

CONFIDENTIAL

UK SMOKE CONSTITUENTS STUDY

ANNEX

Part 7: Validation data Determination of SVC yields in mainstream cigarette smoke

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Validation data for semi-volatile compounds

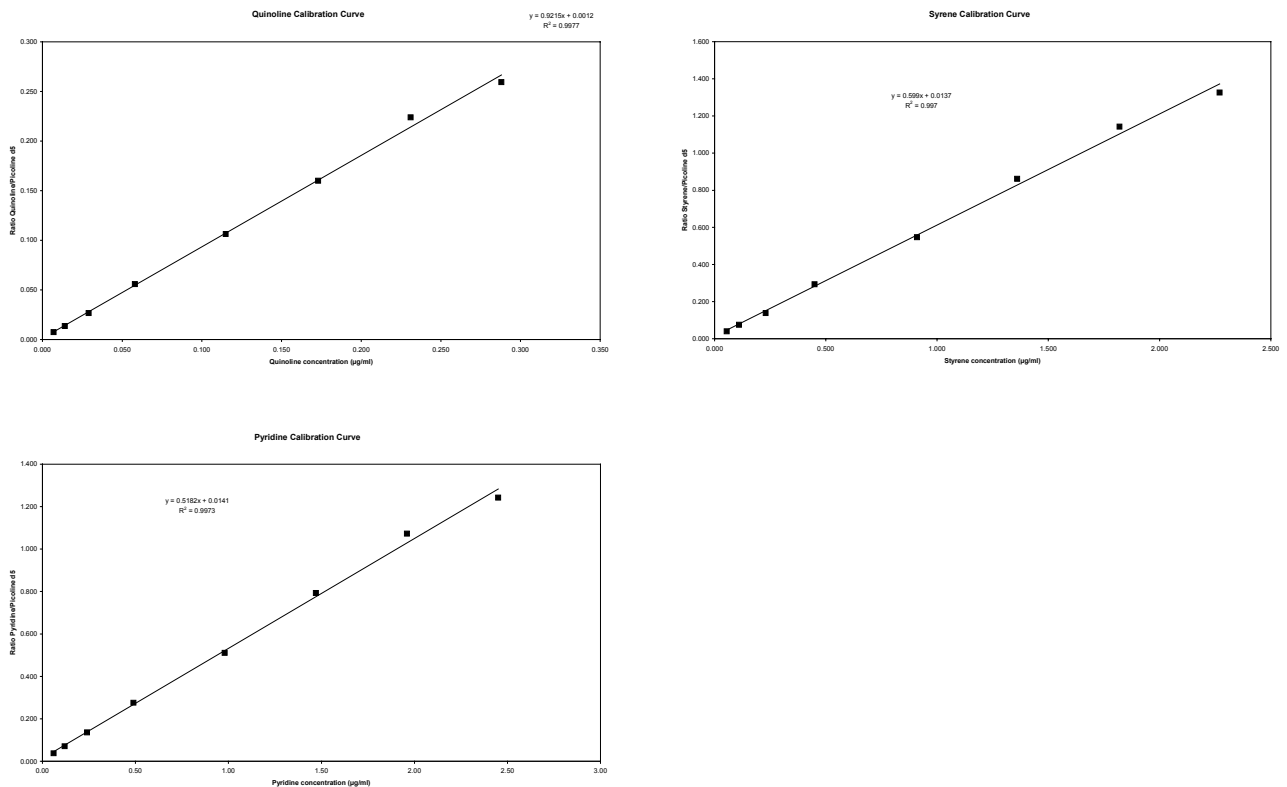
The following is a summary of the validation data obtained when validating the method for pyridine, quinoline and styrene.

1. Overview

For each sample, five conditioned cigarettes are smoked on a Borgwaldt rotary smoking machine. The mainstream smoke is passed through a Cambridge filter pad and the vapour phase passed through a solid sorbent tube (XAD-4). After smoking, the contents of the sorbent tube are added to the pad and methanol added to extract the semi volatile compounds. The solution is analysed by gas chromatography/mass spectrometry.

2. Calibration

Eight standards are used to calibrate the instrument. They are run at the beginning of the run. A QC standard and blank is also run at the end to check that the calibration has not drifted. Typical calibration curves are shown below.

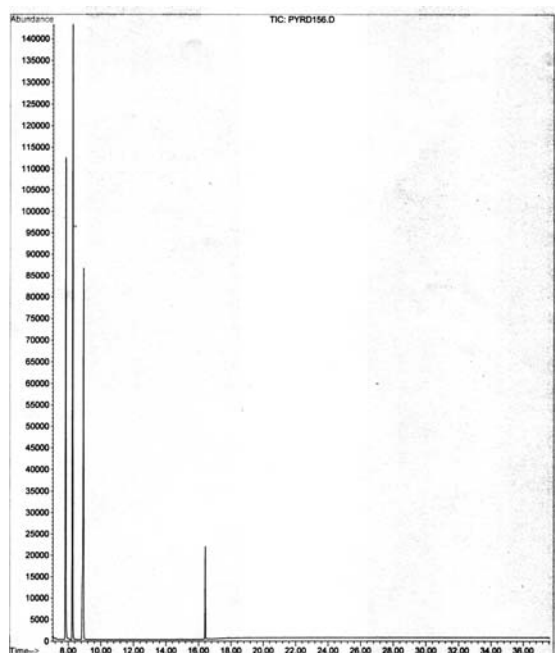


The intercept and slope were found to be reasonably consistent over time, quinoline gave the most variation (analyte present at lower concentration than the other two).

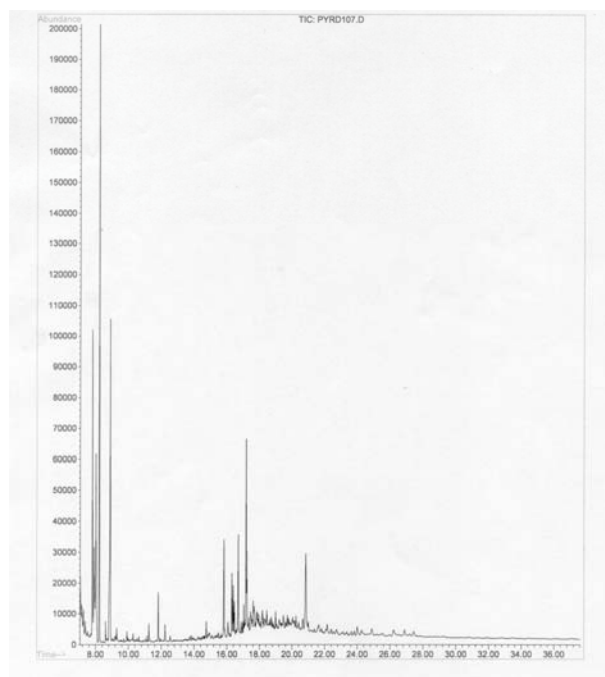
3. Chromatograms

The three chromatograms are (a) standard $\equiv 9 \mu\text{g cig}^{-1}$ for pyridine and styrene; $0.9 \mu\text{g cig}^{-1}$ for quinoline, (b) 1R4F and (c) blank. The peak order in the standard is pyridine, internal standard, styrene and quinoline. 1R4F is the extract solution which includes the deuterated recovery standards and the internal standard. The blank also contains the internal and deuterated recovery standards.

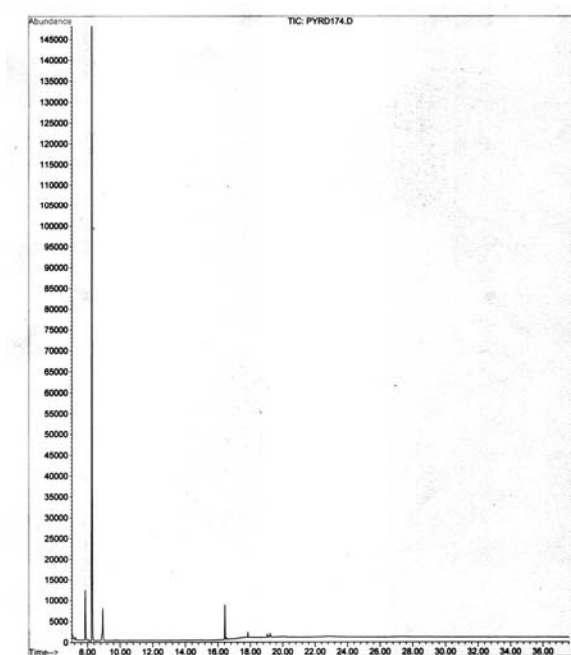
(a)



(b)



(c)



4. Limit of detection/quantitation/reporting

The intercept on the calibration curve changes between runs and effects the limit of detection. Therefore the lowest standard concentration is used to determine a limit of quantitation and reporting limit. With 5 cigarettes and 15 mL* volume, the reporting limit is set at 0.2 µg cig⁻¹ (0.02 µg cig⁻¹ quinoline).

5. Calibration range

Eight standards are used to try and cover the expected wide range of analyte concentrations. Indications are that at least one brand may exceed the top standard. Where appropriate, an aliquot of the sample solution was diluted (1:1) with methanol to keep the sample solution concentration within range.

6. Precision, repeatability and accuracy

1R4F and 1R5F were smoked on the same day to show repeatability within a run. Data from the study was used to show repeatability between runs for these brands. Brand A and B were brands which had recently been used in an inter comparison exercise for a range of analytes including semi volatile compounds. Brands C – G were smoked to demonstrate the robustness of the method when handling a range of brands.

Brand	No. of assays	Mean Pyridine yield µg cig ⁻¹	Mean Quinoline yield µg cig ⁻¹	Mean Styrene yield µg cig ⁻¹	Comments (anticipated values)
1R4F	5	4.63 ± 0.24	0.22 ± 0.01	6.52 ± 0.22	Same day
1R4F	5	4.14 ± 0.24	0.22 ± 0.04	5.63 ± 0.26	Results from study (different days)
1R5F	5	0.73 ± 0.11	0.057 ± 0.005	1.45 ± 0.15	Same day
1R5F	5	1.70 ± 0.20	0.08 ± 0.02	2.93 ± 0.23	Results from study (different days)
Brand A	5	8.09 ± 0.62	0.25 ± 0.01	9.79 ± 0.52	Same day (5.5 – 12.1; 0.35 – 1.42; 2.4 – 23.8) [†]
Brand B	5	2.50 ± 0.35	0.14 ± 0.01	3.44 ± 0.34	Same day (2.2 – 4.4; 0.19 – 0.38; 1.2 – 7.8) [†]
Brand C	2	10.7 ± 0.53	0.38 ± 0.01	6.88 ± 0.02	Plain cigarette (UK blend – high tar)
Brand D	2	18.2 ± 4.91	0.43 ± 0.01	9.56 ± 2.51	Filter cigarette (dark air cured tobacco)
Brand E	2	1.83 ± 0.06	0.12 ± 0.02	2.68 ± 0.27	Filter cigarette (menthol UK blend medium/low tar)
Brand F	2	9.44 ± 0.08	0.30 ± 0.004	9.79 ± 0.34	Filter cigarette (USA blend – high tar)
Brand G	2	0.71 ± 0.13	0.06 ± 0.002	1.22 ± 0.20	Filter cigarette (UK blend – low tar)

* Low NFDPM yield samples (<2.5 mg cig⁻¹) are extracted with 15 mL instead of the normal 30 mL .

[†] Results from inter comparison exercise used different smoking regimes and analytical methods.