Designation: D6646 − 03 (Reapproved 2022)

Standard Test Method for Determination of the Accelerated Hydrogen Sulfide Breakthrough Capacity of Granular and Pelletized Activated Carbon[1]

This standard is issued under the fixed designation D6646; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method is intended to evaluate the performance of virgin, newly impregnated or in-service, granular or pelletized activated carbon for the removal of hydrogen sulfide from an air stream, under the laboratory test conditions described herein. A humidified air stream containing 1 % (by volume) hydrogen sulfide is passed through a carbon bed until 50 ppm breakthrough of H_2S is observed. The H_2S adsorption capacity of the carbon per unit volume at 99.5 % removal efficiency (g H_2S/cm^3 carbon) is then calculated. This test is not necessarily applicable to non-carbon adsorptive materials.

1.2 This standard as written is applicable only to granular and pelletized activated carbons with mean particle diameters (MPD) less than 2.5 mm. See paragraph 5.3 if activated carbons with larger MPDs are to be tested.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D2652 Terminology Relating to Activated Carbon
D2854 Test Method for Apparent Density of Activated Carbon
D2867 Test Methods for Moisture in Activated Carbon
E300 Practice for Sampling Industrial Chemicals

3. Terminology

3.1 Terms relating to this standard are defined in D2652.

4. Summary of Test Method

4.1 Breakthrough capacity is determined by passing a stream of humidified air containing 1 volume % hydrogen sulfide through a sample of granular or pelletized activated carbon of known volume under specified conditions until the concentration of hydrogen sulfide in the effluent gas reaches 50 ppmv.

5. Significance and Use

5.1 This method compares the performance of granular or pelletized activated carbons used in odor control applications, such as sewage treatment plants, pump stations, etc. The method determines the relative breakthrough performance of activated carbon for removing hydrogen sulfide from a humidified gas stream. Other organic contaminants present in field operations may affect the H_2S breakthrough capacity of the carbon; these are not addressed by this test. This test does not simulate actual conditions encountered in an odor control application, and is therefore meant only to compare the hydrogen sulfide breakthrough capacities of different carbons under the conditions of the laboratory test.

5.2 This test does not duplicate conditions that an adsorber would encounter in practical service. The mass transfer zone in the 23 cm column used in this test is proportionally much larger than that in the typical bed used in industrial applications. This difference favors a carbon that functions more rapidly for removal of H_2S over a carbon with slower kinetics. Also, the 1 % H_2S challenge gas concentration used here engenders a significant temperature rise in the carbon bed. This effect may also differentiate between carbons in a way that is not reflected in the conditions of practical service.

5.3 This standard as written is applicable only to granular and pelletized activated carbons with mean particle diameters

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[1] This test method is under the jurisdiction of ASTM Committee D28 on Activated Carbon and is the direct responsibility of Subcommittee D28.04 on Gas Phase Evaluation Tests.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

less than 2.5 mm. Application of this standard to activated carbons with mean particle diameters (MPD) greater than 2.5 mm will require a larger diameter adsorption column. The ratio of column inside diameter to MPD should be greater than 10 in order to avoid wall effects. In these cases it is suggested that bed superficial velocity and contact time be held invariant at the conditions specified in this standard (4.77 cm/s and 4.8 s). Although not covered by this standard, data obtained from these tests may be reported as in paragraph 12 along with additional information about column diameter, volume of carbon, and volumetric flow rate used.

5.4 For pelletized carbons, it is felt that the equivalent spherical diameter of the pellet is the most suitable parameter for determining the appropriate adsorption column inside diameter. The equivalent spherical diameter is calculated according to the following equation.

$$D_{eqv} = \frac{3\,X\,d\,X\,h}{d+2\,X\,h} \tag{1}$$

where:

d = the diameter, and
h = the length of the pellet in mm.

An average of 50 to 100 measurements is recommended to determine the average length of a pellet. Annex A3 is a table to guide the user in selecting bed diameter and flow rates from typical equivalent diameters (or MPD) of pelletized carbon.

6. Apparatus and Materials

6.1 *(5±1) % Hydrogen Sulfide in Nitrogen Mixture*—The concentration of hydrogen sulfide in the gas test mixture must be known. It is recommended that gas cylinders specifically manufactured for holding hydrogen sulfide gas be used. Analyzed and certified hydrogen sulfide in nitrogen gas mixtures can be purchased from specialty gas suppliers. Annex A1 and Annex A2 present methods that may be used to check the hydrogen sulfide concentration of hydrogen sulfide/nitrogen gas mixtures. It is recommended that the hydrogen sulfide concentration be checked if gas cylinders are stored for more than three months, particularly after being partially depleted. Other organic contaminants that may be present in the hydrogen sulfide tank can affect the adsorption capacity of the carbon being tested.

6.2 *Hydrogen Sulfide Detector*—The hydrogen sulfide detector used in this test must be demonstrated to reliably detect 50 ppm hydrogen sulfide in a humidified air stream. In addition to certain "solid state" detectors, electrochemical type hydrogen sulfide sensors, for example, Ecolyzer Model 6400 or Interscan LD-17, have been evaluated and fit this requirement. Other means of hydrogen sulfide detection may be selected, as long as they are carefully calibrated and evaluated for this application.

6.3 *Adsorption Tube*—The adsorption tube is shown in Fig. 1. Adsorption tubes are not commercially available; however, they can be custom fabricated by a scientific glassblower. The perforated support shown is necessary to support the carbon bed and to enhance diffusion of the gases. (Adjust dimensions accordingly from Annex A3, specifically diameter.)

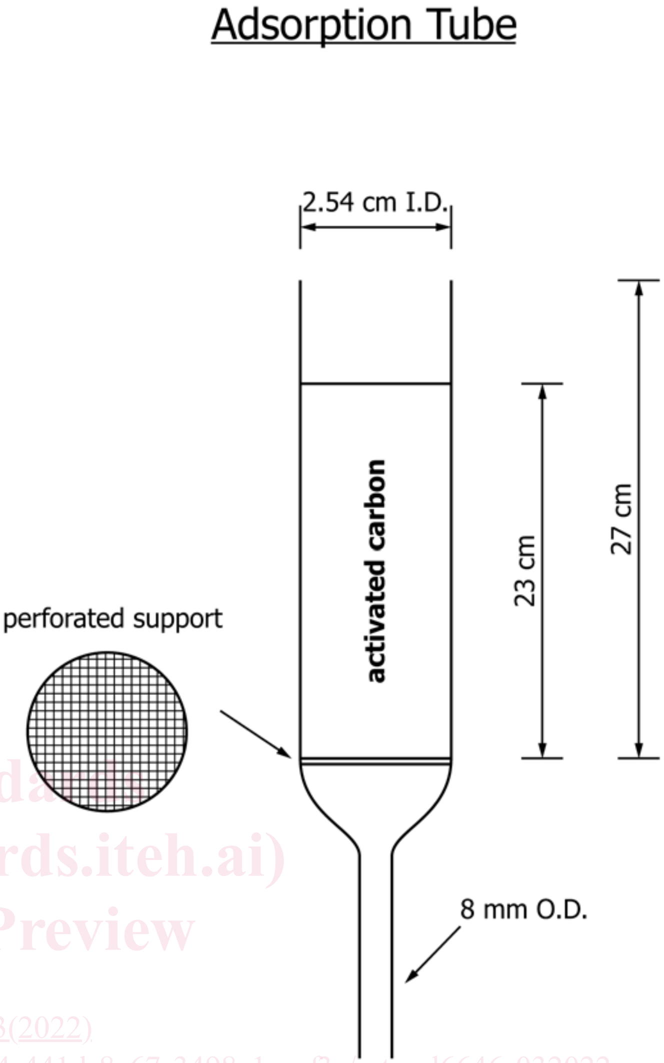

FIG. 1 Schematic of Adsorption Tube

6.4 *Flowmeter (0-500 mL/min Nitrogen; see Annex A3 for Guide to Higher Flow Range for Particles > 2.5 mm MPD)*—For hydrogen sulfide/N_2 control, it is recommended that the wettable parts of this flow meter be made of PTFE or other corrosion resistant material. Rotameter floats should be made from non-metallic materials such as glass or sapphire.

6.5 *Flowmeter (0-2000 mL/min Air; see Annex A3 for Guide to Higher Flow Range for Particles > 2.5 mm MPD)*—

NOTE 1—Mass flow controllers have been found to be more reliable than flowmeters and are highly recommended due to their ability to automatically maintain precise gas flow rates. Rotameters are satisfactory for this method, but may require more frequent attention in maintaining proper test gas flows for the duration of the test.

6.6 *Two Stage Cylinder Regulator, Suitable for Corrosive Gas Service, for Hydrogen Sulfide Gas Cylinder.*

6.7 *Air Line Pressure Regulator—Low Pressure*—To maintain up to 10 psig pressure for up to 2 liters of air/min flow rate (see Annex A3 for guide to airflow for tubes used for particles >2.5 mm MPD)

6.8 *Two Metering Valves*—Suitable valves are the Whitey SS-21-RS4 (H_2S/N_2) and B-21-RS4 (air). Other similar valves may be used. If the rotameters in 6.4 and 6.5 are equipped with their own high quality metering valves, these valves are not needed.

6.9 *Source of Dry, Contaminant-Free Air Capable of Delivering up to 2 liters/min Through the Test System,* (higher flow for larger particles, >2.5 mm MPD, see Table A3.2.)

6.10 *Gas Bubbler*—(Ace Glass cat .#5516 gas washing bottle equipped with gas dispersion fritted tube, cat. #7202, porosity code "C," or equivalent to this.) The glass bubbler should be immersed in a constant temperature bath regulated at 25 °C to ensure the generation of a 80 % RH air stream for the final gas mixture (after mixing with dry H_2S/N_2). The porous bubbler should be immersed under at least 3 in. of water to consistently saturate the air stream with water during the course of the test. (A larger gas washing bottle should be used if larger particles than 2.5 mm (Equivalent Diameter) and a larger bed are used. Increase size proportionately with air flow).

6.11 *Hydrogen Sulfide Calibration Gas Mixture,* 20 to 50 ppmv, in nitrogen, to be used as a span or calibration gas for the hydrogen sulfide detector. (Available from specialty gas supply companies.)

6.12 *Timer*—A count up timer that can be tripped at the 50 ppmv set point of the H_2S monitor and is capable of retaining the tripped time.

6.13 *Vibratory Feeder,* (see Test Method D2854).

6.14 *Powder Funnel.*

6.15 *Temperature Controlled Water Bath,* to maintain the water bubbler at 25 °C ± 2 °C.

6.16 Other miscellaneous hardware needed to set up the apparatus in Fig. 2. Polyethylene tubing is suitable for carrying the H_2S/N_2 flow. Clamped ball and socket joints are convenient for quick connect and disconnect of the absorption column and calibration bubbler (see Annex A2) from the system.

7. Safety Precautions

7.1 Several potential hazards are associated with conducting this test procedure. It is not the purpose of this standard to address all potential health and safety hazards encountered with its use. The user is responsible for establishing appropriate health and safety practices before use of this test procedure. Determine the applicability of Federal and State regulations before attempting to use this standard test method.

7.2 Personnel conducting the hydrogen sulfide adsorption capacity procedure should be aware of potential safety and health hazards associated with the chemicals used in this procedure. The "Material Safety Data Sheet" (MSDS) for each reagent listed in Section 6 should be read and understood. Special precautions to be taken during use of each reagent are included on the MSDS. First aid procedures for contact with a chemical are also listed on its MSDS. The MSDS for each reagent may be obtained from the manufacturer.

7.3 Safety and health hazard information on reagents used in this procedure may also be obtained from:

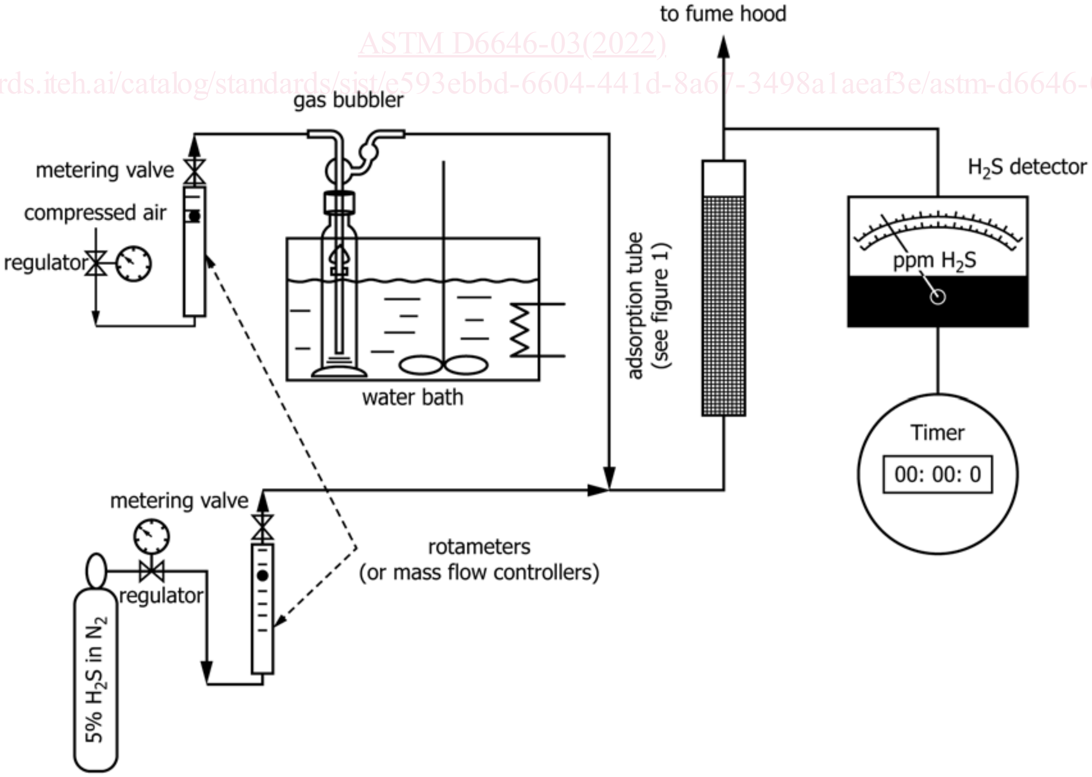

FIG. 2 Schematic of Apparatus for Determination of H_2S Breakthrough Capacity

7.3.1 *Sax's Dangerous Properties of Industrial Materials* / Richard J. Lewis, Sr., New York : J. Wiley, 2000.

7.3.2 *NIOSH/OSHA Pocket Guide to Chemical Hazards*, 1997, U.S. Department of Labor, Occupational Safety and Health Administration, Washington, D.C. Available from U.S. Government Printing Office, Washington, D.C. or at http://www.cdc.gov/niosh/npg/npg.html.

8. Sampling

8.1 Guidance in sampling granular activated carbon is given in recommended Practice E300.

9. Calibration

9.1 Calibration of flowmeters, mass flow controllers, and hydrogen sulfide detectors shall be performed by standard laboratory methods.

NOTE 2—The test apparatus (Fig. 1) has metering valves at the rotameter outlets. This is done to minimize changes in gas flow rates caused by small backpressure changes during this long duration test. However, placement of metering valves in this position invalidates the atmospheric pressure calibration usually supplied by the rotameter manufacturer. The apparatus in A2.4.2 may be used to calibrate the rotameters. During this calibration, the gas delivery pressure must be the same as that used during the actual test.

9.2 Determine the percent H_2S in the H_2S/nitrogen tank using the methods outlined in Annex A1 or Annex A2 if the H_2S/nitrogen tank was not certified by the manufacturer.

10. Procedure

10.1 Assemble the test apparatus as shown in the schematic diagram of Fig. 2.

10.2 Adjust the H_2S/N_2 and air flow rates to generate a 1.0 % H_2S stream at a total flow rate of 1450 cm^3/min at the one-inch diameter adsorption tube (see Annex A3 for higher flowrates with larger than 2.5 mm (Equivalent Diameter) particles). This adjustment will depend on the concentration of H_2S in the H_2S/N_2 gas mixture.

10.3 Determine the H_2S concentration of the actual mixed test gas using method(s) as outlined in Annex A1 or Annex A2 of this procedure. This test should be repeated if any adjustment is made on the flow meter(s).

10.4 Obtain a representative sample of the as-received granular or pelletized activated carbon to be tested. A300 cm^3 sample is sufficient for apparent density, moisture and replicate performance testing. (A larger amount should be used if the particles larger than 2.5 mm (Equivalent Diameter) and a larger diameter bed are used).

10.5 Reduce the sample size to an aliquot for testing using the riffling procedure described in E300.

10.6 Determine the apparent density of the sample by Test Method D2854.

10.7 Use an adsorption tube whose volume has been calibrated to contain 116 mL (see Annex A3 for larger volumes) when filled from the top of the carbon support to a bed depth of approximately 22.9. (The calibrated volume for an adsorption tube can be determined by using a graduated buret to determine the volume of water required to fill the adsorption tube from the top of the carbon support to approximately the 22.9 cm mark.)

10.8 Tare a clean, dry adsorption tube to the nearest 0.1 g. Note and record.

10.9 Fill the adsorption tube with 116 mL of carbon [bed depth of approximately 22.9 cm] using a vibratory feeder. (The apparatus described in Test Method D2854, or equivalent is suitable for filling the adsorption tube.) The vibratory feeder is to be adjusted so the adsorption tube is filled at a rate not less than 0.75 mL/s or exceeding 1.0 mL/s. (See Annex A3 for guide to larger volume if larger than 2.5 mm (Equivalent Diameter) particles are tested.)

10.10 Weigh the filled adsorption tube to the nearest 0.1 gm. Note and record.

10.11 Carefully transfer the filled adsorption tube to the test system and connect it to the test apparatus.

NOTE 3—If a sample of non-impregnated, low moisture, virgin carbon is being evaluated for adsorption capacity, it is advised that it be conditioned for several hours with only humidified air passing through it to equilibrate the moisture content of the carbon with the moisture in the air stream. The moisture content of the carbon will affect the breakthrough capacity

Start the H_2S/air flow and simultaneously start the timer.

10.12 Continue the H_2S/air flow until a breakthrough of 50 ppmv is indicated. Record the time elapsed from the start of H_2S/air flow to 50 ppm breakthrough.

10.13 Repeat 10.2 – 10.12 on replicate portions of the carbon sample. A minimum of one replicate analyses must be performed.

11. Calculation

11.1 Calculate the hydrogen sulfide breakthrough capacity of the test sample using the following equation:

$$\frac{g\ H_2S}{cm^3\ GAC} = \frac{\left(\frac{C}{100}\right) \times F \times T \times \left(\frac{1\ L}{1000\ cm^3}\right) \times \left(\frac{1\ mole}{22.4\ L}\right) \times \left(\frac{34.1\ g\ H_2S}{mole}\right)}{V} \quad (2)$$

where:

C = concentration of hydrogen sulfide in air stream, volume %,
F = total H_2S/air flow rate, cm^3/min (should be 1450 cm^3/min) (Adjust from Annex A3 if necessary),
T = time to 50 ppmv breakthrough, minutes, and
V = actual volume of the carbon bed in the absorption tube, cm^3 (Adjust from Annex A3 if necessary).

NOTE 4—For simplicity and without introducing significant error into the calculation, it can be assumed the gas streams are at standard conditions and corrections for ambient temperature or pressure are unnecessary.)

This equation simplifies to:

$$\frac{g\ H_2S}{cm^3\ GAC} = \frac{(1.52 \times 10^{-5}) \times C \times F \times T}{V} \quad (3)$$

To determine the H_2S breakthrough capacity in g H_2S/g GAC, use the following equation:

$$\frac{g\ H_2S}{g\ GAC} = \frac{g\ H_2S/cm^3\ GAC}{apparent\ density\ (from\ 10.6)} \quad (4)$$

11.2 The hydrogen sulfide breakthrough capacity is determined for each replicate portion of the carbon sample. The average and sample standard deviation for the hydrogen sulfide breakthrough capacities is then calculated using N-1 weighting. If the standard deviation of the analyses is less than or equal to 10 % of the average hydrogen sulfide breakthrough capacity, the average value and standard deviation are reported as the hydrogen sulfide breakthrough capacity. If not, an additional replicate portion must be analyzed until the above criteria is obtained.

12. Report

12.1 Report the following:

12.1.1 Source of the sample,

12.1.2 Type and designation of the sample,

12.1.3 Name of carbon supplier,

12.1.4 Supplier name, lot number, batch number, and

12.1.5 H_2S breakthrough capacity in H_2S g/cm^3 of GAC.

13. Precision and Bias

13.1 *Precision*—A round-robin test of this proposed method was conducted in 1995, with five laboratories testing four different samples of impregnated activated carbon for H_2S removal, each sample being tested in triplicate. The following is a summary of the precision parameters of the round-robin:

material	average	S_r	S_R	r=2.8×S_r	R=2.8×S_R
			g H_2 S/cm^3 GAC		
C	0.0847	0.00683	0.01183	0.01913	0.03313
D	0.1147	0.00516	0.00875	0.01445	0.02451
B	0.1207	0.01211	0.01211	0.03391	0.03391
A	0.1480	0.00966	0.02319	0.02705	0.06493

S_r is the repeatability standard deviation for interlaboratory results, S_R is the reproducibility standard deviation for interlaboratory results. The precision of interlaboratory reproducibility results is indicated by R = 2.8 × S_R, the 95 % confidence limit of the test method. The repeatability of results by this method is indicated by r = 2.8 × S_r, the 95 % confidence limit of interlaboratory repeatability.

13.2 *Bias*—With respect to bias of the method, there seems to be a decline in repeatability and reproducibility with the increase of breakthrough capacity, as indicated by the general upward trend in the confidence limits with the increase in H_2S capacity.

14. Keywords

14.1 activated carbon; breakthrough capacity; hydrogen sulfide

ANNEXES

(Mandatory Information)

A1. ANALYSIS AND CALIBRATION OF H_2S TEST GAS STREAM WITH GAS CHROMATOGRAPHY

A1.1 Scope

A1.1.1 The exact concentration of the hydrogen sulfide test gas stream needs to be known. A gas chromatograph can be used to analyze the gas stream and determine its concentration against an independently certified calibration gas. This method can be used to determine the H_2S gas concentrations in both nitrogen and air mixtures. This method is believed to be more reliable than wet-chemical methods and can indicate the presence of contaminant gases that may be present in some grades of hydrogen sulfide.

A1.2 Summary of Method

A1.2.1 A sample of the gas to be analyzed is taken over a period of several minutes, collected in a one-time use polyvinyl fluoride (PVF) film bag or flow-through gas-sampling bottle. A gas-tight syringe is used to withdraw a sample of the gas and inject it into a previously calibrated gas chromatograph.

A1.3 Apparatus

A1.3.1 Column: 6 ft × 4 mm (ID) glass column, Chromosil 310 (or similar) packing, open bore, or any capillary column suitable for permanent gas separation.

A1.3.2 Conditions: Injector 60 °C.

Column oven: 42 °C to 46 °C (optimize for separation).

Detector: 60 °C (for FID or FPD).

A1.3.3 Detector type: Flame ionization detector, flame photometric detector, or Hall detector optimized for sulfur (most sensitive).

A1.3.4 Gas sampling bag(s) or glass collecting tube.

A1.3.5 Gastight syringe.

A1.3.6 Integrator or computerized data collection to integrate peak areas of sample gases.

A1.4 Procedure

A1.4.1 The GC column is glass, packed with Chromosil 310 (Supelco or similar), 6 ft. × 4 mm ID. The flow rate is set at 40 mL/min, the carrier gas helium. A flame ionization (or flame photometric) detector should be used. The column temperature should be maintained at 42 to 46 °C, depending on the efficiency of separation of the air peak from the hydrogen sulfide peak. Injector and detector temperature should be