APPLICATION NOTE

Infrared Spectroscopy

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Determination of Nicotine Content in E-Liquids using Near-Infrared Spectroscopy

Introduction

Electronic Cigarettes (or e-cigarettes) are battery powered devices designed to imitate the act of smoking by delivering a nicotine-containing aerosol to the user, comprising propylene glycol, vegetable glycerine, flavourings and, of course, nicotine.[1]

The use of E-cigarettes has increased rapidly since their introduction in late 2007, with an increase of 550% in the United States.[2] Furthermore, studies have shown that more than 80% of E-Cigarette users list quitting smoking as a very important reason for using E-Cigarettes.[3]

The PerkinElmer Spectrum Two N™ with Near-Infrared Reflectance Module (NIRM) using Spectrum™ Quant allows for quick, non-destructive determination of the nicotine content of e-liquids.

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Experimental

244 spectra of 21 different varieties of E-Liquid were measured using the PerkinElmer Spectrum Two N with Near-Infrared Reflectance Module (NIRM) (Figure 1) and a 60 mm glass petri dish with a transflectance accessory (Figure 2). This method allows for the E-liquid to be analyzed quickly and non-destructively with minimal sample preparation. The spectra were collected using the experimental parameters shown in Table 1.

Figure 1. PerkinElmer Spectrum Two N with Near-Infrared Reflectance Module (NIRM).

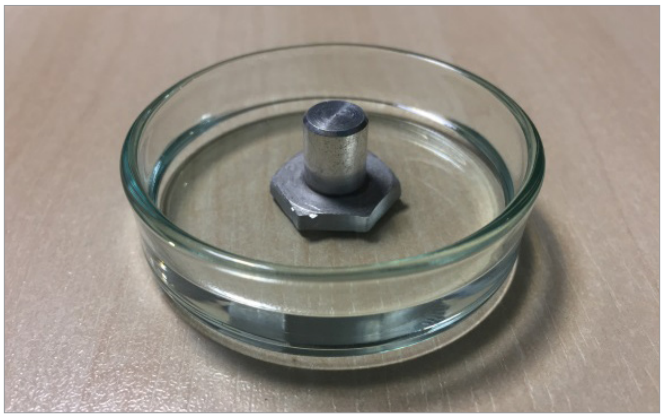

Figure 2. E-Liquids sample with Transflectance Accessory.

Table 1. Experimental parameters for the collection of E-liquid spectra.

Parameter	Value
Resolution	8 cm^{-1}
Number of Scans	32 (30s)
Spectral Range	10000 – 4000 cm^{-1}

Of the 21 varieties measured, the concentration of nicotine ranged from 0 to 2.4 % and the countries of origin were the UK, USA and China. Table 2 shows the various brands of E-liquid investigated, as well as country of origin and nicotine concentration.

The E-liquids measured varied somewhat in color with the majority being colorless but some having a deep color e.g. 'Grape Drank'.

Table 2. Flavor, country of origin, and nicotine content of the E-liquids investigated.

Flavor	Country of Origin	Nicotine Content Label Claim (%) (w/v)
Black Cherry	UK	1.8
Candyfloss	UK	0
Strawberry	China	2.4
Fruit Twist	UK	1.1
Tobacco	China	1.6
Strawberry	China	1.6
Blueberry	UK	1.1
Cherry	China	1.1
Fruit Twist	UK	1.6
Red Cola	China	1.6
Menthol	UK	1.1
Menthol	China	1.8
Cherry	China	1.8
Rhubarb and Custard	UK	1.8
Strawberry Milkshake	UK	1.2
Banana	UK	0.6
Raspberry	UK	1.2
Virginia	UK	0.6
Grape Drank	USA	0.3
Fruit Pastille	UK	0.5
Black Ice	UK	1.8

Of the 244 spectra collected in this study, 238 were used as standards in Spectrum Quant to create a partial least-squares regression model (PLS1). This model used Standard Normal Variate (SNV) and second order derivative (13 point) baseline correction pre-processing and leave-1-out cross validation. The overlaid pre-processed E-Liquid spectra are shown in Figure 3. Figure 4 and Table 3 show the estimated vs. specified % nicotine content and regression summary respectively.

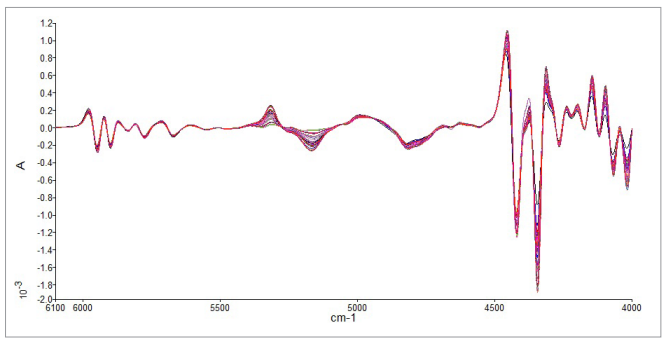

Figure 3. Second order derivative (13 point) and SNV pre-processed E-Liquid spectra.

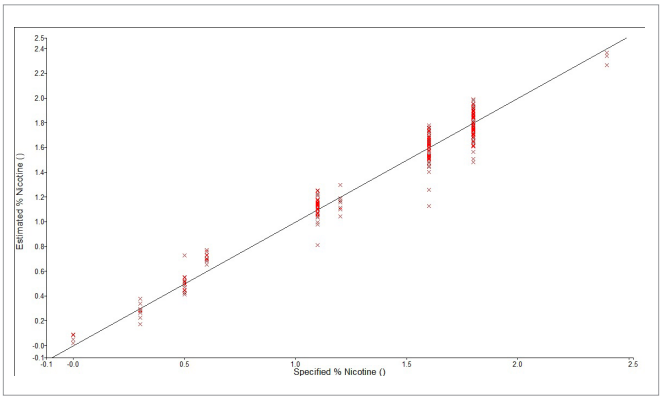

Figure 4. PLS model of E-Liquid samples.

Table 3. PLS model regression summary.

Number of PCs	% Variance (R^2)	Standard Error of Estimate (SEE)	Standard Error of Prediction (SEP)	Cross Validation SEP
10	96.25	0.1028	0.1058	0.1181

Six independent validation samples were prepared consisting of a range of nicotine concentrations in the most common ratio of propylene glycol to vegetable glycerine found in E-Liquids (70:30). The nicotine content of these validation samples was predicted in Spectrum Quant, with the results shown in Table 4.

Table 4. Predicted nicotine content of validation standards.

Actual Nicotine Content (% w/v)	Predicted Nicotine Content (% w/v)	Error in Prediction (% w/v)
0	0.03	0.03
0.15	0.14	0.01
1.20	1.15	0.05
1.50	1.46	0.04
1.80	1.87	0.07
2.10	2.11	0.01

Table 4 demonstrates that Spectrum Quant was able to accurately predict the nicotine content of standard solutions spanning the legal range of concentrations found in E-Liquids, with an average error of prediction of just 0.036 %.

This Quant model may be incorporated into a Spectrum Touch™ method to allow for simplified workflow and quantification of nicotine in E-liquids. Figure 5 shows the result screen which clearly displays the predicted nicotine content (in this case 2.11%) with the option to select 'Quant details' in order to view a more in depth analysis of the prediction, such as residual distance and prediction error.

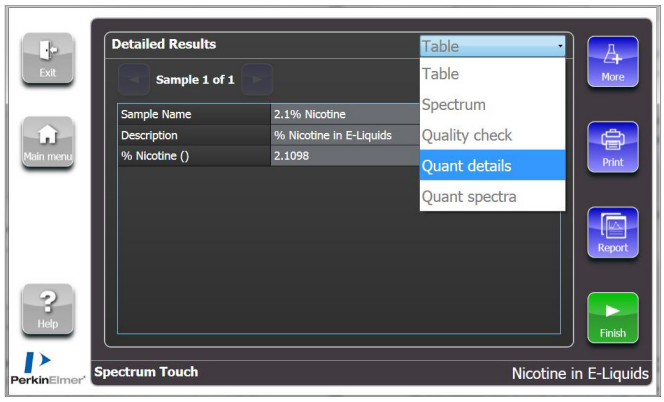

Figure 5. Implementation of the nicotine Quant method into a Spectrum Touch method.

Alternatively, the 'Scanalyze' function in Spectrum 10 allows samples to be scanned and their nicotine content predicted in one click.

Conclusion

The PerkinElmer Spectrum Two N FT-NIR spectrometer and Near-Infrared Reflectance Module (NIRM) provides a quick, non-destructive and accurate method for the quantification of nicotine in E-Liquids when used in conjunction with the chemometric capabilities of PerkinElmer Spectrum Quant software. Additionally, Spectrum Quant methods may be implemented into Spectrum Touch software to achieve a simple workflow based approach to the quantification of nicotine in e-liquids.

References

1. R. Grana, N. Benowitz, S. A. Glantz, *Background Paper on E-cigarettes (Electronic Nicotine Delivery Systems),* Centre for Tobacco Control Research and Education, University of California, San Francisco, 2013
2. J. W. Ayers, K. M. Ribisl, J. S. Brownstein, *Tracking the Rise in Popularity of Electronic Nicotine Delivery Systems (Electronic Cigarettes) Using Search Query Surveillance,* AMPJ, 2011, 40(4), 448 – 453
3. K. D. Volesky, A. Maki, C. Scherf, L. M. Watson, E. Cassol, P. J. Villeneuve, *Characteristics of e-cigarette users and their perceptions of the benefits, harms and risks of e-cigarette use: survey results from a convenience sample in Ottawa, Canada,* Health Promotion and Chronic Disease Prevention in Canada, 2016, 36(7), 130-138

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