ANALYSIS OF ETHYLENE GLYCOL (EG), PROPYLENE GLYCOL (PG), AND DIETHYLENE GLYCOL (DEG) USING GAS CHROMATOGRAPHY AND THE LUMA™ MULTI-CHANNEL VACUUM ULTRAVIOLET (VUV) DETECTOR

VUV ANALYTICS

INTRODUCTION:

The presence and quantification of Ethylene Glycol (EG), Propylene Glycol (PG), and Diethylene Glycol (DEG) in consumer products are a crucial concern due to their potential toxicity to humans. While these compounds are commonly used in a variety of applications, from antifreeze to cosmetics, their ingestion or absorption can have harmful health impacts, with DEG being especially harmful.

Regulatory bodies such as the U.S. Food and Drug Administration (FDA) have provided guidelines to determine the levels of these glycols in consumer products. However, conventional methods can be time-consuming, complex, and may require extensive sample preparation.

This technical brief introduces a streamlined approach to this analysis using LUMA, an advanced gas chromatography detector that leverages vacuum ultraviolet (VUV) spectroscopy. The method uses 1,3-Propanediol as an internal standard, providing accurate and reliable quantification. This approach aligns with the FDA's method but employs LUMA's unique capabilities to simplify the process and increase the robustness of the analysis.

With LUMA, laboratories can gain high sensitivity, a broad linear dynamic range, and straightforward sample preparation procedures. This novel approach enables accurate, reliable, and time-efficient analysis of EG, PG, and DEG in various consumer products. The subsequent sections of this technical brief will provide detailed methodology and analysis results, illustrating the power and effectiveness of LUMA for this critical application.

The Glycols analysis was carried out utilizing an Agilent 8890 Gas Chromatograph coupled to a LUMA Multi-Channel Vacuum Ultraviolet Absorbance Detector powered by OpenLab CDS. Method conditions for this experiment are described in Table 1.

GC Conditions	LUMA Conditions
Injection Volume: 1µL	**Makeup Gas Pressure:** 14 PSI N_2
Inlet Temperature: 250°C	**System Gas Pressure:** 50 psi N_2
Split Ratio: Splitless 0.75 min	**Flow Cell Temperature:** 275°C
Column: Restek Stabilwax (30m x 0.25mm, 0.25µm)	**Transfer Line Temperature:** 275°C
Carrier gas: Hydrogen @ 2.5mL/min	**Acquisition Rate:** 10 Hz
Oven Program: 60°C, hold 0.75 min; 10°C/min to 215°C (0 min)	
Run Time: 16.25 minutes	

Table 1 – Instrument conditions for analysis of Glycols.

ANALYSIS OF ETHYLENE GLYCOL (EG), PROPYLENE GLYCOL (PG), AND DIETHYLENE GLYCOL (DEG) USING GAS CHROMATOGRAPHY AND THE LUMA™ MULTI-CHANNEL VACUUM ULTRAVIOLET (VUV) DETECTOR

RESULTS:

- EG, PG, and DEG were analyzed from a range of 50 ppb – 0.18 % (1,800 ppm) with excellent %RSDs and linearity.
- 12-point LUMA Spectra for the glycols generated unique spectra shapes for all analytes of interest allowing for easy qualitative identification.

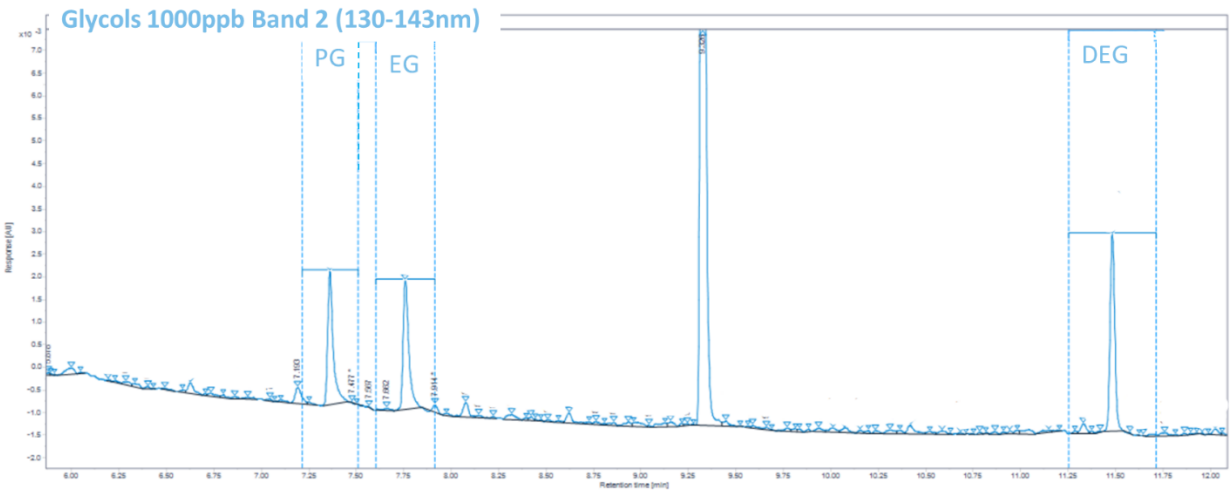

Figure 1 – Glycols PG, EG and DEG @ 1ppm in Band 2 (130-143nm)

Propylene Glycol, Ethylene Glycol, and Diethylene Glycol showed at 1000ppb, for maximum sensitivity band 2 was selected as the quantitation Band for the remainder of the analysis. The other bands served as a qualitative way to ensure the identity of the glycols based on their respective band ratios. With LUMA 3 dimensions of analyte information are obtained, retention time, peak response as well as Band ratios relative to the other Bands in the resulting chromatograms. These 3 dimensions allow greater confidence in results and avoids false positives as can be the case with other detectors where only retention times and peak areas are observed as part of the chromatographic results during the analysis.

Figures 2 and 3 show the LUMA spectra and High-Resolution VUV Spectra for the analytes of interest EG, PG, DEG as well as for the internal standard 1,3-Propanediol.

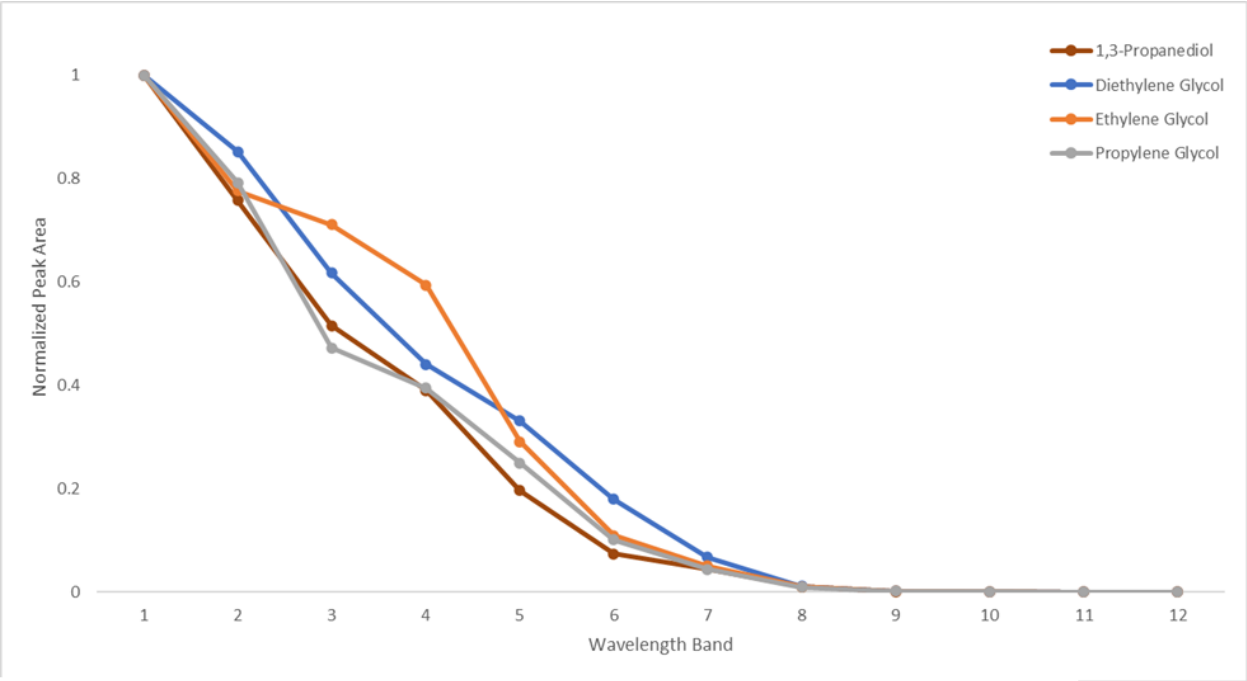

Figure2 - *LUMA Spectra for EG, PG and DEG Analytes and 1,3-Propanediol ISTD*

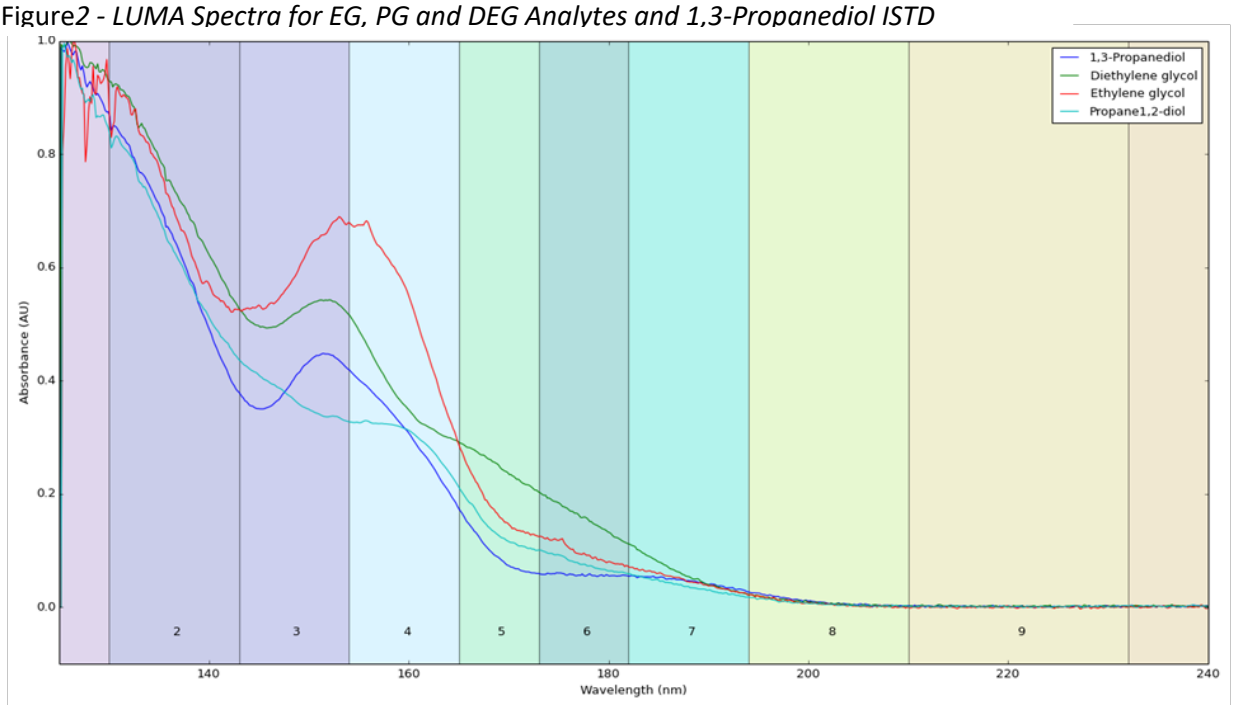

Figure 3 - High-Resolution VUV Spectra for EG, PG, and DEG Analytes and 1,3-Propanediol ISTD

The analytes absorb in Bands 1-6, and any of these bands can be used for quantitation purposes depending on the desired level of sensitivity. For maximum sensitivity select Bands 1 or 2, for less sensitive analysis you may choose to quantitate utilizing Bands 3-6. The other Bands may be used as a qualifier as the ratios between bands will always be the same independent of analyte concentrations.

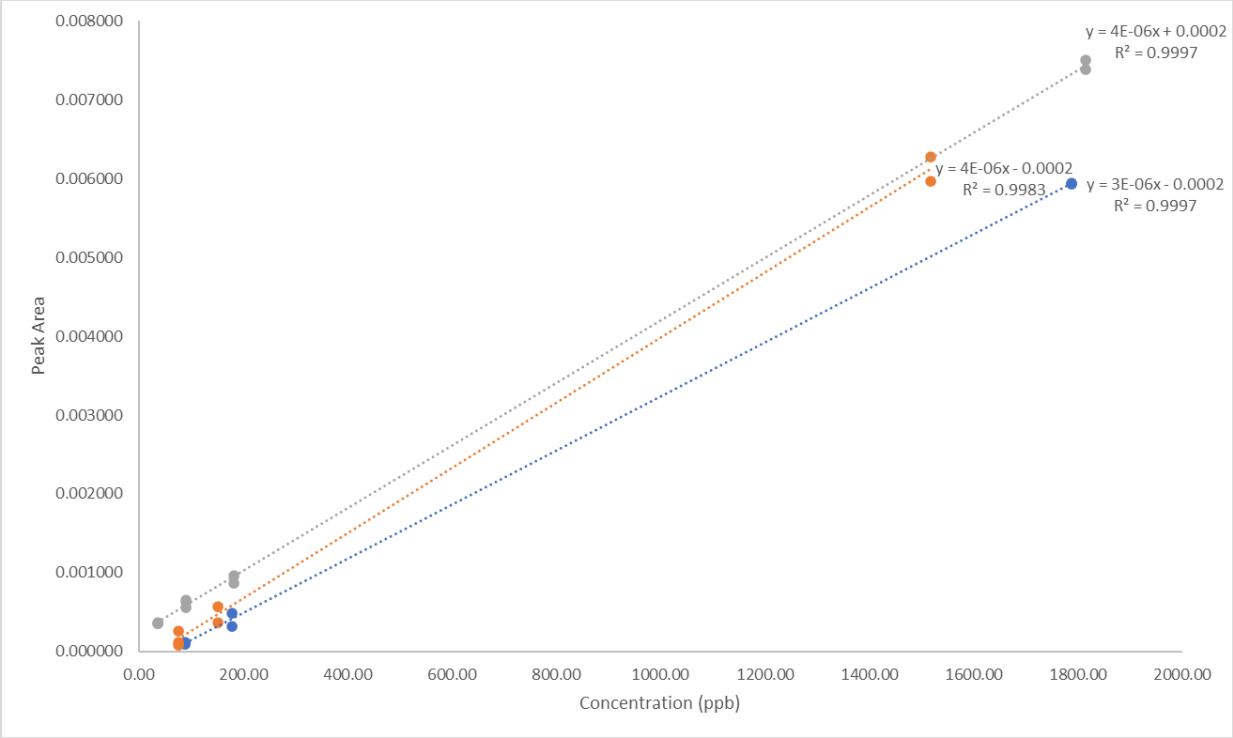

Figure 4 – Linearity of EG, PG, and DEG from 50ppb – 1.8ppm

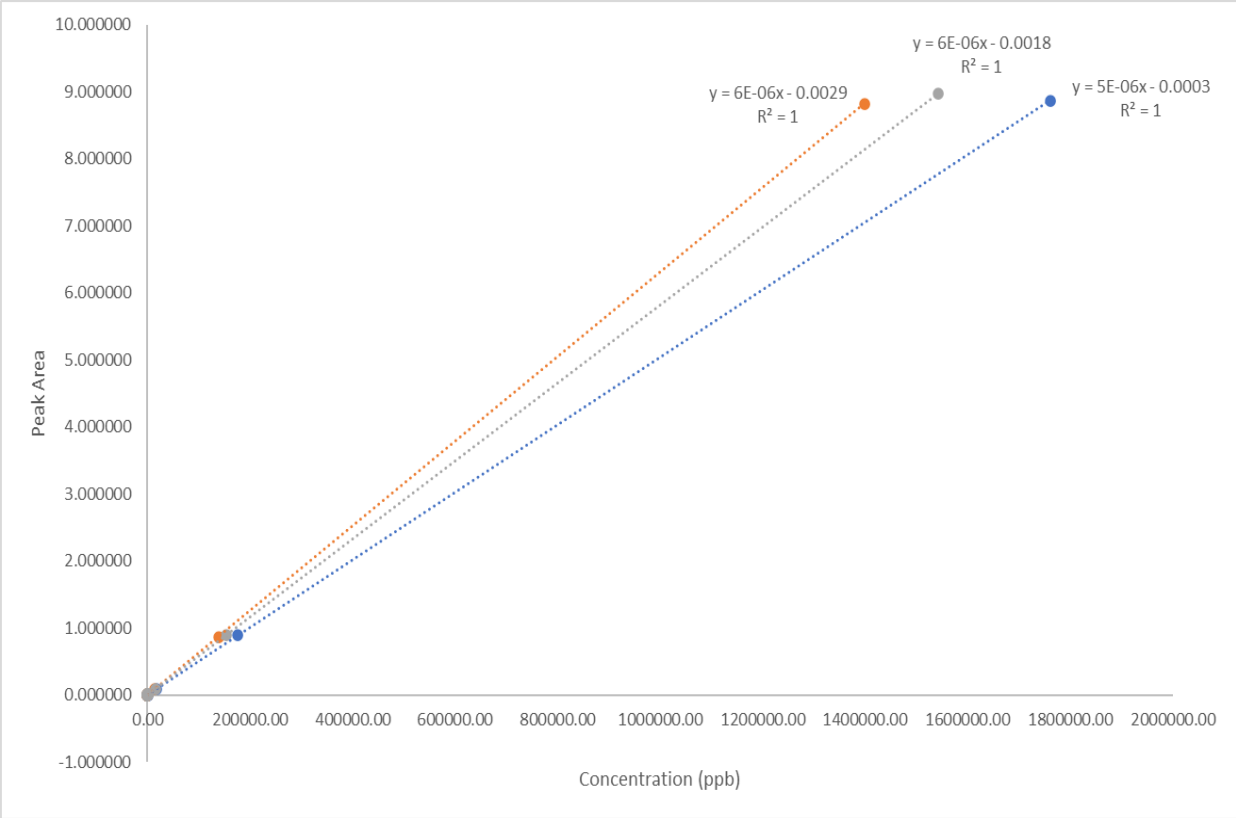

Figure 5 – Linearity of EG, PG, and DEG from 50ppb – 1,800ppm (0.18%)

Repeatability Peak Area Response for EG, PG, and DEG at (150 ppb, 1.5 ppm, and 150 ppm):

EG	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	AVG	%RSD
150 ppm	8.99E-01	8.93E-01	9.16E-01	9.17E-01	9.07E-01	9.00E-01	8.94E-01	9.04E-01	**1.08**
1.5 ppm	9.93E-03	9.80E-03	9.80E-03	9.86E-03	1.05E-02	9.74E-03	9.66E-03	9.90E-03	**2.84**
150 ppb	1.34E-03	1.60E-03	1.49E-03	1.39E-03	1.44E-03	1.76E-03	1.54E-03	1.51E-03	**9.36**

PG	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	AVG	%RSD
150 ppm	1.11E+00	1.10E+00	1.13E+00	1.13E+00	1.12E+00	1.11E+00	1.10E+00	1.11E+00	**1.00**
1.5 ppm	1.23E-02	1.20E-02	1.23E-02	1.13E-02	1.15E-02	1.19E-02	1.21E-02	1.19E-02	**3.28**
150 ppb	9.46E-04	9.72E-04	1.11E-03	1.07E-03	1.14E-03	1.07E-03	1.18E-03	1.07E-03	**7.97**

DEG	Run 1	Run 2	Run 3	Run 4	Run 5	Run 6	Run 7	AVG	%RSD
150 ppm	1.18E+00	1.16E+00	1.18E+00	1.19E+00	1.17E+00	1.16E+00	1.16E+00	1.17E+00	**0.98**
1.5 ppm	1.33E-02	1.32E-02	1.32E-02	1.35E-02	1.35E-02	1.34E-02	1.33E-02	1.33E-02	**0.84**
150 ppb	2.13E-03	2.95E-03	2.85E-03	2.56E-03	2.85E-03	2.94E-03	2.97E-03	2.75E-03	**11.08**

Table 2 – N = 7 replicate injections of glycols at various concentrations.

As can be seen from the above results LUMA provides excellent linearity, sensitivity, and reproducibility for all analytes of interest providing extra confidence in the obtained results.

CONCLUSION:

The application of LUMA in the analysis of Ethylene Glycol (EG), Propylene Glycol (PG), and Diethylene Glycol (DEG) in consumer products, using 1,3-Propanediol as an internal standard, has proven to be exceptionally effective. The results showcased in this technical brief demonstrate that LUMA's unique capabilities significantly enhance the robustness and efficiency of the analysis.

Notably, the excellent reproducibility of the method, as indicated by the low %RSD values, underscores the precision of LUMA. The data further reveals excellent linearity, indicating that the method performs consistently across a wide concentration range. Moreover, the wide dynamic range provided by LUMA ensures that this method is adaptable for both low and high concentration levels without any loss in precision or accuracy. Perhaps most significantly, LUMA offers superb sensitivity, detecting levels of EG, PG, and DEG down to 50 ppb. This sensitivity level is well below the regulatory limits, ensuring safety and compliance with regulations.

In conclusion, LUMA provides a powerful tool for the analysis of EG, PG, and DEG in consumer products. By combining efficiency, precision, and exceptional sensitivity, it allows labs to meet regulatory demands while enhancing their analytical workflow. Through its application, laboratories can ensure the safety of consumer products, protect public health, and ensure regulatory compliance.