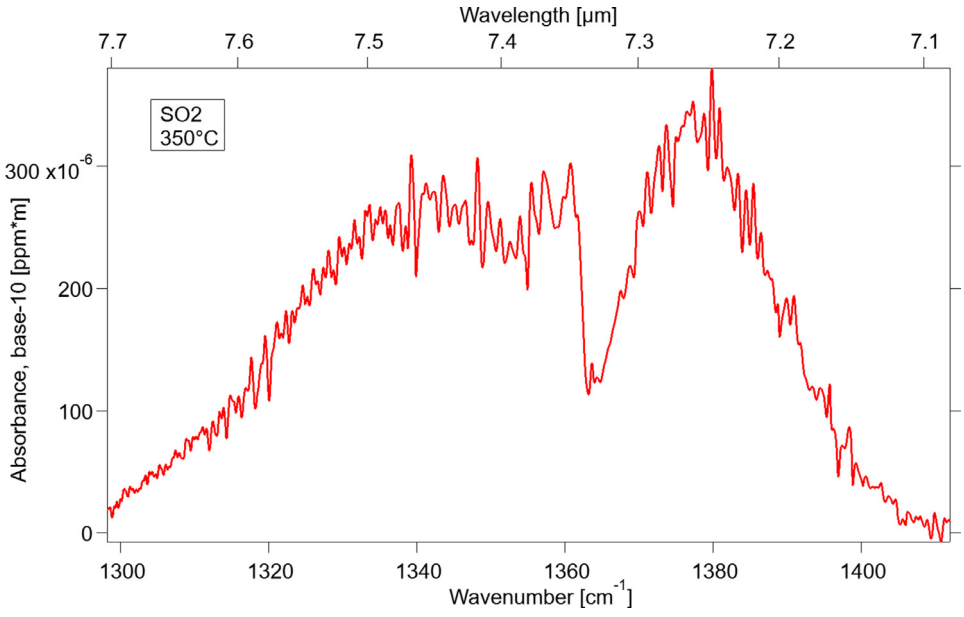


Fig. 2. Infrared absorption spectrum of Sulfur Dioxide (SO₂) taken at a temperature of 350° C in the wavelength range of 7 to 7.7 microns (1298-1412 cm⁻¹). Image file: so2_350c_7 um.pdf; data file: so2_350c_7 um.txt.

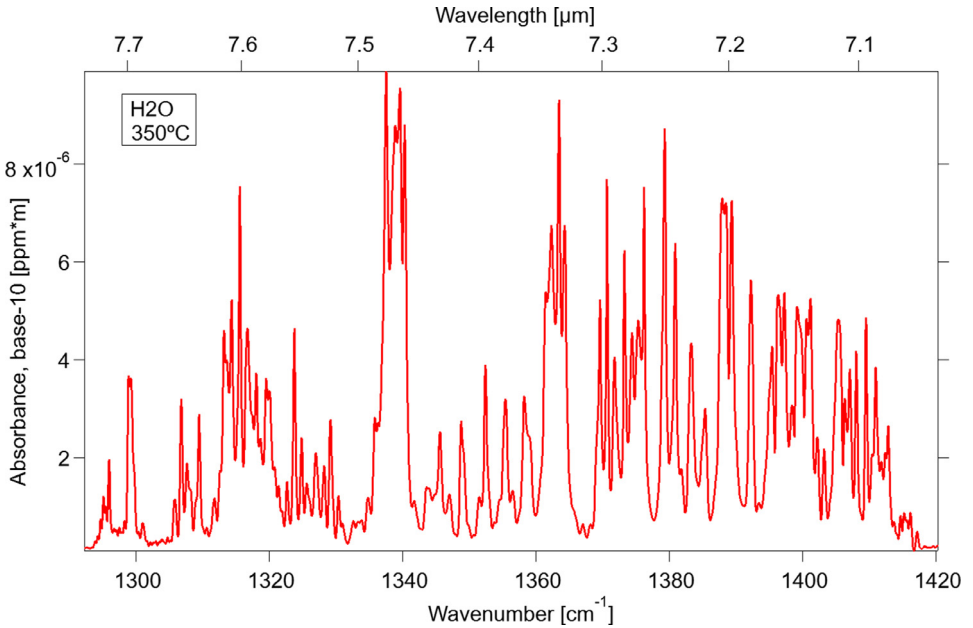


Fig. 3. Infrared absorption spectrum of water (H₂O) taken at a temperature of 350° C in the wavelength range of 7 to 7.7 microns (1292-1420 cm⁻¹). Image file: h2o_350c_7 um.pdf; data file: h2o_350c_7 um.txt.

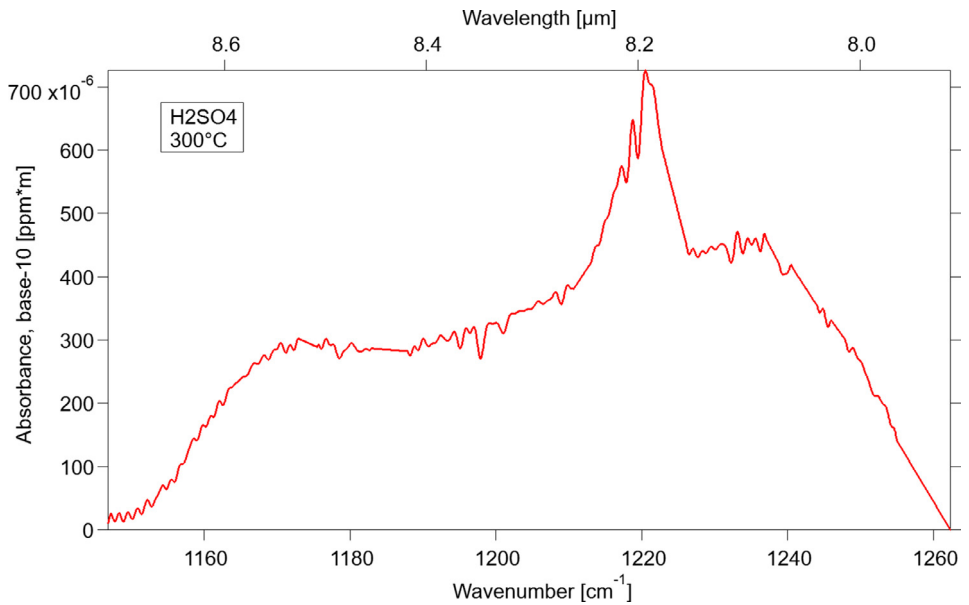


Fig. 4. Infrared absorption spectrum of Sulfuric Acid (H_2SO_4) taken at a temperature of 300°C in the wavelength range of 7.9 to 8.7 microns ($1147\text{--}1262\text{ cm}^{-1}$). Image file: h2so4_300c_8 um.pdf; data file: h2so4_300c_8 um.txt.

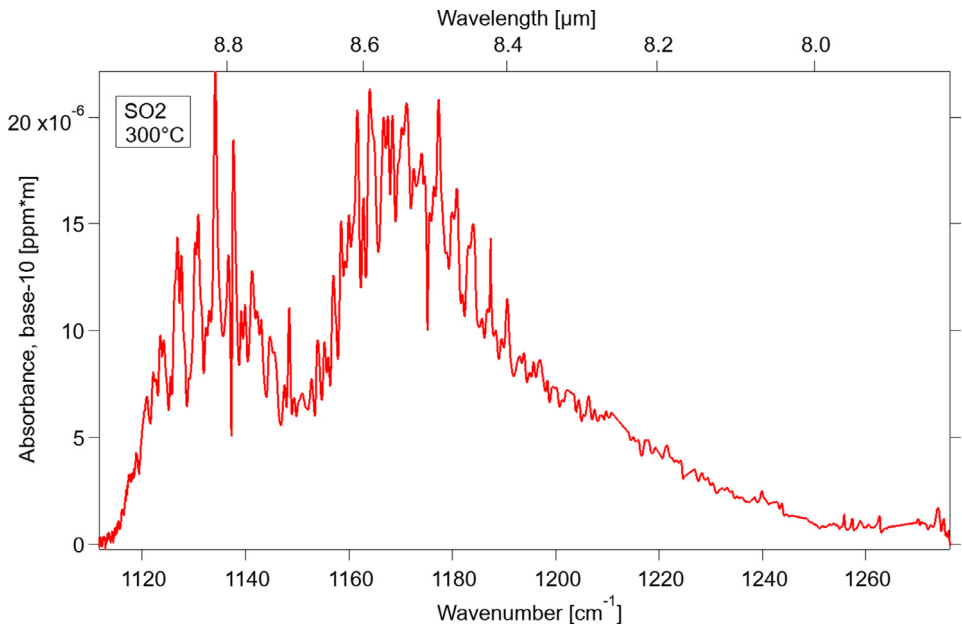


Fig. 5. Infrared absorption spectrum of Sulfur Dioxide (SO_2) taken at a temperature of 300°C in the wavelength range of 7.8 to 9 microns ($1112\text{--}1276\text{ cm}^{-1}$). Image file: so2_300c_8 um.pdf; data file: so2_300c_8 um.txt.

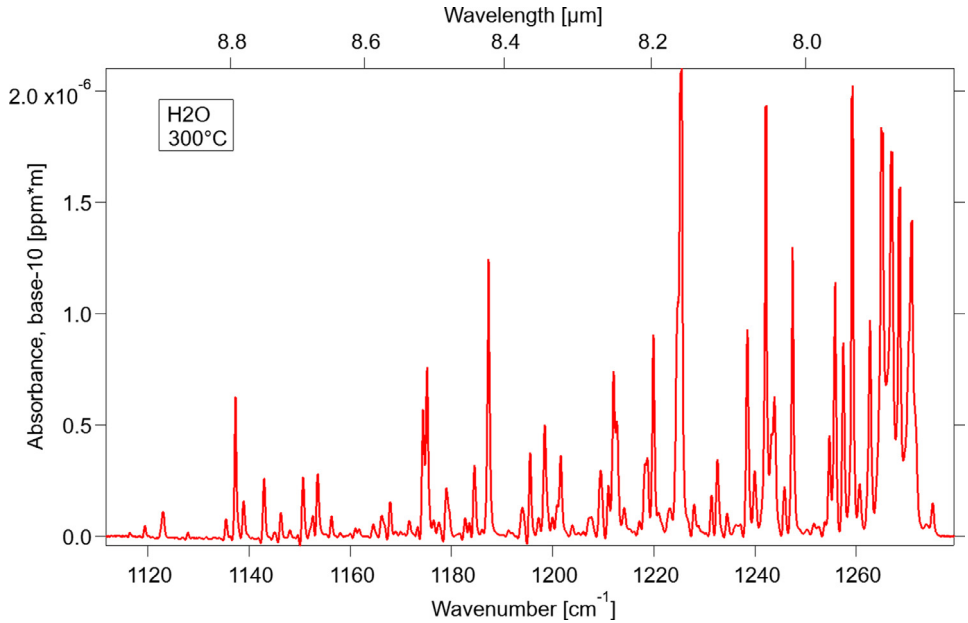


Fig. 6. Infrared absorption spectrum of water (H₂O) spectrum taken at a temperature of 300 °C in the wavelength range of 7.8 to 9 microns (1112-1279 cm⁻¹). Image file: h2o_300c_8 um.pdf; data file: h2o_300c_8 um.txt.

3. Experimental Design, Materials and Methods

The experimental setup was assembled at the University of California, Irvine (UCI) to perform the measurements. The schematic of the experimental apparatus is depicted in Fig. 7. Mechanically the setup consists of a VK58 catalyst bed reactor which converts SO₂ into SO₃, multi-pass

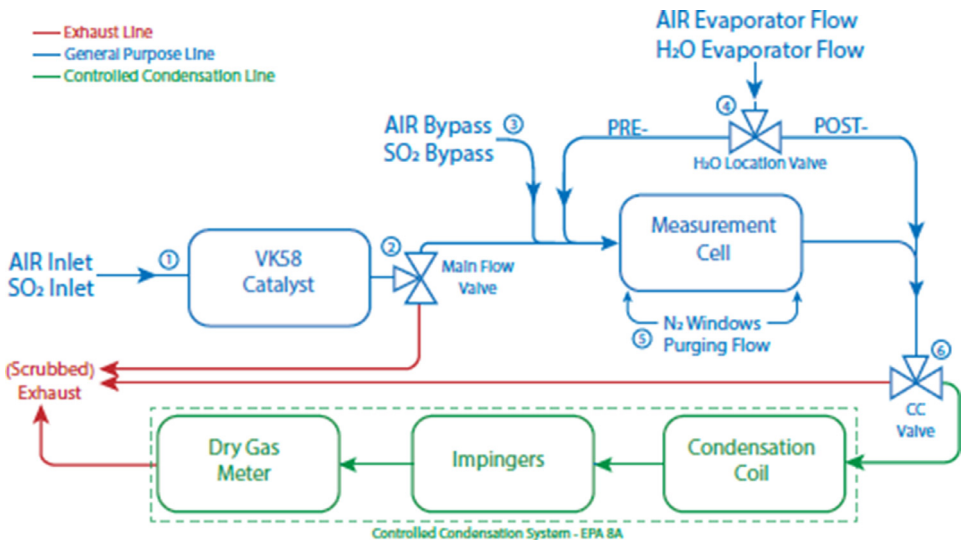


Fig. 7. Experimental apparatus for the high temperature SO₂, SO₃, H₂SO₄ and H₂O measurements [2].

measurement cell, evaporator which produces air flow with controlled humidity and controlled condensation system for industry standard EPA Method 8A measurements of SO_3 and H_2SO_4 combined. Also, there are three major valves in the system. The main flow valve directs gas flow from the catalyst either into the measurement cell or into the scrubber and exhaust. The H_2O location valve switches humidity either into the cell or after the cell depending on the purpose of the performed measurement. The CC valve switches the cell output flow either through the controlled condensation setup or directly into the scrubber and the exhaust. The Air/ SO_2 bypass line serves to provide either dilution with air flow or calibrated SO_2 flow into the cell. All components of the assembly that are in contact with gas flow are made of 316 L stainless steel and coated with Silconert2000 coating to minimize corrosion and loss of sulfur at the surfaces. Also, all the components of the measurement setup are wrapped with heaters and operated at elevated temperatures to avoid any gas condensation during the experiment. Temperature of all the gas delivery lines was kept stable at 400°C for measurements conducted with a 350°C cell temperature and at 330°C for measurements conducted with a 300°C cell temperature. The temperature of the catalyst bed reactor was kept at 475°C . Temperatures were regulated by temperature controllers and monitored with numerous thermocouples installed across the experimental apparatus.

The optical setup for the multi-pass Herriott absorption cell consisted of two gold mirrors installed on an optical bench on both sides, but outside of the measurement cell. One mirror had a single off-axis through hole for the input and output beams. A laser beam bounces back and forth between the two mirrors 16 times with an effective path length of $L = 5.6\text{ m}$. Two broadly tunable external cavity quantum cascade laser (EC-QCL) systems operating around $\lambda = 7\ \mu\text{m}$ and $\lambda = 8\ \mu\text{m}$ wavelength bands were installed and aligned one at a time. Both lasers are proprietary EC-QCL systems custom built by Opto-Knowledge Systems Inc. Uncoated BaF_2 windows were used on the absorption cell to ensure transmission in both $\lambda = 7\ \mu\text{m}$ and $\lambda = 8\ \mu\text{m}$ wavelength bands. The Herriott cell output beam was aligned and focused onto a cooled Vigo MCT detector. Data was acquired with a data acquisition board integrated inside an EC-QCL laser control system.

For SO_3 measurements, SO_2 gas was flowed through the catalyst bed reactor at 5 slm rate. The catalyst oxidized a portion of the SO_2 gas flow into SO_3 with approximately 30% conversion efficiency, and the resulting SO_2/SO_3 mixture then flowed through the measurement cell. For H_2SO_4 measurements, the SO_2/SO_3 flow was mixed with air containing controlled amounts of humidity (H_2O) from the evaporator. When water was added to the flow, a portion of SO_3 hydrates to H_2SO_4 . When SO_2 flow through the catalyst is substituted for dry air flow, only SO_3 , H_2SO_4 and H_2O remain in the gas flow through the absorption cell; this occurs due to SO_3 being flushed out of the catalyst bed. The two species, SO_3 and H_2SO_4 , exist in the flow in a state of equilibrium (Fig. 8). The concentration of both depends on temperature and humidity level. Lower temperatures favor H_2SO_4 while higher temperatures favor SO_3 . By supplying a known amount of H_2O into the gas flow and maintaining stable temperature of the absorption cell, we control the ratio of H_2SO_4 and SO_3 in the gas mixture. SO_3 does not have absorption features in the $8\ \mu\text{m}$ band. Therefore, it does not affect the H_2SO_4 measurements. Furthermore, H_2O features were eliminated from H_2SO_4 spectrum by using the measured H_2O spectrum as a background for absorption calculations. This technique allowed for measurement of the H_2SO_4 library spectrum in presence of the interfering species. The gas flow exiting the measurement cell is directed into the controlled condensation system (EPA Method 8A) where the controlled condensate was collected during the experimental run and processed with ion chromatography to determine combined concentration of SO_3 and H_2SO_4 in a flow. In the case of dry gas, water vapor can be added after the cell through valve 4 (Fig. 7) and controlled condensation (EPA Method 8A) performed to measure SO_3 concentration.

For SO_2 measurements, 100 ppm flow was supplied through the Air/ SO_2 bypass line directly into the measurement cell. The temperature of the multi-pass absorption cell was set to the desired experimental conditions of 300°C or 350°C respectively.

For H_2O measurements, dry air was run through the evaporator to produce 8% humidity level and then supplied into the measurement cell through the separate heated line.

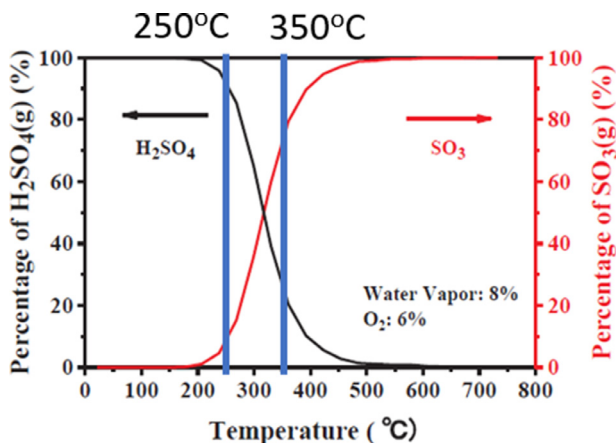


Fig. 8. Equilibrium between SO_3 and H_2SO_4 .

Each experimental data acquisition run consisted of the following sequence. First, the dry air background was measured, then the species were introduced to the flow. For the measurement of SO_3 both SO_2 and SO_3 were present in the flow from the catalyst. However, when SO_2 flow was stopped and switched to the dry air inlet, the dry air continued to flush out SO_3 trapped inside the catalyst without SO_2 being present in the flow. For this approach we exploit the SO_3 adsorption-desorption mechanism typical of vanadia catalyst to temporarily operate on air without SO_2 , to produce an output consisting only of SO_3 and air [5]. This enabled stable SO_3 measurements without any interference from SO_2 for about 15 – 20 min at a time.

Ethics Statements

Neither human subjects nor animals nor social media platforms were involved in the presented work.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data Availability

[Infrared Absorption Spectra of \$\text{H}_2\text{SO}_4\$, \$\text{SO}_3\$, \$\text{SO}_2\$, and \$\text{H}_2\text{O}\$ at 300C and 350C \(Original data\)](#) (EDX NETL's Data eXchange (EDX)).

CRedit Author Statement

Ilya Dunayevskiy: Software, Validation, Data curation, Writing – original draft; **Jason Kriesel:** Conceptualization, Supervision, Project administration, Funding acquisition, Writing – review & editing; **Andrea Biasioli:** Investigation, Validation, Data curation; **Yu-Chien (Alice) Chien:** Supervision; **Derek Dunn-Rankin:** Supervision, Project administration; **Lawrence Muzio:** Resources, Project administration; **Richard Himes:** Conceptualization, Funding acquisition, Project administration.

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