

Industrial Hygiene Report Control of Vapors in a Lab Setting:

Sulfur Dioxide

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Background

Sulfur dioxide (SO₂) — a colorless, noxious gas at room temperature — is a frequently encountered chemical in many industrial applications. The majority of SO₂ is generated as a precursor in the production of aqueous sulfuric acid solutions. It is also found in substantial amounts in the atmosphere as a result of volcanic activity and the burning of fossil fuels.

SO₂, whether encountered in an industrial setting or out in the world at large, can be a significant respiratory hazard. The main physical and environmental danger of SO₂ exposure is the reaction of SO₂ and water vapor to form sulfuric acid. This mechanism is one of several that can produce so-called “acid rain”. In any case, given the prevalence of SO₂ in the ambient atmosphere and its usage in industrial applications, exposure to SO₂ is a fairly common respiratory hazard. Many plastics and organic chemistry applications may generate a SO₂ respiratory hazard as well as most combustion reactions involving fossil fuels. Sometimes exposure can result from food preservation processes where elemental sulfur is burned in an enclosed space to preserve food such as tomatoes. Other common scenarios that might result in SO₂ exposure include lab or industrial usage of sulfuric acid.

The Occupational Safety and Health Administration (OSHA) has set the Permissible Exposure Limit (PEL) for SO₂ exposure to be 5 parts per million (PPM) time weighted average (TWA). This means that operator exposure should not exceed an average concentration of 5 PPM over an 8 hour shift. The [National Institute for Occupational Safety and Health \(NIOSH\)](#), another health and safety standards group, suggests a Recommended Exposure Limit of 2 PPM TWA and a Short Term Exposure Limit (STEL) of 5 PPM. The STEL is essentially the concentration that an operator can be exposed to for a short period of time, generally less than 60 minutes, without short or long term ill effects. NIOSH also lists the Immediately Dangerous to Life and Health (IDLH) limit as 100 PPM. The IDLH is the concentration that is immediately dangerous to operator health and exposure would likely illicit acute and chronic symptoms.

Test Design

Most filter efficiency tests follow the same procedure: Generate a filter “challenge” inside an enclosure and measure “challenge” concentration at the filter inlet, the filter outlet, and in the ambient room air around the test enclosure. SO₂ presents a significant

challenge in this regard as it's a gas at room temperature and is difficult to generate in significant and/or constant quantities.

For this test, a batch reaction of hydrochloric acid (HCl) and sodium bisulfite (NaHSO₃) was used to generate the “challenge gas”. Instead of using a continuous source of the “challenge” as you would when evaporating a liquid in a dish, the SO₂ was generated by staging the reactions in smaller sealed flasks that could be triggered to start the reaction without breaching the flasks seals. One of the reaction flasks was initiated and breached shortly before sampling started. During the course of the test more of the flasks were initiated and then breached to provide a somewhat continuous supply of SO₂ during the test.

The test was based on NIOSH Test Method 6004 which specified a minimum sample volume of 4L and a sampling rate between 0.5-1.5 L/min. The guidelines were used to determine a target sample volume of 30L, while sampling at 1.5L/min was chosen to ensure that the samples contained enough analyte to be measured by the analytical lab.

Test Equipment and Setup

The unit under test in this study was Sentry Air Systems' (SAS) 30-inch wide ductless containment hood ([SS-330-DCH](#)). The SS-330-DCH was configured with a standard carbon pre-filter pad (SS-300-CFP) and a specialty blended 10lb treated activated carbon filter (SS-310-AGF) for “acid-gas” type respiratory hazards. The SS-310-AGF is a specialty treated filter that works via chemisorption in lieu of adsorption as in standard activated carbon.

Air sampling was conducted using SKC-branded personal air samplers (SKC-224-PCXR4), each calibrated with a BIOS International Defender 510 (Defender 510). The flow rate of each sampler was set, measured, and recorded to meet NIOSH and/or OSHA test protocol requirements. The SKC-224-PCXR4 flow rates were recorded both before and after testing and the average of the two values was used. Sample size was determined by using the timer onboard each SKC-224-PCXR4 air sampler along with their average calibrated flow rates.

Test design dictated that each sampler be setup with a flow rate of about 1.5 L/min for a sample time of about 20 minutes. According to the NIOSH test method 6004, a sample train consisting of a 0.8 micrometer cellulose ester membrane and a cellulose + Na₂CO₃ filter were used as sample media.

Test samples were taken at four locations during the test. Definitions of the test points can be found in the table below.

Test Point A (T1A)	Located in the interior of the hood 6 inches below the FFU inlet.
Test Point B (T1B)	Located on the exterior of the FFU just above the outlet. Note: The sample taken at T2B was made with the sampling hose perpendicular to exhaust airflow.
Test Point C & D (T1C & T1D)	Located on a shelf in the lab space approximately 5 feet from the test apparatus and about 5 feet off the floor. Note: T1C was taken before the SO ₂ challenge

	was released and T1D was taken just after the SO ₂ challenge was released.
Blank	This was a sorbent tube from the same lot number as the other sample tubes and was subjected to the same handling procedures as the other samples however no air was pulled through it.

The batch gas generation setup consisted of four 150mL Erlenmeyer flasks each sealed with a rubber balloon. A 20% HCl solution was prepared from a concentrated HCl solution as well as a commercially procured 1kg container of NaHSO₃. The flasks were prepared by initially cleaning and weighing the dried flasks. 10mL of the diluted HCl solution was then added to the flask and about 8 g of NaHSO₃ was measured out and then added to one of the balloons. The balloon-with-reagent was weighed and then pulled over the lip of the flask in such a way that the flask was sealed but the chemicals were not poured into the flask. This apparatus was then also weighed. When the apparatus was needed to generate SO₂, about 2 minutes prior to being needed the balloon was picked up so as to allow the reagents inside to slide into the flask. Then the flask was gently agitated. Two minutes later the balloon was removed to release the gas.

The lab space utilized for testing was an air conditioned, non-vented room approximately 12'Wx12'Lx8'H with the SS-330-DCH located atop a 32" long table in one corner of the room. Note: Ceiling clearance above the unit was approximately 14.5 inches.

After testing was completed all samples were labeled and couriered to a third party analytical lab for next day testing. The lab analyzed the samples for SO₂ composition and reported the results in mg of SO₂. This information was converted to mg/m³ by calculating the total volume of air sampled at each test point.

Mass measurements were made using an MB 2610 triple beam balance.

Procedure

1. Prepare hood for testing. Ensure SS-310-AGF is properly installed and sealed in the fan-filter unit.
2. Prepare "challenge gas" flasks and label E, F, G, & H
3. Arrange sample trains on the SKC-224-PCXR4 and calibrate with the Defender 510 ensuring that flow rate is +/- 5% of the target flow rate
4. Place samplers in their respective locations and move flasks into hood.
5. Prior to initiating Flask E, start initial ambient sample T1C and allow full sample to be taken
6. Initiate Flask E, wait 2 minutes, then breach flask E
7. Wait 3 minutes then begin sampling at test points T1A & T1B and initiate flask F
8. Wait 5 minutes then breach flask F and initiate flask G
9. Wait 5 minutes then breach flask G and initiate flask H
10. Wait 5 minutes then breach flask H
11. Wait 5 minutes then stop samples at test points T1A & T1B
12. Start sampler at test point T1D and wait 20 minutes
13. Disassemble all sample trains, cap and label all samples, and pack for shipping
14. Neutralize all test solutions with Sodium Hydroxide before disposing

Data/Findings

SO ₂ Industrial Hygiene Test Data		HIH Laboratories Analysis Results			
Test Point ID	Sample Time (Minutes)	Sample Flow Rate (mL/min)			SO ₂ (mg)
		Pretest	Posttest	Avg.	
T1A	20	1492.1	1492.2	1492.2	3.6
T1B	20	1462	1478.2	1470.1	<0.004
T1C	26	1450.3	1455.1	1452.7	<0.004
T1D	25	1490.7	1593	1541.85	0.014

Test Point ID	Calculated Results		
	Sample Indicated Concentration		
	Sample Volume (m ³)	SO ₂ (mg/m ³)	SO ₂ (PPM)
T1A	2.9843E-02	120.6313	46.0849
T1B	2.9402E-02	0.1360	0.0519
T1C	3.7770E-02	0.1059	0.0404
T1D	3.8546E-02	0.3632	0.1387

Indicated Performance
SO₂ (% Efficiency)
99.89%

Flask ID	Estimated SO ₂ Generated based on Stoichiometry of reaction		
	Mass of HCl (g)	Mass of NaHSO ₃ (g)	Calculated Mass of SO ₂ (g)
Flask E	2.33	8.00	4.31
Flask F	2.75	7.20	5.09
Flask G	0.86	8.00	1.60
Flask H	2.55	8.60	4.72

Results Summary

HIH Laboratories processed the samples and analyzed for the SO₂ concentration. According to their results, the reporting limit for SO₂ was >0.004 mg of analyte. Meaning any samples made in areas of very low concentration might not register on this analysis and, with certainty, the sample contained less than this amount.

Considering that it is better to underestimate filter performance, the results presented for this test will follow the convention that if a sample has a reported value of “< 0.004 mg” that value will be taken as 0.004 mg. This results in the reported filter efficiency being considerably lower than it’s actual efficiency; however following this convention also implies that the filter’s performance is no worse than the reported value.

Lab analysis found 3.6 mg of SO₂ at test point T1A which corresponds to an enclosure SO₂ concentration of about 120 mg/m³ or 46 PPM. The indicated concentration at the

filter outlets, T1B, was at the detection limit which corresponds to 0.14 mg/m³ or 0.05 PPM.

Based on the measured difference in concentration across the filter stack, the indicated filter efficiency for SO₂ using SAS' acid gas filter media was 99.89%. During the course of the test the room SO₂ concentration only changed by about 0.26 mg/m³ which is well below the STEL or the PEL.

Conclusions and Considerations

Given the results presented here, it is likely that the use of the SS-330-DCH configured with SS-310-AGF for work with SO₂ and related gases would prove beneficial in reducing operator exposure to respiratory hazards presented by of those chemicals. Further, it is indicated that use of this equipment will reduce operator exposure to respiratory hazards below what exposure would be expected if no precautions were taken. As mentioned earlier, it is likely that the acid gas filter efficiency results presented here are actually lower than the "real" value as the SO₂ concentrations at the filter outlet were too low to measure given current test setup limitations.

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