

CONFIDENTIAL

# **UK SMOKE CONSTITUENTS STUDY**

## **Part 9: Determination of Volatile Organic Compounds Yields in Cigarette Smoke**

### **Annex B - Validation**

Commissioned by:  
Tobacco Manufacturers Association  
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## Validation data for Volatile Organic Compounds

### 1. Overview

The determination of Volatile Organic Compounds (VOCs) yields in cigarette smoke for the 25 brands was carried out by Arista Laboratories, USA.

Arista Laboratories USA methods have previously been validated and are in regular use. Therefore a different approach was adopted, compared to previous reports, to show that the method would be suitable for performing the study. Less emphasis has been put on showing the method is suitable but more data was obtained through the study to demonstrate the reliability of the method.

### 2. Repeatability

The following data was achieved for 2R4F throughout the survey

	1,3-butadiene (µg/cigt)	Isoprene (µg/cigt)	Acrylonitrile (µg/cigt)	Benzene (µg/cigt)	Toluene (µg/cigt)	Styrene (µg/cigt)
2R4F	32.8	404	9.92	49.8	84.2	5.32
2R4F	33.0	392	9.56	48.4	84.5	5.76
2R4F	34.9	400	9.60	49.8	85.5	5.72
2R4F	33.9	385	9.00	46.8	78.6	5.20
2R4F	35.4	405	9.44	48.6	81.2	5.36
2R4F	35.4	420	9.28	50.7	89.2	5.56
2R4F	30.9	403	9.28	50.1	88.9	6.20
2R4F	36.8	412	9.88	51.3	87.1	6.20
2R4F	32.4	401	8.12	45.4	78.5	5.64
2R4F	38.6	408	9.32	46.6	79.6	6.00
2R4F	35.8	429	8.36	49.2	85.3	6.24
2R4F	36.6	389	8.12	47.0	78.6	5.04
2R4F	36.7	408	8.28	47.9	78.7	5.24
2R4F	34.2	350	8.00	43.6	75.3	5.12
2R4F	33.9	368	7.92	46.1	76.9	4.84
2R4F	30.5	362	8.92	46.2	82.4	5.24
2R4F	34.6	406	8.28	47.6	81.4	5.80
<b>Average</b>	<b>34.5</b>	<b>397</b>	<b>8.90</b>	<b>47.9</b>	<b>82.1</b>	<b>5.56</b>
<b>sd</b>	<b>2.15</b>	<b>20.4</b>	<b>0.70</b>	<b>2.08</b>	<b>4.21</b>	<b>0.43</b>
<b>%RSD</b>	<b>6.2</b>	<b>5.2</b>	<b>7.8</b>	<b>4.3</b>	<b>5.1</b>	<b>7.8</b>

“Historical data” achieved for 2R4F by Arista (USA) collected over approximately 10 months. Upper and lower control limits are based on the average  $\pm 3 \times$  sample standard deviation and if any monitor falls outside this range, then the result and the entire run would be rejected

<b>Volatile Organics (µg/cigt) n=144</b>	Mean µg/cigt	Standard Deviation µg/cigt
1,3-butadiene	30.8	4.3
Isoprene	374	32
Acrylonitrile	9.3	0.9
Benzene	48.0	3.0
Toluene	82.8	6.2
Styrene	5.8	0.8

### 3. Reproducibility

Data for 1R4F quoted below is as achieved in the survey and compared with recently published data for 1R4F<sup>1</sup>.

Data for Brand A and B were brands which had recently been used in an inter comparison exercise for a range of analytes including vocs.

Brand	No. of assays	1,3 Butadiene yield $\mu\text{g cig}^{-1}$	Isoprene yield $\mu\text{g cig}^{-1}$	Acrylonitrile yield $\mu\text{g cig}^{-1}$	Benzene yield $\mu\text{g cig}^{-1}$	Toluene yield $\mu\text{g cig}^{-1}$	Styrene yield $\mu\text{g cig}^{-1}$	Comments (anticipated values)
1R4F	5	30.3 ± 0.51	333 ± 14.8	8.07 ± 0.67	40.6 ± 2.52	72.3 ± 5.23	5.3 ± 0.46	1,3 butadiene 38.8 ± 3.2 Isoprene 371 ± 24 Acrylonitrile 9.08 ± 1.08 Benzene 39.5 ± 2.6 Toluene 63.8 ± 6.0 Styrene 6.64 ± 6.0 (all $\mu\text{g cig}^{-1}$ ) <sup>1</sup>
1R5F	5	12.6 ± 1.76	136 ± 12.3	2.17 ± 0.42	15.7 ± 1.47	23.0 ± 3.16	1.5 ± 0.28	Results from study
2R4F	17	34.5 ± 2.15	397 ± 20.4	8.90 ± 0.70	47.9 ± 2.08	82.1 ± 4.21	5.56 ± 0.43	Results from study
Brand A	5	42.2 ± 0.76	369 ± 18.6	11.7 ± 1.15	57.5 ± 2.48	97.7 ± 5.69	11.8 ± 0.81	1,3 butadiene 21.9 – 76.5 Isoprene 337 – 489 Acrylonitrile 9.0 – 16.2 Benzene 44.1 – 60.8 Toluene 65.3 – 83.2 Styrene 2.4 – 23.8 (all $\mu\text{g cig}^{-1}$ ) <sup>2</sup>
Brand B	5	25.6 ± 2.41	260 ± 27.5	5.93 ± 0.84	33.1 ± 4.52	54.8 ± 7.37	4.89 ± 1.15	1,3 butadiene 11.3 – 34.6 Isoprene 198 – 361 Acrylonitrile 3.5 – 8.8 Benzene 20.7 – 38.2 Toluene 30.1 – 43.3 Styrene 1.2 – 7.8 (all $\mu\text{g cig}^{-1}$ ) <sup>2</sup>

<sup>1</sup> Stability of yields of Canadian mandated analytes from the Kentucky Reference Cigarette 1R4F: A time series analysis, Paper No ST26, 2002 CORESTA Congress.

<sup>2</sup> Results from inter comparison exercise used different smoking regimes and analytical methods.

#### 4. Limit of Detection and Quantitation

The following limits of detection and quantitation were used for the study

	1,3 butadiene	Isoprene	Acrylonitrile	Benzene	Toluene	Styrene
	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$
Limit of Detection	0.7	0.9	0.4	0.6	0.4	0.2
Limit of Quantitation	2.2	3.1	1.3	1.9	1.2	0.8