

CONFIDENTIAL

# **UK SMOKE CONSTITUENTS STUDY**

## **Part 10: Determination of Mono and Dihydroxy Phenols Yields in Cigarette Smoke**

### **Annex B - Validation Data**

Commissioned by:  
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## Validation data for Phenols

### 1. Overview

The determination of Phenols 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

Client Code	Hydroquinone (µg/cigt)	Resorcinol (µg/cigt)	Catechol (µg/cigt)	Phenol (µg/cigt)	m+p-Cresol (µg/cigt)	o-Cresol (µg/cigt)
2R4F	31.5	0.71	41.7	6.96	5.70	2.08
2R4F	29.3	0.69	38.9	6.92	5.90	2.18
2R4F	31.0	0.73	39.7	6.55	5.57	2.08
2R4F	31.8	0.82	41.9	6.45	5.67	2.12
2R4F	31.3	0.73	40.7	4.81	4.63	1.65
2R4F	32.1	0.79	41.1	4.60	4.50	1.61
2R4F	30.8	0.70	37.7	4.00	3.95	1.41
2R4F	31.5	0.77	40.7	5.15	4.90	1.78
2R4F	31.0	0.79	39.3	4.64	4.42	1.59
2R4F	32.0	0.80	41.1	5.22	4.94	1.79
2R4F	29.4	0.72	38.9	5.02	4.62	1.67
2R4F	30.9	0.73	38.2	4.39	4.22	1.51
2R4F	30.3	0.70	38.6	4.31	4.17	1.50
2R4F	30.5	0.71	36.9	3.83	3.74	1.32
2R4F	29.0	0.68	37.6	4.12	4.03	1.44
2R4F	31.4	0.74	39.8	4.31	4.24	1.52
2R4F	33.7	0.80	43.4	5.66	5.27	1.92
2R4F	31.5	0.71	40.0	4.43	4.42	1.59
2R4F	31.4	0.73	39.6	4.07	4.12	1.45
<b>Average</b>	<b>31.1</b>	<b>0.74</b>	<b>39.8</b>	<b>5.02</b>	<b>4.69</b>	<b>1.69</b>
<b>SD</b>	<b>1.10</b>	<b>0.04</b>	<b>1.67</b>	<b>1.01</b>	<b>0.66</b>	<b>0.26</b>
<b>%RSD</b>	<b>3.5</b>	<b>5.7</b>	<b>4.2</b>	<b>20.2</b>	<b>14.0</b>	<b>15.6</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>Phenolics (µg/cigt) n=148</b>	Mean µg/cigt	Standard Deviation µg/cigt
Hydroquinone	31.1	3.1
Resorcinol	0.7	0.1
Catechol	39.5	3.6
Phenol	6.2	1.3
m/p-cresol	5.4	0.9
o-cresol	2.0	0.3

### 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 phenols.

Brand	No. of assays	Hydroquinone yield $\mu\text{g cig}^{-1}$	Resorcinol yield $\mu\text{g cig}^{-1}$	Catechol yield $\mu\text{g cig}^{-1}$	Phenol yield $\mu\text{g cig}^{-1}$	m+p-cresol yield $\mu\text{g cig}^{-1}$	o-cresol yield $\mu\text{g cig}^{-1}$	Comments (anticipated values)
1R4F	5	$34.2 \pm 2.32$	$0.64 \pm 0.06$	$38.0 \pm 2.68$	$7.54 \pm 0.88$	$5.88 \pm 0.60$	$2.31 \pm 0.25$	hydroquinone $39.6 \pm 3.0$ resorcinol $0.54 \pm 0.06$ catechol $38.5 \pm 2.5$ phenol $8.89 \pm 1.04$ m+p-cresol $7.52 \pm 0.73$ o-cresol $2.93 \pm 0.29$ (all $\mu\text{g cig}^{-1}$ ) <sup>1</sup>
1R5F	5	$6.66 \pm 0.71$	<0.33	$8.42 \pm 0.50$	<0.72	$0.64 \pm 0.02$	<0.41	Results from study
2R4F	19	$31.1 \pm 1.10$	$0.74 \pm 0.04$	$39.8 \pm 1.67$	$5.02 \pm 1.01$	$4.69 \pm 0.66$	$1.69 \pm 0.26$	Results from study
Brand A	5	$62.7 \pm 2.99$	$1.46 \pm 0.06$	$67.6 \pm 3.50$	$15.3 \pm 0.19$	$9.08 \pm 0.46$	$3.69 \pm 0.21$	hydroquinone 68.4 – 81.7 resorcinol 0.7 – 8.1 catechol 56.3 – 83.1 phenol 16.2 – 19.3 m+p-cresol 6.4 – 12.8 o-cresol 2.8 – 7.4 (all $\mu\text{g cig}^{-1}$ ) <sup>2</sup>
Brand B	5	$32.6 \pm 4.85$	$0.71 \pm 0.12$	$38.1 \pm 5.48$	$8.75 \pm 1.54$	$5.57 \pm 0.81$	$2.27 \pm 0.34$	hydroquinone 33.1 – 61.2 resorcinol 0.6 – 3.2 catechol 29.1 – 43.5 phenol 8.6 – 9.9 m+p-cresol <2 – 3.7 o-cresol 3.8 – 6.9 (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. Phenol set of results for Brand A & B excludes one set of suspect results

#### 4. Limit of Detection and Quantitation

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

	hydroquinone	resorcinol	catechol	phenol	m+p-cresol	o-cresol
	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$	$\mu\text{g cig}^{-1}$
Limit of Detection	0.12	0.11	0.27	0.33	0.14	0.17
Limit of Quantitation	0.36	0.33	0.81	0.72	0.32	0.41