

CONFIDENTIAL

UK SMOKE CONSTITUENTS STUDY

Part 8: Determination of Tobacco Specific Nitrosamines Yields in Cigarette Smoke

Annex B - Validation

Commissioned by:
Tobacco Manufacturers Association
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Validation data for tobacco specific nitrosamines

1. Overview

The determination of Tobacco Specific Nitrosamines 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 (Part 2 – 7), 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	NAB (ng/cigt)	NAT (ng/cigt)	NNK (ng/cigt)	NNN (ng/cigt)
2R4F	13.8	107.4	108.7	134.3
2R4F	16.0	133.6	135.9	158.3
2R4F	15.0	112.7	127.6	142.2
2R4F	14.0	107.5	120.2	136.1
2R4F	15.1	119.5	127.8	133.0
2R4F	14.9	119.9	132.6	140.6
2R4F	14.0	114.5	120.4	142.8
2R4F	14.2	101.4	122.8	142.0
2R4F	13.7	104.8	118.1	129.5
2R4F	15.0	107.8	125.8	141.1
2R4F	14.1	98.4	107.7	135.0
2R4F	13.4	92.9	114.0	139.5
2R4F	13.3	107.4	111.7	144.8
2R4F	15.2	136.5	125.7	150.8
2R4F	16.8	153.2	139.9	160.9
2R4F	15.4	120.3	139.4	170.9
2R4F	13.8	119.8	121.3	145.3
2R4F	14.1	112.9	116.3	143.1
Average	14.5	115	123	144
sd	0.94	14.6	9.72	10.5
%RSD	6.4	12.7	7.9	7.3

“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

Nitrosamines (ng/cigt) n=88	Mean	Standard deviation
NAB	14.3	1.5
NAT	114	16
NNK	120	11
NNN	137	13

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 tsnas.

Brand	No. of assays	NNN yield ng cig ⁻¹	NNK yield ng cig ⁻¹	NAT yield ng cig ⁻¹	NAB yield ng cig ⁻¹	Comments (anticipated values)
1R4F	5	107 ± 4.99	93.0 ± 5.93	113 ± 12.5	16.1 ± 1.12	NNK 86.8 ± 7.3 ng cig ⁻¹ NNN 104 ± 8 ng cig ⁻¹ NAT 120 ± 10 ng cig ⁻¹ NAB 22.8 ± 2.6 ng cig ⁻¹ ¹
1R5F	5	53.6 ± 4.39	23.9 ± 2.59	52.6 ± 6.51	7.2 ± 0.72	Results from study
2R4F	18	144 ± 10.9	123 ± 9.72	115 ± 14.6	14.5 ± 0.94	Results from study
Brand A	5	22.8 ± 3.88	30.2 ± 5.88	31.5 ± 1.86	5.2 ± 0.56	NNK 31.6 – 41.5 ng cig ⁻¹ NNN 16.3 – 24.2 ng cig ⁻¹ NAT 34.5 – 45.6 ng cig ⁻¹ NAB 8.3 – 3.7 ng cig ⁻¹ ²
Brand B	5	11.4 ± 0.29	17.2 ± 3.37	16.3 ± 0.46	2.6 ± 0.32	NNK 13.6 – 19.2 ng cig ⁻¹ NNN 7.1 – 17.4 ng cig ⁻¹ NAT 17.0 – 22.4 ng cig ⁻¹ NAB 5.8 – 3.1 ng cig ⁻¹ ²

4. Limit of Detection and Quantitation

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

	NAB	NAT	NNK	NNN
	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹	ng cig ⁻¹
Limit of Detection	0.8	1.4	0.4	1.0
Limit of Quantitation	2.5	4.5	1.4	3.5

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.