

CONFIDENTIAL

UK SMOKE CONSTITUENTS STUDY

Part 11: Determination of Metal Yields in Cigarette Smoke

Annex B - Validation Data

Commissioned by:
Tobacco Manufacturers Association
55 Tufton Street
London SW1P 3QL

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Validation data for Metals

1. Overview

The determination of metals' 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 (Mercury) was achieved for 2R4F throughout the survey

Client Code	Mercury (ng/cigt)
2R4F	2.12
2R4F	2.39
2R4F	2.38
2R4F	2.31
2R4F	2.37
2R4F	2.47
2R4F	2.57
2R4F	2.52
2R4F	2.51
2R4F	2.46
2R4F	2.25
2R4F	2.49
2R4F	2.40
2R4F	2.45
2R4F	2.40
2R4F	2.31
2R4F	2.43
2R4F	2.52
2R4F	2.42
2R4F	2.45
2R4F	2.43
2R4F	2.54
2R4F	2.49
2R4F	2.44
2R4F	2.38
Average	2.42
SD	0.10
%RSD	4.1

“Historical data” achieved for 2R4F by Arista (USA)

Metals II (ng/cigt) n=164	Mean	Standard Deviation
Mercury	2.4	0.2

The following data (metals excluding mercury) was achieved for 2R4F throughout the survey

Client Code	Chromium (ng/cigt)	Selenium (ng/cigt)	Arsenic (ng/cigt)	Nickel (ng/cigt)	Lead (ng/cigt)	Cadmium (ng/cigt)
2R4F	<LOD	<LOQ	3.23	<LOD	11.6	39.9
2R4F	<LOD	<LOQ	3.45	<LOD	12.6	40.2
2R4F	<LOD	<LOQ	3.25	<LOD	12.0	41.4
2R4F	<LOD	<LOQ	3.14	<LOD	11.5	40.6
2R4F	<LOD	<LOQ	3.40	<LOD	11.7	41.7
2R4F	<LOD	<LOQ	3.36	<LOD	11.7	40.5
2R4F	<LOD	<LOQ	3.39	<LOD	11.7	41.3
2R4F	<LOD	<LOQ	3.71	<LOD	13.0	47.0
2R4F	<LOD	<LOD	3.56	<LOD	13.3	48.6
2R4F	<LOD	<LOD	3.23	<LOD	12.1	40.1
2R4F	<LOD	<LOD	3.31	<LOD	13.4	47.4
2R4F	<LOD	<LOD	3.69	<LOD	13.7	50.0
2R4F	<LOD	<LOD	3.46	<LOD	14.2	52.0
2R4F	<LOD	<LOD	2.97	<LOD	12.1	49.1
2R4F	<LOD	<LOQ	3.56	<LOD	15.1	54.0
2R4F	<LOD	<LOD	3.37	<LOD	14.8	51.6
2R4F	<LOD	1.4	4.03	<LOD	14.5	51.9
2R4F	<LOD	1.5	4.33	<LOD	15.8	62.8
2R4F	<LOD	1.6	4.17	<LOD	15.8	62.2
2R4F	<LOD	1.3	4.04	<LOD	15.8	55.8
2R4F	<LOD	1.2	3.75	<LOD	14.4	53.1
2R4F	<LOD	1.2	3.67	<LOD	14.5	52.6
2R4F	<LOD	1.1	3.88	<LOD	14.7	48.2
2R4F	<LOD	1.4	3.91	<LOD	15.3	58.7
2R4F	<LOD	1.4	4.11	<LOD	15.8	57.1
2R4F	<LOD	1.7	4.05	<LOD	15.7	63.1
Average	<LOD	<LOQ	3.62	<LOD	13.7	49.7
SD	na	na	0.36	na	1.6	7.5
%RSD	na	na	10.0	na	11.5	15.1

Note: Values for LODs and LOQs are given on Page 5

“Historical data” achieved for 2R4F by Arista (USA) collected over approximately 10 months. Where appropriate, 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

Metals I (ng/cigt) n=164	Mean	Standard Deviation
Chromium	<LOD	na
Selenium	1.2**	0.2
Arsenic	3.6	0.4
Nickel	<LOD	na
Lead	13.6	1.4
Cadmium	53.9	6.8
(**Note: this value is between the LOD and the Method LOQ)		

3. Reproducibility

Data for 1R4F quoted below is as achieved in the survey and compared with recently published data for 1R4F¹.

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

Brand	No. of assays	Arsenic yield ng cig ⁻¹	Cadmium yield ng cig ⁻¹	Chromium yield ng cig ⁻¹	Lead yield ng cig ⁻¹	Mercury yield ng cig ⁻¹	Nickel yield ng cig ⁻¹	Selenium yield ng cig ⁻¹	Comments (anticipated values)
1R4F	5	6.5 ± 0.30	62.8 ± 10.7	nd (not detected)	41.4 ± 3.22	4.3 ± 0.32	nd (not detected)	<2.3	Arsenic 5.22 ± 1.18 Cadmium 62.4 ± 5.7 Chromium 5.15 ± 1.67 Lead 34.8 ± 3.9 Mercury 5.44 ± 0.54 Nickel 4.94 ± 2.42 Selenium 0.96 ± 0.74 (all ng cig ⁻¹) ¹
1R5F	5	nd	9.6 ± 0.77	nd	4.4 ± 0.43	1.4 ± 0.09	nd	nd	Results from study
2R4F	26	3.62 ± 0.36	49.7 ± 7.5	nd	13.7 ± 1.6	2.4 ± 0.10	nd	<2.3	Results from study
Brand A	5	<2.7	21.9 ± 4.19	nd	19.3 ± 2.68	2.2 ± 0.13	nd	<2.3	Arsenic 2.2 – 1.1 Cadmium 40.2 – 23.3 Chromium 12.4 - <2 Lead 29.2 – 12.0 Mercury 4.1 – 0.4 Nickel 4.6 - <2 Selenium 1.1 - <1.3 (all ng cig ⁻¹) ²
Brand B	5	nd	8.8 ± 1.70	8.3 ± 0.80	nd	1.3 ± 0.04	nd	nd	Arsenic 1.1 – <0.7 Cadmium 13.3 – 6.4 Chromium 5.7 - <2 Lead 22.6 – 7.0 Mercury 5.0 – 0.4 Nickel 5.2 - <2 Selenium 1.0 - <1.3 (all ng cig ⁻¹) ²

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

² 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

	Arsenic	Cadmium	Chromium	Lead	Mercury	Nickel	Selenium
	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹
Limit of Detection	1.0	0.5	1.7	0.7	0.15	1.8	0.9
Limit of Quantitation	2.7	1.2	4.2	2.0	0.25	4.7	2.3