

FT-IR Spectroscopy

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Verifying Adulterant Screen Detection Limits of Glycerol Adulteration with Diethylene Glycol

properties allowing it to act as a sweetener, lubricant and humectant.¹ The glycerol industry is a growing and profitable market with recent estimations predicting that it will be worth more than \$5 billion by 2024.²

Unscrupulous suppliers may adulterate glycerol to increase their profits. Diethylene glycol (DEG), the most common adulterant, has a number of properties similar to glycerol and is roughly three times cheaper. However, ingestion of DEG is toxic and thousands of reported cases of DEG poisoning have resulted in illness and fatalities worldwide.³

The current FDA safety limit for DEG in glycerol is 0.1 % by weight.⁴ As such, detection methods for DEG adulteration in glycerol must be able to identify DEG present at this concentration. Current detection methods tend to involve chromatographic techniques which provide low limits of detection but can require time-consuming sample preparation and may produce hazardous chemical waste.

Introduction

Glycerol (or glycerin) is a colorless, odorless liquid that is generally safe for human exposure and consumption. It has many uses in the food, pharmaceutical and personal care industries due to its range of versatile

Mid-infrared spectroscopy with Adulterant Screen™ technology can rapidly identify and semi-quantitatively estimate the level of DEG adulteration present in glycerol samples, without the need for solvents. Adulterant Screen also provides a detection limit estimate that the method should be capable of reaching.

Previous studies using mid-infrared spectroscopy to determine DEG adulteration were unable to detect down to the necessary 0.1 % safety limit due to an insufficient number of calibration standards being available.⁵ The present study uses a greater number of calibration standards to achieve a sub per cent level detection limit and prove that Adulterant Screen can detect DEG adulteration in glycerol at this level.

Experimental

36 spectra of pure glycerol (12 spectra from three different brands) and one spectrum of diethylene glycol were collected using a PerkinElmer Spectrum Two™ FT-IR Spectrometer with a Universal Attenuated Total Reflectance (UATR) accessory, using the parameters shown in Table 1.



Figure 1. PerkinElmer Spectrum Two with Universal Attenuated Total Reflectance accessory.

Table 1. Scanning parameters for the analysis of glycerol and diethylene glycol.

Scanning Parameters	
Spectral Range	4000 – 450 cm ⁻¹
Resolution	4 cm ⁻¹
Number of Scans	8

Table 2. Adulterant Screen results for a series of glycerol samples adulterated with diethylene glycol.

Specified Diethylene Glycol Concentration (%)	Average Predicted Diethylene Glycol Concentration (%)	Standard Deviation (%)	Detection Limit (%)	Adulterant Screen Pass/Fail
10.60	10.24	1.604	0.043	Fail
5.34	5.69	0.229	0.043	Fail
2.69	2.95	0.098	0.043	Fail
1.37	1.54	0.089	0.043	Fail
0.69	0.63	0.049	0.043	Fail
0.35	0.35	0.017	0.043	Fail
0.17	0.21	0.009	0.043	Fail
0.09	0.07	0.019	0.043	Fail
0.05	0.03	0.023	0.043	Fail
0.00	-	-	-	Pass

The Adulterant Screen algorithm detects, identifies and semi-quantitatively estimates the level of adulterant present in a sample, without needing to develop a quantitative calibration model. An Adulterant Screen method simply requires spectra from a representative population of pure glycerol samples and one spectrum of each possible adulterant. The unadulterated material spectra should represent as much variation as possible, such as samples from different batches or suppliers.

An Adulterant Screen method was created by inputting all pure glycerol spectra as 'Material Spectra' and the diethylene glycol spectrum as 'Adulterant Spectra' within Spectrum 10 software.

The spectra were pre-processed using a first derivative baseline correction. The method was then tested by running a series of glycerol samples adulterated with known levels of DEG (10.60 – 0.05 % (w/w)). Each sample was run in triplicate and an average predicted adulteration level was calculated. The results are shown in Table 2.

The Adulterant Screen method correctly failed all adulterated samples and passed the pure glycerol sample. The method also provided a detection limit of 0.043 %. The results show that Adulterant Screen was able to detect and semi-quantitatively estimate the level of DEG adulteration down to sub per cent levels of 0.05 %, therefore demonstrating that it is capable of reaching the detection limit provided by the algorithm. The results also show that Adulterant Screen technology is capable of detecting DEG adulteration in line with the FDA safety limit of 0.1 %.

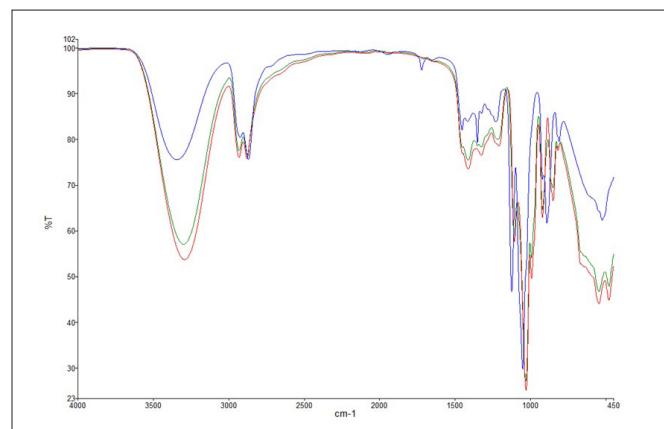


Figure 2. Mid-IR spectra of pure glycerol (red), diethylene glycol (blue) and 10.60 % diethylene glycol in glycerol (green).

Adulterant Screen methods can be incorporated into the Spectrum Touch™ software to provide a user-friendly interface for use by routine operators. Figure 3 shows the simple design of the guided user workflow and the results provided by the software. The estimated level of DEG and the detection limit are given as a decimal.

The figure consists of three vertically stacked screenshots of the Spectrum Touch software interface:

- Screenshot 1 (Top):** A welcome screen titled "Diethylene Glycol Adulteration in Glycerol". It features a photograph of a glass flask containing a clear liquid, labeled "DEG in Glycerol". To the right is a text box with "Instructions" and a "Next" button at the bottom right.
- Screenshot 2 (Middle):** A "Detailed Results" screen showing "Sample 1 of 1". The table contains the following data:

Sample Name	Sample 9 Rep 1 (0.05 % DEG)
Description	0.05 % Diethylene Glycol in Glycerol
Adulterant Screen Pass/Fail	Fail
Adulterant	Diethylene Glycol
Confidence	Possible

 On the right side of the screen are buttons for "More", "Print", and "Report", along with a "Next" button at the bottom right.
- Screenshot 3 (Bottom):** A "Detailed Results" screen showing "Sample 1 of 1". The table contains the following data:

Sample Name	Sample 9 Rep 1 (0.05 % DEG)
Description	0.05 % Diethylene Glycol in Glycerol
Material Name	Diethylene Glycol
Adulterant	Diethylene Glycol
Level	0.00058
Detection Limit	0.00043
Confidence	Possible

 An additional column titled "Adulterant Screen details" is present, showing "(Best hit) Diethylene Glycol". On the right side of the screen are buttons for "More", "Print", and "Report", along with a "Next" button at the bottom right.

Figure 3. Example of Spectrum Touch workflow and Adulterant Screen results for diethylene glycol adulteration in glycerol.

Conclusion

The results show that mid-infrared spectroscopy with Adulterant Screen technology can provide a rapid screening technique to detect DEG adulteration in glycerol. The Adulterant Screen method was able to detect and semi-quantitatively estimate DEG adulteration down to sub per cent levels, therefore meeting the FDA safety limit of 0.1 % and the detection limit provided by the algorithm.⁴ The incorporation of the method within Spectrum Touch software allows quick and easy routine checks to be carried out by untrained operators.

References

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