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Reduced Sulfur Analysis by ASTM 5504



Reduced sulfur compounds, such as sulfides and mercaptans, tend to have very distinctive odors that may be detected at extremely low concentrations. These odors are offensive to many individuals, and in some cases may result in irritative symptoms, such as headaches and nausea. ASTM D5504 is a useful method for the differentiation and quantification of many of these odor-causing sulfur compounds.

Sources and Common Compounds

Columbia Analytical's air quality laboratory performs ASTM D5504 to identify and quantify sulfur compounds from a variety of sources, such as:

Source	Common Sulfur Compounds	S

Sewers and Wastewater
Treatment Facilities

Hydrogen sulfide, mercaptans, dimethyl sulfide

Landfills Hydrogen sulfide

Petrochemical Refineries Hydrogen sulfide, carbonyl sulfide, carbon disulfide

Odorants of Liquid Propane Ethyl mercaptan, dimethyl sulfide, tetrahydrothiophene

Odorants of Natural Gas tert-Butyl mercaptan, isopropyl mercaptan, n-propyl mercaptan, ethyl methyl

sulfide

Pulp and Paper Mills Hydrogen sulfide, methyl mercaptan, dimethyl disulfide, dimethyl sulfide

Composting Facilities

Hydrogen sulfide, ethyl mercaptan, carbon disulfide, dimethyl sulfide,

dimethyl disulfide, methyl mercaptan, propyl mercaptan

Livestock Activities Hydrogen sulfide, dimethyl disulfide, dimethyl trisulfide, carbonyl sulfide

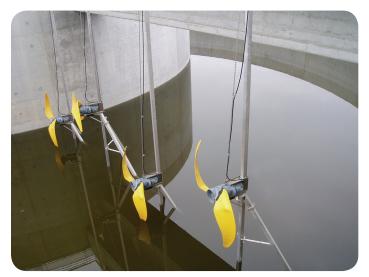


Applications

Use of this method is appropriate in situations where sulfur odors are a concern and differentiation between them is desired. It also may be a useful tool during odor investigations when a complex matrix is present or the human receptors are unable to adequately characterize the odor.

Collection, Shipping and Analysis

Since many sulfur compounds are not stable in stainless steel canisters, such as Summa canisters, air samples for this analysis should be collected in Tedlar bags. When the odor is intermittent, unpredictable, or at a remote location,



Wastewater Treatment Facility

Columbia Analytical may recommend the use of a Silco (glass lined) canister. Please contact the lab for more information on sampling media for sulfur compounds.

Due to the unstable and reactive nature of many sulfur compounds, full compliance of the method requires analysis of the samples within 24 hours of collection. Despite this short holding time, Columbia Analytical receives samples from all over North America and performs analysis within holding time on a daily basis.

We recommend that our clients collect their sample as late in the day as their sampling plan allows, then ship the sample to the lab via priority overnight delivery. The sample then arrives at our location early in the day, ensuring analysis within the holding time. Analysis is performed following ASTM D5504, which is a gas chromatography method with sulfur chemiluminescence detection.

Limitations of ASTM D5504

The short holding time for the samples is one limitation of this method. Another is the fact that the odor thresholds (i.e. the concentrations at which these compounds can be observed by an individual) are lower than the analytical detection limits for some compounds.

The method may be obtained online from www.astm.org.

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Table 1. Reduced Sulfur Compounds, Reporting Limits And Available Odor Thresholds

Compound	CAS No.	Reporting Limit PPBV	Character
Hydrogen Sulfide	7783-06-4	5.00	Rotten eggs
Carbonyl Sulfide	463-58-1	5.00	Pungent
Methyl Mercaptan	74-93-1	5.00	Rotten cabbage
Ethyl Mercaptan	75-08-1	5.00	Rotten cabbage
Dimethyl Sulfide	75-18-3	5.00	Decayed vegetables
Carbon Disulfide	75-15-0	2.50	Vegetable sulfide
Isopropyl Mercaptan	75-33-2	5.00	Skunk
tert-Butyl Mercaptan	75-66-1	5.00	Skunk
n-Propyl Mercaptan	107-03-9	5.00	Cabbage
Ethyl Methyl Sulfide	624-89-5	5.00	Sulfurous, garlic
Thiophene	110-02-1	5.00	Sweet
Isobutyl Mercaptan	513-44-0	5.00	Skunk
Diethyl Sulfide	352-93-2	5.00	Sharp, garlic
n-Butyl Mercaptan	109-79-5	5.00	Skunk
Dimethyl Disulfide	624-92-0	2.50	Putrid, decayed vegetables
3-Methylthiophene	616-44-4	5.00	
Tetrahydrothiophene	110-01-0	5.00	Sharp, pungent
2,5-Dimethylthiophene	638-02-8	5.00	
2-Ethylthiophene	872-55-9	5.00	
Diethyl Disulfide	110-81-6	2.50	
Dimethyl Trisulfide	3658-80-8	2.50	Rotten cabbage

Sources

- AIHA 1989. Odor Thresholds for Chemicals with Established Occupational Standards
- ASTM 1978. Compilation of Odor and Taste Threshold Values Data, F.A. Fazzalari, (ed.) ASTM DS 48A Dravnieks, A. 1985. Atlas of odor profiles. ASTM DS 61.
- NLM 2004. Hazardous substances data bank (http://toxnet.nlm.nih.gov/cgi-bin/sis/htmlgen?HSDB) Ruth, J. 1986. AIHAJ 47: A142-A151.
- Sandmeyer, A., 1981. Organic sulfur compounds. In Patty's Industrial Hygiene, 3rd Revised Edition, Clayton, G. & F. (ed.), John Wiley & Sons, New York.
- Van Gemert, L.J. and A.H. Nettenbreijer. 1977. Compilation of odour threshold values in air and water. Zahn et al., 2001. J. Environ. Qual. 30:624-632.



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