

Determination of Diacetyl in E-vapor Products by Gas Chromatography-Mass Spectrometry

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INTRODUCTION

Diacetyl (2,3-butanedione)

- Is a volatile, yellow liquid with an intense buttery flavor (chemical formula $(CH_3CO)_2$)
- Is a natural byproduct of fermentation and is present in alcoholic beverages¹
- Has been previously detected in e-cigarette formulations²

OBJECTIVE

- Develop a sensitive and selective method for quantitative analysis of diacetyl in e-liquids by using gas chromatography-mass spectrometry (GC-MS)
- Analyze commercially available e-liquids (refill products) by using gas chromatography-mass spectrometry (GC-MS) for the presence of diacetyl in order to determine if the method is fit for purpose

METHOD

Parameter	Description
GC Column	Restek StabilWax, 30 m x 0.25 mm x 0.5 μ m
Oven Temperature Program	35 °C hold 8 min ramp 10 °C/min to 100 °C, hold 1 min ramp 100 °C to 250 °C, hold 15 min
Flow Rate	1.5 mL/min
Injector Temperature	240 °C
Selected Ion Monitoring (SIM)	m/z 43 & 86 for diacetyl m/z 44 & 42 for ISTD
Ion Source Temperature	230 °C
Interface Temperature	250 °C
Method RunTime	32 min
Sample Requirement	1 μ L e-vapor formulation (e-liquid) injected with a 10:1 split

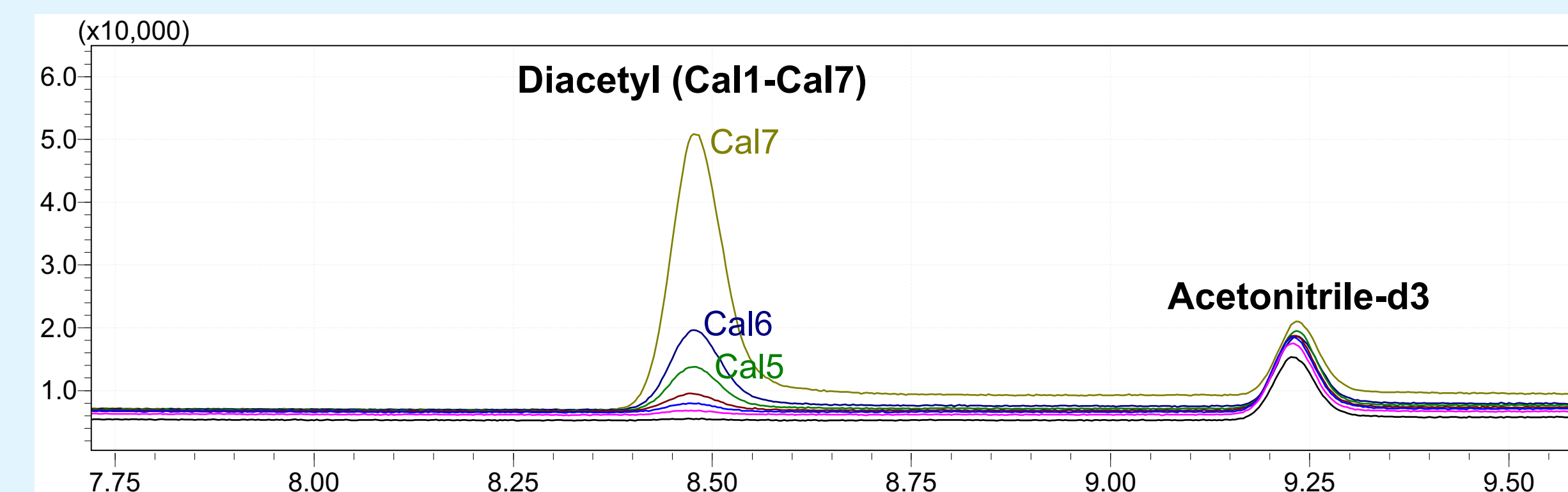
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- Hayasaka Y & Bartowsky EJ. (1999) Analysis of diacetyl in wine using solid-phase microextraction combined with gas chromatography-mass spectrometry. J Agr Food Chem 47:612–617.
- Farsalinos KE et al. (2015) Evaluation of electronic cigarette liquids and aerosol for the presence of selected inhalation toxins. Nicotine Tob Res 17:168-174.

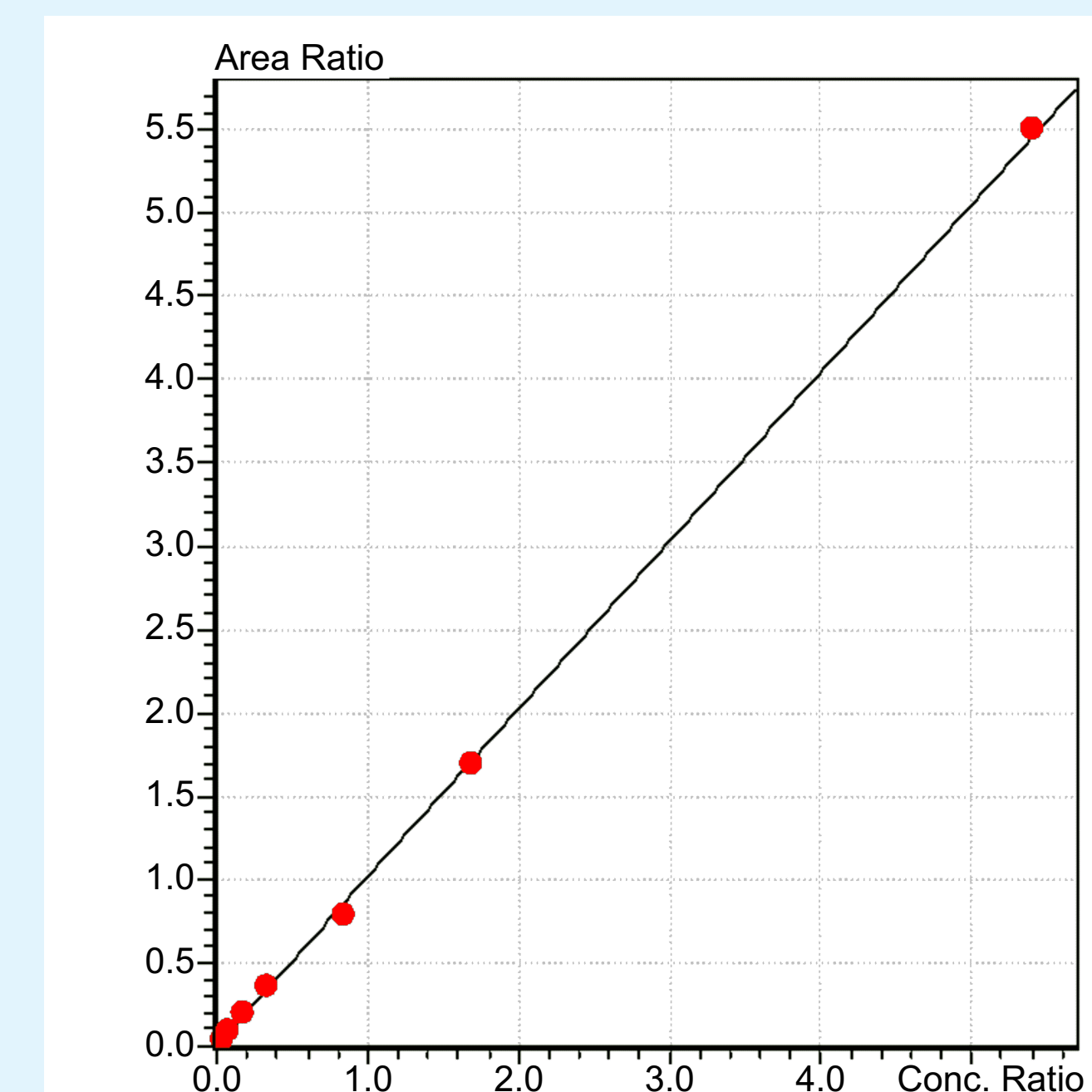
ABSTRACT

While diacetyl is approved for food use, the United States National Institute for Occupational Safety and Health (NIOSH) has suggested it may be associated with respiratory disease when heated and inhaled. NIOSH has defined safety limits for occupational exposure to diacetyl. Farsalinos and coworkers recently investigated 159 sweet-flavored e-vapor refill formulations where they observed that 110 contained measurable amounts of diacetyl with many exceeding NIOSH limits.² The authors used a modified method that was originally developed for the analysis of carbonyls in mainstream cigarette smoke. This method involved derivatization using 2,4-dinitrophenylhydrazine (DNPH) and analysis by high performance liquid chromatography (HPLC) with ultraviolet (UV) detection. The purpose of this work was to develop a sensitive and selective method for the analysis of diacetyl using gas chromatography-mass spectrometry (GC-MS) that does not require derivatization.

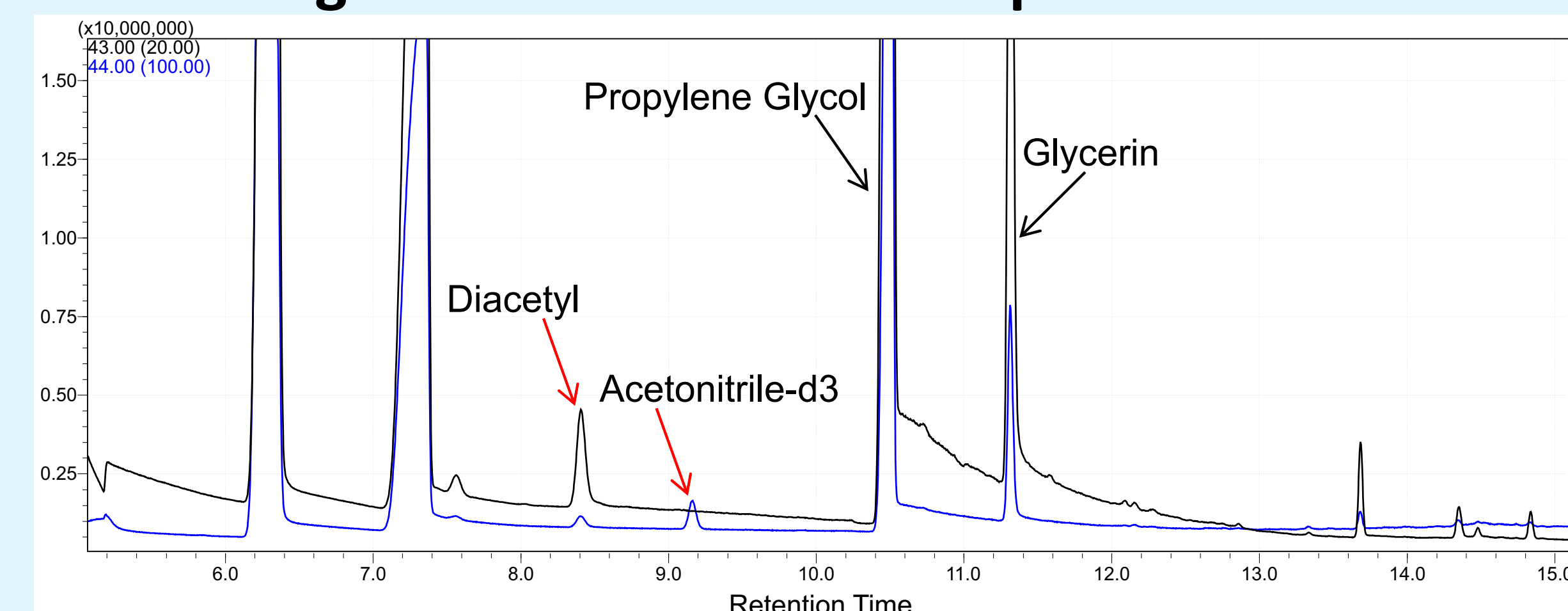
Calibration Standards



Calibration Curve

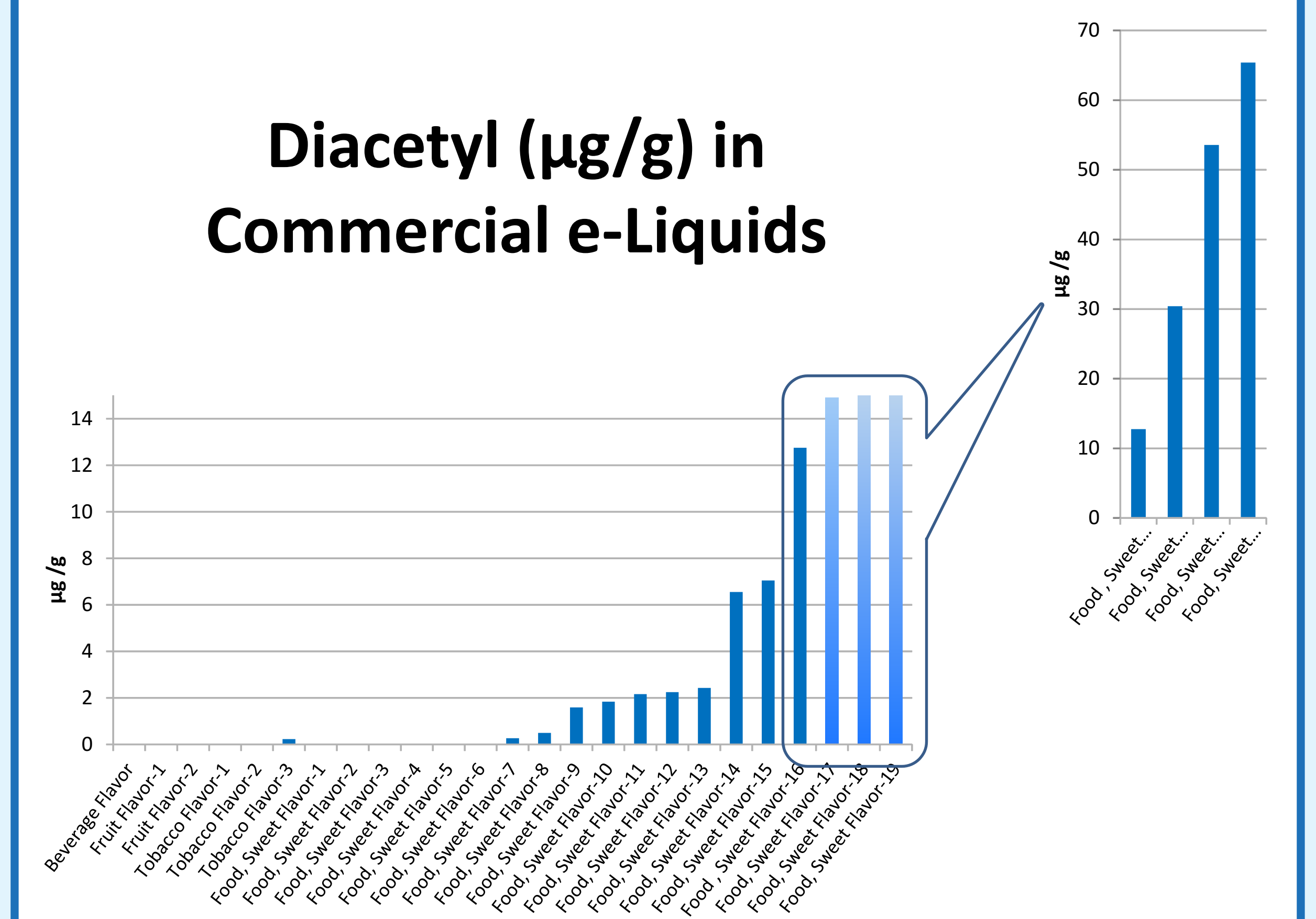


Chromatogram of Commercial E-liquid



RESULTS

Diacetyl (μ g/g) in Commercial e-Liquids



14 of 25 commercial e-liquids (refill products) selected for this study contained measurable levels of diacetyl ($>0.1 \mu$ g/g) and 3 were greater than 30 μ g/g

Validation Summary

Parameter	Result
Calibration Range	0.1 μ g/g – 16.2 μ g/g
Linearity	$R^2 > 0.995$
Accuracy	92.5 % to 109 % (3 levels)
Precision	< 8.8 % RSD
Limit of Detection	0.03 μ g/g

SUMMARY

- A method was developed for the analysis of diacetyl in e-formulations (e-liquids)
 - It does not require derivatization
 - It can be used to quantitate diacetyl in e-vapor products to estimate potential human exposure
- Of the 25 commercial e-liquids analyzed in this study, 14 contained measurable amounts of diacetyl ranging from 0.2 μ g/g to 65 μ g/g

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