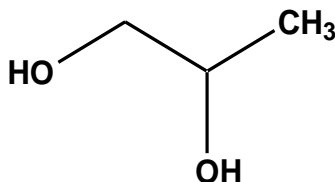


# Certificate of Analysis

**ISO GUIDE 34**  
ACCLASS Cert# AR-1470

**ISO/IEC 17025**  
ACCLASS Cert# AT-1467

## PROPYLENE GLYCOL CERTIFIED REFERENCE MATERIAL



**CERTIFIED PURITY: 99.9%**,  $U_{\text{crm}} = \pm 0.2\%$   $k = 2$   
(Mass Balance/as is basis)

**NOMINAL PACKAGE SIZE:** 1.5g

**CATALOG #:** PHR1051

**LOT #:** LRAA5984

**CERTIFICATE VERSION:** LRAA5984.1

**ISSUE DATE:** 10 November 2014

*Note: Certificates may be updated due to Pharmacopeial Lot changes or the availability of new data.  
Check our website at: [www.sigma-aldrich.com](http://www.sigma-aldrich.com) for the most current version.*

**CRM EXPIRATION:** 31 December 2019 (Proper Storage and Handling Required).

**RECEIPT DATE:** \_\_\_\_\_

Note: this space is provided for convenience only and its use is not required.

**STORAGE:** Store at Room Temperature. Transfer unused portions to a tightly closed container. Protect from humidity.

**CHEMICAL FORMULA:** C<sub>3</sub>H<sub>8</sub>O<sub>2</sub>

**MW:** 76.1

**PHYSICAL DESCRIPTION:** Clear liquid in amber ampule **CAS #:** 57-55-6

**HAZARDS:** Read Safety Data Sheet before using. All chemical reference materials should be considered potentially hazardous and should be used only by qualified laboratory personnel.

**INSTRUCTIONS FOR USE:** Do not dry, us as is. The internal pressure of the container may be slightly different from the atmospheric pressure at the user's location. Open slowly and carefully to avoid dispersion of the material. This material is intended for R&D use only. Not for drug, household or other uses.

## TRACEABILITY ASSAY

Comparative assay demonstrates direct traceability to Pharmacopeial Standards  
Specification: Not less than 99.5% (USP)

**METHOD: GC (ref.: USP 36; Adapted from Propylene Glycol and Glycerin Monographs)**

### ***ASSAY vs. USP REFERENCE STANDARD (as is basis)***

ASSAY VALUE

**99.5%**

vs. USP LOT

**L0J287**

Labeled Content = 1.00mg/mg

### ***ASSAY vs. EP CRS (as is basis)***

ASSAY VALUE

**99.5%**

vs. EP BATCH

**1.0**

Labeled Content = None

Assigned Content = 98.4%\*

\*Assigned content was determined by comparison against the USP Reference Standard

Column: SPB-624 30m x 0.53 mm x 1.40 $\mu$ m

Detector/Temperature: FID/250 $^{\circ}$ C

Carrier Gas: H<sub>2</sub>

Flow: 1.2mL/min

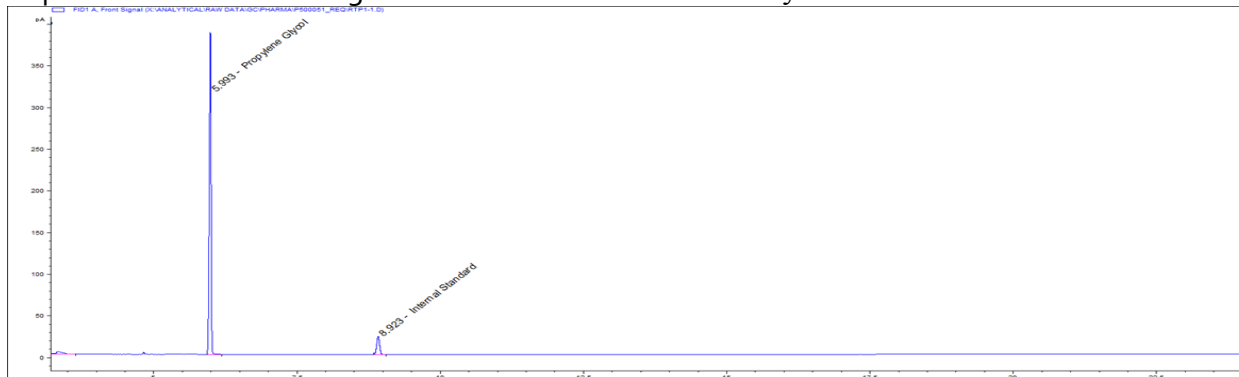
Split Ratio: 1:5

Injection/Temperature: 1 $\mu$ l/220 $^{\circ}$ C

Temperature Program: 40 $^{\circ}$ C for 1min, 25 $^{\circ}$ C /min to 120 $^{\circ}$ C, hold 10 min, 25 $^{\circ}$ C to 220 $^{\circ}$ C, hold 6min

Internal Standard: 2,2,2-Trichloroethanol

Representative Chromatogram from Lot: LRAA5984 Analysis

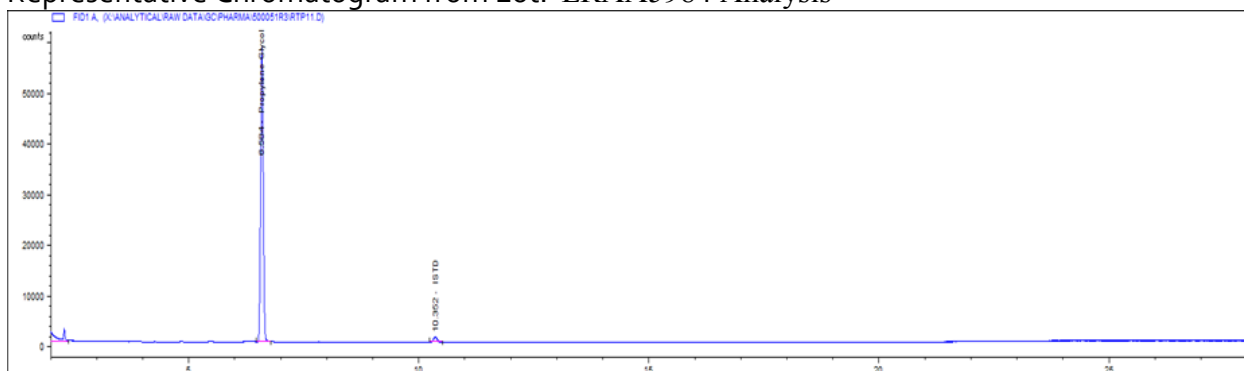


## ASSAY vs. BP CRS (as is basis)

<u>ASSAY VALUE</u>	<u>vs. BP BATCH</u>
<b>99.6%</b>	<b>3082</b>
	Labeled Content = 99.9%

Column: DB-624 30m x 0.53 mm x 3.00µm  
 Detector/Temperature: FID/250°C  
 Carrier Gas: He  
 Flow: 4 mL/min  
 Split Ratio: 1:10  
 Injection/Temperature: 1 µl/220°C  
 Temperature Program: 40°C for 1min, 40°C to 100°C at 25°C/min, 25°C /min to 120°C, hold 10 min, 25°C to 220°C, hold 6min  
 Internal Standard: 2,2,2-Trichloroethanol

Representative Chromatogram from Lot: LRAA5984 Analysis



## PURITY DETERMINATION BY MASS BALANCE

### CHROMATOGRAPHIC IMPURITY ANALYSIS

**METHOD:** GC

Column: DB-Wax 30m x 0.32 mm x 0.50µm

Detector/Temperature: FID/240°C

Carrier Gas: N<sub>2</sub>

Flow: 5 mL/min

Split Ratio: 1:11

Injection/Temperature: 1 µl/200°C

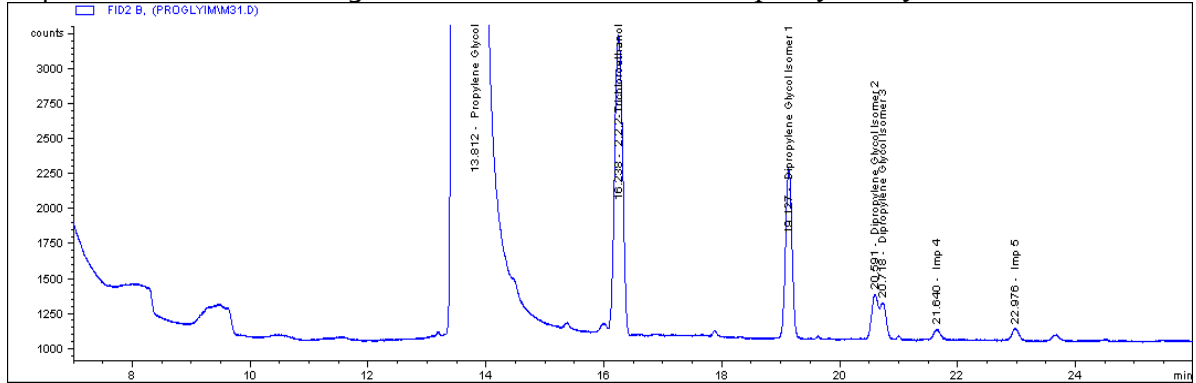
Temperature Program: 120°C to 200°C at 5°C/min, hold at 200°C for 10 min

### Impurities Detected

Dipropylene Glycol Isomer 1:	<b>0.04%</b>
Dipropylene Glycol Isomer 2:	<b>0.01%</b>
Dipropylene Glycol Isomer 3:	<b>0.006%</b>
Unknown Impurity 4:	<b>0.005%</b>
Unknown Impurity 5:	<b>0.007%</b>

Total Chromatographic Impurities: **0.07%**

### Representative Chromatogram from Lot: LRAA5984 Impurity Analysis



### RESIDUAL SOLVENTS

Method: GC-MS Headspace (ref.: Residual Solvents <467>, USP34)

Column: DB-1301

Carrier gas: He

Flow: 1.2mL/min

Split Ratio: 1:5

Injection/Temperature: 1µl/250°C

Temperature Program: 40°C for 20min, 10°C/min to 240°C, hold 20min

Solvents Detected: None

### WATER DETERMINATION

Method: Karl Fisher titration

Mean of three measurements, Water Content = **0.05%**

### RESIDUE ANALYSIS

Method: Sulfated Ash

Sample Size: ~25 g

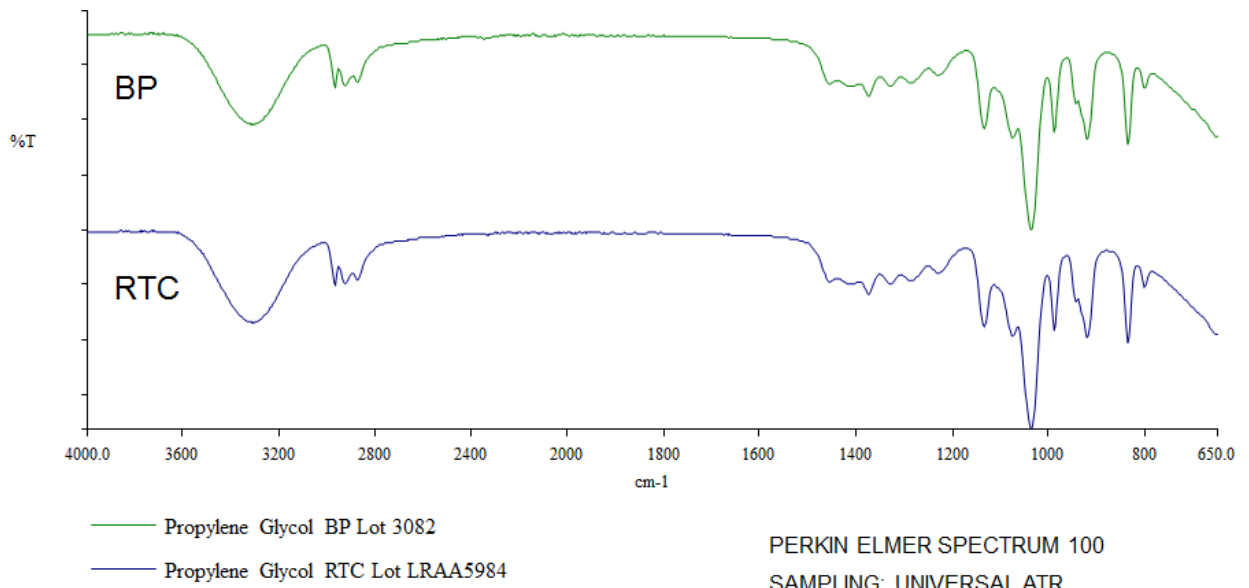
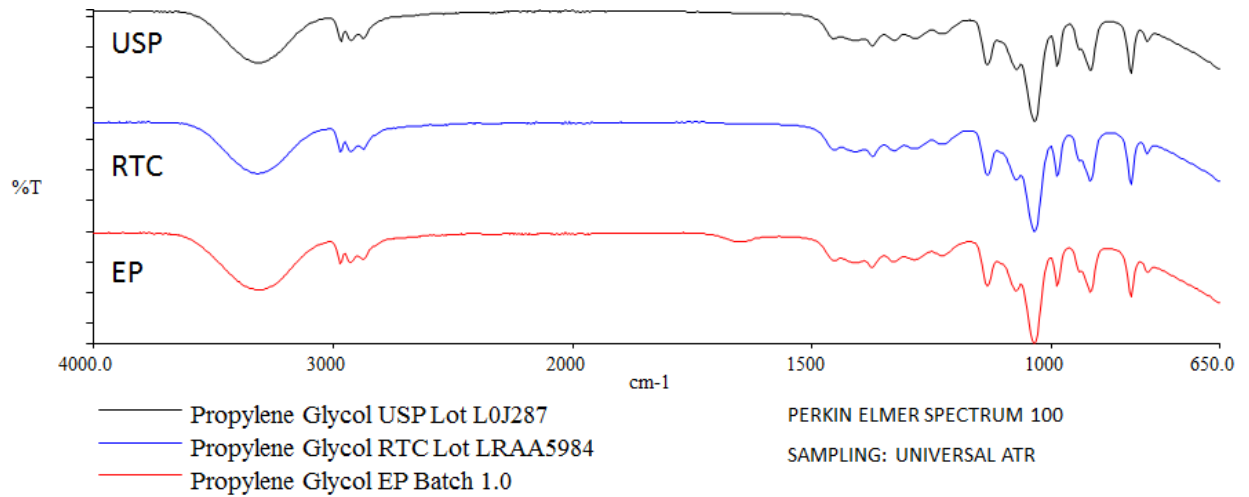
Mean of three determinations, Residue = **0.0009%**

### CERTIFIED PURITY BY MASS BALANCE [100% - Impurities (normalized)]

**99.9%**  $U_{\text{crm}} = \pm 0.2\%$ ,  $k = 2$   
(as is basis)

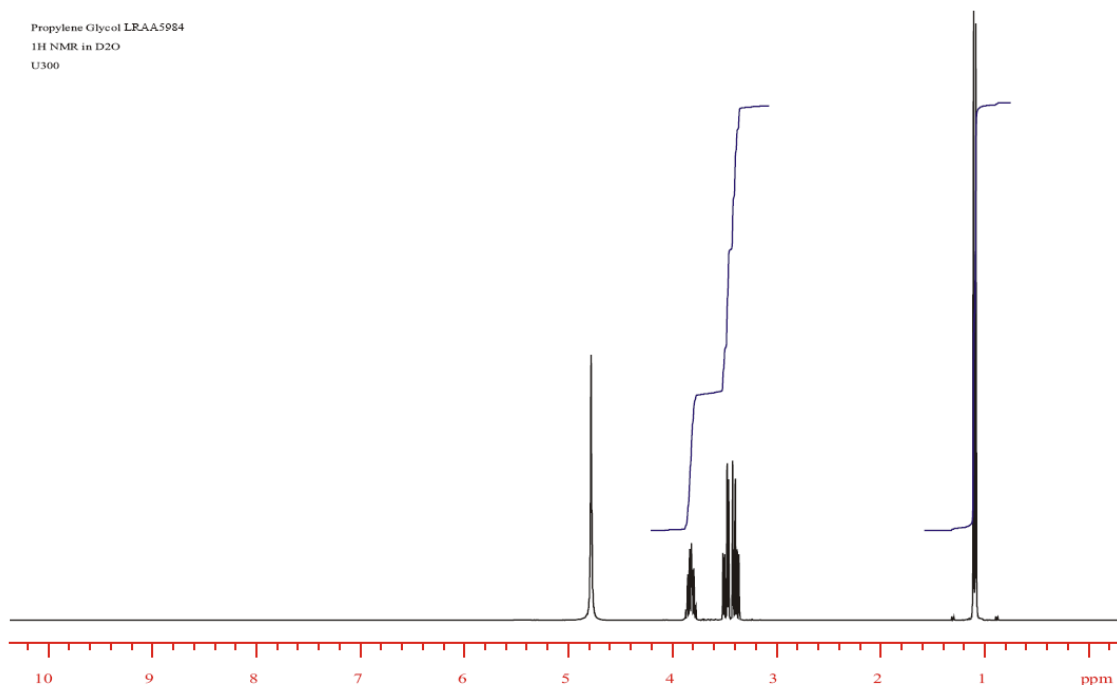
## IDENTIFICATION TESTS

**INFRARED SPECTROPHOTOMETRY** (Comparative identification analysis demonstrates direct traceability to Pharmacopeial standards)



**<sup>1</sup>H NMR** (Data provided by an external laboratory; not in scope of accreditation)

Propylene Glycol LRAA5984  
<sup>1</sup>H NMR in D<sub>2</sub>O  
 U300



Consistent with structure

**ELEMENTAL ANALYSIS** (Data provided by an external laboratory; not in scope of accreditation)

Exeter Analytical 440 Elemental Analyzer

Combustion method

%	Theoretical	Result 1	Result 2	Mean
C	47.36	47.39	47.40	47.40
H	10.60	10.51	10.52	10.52

**BOILING POINT**

Specification: 184°C to 189°C (EP)

Mettler Toledo FP900 Thermosystem with FP81 Measuring Cell

Mean of three measurements = **187.6 °C (corrected)**

**REFRACTIVE INDEX**

Specification: 1.431 to 1.433 (EP)

Mettler Toledo RA-510M Refractoanalyzer

Temperature: 20°C

Mean of three measurements = **1.432**

### HOMOGENEITY ASSESSMENT

Homogeneity was assessed in accordance with ISO Guide 35. Completed units were sampled using a random stratified sampling protocol. The results of chemical analysis were then compared by Single Factor Analysis of Variance (ANOVA). The uncertainty due to homogeneity was derived from the ANOVA. Heterogeneity was not detected under the conditions of the ANOVA.

Analytical Method: GC

Sample size: ~50mg

### UNCERTAINTY STATEMENT

Uncertainty values in this document are expressed as Expanded Uncertainty ( $U_{\text{crm}}$ ) corresponding to the 95% confidence interval.  $U_{\text{crm}}$  is derived from the combined standard uncertainty multiplied by the coverage factor  $k$ , which is obtained from a  $t$ -distribution and degrees of freedom. The components of combined standard uncertainty include the uncertainties due to characterization, homogeneity, long term stability, and short term stability (transport). The components due to stability are generally considered to be negligible unless otherwise indicated by stability studies.

### STABILITY ASSESSMENT

Significance of the stability assessment will be demonstrated if the analytical result of the study and the range of values represented by the Expanded Uncertainty do not overlap the result of the original assay and the range of its values represented by the Expanded Uncertainty. The method employed will usually be the same method used to characterize the assay value in the initial evaluation.

Long Term Stability Evaluation - An assessment, or re-test, versus a Compendial Reference Standard may be scheduled, within the 3 year anniversary date of a release of a Secondary Standard. The re-test interval will be determined on a case-by-case basis.

Short Term Stability Study - It is useful to assess stability under reasonably anticipated, short term transport conditions by simulating exposure of the product to humidity and temperature stress. This type of study is conducted under controlled conditions of elevated temperature and humidity.



Operations Manager



QA Supervisor

### APPENDIX

Original Release Date: 10 November 2014