



TRS and Mercaptan Analysis

Applicable Methods:

Review: A number of analytical techniques are available for determination of sulfur compounds. Gas Chromatography with Flame Photometric Detection (FPD) and with Sulfur Chemiluminescence (SCD) detection are the most widely used. Detection limits ranging from .055 ppm using FPD and .007 ppm using SCD are typical. A detection limit of (.0021 ppb) has been reported for GC-Electrochemical detection. Other less sensitive methods are available such as EPA method 16 using the barium-thorin titration procedure and methods using methylene blue techniques have been widely utilized for continuous monitoring of hydrogen sulfide in air and are sensitive to concentrations to .001 ppm to .003 ppm in ambient air(NIOSH). The amount of sulfide is determined by spectrophotometric or colormetric measurement of methylene blue. Both methods described above are subject to other sulfur compound interferences. NIOSH method 6013 measures hydrogen sulfide in air by ion chromatography. And has a detection limit of 11 ug/sample or .9 mg/cubic meter based upon a 20 liter sample. Iodometric titration has been used with a detection limit of .5 ppb based upon a 30 liter sample. Numerous other methods utilizing impregnated lead acetate papers or tapes have been used.

ASTM D6228-98 (2003) and Method 15 (Offsite)

1. Scope

1.1 This test method covers the determination of individual volatile sulfur-containing compounds in gaseous fuels by gas chromatography (GC) with flame photometric detection (FPD). The detection range for sulfur compounds is from 20 to 20 000 picograms (pg) of sulfur. This is equivalent to 0.02 to 20 mg/m³ or 0.014 to 14 ppmv of sulfur based upon the analysis of a 1-mL sample.

1.2 This test method describes a GC-FPD method using a specific capillary GC column. Other GC-FPD methods, with differences in GC column and equipment setup and operation, may be used as alternative methods for sulfur compound analysis with different range and precision, provided that appropriate separation of the sulfur compounds of interest can be achieved.

1.3 This test method does not intend to identify all individual sulfur species. Total sulfur content of samples can be estimated from the total of the individual compounds determined. Unknown compounds are calculated as monosulfur-containing compounds.

List Compounds:

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|------------------|---------------------|
| Hydrogen sulfide | n-Propyl mercaptan |
| Carbonyl sulfide | t-Butyl mercaptan |
| Methyl mercaptan | iso-Butyl mercaptan |
| Ethyl mercaptan | Butyl mercaptan |
| Dimethyl sulfide | Dimethyl disulfide |
| Carbon disulfide | |