CONFIDENTIAL

UK SMOKE CONSTITUENTS STUDY

Part 5 : Determination of Ammonia Yields in Cigarette Smoke

COMMISSIONED BY :

Tobacco Manufacturers Association

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UK SMOKE CONSTITUENTS TESTING STUDY PROTOCOL

Determination of Ammonia 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(s) has been smoked as part of the study.

This report details the results for Ammonia.

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 included in this part of the study.

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[1] at a temperature[*] of 22 ± 1°C and 60 ± 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[2]. 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 ± 2°C and 60 ± 5 % relative humidity[1].

4. Smoking

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

8 cigarettes were sub-sampled from packets chosen on a random basis and smoked to determine the yield of ammonia using the method given below (see section 5) Five determinations were performed for each of the 25 brands & 1R4F. As far as was practicable sub-samples of each brand were smoked on different days.

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

ISO conditions[3] for smoking cigarettes were used. The smoking machine puffing parameters was 35 ± 0.2 cm^3 puff volume* with 2.0 ± 0.02 second puff duration once every 60.0 ± 0.5 seconds.

5. Method and Validation

This method is applicable to the quantitative determination of ammonia† in mainstream smoke of cigarettes of a range of deliveries. Eight conditioned cigarettes were smoked using a 20 channel linear smoking machine. The mainstream smoke was passed through a 44 mm Cambridge filter pad and the vapour phase through a bubbler containing a solution of malic acid. The pad and solution were combined and the resulting solution shaken for 20 minutes. The solution was filtered through a syringe filter (0.45 μm) and analysed within 12 hours using a Dionex ion chromatograph fitted with a conductivity cell.

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 each set of results.

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

Due to an auto-sampler malfunction, some sample solutions were over 24 hours old when analysed and it was observed that the ammonium ion concentrations had increased. The standard operating procedure states that analysis should take place within 12 hours. Therefore, these runs were repeated (denoted by an "r" at the end of the data).

One cigarette brand consistently gave sample solution concentrations that were greater than the top standard (2.5 μg mL^{-1}). An aliquot of each sample solution for this brand was diluted (1:1) with malic acid and analysed at the end of the run. It was found that the ammonium ion concentration had increased by ca 15 - 20%. However, it is not known if this increase would be due to ageing of the sample solution (see Annexe - validation data), a change in equilibrium conditions due to dilution of the sample, an effect specific to this brand, etc. Earlier validation work had shown that a reasonable fit for the calibration curve had been achieved with a wider range of ammonium standards (r^2 = 0.995, calibration range 0.25 - 4 μg mL^{-1}) Therefore, it was felt more appropriate to report the result for the original undiluted solution.

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

† In this report, ammonia is used to describe the 'analyte' in the vapour phase and ammonium ions to describe the 'analyte' in solution.

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 Annex

Appendix 1 contains technical opinions and interpretations about the method, validation data and the results [4].

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.

The Annex to this report lists the method used to determine the ammonia yields in cigarette smoke. It also contains a summary of the validation data used to show that the method was suitable for the purposes of the bench mark study.

9. Acknowledgement

The author would like to acknowledge the help given by Clare Hughes and Steve Purkis at ITL for providing advice on sample stability and method reliability.

10. June 2003 revision

A calculation error was found on Page 28 of the report. This error has been corrected and the revised report issued in June 2003

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[1] ISO 3402: 2000 - Tobacco and tobacco products – atmosphere for conditioning and testing
[2] 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
[3] ISO 3308:2000 – Routine analytical cigarette smoking machine: Definitions and standard conditions
[4] 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.

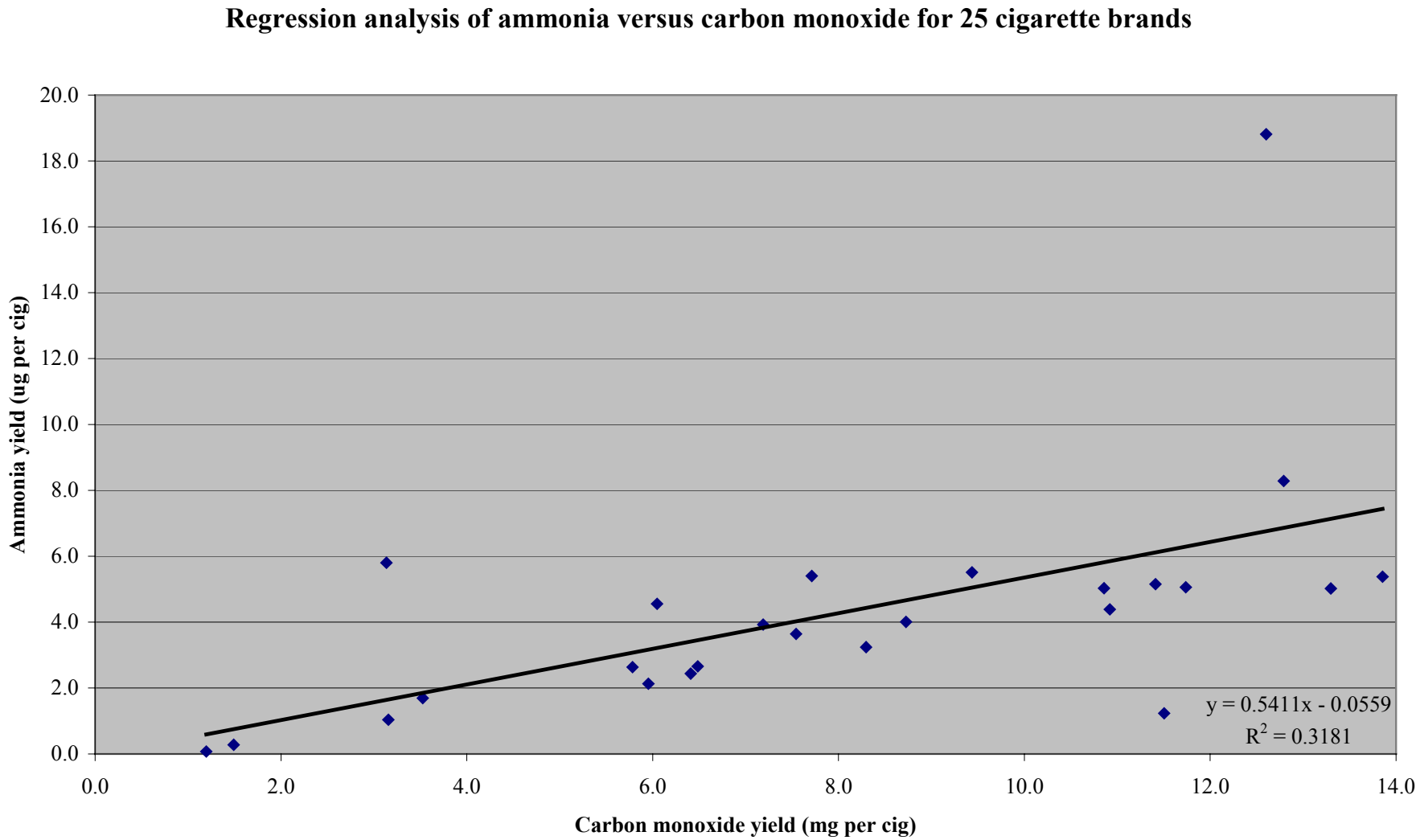
Tables

Summary of mean results for 25 cigarette brands plus 1R4F

Brand	Ammonia	Ammonia results for linear regression analysis	NFDPM	Carbon Monoxide
	ug/cig	ug/cig	mg/cig	mg/cig
1R4F	6.2	6.2	9.06	12.26
Benson & Hedges King Size	5.1	5.1	10.30	11.74
Berkely Superkings	<1.3	1.2	9.69	11.50
Camel Ultra Lights	5.8	5.8	3.09	3.13
Consulate Menthol	3.2	3.2	7.06	8.30
Gitanes Caporal Filter	18.8	18.8	12.00	12.60
Lambert & Butler King Size	5.0	5.0	11.93	13.30
Lambert & Butler Lights King Size	2.7	2.7	5.24	6.48
Lambert & Butler Ultra Lights	<1.3	0.3	1.61	1.49
Marlboro King Size	8.3	8.3	12.69	12.79
Marlboro Lights King Size	3.9	3.9	6.10	7.19
Mayfair Lights King Size	4.0	4.0	7.23	8.73
Mayfair Menthol King Size	2.1	2.1	4.65	5.95
Red Band Lights King Size	2.4	2.4	5.55	6.41
Regal Filter	4.4	4.4	10.65	10.92
Regal King Size	5.4	5.4	11.96	13.86
Rothman Royals 120s	5.5	5.5	10.39	9.44
Rothman Royals King Size	5.0	5.0	11.00	10.86
Senior Service	5.4	5.4	11.92	7.71
Silk Cut Extra Mild	<1.3	1.0	2.67	3.16
Silk Cut King Size	2.6	2.6	5.62	5.78
Silk Cut Ultra King Size	<1.3	0.1	1.01	1.20
Superkings	5.2	5.2	10.71	11.41
Superkings Lights	3.6	3.6	8.09	7.54
Superkings Ultra Lights	1.7	1.7	3.08	3.53
Vogue Superslims	4.6	4.6	7.38	6.05

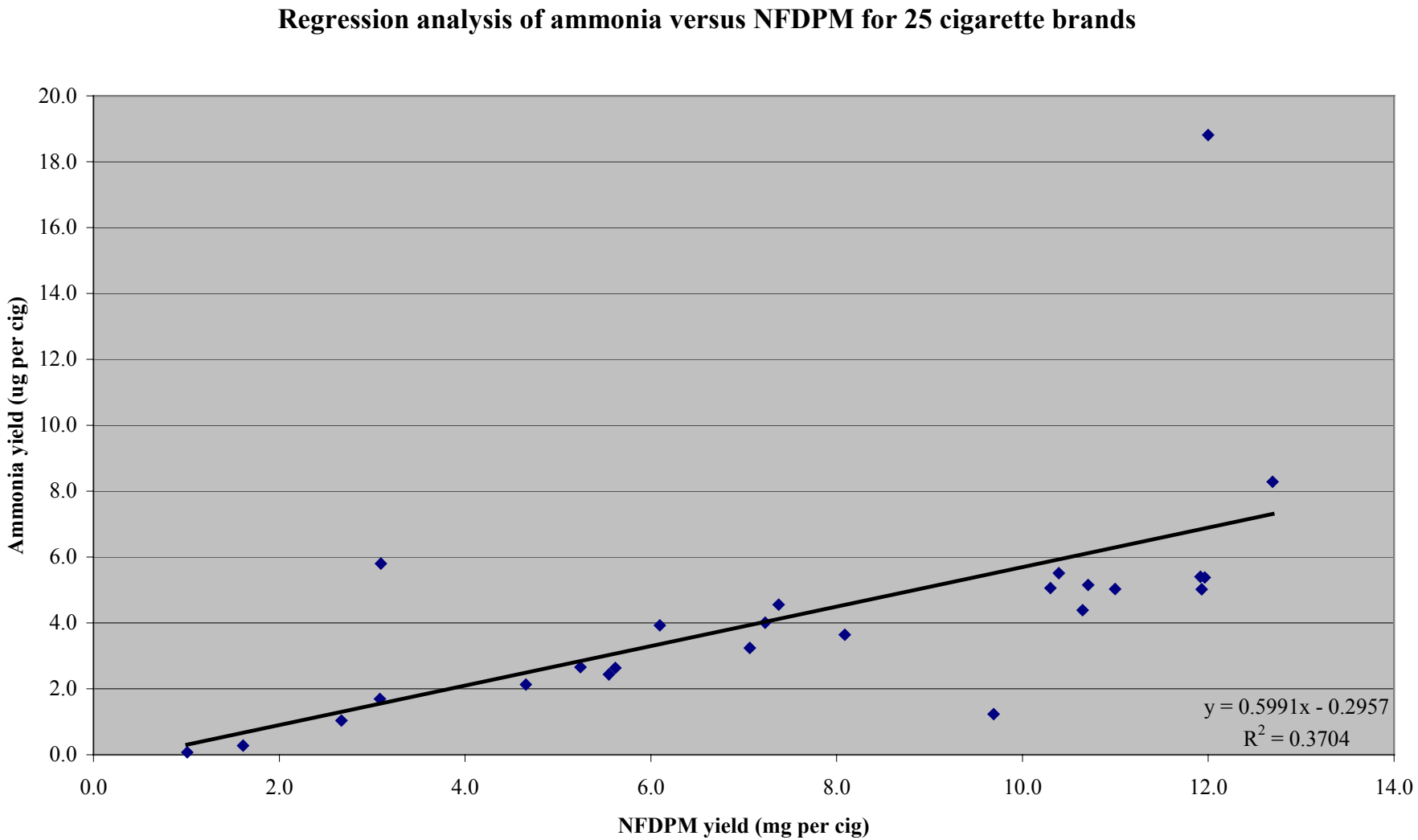
Note: Four of the brands gave ammonia yields <1.3 μg cig^{-1}. In these cases, the original ammonia results have been used to perform the regression analysis (shown in grey).

Regression analysis of ammonia versus carbon monoxide



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

Regression analysis of ammonia versus NFDPM



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

1R4F

Brand	Sample Number	Ammonia yield (ug per cigarette)
1R4F	1810	7.7
1R4F	1908	5.4
1R4F	2205	6.3
1R4F	2502	6.4
1R4F	2709	4.9
Mean (ug/cig)		6.2
Standard Deviation		1.10
CV (%)		17.8

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	5.568416208
	6.317086964
	6.447735533
	7.73928106
	10.59551015
Statistical test applied	
Dixons low end test	0.148927147
Outlier detected at 95%	
Dixons high end test	0.568167041
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)	6.2
Standard Deviation	1.10
CV (%)	17.8

Benson & Hedges King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Benson & Hedges King Size	1708	5.9
Benson & Hedges King Size	2005	4.4
Benson & Hedges King Size	2302	5.6
Benson & Hedges King Size	2509	4.9
Benson & Hedges King Size	2807	4.6
Mean (ug/cig)		5.1
Standard Deviation		0.65
CV (%)		12.8

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.57400168089
	4.93024032238
	5.55328981912
	5.88915434082
	8.25728855469
Statistical test applied	
Dixons low end test	0.096717593
Outlier detected at 95%	
Dixons high end test	0.642940475
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)	5.1
Standard Deviation	0.65
CV (%)	12.8

Berkely Superkings

Brand	Sample Number	Sample solution diluted	Ammonia yield (ug per cigarette)	
Berkely Superkings	1906	no	<1.3	r
Berkely Superkings	2203	no	1.4	
Berkely Superkings	2410	no	1.5	
Berkely Superkings	2603	no	<1.3	
Berkely Superkings	2707	no	<1.3	
Mean (ug/cig)			1.2	*
Standard Deviation			0.09	
CV (%)			7.2	

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	1.033874962
	1.039657707
	1.199531734
	1.388014492
	1.514222145
Statistical test applied	
Dixons low end test	0.012038678
Outlier detected at 95%	
Dixons high end test	0.26274257
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)	1.2
Standard Deviation	0.09
CV (%)	7.2

Report as	
Mean (ug/cig)	<1.3
Standard Deviation	n/a
CV (%)	n/a

* The mean, standard deviation and CV have been calculated using all 5 results. As the mean value is below the reporting limit set in the method, the result has been reported as < 1.3 ug/cig

Camel Ultra Lights

Brand	Sample Number	Ammonia yield (ug per cigarette)
Camel Ultra Lights	1702	6.2
Camel Ultra Lights	1905	5.4
Camel Ultra Lights	2202	6.4
Camel Ultra Lights	2409	6.1
Camel Ultra Lights	2706	4.9
Mean (ug/cig)		5.8
Standard Deviation		0.65
CV (%)		11.2

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.851665066
	5.411193132
	6.116451205
	6.228542764
	6.388635065
Statistical test applied	
Dixons low end test	0.364046186
Outlier detected at 95%	
Dixons high end test	0.104160979
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)	5.8
Standard Deviation	0.65
CV (%)	11.2

Consulate Menthol

Brand	Sample Number	Ammonia yield (ug per cigarette)
Consulate Menthol	1902	2.8
Consulate Menthol	2109	4.3
Consulate Menthol	2406	3.1
Consulate Menthol	2703	3.3
Consulate Menthol	2910	2.7
Mean (ug/cig)		3.2
Standard Deviation		0.67
CV (%)		20.6

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	2.657002006
	2.781533152
	3.126335642
	3.311090125
	4.343739713
Statistical test applied	
Dixons low end test	0.073829586
Outlier detected at 95%	
Dixons high end test	0.612217053
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)	3.2
Standard Deviation	0.67
CV (%)	20.6

Gitanes Caporal Filter

Brand	Sample Number	Ammonia yield (ug per cigarette)
Gitanes Caporal Filter	1703	21.9
Gitanes Caporal Filter	1909	18.3
Gitanes Caporal Filter	2207	19.4
Gitanes Caporal Filter	2504	20.1
Gitanes Caporal Filter	2802	14.3
Mean (ug/cig)		18.8
Standard Deviation		2.82
CV (%)		15.0

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	14.3456486
	18.31139504
	19.4089542
	20.07361187
	21.91578485
Statistical test applied	
Dixons low end test	0.523867248
Outlier detected at 95%	
Dixons high end test	0.2433474
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)	18.8
Standard Deviation	2.82
CV (%)	15.0

Lambert & Butler King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Lambert & Butler King Size	1802	5.7
Lambert & Butler King Size	1910	4.9
Lambert & Butler King Size	2306	5.0
Lambert & Butler King Size	2604	4.8
Lambert & Butler King Size	2901	4.7
Mean (ug/cig)		5.0
Standard Deviation		0.42
CV (%)		8.3

Outlier Test
Dixons outlier test was applied to the above data

Data sorted		4.682110565
		4.804684311
		4.905470118
		4.977448164
		5.744684318
Statistical test applied		
Dixons low end test		0.115355518
Outlier detected at 95%		
Dixons high end test		0.722054494
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)	4.8
Standard Deviation	0.13
CV (%)	2.6

Lambert & Butler Lights King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Lambert & Butler Lights King Size	1805	2.8
Lambert & Butler Lights King Size	2103	3.1
Lambert & Butler Lights King Size	2309	2.8
Lambert & Butler Lights King Size	2607	2.4
Lambert & Butler Lights King Size	2904	2.2
Mean (ug/cig)		2.7
Standard Deviation		0.36
CV (%)		13.6

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	2.155024003
	2.436338745
	2.781009889
	2.844224962
	3.074874338
Statistical test applied	
Dixons low end test	0.305826646
Outlier detected at 95%	
Dixons high end test	0.250746635
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)	2.7
Standard Deviation	0.36
CV (%)	13.6

Lambert & Butler Ultra Lights

Brand	Sample Number	Ammonia yield (ug per cigarette)	
Lambert & Butler Ultra Lights	1701	<1.3	
Lambert & Butler Ultra Lights	2008	<1.3	
Lambert & Butler Ultra Lights	2305	<1.3	
Lambert & Butler Ultra Lights	2602	<1.3	
Lambert & Butler Ultra Lights	2810	<1.3	
Mean (ug/cig)		0.3	*
Standard Deviation		0.17	
CV (%)		62.0	

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	0.126412847
	0.156164717
	0.178515422
	0.442878365
	0.489050554
Statistical test applied	
Dixons low end test	0.082042957
Outlier detected at 95%	
Dixons high end test	0.127323187
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)	0.3
Standard Deviation	0.17
CV (%)	62.0

Report as	
Mean (ug/cig)	<1.3
Standard Deviation	n/a
CV (%)	n/a

* The mean, standard deviation and CV have been calculated using all 5 results. As the mean value is below the reporting limit set in the method, the result has been reported as < 1.3 ug/cig

Marlboro King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Marlboro King Size	1809	8.3
Marlboro King Size	2107	10.6
Marlboro King Size	2404	7.2
Marlboro King Size	2701	7.8
Marlboro King Size	2908	7.5
Mean (ug/cig)		8.3
Standard Deviation		1.37
CV (%)		16.6

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	7.157686174
	7.503101444
	7.809759951
	8.333877041
	10.61094405
Statistical test applied	
Dixons low end test	0.100025912
Outlier detected at 95%	
Dixons high end test	0.659396747
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)	8.3
Standard Deviation	1.37
CV (%)	16.6

Marlboro Lights King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Marlboro Lights King Size	1808	4.5
Marlboro Lights King Size	2106	4.7
Marlboro Lights King Size	2403	3.7
Marlboro Lights King Size	2610	3.7
Marlboro Lights King Size	2907	3.0
Mean (ug/cig)		3.9
Standard Deviation		0.69
CV (%)		17.7

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	3.009974213
	3.656431271
	3.724251983
	4.522502715
	4.708444405
Statistical test applied	
Dixons low end test	0.380611365
Outlier detected at 95%	
Dixons high end test	0.109475981
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)	3.9
Standard Deviation	0.69
CV (%)	17.7

Mayfair Lights King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Mayfair Lights King Size	1707	4.6
Mayfair Lights King Size	2004	3.2
Mayfair Lights King Size	2301	4.2
Mayfair Lights King Size	2508	4.2
Mayfair Lights King Size	2806	3.8
Mean (ug/cig)		4.0
Standard Deviation		0.51
CV (%)		12.8

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	3.236140023
	3.837544364
	4.153732178
	4.188650963
	4.616133484
Statistical test applied	
Dixons low end test	0.435802312
Outlier detected at 95%	
Dixons high end test	0.309771411
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)	4.0
Standard Deviation	0.51
CV (%)	12.8

Mayfair Menthol King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)	
Mayfair Menthol King Size	1709	2.7	
Mayfair Menthol King Size	2006	<1.3	r
Mayfair Menthol King Size	2303	2.1	
Mayfair Menthol King Size	2510	2.2	
Mayfair Menthol King Size	2808	2.4	
Mean (ug/cig)		2.3	*
Standard Deviation		0.26	
CV (%)		10.9	

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	1.272758411
	2.099718675
	2.24425027
	2.353738692
	2.7001329
Statistical test applied	
Dixons low end test	0.579357604
Outlier detected at 95%	
Dixons high end test	0.242679276
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)	2.3
Standard Deviation	0.26
CV (%)	10.9

* The mean, standard deviation and CV have been calculated using all 5 results.

Red Band Lights King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Red Band Lights King Size	1710	2.9
Red Band Lights King Size	2007	1.9
Red Band Lights King Size	2304	2.6
Red Band Lights King Size	2601	2.3
Red Band Lights King Size	2809	2.5
Mean (ug/cig)		2.4
Standard Deviation		0.35
CV (%)		14.6

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	1.922731994
	2.288828793
	2.480454369
	2.619916767
	2.862419689
Statistical test applied	
Dixons low end test	0.389594118
Outlier detected at 95%	
Dixons high end test	0.258067572
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)	2.4
Standard Deviation	0.35
CV (%)	14.6

Regal Filter

Brand	Sample Number	Ammonia yield (ug per cigarette)
Regal Filter	1801	4.7
Regal Filter	2009	4.7
Regal Filter	2206	4.9
Regal Filter	2503	4.3
Regal Filter	2710	3.3
Mean (ug/cig)		4.4
Standard Deviation		0.65
CV (%)		14.8

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	3.294871647
	4.30643282
	4.688599823
	4.714850051
	4.924263137
Statistical test applied	
Dixons low end test	0.620821441
Outlier detected at 95%	
Dixons high end test	0.128522266
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)	4.4
Standard Deviation	0.65
CV (%)	14.8

Regal King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Regal King Size	1803	5.9
Regal King Size	2101	6.6
Regal King Size	2307	5.2
Regal King Size	2605	4.7
Regal King Size	2902	4.5
Mean (ug/cig)		5.4
Standard Deviation		0.88
CV (%)		16.3

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.486241022
	4.678870882
	5.2133151
	5.898074114
	6.603951378
Statistical test applied	
Dixons low end test	0.090961382
Outlier detected at 95%	
Dixons high end test	0.333320967
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)	5.4
Standard Deviation	0.88
CV (%)	16.3

Rothman Royals 120s

Brand	Sample Number	Ammonia yield (ug per cigarette)	
Rothman Royals 120s	1904	5.2	r
Rothman Royals 120s	2010	5.4	r
Rothman Royals 120s	2201	6.5	
Rothman Royals 120s	2408	5.9	
Rothman Royals 120s	2705	4.5	
Mean (ug/cig)		5.5	
Standard Deviation		0.73	
CV (%)		13.2	

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.542953955
	5.220598354
	5.400911106
	5.944997725
	6.46197904
Statistical test applied	
Dixons low end test	0.353119094
Outlier detected at 95%	
Dixons high end test	0.269397893
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)	5.5
Standard Deviation	0.73
CV (%)	13.2

Rothman Royals King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Rothman Royals King Size	1903	4.3
Rothman Royals King Size	2110	6.4
Rothman Royals King Size	2310	4.9
Rothman Royals King Size	2407	4.9
Rothman Royals King Size	2704	4.6
Mean (ug/cig)		5.0
Standard Deviation		0.81
CV (%)		16.2

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.312649107
	4.564804118
	4.907259064
	4.948005428
	6.408768476
Statistical test applied	
Dixons low end test	0.120296112
Outlier detected at 95%	
Dixons high end test	0.696889247
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)	5.0
Standard Deviation	0.81
CV (%)	16.2

Senior Service

Brand	Sample Number	Ammonia yield (ug per cigarette)
Senior Service	1705	6.0
Senior Service	2002	5.1
Senior Service	2209	6.5
Senior Service	2506	4.6
Senior Service	2804	4.8
Mean (ug/cig)		5.4
Standard Deviation		0.80
CV (%)		14.7

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.6064815
	4.821494363
	5.139028335
	5.956177178
	6.492647475
Statistical test applied	
Dixons low end test	0.113994667
Outlier detected at 95%	
Dixons high end test	0.284423696
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)	5.4
Standard Deviation	0.80
CV (%)	14.7

Silk Cut Extra Mild

Brand	Sample Number	Ammonia yield (ug per cigarette)
Silk Cut Extra Mild	1901	<1.3
Silk Cut Extra Mild	2108	1.5
Silk Cut Extra Mild	2405	<1.3
Silk Cut Extra Mild	2702	<1.3
Silk Cut Extra Mild	2909	<1.3
Mean (ug/cig)		1.0
Standard Deviation		0.27
CV (%)		26.1

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	0.739358435
	0.91088704
	0.994539745
	1.081365687
	1.46734324
Statistical test applied	
Dixons low end test	0.235621133
Outlier detected at 95%	
Dixons high end test	0.530200013
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)	1.0
Standard Deviation	0.27
CV (%)	26.1
Report as	
Mean (ug/cig)	<1.3
Standard Deviation	n/a
CV (%)	n/a

* The mean, standard deviation and CV have been calculated using all 5 results. As the mean value is below the reporting limit set in the method, the result has been reported as < 1.3 ug/cig

Silk Cut King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)
Silk Cut King Size	1706	2.9
Silk Cut King Size	2003	2.4
Silk Cut King Size	2210	2.8
Silk Cut King Size	2507	2.6
Silk Cut King Size	2805	2.5
Mean (ug/cig)		2.6
Standard Deviation		0.20
CV (%)		7.7

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	2.429577366
	2.497490584
	2.575334188
	2.756433011
	2.930533882
Statistical test applied	
Dixons low end test	0.135567092
Outlier detected at 95%	
Dixons high end test	0.347536893
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)	2.6
Standard Deviation	0.20
CV (%)	7.7

Silk Cut Ultra King Size

Brand	Sample Number	Ammonia yield (ug per cigarette)	
Silk Cut Ultra King Size	1704	<1.3	
Silk Cut Ultra King Size	2001	<1.3	r
Silk Cut Ultra King Size	2208	<1.3	
Silk Cut Ultra King Size	2505	<1.3	
Silk Cut Ultra King Size	2803	<1.3	
Mean (ug/cig)		0.1	*
Standard Deviation		0.13	
CV (%)		181	

Outlier Test
Dixons outlier test was applied to the above data

Data sorted		0
		0.002424846
		0.018251053
		0.035757533
		0.308864051
Statistical test applied		
Dixons low end test		0.007850852
Outlier detected at 95%		
Dixons high end test		0.884228892
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)	0.0
Standard Deviation	0.02
CV (%)	117

Report as	
Mean (ug/cig)	<1.3
Standard Deviation	n/a
CV (%)	n/a

* The mean, standard deviation and CV have been calculated using all 5 results. As the mean value is below the reporting limit set in the method, the result has been reported as < 1.3 ug/cig

Superkings

Brand	Sample Number	Ammonia yield (ug per cigarette)
Superkings	1806	5.8
Superkings	2104	5.5
Superkings	2401	5.0
Superkings	2608	4.4
Superkings	2905	5.0
Mean (ug/cig)		5.2
Standard Deviation		0.53
CV (%)		10.2

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	4.408281874
	5.019360273
	5.044879289
	5.511915353
	5.777992285
Statistical test applied	
Dixons low end test	0.446136931
Outlier detected at 95%	
Dixons high end test	0.194257801
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)	5.2
Standard Deviation	0.53
CV (%)	10.2

Superkings Lights

Brand	Sample Number	Ammonia yield (ug per cigarette)
Superkings Lights	1807	4.2
Superkings Lights	2105	4.7
Superkings Lights	2402	3.4
Superkings Lights	2609	3.3
Superkings Lights	2906	2.6
Mean (ug/cig)		3.6
Standard Deviation		0.82
CV (%)		22.4

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	2.568133009
	3.320081435
	3.448882487
	4.197751931
	4.67463241
Statistical test applied	
Dixons low end test	0.356965887
Outlier detected at 95%	
Dixons high end test	0.226385291
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)	3.6
Standard Deviation	0.82
CV (%)	22.4

Superkings Ultra Lights

Brand	Sample Number	Ammonia yield (ug per cigarette)	
Superkings Ultra Lights	1804	2.0	
Superkings Ultra Lights	2102	2.2	
Superkings Ultra Lights	2308	1.6	
Superkings Ultra Lights	2606	<1.3	
Superkings Ultra Lights	2903	1.5	
Mean (ug/cig)		1.7	*
Standard Deviation		0.40	
CV (%)		23.5	

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	1.199980105
	1.503270849
	1.572991759
	1.994369568
	2.191015252
Statistical test applied	
Dixons low end test	0.306034296
Outlier detected at 95%	
Dixons high end test	0.19842453
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)	1.7
Standard Deviation	0.40
CV (%)	23.5

* The mean, standard deviation and CV have been calculated using all 5 results.

Vogue Superslims

Brand	Sample Number	Ammonia yield (ug per cigarette)
Vogue Superslims	1907	3.9
Vogue Superslims	2204	5.1
Vogue Superslims	2501	4.5
Vogue Superslims	2708	4.3
Vogue Superslims	2801	5.0
Mean (ug/cig)		4.6
Standard Deviation		0.49
CV (%)		10.7

Outlier Test
Dixons outlier test was applied to the above data

Data sorted	3.899856413
	4.314679441
	4.516867193
	4.983944482
	5.075745457
Statistical test applied	
Dixons low end test	0.352773954
Outlier detected at 95%	
Dixons high end test	0.07806942
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)	4.6
Standard Deviation	0.49
CV (%)	10.7

Appendix 1: Technical opinions and interpretations

The following comments are of a technical nature about the method, validation data and results obtained during the study. They are designed to help put the results in context.

Trapping system

An ideal trapping system should be capable of trapping 100% of the analyte(s) under investigation. It should also not significantly effect the way that the cigarette smokes – i.e. cigarette should be smoked to ISO conditions.

In practice with the trapping system used, virtually all of the ammonia was trapped on the pad and very little on the bubbler. There was some effect on the puff profile (see Annexe) due to the bubbler. Therefore, it may be a better alternative to leave out the bubbler altogether.

Method sensitivity and reporting limit

The study showed that the reporting limit set was higher than the ammonia yields for some low tar brands. NB The reporting limit of < 1.3 mg cig^{-1} is based on the bottom standard (Ammonium ion concentration = 0.25 mg mL^{-1}, 40 mL solution, 8 cigarettes). The slope of the calibration curve varies slightly from day to day (changing the intercept) so that it was not practicable to determine ammonia yields below the bottom standard in the calibration range. To improve the method sensitivity, the bubbler could be left out and a smaller extraction volume of malic acid (15 mL) used for low tar brands. This would improve the sensitivity of the method from 1.25 mg cig^{-1} to 0.5 mg cig^{-1}.

Free and complex ammonia

It was observed that for some sample solutions (pad plus malic acid) the ammonium ion concentration increased with time. For the purpose of this study, ammonia present in the cigarette smoke is treated as free ammonia (easily liberated) and complex ammonia (bound to the matrix and not readily available). The method has been was set up to measure the free ammonia – i.e. to measure the ammonium ion concentration in a sample solution as soon as practicable after smoking.

Simple data analysis was performed on the results as a check that ageing of sample solutions analysed towards the end of the run did not lead to significantly higher ammonium ion concentrations. It was found that approximately half the brands gave higher results and the other half lower results when two samples were compared for each brand (comparing lowest and highest channel number).

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) trapping the smoke and (c) the analytical method.

From the results of the study, it would appear that the largest uncertainty is associated with the measurement of the ammonium ions. In addition, there is the issue of what is being measured. For this study, it was felt more relevant to measure the ammonium ion concentration in the filtered sample solution due to ammonia in cigarette smoke that was easily liberated.

Appendix 2: Selected smoke constituents for UK study

Type	Specific analyte(s)
	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-nitrosonomicotine (nnn)
	n-nitrosoanatabine (nat)
	n-nitrosanabasine (nab)
	n-nitrosonormicotine 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

Term/Definition	Meaning
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^{-1}	per cigarette
ppm	Parts per million
v/v (ppm)	Volume/volume expressed in parts per million (sometimes called vpm)
NH_3	Ammonia (in gaseous form)
NH_4^+	Ammonium ions (ammonia in aqueous solution)

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

Brand	Length (mm)	Butt length used for the study (mm)	Description
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