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Committee D16 on Aromatic Hydrocarbons and Related Chemicals Subcommittee D16.16 on Industrial & Specialty Product Standards

Research Report: D16-1074

Interlaboratory Study to Establish Precision Statements for ASTM D8311, Impurities in Monoethylene Glycol by Gas Chromatography with Normalization

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1. Introduction:

1.1 This test method covers the gas chromatographic determination of the purity and impurities in MEG with normalization. The impurities to be determined includes 1,3-Dioxane-2-methanol, diethylene glycol (DEG) and triethylene glycol (TEG) with the LOD of 0.0002 mass %.

2. Test Method:

2.1 A MEG sample is injected into the gas chromatography. The components in sample are separated with a capillary column and detected with a flame ionization detector (FID). All the peak areas on the chromatogram are collected and calibrated with calibration factors measured with calibration solution. The calibrated peak areas are used to calculate the purity of MEG and the amount of the impurities with normalization

3. Participating Laboratories:

3.1. The following laboratory participated in this study:

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4. Description of Samples:

4.1 Two MEG samples are tested in this study. The samples are prepared by adding the typical impurities including DEG, TEG and 1,3-dioxane-2-methanol into a blank MEG.

5. Inter-laboratory Study Instructions:

5.1. The concentration of the components in the test samples are listed in Table 1.

	Sample I	Sample II
EG	99.9363	99.9450
1,3-Dioxane-2-methanol	0.0052	0.0033
DEG	0.0126	0.0044
TEG	0.0102	0.0011
Water	0.0307	0.0414

Table 1. The concentration of the components

5.2. Operating conditions of gas chromatography are listed in Table 2.

1	0 011			
Inlet	Split			
Temperature, °C	300			
Column				
Material	fused silica			
Stationary Phase	6 % Cyanopropyl-phenyl -94 % dimethyl polysiloxane			
Length, m	30			
Internal diameter, mm	0.32			
Film thickness, µm	1.8			
Column temperature program				
Initial temperature, °C	80			
Initial time, min	0.1			
Programming rate, °C/min	25			
Final, °C	240			
Time 2, min	10			
Carrier gas	Helium			
Flow velocity, mL/min	1.5 Helium			
Split ratio	30:1			
Sample size, µL	1.0			
Detector	FID			
Temperature, °C	300			

Table 2. The o	peration	conditions	of gas	chromatography

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