1,3,5-Triglycidyl Isocyanurate (TGI)

Method no.:	PV2055
Matrix:	Air
Target concentration:	0.25 mg/m^3
Procedure:	Samples are collected by drawing known volumes of air through hydrobromic acid treated glass fiber filters. Samples are desorbed with dimethylformamide (DMF), derivatized with heptafluorobutyric anhydride (HFBAnh) and analyzed by gas chromatography (GC) using an electron capture detector (ECD).
Recommended air volume and sampling rate:	60 L at 1.0 L/min
Detection limit of the overall Procedure based on the recommended air volume:	23.3 µg/m^3
Status of method:	Stopgap method. This method has been only partially evaluated and is presented for information and trial use.

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1. General Discussion

1.1. Background

1.1.1. History of procedure

The OSHA Analytical Laboratory received a set of samples requesting the analysis of 1,3,5-triglycidyl isocyanurate (TGI) from glass fiber filters. Storage studies on glass fiber filters yielded poor recoveries of TGI. Therefore, this report describes the preliminary validation of a sampling and analytical method using glass fiber filters coated with hydrobromic acid (HBr).

1.1.2. Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.) (Ref. 5.3.)

The oral LD_{50} for rats is 400 mg/kg. The dermal LD_{50} for rats is >3000 mg/kg.

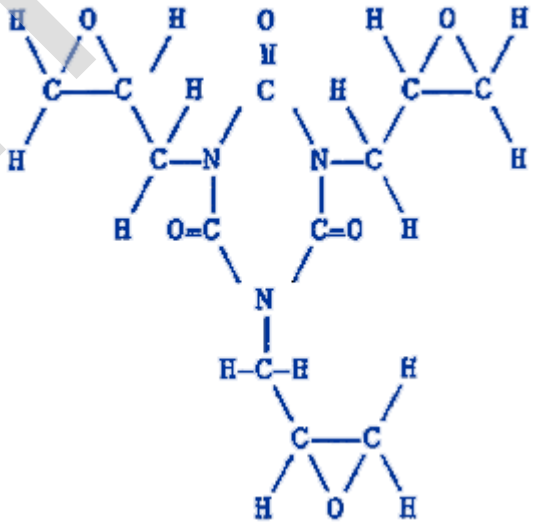
1.1.3. Potential workplace exposure

No estimate of worker exposure to TGI could be found. TGI is a solid resin that provides superior thermal, electrical, and mechanical properties and is recommended for laminates, insulating varnishes, coatings, and adhesives. (Ref. 5.2.)

1.1.4. Physical properties (Ref. 5.1. to 5.3.)

Molecular weight:	297.3
Molecular formula:	$C_{12}H_{15}N_3O_6$
CAS #:	2451-62-9
Specific gravity:	1.4 at 25 ℃
Melting point:	5-110 ℃
Solubility:	soluble in dimethylformamide, dimethylsulfoxide
Synonyms:	tris(2,3-epoxypropyl)isocyanurate
Description:	white crystalline powder

Structure:



1.2. Limit defining parameters

The detection limit of the analytical procedure is 0.9 pg per injection. This is the amount of analyte which will give a peak whose height is approximately five times the baseline noise. (Figure 1)

2. Sampling procedure

2.1. Apparatus

2.1.1. Samples are collected by using a personal sampling pump that can be calibrated to within ± 5% of the recommended flow rate with the sampling device in line.

2.1.2. Samples are collected with 37 mm Gelman type A/E glass fiber filters treated with HBr. The filters are prepared by soaking each filter with 0.5 mL of HBr solution (25 mL of 48% hydrobromic acid solution in 75 mL of acetonitrile) and allowing them to air dry overnight. These filters are further dried by placing them in a vacuum oven at 60°C for two hours. The filters are assembled in two-piece 37 mm polystyrene cassettes with backup pads. The cassettes are sealed with shrink bands and the ends are plugged with plastic plugs.

2.2. Reagents

No sampling reagents are required.

2.3. Sampling technique

2.3.1. Immediately before sampling, remove the plastic plugs from the filter cassettes.

2.3.2. Attach the cassette to the sampling pump with flexible tubing.

2.3.3. Attach the cassette vertically in the employee's breathing zone in such a manner that it does not impede work performance.

2.3.4. After sampling for the appropriate time, remove the cassette and seal with plastic plugs.

2.3.5. Wrap each sample end-to-end with an OSHA seal (Form 21).

2.3.6. Submit at least one blank for each set of samples. Handle the blank in the same manner as the samples, except no air is drawn through it.

2.3.7. Record the air volume (in liters of air) for each sample, and list any possible interferences.

2.3.8. Submit bulk samples for analysis in a separate container.

2.4. Extraction efficiency

Five treated glass fiber filters were each liquid spiked with 8 µL of a 2.005 mg/mL TGI standard. After two hours, the samples were extracted with 3 mL of dimethylformamide (DMF) by shaking them for 30 min and then analyzed as per section 3.5. The results are listed in the table below.

Table 2.4. Extraction Efficiency			
Sample #	Amount Spiked, µg	Amount Found, µg	% Recovered
Ex1	16.04	15.81	98.6
Ex2	16.04	17.75	110.6
Ex3	16.04	16.57	103.3
Ex4	16.04	17.39	108.4
Ex5	16.04	17.54	109.4
			Average 106

2.5. Retention efficiency

Six treated glass fiber filters were liquid spiked with 8 µL of a 2.005 mg/mL standard and humid air (~80% relative humidity) was drawn through each filter at 1 L/min for 60 minutes. The filters were extracted with 3 mL of DMF by shaking them for 30 min and then analyzed as per section 3.5. The results are listed in the table below.

Table 2.5. Retention Efficiency			
Sample	Amount Spiked, µg	Amount Found, µg	% Recovered
R1	16.04	12.62	78.7
R2	16.04	14.28	89.0
R3	16.04	15.07	93.9
R4	16.04	16.97	105.8
R5	16.04	15.85	98.6
R6	16.04	16.43	102.4
			Average = 94.8

2.6. Sample storage

Twelve treated filters were liquid spiked with 8 µL of a 2.005 mg/mL standard and humid air (~80% relative humidity) was drawn through each filter at 1 L/min for 60 minutes. Six of the samples were stored at ambient temperature in a drawer, and six were stored in a freezer. After four days of storage, three samples from each group were extracted with 3 mL of DMF by shaking for 30 min and then analyzed as per section 3.5. The remaining samples were desorbed and analyzed after eight days of storage. The results are given in the tables below.

Table 2.6.1. Ambient Storage			
Days Stored	Amount Spiked, µg	Amount Found, µg	% Recovered
4	16.04	16.27	101.4
4	16.04	15.64	97.5
4	16.04	Lost in analysis	
8	16.04	13.23	82.5
8	16.04	12.85	80.1
8	16.04	17.11	106.7
		Average of 4 days	99.4
		Average of 8 days	89.8

Table 2.6.2. Refrigerated Storage			
Days Stored	Amount Spiked, µg	Amount Found, µg	% Recovered
4	16.04	17.16	107
4	16.04	17.15	107
4	16.04	17.2	107.2
8	16.04	13.06	81.4
8	16.04	17.1	106.6
8	16.04	15.84	98.8
		Average of 4 days	107
		Average of 8 days	95.6

2.7. Recommended air volume and sampling rate

2.7.1. The recommended air volume is 60 L.

2.7.2. The recommended flow rate is 1.0 L/min.

2.8. Interferences

It is not known if any compounds will interfere with the collection of TGI. Any collected compound that reacts with the HBr may compete for the derivatizing reagent on the filter.

2.9. Safety precautions

2.9.1. Attach the sampling equipment in such a manner that it will not interfere with work performance or employee safety.

2.9.2. Follow all safety practices that apply to the work area being sampled.

3. Analytical procedure

3.1. Apparatus

3.1.1. A balance capable of weighing to the nearest tenth of a milligram. A Mettler HL52 balance was used in this evaluation.

3.1.2. Mechanical shaker.

3.1.3. A gas chromatograph (GC) equipped with an electron capture detector (ECD). A Hewlett Packard 5890 was used in this evaluation.

3.1.4. A GC column capable of separating TGI from any interferences. A 10 m x .32 mm i.d. (1.0 µm film) DB-5 column was used in this evaluation.

3.1.5. An electronic integrator, or some other suitable method for measuring detector response. The Hewlett-Packard 3357 Laboratory Data System was used in this evaluation.

3.1.6. Volumetric flasks and pipets.

3.1.7. Vials, 4-mL with Teflon-lined caps.

3.1.8. Vials, 2-mL suitable for use on GC autosamplers.

3.2. Reagents

3.2.1. Dimethylformamide (DMF), high purity Burdick and Jackson

3.2.2. 1,3,5-Triglycidyl isocyanurate, Polysciences, Inc.

3.2.3. Iso-octane, HPLC grade, Fisher Scientific Co.

3.2.4. Heptafluorobutyric anhydride (HFBAnh), Pierce Chemical Co.

3.2.5. High purity water, Milli-Q filtered water, Millipore Inc.

3.2.6. Hydrobromic acid, 48% solution, Fisher Scientific Co.

3.3. Standard preparation

Prepare stock TGI standards by weighing 10 to 14 mg of TGI. Transfer the TGI to separate 10-mL volumetric flasks, and add DMF containing HBr (three drops 48% HBr in 10 mL of DMF) to the mark. Make working range standards of 0.4 to 10 µg/mL by pipet dilutions of the stock standards with DMF. This range corresponds to 1.2 to 30 µg per sample when an extraction volume of 3 mL is used. Store stock and dilute standards in a freezer.

3.4. Sample preparation

3.4.1. Transfer the glass fiber filter of each cassette to a 4-mL vial.

3.4.2. Pipet 3.0 mL of DMF into each vial and seal with PTFE-lined caps.

3.4.3. Shake the vials for 30 minutes on a mechanical shaker.

3.5. Derivatization of samples and standards

3.5.1. Transfer 20 µL of each sample and standard to 4-mL vials containing 2 mL of iso-octane.

3.5.2. Add 25 µL of HFBAnh to each vial.

3.5.3. Cap the vials and shake for 10 to 15 s to ensure mixing and allow them to sit at room temperature for 15 min.

3.5.4. Add 1 mL of filtered water to each vial.

3.5.5. Recap the vials and shake for 15 s.

3.5.6. After allowing the layers to separate, transfer the iso-octane (upper) layer to 2-mL vials for analysis by GC.

3.6. Analysis

3.6.1. Instrument conditions

Column:	DB-5, 1.0 µm film, 10 m x 0.32 mm i.d.
Injector temperature:	235 °C
Column temperature:	225 °C
Detector temperature:	300 °C
Gas flows:	Column 8.6 mL/min hydrogen
Make up:	42 mL/min nitrogen
Injection volume:	1.0 µL
Split ratio:	5:1
Retention time:	5.99 min

3.6.2. Chromatogram (see Figure 2)

3.7. Interferences

3.7.1. Any collected compound having a similar retention time and responds to an ECD is an interference.

3.7.2. Any compound that reacts with HFBAnh is an interference.

3.7.3. GC conditions may be varied to circumvent an interference.

3.7.4. Retention time alone is not proof of chemical identity. Analysis by an alternate GC column and confirmation by mass spectrometry are additional means of identification.

3.8. Calculations

3.8.1. A calibration curve (Figure 3) is constructed by plotting detector response versus total µg of TGI in 3 mL of volume.

3.8.2. The amount of TGI in a sample is determined from the calibration curve.

3.8.3. The air concentration is then determined by the following formula.

$$\text{mg/m}^3 = \frac{(\text{total µg in sample})}{(\text{air volume sampled}) \times (\text{extraction efficiency})}$$

3.9. Safety precautions

3.9.1. Avoid skin contact and air exposure to TGI.

3.9.2. Avoid skin contact with all solvents.

3.9.3. Wear safety glasses at all times.

4. Recommendation for further study

The method should be fully validated.

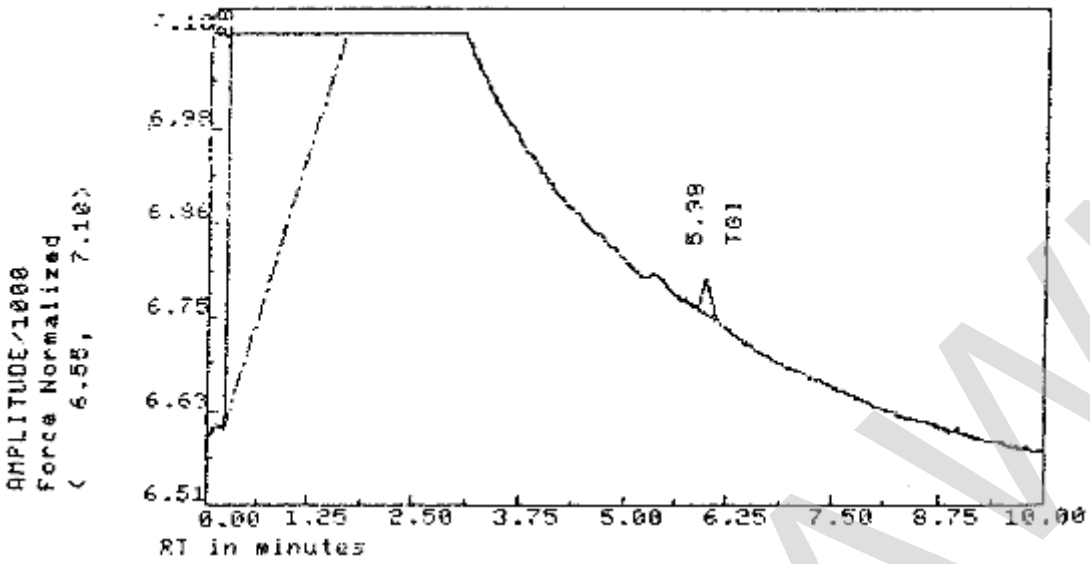


Figure 1
Chromatogram at the Detection Limit

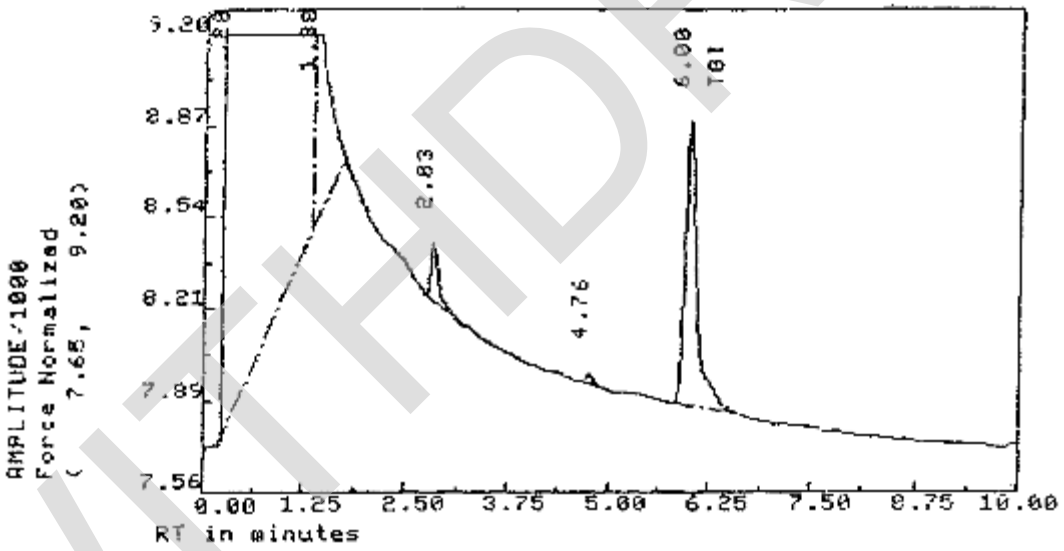


Figure 2
Chromatogram of TGI

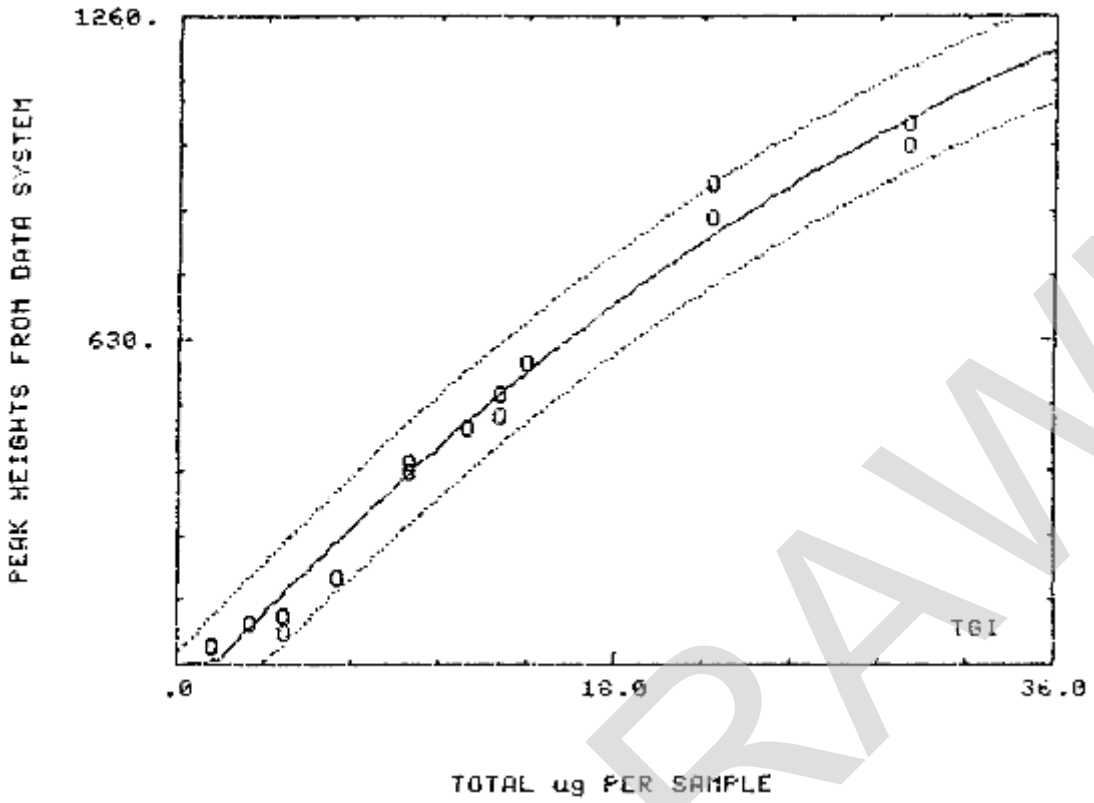


Figure 3
Calibration Curve

5. References

5.1. *Kirk-Othmer Encyclopedia of Chemical Technology*; John Wiley & Sons: New York, 1981, Volume 7, PP 400-401.

5.2. *Kirk-Othmer Encyclopedia of Chemical Technology*; John Wiley & Sons: New York, 1981, Volume 9, PP 272-277.

5.3. *"1,3,5-Triglycidyl Isocyanurate"* Material Safety Data Sheet, Polysciences Inc., Warrington, Pennsylvania