Application Note No. 095

Analysis of Ethylene Glycol in Wastewater by Direct Aqueous Injection

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Introduction

For many years glycol based products have been used for freeze up, boil over and corrosion protection in most vehicle's cooling systems. The majority of antifreezes in use today are based on ethylene glycol, but propylene glycol products are becoming more common. They both have a high boiling point, low vapour pressure, excellent heat transfer abilities and the ability to depress the freezing point of water, but ethylene glycol is a slightly more effective freezing point depressant.

Ethylene glycol is a colourless, odourless, syrupy liquid and is completely soluble in water. It has a maximum freeze protection concentration of 67% ethylene glycol in water. However, ethylene glycol is poisonous. It can cause irritation, vomiting and headaches and may be a teratogen[1]. It is necessary to test for contamination of the waste stream in sites where antifreeze is stored or waste coolant is disposed of, as increased levels of glycols in rivers can cause an increase in the Biological Oxygen Demand (BOD). In severe cases of contamination, aquatic life can be killed through oxygen starvation rather than direct poisoning. Presented here is a method for the direct aqueous injection of wastewater to analyse for ethylene glycol.

Instrumentation

- Optic 3 Programmable Injector
- Focus Robotic Sample Processor
- Agilent 5890 GC with FID
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Analysis Method

- Dilute ethylene glycol in HPLC grade water to make calibration standards
- Inject 2uL of calibration standard or a water sample under cold split conditions
- Separate glycols on an SGE SolGel Wax column

Conditions

Optic Conditions:

Injection volume:	2 µL
Liner style:	Fritted
Initial temperature:	60°C
Ramp rate:	16 °C/s
Final temperature:	250 °C
Transfer column flow:	2.3 mL/min
Transfer time:	2 mins
Initial column flow:	2.3 mL/min
Final column flow:	2.3 mL/min
Split flow:	100 mL/min
Saver split flow:	50 mL/min

GC Conditions:

Column: SGE SolGel Wax, 30 m x 0.25 mm i.d. x 0.25 µm

Oven program:	100 °C (hold 2 mins)
	10 °C/min to 230 °C
Carrier gas:	Helium

FID Conditions:

Temperature:	250°C
Hydrogen flow:	40 mL/min
Air flow:	400 mL/min

Results

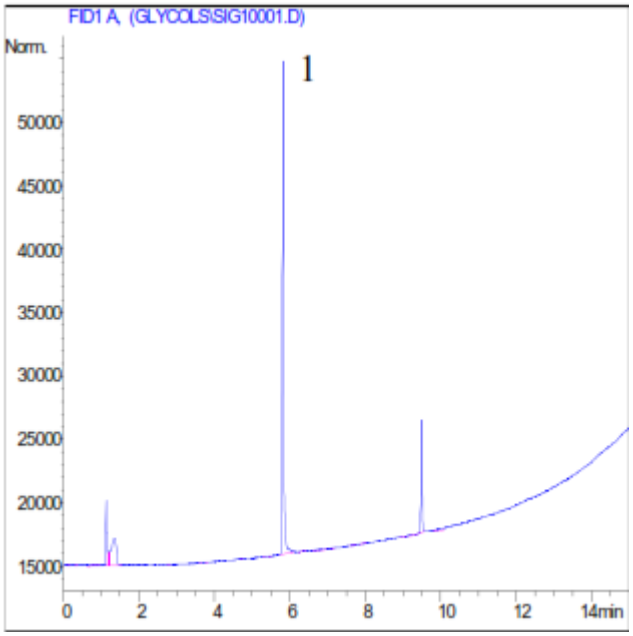

Figure 1: *2uL cold split injection of 5ppm ethylene glycol (1) standard*

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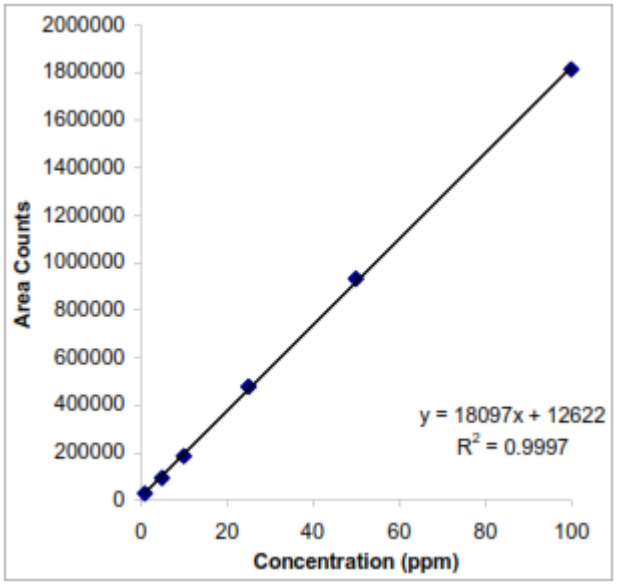

Figure 2: *Calibration of ethylene glycol, concentration from 1 ppm to 100 ppm*

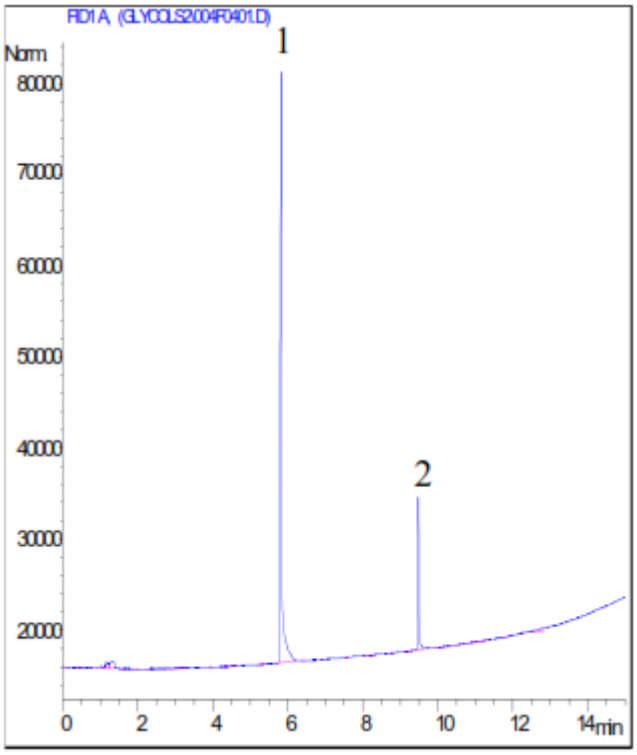

Figure 3: *2uL cold split injection of waste water: (1) ethylene glycol; (2) diethylene glycol*

Table 1: Repeatability

Injection	Area counts
1	194617.2
2	198166.3
3	196152.9
4	202825.3
5	204092
6	204989.5
7	202210.7
8	200436
9	205781.6
Mean	**201030.2**
StdDev	**3964.09**
%RSD	**1.97**

Conclusions

The direct aqueous injection using the Optic 3 Programmable Injector provides a successful method for the analysis of ethylene glycol in wastewater.

The calibration is linear between 1 ppm and 100 ppm, with a correlation coefficient of 0.9997 and the repeatability on a wastewater sample is good, with a %RSD of less than 2%. The limit of detection was by no means stretched; looking at these levels a 1uL injection would be adequate.

By keeping the oven hot during injection, condensation of the water on the column is prevented, and using the SolGel Wax column with a fast column flow the peak shape of the glycols is good.

This application could easily be transformed into an online method using the Focus Flowcell. The effluent could be pumped through the flowcell and a Focus equipped with a 10uL syringe would inject the sample directly from the stream into the injection port.

Acknowledgements

We would like to thank Duncan Taylor from SGE for providing the column.

References

(1) New Jersey Department of Health and Senior Services, Hazardous Substances Data Sheet