

CONFIDENTIAL

UK SMOKE CONSTITUENTS STUDY

ANNEX

Part 12: Validation

Determination of polycyclic aromatic amines yields in cigarette smoke

Commissioned by:

Tobacco Manufacturers Association
55 Tufton Street
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Validation data for Aromatic Amines

1. Overview

The determination of Aromatic amines 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	1-Naphthyl Amine (ng/cigt)	2-Naphthyl Amine (ng/cigt)	3-Amino Biphenyl (ng/cigt)	4-Amino Biphenyl (ng/cigt)
2R4F	9.12	5.59	1.27	0.96
2R4F	7.43	5.40	1.38	1.06
2R4F	9.65	5.80	1.31	1.04
2R4F	7.99	5.01	1.38	0.99
2R4F	8.20	5.65	1.66	1.12
2R4F	6.61	5.33	1.42	1.14
2R4F	6.61	4.83	1.34	1.15
2R4F	9.17	6.95	1.51	1.24
2R4F	8.48	5.79	1.70	1.11
2R4F	9.53	6.13	1.58	1.27
2R4F	6.63	3.46	0.90	0.72
2R4F	10.0	5.43	1.37	0.86
2R4F	8.01	4.67	1.32	0.91
2R4F	9.48	5.88	1.41	1.13
2R4F	9.18	5.98	1.63	1.29
2R4F	7.79	5.97	1.37	0.98
2R4F	8.36	6.52	1.41	1.09
2R4F	8.45	6.12	1.46	1.18
2R4F	6.62	5.13	1.23	0.91
2R4F	6.92	5.47	1.26	0.95
2R4F	7.48	5.54	1.22	0.97
2R4F	10.5	6.75	1.44	1.17
2R4F	8.34	6.76	1.56	1.26
2R4F	7.07	4.75	1.24	0.90
Average	8.23	5.62	1.39	1.06
sd	1.16	0.78	0.17	0.15
% RSD	14.1	13.8	12.3	13.9

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

Aromatic Amines (ng/cigt) n=144	Mean	Standard Deviation
1-aminonaphthalene	9.0	1.2
2-aminonaphthalene	5.7	0.7
3-aminobiphenyl	1.5	0.2
4-aminobiphenyl	1.2	0.2

3. Reproducibility

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

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-aminonaphthalene yield ng cig ⁻¹	2-aminonaphthalene yield ng cig ⁻¹	3-aminobiphenyl yield ng cig ⁻¹	4-aminobiphenyl yield ng cig ⁻¹	Comments (anticipated values)
1R4F	5	8.74 ± 1.01	6.71 ± 0.70	1.66 ± 0.14	1.30 ± 0.12	1-aminonaphthalene 17.3 ± 2.2 2-aminonaphthalene 11.7 ± 1.3 3-aminobiphenyl 2.91 ± 0.23 4-aminobiphenyl 2.20 ± 0.20 (all ng cig ⁻¹) ¹
1R5F	5	2.04 ± 0.29	1.57 ± 0.10	0.50 ± 0.05	0.42 ± 0.02	Results from study
2R4F	5	8.23 ± 1.16	5.62 ± 0.78	1.39 ± 0.17	1.06 ± 0.15	Results from study
Brand A	5	6.58 ± 0.91	3.38 ± 0.42	0.84 ± 0.13	0.63 ± 0.09	1-aminonaphthalene 11.9 – 14.1 2-aminonaphthalene 3.9 – 8.5 3-aminobiphenyl 1.5 – 2.5 4-aminobiphenyl 1.0 – 1.8 (all ng cig ⁻¹) ²
Brand B	5	4.60 ± 0.65	2.77 ± 0.24	0.72 ± 0.06	0.54 ± 0.06	1-aminonaphthalene 6.7 – 8.1 2-aminonaphthalene 2.7 – 5.0 3-aminobiphenyl 0.9 – 1.4 4-aminobiphenyl 0.6 – 1.1 (all ng cig ⁻¹) ³

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.

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4. Limit of Detection and Quantitation

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

	1-aminonaphthalene	2-aminonaphthalene	3-aminobiphenyl	4-aminobiphenyl
	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹
Limit of Detection	0.03	0.04	0.01	0.01
Limit of Quantitation	0.10	0.14	0.02	0.02