Direct Thermal Desorption Gas Chromatographic Determination of Toxicological Relevant Concentrations of Ethylene Glycol in Whole Blood

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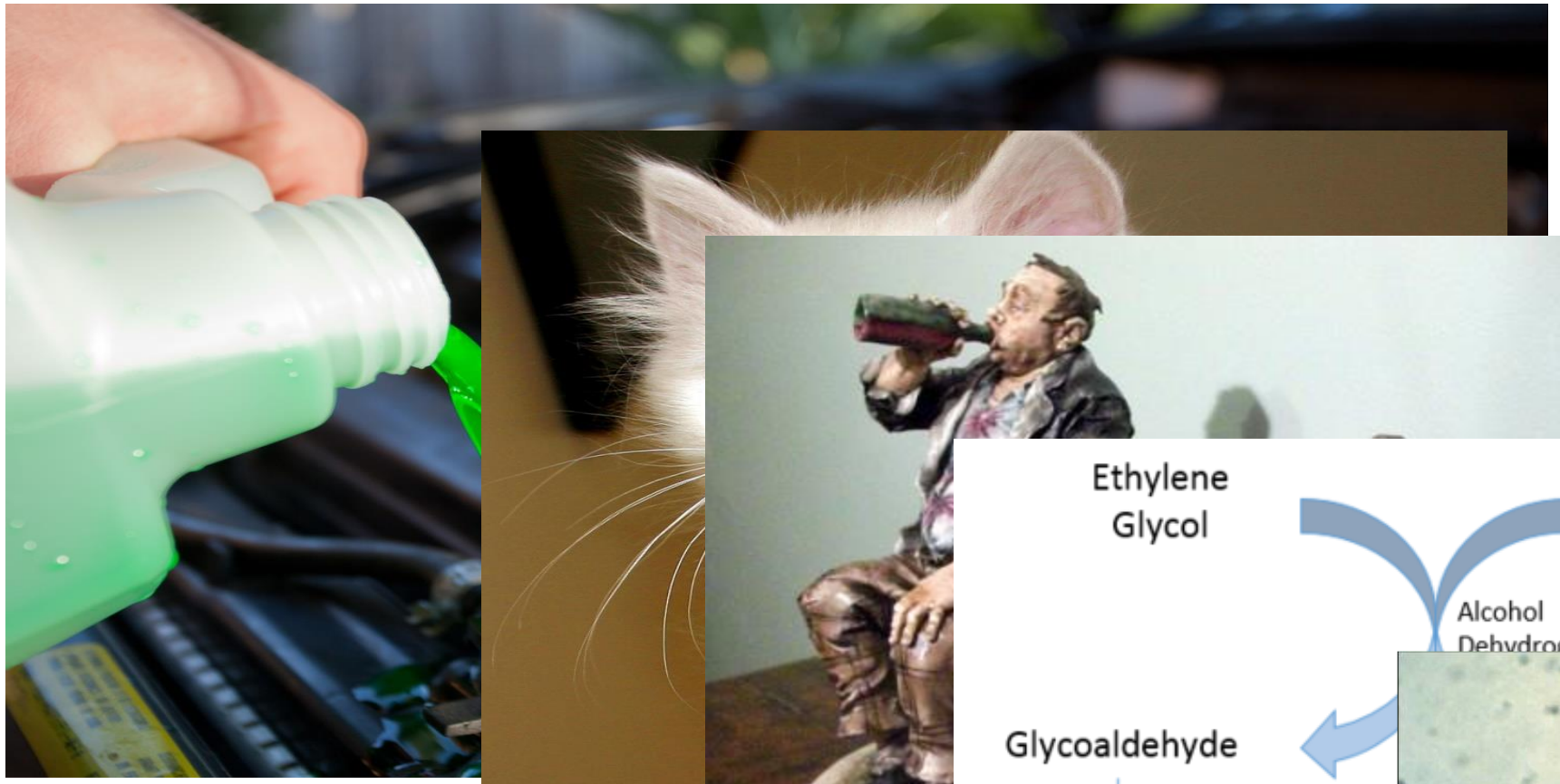
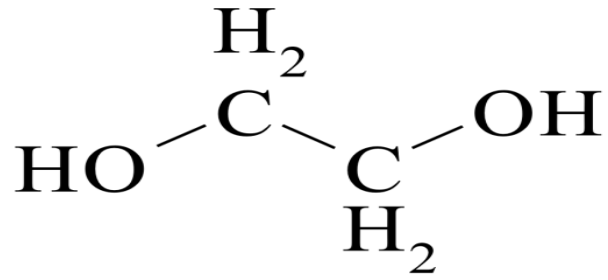
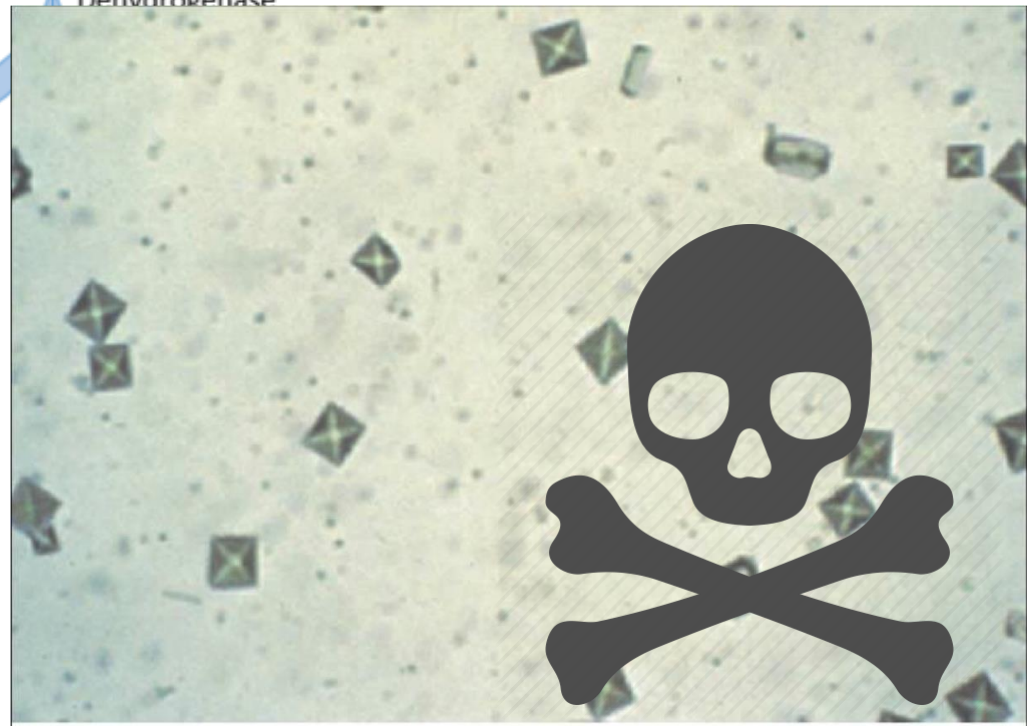
Outline of talk

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





Ethylene Glycol



Problems of Analysis

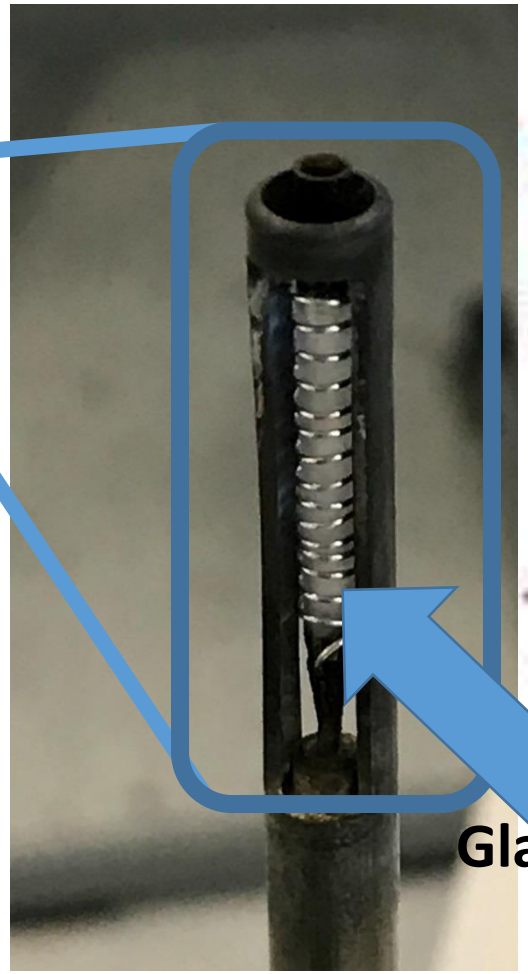
- **Detection of ethylene glycol is very analytical challenging**
  - Analysis of serum osmol and anion gap.
  - Detection of other common components of ethylene glycol antifreeze formulations, such as fluorescein have been reported.

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

- **Does not lend itself to LC/MS or HPLC**
  - 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



Glass wool

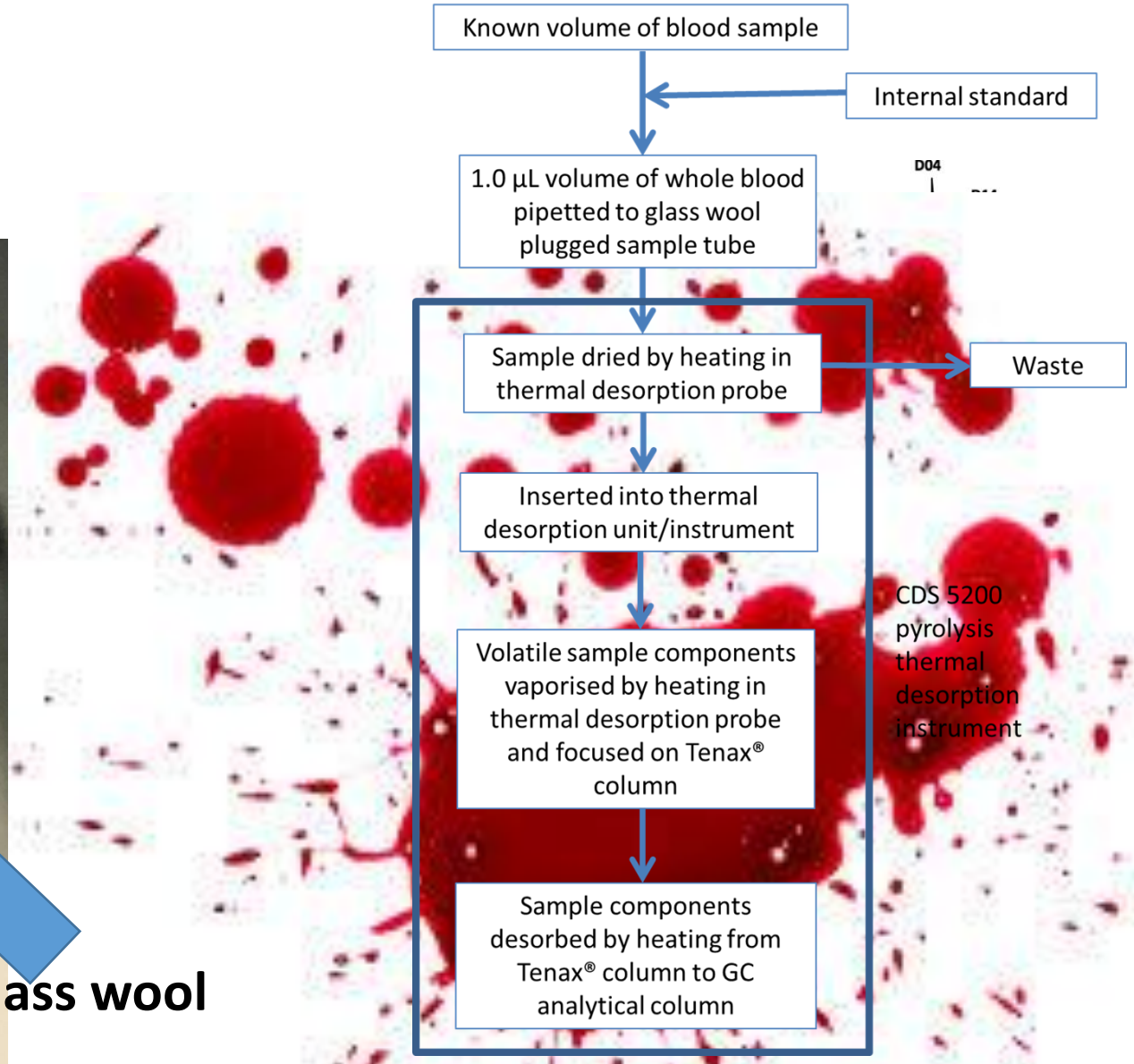


Figure 1. Flow diagram of ethylene glycol determination in whole blood sample by TD-GC.

M. Traoré *et al, J. Anal. Appl. Pyrolysis*, 2017, 126, 1-13.

Optimisation studies on aqueous 25 mM ethylene glycol

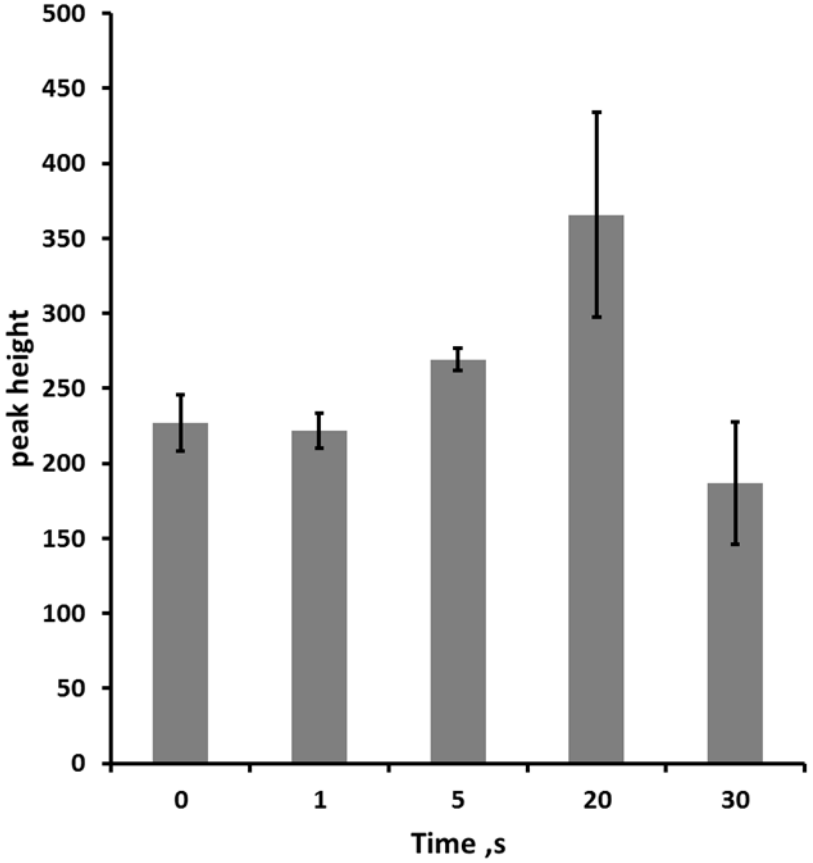


Figure 2. Effect of drying time at 100 °C on ethylene glycol chromatographic peak height.

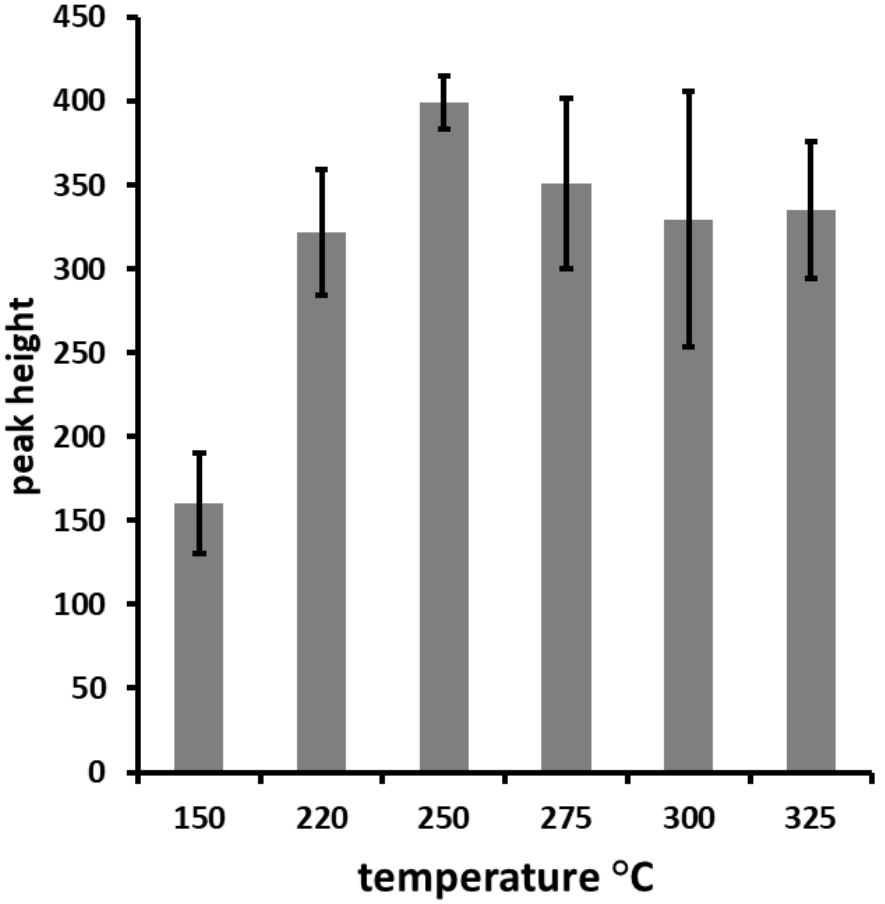


Figure 3. Effect of desorption temperature on ethylene glycol chromatographic peak height.

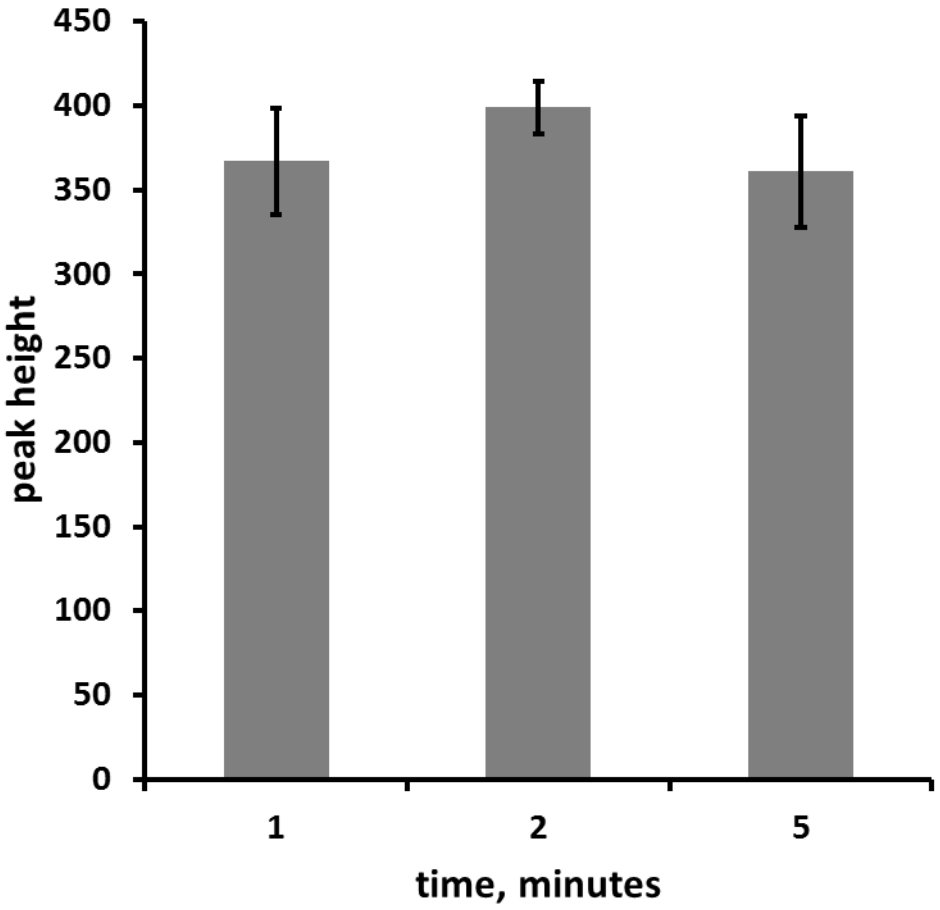


Figure 4. Effect of desorption time based on resulting ethylene glycol chromatographic peak height.

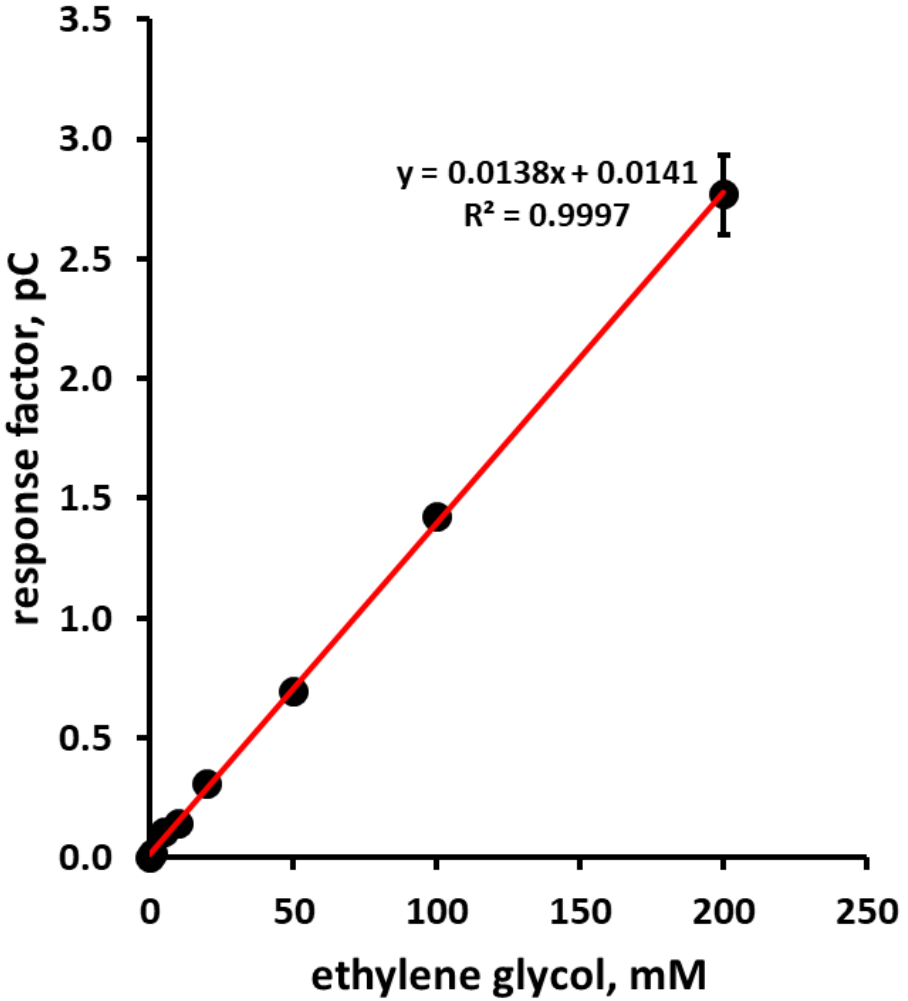
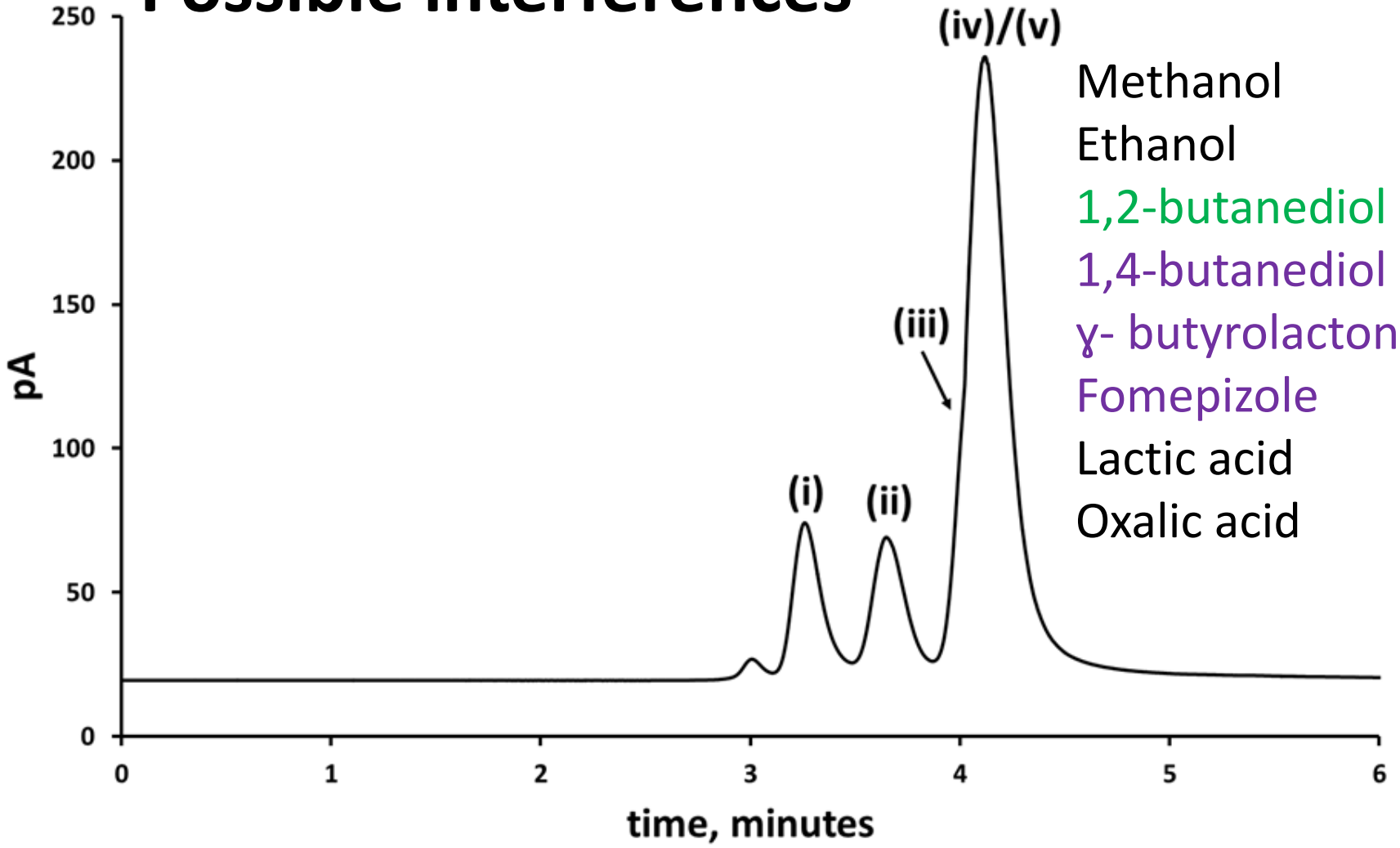


Figure 5. Calibration curve for ethylene glycol.

Theoretical limit of detection, based on 3 σ, 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.

Possible interferences



Methanol	ND
Ethanol	ND
1,2-butanediol	(ii)
1,4-butanediol	(iii)
ɣ- butyrolactone	(iv)
Fomepizole	(v)
Lactic acid	ND
Oxalic acid	ND

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), ɣ- butyrolactone (iv) and fomepizole (v); 45 mM of each compound. No further peaks were detected after 6 minutes. ND = not detected.

Whole Blood Samples

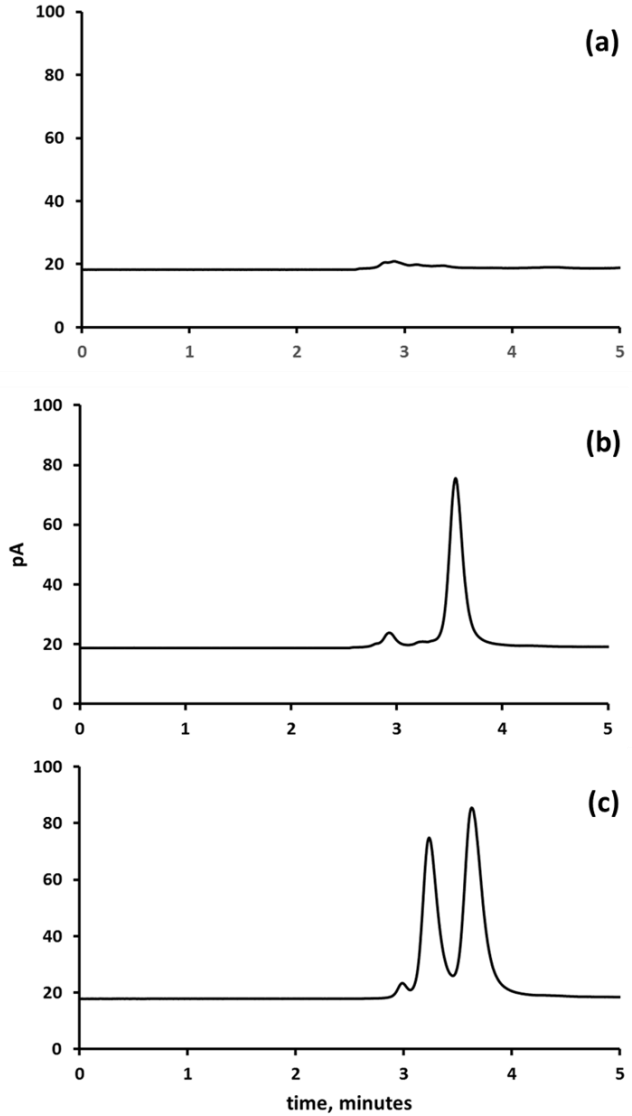


Figure 7. Representative chromatograms of whole blood samples obtained by TD-GC for (a) whole blood (b) whole blood with internal standard (1,2-butanediol) 3.6 minutes (c) whole blood with ethylene glycol (3.2 minutes) and internal standard.

Blood Sample	Native	Added, mM	Mean Found, mM	% Mean Recovery	%CV
1	ND	12.5	10.5	**84.8**	**4.4**
2	ND	20.0	19.5	**96.7**	**2.3**
3	ND	32.2	29.4	**94.3**	**5.8**
4	ND	100	107	**107**	**3.9**
5	ND	200	209	**105**	**1.7**

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

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Reference

Robson, J., Townsend, S., Bowdler, P. and Honeychurch, K. C., Direct thermal desorption gas chromatographic determination of toxicological relevant concentrations of ethylene glycol in whole blood. *Analyst*, 2018, 143, 963-969.
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