
We recommend you cite the published version.
The publisher’s URL is: http://eprints.uwe.ac.uk/35854/

Refereed: Yes

(no note)

Disclaimer

UWE has obtained warranties from all depositors as to their title in the material deposited and as to their right to deposit such material.

UWE makes no representation or warranties of commercial utility, title, or fitness for a particular purpose or any other warranty, express or implied in respect of any material deposited.

UWE makes no representation that the use of the materials will not infringe any patent, copyright, trademark or other property or proprietary rights.

UWE accepts no liability for any infringement of intellectual property rights in any material deposited but will remove such material from public view pending investigation in the event of an allegation of any such infringement.

PLEASE SCROLL DOWN FOR TEXT.
Direct Thermal Desorption Gas Chromatographic Determination of Toxicological Relevant Concentrations of Ethylene Glycol in Whole Blood

James Robson, Stephen Townsend, Paul Bowdler and Kevin C. Honeychurch*

Department of Applied Sciences, Faculty of Health and Life Sciences, University of the West of England, Frenchay Campus, Coldharbour Lane, Bristol, BS16 1QY, UK. *Kevin.honeychurch@uwe.ac.uk, Tel. +44(0)1173287357
Outline of talk

- Ethylene glycol
- Problems of analysis
- Our new approach
- Optimisation studies
- Analytical performance characteristics
- Analytical application
- Areas of future work
- Conclusions
- Acknowledgements
Ethylene Glycol

\[
\begin{align*}
\text{H}_2 & \quad \text{C} \quad \text{C} \quad \text{OH} \\
\text{HO} & \quad \text{C} \quad \text{C} \quad \text{OH} \\
\text{H}_2
\end{align*}
\]

Ethylene Glycol

Chemical pathway:

- Ethylene Glycol
- Alcohol Dehydrogenase
- Glycoaldehyde
- Glycolate
- Glyoxylate
- Oxalate

Toxicity warning:

Skull and crossbones icon
Problems of Analysis

• Detection of ethylene glycol is very analytical challenging
  o Analysis of serum osmol and anion gap.
  o Detection of other common components of ethylene glycol antifreeze formulations, such as fluorescein have been reported.

• Enzyme-based assays poor sensitivity (300 mg/dL).

• Does not lend itself to LC/MS or HPLC
  o Small molecular mass and lack of a chromophore. Refractive Index detection, lacks specificity and sensitivity.

• Gas chromatography (GC) is the most commonly employed laboratory based approach. However, methods are laborious and problematic; based on headspace, direct aqueous injection, or requiring complex derivatisation steps.
Our New Approach

Figure 1. Flow diagram of ethylene glycol determination in whole blood sample by TD-GC.
Optimisation studies on aqueous 25 mM ethylene glycol

**Figure 2.** Effect of drying time at 100 °C on ethylene glycol chromatographic peak height.

**Figure 3.** Effect of desorption temperature on ethylene glycol chromatographic peak height.
Figure 4. Effect of desorption time based on resulting ethylene glycol chromatographic peak height.

Figure 5. Calibration curve for ethylene glycol. Theoretical limit of detection, based on 3σ, was calculated as 50.2 µM (3.11 mg/L) of ethylene glycol with the limit of quantification defined as 1.0 mM (62.1 mg/L) for a 1 µL sample.
**Figure 6.** Gas chromatogram showing the separation of ethylene glycol (i) and the internal standard, 1,2-butanediol (ii) in presence of methanol (ND), ethanol (ND), 1,4-butanediol (iii), γ-butyrolactone (iv) and fomepizole (v); 45 mM of each compound. No further peaks were detected after 6 minutes. ND = not detected.
Whole Blood Samples

Figure 7. Representative chromatograms of whole blood samples obtained by TD-GC for (a) whole blood (b) whole blood with internal standard (1,2-butanediol) 3.6 minutes (c) whole blood with ethylene glycol (3.2 minutes) and internal standard.

<table>
<thead>
<tr>
<th>Blood Sample</th>
<th>Native</th>
<th>Added, mM</th>
<th>Mean Found, mM</th>
<th>% Mean Recovery</th>
<th>%CV</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>ND</td>
<td>12.5</td>
<td>10.5</td>
<td>84.8</td>
<td>4.4</td>
</tr>
<tr>
<td>2</td>
<td>ND</td>
<td>20.0</td>
<td>19.5</td>
<td>96.7</td>
<td>2.3</td>
</tr>
<tr>
<td>3</td>
<td>ND</td>
<td>32.2</td>
<td>29.4</td>
<td>94.3</td>
<td>5.8</td>
</tr>
<tr>
<td>4</td>
<td>ND</td>
<td>100</td>
<td>107</td>
<td>107</td>
<td>3.9</td>
</tr>
<tr>
<td>5</td>
<td>ND</td>
<td>200</td>
<td>209</td>
<td>105</td>
<td>1.7</td>
</tr>
</tbody>
</table>

ND = not detected; %CV = percentage coefficient of variation

Table 1. Recovery and precision data for ethylene glycol obtained on whole blood.
Conclusions

• Extraction and derivatisation free method which is much faster, easier and cheaper.

• Free from interference from common endogenous blood components or other structurally similar compounds.

• It would be readily simple to also determine ethylene glycol concentrations in dry blood samples.

• As far as we are aware, this is the first report to describe this approach for the detection of any glycol.

• The assay could form the basis of a generic approach for the analysis of other alcohols, toxins and drugs.

• The small volumes of blood (nL-μL) utilised offer advantages for health and safety.
Acknowledgements

• We would like to thank the support of the Royal Society of Chemistry Analytical Chemistry Trust Fund.
• Kevin Sudlow and Sarah Almond are thanked for their technical assistance.
• Dr. Sophie Adamantos (University of Bristol) is thanked for her advise and help in areas of the veterinary sciences.

Reference

Available from: http://eprints.uwe.ac.uk/34416

Thank you for listening