Method number:	PV2141
Version	1.0
Target concentration:	5 mg/m^3
OSHA PEL:	none
ACGIH TLV:	5 mg/m^3
Procedure:	An air sample is collected by drawing a known volume of air through a 37-mm closed-face cassette containing a glass fiber filter. The sample is extracted with 2 mL of acetone and analyzed by gas chromatography using a flame ionization detector.
Recommended sampling time and sampling rate:	100 min at 1 L/min (100 L)
Reliable quantitation limit:	420 µg/m^3
Status of method:	Partially validated method. This method has been subjected to established evaluation procedures of the Methods Development Team and is presented for information and trial use.

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Karl J. Walker
Donna LaGarde

Organic Analytical Services
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 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

This work was performed because OSHA has no sampling and analytical method for triethanolamine.

1.1.2 Toxic effects (This section is for information only and should not be taken as the basis of OSHA policy.)[1]

Acute oral LD_{50} values are reported as 8680 and 9110 mg/kg for rats; 1450 mg/kg for mice and 8000 mg/kg for guinea pigs.

"Triethanolamine has been identified as causing allergic contact dermatitis, erythematous vesicular lesions, eczema, contact dermatitis, and irritation in workers exposed to triethanolamine in their occupations."

"A TLV-TWA of 5 mg/m^3 is recommended to minimize the potential for skin and eye irritation, and acute and chronic effects, following exposure to triethanolamine."

1.1.3 Workplace exposure[2]

"Triethanolamine is used in dry cleaning and wool scouring. It is found in cosmetics, household detergents, metalworking fluids, polishes and emulsions. Triethanolamine is used as an antifoam agent, water repellant, dispersion agent, corrosion inhibitor, softening agent, emulsifier, humectant, plasticizer, chelating agent, solvent, rubber accelerator and pharmaceutical alkalizing agent."

1.1.4 Physical properties and other descriptive information[3]

CAS number:	102-71-6
IMIS[4]:	T185
molecular weight:	149.22
vapor density:	5.14 (air = 1.0)
specific gravity:	1.12 g/mL at 20 °C
vapor pressure:	< 0.01 torr at 20 °C
melting point:	21.2 °C
color:	colorless to pale yellow
odor:	slight ammonia-like odor
state:	liquid but solid below 21.2 °C

[1] Documentation of the Threshold Limit Values and Biological Exposure Indices, 7th ed. American Conference of Governmental Industrial Hygienists: Cincinnati, OH, 2001; Vol. 3.

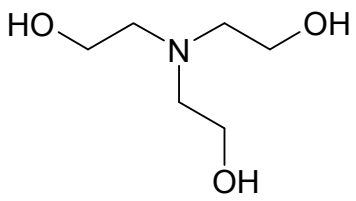
[2] Documentation of the Threshold Limit Values and Biological Exposure Indices, 7th ed. American Conference of Governmental Industrial Hygienists: Cincinnati, OH, 2001; Vol. 3.

[3] Documentation of the Threshold Limit Values and Biological Exposure Indices, 7th ed. American Conference of Governmental Industrial Hygienists: Cincinnati, OH, 2001; Vol. 3.

[4] Triethanolamine (Chemical Sampling Information), 2005. U.S. Department of Labor, Occupational Safety and Health Administration Web Site. http://www.osha.gov/dts/chemicalsampling/data/CH_273550.html (accessed July 2008).

boiling point:	335 °C; decomposition
flash points:	355 °C, (closed cup); 375 °C (open cup) combustible
solubility:	soluble in chloroform, benzene, and ether; miscible with acetone, methanol, and water
reactivity:	reacts vigorously with oxidizing materials
synonyms:	trihydroxytriethylamine; Daltogen; Sterolamide; 2,2,2-nitrilo-triethanol; TEA

structural formula:



This method was evaluated according to the OSHA SLTC "Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis"[5]. The Guidelines define analytical parameters, specify required laboratory tests, statistical calculations and acceptance criteria. The analyte air concentrations throughout this method are based on the recommended sampling and analytical parameters.

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

The DLOP and RQL are measured as mass per sample and expressed as an equivalent air concentration based on the recommended sampling parameters. Ten samplers were spiked with equally descending increments of triethanolamine, such that the highest sampler loading was 130.68 µg. The highest sampler loading is the amount that would produce a peak about 10 times the response for a sample blank. These spiked samplers were analyzed with the recommended analytical parameters and the data obtained were used to calculate a regression line, for which the standard error of estimate (SEE) was determined to be 550 and the slope was determined to be 131.5. The DLOP is calculated as 3 times the SEE divided by the slope, and the RQL is calculated as 10 times the SEE divided by the slope, providing 75% to 125% of triethanolamine is recovered. The DLOP and RQL were 12.6 µg (126 µg/m^3) and 42.0 µg (420 µg/m^3) respectively. The recovery at a mass near the RQL was 87.7%.

[5] Burright, D.; Chan, Y.; Eide, M.; Elskamp, C.; Hendricks, W.; Rose, M. C. *Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis;* OSHA Salt Lake Technical Center, U.S. Department of Labor, Salt Lake City, UT, 1999.

Table 1.2
Detection Limit of the Overall Procedure for Triethanolamine

mass per sample (µg)	area counts (µV·s)
0.00	54
7.26	49
14.52	998
29.04	2557
43.56	4043
58.08	6353
72.60	7828
87.12	10348
101.64	12581
116.60	14548
130.68	16787

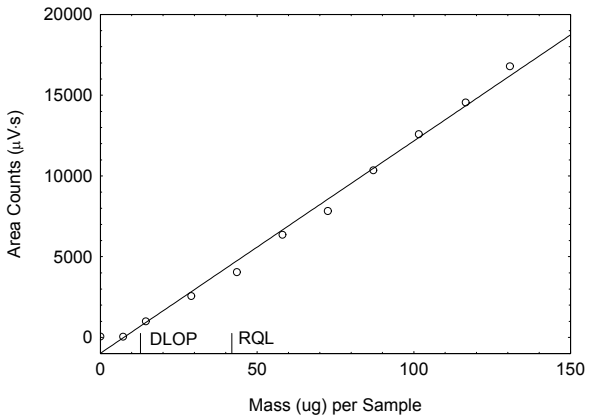


Figure 1.2.1. Plot of data to determine DLOP/RQL. (y = 131.5x - 979)

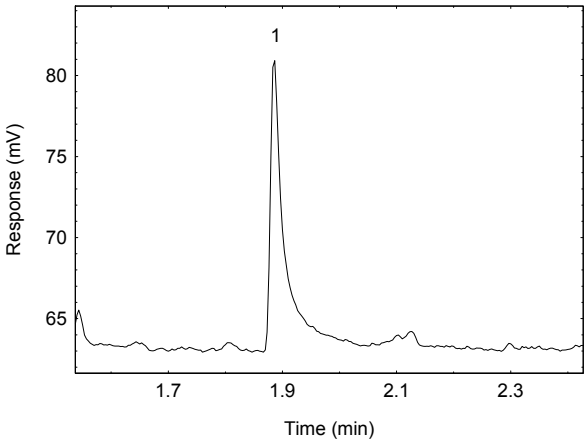


Figure 1.2.2. Chromatogram of triethanolamine at the RQL. [Key: (1 = triethanolamine)]

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 such a manner that it will not interfere with work performance or safety.

2.1 Apparatus

Samples are collected using a personal sampling pump calibrated, with the sampling device attached, to within ±5% of the recommended flow rate.

Samples are collected with 2-piece closed-face cassettes containing glass fiber filters (GFF); Type A/E; 37 mm diameter; Pall Life Sciences; part no. 61652 (lot 70361).

2.2 Reagents

None required

2.3 Technique

Remove the plastic end plugs from the filter cassette immediately before sampling.

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 it does not impede work performance or safety.

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

After sampling for the appropriate time, remove the sample and seal the cassette 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 (L/min) for each sample, along with potential interferences.

Submit the samples to the laboratory for analysis as soon as possible after sampling. If a delay is unavoidable, store the samples in a refrigerator. Ship any bulk samples separate from the air samples.

2.4 Extraction efficiency

Dry media

The extraction efficiency was determined by spiking six dry GFFs with triethanolamine at 0.1 to 2 times the target concentration. These samples were stored at ambient temperature overnight, extracted for 30 min on a tube rotator, and analyzed. The mean extraction efficiency over the studied range was 95.7%.

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Table 2.4.1
Extraction Efficiency (%) of Triethanolamine From Dry GFF

×target concn	µg per sample	1	2	3	4	5	6	mean
0.1	51.3	93.7	92.3	92.6	92.1	93.5	93.9	93.0
0.5	258	96.2	94.6	95.9	95.5	97.7	97.9	96.3
1.0	501	96.0	94.9	94.0	95.1	96.4	95.9	95.4
2.0	1001	98.5	97.8	98.2	97.9	97.8	98.3	98.1

Wet media for cassette wipes

The extraction efficiency was determined by spiking six wet GFFs with triethanolamine at 0.1 to 2 times the target concentration. The wet media were prepared by spiking the GFFs with 0.1 mL of deionized water. The samples were then spiked with triethanolamine and analyzed immediately after spiking (no drying). This study was performed to determine the extraction efficiency for cassette wipes. The mean extraction efficiency over the studied range was 103.0%.

Table 2.4.2
Extraction Efficiency (%) of Triethanolamine From Wet GFF

×target concn	µg per sample	1	2	3	4	5	6	mean
0.1	50.1	96.6	108.4	103.6	105.4	108.0	106.3	104.7
0.5	250	94.9	100.1	103.4	106.0	106.0	106.5	102.8
1.0	501	102.3	103.6	104.6	105.0	106.0	104.8	104.4
2.0	1002	99.4	99.6	101.5	102.4	98.9	98.3	100.0

2.5 Retention efficiency

Six samplers were assembled by placing another filter in series with the front filter. The back-up filter was separated from the front filter with a cassette ring. The front filters of the six samplers were each spiked with 1049 µg of triethanolamine and allowed to equilibrate for 60 hours. Each spiked sampler had 150 L humid air (about 82% relative humidity at 23 °C) pulled through them at 1 L/min. The samplers were extracted and analyzed. The mean recovery was 95.5%. There was no triethanolamine found on the back-up filters of any of the samplers.

Table 2.5
Retention Efficiency (%) of Triethanolamine

	sample number						
section	1	2	3	4	5	6	mean
front filter	96.5	94.4	94.0	95.9	95.2	97.0	95.5
back filter	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Total	96.5	94.4	94.0	95.9	95.2	97.0	95.5

2.6 Sample storage

Fifteen samplers were each spiked with 513 µg of triethanolamine. They were allowed to equilibrate for 4 h, then 100 L of air, with an absolute humidity of 15.7 milligrams of water per liter of air (about 80% relative humidity at 23 °C), was drawn through them. Three samplers were analyzed immediately, and the rest were sealed. Six were stored at room temperature (23 °C), while the other six were stored at refrigerated temperature (4 °C). Three samples stored at room temperature and three samples stored at refrigerated temperature were analyzed after seven days and the remaining six after 14 days. The amounts recovered, indicate good storage stability for the time period studied.

Table 2.6
Storage Test for Triethanolamine

time (days)	ambient storage recovery (%)			refrigerated storage recovery (%)		
0	103.0	94.6	96.5			
7	101.0	105.0	lost	100	98.0	100.0
14	95.0	100.0	100.0	104	95.0	99.0

2.7 Cassette wipes

Blank cassettes were spiked with solutions of triethanolamine in deionized water. The cassettes were allowed to air dry and then they were wiped with 37-mm GFFs that had been moistened with 0.1 mL of deionized water.

Table 2.7
Recovery of Triethanolamine From Spiked Cassette Walls

mass per cassette (µg)	recovery (%)				mean recovery (%)
50.1	95.7	88.1	86.9	98.3	92.2
501	90.4	93.6	95.0	96.4	93.8

2.8 Recommended air volume and sampling rate

Based on the data collected in this evaluation, 100-L air samples should be collected at a sampling rate of 1 L/min for 100 minutes.

2.9 Interferences (sampling)

There are no known compounds that will interfere with the collection of triethanolamine.

Suspected interferences should be reported to the laboratory with submitted samples.

3. Analytical procedure

Adhere to the rules set down in your Chemical Hygiene Plan[6]. Avoid skin contact and inhalation of all chemicals and review all appropriate MSDSs.

3.1 Apparatus

A gas chromatograph equipped with a FID detector. An Agilent 6890 gas chromatograph with a FID detector and an autosampler was used in this evaluation.

A GC column capable of separating triethanolamine from the extraction solvent, the internal standard and potential interferences. A RTX-5 amine 15 m × 0.32 mm i.d. x 1 µm df capillary column was used in this evaluation. A Restek Siltek deactivated injector liner (part no. 21700) was used in combination with the GC column.

An electronic integrator or some other suitable means of measuring peak areas. Waters Millennium[32] and Empower 2 data systems were used in this evaluation.

Glass vials with poly(tetrafluoroethylene)-lined caps. For this evaluation 2 and 4-mL vials were used.

Volumetric pipettes, 2.0-mL and other convenient sizes.

Volumetric flasks, 10-mL and other convenient sizes for preparing standards.

Syringes, calibrated 10-µL and 20-µL syringes for preparing standards.

A tube rotator or some other suitable means to extract the samples. A Fisher Roto-Rack tube rotator was used for this evaluation.

3.2 Reagents

Triethanolamine, [CAS no. 102-71-6], reagent grade. Fisher lot no. 0225792, 100.7% purity was used in this evaluation.

[6] Occupational Exposure to Hazardous Chemicals in Laboratories. *Code of Federal Regulations*, Part 1910.1450, Title 29, 2003.

Acetone, [CAS no. 67-64-1], reagent grade, Acros lot no. B0573018, ≥99.8% purity, was used in this evaluation.

1-Octanol [CAS no. 111-87-5], reagent grade. Acros lot no. B00500477, 99+% purity was used in this evaluation.

Extraction solution: 1µL/mL 1-octanol in acetone.

Deionized water; 18 MΩ-cm. A Barnstead NANOpure Diamond system was used to purify the water for this evaluation.

3.3 Standard preparation

Prepare a concentrated stock standard by weighing triethanolamine into a 10-mL volumetric flask and then diluting with extraction solution. Prepare working analytical standards by injecting microliter amounts of the stock standard into 4-mL vials containing 2 mL of extraction solution delivered from the same dispenser used for extracting the samples. For this evaluation, working standards in the range of 38 to 513 µg/sample were used.

Bracket 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 acetone and reanalyze the diluted samples.

3.4 Sample preparation

Transfer each GFF sample filter to a separate 4-mL vial.

Wipe the interior walls of each cassette top section with a clean GFF that has been moistened with 0.1 mL of deionized water. Use forceps to grasp the moistened filter and gently wipe all the interior surfaces on which TEA could accumulate. Place the filter in a 4-mL glass vial. Do not wad or crumple the filter. Wipe the blank sample in the same manner as the air sample.

Add 2.0 mL of extraction solution to each vial using the same dispenser as used for preparation of standards.

Immediately seal the 4-mL vials with poly(tetrafluoroethylene)-lined caps.

Rotate the GFFs in the vials on a tube rotator for 30 min.

Transfer the solutions to 2-mL GC autosampler vials and seal them with poly(tetrafluoroethylene)-lined caps.

3.5 Analysis

GC conditions

column temperature:	initial 125 °C, program at 30 °C /min to 200 °C, hold for 1.5 min
zone temperatures:	250 °C (injector) 300 °C (detector)
run time:	4 min
column gas flow:	4 mL/min (hydrogen)
injection size:	1.0 µL (20:1 split)
column:	RTX-5 amine 15 m x 0.32 mm i.d. x 1 µm df capillary
retention times:	0.4 min (acetone) 0.9 min ISTD (1-octanol)

1.9 min (triethanolamine)

FID conditions

hydrogen flow:	4 mL/min (constant flow)
air flow:	450 mL/min
nitrogen makeup flow:	45 mL/min

Measure peak area with an integrator or other suitable means. A calibration curve can be constructed by plotting standard injections versus micrograms of triethanolamine per sample. Bracket the samples with freshly prepared analytical standards over the range of concentrations.

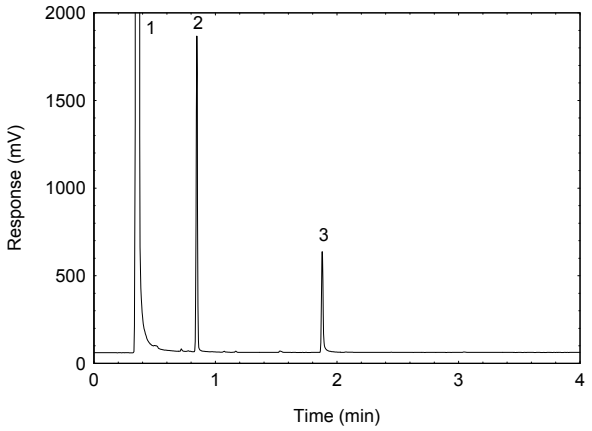

Figure 3.5.1. Chromatogram at the target concentration. [Key: (1) acetone, (2) 1-octanol, (3) triethanolamine]

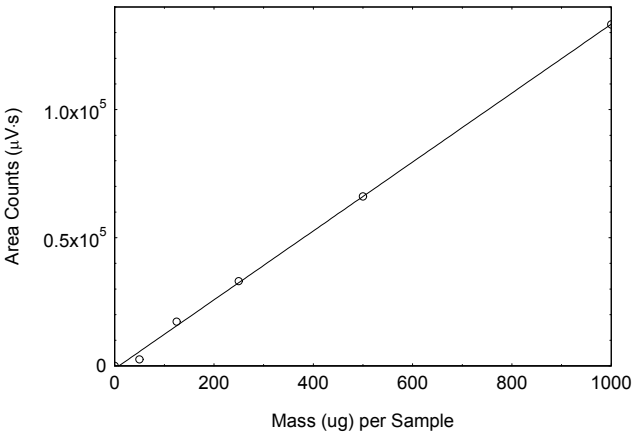

Figure 3.5.2. Calibration curve for triethanolamine. (y = 134x – 1137)

3.6 Interferences (analytical)

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

When necessary, the identity or purity of triethanolamine may be confirmed by GC-mass spectroscopy.

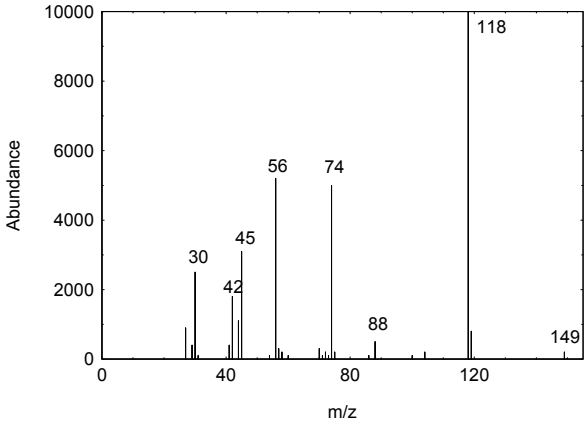

Figure 3.6. Mass spectrum for triethanolamine.

3.7 Calculations

The amount of triethanolamine per sampler is obtained from the appropriate calibration curve in terms of micrograms per GFF sample and micrograms per cassette wipe both uncorrected for extraction efficiency. These amounts are then corrected by subtracting the total amount (if any) found on the respective blanks. The blank corrected amounts are then corrected by dividing the amounts by their respective extraction efficiencies. The air concentration is calculated using the following formula:

$$C_M = \frac{\left(M_{GFF} / E_{GFF}\right) + \left(M_{wipe} / E_{wipe}\right)}{V}$$

where: C_M is concentration in mass per volume of air
M_{GFF} is micrograms per GFF sample (blank corrected)
M_{wipe} is micrograms per wipe sample (blank corrected)
E_{GFF} is extraction efficiency, in decimal form, *for GFF*
E_{wipe} is extraction efficiency, in decimal form, for wipe
V is liters of air sampled

4. Recommendations for further study

The possibility of developing a method capable of determining ethanolamine, diethanolamine, and triethanolamine from the same sample should be explored. It may be possible to sample with an acid-coated filter and to analyze samples by ion chromatography.