

Detecting Ethylene Oxide by Packed Column GC

EtO is suspected of being a carcinogenic agent at concentrations above 1ppm. In 1983, OSHA issued an emergency health standard, which later became permanent, that limited worker exposure from 50ppm to 1ppm of EtO. We detected EtO at levels below 1ppm using a Carbowax C/0.8% THEED column. Subsequently, in 1988, ANSI/AAMI put out standards for determining residual EtO and its byproducts in medical devices. For these methods, we detected 10ppm EtO on the 3% Carbowax 20M on 80/100 Chromosorb column.

Key Words

- ethylene oxide • ethylene chlorohydrin • ethylene glycol

Ethylene oxide (EtO) is commonly used in the medical field to sterilize supplies and instruments that come in contact with patients. However, due to its carcinogenic affect, the Occupational Safety and Health Administration (OSHA) limited worker exposure to 1ppm (1). OSHA sampling procedures recommend pre-concentrating EtO from air onto a charcoal adsorbent tube, desorbing the tube in carbon disulfide (CS₂), and analyzing it by gas chromatography (GC) (2). We analyzed 1ppm EtO using a Carbowax™ C/0.8% tetrahydroxyethylethylene diamine (THEED) column. Figure A illustrates the analyses of EtO in CS₂ and H₂O.

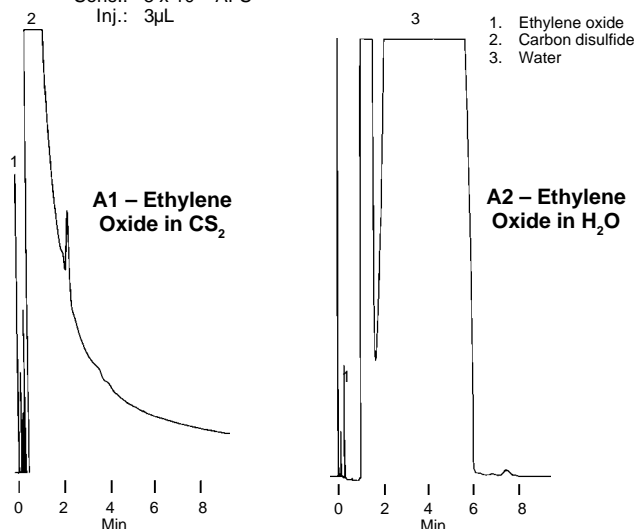
Potential interference by other airborne contaminants was investigated, and only one compound was found to coelute with EtO — bromomethane. On the Carbowax C/0.8% THEED column, EtO was resolved from Freon®-12, a material with which EtO is frequently used, with a retention time of 0.43 minutes. Other compounds investigated were the C1 to C6 hydrocarbons; homologous series of aldehydes, esters, ketones, alcohols, and ethers; and chlorinated hydrocarbons.

Many sterilization procedures culminate in a final flushing of EtO from the sterilized product with water or acetone. To ensure elimination of a potential health hazard, the residual EtO levels of these flush solutions must be monitored. Figure A shows that a Carbowax C/0.8% THEED column can detect very low ppm concentrations of EtO in air or in a flush solution without interference.

The Carbowax C/0.8% THEED packing also can be used at 115°C to determine levels of ethylene chlorohydrin (EC) and ethylene glycol (EG) — byproducts of the EtO sterilization process. The versatility of this packing eliminates the need to use two columns for analyzing EtO and its byproducts. Analyses for low ppm quantities of all three compounds can be conducted with most sample matrices (Figure B).

Figure A. Ethylene Oxide at 1ppm in CS₂ and H₂O

Sample: 1ppm ethylene oxide in CS₂ (Figure A1) or H₂O (Figure A2)
Packing: **80/100 Carbowax C/0.8% THEED**
Cat. No.: **11880-U** (packing)
Column: 3' x 2mm ID glass
(stock column available)
Oven: 40°C
Carrier: helium, 20mL/min
Det.: FID, 40°C
Sens.: 8 x 10⁻¹² AFS
Inj.: 3μL



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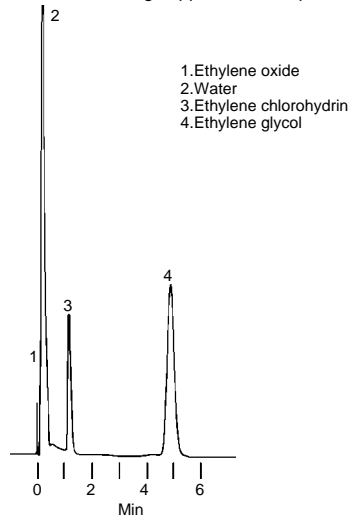
ANSI/AAMI Methods

In addition to OSHA standards protecting workers, the American National Standards Institute/Association for the Advancement of Medical Instrumentation (ANSI/AAMI) issued ST29 and ST30 to protect patients — *Recommended Practice for Determining Residual Ethylene Oxide in Medical Devices* and *Determining Residual Ethylene Chlorohydrin and Ethylene Glycol in Medical Devices*, respectively. These standards define testing procedures for residual EtO, EC, and EG from medical devices. ANSI/AAMI suggests using a 3% Carbowax® 20M on 80/100 Chromosorb® 101 packing to separate these compounds in water or headspace. We tested this method using 10ppm each compound and two temperatures (Figure C). All compounds separated well on the 3% Carbowax 20M on 80/100 Chromosorb 101 packing.

The Carbowax C/0.8% THEED and the 3% Carbowax 20M on 80/100 Chromosorb 101 packings are available as stock items, or in custom-packed columns. Refer to the Supelco catalog for more information about ordering glass columns packed with these materials.

Figure B. Ethylene Oxide and Ethylene Oxide Residues

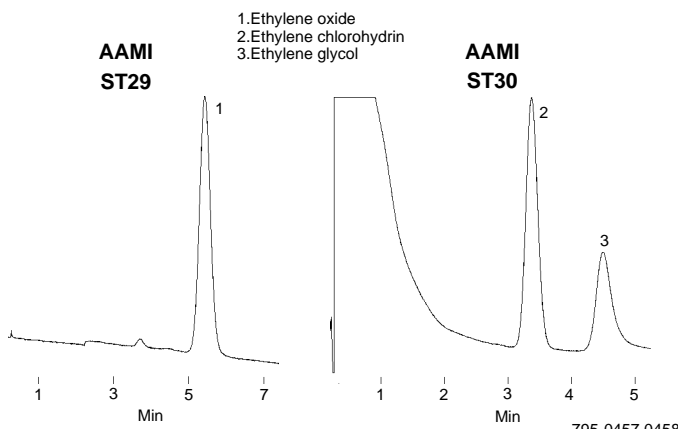
Packing: **80/100 Carbowax C/0.8% THEED**
 Cat. No.: **11880-U** (packing)
 Column: 1m x 2mm ID glass
 (stock column available)
 Oven: 115°C
 Carrier: nitrogen, 20mL/min
 Det.: FID
 Inj.: 1µL water containing 50ppm each component



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Figure C. Ethylene Oxide and Ethylene Oxide Residues by AAMI Methods

Packing: **3% Carbowax 20M on 80/100 Chromosorb 101**
 Cat. No.: **11780-U** (20g)/bottle
 Column: 6' x 2mm ID glass
 Oven: 70°C (ST29) or 160°C (ST30)
 Carrier: nitrogen, 30mL/min
 Det.: FID (250°C)
 Inj.: 3µL water containing 10ppm each analyte (200°C)



795-0457,0458

Contact our Technical Service Department
 (phone 800-359-3041 or 814-359-3041, FAX 814-359-5468)
 for expert answers to your questions.

Ordering Information:

Description	Cat. No.
80/100 Carbowax C/0.8% THEED packing, 15g	11880-U
3% Carbowax 20M on 80/100 Chromosorb 101, 20g	11780-U

Stock Packed Glass Columns

80/100 Carbowax C/ 0.8% THEED 3' x 2mm ID glass	1m x 2mm ID TightSpec	3% Carbowax 20M on 80/100 Chromosorb 101 6' x 2mm ID glass	2m x 2mm ID TightSpec
Hewlett-Packard 5700, 5992-3 GC-MS			
26065	26056	26083	26074
Hewlett-Packard 5880, 5890, 5987, 6890			
26066	26057	26084	26075-U
Perkin Elmer 900, 3920, Sigma 1,2,3 (not on-col. inj.)			
26067	26058	26085	26076
Perkin-Elmer, Sigma (on-col. inj.)			
26068	26059	26086	26077-U
Perkin Elmer 8300, 8400, 8500, 8600, 8700, Auto System (not on-col. inj.)			
26069	26060-U	26087	26078
Shimadzu GC14A, GC15A, GC16A [■]			
26070	26061	26088	26079
Tracor 560, 565, 570			
26071	26062	26089	26080-U
Varian 3300/3400 Vista Series (FID)			
26072	26063	26090-U	26081
Varian 3700 Vista Series (FID)			
26073	26064	26091	26082

[■] Changed to circular configuration. 5mm OD x 2.6mm ID.

TightSpec Versus Nominal Length Glass Columns

TightSpec metric length columns conform to within ±6mm of their stated lengths. No matter which instrument you use, your column will have the same length, and you will have the best chance of repeating your results from one instrument to another.

Supelco nominal length columns conform to within ±6mm of the instrument manufacturer's length specifications. When you compare actual lengths of these columns among instruments, you will find large differences, which will cause problems when you try to reproduce retention times among different instruments. We recommend using nominal length columns only when you are trying to duplicate a method on the same model of instrument as was originally used to develop the method.

References

1. Chemical & Engineering News, 61 (3), p. 9 (Jan. 17, 1983).
2. NIOSH Manual of Analytical Methods, 2nd edition, Vol. 3, Method No. S286 (NIOSH Publication No. 77-157-C, 1977).

References not available from Supelco.

Trademarks

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 Chromosorb — Manville Corp.
 Freon — E.I. du Pont de Nemours & Co., Inc.

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