New Vapes: Solutions for VOC Analysis

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Introduction

New analytical challenges are constantly being presented by the vaping industry and through collaboration these can be effectively and quickly solved.

Recently a CRO, a customer of Cerulean, experienced difficulties with their vaping machine, the puff volumes were wildly wrong and sometimes they did not get puffs at all!

Contacting Cerulean was a first step to providing a working solution to their problems.

The Problem

The laboratory in question were new to testing vaping products and needed to examine volatile organic species (VOC) that are transferred into the aerosol of a vape. VOC's are expected partly to be essential oils and terpenes that impart a characteristic flavour to the vapour as well as being characteristic of the organic roots of the vaping liquid.

The laboratory wanted to use a thermal desorption mass spectrometer, an ideal tool for characterising volatile compounds. This works by heating a sample to release volatile compounds that are "sniffed" by the spectrometer and the mass and chemistry of the volatile determined. Selectivity is improved by "ramping" the heating temperature and releasing volatiles one at a time for analysis.

E-liquids are often "sniffed" from the head space above the liquid, but vaping may change the profile of the volatiles so a different method is needed. In this case a sorbent tube, a tube packed with high surface area volatile capture medium, was placed between the e-cigarette and the puff engine of the CETI8. After puffing, the sorption tube could be placed directly in the TDMS for further analysis.

The problem the laboratory encountered was the flow regulated e-cigarette would not always trigger and the tubing between device and the puff engine could collapse causing the engine to stall.

The experiment could not be conducted.

The Analysis

The first step was to document exactly what was happening and what equipment was being used.

The sorption tube used was a Markes type C3-CAXXX5266 (Inert Universal). This was connected to the puffing port with thin walled silicon tube.

Step 1: Measure the pressure drop of the Markes sorption tube.

This was measured at approximately 1700mmWG (measured at a flow rate of 17.5ml/sec to ISO2965). Normally the pressure drop of an e-cigarette would be 100-200mmWG. However the CETI8 can tolerate higher pressure drops before the engine stalls.

Figure 2: Markes sorption tube used in experiments

Figure 1: Cerulean CET8 8 port vaping machine fitted with End Point Detection system

Step 2: Evaluate the role of the collapsing tube.

The tube used was thin walled, soft and flexible, used for joining different parts of the experimental apparatus. A simple vacuum test showed this would easily collapse but thicker walled (3mm) silicon tube or Tygon tubing could withstand vacuums in excess of 90kPa (~9000mmWg). These tubes were equally suitable for joining the apparatus and did not collapse in use.

Step 3: Investigate the failure to trigger a puff in flow activated devices

A standard ISO20768 square puff has a flow rate of 18.33ml/sec and the Markes sorbent tube requires a flow rate of 0.5 to 1 l/min. Flow is adequate.

A comparison of the puff profile was made between a CETI8 with Markes tube fitted and one with a more representative 100mmWg pressure drop in line.

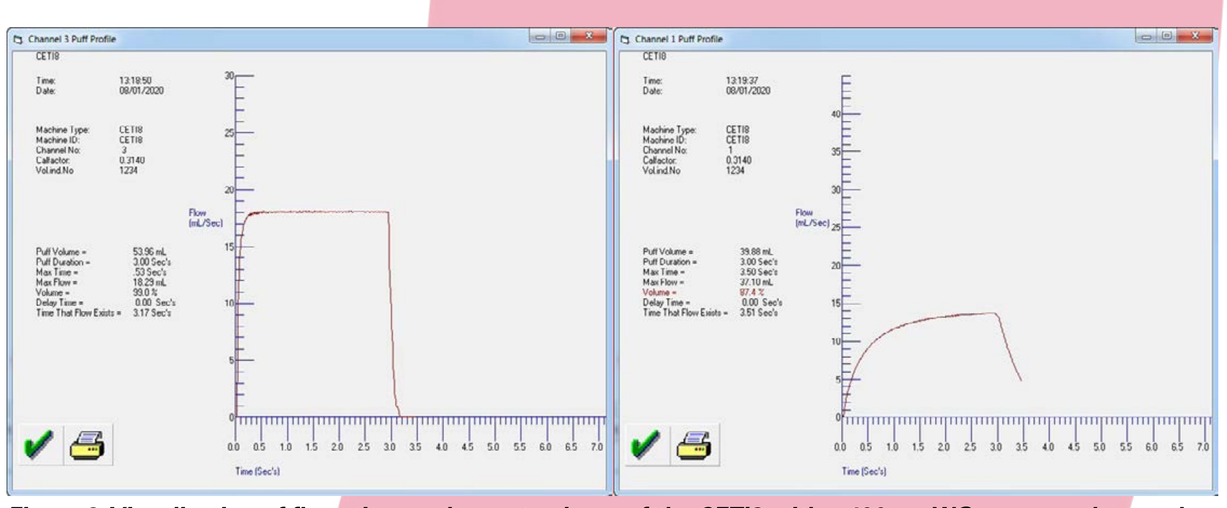
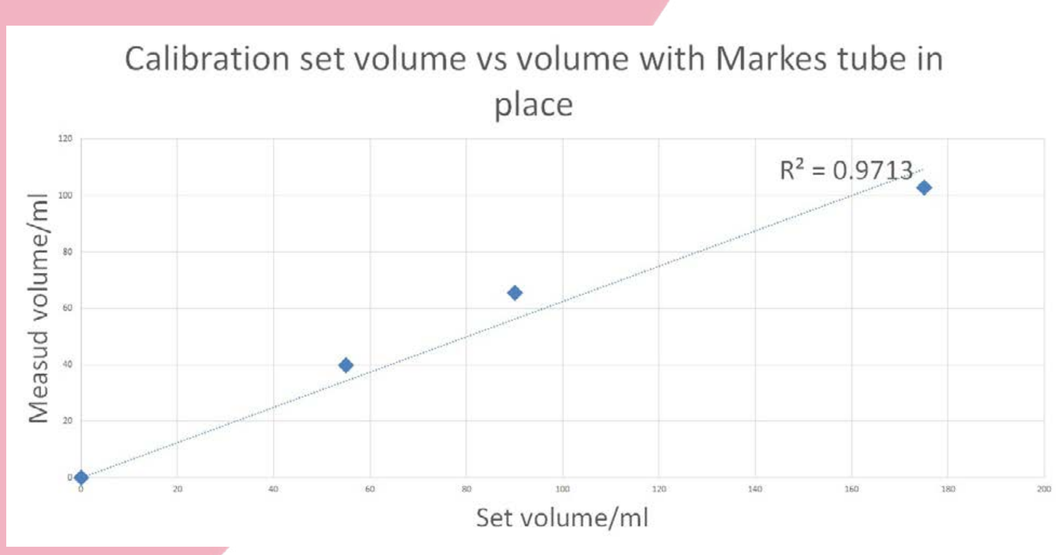


Figure 3: Visualisation of flow, time and swept volume of the CETI8 with a 100mmWG pressure drop and Markes TD tube in series. Nominal volume 55ml, puff duration 3 seconds. Measurements made with Cerulean's VFA450RH.

The above plots show the fundamental problem of this high pressure drop device, flow rates are truncated and this can prevent the e-cigarette vaping correctly. The high pressure drop "smears" out the profile puffed. There is a significant lag in the puff drawn and the 55ml puff is truncated to a sub 40ml puff. The peak flow rate drops from 18ml/sec to 13ml/sec. This is caused by a partial vacuum being created in the tubing /Markes tube and the puff valve shutting off before all the air for the puff is drawn.

The Solution

Firstly tubing was replaced with a more robust section to prevent tube collapse. Next the puff volume was modified to allow for the partial vacuum being created in the Markes tube. A simple calibration curve was created of set volume vs true volume (and peak flow rate). This allowed a volume of 55ml to be drawn through the Markes tube by setting the CETI8 to an approximate 85ml volume.



The system then worked correctly.

Conclusion

A simple pragmatic solution was found for the capture of terpenes and VOC's by TD tubes considering the physical and operational limitations of the CETI8 and ancillary equipment. The flexibility that the CETI8 offered was used to prepare samples suitable for later analysis equipment. Consultation between equipment manufacturer and user yielded a solution that proved fit for purpose.

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