Method number:	1024
Version:	1.0
Target concentration:	0.05 mg/m^3
OSHA PEL:	Not established
ACGIH TLV:	0.05 mg/m^3 8-hour TWA
Procedure:	Active samples are collected by drawing workplace air through cassettes containing 37-mm glass fiber filters with personal sampling pumps. Samples are extracted with acetonitrile and analyzed by gas chromatography (GC) using a flame ionization detector (FID).
Recommended sampling time:	180 min
Sampling rate:	1.0 L/min (180 L)
Reliable quantitation limit:	4.94 μg/m^3
Standard error of estimate:	6.0%
Special requirements:	Samples must be shipped cold overnight and stored in a freezer until analysis.
Status of method:	Fully validated method. This method has been subjected to the established validation procedures of the Methods Development Team.

June 2020

Radhakrishnan Ukkiramapandian

Methods Development Team
Industrial Hygiene Chemistry Division
OSHA Salt Lake Technical Center
Sandy UT 84070-6406

1. General Discussion

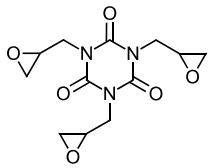
For assistance with accessibility problems in using figures and illustrations presented in this method, please contact the Salt Lake Technical Center (SLTC) at (801) 233-4900. This procedure was designed and tested for internal use by OSHA personnel. Mention of any company name or commercial product does not constitute endorsement by OSHA.

1.1 Background

1.1.1 History

OSHA has routinely collected samples for 1,3,5-triglycidyl-s-triazinetrione (TGIC) using OSHA method PV2055 which uses hydrobromic acid coated glass fiber filters (HBr/GFF).[1] The samples are extracted with *N,N*-dimethylformamide (DMF), further derivatized with heptaflurobutyric anhydride (HFBA), and analyzed using gas chromatography with electron capture detection.[1] The derivatization steps were included to improve the stability of the analyte, and analytical sensitivity. Upon later study of this TGIC analysis process, it was observed that when using HBr/GFF media the TGIC derivatization does not occur on the filter during and after sample collection, but happens only during the extraction process. Also, these studies led to the finding that underivatized TGIC is reasonably stable on GFF media obtained from one of two commercial sources, and collection of TGIC on uncoated GFF media followed by analysis of the underivatized chemical using gas chromatography with flame ionization detection (GC-FID) was possible. The GFF media obtained from the second source gave unsatisfactory storage stability results.

1.1.2 Physical properties and descriptive information[2,]

analyte:	1,3,5-triglycidyl-s-triazinetrione
synonyms:	1,3,5-triglycidyl isocyanurate (TGIC), tris(2,3-epoxypropyl)isocyanurate
solubility:	soluble in *N,N*-dimethylformamide, acetonitrile, dimethyl sulfoxide
IMIS number:	T405
CAS number:	2451-62-9
molecular weight:	297.26
melting point:[2]	95 °C
formula:	$C_{12}H_{15}N_3O_6$
appearance:	white powder
structure:	

[1] Lee, D. 1,3,5-Triglycidyl Isocyanurate (TGIC) (OSHA Method PV2055), 1988. United States Department of Labor, Occupational Safety & Health Administration website. https://www.osha.gov/dts/sltc/methods/partial/pv2055/2055.pdf (accessed June 2020).

[2] Safety Data Sheet 379506, Tris(2,3-epoxypropyl) isocyanurate (TGIC), Sigma-Aldrich, St. Louis, MO. May 27, 2016. https://www.sigmaaldrich.com/MSDS/MSDS/DisplayMSDSPage.do?country=US&language=en&productNumber=379506&brand=ALDRICH&PageToGoToURL=https%3A%2F%2Fwww.sigmaaldrich.com%2Fcatalog%2Fproduct%2Faldrich%2F379506%3Flang%3Den. (accessed June 2020)

2. Sampling Procedure

All safety practices that apply to the work area being sampled should be followed. The sampling equipment should be attached to the worker in a manner that will not interfere with work performance or safety.

2.1 Apparatus

Collect active samples with a 2-piece closed-face cassette, containing a 37-mm diameter type A/E glass fiber filter (GFF) and a support pad. For this validation, commercially prepared filters were purchased from SKC, Inc., catalog no. 225-7, lot no. 17206-7E0-179. While the use of other similar GFF media is possible, verification of sample storage stability is required.

Collect samples using a personal sampling pump calibrated to within ±5% of the recommended flow rate with the sampling device in-line.

2.2 Reagents

None required

2.3 Technique

Immediately before sampling, remove the plastic end plugs from the cassette.

Attach the cassette to the sampling pump so that it is in an approximately vertical position with the inlet facing down during sampling. Position the sampling pump, cassette, and tubing so they do not impede work performance or safety.

Draw the air to be sampled directly into the inlet of the cassette. The air being sampled should not pass through any hose or tubing before entering the cassette.

Sample for up to 180 min at 1.0 L/min (180 L) to collect time weighted average samples.

After sampling for the appropriate time, remove the cassette and seal with plastic end plugs. Seal each sample end-to-end with a Form OSHA-21 as soon as possible.

Submit at least one blank sample with each set of samples. Handle the blank sample in the same manner as the other samples except draw no air through it.

Record sample air volume (liters), sampling time (min) and sampling rate (mL/min) for each sample, along with any potential interferences on the Form OSHA-91A.

Submit the samples to the laboratory for analysis as soon as possible after sampling using next day delivery with freezer packs. If a delay is unavoidable, store the samples in a freezer.

3. Analytical Procedure

3.1 Apparatus

Gas chromatograph equipped with a flame ionization detector. An Agilent 6890 Series GC System was used in this validation.

GC injection port liner. Restek Sky 4.0-mm ID Topaz, Split Precision Inlet Liner w/wool (Restek catalog no. 23305, or equivalent) was used in this validation.

GC column capable of separating TGIC from the extraction solvent, potential interferences, and internal standard. An Agilent J&W HP-5, 30-m × 0.32-mm i.d. (film thickness 0.25-µm) capillary column was used in this validation.

Electronic integrator or other suitable means of measuring GC detector response. A Waters Empower 3 Data System was used in this validation.

Glass vials (clear) with PTFE-lined crimp caps. In this validation, 2-mL vials were used.

Glass vials (amber) with PTFE-lined screw caps. In this validation, 4-mL vials were used.

A dispenser capable of delivering 3.00 mL of extraction solvent to prepare standards and samples. If a dispenser is not available, 3-mL volumetric pipettes can be used.

Class A volumetric flasks of convenient sizes for standard preparation. In this validation 5-mL, and 10-mL flasks were used.

Calibrated microliter syringes of convenient sizes for standard preparation. Several SGE syringes of 10-µL, 25-µL and 100-µL volumes were used in this validation.

Mechanical rotator. A Thermo Scientific Labquake Shaker Rotisserie, Model 415110 was used in this validation.

3.2 Reagents

Acetonitrile (ACN), [CAS no. 75-05-8], reagent grade or better.

p-Cymene, [CAS no. 99-87-6], reagent grade or better.

1,3,5-triglycidyl-s-triazinetrione (TGIC), [CAS no. 2451-62-9], reagent grade or better.

Extraction solvent. The extraction solvent used in this method consists of 0.1 µL/mL of *p*-cymene in ACN. The *p*-cymene was added as an internal standard (ISTD). The extraction efficiency is affected by the extraction solvent, the ISTD, the sampling medium, and the technique used to extract the samples. Reagents and other techniques than those described in this method can be used provided they are tested as specified in the validation guidelines.[3]

3.3 Standard preparation

Prepare a concentrated stock solution of TGIC by weighing 9.00 mg of TGIC into a 10-mL volumetric flask and then fill to the mark with ACN. Prepare working analytical standards by injecting microliter amounts of concentrated stock solution into 4-mL vials containing 3.00 mL of extraction solvent delivered from the same dispenser used to extract samples. For example, to prepare a target level standard based on sampling at the recommended sampling rate for the recommended time, inject 10.00 µL of a stock solution containing 0.90 mg/mL of TGIC into a vial containing 3.00 mL of extraction solvent. Prepare five calibration standards for TGIC with concentration between 0.900 to 25.0 µg/sample.

Bracket the sample concentrations with standard concentrations. If upon analysis, sample concentrations fall outside the range of prepared standards, prepare and analyze additional standards to confirm instrument response, or dilute high samples with extraction solvent and reanalyze the diluted samples.

[3] Eide, M.; Simmons, M.; Hendricks, W. Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis, 2010. United States Department of Labor, Occupational Safety & Health Administration Web site. http://www.osha.gov/dts/sltc/methods/chromguide/chromguide.pdf (accessed June 2020).

3.4 Sample preparation

Remove the plastic end plugs, open the cassette, and carefully transfer the glass fiber filter to a 4-mL glass vial so that the filter is flat against the inside surface of the vial (not folded or crumpled). The exposed filter surface must face inward in contact with the extraction solvent, not toward the glass.

Wet a glass fiber filter with 100.0 µL of acetonitrile, wipe the cassette walls, and place in a separate vial as above.

Add 3.00 mL of extraction solvent to each vial and immediately seal the vials with PTFE-lined caps.

Extract the sample by rotating for 60 min in a mechanical rotator at 40 rpm.

Fill a 2-mL vial with the sample extract and seal with a PTFE-lined cap.

3.5 Analysis

3.5.1 Analytical conditions

GC conditions

oven temperature:	80 ˚C (hold 1 min), ramp at 20 ˚C/min to 240 ˚C (hold 5.0 min)
injection conditions:	250 ˚C, 1.0 µL, 10 to 1 split
run time:	14 min
column:	Agilent J&W HP-5 capillary column, 30-m × 0.32 mm i.d., d_f = 0.25 µm, or equivalent
column mode:	constant pressure (7.9 psi)
initial column gas flow:	3.0 mL/min (hydrogen)
septum purge:	3.0 mL/min (hydrogen)
inlet liner:	Restek Sky 4.0-mm ID Topaz, Split, Precision Inlet Liner w/wool (Restek catalog no. 23305, or equivalent)
retention times:	1.09 min (ACN) 2.57 min (ISTD) 11.95 min (TGIC)

FID conditions

detector temperature:	300 ˚C
hydrogen flow:	40 mL/min
air flow:	450 mL/min
nitrogen make up flow:	45 mL/min

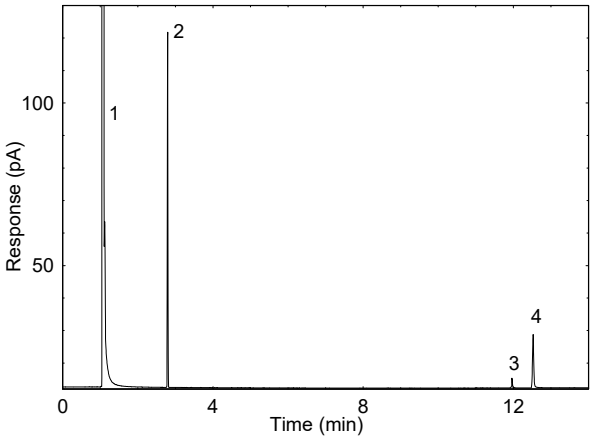


Figure 3.5.1. Chromatogram obtained at the target concentration for samples spiked on filters with the recommended analytical conditions (1: ACN; 2: p-Cymene (ISTD); 3: TGIC; 4: di(2-ethylhexyl) adipate or DEHA, CAS no. 103-23-1, artifact present on sampling media).

3.5.2 Calibration

An ISTD calibration method is used. A linear calibration curve can be constructed by plotting ISTD-corrected response of standard injections versus micrograms of analyte per sample. When adding reporting limit standards to the calibration, the curves can be weighted to reduce bias at the reporting limit. Bracket the samples with freshly prepared analytical standards over a range of concentrations.

3.6 Interferences (analytical)

3.6.1 Any compound that produces a GC response and has a similar retention time as the analyte or internal standard is a potential interference. If any potential interferences are reported, they should be considered before samples are extracted. Generally, chromatographic conditions can be altered to separate interferences from the analyte.

3.6.2 When necessary, the identity of an analyte peak can be confirmed with additional analytical data or procedures (Section 4.11).

3.7 Calculations

Obtain the micrograms per sample value (M) for each analyte. If any analyte is found on the cassette wipe filter, it is added to the amount found in the sampled filter. This total amount (M_{obs}) is corrected by subtracting the amount (if any) found on the blank. The air concentration C_M is calculated using the following formulas.

$$M = \frac{M_{obs} - M_{blk}}{E_E}$$

Where
M is micrograms per sample blank corrected and extraction corrected
M_{obs} is micrograms per sample
M_{blk} is micrograms per sample blank
E_E is extraction efficiency, in decimal form

$$C_M = \frac{M}{V}$$

Where
C_M is concentration by weight in air (mg/m^3)
V is liters of air sampled

4. Method Validation

General instruction for the laboratory validation of OSHA sampling and analytical methods that employ chromatographic analysis is presented in "Validation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis."[3] These Guidelines detail required validation tests, show examples of statistical calculations, list validation acceptance criteria, and define analytical parameters. Air concentrations listed in ppm are referenced to 25.0 ˚C and 760 mmHg (101.3 kPa).

4.1 Detection limit of the analytical procedure (DLAP)

The DLAP is measured as the mass of analyte introduced onto the chromatographic column. Ten analytical standards were prepared with equally descending increments of TGIC with the highest standard containing 0.507 µg/mL. This is the concentration that would produce a peak approximately 10 times the response of a reagent blank at or near the chromatographic retention time of the analyte. These standards and the reagent blank were analyzed with the recommended analytical parameters (1.0-µL injection with a 10:1 split). The data obtained were used to determine the required parameters (standard error of estimate and slope) for the calculation of the DLAP. Values of 4.22 and 9.12 were obtained for the slope and standard error of estimate, respectively. The DLAP was calculated to be 6.48 pg.

Table 4.1
Detection Limit of the Analytical Procedure data for TGIC

concentration (µg/mL)	mass on column (pg)	area counts (µV●s)
0.00	0.00	00.0
0.0500	5.00	13.6
0.100	10.0	17.7
0.150	15.0	61.0
0.203	20.3	62.0
0.253	25.3	84.1
0.303	30.3	118.4
0.353	35.3	135.2
0.403	40.3	157.0
0.453	45.3	183.5
0.507	50.7	209.4

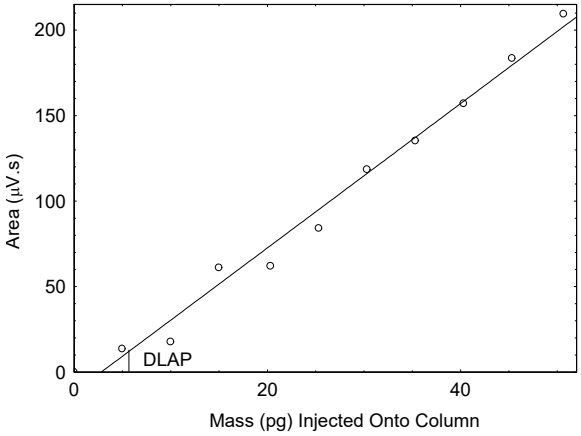


Figure 4.1. Plot of data to determine DLAP for TGIC (y = 4.22x – 11.8).

4.2 Detection limit of the overall procedure (DLOP) and reliable quantitation limit (RQL)

The DLOP is measured as mass per sample and expressed as an equivalent air concentration based on the recommended sampling parameters. Ten GFF samplers were spiked with equally descending increments of analyte, such that the highest sampler loading for TGIC was 3.61 µg/sample. This is the amount spiked on a sampler that would produce a peak approximately 10 times the response of a sample blank at or near the chromatographic retention time of the analyte. These spiked samplers and the sample blank were analyzed with the specified analytical parameters, and the data obtained were used to calculate the standard error of estimate and the slope used for calculation of the DLOP. Values of 148 and 13.2 were obtained for the slope and standard error of estimate, respectively. The DLOP was calculated to be 0.267 µg/sample (1.48 $\mu g/m^3$).

Table 4.2
Detection Limit of the Overall Procedure data for TGIC

mass per sample (µg)	area counts (µV•s)
0.00	00.0
0.361	53.3
0.722	135.8
1.08	140.2
1.44	207.8
1.81	273.0
2.17	334.0
2.53	381.8
2.89	425.0
3.25	493.4
3.61	530.3

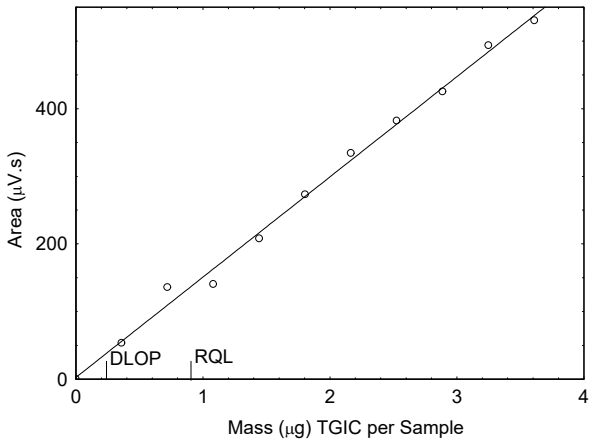


Figure 4.2.1. Plot of data to determine the DLOP for TGIC (y = 148x + 2.82).

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters that were obtained for the calculation of DLOP, providing 75% to 125% of the analyte is recovered. The RQL was calculated to be 0.889 µg/sample (4.94 µg/m^3). Recovery at this concentration was 89.7%.

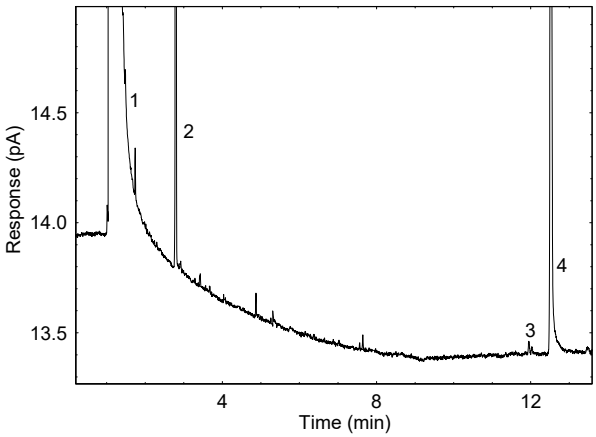


Figure 4.2.2. Chromatogram of the RQL
(1: ACN; 2: p-cymene (ISTD); 3: TGIC, 4: DEHA).

4.3 Precision of the analytical method

The precision of the analytical method, measured as the mass equivalent to the standard error of estimate was determined from the linear regression of data points from standards over a range that covers approximately 0.1 to 2× the target concentration. Calibration curves were constructed from three injections of five standards and were not weighted. The standard error of estimate was determined to be 0.671 µg.

Table 4.3.
Instrument Calibration Area Ratio for TGIC

× target concn (µg/sample)	0.1× 0.939	0.5× 4.69	1.0× 9.36	1.5× 14.0	2.0× 18.6
Peak/ISTD	0.00100	0.00574	0.01226	0.01925	0.02389
	0.00091	0.00544	0.01221	0.01865	0.02500
	0.00128	0.00558	0.01162	0.01924	0.02367

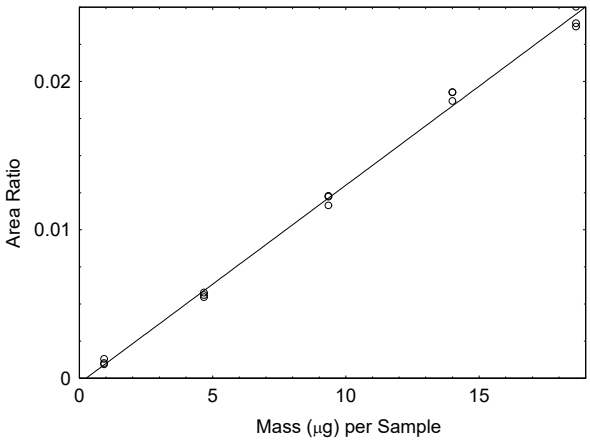


Figure 4.3. Calibration curve for TGIC
(y = 0.00130x – 0.000400).

4.4 Storage stability test

Storage samples for TGIC were prepared by liquid spiking samplers with TGIC, equivalent to sampling for at the specified flow rate and specified time at the target concentration. Following spiking, humidified air (81.0% relative humidity at 22.0 ˚C) was drawn through each cassette for 180 minutes at a flow rate of 1.0 L/min. Freezer storage was studied after ambient temperature and refrigerated storage tests demonstrated a lack of sample stability with those storage conditions. Thirty-three storage samples were prepared for this test. Three samples were analyzed on the day of generation. Fifteen samples were stored at reduced temperature (-20.0 ˚C) and the other fifteen were stored in a closed drawer at ambient temperature (about 22.0 ˚C). At 3-4 day intervals, three samples were selected from each of the two storage sets and analyzed. Sample results are not corrected for extraction efficiency. Samples stored at ambient temperature were stable for 3 days, while freezer-stored TGIC samples were stable for 17 days.

Using the regression equation obtained from the freezer (-20.0 ˚C) storage test, the calculated TGIC recovery was 99.3% at day 17 (not corrected for extraction efficiency).

Table 4.4
Storage Test for TGIC

time (days)	ambient storage recovery (%)			freezer storage recovery (%)		
0	97.6	97.1	100.3	97.6	97.1	100.3
3	95.6	97.1	99.7	104.1	106.4	103.4
7	86.5	83.3	88.2	103.4	101.8	102.9
10	81.1	82.6	80.0	103.4	103.4	102.1
13	81.6	67.1	69.1	100.6	97.6	98.1
17	57.2	62.4	61.5	94.0	96.2	102.4

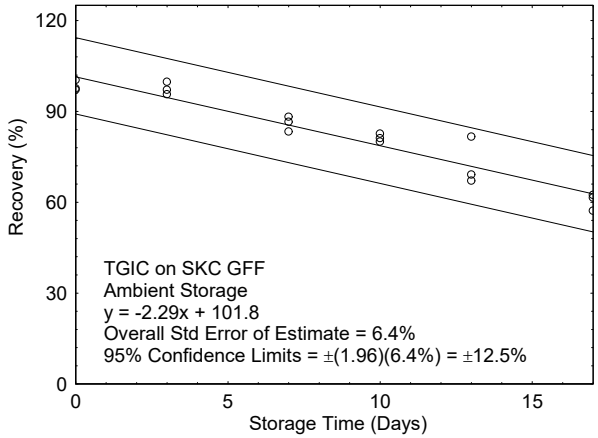


Figure 4.4.1. Ambient storage test for TGIC on SKC 225-7 GFF.

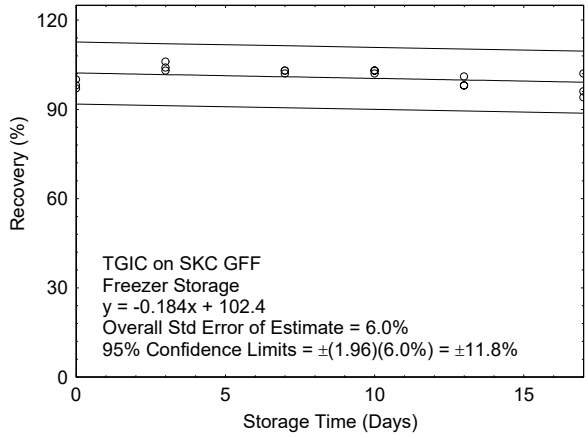


Figure 4.4.2. Freezer storage test for TGIC on SKC 225-7 GFF.

4.5 Precision (overall procedure)

The precision of the overall procedure (overall standard error of estimate) statistic considers variability in sampling, filter handling and solvent extraction, and instrumental analysis. The standard error of estimate related to the variation about the freezer storage test regression line described in Section 4.4 ($S_{y/x(sto)}$, 3.24%) and sampling pump variability (V_{sp}, 5%) were used to determine the precision of the overall procedure.

The precision of the overall procedure was calculated by taking the square root of the combined value of the squared freezer stability study standard error of estimate ($S^2_{y/x(sto)}$), and the squared sampling pump variability value (V^2_{sp}). The precision of the overall procedure for the -20.0 ˚C, 17-day storage test (at the target concentration) for TGIC on GFF was determined to be ± 6.0%. The precision of the overall procedure at the 95% confidence level was obtained by multiplying this value by 1.96 (the z-statistic from the standard normal distribution at the 95% confidence level), to arrive at a value of ± 11.4%. The 95% confidence intervals for the regression lines in both storage stability test figures provided in Section 4.4 were drawn following this approach.

4.6 Reproducibility

Six samples were prepared by liquid spiking samplers with TGIC, equivalent to sampling at the specified flow rate for the specified time at the target concentration. Following spiking, humidified air (81.0% relative humidity at 22.0 ˚C) was drawn through each cassette for 180 minutes at a flow rate of 1.0 L/min. The samples were submitted to the OSHA Salt Lake Technical Center for analysis using the procedure described in this method. The samples were stored for 7 days at -20.0 ˚C before analysis. Sample results were corrected for extraction efficiency. No sample result for TGIC deviated beyond the 95% confidence interval precision boundaries of the overall procedure as determined in Section 4.5.

Table 4.6
Reproducibility Data for TGIC

theoretical (µg/sample)	Recovered (µg/sample)	recovery (%)	deviation (%)
8.40	7.56	90.0	-10.0
8.40	8.23	98.0	-2.0
8.40	7.88	93.8	-6.2
8.40	8.17	97.3	-2.7
8.40	7.98	95.0	-5.0
8.40	7.64	91.0	-9.0

4.7 Sampler capacity

A controlled test atmosphere was not generated for this aerosol-type analyte. See Section 4.9 for retention data that support the recommended air sample volume.

4.8 Extraction efficiency and stability of extracted samples

The extraction efficiency is affected by the extraction solvent, the ISTD, the sampling medium, and the technique used to extract the samples. Other reagents and techniques than described in this method can be used provided they are tested as specified in the validation guidelines.[3]

4.8.1 Extraction efficiency

The extraction efficiency of TGIC was determined by liquid-spiking four glass fiber filters with TGIC, equivalent to sampling at the specified flow rate for the specified time at each concentration level tested. Wet filters were also prepared by drawing humid air (81.0% RH and 22.0 ˚C) through filters at 1.0 L/min for 180 min prior to spiking. Samples were stored overnight in a freezer and then analyzed. The overall mean extraction efficiency over the working range of 0.1 to 2 times the target concentration was 102.1%. The extraction efficiency at the RQL was 99.0%. The presence of water did not have an unacceptable effect on extraction efficiency. The extraction efficiencies at the RQL and for wet samplers were not included in the overall mean. The data obtained are shown in Table 4.8.1.

Table 4.8.1
Extraction Efficiency of TGIC

Level		sample number				
× target Concn	µg per sample	1	2	3	4	mean
0.1	0.940	97.7	97.3	102.3	101.0	99.6
0.25	2.35	102.9	101.7	101.7	97.9	101.1
0.5	4.70	101.7	101.9	104.8	100.8	102.3
1.0	9.39	100.5	102.3	101.9	101.4	101.5
1.5	14.1	105.6	106.5	102.1	102.9	104.3
2.0	18.8	101.4	101.4	106.2	105.7	103.7
RQL	0.880	94.6	104.1	96.8	100.3	99.0
1.0 (wet)	9.39	104.4	105.8	104.0	106.2	105.1

4.8.2 Stability of extracted samples

The stability of extracted samples was examined by reanalyzing the 1.0× target concentration samples 24, 48, and 72 hours after the initial analysis. After the original

analysis was performed, two vials were recapped with new septa, which were replaced after each reanalysis. The remaining two vials retained their sequentially punctured septa throughout the test. All samples were allowed to stand at room temperature in the autosampler tray at 21.0 ˚C. The samples were reanalyzed using freshly prepared standards for each reanalysis. The term "diff" in Table 4.8.2 refers to the difference in percent recovery between the initial analysis and the subsequent analysis. Each septum was punctured four times for each injection (three syringe rinses and one syringe fill that was injected for analysis).The data obtained are shown in Table 4.8.2.

Table 4.8.2
Extracted Solutions Stability of TGIC

punctured septa replaced							punctured septa retained						
initial (%)	24h (%)	diff (%)	48h (%)	diff (%)	72h (%)	diff (%)	initial (%)	24h (%)	diff (%)	48h (%)	diff (%)	72h (%)	diff (%)
100.5	99.1	-1.4	100.3	-0.2	105.0	4.5	101.9	102.9	1.0	99.8	-2.1	105.4	3.5
102.3	102.8	0.5	97.8	-3.5	97.4	-3.9	101.4	104.1	2.7	102.4	1.0	102.3	0.9
			Mean							Mean			
101.4	101.0	-0.4	99.1	-1.8	101.2	0.3	101.6	103.5	1.8	101.1	-0.6	103.8	2.2

4.8.3 Support pad

Five support pads were spiked with 0.861 μg of TGIC. The spiked support pads were extracted with 3.00 mL of extraction solution. Analysis results provided recoveries of 116.1%, 114.9%, 94.6%, 96.7% and 118.6%, with a mean recovery value of 108.2%.

4.9 Sampling Interferences

Sampling interferences were tested using retention, low humidity and low concentration tests mentioned in the validation guidelines[3] and the results are reported here.

4.9.1 Retention

Retention was tested by spiking six glass fiber filters with TGIC, equivalent to sampling at the specified flow rate for the specified time at 2.0× the target concentration (22.0 μg). The TGIC was loaded into separate 3-piece cassettes, each with a back filter and a support pad. The spiked samplers were then used to sample 210 L of humid air (80.0% relative humidity at 22.5 ˚C) at 1.0 L/min. Both filters in each cassette were immediately extracted and analyzed separately. The average retention of TGIC on the 1st filters was 97.7%, and no breakthrough to the back filters was observed. The data obtained are shown in Table 4.9.1.

Table 4.9.1
Retention Efficiency of TGIC

	recovery (%)						
Set	1	2	3	4	5	6	Mean
front	96.4	94.8	97.1	99.3	101.0	97.5	97.7
back	0	0	0	0	0	0	0
total	96.4	94.8	97.1	99.3	101.0	97.5	97.7

4.9.2 Low humidity

The effect of low humidity was tested by spiking four glass fiber filters with TGIC, equivalent to sampling at the specified flow rate for the specified time at 2.0× the target concentration (22.0 µg). The TGIC was loaded into separate 3-piece cassettes, each with a back filter and a support pad. Dry air was then drawn through the spiked samplers at 1.0 L/min for 180 min (20.0% relative humidity at 21.8 ˚C). Both filters in each cassette were immediately extracted and analyzed separately. Analysis results provided recoveries of 92.3%, 93.3%, 94.2%, and 93.8%. The average recovery of TGIC from the 1st filters was 93.4%, and no breakthrough to the back filters was observed.

4.9.3 Low concentration

The effect of low concentration was tested by spiking six glass fiber filters with TGIC, equivalent to sampling at the specified flow rate for the specified time at 0.1× the target concentration (1.10 µg). The TGIC was loaded into separate 3-piece cassettes, each with a back filter and a support pad. Humid air was then drawn through the spiked samplers at 1.0 L/min for 180 min (83.0% relative humidity at 20.1 ˚C). Both filters in each cassette were immediately extracted and analyzed separately. Analysis results provided recoveries of 91.5%, 97.0%, 94.3%, 91.4%, 95.6% and 94.0%. The average recovery of TGIC from the 1st filters was 94.0%, and no breakthrough to the back filters was observed.

4.10 Cassette wipe

Four blank cassettes were spiked with an ACN solution of 14.4 µg of TGIC (10.00 µL of a 1.44 mg/mL TGIC solution). The cassettes were allowed to air dry and then stored in the freezer overnight. Each cassette was wiped with 37-mm glass fiber filters wetted with 0.1 mL of ACN. Cassette walls were wiped multiple times to determine how many wipes would be needed to recover 75% of the spiked material. The filters were placed in separate vials, extracted and analyzed as described in section 3.5. The results obtained are presented in Table 4.9.2.

Table 4.9.2
Cassette Wipe Recovery of TGIC

cassette	wipe	recovery (%)
1	first	93.0
	second	0.0
	third	0.0
2	first	93.8
	second	0.0
	third	0.0
3	first	91.3
	second	0.0
	third	0.0
4	first	95.3
	second	0.0
	third	0.0

4.11 Qualitative analysis

When necessary, the identity or purity of an analyte peak can be confirmed by gas chromatography-mass spectrometry (GC-MS) or by another analytical procedure. For the levels analyzed in this method, use of selective ion monitoring mode (SIM) is recommended. The SIM chromatogram for TGIC obtained at the target concentration using SIM parameters is shown in

Figure 4.11.1.

A diagnostic mass spectrum of TGIC may be obtained using scan mode, if sufficient analyte is present. A total ion chromatogram (TIC) and mass spectrum are shown in Figures 4.11.2 and 4.11.3 respectively.

GC conditions

oven temperature:	80 °C (hold 1 min), ramp to 240 °C at 35 °C/min (hold 11 min)
injection conditions:	250 °C, 1.0 µL, 5 to 1 split
run time:	16.6 min
column:	Agilent HP-5 MS, 30-m × 0.25-mm i.d., (0.25-µm d_f), or equivalent
column mode:	constant flow (initial avg. velocity 35 cm/sec)
initial column gas flow:	0.9 mL/min (helium)
septum purge:	3.0 mL/min (helium)
inlet liner:	Restek Sky 4.0 mm ID Low Pressure Drop Precision Inlet Liner w/wool (Restek catalog no. 23309.1, or equivalent)
retention times:	10.6 min (TGIC) 11.1 min (DEHA)

MS conditions

mode:	Electron Ionization
solvent delay:	2.0 min
timed events:	None
EMV mode:	gain factor 5
MS source:	230 °C
MS quadrupole:	150 °C
MSD transfer line:	300 °C
scan parameters:	*m/z* 10-500
SIM parameters:	*m/z* 297, 255
dwell time:	250 sec
threshold:	150

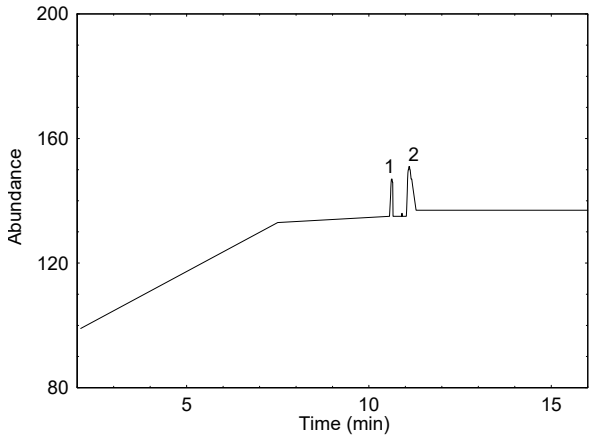


Figure 4.11.1. SIM chromatogram of TGIC at 8 µg/sample (1. TGIC, 2. DEHA)

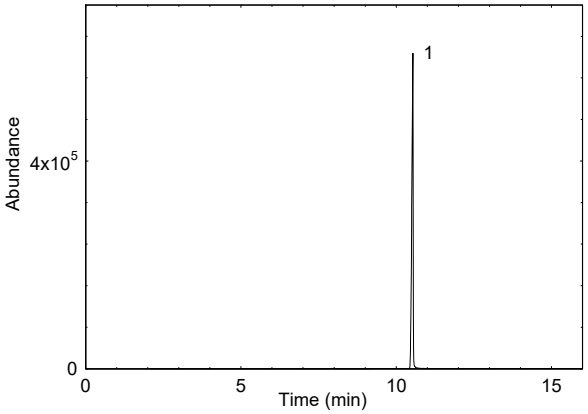


Figure 4.11.2. TIC obtained at the concentration of 3 mg/sample in ACN with the recommended analytical conditions (1: TGIC).

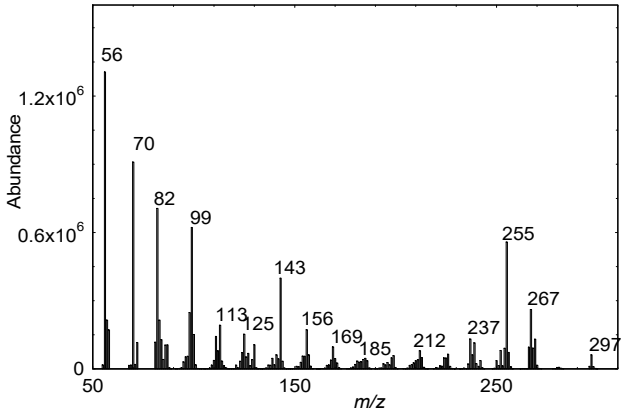


Figure 4.11.3. Mass spectrum of TGIC (scan).