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Determination of Glycols in Air: Development of Sampling and Analytical Methodology and Application to Theatrical Smokes

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Glycol-based fluids are used in the production of theatrical smokes in theaters, concerts, and other stage productions. The fluids are heated and dispersed in aerosol form to create the effect of a smoke, mist, or fog. There have been reports of adverse health effects such as respiratory irritation, chest tightness, shortness of breath, asthma, and skin rashes. Previous attempts to collect and quantify the aerosolized glycols used in fogging agents have been plagued by inconsistent results, both in the efficiency of collection and in the chromatographic analysis of the glycol components. The development of improved sampling and analytical methodology for aerosolized glycols was required to assess workplace exposures more effectively. An Occupational Safety and Health Administration versatile sampler tube was selected for the collection of ethylene glycol, propylene glycol, 1,3-butylene glycol, diethylene glycol, triethylene glycol, and tetraethylene glycol aerosols. Analytical methodology for the separation, identification, and quantitation of the six glycols using gas chromatography/flame ionization detection is described. Limits of detection of the glycol analytes ranged from 7 to 16 μg/sample. Desorption efficiencies for all glycol compounds were determined over the range of study and averaged greater than 90%. Storage stability results were acceptable after 28 days for all analytes except ethylene glycol, which was stable at ambient temperature for 14 days. Based on the results of this study, the new glycol method was published in the *NIOSH Manual of Analytical Methods.*

Keywords: gas chromatography, glycols, theatrical fogging agents

Mention of company names or products does not constitute endorsement by the Centers for Disease Control and Prevention.

Recently, the National Institute for Occupational Safety and Health (NIOSH) has received requests from the Actors Equity Association and the League of American Theatres and Producers, Inc. to evaluate possible adverse health effects arising from the use of glycol-based fogging agents. The components of these fogging agents include one or more of the following polyfunctional alcohols in various combinations: ethylene glycol, propylene glycol, 1,3-butylene glycol, diethylene glycol, triethylene glycol, and tetraethylene glycol. Mixtures of these glycols are heated and dispensed as aerosols during live theatrical, musical, dance, and motion picture performances to provide the illusion of fogs, smokes, or mists.

Initial industrial hygiene evaluations of three Broadway plays in which glycol-based aerosols were used indicated that actors were exposed to the aerosolized fogging agents for durations varying from several minutes to a few hours. Actors and stage hands exposed to the glycol aerosols complained of respiratory irritation, coughing, shortness of breath, wheezing, chemical asthma, and chest tightness.[1] Questionnaires administered by NIOSH industrial hygienists to exposed actors and unexposed controls indicated a greater percentage of complaints in the exposed populations.[1] Additionally, these questionnaires detailed the frequency and severity of irritation and respiratory symptoms.

There currently is no Occupational Safety and

Health Administration (OSHA) permissible exposure limit (PEL) for ethylene glycol, nor has NIOSH established a recommended exposure level for ethylene glycol or for any of the other glycols.[2] However, NIOSH has provided testimony to OSHA that questioned whether the proposed PEL for ethylene glycol (ceiling, 50 ppm) was adequate to protect workers from recognized health hazards.[3] The American Conference of Governmental Industrial Hygienists has established a ceiling threshold limit value (TLV®) of 39.4 ppm for ethylene glycol.[4] However, it is important to recognize that in live theatrical productions, these relatively nonvolatile glycols are generated as aerosols, which may present a different and potentially significant mode of exposure.

Historically, *NIOSH Manual of Analytical Methods* (NMAM) 5500 (ethylene glycol) and various modifications of NMAM 5500, when applied to the sampling and analysis of both glycol vapors and glycol aerosols, have yielded lower results than expected, particularly when the samplers were placed in the middle of concentrated and readily visible mists.[5–13] Several of these studies reported recoveries of ethylene glycol spiked onto silica gel tubes (33–481 μg) ranging from 60–89%.[5,6,8–10] During the initial phases of this investigation, ethylene glycol and propylene glycol recoveries were found to be significantly lower when the analytes were spiked onto glass fiber filters in series with silica gel tubes and air was pulled through the sampling train for 1 hour. The results (lower recovery values) differed from those reported during the development of NMAM 5500.[14] A survey of the literature identified a number of analyses that exhibited limited success in isolating and quantitating some of the glycols used to generate theatrical smokes.[15–21] None of these approaches were considered suitable procedures for the collection and analysis of aerosolized glycol fogging agents.

Based on the need for efficient collection and analysis of the various glycol components of the fogging agents, this research was directed at (1) identifying a sampling device that is efficient at the collection of aerosolized glycols, and (2) development of an analytical method that can identify and quantitate any of the six glycols that may be present in fogging agents.

MATERIALS AND METHODS

Chemicals and Materials

Ethylene glycol (99.8%), propylene glycol (99.5%), 1,3-butylene glycol (99+%), diethylene glycol (99%), triethylene glycol (99%), and tetraethylene glycol (99%) standards were purchased from Aldrich Chemical Co., Inc. (Milwaukee, Wisc.). High-performance liquid chromatography grade methanol (Burdick & Jackson Muskegon, Mich.) was used as the desorption solvent. Glycol-containing aerosols (propylene glycol, 1,3-butylene glycol, and triethylene glycol) were generated using ROSCO™ Formulation Fluid #8207 (Rosco Laboratories, Inc., Cleveland, Ohio). A Rosco model 1500 Fog Machine (Rosco Laboratories, Inc., Port Chester, N.Y.) was used for generation of the aerosol.

Six commercially available sampling tubes were obtained from SKC Inc., (Eighty Four, Pa.). The OVS-7 tube #226–57 contained a 13-mm glass fiber filter (GFF) and two sections of XAD-7 resin (200-mg front section, a polyurethane foam [PUF] separator, and a 100-mg back section). The OVS-2 tube #226–58 contained a 13-mm GFF and two sections of XAD-2 resin (270-mg front section, 140-mg back section separated by PUF). Also obtained from SKC were the Ambersorb XE-348, catalog # 226–62–348 (140-mg front section, 70-mg back section separated by PUF); Anasorb 727, catalog #226–75 (300-mg front section, 150-mg back section separated by PUF); Anasorb 747, catalog # 226–82 (400-mg front section, 200-mg back section separated by PUF); and XAD-2, catalog #226–30–06 (400-mg front section, 200-mg back section separated by glass wool).

TABLE I. Solid Sorbent Tubes Evaluated for Use in the Glycol Sampling Train

Sorbent Tube	Composition	Mg Sorbent/ Section
XAD-2 (OVS)	styrene divinylbenzene copolymer	270/140
XAD-7 (OVS)	acrylate polymer	200/100
Ambersorb XE-348	partially carbonized polymer resin	140/70
Anasorb 727	cross-linked styrene synthetic porous beaded polymer	400/200
Anasorb 747	beaded synthetic carbon (low ash content)	400/200

Aerosol Generation

The aerosol chamber used for generation of the glycol aerosols was 12 feet (3.65 m) wide by 18 feet (5.49 m) long by 8 feet (2.44 m) high. Each end wall (8 ft (2.44 m) × 12 ft (3.65 m)) had a plenum made of masonite pegboard with quarter-inch holes. Using the Rosco fogger, a heavy mist of the Rosco fogging agent was generated and introduced into the aerosol chamber via a 3-inch hose. A constant airflow was provided by an exhaust fan positioned behind the exit plenum wall. This provided approximately 30 room air exchanges per hour. In addition, a room fan was placed directly behind the outlet hose of the Rosco fogger to ensure homogeneity of the aerosolized glycols. Samplers were prepared by connecting silica gel tubes to filter cassettes (13- or 37-mm GFF) or by using silica gel tubes. Each sampler was connected to a GilAir5™ portable air sampling pump calibrated at either 0.2 or 1.0 L/min for 60 minutes. Thirty-two pumps and sampling trains were arranged in random order on a 4 ft (1.21 m) × 8 ft (2.44 m) table. A minimum distance of 6 inches was maintained between samplers. The filter inlets were covered with parafilm to prevent aerosol collection prior to initiation of the sampling experiment. After the room had reached a (visually determined) uniform mist concentration, the parafilm was removed, each sampling pump was turned on, and the air was sampled for 60 minutes.

Analytical Parameters

Gas chromatographic analysis was performed using a Hewlett-Packard 5890 Series II gas chromatograph (GC) equipped with a flame ionization detector, and a 7673A autosampler (Hewlett-Packard Co., Avondale, Pa.). A 30-m Rtx®-35 fused silica capillary column (0.53 mm i.d., 3-μm film) (Restek Corp., Bellefonte, Pa.) was used for all separations. Chromatographic data was collected and analyzed with an AI-450 Dionex Data System (Dionex Corp., Sunnyvale, Calif.).

Baseline separation of the six glycols was achieved using the following gas chromatographic parameters: 30-m Rtx-35 fused silica capillary column (0.53 mm i.d., 3-μm film); a temperature program ramped from 40 to 230°C (8°C/min); an injection port temperature of 250°C, and a detector temperature of 300°C. The

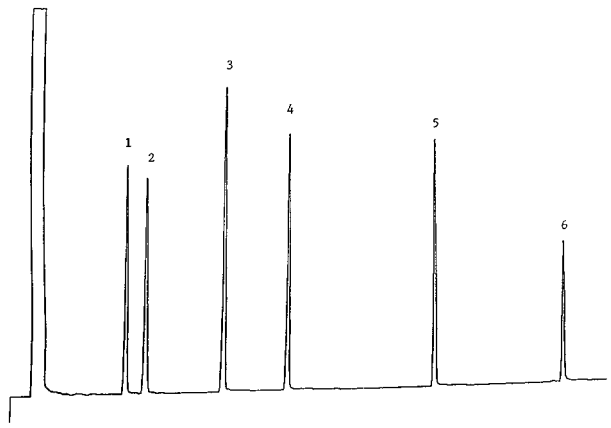

FIGURE I. Baseline separation of (1) ethylene glycol, (2) propylene glycol, (3) 1,3-butylene glycol, (4) diethylene glycol, (5) triethylene glycol, and (6) tetraethylene glycol on an Rtx-35 fused silica capillary column

carrier gas was helium (3 mL/min) and the sample injection volume was 1 μL, splitless mode.

Sampler Selection

NMAM 5500 Sampling Train

Initial investigations focused on using the sampling train described in NMAM 5500 for the collection and analysis of the six glycol analytes associated with theatrical smokes. The sampling train consisted of a Gelman 37-mm GFF in series with a silica gel tube (520/260 mg).[22] Filters of the test sampling trains were spiked with solutions of the six glycol analytes at three concentration levels (N=6) ranging from 0.028 to 0.237 mg/sample. Air was drawn though the samplers at 1 L/min for 30 minutes. The sampling trains were disassembled, capped, and allowed to stand at room temperature overnight. The filter, front section, and backup section were each extracted separately in 1 mL of methanol. An ultrasonic water bath was used to facilitate desorption.

Impinger Samplers

Midget impingers containing 15 mL of methanol were spiked with 30 μL of a stock solution (200–300 μg/mL) of glycol compounds. Air was drawn through the impingers for 30 minutes to simulate sampling conditions.

Solid Sorbent Sampler

Six commercially available solid sorbents were selected for preliminary evaluation based on the requirements of high polarity, maximum surface area, and increased mass of sorbent. The sorbents selected included XAD-2, XAD-2 (OVS), XAD-7, Ambersorb xe-348, Anasorb 727, and Anasorb 747. See Table I for specifications of the sorbents. Each sorbent tube was spiked at a single level (N=3) with a mixture of the six glycols. Using Gil-Air 5 portable sampling pumps (Sensidyne, Inc., Clearwater, Fl.), air was drawn through each solid sorbent tube sampler for 30 minutes at 1 L/min. After standing overnight, the filter, the front, and the back sorbent sections were separately extracted with 2 mL of methanol, placed in an ultrasonic bath for 30 minutes, and analyzed using the gas chromatographic conditions previously described.

TABLE II. Analytical Limits of Detection and Limits of Quantitation for Glycols as Derived from Media Standards

Analyte	LOD (μg/sample)	LOQ (μg/sample)	RSD
Ethylene glycol	7	22	0.07
Propylene glycol	8	24	0.05
1,3-Butylene glycol	6	18	0.03
Diethylene glycol	16	48	0.03
Triethylene glycol	14	42	0.02
Tetraethylene glycol	14	42	0.08

Desorption Efficiency Study

The GFFs in the XAD-7 OVS sorbent tubes (SKC # 226–57, Lot 904) were spiked with 10-μL aliquots of solutions containing ethylene glycol (0.033–0.218 mg/sample), propylene glycol (0.026–0.190 mg/sample), 1,3-butylene glycol (0.034–0.178 mg/sample), diethylene glycol (0.066–0.220 mg/sample), triethylene glycol (0.033–0.201 mg/sample), and tetraethylene glycol (0.032–0.197 mg/sample). Three levels (N=6 at each level) in the range of approximately 1.5× to 10× the limit of quantitation (LOQ) were prepared. Using portable sampling pumps, air was drawn through the sampling tubes at 1 L/min for 60 minutes. The samplers were then capped and allowed to stand at room temperature overnight. The filter and front section of the XAD-7 sorbent were placed in 5-mL screw capped vials, and extracted in 2 mL of methanol for 30 minutes. An ultrasonic water bath was used to faciltate desorption. The backup section of XAD-7 was extracted separately to check for breakthrough. One-mL aliquots were transferred to GC autosampler vials for analysis.

Storage Stability Study

The GFFs in the XAD-7 OVS sorbent tubes were spiked at three levels (N = 18 at each level, for a total of 54 samplers) in the range of approximately 1.5× to 15× LOQ with 10-μL aliquots

TABLE III. Average Recoveries of Glycols Spiked on 13-mm GFF in Series with SiO_2 Sorbent Tubes

Analyte	Spike Level (μg)	Filter Recovery (%)	SiO_2 Recovery (%)	Total Recovery (%)	RSD
Ethylene glycol	33.6	0.0	38.2	38.2	0.35
	109.3	0.0	34.0	34.1	0.19
	218.6	6.7	37.4	44.0	0.14
Propylene glycol	28.2	0.0	54.5	54.5	0.21
	91.8	1.1	46.9	47.9	0.11
	183.5	6.1	46.5	52.6	0.08
1,3-Butylene glycol	34.6	0.0	49.9	49.9	0.34
	112.4	1.8	30.6	32.4	0.19
	224.9	10.2	30.4	40.6	0.15
Diethylene glycol	34.0	47.2	21.4	68.6	0.56
	110.5	68.6	25.3	93.9	0.08
	221.0	77.6	10.2	87.7	0.07
Triethylene glycol	36.4	85.8	0.0	85.8	0.06
	118.3	108.1	0.0	108.1	0.09
	236.7	106.0	0.0	106.0	0.06
Tetraethylene glycol	31.2	<LOQ	<LOQ	<LOQ	
	101.3	96.8	0.0	96.8	0.13
	202.7	101.1	0.0	101.1	0.09

Note: Air drawn through spiked sampling trains at 1 L/min. N = 6 for each level.

TABLE IV. Glycol Solid Sorbent Collection Efficiency Study

Sorbent ID	Analyte	Recovery (from Filter) (%)	Recovery (from Sorbent) (%)	Recovery (Total) (%)
XAD-2	ethylene glycol	6.8	19.6	26.4
XAD-2	propylene glycol	3.7	51.5	55.2
XAD-2	1,3-butylene glycol	4.3	85.1	89.4
XAD-2	diethylene glycol	45.2	45.3	90.5
XAD-2	triethylene glycol	87.4	0.0	87.4
XAD-2	tetraethylene glycol	105.1	0.0	105.1
XAD-2 (OVS)	ethylene glycol	0.0	28.4	28.4
XAD-2 (OVS)	propylene glycol	0.0	53.7	53.7
XAD-2 (OVS)	1,3-butylene glycol	0.0	92.4	92.4
XAD-2 (OVS)	diethylene glycol	11.5	87.3	98.8
XAD-2 (OVS)	triethylene glycol	36.4	54.1	90.5
XAD-2 (OVS)	tetraethylene glycol	68.5	41.2	109.7
XAD-7 (OVS)	ethylene glycol	0.0	95.1	95.1
XAD-7 (OVS)	propylene glycol	0.0	98.2	98.2
XAD-7 (OVS)	1,3-butylene glycol	0.0	101.1	101.1
XAD-7 (OVS)	diethylene glycol	0.0	91.1	91.1
XAD-7 (OVS)	triethylene glycol	20.7	66.6	87.3
XAD-7 (OVS)	tetraethylene glycol	45.1	65.7	110.8
Ambersorb XE-348	ethylene glycol	0.0	99.9	99.9
Ambersorb XE-348	propylene glycol	0.0	96.8	96.8
Ambersorb XE-348	1,3-butylene glycol	0.0	91.4	91.4
Ambersorb XE-348	diethylene glycol	30.6	51.4	82.0
Ambersorb XE-348	triethylene glycol	88.0	0.0	88.0
Ambersorb XE-348	tetraethylene glycol	104.5	0.0	104.5
Anasorb-727	ethylene glycol	0.0	69.2	69.2
Anasorb-727	propylene glycol	0.0	91.3	91.3
Anasorb-727	1,3-butylene glycol	0.0	94.7	94.7
Anasorb-727	diethylene glycol	33.5	47.7	81.2
Anasorb-727	triethylene glycol	86.1	0.0	86.1
Anasorb-727	tetraethylene glycol	102.1	0.0	102.1
Anasorb-747	ethylene glycol	0.0	91.9	91.9
Anasorb-747	propylene glycol	0.0	84.4	84.4
Anasorb-747	1,3-butylene glycol	0.0	77.2	77.2
Anasorb-747	diethylene glycol	39.3	36.2	75.5
Anasorb-747	triethylene glycol	93.1	0.0	93.1
Anasorb-747	tetraethylene glycol	109.9	0.0	109.9

Note: Flow rate of 1 L/min. N = 3 for each type of sorbent tube tested. Spiking levels were approximately 5× LOQ.

of solutions containing ethylene glycol (0.034 to 0.342 mg/sample), propylene glycol (0.029 to 0.285 mg/sample), 1,3-butylene glycol (0.030 to 0.303 mg/sample), diethylene glycol (0.033 to 0.329 mg/sample), triethylene glycol (0.031 to 0.309 mg/sample), and tetraethylene glycol (0.033 to 0.326 mg/sample). Using portable sampling pumps, air (ambient temperature and 28% relative humidity) was drawn through the sampling tubes at 1 L/min for 60 minutes. The samplers were capped and stored at room temperature in the dark. Six samples at each of the three levels (18 total) were analyzed after 7 days; another 18 were analyzed after 14 days; and the last 18 analyzed after 28 days.

RESULTS AND DISCUSSION

The gas chromatographic analytical parameters identified in the experimental section were able to separate trace levels of six homologous glycols: (ethylene glycol, propylene glycol, 1,3-butylene glycol, diethylene glycol, triethylene glycol, and tetraethylene glycol). Hexylene glycol was also resolved but was not included in the evaluation of this method as it is not a component of theatrical fogging agents. Baseline separation of all the glycol analytes was achieved in less than 25 minutes using an Rtx-35 fused silica capillary column (see Figure 1). A 12-point calibration curve (in duplicate) ranging from 1.4 to 337.5 μg/sample was employed in this method development. The limits of detection (LOD), limits of quantitation (LOQ), and relative standard deviation (RSD) values were determined for each glycol analyte[23] (see Table II). For the purposes of this method development, the LOD is defined as the mass of an analyte that gives a signal three sigma above the mean blank signal, where sigma is the standard deviation of the blank signal, while the LOQ is defined as the mass corresponding to the mean blank signal + $10\delta_b$ or the mass above which recovery is ≥75%.[23]

Initial efforts focused on adapting the sampling train specified in NMAM 5500 for the collection and analysis of the six glycols potentially present in theatrical fogging agents. Four of the glycols—1,3-butylene glycol, diethylene glycol, triethylene glycol, and tetraethylene glycol—exhibited poor recovery (36–65%) when desorbed with 98:2 water/isopropanol. Methanol was then considered as the desorption solvent and appeared to be more effective as a desorption agent for all six glycol analytes. In addition, the high water content had a detrimental effect on capillary column performance (peak broadening, coeluting peaks, and flow rate limitations) and frequently extinguished the detector flame.

TABLE V. Glycol Desorption Efficiencies from OVS XAD-7 Solid Sorbent Tubes

Analyte	Spike Level (μg)	Recovery (%)	RSD (%)
Ethylene glycol	33.3	80.5	6.3
	105.7	84.8	3.0
	217.7	89.5	2.4
Propylene glycol	25.7	105.0	4.9
	113.3	91.7	9.3
	217.7	93.7	2.2
1,3-Butylene glycol	33.5	102.3	8.5
	106.4	98.8	2.8
	177.9	97.4	2.8
Diethylene glycol	67.6	98.4	5.7
	107.6	103.5	5.1
	219.3	97.4	2.6
Triethylene glycol	33.0	107.6	11.7
	103.7	89.8	4.8
	200.8	90.5	3.0
Tetraethylene glycol	32.2	139.7	25.3
	95.2	115.7	0.1
	197.3	105.2	4.9

Note: Sampling flow rate of 1 L/min. N = 6 for each level.

At this point in the method development it was determined that it was necessary to conduct an experiment in which glycol aerosols (propylene, 1,3-butylene, and triethylene glycol) were generated using the Rosco model 1500 Fog Machine to ascertain the efficiency of the NMAM 5500 sampler and the effect of sampler orientation and flow rate. Glycol aerosols were sampled for 1 hour using several different sampling systems and a direct comparison of the relative total mass of glycols collected (not absolute recovery from a known initial concentration) was conducted. The results indicated that a 13-mm GFF in series with a silica gel sorbent tube was approximately 2–3 times more effective at glycol aerosol collection than a 37-mm GFF in series with a silica gel sorbent tube. Collection of propylene and 1,3-butylene glycol aerosols on silica gel sorbent tubes alone was comparable with the results achieved using the 37-mm GFF in series with the silica gel sorbent tube. No triethylene glycol was detected on the single silica gel sorbent tubes. Sample collection at a rate of 1 L/min was approximately 10 times more effective than a rate of 0.2 L/min.

TABLE VI. Glycol Recovery Results from Storage Stability Study

Analyte	Spiking Level (μg)	9-Day Storage Time Recovery (%)	14-Day Storage Time Recovery (%)	28-Day Storage Time Recovery (%)	Averaged RSD (%)
Ethylene glycol	34.2	80.9	75.5	60.4	4.3
	102.7	82.3	78.5	62.0	4.1
	342.2	90.4	92.5	71.6	1.5
Propylene glycol	28.5	96.3	88.5	90.7	6.7
	85.4	92.0	87.5	87.8	4.9
	284.6	91.4	95.8	96.5	1.8
1,3-Butylene glycol	30.3	91.1	77.0	83.4	5.8
	90.8	93.2	85.1	89.0	4.9
	302.8	91.8	97.8	99.5	1.4
Diethylene glycol	32.9	87.3	73.4	75.4	12.2
	98.7	89.4	82.0	77.0	4.6
	302.8	94.9	84.5	87.5	2.1
Triethylene glycol	30.9	91.3	97.0	92.0	12.1
	92.6	91.2	85.8	90.0	5.9
	308.6	92.0	98.7	99.2	2.9
Tetraethylene glycol	32.6	97.9	95.1	93.2	11.5
	97.7	81.4	76.5	82.7	5.3
	325.5	86.5	86.5	85.8	5.2

Note: Sampler flow rate was 1 L/min. N = 6 at each level.

All six glycol analytes exhibited nearly quantitative recovery (>95%) when spiked onto silica gel tubes, allowed to stand overnight, and desorbed with methanol using agitation. However, when the glycols were spiked directly onto the GFF and air was drawn through the sampler prior to desorption and analysis (filter and sorbent section were both analyzed); ethylene glycol, propylene glycol, and 1,3-butylene glycol exhibited poor recoveries (<40%). Diethylene glycol and tetraethylene glycol exhibited poor recoveries at spiking levels near the LOQ. The results are depicted in Table III.

Because of the poor recoveries achieved when the NMAM 5500 sampler was used, a sampler with better recovery characteristics was required. Impingers containing 15 mL of methanol were spiked with 30 μL of a stock solution (200–300 μg/mL) of the glycol analytes. Air was drawn through the impingers for 30 minutes to simulate sampling conditions. Quantitative recoveries over a range of 208 to 297 μg/sample were obtained for all analytes. However, approximately one-half of the methanol evaporated during the sampling period. Additional experiments indicated that impingers containing 15–20 mL of methanol, when connected to pumps pulling air through the impingers at a flow rate of 1 L/min for 90 minutes, exhibited evaporative losses of 60–72%. Since there was no practical way of refilling the impingers during the 2-hour sampling period of a theatrical performance, coupled with problems encountered such as worker resistance to wearing the bulky impingers, subsequent studies were directed toward the selection of a suitable solid sorbent sampler.

Six commercially available solid sorbents were selected for preliminary evaluation based on polarity, surface area, and mass of sorbent. Of these, the XAD-7 OVS tube spiked with the six glycol analytes exhibited the best overall recovery results (>98%). Five of the six glycols tested had recovery efficiencies greater than 91%, while that for triethylene glycol was slightly lower (87%) (see Table IV). Ambersorb-348 had results similar to the XAD-7 OVS tube, but two of the glycol analytes had recoveries below 90% (82 and 87%). The XAD-2 based solid sorbent samplers exhibited the poorest overall recovery results (<56%).

XAD-7 OVS Sorbent

Based on the preliminary recovery results achieved for the glycol analytes using the XAD-7 OVS sorbent, a full-scale recovery study was conducted. Using a sampling time (1–2 hours) representative of that most likely to be encountered in live theatrical productions, recoveries for the glycol analytes, with the exception of ethylene glycol, were greater than 98% (see Table V). Recoveries for ethylene glycol ranged from 81% (33 μg) to 90% (218 μg). The lower recoveries for ethylene glycol could possibly be attributed to increased volatility when sampled at a flow rate of 1 L/min for 60 minutes. Additionally, the results of this study indicated that the more volatile glycols such as ethylene, propylene, and 1,3-butylene glycol were primarily collected on the XAD-7 sorbent bed while the less volatile glycols such as diethylene, triethylene, and tetraethylene glycol were collected on the 13-mm GFF. At a spiking level near the LOQ tetraethylene glycol had a high recovery (139.7% with an RSD of 25.3%) due to a wide variance among sample recoveries.

The final phase in this study was a 28-day storage stability evaluation (range was approximately 30 to 300 μg) at 5°C. The results are depicted in Table VI. All six glycol analytes exhibited acceptable storage stability recoveries (>75% recovery) when analyzed after 14 days. Ethylene glycol was the only analyte to exhibit unacceptable recoveries after 28 days. Corrections for analyte recovery would be required when recovery falls below 95%. Recovery below 75% is considered unacceptable for reporting as quantitative data.[23]

SUMMARY AND CONCLUSIONS

A sampling and analytical method for the determination of aerosolized glycol fogging agents has been described. Glycol fogging agents containing ethylene, propylene, 1,3-butylene, diethylene, triethylene, and tetraethylene glycol are efficiently recovered from an OVS sampler tube containing a 13-mm GFF in contact with a two-stage solid sorbent tube containing XAD-7. The more volatile glycols, such as ethylene, propylene, and 1,3-butylene glycol, were primarily collected on the XAD-7 sorbent while the less volatile glycols, such as diethylene, triethylene, and tetraethylene glycol, were largely collected on the GFF. Desorption efficiencies and recoveries were acceptable (overall average was >97%), and no breakthrough was found after sampling for 60 minutes. All analytes were stable on the XAD-7 OVS tubes for 14 days at room temperature.

Overall, sampling on the XAD-7 OVS tubes, coupled with separation and analysis using gas chromatography provides a comprehensive method for monitoring and identifying exposures to the glycol components of theatrical fogging agents.

Although the author believes that the sampling and analytical methodology for glycol aerosols, as presented herein, represents a significant improvement over previous techniques, it is recognized that the collection of liquid aerosols presents difficulties associated with sampling. Possible explanations are the evaporative loss of glycol particles from the filter during sampling and/or adhesion of glycol particles to the interior surfaces of the sampler. Possible areas for future research should focus on minimizing these losses.

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