

# UK SMOKE CONSTITUENTS TESTING STUDY PROTOCOL

## Part 4 : Determination of Benzo[a]pyrene Yields in Cigarette Smoke

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*Setting standards  
in analytical science*

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## UK SMOKE CONSTITUENTS TESTING STUDY PROTOCOL

### Determination of Benzo[a]pyrene Yields in Cigarette Smoke

#### 1. Introduction

This work was undertaken by LGC Limited at the request of the Tobacco Manufacturers' Association in accordance with the Study Protocol provided by, and agreed with, the UK Department of Health.

#### 2. Summary

The objective of this study is to determine the yield ratings of selected smoke constituents (Appendix 1) in mainstream cigarette smoke as identified by the United Kingdom Department of Health. The study encompassed 25 brands of cigarettes representing a 58% market share (July 2001) of the UK market. In addition a Kentucky reference cigarette 1R4F has been smoked as part of the study.

This report details the results for benzo[a]pyrene.

#### 3. Samples

25 brands of cigarettes were selected because their design parameters are representative of the brands in the UK market place. The selection criteria include a range of "tar" values, ventilation, paper permeability, circumference, length, tobacco weight, blend and market share. The Kentucky reference cigarette 1R4F was also included in the study and for some determinations Kentucky 1R5F has been included.

2000 cigarettes of each brand were obtained from a single production batch of current specification (November/December 2001), and stored in plastic containers at 4°C. Cigarettes were selected from packets on a random basis for testing.

Cigarettes were conditioned<sup>1</sup> at a temperature\* of  $22 \pm 1^\circ\text{C}$  and  $60 \pm 2\%$  relative humidity\* for a minimum of 48 hours but not exceeding 10 days.

Butt marking was done in accordance with ISO butt length specifications<sup>2</sup>. Filtered cigarettes were smoked to a measured butt length equal to either the tipping paper + 3 mm or filter length + 8 mm whichever was longer. The minimum butt length was 23 mm and this was used for non filter brands. All smoking was conducted in an environment of temperature  $22 \pm 2^\circ\text{C}$  and  $60 \pm 5\%$  relative humidity<sup>1</sup>.

#### 4. Smoking

The cigarettes were smoked on a 20 channel Filtrona SM350 smoking machine or a Filtrona SM400 smoking machine.

5 cigarettes were sub-sampled from packets chosen on a random basis and smoked to determine the yield of benzo[a]pyrene using the method given below (see section 5) Five determinations were performed for each of the 25 brands & 1R4F. In addition the Kentucky

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\* The parameter is slightly more stringent than that specified in ISO

Reference cigarette 1R5F was also tested. As far as was practicable brands were smoked on different channels on different smoking runs.

ISO conditions<sup>3</sup> for smoking cigarettes were used. The smoking machine puffing parameters was  $35 \pm 0.2 \text{ cm}^3$  puff volume\* with  $2.0 \pm 0.02$  second puff duration once every  $60.0 \pm 0.5$  seconds.

Carbon monoxide was determined in the vapour phase using a non-dispersive infrared analyser using the method described in ISO 8454: 1995<sup>4</sup>.

## 5. Method and Validation

This method is applicable to the quantitative determination of benzo[a]pyrene in mainstream smoke of cigarettes of a range of deliveries. Five conditioned cigarettes are smoked using a 20 channel linear smoking machine. The mainstream smoke is collected onto a 44 mm (Cambridge) glass fibre filter pad. The pad is extracted with cyclohexane and water is added to remove some of the polar compounds present. An aliquot is taken and the solution concentrated under nitrogen. The extract is quantitatively transferred to a silica column. The column is eluted with hexane and the extract solution concentrated to approximately 1 mL under nitrogen. The solution is injected onto a gas capillary column and analysed. The instrument is calibrated with a set of benzo[a]pyrene standards. An internal standard is used to compensate for variations in injection volume and a surrogate standard to compensate for losses in the extraction procedure. QC and blank solutions were analysed with each batch of samples to check that the analysis was under control. The full method is given in an Annexe to this report.

Before use, the method was validated to show that it was suitable for carrying out the bench mark study. The validation data is given in an Annexe to this report.

## 6. Results & Discussion

The results were tabulated for each brand (see Tables). The mean, standard deviation and relative standard deviation were determined for the sets of results for each brand.

A summary of the results is included at the beginning (Page 6). Linear least squares regression analysis has been carried out for benzo[a]pyrene yield versus NFDPM (Page 7) and benzo[a]pyrene yield versus carbon monoxide (Page 8) for the twenty five cigarette brands (excluding 1R4F & 1R5F).

Repeat analyses were only performed where a problem had been identified. In this study there were three runs that had to be repeated because the results for the QC and/or blank sample showed that the calibration had drifted. The problem with maintaining a satisfactory calibration was linked with a decrease in sensitivity of the GC/MS. A programme of cleaning was introduced as it was found that analysis of a large number of smoke solutions caused the injector and detector to become dirty leading to a deterioration in performance. The repeat runs have been identified with an r.

## 7. Outlier Test

It was agreed as part of the study protocol that Dixon's outlier test would be performed on each set of results. This has been carried out and where an outlier has been detected then the result has been flagged "95%". A judgement was then made as to whether to use the original

results or recalculate the mean excluding the outlier. The CV values for each analyte across the 25 brands were examined before making this judgement. As a result of this, the original results have been used in the summary table as comparison of the CV values did not confirm that there are true outliers present in the original data.

## **8. Information provided in the Appendices and Annexes**

Appendix 1 contains technical opinions and interpretations about the method, validation data and the results.<sup>5</sup>

Appendix 2 lists the specific analytes to be determined in the study.

Appendix 3 contains a brief glossary of selected abbreviations and terms used in this report

Appendix 4 contains a brief description of the cigarettes used in this survey. It also lists the butt lengths determined for each brand of cigarette.

There are two annexes to this report. One describes the method used to determine benzo[a]pyrene yields in cigarette smoke. The other contains a summary of the validation data used to show that the method was suitable for the purposes of the bench mark study.

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<sup>1</sup> ISO 3402: 2000 - Tobacco and tobacco products – atmosphere for conditioning and testing

<sup>2</sup> ISO 4387: 2000 - Methods for chemical analysis of tobacco and tobacco products: Determination of total and nicotine- free dry particulate matter using a routine analytical smoking machine

<sup>3</sup> ISO 3308:2000 – Routine analytical cigarette smoking machine: Definitions and standard conditions

<sup>4</sup> ISO 8454:1995 – Chemical analysis of tobacco and tobacco products: Determination of carbon monoxide in the vapour phase cigarette smoke (NDIR method)

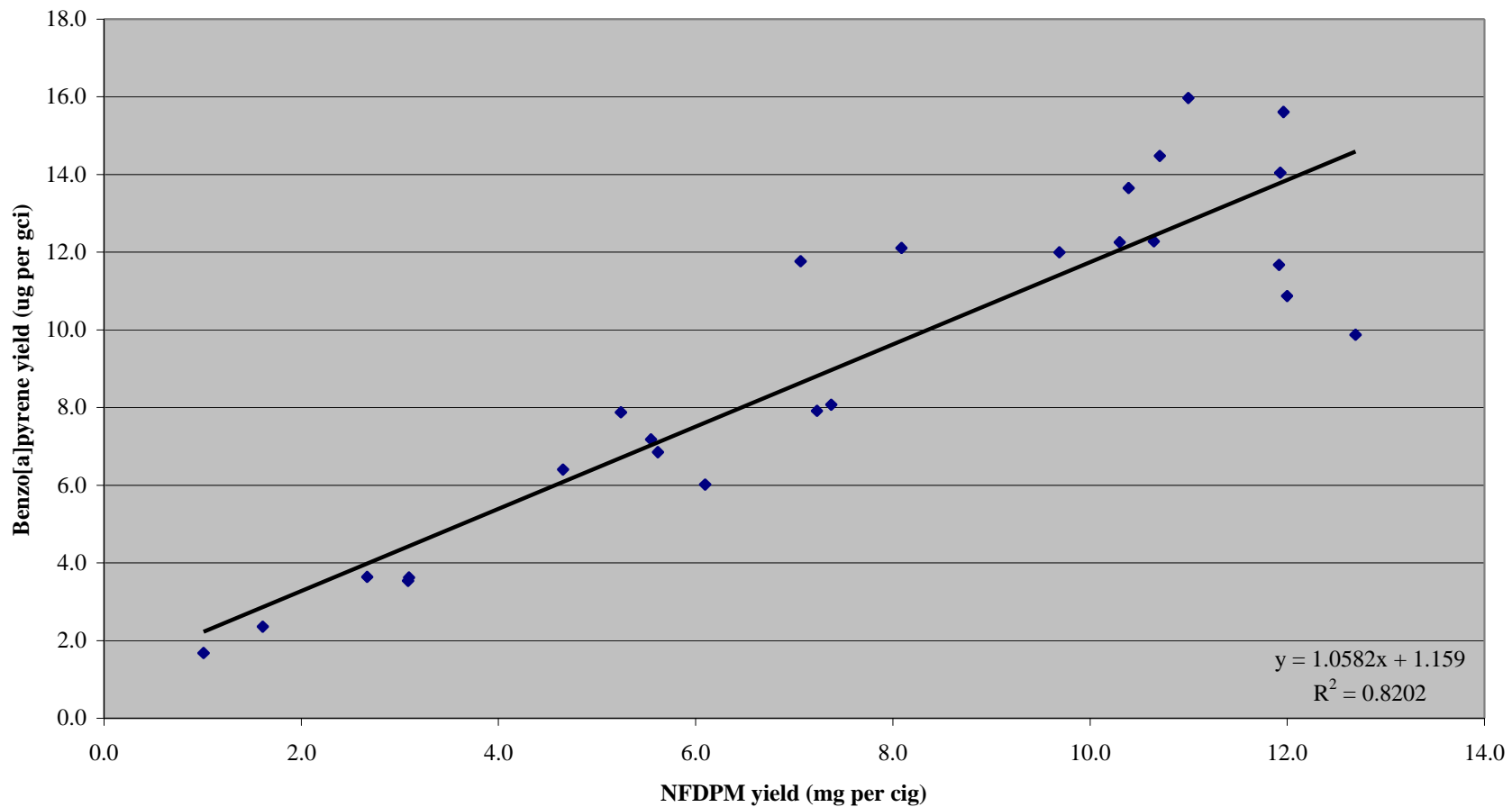
<sup>5</sup> NB When evaluating a set of results obtained using a particular method it is important to put the results in context and this is what we have set out to do in this Appendix.

**Tables****Summary of mean results for 25 cigarette brands plus 1R4F & 1R5F**

<b>Brand</b>	<b>Benzo[a]pyrene</b>	<b>NFDPM</b>	<b>Carbon Monoxide</b>
	ng/cig	mg/cig	mg/cig
<b>1R4F</b>	7.07	9.06	12.26
<b>1R5F</b>	1.80	1.92	3.36
<b>Benson &amp; Hedges King Size</b>	12.3	10.30	11.74
<b>Berkely Superkings</b>	12.0	9.69	11.50
<b>Camel Ultra Lights</b>	3.63	3.09	3.13
<b>Consulate Menthol</b>	11.8	7.06	8.30
<b>Gitanes Caporal Filter</b>	10.9	12.00	12.60
<b>Lambert &amp; Butler King Size</b>	14.0	11.93	13.30
<b>Lambert &amp; Butler Lights King Size</b>	7.88	5.24	6.48
<b>Lambert &amp; Butler Ultra Lights</b>	2.36	1.61	1.49
<b>Marlboro King Size</b>	9.88	12.69	12.79
<b>Marlboro Lights King Size</b>	6.02	6.10	7.19
<b>Mayfair Lights King Size</b>	7.91	7.23	8.73
<b>Mayfair Menthol King Size</b>	6.40	4.65	5.95
<b>Red Band Lights King Size</b>	7.18	5.55	6.41
<b>Regal Filter</b>	12.3	10.65	10.92
<b>Regal King Size</b>	15.6	11.96	13.86
<b>Rothman Royals 120s</b>	13.6	10.39	9.44
<b>Rothman Royals King Size</b>	16.0	11.00	10.86
<b>Senior Service</b>	11.7	11.92	7.71
<b>Silk Cut Extra Mild</b>	3.64	2.67	3.16
<b>Silk Cut King Size</b>	6.85	5.62	5.78
<b>Silk Cut Ultra King Size</b>	1.68	1.01	1.20
<b>Superkings</b>	14.5	10.71	11.41
<b>Superkings Lights</b>	12.1	8.09	7.54
<b>Superkings Ultra Lights</b>	3.54	3.08	3.53
<b>Vogue Superslims</b>	8.07	7.38	6.05

**Regression analysis of benzo[a]pyrene versus NFDPM**

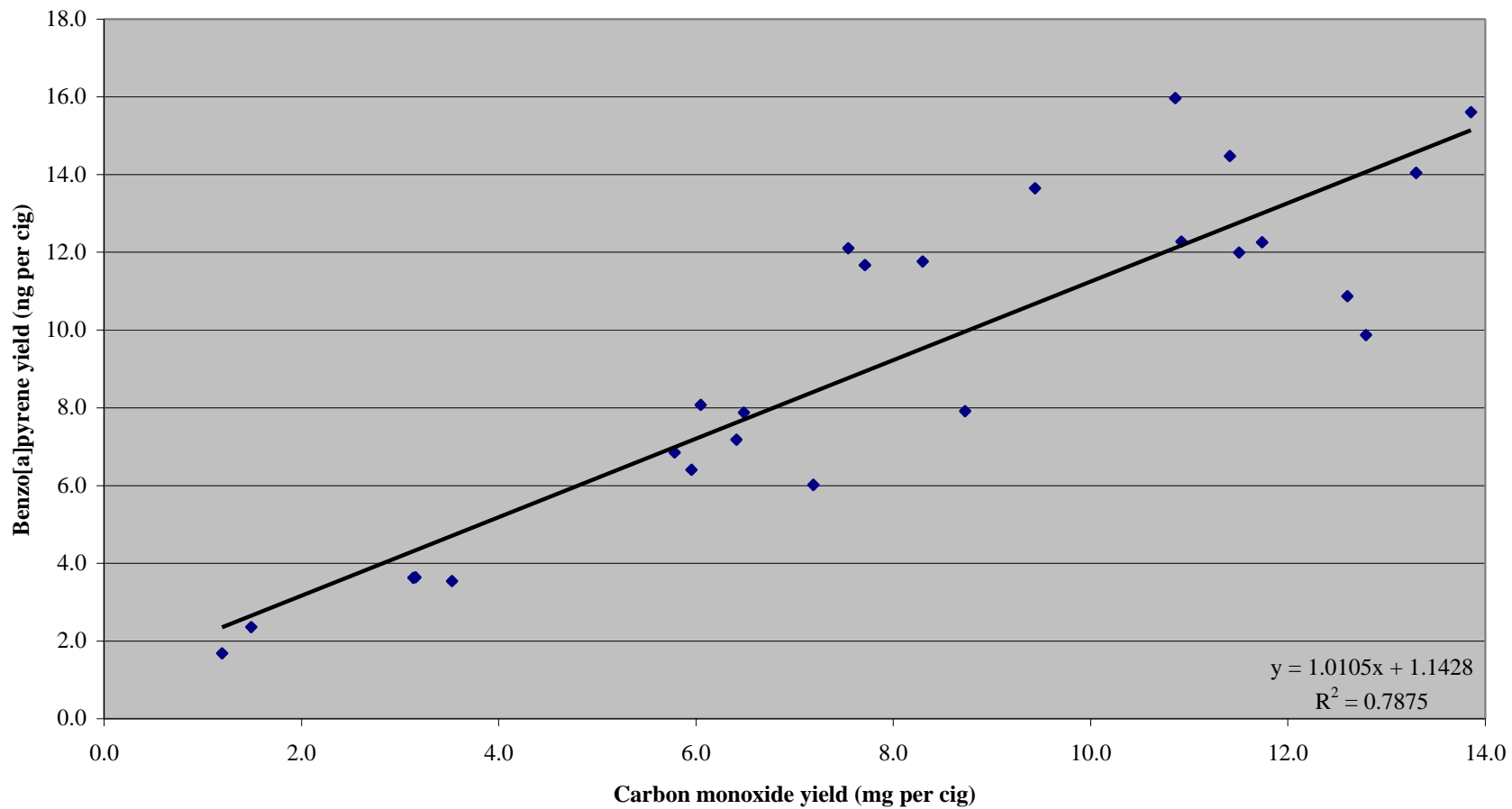
**Regression analysis of benzo[a]pyrene versus NFDPM for 25 cigarette brands**



The regression analysis trend line has been calculated on the basis of a linear relationship ( $y = mx + c$ )

**Regression analysis of benzo[a]pyrene versus carbon monoxide**

**Regression analysis of benzo[a]pyrene versus carbon monoxide for 25 cigarette brands**



The regression analysis trend line has been calculated on the basis of a linear relationship ( $y = mx + c$ )

**1R4F**

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
1R4F	5	11	8.65
1R4F	12	5	6.67
1R4F	8	1	6.97
1R4F	9	17	6.70
1R4F	10	8	6.38
<b>Mean (ug/cig)</b>			7.07
<b>Standard Deviation</b>			0.91
<b>CV (%)</b>			12.8

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

6.376072264  
6.666176961  
6.70182457  
6.968532445  
8.64680418

Statistical test applied

Dixons low end test

Outlier detected at 95%

Dixons high end test

Outlier detected at 95%

0.127758233  
0.739088451  
95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

6.68

**Standard Deviation**

0.24

**CV (%)**

3.6

**1R5F**

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
1R5F	5	2	2.60
1R5F	8	11	1.66
1R5F	9	3	1.53
1R5F	10	18	1.42

**Mean (ug/cig)** 1.80  
**Standard Deviation** 0.54  
**CV (%)** 29.9

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted 1.417862685  
 1.530781218  
 1.661613463  
 2.595955979

Statistical test applied

Dixons low end test 0.095848549

Outlier detected at 95%

Dixons high end test 0.79309722

Outlier detected at 95% 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)** 1.54

**Standard Deviation** 0.12

**CV (%)** 7.94

**Benson & Hedges King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Benson & Hedges King Size	4	11	11.5	
Benson & Hedges King Size	5	5	16.1	
Benson & Hedges King Size	12	18	10.8	r
Benson & Hedges King Size	8	14	10.1	r
Benson & Hedges King Size	9	14	12.8	r
<b>Mean (ug/cig)</b>			12.3	
<b>Standard Deviation</b>			2.37	
<b>CV (%)</b>			19.3	
<i>Outlier Test</i>				
Dixons outlier test was applied to the above data				
Data sorted			10.10595519	
			10.78590351	
			11.49349221	
			12.78482845	
			16.10596074	
Statistical test applied				
Dixons low end test			0.113324616	
Outlier detected at 95%				
Dixons high end test			0.553521536	
Outlier detected at 95%				
<i>If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier</i>				
<b>Summary of Results</b>				
<b>Mean (ug/cig)</b>			12.3	
<b>Standard Deviation</b>			2.37	
<b>CV (%)</b>			19.3	

### Berkely Superkings

Brand	Run	Channel	Benz[a]pyrene yield (ng per cigarette)	
Berkely Superkings	5	1	15.3	
Berkely Superkings	12	14	8.81	r
Berkely Superkings	7	10	13.7	
Berkely Superkings	9	12	11.7	r
Berkely Superkings	10	17	10.5	
<b>Mean (ug/cig)</b>			12.0	
<b>Standard Deviation</b>			2.58	
<b>CV (%)</b>			21.5	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

8.810414394  
 10.47096705  
 11.67826287  
 13.70687258  
 15.31580208

Statistical test applied

Dixons low end test

0.255258062

Outlier detected at 95%

Dixons high end test

0.247322617

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	12.0
<b>Standard Deviation</b>	2.58
<b>CV (%)</b>	21.5

**Camel Ultra Lights**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Camel Ultra Lights	4	20	3.20	
Camel Ultra Lights	12	4	2.96	r
Camel Ultra Lights	7	9	4.66	
Camel Ultra Lights	9	7	3.52	r
Camel Ultra Lights	10	7	3.80	
<b>Mean (ug/cig)</b>			3.63	
<b>Standard Deviation</b>			0.66	
<b>CV (%)</b>			18.2	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

2.960796194  
 3.195916859  
 3.523365188  
 3.800763437  
 4.663389268

Statistical test applied

Dixons low end test

0.138095631

Outlier detected at 95%

Dixons high end test

0.506654141

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	3.63
<b>Standard Deviation</b>	0.66
<b>CV (%)</b>	18.2

**Consulate Menthol**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Consulate Menthol	4	18	10.5	
Consulate Menthol	12	2	10.2	r
Consulate Menthol	7	15	15.4	
Consulate Menthol	9	6	12.3	r
Consulate Menthol	10	5	10.4	
<b>Mean (ug/cig)</b>			11.8	
<b>Standard Deviation</b>			2.21	
<b>CV (%)</b>			18.8	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

10.23171164  
 10.37369067  
 10.48212862  
 12.33485251  
 15.41817155

Statistical test applied

Dixons low end test

0.02737494

Outlier detected at 95%

Dixons high end test

0.59449395

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

11.8

**Standard Deviation**

2.21

**CV (%)**

18.8

### Gitanes Caporal Filter

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Gitanes Caporal Filter	5	12	13.1
Gitanes Caporal Filter	12	6	9.70
Gitanes Caporal Filter	8	2	10.1
Gitanes Caporal Filter	9	8	10.7
Gitanes Caporal Filter	10	9	10.8
<b>Mean (ug/cig)</b>			10.9
<b>Standard Deviation</b>			1.30
<b>CV (%)</b>			12.0

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

9.697397506  
 10.13722504  
 10.67496515  
 10.77200483  
 13.06474344

Statistical test applied

Dixons low end test

0.130615488

Outlier detected at 95%

Dixons high end test

0.680874095

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	10.9
<b>Standard Deviation</b>	1.30
<b>CV (%)</b>	12.0

**Lambert & Butler King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>
Lambert & Butler King Size	4	4	11.6
Lambert & Butler King Size	5	17	16.9
Lambert & Butler King Size	7	1	15.7
Lambert & Butler King Size	8	7	12.2
Lambert & Butler King Size	9	20	13.9
<b>Mean (ug/cig)</b>			14.0
<b>Standard Deviation</b>			2.26
<b>CV (%)</b>			16.1
<i>Outlier Test</i>			
Dixons outlier test was applied to the above data			
Data sorted			11.5853769
			12.16725294
			13.8765575
			15.65768182
			16.92480991
Statistical test applied			
Dixons low end test			0.108977121
Outlier detected at 95%			
Dixons high end test			0.237315102
Outlier detected at 95%			
<i>If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier</i>			
<b>Summary of Results</b>			
<b>Mean (ug/cig)</b>			14.0
<b>Standard Deviation</b>			2.26
<b>CV (%)</b>			16.1

**Lambert & Butler Lights King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>
Lambert & Butler Lights King Size	4	15	6.19
Lambert & Butler Lights King Size	5	9	10.2
Lambert & Butler Lights King Size	7	12	9.87
Lambert & Butler Lights King Size	8	18	6.71
Lambert & Butler Lights King Size	10	2	6.41
<b>Mean (ug/cig)</b>			7.88
<b>Standard Deviation</b>			1.99
<b>CV (%)</b>			25.2
<i>Outlier Test</i>			
Dixons outlier test was applied to the above data			
Data sorted			6.18641786
			6.414922844
			6.70641548
			9.870590908
			10.20859616
Statistical test applied			
Dixons low end test			0.056811252
Outlier detected at 95%			
Dixons high end test			0.084035374
Outlier detected at 95%			
<i>If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier</i>			
<b>Summary of Results</b>			
<b>Mean (ug/cig)</b>			7.88
<b>Standard Deviation</b>			1.99
<b>CV (%)</b>			25.2

**Lambert & Butler Ultra Lights**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>
Lambert & Butler Ultra Lights	4	3	2.59
Lambert & Butler Ultra Lights	5	16	3.21
Lambert & Butler Ultra Lights	12	10	1.76
Lambert & Butler Ultra Lights	8	6	2.15
Lambert & Butler Ultra Lights	9	10	2.09
<b>Mean (ug/cig)</b>			2.36
<b>Standard Deviation</b>			0.56
<b>CV (%)</b>			23.7

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

1.764651552  
 2.090653295  
 2.146529744  
 2.588532827  
 3.21219169

Statistical test applied

Dixons low end test

0.225210849

Outlier detected at 95%

Dixons high end test

0.430840462

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	2.36
<b>Standard Deviation</b>	0.56
<b>CV (%)</b>	23.7

**Marlboro King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Marlboro King Size	4	17	8.53	
Marlboro King Size	12	1	9.02	r
Marlboro King Size	7	14	13.5	
Marlboro King Size	8	20	9.47	r
Marlboro King Size	10	4	8.89	
<b>Mean (ug/cig)</b>			9.88	
<b>Standard Deviation</b>			2.04	
<b>CV (%)</b>			20.7	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

8.527187642  
 8.885425863  
 9.021008138  
 9.472667244  
 13.47394737

Statistical test applied

Dixons low end test

0.072418763

Outlier detected at 95%

Dixons high end test

0.808868905

Outlier detected at 95%

95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

8.98

**Standard Deviation**

0.39

**CV (%)**

4.4

**Marlboro Lights King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Marlboro Lights King Size	4	7	5.98	
Marlboro Lights King Size	12	15	5.76	r
Marlboro Lights King Size	7	4	7.30	
Marlboro Lights King Size	8	10	5.12	r
Marlboro Lights King Size	10	13	5.92	
<b>Mean (ug/cig)</b>			6.02	
<b>Standard Deviation</b>			0.79	
<b>CV (%)</b>			13.2	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

5.124811057  
5.763554949  
5.922629869  
5.977316021  
7.299169561

Statistical test applied

Dixons low end test

0.293761995

Outlier detected at 95%

Dixons high end test

0.607928057

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	6.02
<b>Standard Deviation</b>	0.79
<b>CV (%)</b>	13.2

**Mayfair Lights King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Mayfair Lights King Size	4	1	8.01	
Mayfair Lights King Size	5	14	10.2	
Mayfair Lights King Size	12	8	6.76	r
Mayfair Lights King Size	8	4	7.00	r
Mayfair Lights King Size	9	9	7.64	r
<b>Mean (ug/cig)</b>			7.91	
<b>Standard Deviation</b>			1.36	
<b>CV (%)</b>			17.1	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

6.756228983  
 6.998792391  
 7.636249092  
 8.008960322  
 10.172183

Statistical test applied

Dixons low end test

0.071008979

Outlier detected at 95%

Dixons high end test

0.633270432

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	7.91
<b>Standard Deviation</b>	1.36
<b>CV (%)</b>	17.1

**Mayfair Menthol King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Mayfair Menthol King Size	4	2	6.37	
Mayfair Menthol King Size	5	15	9.64	
Mayfair Menthol King Size	12	9	4.63	r
Mayfair Menthol King Size	8	5	5.96	r
Mayfair Menthol King Size	9	19	5.42	r
<b>Mean (ug/cig)</b>			6.40	
<b>Standard Deviation</b>			1.92	
<b>CV (%)</b>			30.0	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

4.628712009  
5.420329337  
5.961533564  
6.368974903  
9.64151724

Statistical test applied

Dixons low end test

0.157919028

Outlier detected at 95%

Dixons high end test

0.652836523

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

6.40

**Standard Deviation**

1.92

**CV (%)**

30.0

**Red Band Lights King Size**

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Red Band Lights King Size	4	12	7.16
Red Band Lights King Size	5	6	8.72
Red Band Lights King Size	12	19	6.22
Red Band Lights King Size	8	15	6.34
Red Band Lights King Size	9	5	7.45
<b>Mean (ug/cig)</b>			7.18
<b>Standard Deviation</b>			1.01
<b>CV (%)</b>			14.1

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

6.220221352  
 6.343078202  
 7.164627695  
 7.445388929  
 8.722806375

Statistical test applied

Dixons low end test

0.049091978

Outlier detected at 95%

Dixons high end test

0.510439179

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

7.18

**Standard Deviation**

1.01

**CV (%)**

14.1

**Regal Filter**

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Regal Filter	4	13	10.8
Regal Filter	5	7	13.8
Regal Filter	12	20	12.2
Regal Filter	8	16	11.3
Regal Filter	9	15	13.2
<b>Mean (ug/cig)</b>			12.3
<b>Standard Deviation</b>			1.26
<b>CV (%)</b>			10.2

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted	10.84063609
	11.29454882
	12.21270014
	13.21265832
	13.82981307

Statistical test applied

Dixons low end test 0.151852074

Outlier detected at 95%

Dixons high end test 0.2064631

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	12.3
<b>Standard Deviation</b>	1.26
<b>CV (%)</b>	10.2

**Regal King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>
Regal King Size	4	14	13.5
Regal King Size	5	8	21.6
Regal King Size	7	11	16.2
Regal King Size	8	17	12.0
Regal King Size	10	1	14.7
<b>Mean (ug/cig)</b>			15.6
<b>Standard Deviation</b>			3.69
<b>CV (%)</b>			23.6

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

12.04495699  
 13.49642553  
 14.69293256  
 16.19800041  
 21.62150307

Statistical test applied

Dixons low end test

0.15156493

Outlier detected at 95%

Dixons high end test

0.566331809

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	15.6
<b>Standard Deviation</b>	3.69
<b>CV (%)</b>	23.6

**Rothman Royals 120s**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Rothman Royals 120s	4	10	13.5	
Rothman Royals 120s	12	13	12.7	r
Rothman Royals 120s	7	8	16.9	
Rothman Royals 120s	9	2	11.5	r
Rothman Royals 120s	10	16	13.7	
<b>Mean (ug/cig)</b>			13.6	
<b>Standard Deviation</b>			1.99	
<b>CV (%)</b>			14.6	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

11.54190592  
 12.67069672  
 13.45065252  
 13.7163829  
 16.86854383

Statistical test applied

Dixons low end test

0.21191431

Outlier detected at 95%

Dixons high end test

0.591773082

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	13.6
<b>Standard Deviation</b>	1.99
<b>CV (%)</b>	14.6

**Rothman Royals King Size**

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)	
Rothman Royals King Size	4	19	14.2	
Rothman Royals King Size	12	3	14.0	r
Rothman Royals King Size	7	7	20.2	
Rothman Royals King Size	9	16	16.0	r
Rothman Royals King Size	10	6	15.5	
<b>Mean (ug/cig)</b>			16.0	
<b>Standard Deviation</b>			2.50	
<b>CV (%)</b>			15.7	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

13.98717641  
 14.19166114  
 15.4792968  
 16.02888745  
 20.1726168

Statistical test applied

Dixons low end test

0.033059041

Outlier detected at 95%

Dixons high end test

0.669916624

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	16.0
<b>Standard Deviation</b>	2.50
<b>CV (%)</b>	15.7

**Senior Service**

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Senior Service	5	13	14.9
Senior Service	12	7	10.9
Senior Service	8	3	11.2
Senior Service	9	18	11.3
Senior Service	10	10	10.0
<b>Mean (ug/cig)</b>			11.7
<b>Standard Deviation</b>			1.86
<b>CV (%)</b>			15.9

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

10.04316797  
 10.89605807  
 11.22943792  
 11.3248633  
 14.87189758

Statistical test applied

Dixons low end test

0.176628258

Outlier detected at 95%

Dixons high end test

0.734568834

Outlier detected at 95%

95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

10.9

**Standard Deviation**

0.58

**CV (%)**

5.4

**Silk Cut Extra Mild**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Silk Cut Extra Mild	4	8	3.66	
Silk Cut Extra Mild	12	11	2.55	r
Silk Cut Extra Mild	7	5	5.00	
Silk Cut Extra Mild	9	1	3.51	r
Silk Cut Extra Mild	10	14	3.47	
<b>Mean (ug/cig)</b>			3.64	
<b>Standard Deviation</b>			0.88	
<b>CV (%)</b>			24.1	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

2.552791299  
 3.472345015  
 3.513731111  
 3.65567709  
 4.99872464

Statistical test applied

Dixons low end test

0.375952075

Outlier detected at 95%

Dixons high end test

0.549094093

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	3.64
<b>Standard Deviation</b>	0.88
<b>CV (%)</b>	24.1

**Silk Cut King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Silk Cut King Size	5	4	8.52	
Silk Cut King Size	12	17	6.40	r
Silk Cut King Size	8	13	6.53	r
Silk Cut King Size	9	4	6.47	r
Silk Cut King Size	10	20	6.33	
<b>Mean (ug/cig)</b>			6.85	
<b>Standard Deviation</b>			0.94	
<b>CV (%)</b>			13.7	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

6.333668122  
6.395428529  
6.470900029  
6.52681659  
8.523030267

Statistical test applied

Dixons low end test

0.028209315

Outlier detected at 95%

Dixons high end test

0.911778658

Outlier detected at 95%

95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

6.43

**Standard Deviation**

0.08

**CV (%)**

1.3

**Silk Cut Ultra King Size**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Silk Cut Ultra King Size	5	3	2.62	
Silk Cut Ultra King Size	12	16	1.36	r
Silk Cut Ultra King Size	8	12	1.32	r
Silk Cut Ultra King Size	9	13	1.39	r
Silk Cut Ultra King Size	10	19	1.73	
<b>Mean (ug/cig)</b>			1.68	
<b>Standard Deviation</b>			0.55	
<b>CV (%)</b>			32.7	
<i>Outlier Test</i>				
Dixons outlier test was applied to the above data				
Data sorted			1.315224146	
			1.360165031	
			1.385811155	
			1.732496989	
			2.620252094	
Statistical test applied				
Dixons low end test			0.034436723	
Outlier detected at 95%				
Dixons high end test			0.680257543	
Outlier detected at 95%				
<i>If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier</i>				
<b>Summary of Results</b>				
<b>Mean (ug/cig)</b>			1.68	
<b>Standard Deviation</b>			0.55	
<b>CV (%)</b>			32.7	

### Superkings

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Superkings	4	6	11.9
Superkings	5	19	17.4
Superkings	7	3	18.2
Superkings	8	9	13.5
Superkings	10	12	11.3
<b>Mean (ug/cig)</b>			14.5
<b>Standard Deviation</b>			3.17
<b>CV (%)</b>			21.9

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

11.34597942  
 11.87692388  
 13.5427751  
 17.41681689  
 18.22530473

Statistical test applied

Dixons low end test

0.077179728

Outlier detected at 95%

Dixons high end test

0.117524293

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

14.5

**Standard Deviation**

3.17

**CV (%)**

21.9

### Superkings Lights

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Superkings Lights	4	16	10.6
Superkings Lights	5	10	13.9
Superkings Lights	7	13	12.9
Superkings Lights	8	19	12.7
Superkings Lights	10	3	10.5
<b>Mean (ug/cig)</b>			12.1
<b>Standard Deviation</b>			1.52
<b>CV (%)</b>			12.5

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

10.48370015  
 10.55839796  
 12.68523082  
 12.89372556  
 13.90075794

Statistical test applied

Dixons low end test

0.02186027

Outlier detected at 95%

Dixons high end test

0.294707448

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

**Mean (ug/cig)**

12.1

**Standard Deviation**

1.52

**CV (%)**

12.5

### Superkings Ultra Lights

Brand	Run	Channel	Benzo[a]pyrene yield (ng per cigarette)
Superkings Ultra Lights	4	5	3.01
Superkings Ultra Lights	5	18	4.25
Superkings Ultra Lights	7	2	4.35
Superkings Ultra Lights	8	8	3.03
Superkings Ultra Lights	10	11	3.06
<b>Mean (ug/cig)</b>			3.54
<b>Standard Deviation</b>			0.69
<b>CV (%)</b>			19.6
<i>Outlier Test</i>			
Dixons outlier test was applied to the above data			
Data sorted			3.009157927
			3.032450965
			3.055126185
			4.245383198
			4.349654445
Statistical test applied			
Dixons low end test			0.017376426
Outlier detected at 95%			
Dixons high end test			0.077785541
Outlier detected at 95%			
<i>If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier</i>			
<b>Summary of Results</b>			
<b>Mean (ug/cig)</b>			3.54
<b>Standard Deviation</b>			0.69
<b>CV (%)</b>			19.6

**Vogue Superslims**

<b>Brand</b>	<b>Run</b>	<b>Channel</b>	<b>Benzo[a]pyrene yield (ng per cigarette)</b>	
Vogue Superslims	4	9	8.12	
Vogue Superslims	12	12	6.35	r
Vogue Superslims	7	6	11.3	
Vogue Superslims	9	11	7.83	r
Vogue Superslims	10	15	6.78	
<b>Mean (ug/cig)</b>			8.07	
<b>Standard Deviation</b>			1.94	
<b>CV (%)</b>			24.1	

*Outlier Test*

Dixons outlier test was applied to the above data

Data sorted

6.345466639  
6.777738011  
7.833835183  
8.116218389  
11.29917289

Statistical test applied

Dixons low end test

0.087262213

Outlier detected at 95%

Dixons high end test

0.642540018

Outlier detected at 95%

*If an outlier is detected then the mean, standard deviation and CV have been recalculated excluding the outlier*

**Summary of Results**

<b>Mean (ug/cig)</b>	8.07
<b>Standard Deviation</b>	1.94
<b>CV (%)</b>	24.1

## **Appendix 1: Technical opinions and interpretations**

The following notes have been compiled to point up technical issues encountered in developing and validating the method to determine benzo{a}pyrene in cigarette smoke.

### **Specificity**

The specificity of the method is important to ensure that the target analyte is being measured rather than the many other chemicals present in cigarette smoke. For the determination of benzo[a]pyrene, a gas chromatograph with mass spectrometer detector was used to target the ion specific for BaP in conjunction with reference materials to ensure specificity.

### **Blank correction**

It was found that the calibration curve gave a positive intercept which varied between runs. However, analysis of the blank sample showed no peak in the chromatogram. To negate artificially lower values the results have not been blank corrected. If the value for the intercept was greater than  $1 \text{ ng mL}^{-1}$ , the run was repeated.

### **Measurement uncertainty**

All measurements have an uncertainty associated with them. There are three components in the uncertainty of each result (a) sample (smoking of the cigarette), (b) extraction & clean up and (c) analysis by GC-MS.

The results of the study would suggest that the largest uncertainty is associated with the calibration of the instrument. The slope varied from run to run and it was observed that Run 5 (generally) gave higher yields than any other run. However, as the QC and blank samples gave satisfactory results, there is no evidence to suggest that the calibration for any particular run in the study was faulty.

There is some evidence that samples containing low concentrations of benzo[a]pyrene gave larger uncertainties than “high yield” brands – this would be expected as sample solution concentrations at the bottom end of the calibration range are close to the intercept which varies from run to run.

### **Internal and recovery standards**

It was observed that the internal standard areas could vary significantly throughout a run. It was also noticed that the variation was dependent on the sample matrix, e.g. standards and blank sample solutions gave lower internal standard peak areas than smoke solutions in the same run. In our opinion, the sample matrix is the major reason for the differences in the internal standard peak areas (see Annex – validation data).

Recoveries were found to be variable – typically from 80 to 125% with a mean around the 100%. It was also observed that the blank samples typically gave the lowest peak areas for the recovery standard. In our opinion, the wide range of recoveries is due to the difference in the sample matrix.

**Appendix 2: Selected smoke constituents for UK study**

<b>Type</b>	<b>Specific analyte(s)</b>
	nicotine free dry particulate matter
	nicotine
	carbon monoxide
	ammonia
	hydrogen cyanide
	nitrogen monoxide
Aromatic amines	1-aminonaphthalene
	2-aminonaphthalene
	3-aminobiphenyl
	4-aminobiphenyl
Aldehydes & Ketones	formaldehyde
	acetaldehyde
	acetone
	acrolein
	propionaldehyde
	crotonaldehyde
	methyl ethyl ketone
	butyraldehyde
Nitrosamines	n-nitrosomonicotine (nnn)
	n-nitrosoanatabine (nat)
	n-nitrosanabasine (nab)
	n-nitrososnormicotine ketone (nnk)
Phenols	phenol
	catchechol
	hydroquinone
	resorcinol
	ortho-cresol
	meta-cresol
	para-cresol
Polycyclic aromatic hydrocarbons	benzo[a]pyrene
Semi Volatile Compounds	pyridine
	quinoline
	styrene
Trace Metals	arsenic
	cadmium
	chromium
	lead
	mercury
	nickel
	selenium
Volatile Organic Compounds	benzene
	toluene
	1,3-butadiene
	isoprene
	acrylonitrile

**Appendix 3: Selected abbreviations and terms used in this report**

<b>Term/Definition</b>	<b>Meaning</b>
BaP	Benzo[a]pyrene
Channel	The channel of the smoking machine that the cigarette was smoked on
CO	Carbon Monoxide
CO(%v/v)	Percentage volume of carbon monoxide in the total volume of mainstream smoke corrected for any clearing puffs
Overwrap	The wrapper applied to the mouth end of the cigarette
Run	The smoking run that the cigarette was smoked in
TPM	Total Particulate Matter
Yield	The concentration of analyte measured in the smoke (normally per cigarette)
°C	Degree Celsius
ng	Nanogram
µg	Microgram
mg	Milligram
mL	Millilitre
L	Litre
mm	Millimetre
cig <sup>-1</sup>	per cigarette

**Appendix 4: Description of brands (sold in the UK - Nov/Dec 2001) used in the benchmark study**

<b>Brand</b>	<b>Length (mm)</b>	<b>Butt length used for the study (mm)</b>	<b>Description</b>
Benson & Hedges King Size	84	28	filter – typical UK blend
Berkeley Superkings	99	33	filter – typical UK blend
Camel Ultra Lights	84	35	filter – typical American blend
Consulate Menthol	84	35	filter – typical UK blend – menthol
Gitanes Caporal Filter	70	23	filter – dark air cured blend
Lambert & Butler King Size	84	30	filter – typical UK blend
Lambert & Butler Lights King Size	84	34 (overwrap + 3 mm)	filter – typical UK blend
Lambert & Butler Ultra Lights	84	34 (overwrap + 3 mm)	filter – typical UK blend
Marlboro King Size	84	29	filter – typical American blend
Marlboro Lights King Size	84	35	filter – typical American blend
Mayfair Lights King Size	84	28	filter – typical UK blend
Mayfair Menthol King Size	84	33	filter – typical UK blend – menthol
Red Band Lights King Size	84	33	filter – typical UK blend
Regal Filter	71	26	filter – typical UK blend
Regal King Size	84	30	filter – typical UK blend
Rothman Royals 120s	120	38	filter – typical UK blend
Rothman Royals King Size	84	30	filter – typical UK blend
Senior Service	69	23	plain - typical UK blend
Silk Cut Extra Mild	84	33	filter – typical UK blend
Silk Cut King Size	84	28	filter – typical UK blend
Silk Cut Ultra King Size	84	33 (overwrap + 3 mm)	filter – typical UK blend
Superkings	99	34	filter – typical UK blend
Superkings Lights	99	33	filter – typical UK blend
Superkings Ultra Lights	99	34	filter – typical UK blend
Vogue Superslims	99	38	filter – typical American blend