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DETERMINATION OF INTENSE SWEETENERS IN FOODSTUFFS: COLLABORATIVE TRIAL

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SUMMARY

The results of a collaborative trial involving 12 laboratories are reported. The method tested comprised aqueous extraction, clean-up on solid phase extraction or with clarification reagents and separation by reversed-phase HPLC and UV detection at 220 nm. The precision parameters calculated from the trial for the determination of acesulfame-K and saccharin were satisfactory when determining these analytes at levels near to the limits prescribed in The Sweeteners in Food Regulations 1995.

INTRODUCTION

Intense (previously known as "artificial") sweeteners are a group of chemically unrelated compounds with "intense" sweetening action which are finding widespread application as sucrose-replacements in a variety of foodstuffs, particularly low-calorie food products. Their use arises in part due to a perceived need to reduce the consumption of sucrose for dietary and health reasons, and because of developments in food processing technologies.

Saccharin, an acidic cyclic sulphonamide, the first of these synthetic compounds known to possess such sweetening properties, is 300 to 500 times sweeter than sucrose. Its chemical structure, 3-oxo-2,3-dihydrobenzo [d]isothiazole-1,1-dioxide, contains an imidic anion, allowing the sodium, potassium and calcium salts of saccharin to also be added to foodstuffs without loss of sweetening action. Saccharin and its salts, however, convey a slightly bitter aftertaste to foods and more recently other synthetic sweeteners such as cyclamic acid (N-cyclohexylsulphamic acid) and its salts, aspartame (L-aspartyl-L-phenylalanine methyl ester), and acesulfame-K (the potassium salt of 6-methyl-1,2,3-oxathiazin-4-one-2,2-dioxide) have been used instead of, or in combination with, saccharin. Major applications of the use of the different

sweeteners by the food industry throughout Europe include soft drinks, milk-based products and confectionery.

Evaluation of the safety-in-use of these compounds has led to restrictions being placed on the use of intense sweeteners for addition to foodstuffs. In the United Kingdom the use of sweeteners is currently controlled by the Sweeteners in Food Regulations (1995)[1], following adoption of the EC Directive 94/35 in 1994[2]. The Regulations prescribe limits for the use of various sweeteners in specified categories of food.

As a consequence of the implementation of this Directive, there is a need to have available validated methods of analysis for the determination of these sweeteners in foodstuffs in order to allow effective enforcement of the prescribed limits.

'Official' methods have previously only been described for the determination of saccharin or cyclamates in foodstuffs, the analyses being based on techniques such as polarography[3], colorimetry[4,5] or gravimetric end-point determination[6,7] and, in the case of the determination of saccharin in soda beverages, on high-performance liquid chromatography[8].

More recently high-performance liquid chromatographic procedures, capable of determining other sweeteners and saccharin simultaneously in various food matrices, have been reported[9-12]. Collaborative trials of such methods carried out by the UK and other Member States, however, have resulted in poor method performance characteristics particularly when applied to the determination of saccharin[13,14]. In view of the lack of suitable methods for determining these sweeteners, a critical assessment of the existing methodologies was carried out at the MAFF Food Science Laboratory and, as a result, a high-performance liquid chromatographic method has been developed and evaluated 'in-house' for two of the more commonly used sweeteners, saccharin and acesulfame-K. The present work was carried out to formally validate this procedure and thereby provide enforcement and other analysts with a validated method for the determination of these sweeteners in food.

METHOD OF ANALYSIS TO BE COLLABORATIVELY TESTED

The method tested was developed at the Food Science Laboratory, Norwich. It comprised aqueous extraction of the sample, clean-up on a solid phase extraction column or with clarification reagents, followed by chromatography on a reversed-phase HPLC column and photometric detection at 220 nm. The method tested is described in Appendix I. It allowed participants some choice in chromatographic conditions, the various chromatographic conditions used in the trial by participants are given in Table 1.

TABLE 1

CHROMATOGRAPHY CONDITIONS USED BY PARTICIPANTS FOR ANALYSIS OF THE TRIAL SAMPLES.

Laboratory Number	HPLC Column	Dimensions	Flow Rate (mL/min)	Changes Made to the Elution Programme of the Method (Times in minutes)
1	Waters C18	300 mm × 3.9 mm	1.3	None
2	Partisil 10 ODS 3 10μm	25 cm × 4.6 mm i.d.	1.3	None
3	Partisil 10 ODS 3 10μm	25 cm × 4.6 mm i.d.	1.3	None
4	5 μm ODS 2	100 mm × 4.6 mm i.d.	0.65	-
5	Spherisorb ODS 2 5 μm	25 cm × 4.6 mm i.d.	1.3	100% A 0-9, 100% B 10-15, 100% A 16-21
6	Partisil 10 ODS 3	250 mm × 4.6 mm i.d.	1.3	100% A 0-13, 50% A/B 14, 50% A/B 17, 100% A 18
7	Partisil 10 ODS 3 10μm	25 cm	1.3	None
8	Spherisorb ODS 2 5 μm	25 cm × 4.6 mm i.d.	1.3	100% A for 8 to 100% B in 1 min. 100% B for 5 to 100% A in 1 min. 100% A for 4 min.
9	Partisil 10 ODS 2	250 mm × 4 mm	1.3	100% A for 13
10	Partisil 10 ODS 3 10μm	250 mm × 4.6 mm	1.3	None
11	ODS 2	25cm, 5 μm	1.3	-
12	ODS 3,10μm	25 × 0.46cm	1.3	100% A for 25

COLLABORATIVE TRIAL ORGANISATION, SAMPLES AND RESULTS

Participants

Twelve U.K. Public Analyst laboratories took part in the study. The method to be used in the trial was sent to participants for comment in advance of the pre-trial .

Pre-trial

Participants were sent one test material comprising of soft drink fortified with 200 mg/L accsulfame-K and 200 mg/L sodium saccharin. Participants were asked to analyse this sample in duplicate after they had familiarised themselves with the method and carried out successfully spiked recovery experiments using a base material supplied with the pre-trial sample. Several comments were received and minor changes to the written method were made at this stage.

Main Trial

Twelve Public Analyst laboratories participated in the main trial and received ten test materials comprising two blind duplicate and eight split level samples. The test materials were prepared by adding combinations of acesulfame-K and sodium saccharin at varying concentrations as shown in Table 2. For each sample type a blank matrix, containing no added sweeteners, was also supplied to participants to allow them to determine recoveries for both analytes.

Sample Preparation

All the samples were prepared at the Food Science Laboratory, Norwich.

Sufficient amounts of each commodity to be used to form the test materials were purchased from retail outlets. The sample commodities were homogenised and sub-samples taken for analysis to verify they were free of detectable acesulfame-K and saccharin. Portions of each sample type were fortified with acesulfame-K and sodium saccharin at the concentrations shown in Table 2 and each fortified material homogenised. Sub-samples for analysis were dispatched to participants after satisfactory homogeneity testing was completed.

TABLE 2

SAMPLE SCHEME USED IN TRIAL

Sample	Sample Code Number	Concentration of acesulfame-K added	Concentration of sodium saccharin added
Orange soft drink	231	350 mg/L	80 mg/L
(split level)	769	370 mg/L	100 mg/L
Cola	558	330 mg/L	75 mg/L
(split level)	307	350 mg/L	95 mg/L
Cream	897	380 mg/kg	84 mg/kg
(split level)	190	358 mg/kg	104 mg/kg
Yoghurt	456	300 mg/kg	80 mg/kg
(split level)	644	325 mg/kg	120 mg/kg
Orange Juice (blind duplicate)	353 625	20 mg/kg	25 mg/kg

The samples were prepared as follows:

Soft drink samples 231 & 769

500 mL of Orange soft drink was placed in a 1 L glass beaker and was stirred for 90 minutes using a magnetic stirrer. 175 mg of acesulfame-K and 40 mg of sodium saccharin, for sample 231, and 185 mg of acesulfame-K and 50 mg of sodium saccharin, for sample 769, were added to the base material which was then stirred for a further 2 hours.

10 mL portions were dispensed into a series of 10 mL vials.

Soft drink samples 558 & 307

500 mL of 'Cola' drink was placed in a 1 L glass beaker and was stirred for 90 minutes using a magnetic stirrer. 165 mg of acesulfame-K and 37.5 mg of sodium saccharin, for sample 558, and 175 mg of acesulfame-K and 47.5 mg of sodium saccharin, for sample 307, were added to the base material which was then stirred for a further 2 hours.

10 mL portions were dispensed into a series of 10 mL vials.

Cream samples 897 & 190

100 g of Single Cream was placed in a 250 mL glass beaker and was stirred for 90 minutes using a magnetic stirrer. 38 mg of acesulfame-K and 8.4 mg of sodium saccharin, for sample 897, and 35.8 mg of acesulfame-K and 10.4 mg of

sodium saccharin, for sample 190, were added to the base material which was then stirred for a further 2 hours.
2 g portions were dispensed into a series of 5 mL vials.

Yoghurt samples 456 & 644
100 g of Natural Yoghurt was placed in a 250 mL glass beaker and was stirred for 90 minutes using a magnetic stirrer. 30 mg of acesulfame-K and 8 mg of sodium saccharin, for sample 456, and 32.5 mg of acesulfame-K and 12 mg of sodium saccharin, for sample 644, were added to the base material which was then stirred for a further 2 hours.
2 g portions were dispensed into a series of 5 mL vials.

Orange Juice samples 353 & 625
500 g of fresh Orange Juice was placed in a 1 L glass beaker and was stirred for 90 minutes using a magnetic stirrer. 10 mg of acesulfame-K and 12.5 mg of sodium saccharin, for sample 353, and 10 mg of acesulfame-K and 12.5 mg of sodium saccharin, for sample 625, were added to the base material which was then stirred for a further 2 hours.
2 g portions were dispensed into a series of 5 mL vials.

Storage of Material

All prepared test materials were stored at +4°C, pending dispatch to participants.

Verification of Homogeneity

Homogeneity was assessed using procedures described in The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories [15]. A minimum of six randomly selected vials of each test sample were analysed in duplicate using the method to be collaboratively tested.
Results obtained for the verification of homogeneity can be found in Table 3.

Table 3

Homogeneity Test Results of Trial Test Materials

Vial Number	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	(1)	(2)	(1)	(2)
1	218	216	167	165
2	200	198	152	152
3	198	202	150	153
4	204	210	156	159
5	218	206	165	158
6	200	202	147	148
7	202	203	150	149
8	202	203	149	149
9	215	215	158	158
Mean	206.22		154.72	
F-test	0.07		0.75	
F-critical	3.68		3.68	

Trial Proper
Orange Soft Drink sample 231

Vial Number	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	(1)	(2)	(1)	(2)
1	370	367	70	71
2	368	367	70	71
3	369	368	71	70
4	366	367	70	68
5	367	368	69	65
6	365	372	71	69
Mean	367.3		69.58	
F-test	0.258		1.726	
F-critical	4.387		4.387	

Orange Soft Drink sample769

Vial Number	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	(1)	(2)	(1)	(2)
1	399	399	97	91
2	405	400	87	89
3	404	402	90	89
4	404	398	85	88
5	398	398	83	94
6	399	400	83	86
Mean	400.50		88.50	
F-test	1.454		1.373	
F-critical	4.387		4.387	

Cola Soft Drink sample 558

Vial Number	Acesulfame-K (mg/L) (1)		Saccharin (mg/L) (2)	
1	333	331	58	58
2	334	330	58	60
3	328	327	58	58
4	334	328	58	61
5	330	328	58	55
6	332	326	58	58
Mean	330.08		58.17	
F-test	0.851		1.164	
F-critical	4.387		4.387	

Cola Soft Drink sample 307

Vial Number	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	(1)	(2)	(1)	(2)
	335	336	68	69
	335	335	69	69
	337	348	68	71
	335	342	69	71
	343	343	70	71
	344	344	71	71
Mean	339.75		69.75	
F-test	2.200		1.400	
F-critical	4.387		4.387	

Cream sample 897

Vial Number	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	(1)	(2)	(1)	(2)
1	456	415	75	89
2	425	431	68	76
3	436	418	75	71
4	359	389	63	68
5	357	438	61	74
6	415	359	64	58
Mean	408.17		70.17	
F-test	1.225		2.508	
F-critical	4.387		4.387	

Cream sample 190

Vial Number	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	(1)	(2)	(1)	(2)
1	343	344	79	77
2	347	344	79	78
3	335	364	77	83
4	385	343	89	74
5	374	367	85	85
6	410	365	94	85
Mean	360.08		82.08	
F-test	1.510		1.352	
F-critical	4.387		4.387	

Yoghurt sample 456

Vial Number	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	(1)	(2)	(1)	(2)
1	289	273	65	63
2	296	271	68	63
3	273	292	64	65
4	275	277	63	62
5	278	226	61	50
6	279	278	64	68
Mean	275.58		63.00	
F-test	0.856		2.143	
F-critical	4.387		4.387	

Yoghurt sample 644

Vial Number	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	(1)	(2)	(1)	(2)
1	277	273	83	79
2	289	303	83	87
3	296	284	88	85
4	290	287	85	83
5	294	282	84	83
6	290	291	83	87
Mean	288.00		84.17	
F-test	2.292		1.342	
F-critical	4.387		4.387	

Orange Juice samples 353 and 625

Vial Number	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	(1)	(2)	(1)	(2)
1	22	25	18	19
2	24	24	22	21
3	24	23	21	21
4	20	21	17	20
5	22	22	20	20
6	23	24	15	20
Mean	22.83		19.50	
F-test	3.533		1.667	
F-critical	4.387		4.387	

Recovery experiments

Participants were asked to determine recoveries for the analytes spiked into each base material. For the pre-trial sample the spiking levels were 175 mg/L of each analyte. For the main trial, recoveries were determined at two spiking levels as shown in Table 4.

TABLE 4
SPIKING LEVELS USED BY PARTICIPANTS TO DETERMINE RECOVERY IN THE MAIN TRIAL

Matrix	Acesulfame-K	Saccharin
Orange juice	20 and 100 mg/kg	20 and 100 mg/kg
Orange/cola soft drink	150 and 400 mg/L	50 and 100 mg/L
Yoghurt	150 and 400 mg/kg	50 and 120 mg/kg
Cream	150 and 400 mg/kg	50 and 120 mg/kg

The results obtained by participants are given in Table 5, for the pre-trial and Tables 6 and 7 for the main trial.

TABLE 5
RECOVERY DATA:
PRE-TRIAL RECOVERY DATA FROM PARTICIPANTS FOR BASE MATERIAL SPIKED AT 175 mg/L FOR BOTH ANALYTES

Laboratory number	% recovery	
	Acesulfame-K	Saccharin
1	96.5	108.7
2	99.0	97.0
3	85.1	84.5
4	99.3	95.5
5	96.0	95.0
6	79.9	97.1
7	-	-
8	85.0	95.0
9	101.0	105.0
10	99.0	100.0
11	96.8	99.8
12	99.1	100.4

- Participant did not report any data

TABLE 6

RECOVERY DATA: RESULTS OBTAINED BY PARTICIPANTS FOR RECOVERY DATA FOR BASE MATERIALS SPIKED AT VARYING LEVELS FOR ACESULFAME-K IN MAIN TRIAL

Lab No	% recovery									
	Orange soft drink spiking levels (mg/L)		Cola soft drink spiking levels (mg/L)		Cream spiking levels (mg/kg)		Yoghurt spiking levels (mg/kg)		Orange juice spiking levels (mg/kg)	
	150	400	150	400	150	400	150	400	20	100
1	103	102	98	101	94	97	90	87	110	110
	-	-	-	-	-	-	-	-	-	-
2	103	101	105	102	90	90	90	92	132	108
	103	101	105	102	90	90	90	92	132	108
3	108	93.5	108	93.5	97.5	96.5	97.5	96.5	100	82
	108	93.5	108	93.5	97.5	96.5	97.5	96.5	100	82
4	109.8	98.8	102.2	103.4	81.9	82.5	81.2	80.3	100.3	106.3
	107.3	98.8	102.2	103.4	81.9	82.5	81.2	80.3	100.3	106.3
5	97.6	100	80	92.5	84.0	66.1	56	64	68.8	81.5
	97.6	100	80	92.5	45.8	66.1	56	64	68.8	81.5
6	102.2	97.7	103.3	99.1	96.8	95.2	91	80.9	-	-
	-	-	-	-	-	-	-	-	188	111.4
7	-	-	-	-	-	-	-	-	74.2	84
	101.0	101.2	100.3	99.5	97.2	98.1	74	77.2	-	-
8	102	104	106	98	99	99	93	-	95	102
	102	104	106	98	99	99	93	-	95	102
9	103	103	101	104	77	81	75	82	55	68
	103	103	101	104	77	81	75	82	55	68
10	98.9	95.0	88.7	92.6	*	93.7	**	92.3	***	***
	98.9	95.0	88.7	92.6	*	93.7	**	92.3	***	***
11	112	112	112	112	-	-	-	-	-	-
	112	112	112	112	-	-	-	-	-	-
12	112	107	110	107	106	99	92	94	109	103
	112	107	110	107	106	99	92	94	109	103

\- Participant did not report any data.

* Participant reported detector problems which prevented recoveries being submitted.

** Participant reported chromatography problems which prevented recoveries being submitted.

*** Participant reported suspected deterioration of spiked materials and had insufficient time to follow up.

TABLE 7

RECOVERY DATA: RESULTS OBTAINED BY PARTICIPANTS FOR RECOVERY DATA FOR BASE MATERIALS SPIKED AT VARYING LEVELS FOR SACCHARIN IN MAIN TRIAL

Lab No	% recovery									
	Orange soft drink spiking levels (mg/L)		Cola soft drink spiking levels (mg/L)		Cream spiking levels (mg/kg)		Yoghurt spiking levels (mg/kg)		Orange juice spiking levels (mg/kg)	
	50	100	50	100	50	120	50	120	20	100
1	106	105	99	100	100	100	80	78	109	96
	-	-	-	-	-	-	-	-	-	-
2	121	112	101	104	93	93	89	91	113	98
	121	112	101	104	93	93	89	91	113	98
3	84	75	84	75	106	95	106	95	110	90
	84	75	84	75	106	95	106	95	110	90
4	107.9	107.3	103.4	103.7	87.5	107.3	87.6	105.2	102.2	100.7
	107.9	109.8	103.4	103.7	87.5	107.3	87.6	105.2	102.2	100.7
5	97.2	102	54.8	82.6	67.5	45.8	38	49	33.8	52.5
	97.2	102	54.8	82.6	67.5	84	38	49	33.8	52.5
6	117.4	110.8	99	99.8	88.7	101.8	135.2	110.9	-	-
	-	-	-	-	-	-	-	-	39.8	85.5
7	-	-	-	-	-	-	-	-	96.1	98.4
	111.5	107.0	99.7	100.4	95.3	98.9	94.2	98.2	-	-
8	107	107	87	91	87	96	84	-	79	87
	107	107	87	91	87	96	84	-	79	87
9	112	105	96	98	65	81	55	69	21	75
	112	105	96	98	65	81	55	69	21	75
10	103.5	99.1	90.8	94.9	*	111.0	**	106.9	***	***
	103.5	99.1	90.8	94.9	*	111.0	**	106.9	***	***
11	135	130	135	130	-	-	-	-	-	-
	135	130	135	130	-	-	-	-	-	-
12	116	102	100	106	104	102	90	94	107	105
	116	102	100	106	104	104	90	94	107	105

\- Participant did not report any data.

* Participant reported detector problems which prevented recoveries being submitted.

** Participant reported chromatography problems which prevented recoveries being submitted.

*** Participant reported suspected deterioration of spiked materials and had insufficient time to follow up.

RESULTS

The results obtained by the participants for the pre-trial are given in Table 8 and the results for the main trial are given in Tables 9 to 16.

TABLE 8

RESULTS REPORTED FOR PRE-TRIAL SAMPLE ANALYSED IN DUPLICATE

Laboratory No.	Acesulfame-K Concentration (mg/L)		Saccharin Concentration (mg/L)	
	(1)	(2)	(1)	(2)
1	204	205	175	179
2	203	200	153	152
3	180	177	185	186
4	196	196	147	149
5	177	182	141	145
6	153	148	145	142
7	200	198	172	170
8	166	163	163	159
9	210	198	166	158
10	186	189	155	147
11	139(a)	166(a)	129	150
12	212	215	196	188
Mean	189.00		160.55	
r	9		15	
S_r	3.330		5.401	
RSD_r	2		3	
Ho_r	0.4		0.7	
R	54		50	
S_R	19.265		17.862	
RSD_R	10		11	
Ho_R	1.4		1.5	

For key, see Table 16

TABLE 9

RESULTS FOR ORANGE SOFT DRINK SAMPLES 231 AND 769

Lab No	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	Sample 231 fortified at 350 mg/L	Sample 769 fortified at 370 mg/L	Sample 231 fortified at 80 mg/L	Sample 769 fortified at 100 mg/L
1	369	397	86	107
2	362	391	74	93
3	325	350	59(b)	59(b)
4	368	397	72	88
5	316(c)	351(c)	54(c)	72(c)
6	347	376	71	88
7	361	387	74	90
8	377	398	71	85
9	343	383	67	83
10	315	357	66	87
11	380	448	79	97
12	337	398	70	82
Mean	353.09	389.27	73.00	90.00
r	30		6	
S_r	10.857		2.028	
RSD_r	3		2	
Ho_r	0.7		0.5	
R	66		19	
S_R	23.536		6.716	
RSD_R	6		8	
Ho_R	1.0		1.0	

For key, see Table 16

TABLE 10

RESULTS FOR COLA SAMPLES 558 AND 307

Lab No	Acesulfame-K (mg/L)		Saccharin (mg/L)	
	Sample 558 fortified at 330 mg/L	Sample 307 fortified at 350 mg/L	Sample 558 fortified at 75 mg/L	Sample 307 fortified at 95 mg/L
1	340	358	71	87
2	347	367	62	76
3	329	334	36	43
4	350	365	59	72
5	293(c)	316(c)	43(c)	56(c)
6	336	351	58	72
7	347	365	61	75
8	374	389	53	66
9	309	350	51	69
10	322	321	56	69
11	366	384	71	84
12	358	376	60	76
Mean	343.46	360.00	58.00	71.73
r	20		5	
S_r	7.303		1.952	
RSD_r	2		3	
Ho_r	0.5		0.5	
R	55		30	
S_R	19.747		10.569	
RSD_R	6		16	
Ho_R	0.8		1.9	

For key, see Table 16

TABLE 11

RESULTS FOR CREAM SAMPLES 897 AND 190

Lab No	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	Sample 897 fortified at 380 mg/kg	Sample 190 fortified at 358 mg/kg	Sample 897 fortified at 84 mg/kg	Sample 190 fortified at 104 mg/kg
1	365	342	74	94
2	350	333	62	77
3	400	367	51	65
4	258	238	60	74
5	247(c)	222(c)	41(c)	41(c)
6	309	288	58	62
7	278(b)	335(b)	58	75
8	513(b)	359(b)	87	82
9	292	279	48	63
10	357	350	73	83
11	-	-	-	-
12	271	251	50	71
Mean	325.25	306.00	62.10	74.60
r	15		15	
S_r	5.355		5.530	
RSD_r	2		8	
Ho_r	0.4		1.4	
R	138		32	
S_R	49.286		11.287	
RSD_R	16		17	
Ho_R	2.3		1.9	

For key, see Table 16

TABLE 12

RESULTS FOR YOGHURT SAMPLES 456 AND 644

Lab No	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	Sample 456 fortified at 300 mg/kg	Sample 644 fortified at 325 mg/kg	Sample 456 fortified at 80 mg/kg	Sample 644 fortified at 120 mg/kg
1	277	290	56	87
2	275	298	63	92
3	309	338	57	69
4	197	209	65	81
5	164(c)	187(c)	27(c)	40(c)
6	237	255	79	101
7	266	300	54	85
8	282	326	58	92
9	207	226	39	57
10	311	292	98	90
11	-	-	-	-
12	188	190	39	66
Mean	254.90	272.40	60.80	82.00
r	35		25	
S_r	12.388		8.899	
RSD_r	5		12	
Ho_r	1.0		2.2	
R	133		44	
S_R	47.621		15.793	
RSD_R	18		22	
Ho_R	2.6		2.6	

For key, see Table 16

TABLE 13

RESULTS FOR ORANGE JUICE SAMPLES 353 AND 625

Lab No	Acesulfame-K (mg/kg)		Saccharin (mg/kg)	
	Sample 353 fortified at 20 mg/kg	Sample 625 fortified at 20 mg/kg	Sample 353 fortified at 25 mg/kg	Sample 625 fortified at 25 mg/kg
1	49(a)	17(a)	29	22
2	27	23	22	22
3	27	26	19	18
4	38	40	17	19
5	47(c)	14(c)	-(c)	8(c)
6	40	40	11	9
7	<25(d)	<25(d)	<25(d)	<25(d)
8	21	16	18	13
9	11	11	4	4
10	168(a)	57(a)	85(a)	47(a)
11	-	-	-	-
12	11	9	15	15
Mean	24.29		16.06	
r	6		6	
S_r	1.890		2.278	
RSD_r	8		14	
Ho_r	1.2		2.0	
R	34		19	
S_R	12.204		6.927	
RSD_R	50		43	
Ho_R	5.1		4.1	

For key, see Table 16

TABLE 14

SUMMARY OF CALCULATED STATISTICAL PARAMETERS FOR ACESULFAME-K IN MAIN TRIAL SAMPLES

sample type	fortification level	mean (obs)	n	r	S_r	RSD_r	Ho_r	R	S_R	RSD_R	Ho_R
orange soft drink	350 mg/L 370 mg/L	353.09 389.27	11	30	10.857	3	0.7	66	23.536	6	1.0
cola soft drink	330 mg/L 350 mg/L	343.46 360.00	11	20	7.303	2	0.5	55	19.747	6	0.8
cream	380 mg/kg 358 mg/kg	325.25 306.00	8	15	5.355	2	0.4	138	49.286	16	2.3
yoghurt	300 mg/kg 325 mg/kg	254.90 272.40	10	35	12.388	5	1.0	133	47.621	18	2.6
orange juice	20 mg/kg	24.29	7	6	1.890	8	1.2	34	12.204	50	5.1

For key, see Table 16

TABLE 15

SUMMARY OF CALCULATED STATISTICAL PARAMETERS FOR SACCHARIN IN MAIN TRIAL SAMPLES

sample type	fortification level	mean (obs)	n	r	S_r	RSD_r	Ho_r	R	S_R	RSD_R	Ho_R
orange soft drink	80 mg/L 100 mg/L	73.00 90.00	10	6	2.028	2	0.5	19	6.716	8	1.0
cola soft drink	75 mg/L 95 mg/L	58.00 71.73	11	5	1.952	3	0.5	30	10.569	16	1.9
cream	84 mg/kg 104 mg/kg	62.10 74.60	10	15	5.530	8	1.4	32	11.287	17	1.9
yoghurt	80 mg/kg 120 mg/kg	60.80 82.00	10	25	8.899	12	2.2	44	15.793	22	2.6
orange juice	25 mg/kg	16.06	8	6	2.278	14	2.0	19	6.927	43	4.1

For key, see Table 16

TABLE 16

KEY TO TABLES 8 TO 15

-	Participant did not report any data
(a)	An outlying result as determined by Cochran's Test at P<0.01 level, not used in calculation of statistical parameters.
(b)	An outlying result as determined by Grubb's Test
(c)	Result excluded due to participant reporting chromatographic problems and a majority of recoveries outside the range prescribed in the method.
(d)	Result not included in statistical analysis.
obs.	The observed mean, the mean obtained from the collaborative trial data.
r	Repeatability (within laboratory variation). The value below which the absolute difference between two single test results obtained with the same method on identical test material under the **same conditions** may be expected to lie with 95% probability.
S_r	The standard deviation of the repeatability.
RSD_r	The relative standard deviation of the repeatability ($S_r \times 100/MEAN$).
Ho_r	The HORRAT value for repeatability is the observed RSD_r divided by the RSD_r value estimated from the Horwitz equation using the assumption r = 0.66R.
R	Reproducibility (between-lab variation). The value below which the absolute difference between two single test results obtained with the same method on the identical test material under **different conditions** may be expected to lie with 95% probability.
S_R	The standard deviation of the reproducibility.
RSD_R	The relative standard deviation of the reproducibility ($S_R \times 100/MEAN$).
Ho_R	The HORRAT value for reproducibility is the observed RSD_R value divided by the RSD_R value calculated from the Horwitz equation.

Statistical analysis of results

The trial results were examined for evidence of individual systematic error ($p<0.01$) using Cochran's and Grubbs' test progressively, by procedures described in the internationally agreed Protocol for the Design, Conduct and Interpretation of Collaborative Studies[16].

Repeatability and Reproducibility

Calculations for repeatability (r) and reproducibility (R) as defined by that protocol[16] were carried out on those results remaining after removal of outliers. These are given in Tables 8 to 13 and summarised in Tables 14 and 15.

Horwitz Predicted Precision Parameters

There is often no validated reference or statutory method with which to compare precision criteria when assessing a new method. In such cases it is useful to compare the precision data obtained from a collaborative trial with predicted acceptable levels of precision. These levels, as predicted by the Horwitz equation, give an indication as to whether the method is sufficiently precise for the level of analyte being measured[17].

The Horwitz predicted value is calculated from the Horwitz equation[17]:

$$RSD_R = 2^{(1-0.5\log C)}$$

C is the measured concentration of analyte expressed as a decimal, i.e. 1 g/100g = 0.01

Horrat Value (Ho)

The Horrat[18] value gives a comparison of the actual precision measured with the precision predicted by the Horwitz equation for a method measuring at that particular level of analyte. It is calculated as follows:

$$Ho_R = RSD_R \text{ (measured)}/RSD_R \text{ (Horwitz)}$$

A Ho_R value of 1 usually indicates satisfactory interlaboratory precision, while a value of >2 indicates unsatisfactory precision i.e. one that is too variable for most analytical purposes or where the variation obtained is greater than that expected for the type of method employed. Similarly Ho_r is calculated, and used to assess intralaboratory precision, using the approximation RSD_r (Horwitz) = $0.66RSD_R$ (Horwitz). (This assumes the approximation $r = 0.66R$) The Horwitz values calculated from the results of this trial are given in Tables 8 to 15.

DISCUSSION

The results from the pre-trial demonstrated that laboratories were performing the analysis satisfactorily and so participants were advised to proceed to the analysis of the trial samples proper.

The statistical results reveal that the method gave satisfactory precision for the split level samples at the higher sweetener concentrations (276-408 mg/kg for acesulfame-K and 58-89 mg/L for saccharin) representative of the limits set for the analytes in EC Directive 94/35. The values of the within-laboratory precision parameter, the repeatability (r), and the between-laboratory precision parameter, the reproducibility (R), calculated from the experimental data for both analytes at these levels were near to the values predicted by the Horwitz equation.

For samples of orange juice, not permitted to contain added sweeteners, the precision of the method was evaluated using blind duplicate samples at low sweetener concentrations in order to assess the ability of the method to reliably detect low level addition of these compounds. In this case the Ho_r and Ho_R values, 7 and 5 for acesulfame-K and 2 and 4 for saccharin respectively probably reflect the performance of the method near to its detection limit, estimated to be 15 mg/L.

For the soft drink samples analysed, the performance characteristics of the method trialled here are comparable to those from an earlier trial of a method for the analysis of soda beverages which utilised acetate buffer mobile phase in conjunction with reversed-phase high-performance liquid chromatography, and which was adopted by the AOAC[8]. In the more recent trials conducted by CEN, phosphate buffer mobile phase has been used. For all the food matrices included in this collaborative trial the precision of the method is generally better than that obtained using either of the procedures that were assessed by collaborative trial by CEN[13,14]. The present method bears similarities to these earlier CEN methods in that the method used a phosphate buffer mobile phase; however experimental conditions were modified to allow 20 mM buffer to be used at pH 3.5. The validity of the present approach was verified by in-house assessment of the candidate method at the Food Science Laboratory, prior to conducting the trial.

The present method is considerably more precise in its performance, particularly as regards saccharin, than that of a method, based on ion-pair hplc, which was previously assessed by collaborative trial by MAFF in 1991. One problem which was highlighted by the results of that trial was the need to

accurately determine the water content of the sodium saccharin standard material. Sodium saccharin is efflorescent, theoretically its water content should be 15 % for the dihydrate but is thought to vary in practice between 3-15 %. The drying conditions used by participants are given in Table 17 together with the percentage of moisture found.

Table 17

DRYING CONDITIONS USED BY PARTICIPANTS FOR SODIUM SACCHARIN SOLID MATERIAL

Laboratory Number	Temperature Used (°C)	Time Used (hrs)	Percentage Water Calculated
1	103	6	6.9
2	102±2	6	12.7
3	102	6	12.5
4	102	8	5.57
5	100	6	9.7
6	101±2	6	6.86
7	103±2	6	5.53
8	102	4	5.5
9	102±2	5	0.23
10	105±2	Overnight	-
11	103±2	4	12.1
12	102	3	6.05 w/w

Of the AOAC methods only the polarographic method[3] stipulates the need to correct for water content by measuring the water of crystallisation, this being by Karl Fischer titration. In the present study, participants were asked to correct for the water content of the standard material after heating to constant mass and also to report the E 1% 1 cm value for the sodium saccharin standard. The data supplied by participants showed that the percentage water content could vary widely and making satisfactory allowance for this in the present trial may contribute to the better performance characteristics now obtained. The E 1% 1 cm value which participants reported for the purity of their sodium saccharin standard as an alternative means of calculating the moisture content showed large variation and further definition of the procedure is needed before this could be considered for inclusion in the method. One laboratory reported assaying the nitrogen content of its saccharin standard to determine purity, while another questioned whether free saccharin, which does not contain water

of crystallisation would give more consistent results; however solubility problems with the free acid are generally thought to mitigate against the use of this material.

Of the twelve laboratories who agreed to take part in the study, one laboratory only reported results for the soft drink samples.

Although the need for the use of a solid phase extraction cartridge to clean-up soft drink samples was queried, it was found that this approach removed much of the colour from these samples. The earlier trial conducted for soda beverages[8] involved direct injection of the degassed, filtered sample but the method was claimed to suffer from interferences arising from the presence of artificial colours and sorbates. Other problems raised by participants related to the use of a gradient step in the method and the potential interference problems due to aspartame breakdown products. One difficulty in particular with analysing in a single run three of the more common sweeteners, acesulfame-K, saccharin and aspartame, currently permitted for use in foodstuffs in the United Kingdom, is that aspartame is unstable and its breakdown products phenylalanine and aspartylphenylalanine can co-elute with saccharin and acesulfame-K during reversed-phase hplc. Although conditions giving baseline resolution of these compounds have been reported by Prodolliet et al [12], such resolution could not be attained during method evaluation at the Food Science Laboratory. A decision was therefore taken to exclude samples containing aspartame from the present study in order not to compromise the performance characteristics of acesulfame-K and saccharin by the possible presence of these co-eluting breakdown products when a single wavelength 220 nm is used for detection. Nevertheless, since these breakdown products make only a small contribution to the acesulfame-K and saccharin peak response, the method could probably be used as a screening procedure at this wavelength for the analysis of foodstuffs containing aspartame, with quantitation of the compounds being carried out at alternative wavelengths e.g. 200 or 230 nm. The use of isocratic elution as described in methods previously assessed by collaborative trial and also as reported in the paper by Prodolliet, was additionally found during the in-house evaluation to be prone to 'ghost peaks' appearing in the chromatograms. These 'ghost peaks' arose in the chromatograms of subsequent runs from components retained on the hplc column even after sufficient time had elapsed for co-extracted preservatives such as benzoic acid to be eluted. For this reason the method used in this trial included a gradient step operating following the elution of the analytes of interest, although the conditions were modified slightly before the trial proper to meet certain concerns of participants. Nevertheless one participant elected to use isocratic conditions throughout.

Recoveries reported by participants in this trial are given in Tables 5 to 7 and were generally similar to those obtained by the co-ordinating laboratory during the in-house validation work both with this method and during LC-MS confirmation studies. Poor recoveries reported for low concentration spikes appeared to be due to the spiking levels being at or near the detection limit. Laboratory number 5 reported problems with changes in retention times between samples and this, together with the fact that the majority of their recoveries were outside the range prescribed in the method led to the decision not to include their results in the subsequent statistical evaluation.

Further work is required before the need for a simple isocratic method capable of simultaneously determining all permitted sweeteners can be met. Continuing improvements in chromatographic technologies such as columns which have recently become available based on inert silica may, as suggested, prove to give the selectivity needed to reliably separate acesulfame-K and saccharin from the breakdown products of aspartame, enabling the quantitation of these compounds with acceptable precision. However, future investigations should also assess the routine use of photodiode array detectors, capable of monitoring two or more wavelengths, for extending the range of sweeteners which can be determined, especially since it is anticipated that other compounds with intense sweetening action such as alitame and sucralose may be approved for use in foodstuffs in the future.

CONCLUSION

The precision obtained for the method tested in this trial was acceptable for determining acesulfame-K and saccharin at the maximum concentrations specified in The Sweeteners in Food Regulations 1995 for their use in foodstuffs. The procedure will be published as a MAFF validated method.

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APPENDIX I
DETERMINATION OF ACESULFAME-K AND SACCHARIN IN FOODSTUFFS

1. SCOPE AND FIELD OF APPLICATION

The method allows for the determination of acesulfame-K and saccharin in the following foodstuffs: soft drinks, fruit juices, cream, yoghurt, biscuits, jam, chocolate and custard powder. It is not suitable for the analysis of foodstuffs which contain high levels of aspartame breakdown products.

2. PRINCIPLE

Aqueous extraction of the sample, clean-up on a solid phase extraction column or with clarification reagents. Chromatography on a reversed phase HPLC column and photometric detection at 220 nm.

3. REAGENTS

Use only reagents of recognised analytical grade for the HPLC analysis. The water used should be distilled water, or water of at least equivalent purity.

3.1 Acetonitrile, for HPLC, filtered through a 0.45 μm filter (**4.14**) before use.

3.2 Potassium dihydrogenphosphate (KH_2PO_4).

3.3 Orthophosphoric acid (H_3PO_4), (1.75 S.G).

3.3.1 20 mM orthophosphoric acid solution, prepared as follows:

Weigh out 1.96 g of orthophosphoric acid (**3.3**), carefully dissolve in water, mix well and make the volume of solution up to the mark in a 1 L volumetric flask.

3.4 Clarification solution No 1, prepared by dissolving 3.6 g potassium hexacyanoferrate (II) ($K_4[Fe(CN)_6] \cdot 3\ H_2O$) in water, mixing well and making up to 100 mL.

3.5 Clarification solution No 2, prepared by dissolving 7.2 g zinc sulphate ($ZnSO_4 \cdot 7\ H_20$) in water, mixing well and making up to 100mL.

3.6 Phosphate buffer solution, 20 mM at pH 3.5, prepared as follows:

Weigh out 2.72 g of potassium dihydrogenphosphate (**3.2**) and place in a 1 L beaker with 500 mL of water. Stir using a magnetic stirrer until all the solid has dissolved. Adjust the pH of this solution to pH 3.5 by the addition of orthophosphoric acid solution (**3.3.1**). Transfer the contents of the beaker to a 1 L volumetric flask and make up to volume using distilled water. Pass the phosphate buffer solution through a 0.45 µm filter before use.

3.7 HPLC mobile phase A, acetonitrile/phosphate buffer (5:95, v/v).

To prepare 1 L of mobile phase A combine 50 mL of acetonitrile (3.1) and 950 mL of phosphate buffer solution (**3.6**). Mix thoroughly. Degas the mobile phase thoroughly before use, e.g. by sonication in an ultrasonic bath for approximately 5 min. Prepare freshly on the day of use.

3.8 HPLC mobile phase B, acetonitrile/phosphate buffer (50:50, v/v).

To prepare 1 L of mobile phase B combine 500 mL of acetonitrile (**3.1**) and 500 mL of phosphate buffer solution (**3.6**). Mix thoroughly. Degas the mobile phase thoroughly before use by sonication in an ultrasonic bath for approximately 5 min. Prepare freshly on the day of use.

Note 1: In order to avoid corrosion problems due to prolonged contact with phosphate-containing eluents and as a precaution against blockages occurring due to precipitation of phosphate, water should be pumped through the HPLC equipment after carrying out this method.

3.9 Acesulfame-K - crystalline (e.g. Hoechst).

3.10 Sodium saccharin - crystalline (e.g. Sigma).

3.10.1 Determination of drying loss of sodium saccharin (to correct for water content).

Weigh approximately 1 g, to the nearest 0.1 mg, of finely ground sodium saccharin (**3.10**). Dry this amount at 100°C to 105°C for 4 h then remove from the oven, cool to room temperature in a desiccator and determine the drying loss by weighing to the nearest 0.1 mg. Repeat the heating, cooling and weighing operations until the results of two successive weighings, separated by 1 h of heating, do not differ by more than 1 mg.

3.11 Standard solutions

3.11.1 Prepare a combined standard solution (1000 mg/L) by weighing, to the nearest 0.1 mg, 250 mg acesulfame-K (3.9) and 250 mg sodium saccharin (**3.10**). Transfer to a 250 mL volumetric flask. Dissolve, mix well and make up to the mark with water.

Note 2: It is not necessary to use the dried standard in the preparation of the standard solutions, however account should be taken of the moisture content when calculating the concentration of anhydrous saccharin in the sample.

3.11.2 Using a burette or pipettes measure 0.1, 1.0, 2.0, 5.0, 10.0, 20.0, 30.0 and 40.0 mL of the combined standard solution (**3.11.1**) into separate 100 mL volumetric flasks, make each up to the calibration mark with water and mix well. The concentrations of these solutions are respectively 1, 10, 20, 50, 100, 200, 300 and 400 mg/L of both acesulfame-K and sodium saccharin.

4. APPARATUS

Normal laboratory apparatus and in particular the following:

4.1 Analytical balance.

4.2 High speed blender, or homogenizer.

4.3 Volumetric flasks 1000 mL, 250 mL, 100 mL and 25 mL.

4.4 Conical flask, 100 mL and 25 mL.

4.5 Beaker, 1000 mL.

4.6 Burette, 100 mL or pipettes, 100 mL, 40 mL, 30 mL, 25 mL, 20 mL, 10 mL, 5 mL and 1 mL.

4.7 Micropipette, 1000 μL.

4.8 Graduated cylinders, 1000 mL and 500 mL.

4.9 Funnel (e.g. 75 mm).

4.10 Fluted filter papers, medium fast, qualitative (e.g. Whatman No. 4, 150 mm).

4.11 Thermostatted water bath.

4.12 Ultrasonic bath.

4.13 Degassing system, for solvents.

4.14 Membrane filters, polyvinylidene fluoride, pore size 0.45 μm.

4.15 Filter holder for membrane filters.

4.16 10 mL disposable syringe.

4.17 Syringe filters, PTFE, 25 mm, pore size 0.45 μm.

4.18 Solid phase extraction cartridges (C 18), Mega Bond Elut, 6 cc, 1 g.

4.19 Desiccator, containing an efficient desiccant (e.g. self-indicating silica gel).

4.20 High performance liquid chromatograph, capable of gradient operation, consisting of a UV detector and a recorder or integrator which allows the measurement of peak heights and peak areas.

The following conditions have been found to be satisfactory:

Guard column	Partisil 10 ODS 3, C18 10 μm cartridge (10 × 3.2 mm) and holder
Column	Partisil 10 ODS 3, C18 10 μm 25 × 4.6 cm 10.5 % carbon loading
Mobile phase A	Acetonitrile/phosphate buffer (5:95, v/v)
Mobile phase B	Acetonitrile/phosphate buffer (50:50, v/v)
Elution programme	100 % A at 0 min 100 % A at 11 min 100 % B at 12 min 100 % B at 17 min 100 % A at 18 min to 25 min to achieve re-equilibraton
Flow rate	1.3 mL/min
Injection volume	20 μL
Column temperature	ambient
Detector (UV) wavelength	220 nm

Note 3: Gradient analysis is required in order to remove co-extracted materials (e.g. sorbic acid) which may otherwise elute much later in the isocratic run or remain on the column to elute in subsequent runs. Under these conditions, acesulfame-K and sodium saccharin should be baseline resolved from neighbouring peaks and elute after 5.7 min and 10 min respectively. Analysis at room temperature has been found to be satisfactory. Equivalent analytical columns may be used provided that baseline resolution is achieved for the compounds of interest.(resolution = 1.3). A typical chromatogram is shown as Figure 1.

Note 4: Depending on the chromatographic conditions employed, it may be found that sodium saccharin elutes later than 11 min. Under these circumstances isocratic conditions, using mobile phase A, should be continued beyond 11 min until both analytes have been eluted.

5. PROCEDURE

5.1 Preparation of the sample test solution.

5.1.1. Soft drinks (eg lemonades, cola, beverages). Degas the sample and dilute a 5 mL aliquot to 10 mL in a volumetric flask with water. Pass through an unconditioned C18 solid phase extraction cartridge at approximately 2 mL/min. Pass solution through a syringe filter of pore size 0.45 μm (**4.17**) before injection.

5.1.2 Fruit juices.

Shake the sample, weigh about 1 g to the nearest 0.1 mg into a 25 mL conical flask and add 10 mL water. Incubate in a water bath at 60°C for 15 minutes. Transfer to a 25 mL volumetric flask and allow to cool to room temperature. Add 5 mL clarification solution No 1 (**3.4**) and shake Add 5 mL clarification solution No 2 (**3.5**) and shake vigorously. Make up to the mark with water. Filter through a fluted filter paper. Pass the filtrate through a syringe filter of pore size 0.45 μm (**4.17**) before injection.

5.1.3 Yoghurt products, cream and milk products.

Weigh about 1 g of the thoroughly homogenised sample, to the nearest 0.1 mg, into a 25 mL conical flask. Add about 10 mL water and place the conical flask in a water bath at 60°C for 15 min. Transfer the solution to a 25 mL volumetric flask and allow to cool to room temperature. Add 5 mL of clarification solution No 1 (**3.4**) and shake. Add 5 mL of clarification solution No 2 (**3.5**) and shake vigorously. Make up to the mark with water, and filter the solution through a fluted filter paper. Pass the filtrate through a syringe filter of pore size 0.45 μm (**4.17**) before injection.

5.1.4 Jam, custard powder, biscuits and chocolate.

Weigh about 1 g of the thoroughly homogenised sample, to the nearest 0.1 mg, into a 100 mL conical flask. Add about 80 mL water and place the conical flask in a water bath at 60°C for 15 min. Transfer the solution to a 100 mL volumetric flask and allow to cool to room

temperature. Add 5 mL of clarification solution No 1 (**3.4**) and shake. Add 5 mL of clarification solution No 2 (**3.5**) and shake vigorously. Make up to the mark with water, and filter the solution through a fluted filter paper. Pass the filtrate through a syringe filter of pore size 0.45 μm (**4.17**) before injection.

5.2 Identification

Inject 20 μL aliquots of the sample extracts onto the HPLC system and identify the intense sweeteners in the sample solution by comparing the retention times of the analyte concerned in the sample test solution with that of the standard substance.

Note 5: The identity of the peaks of interest can be checked for interferences either by using a diode array detector or by carrying out the measurement at another wavelength (e.g. 200 or 230 nm).

5.3 Preparation of calibration graphs

Inject 20 μL of each of the standard solutions (**3.11.2**). Plot the peak area obtained for acesulfame-K and saccharin in each standard solution on the vertical axis versus the corresponding analyte concentration in mg/L along the horizontal axis

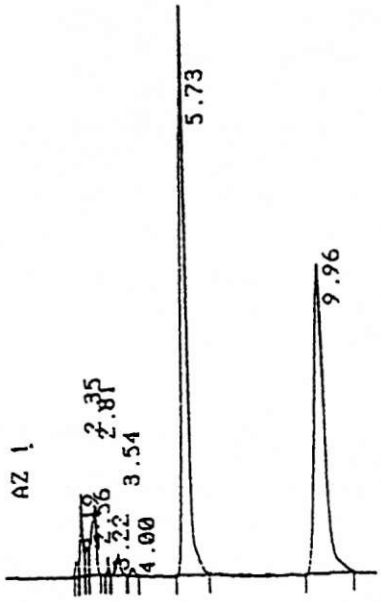
6 CALCULATION

Determine the mean value of two peak areas for each analyte obtained from two 20 μL injections made for each sample extract. Using this mean value obtain from the calibration graph (**5.3**) the concentration of acesulfame-K and saccharin in the extract. Hence calculate for the intense sweetener concerned the mass fraction expressed in mg/kg or the mass concentration in mg/L in the original sample. The saccharin concentration should be corrected for the moisture content of the calibration standard (determined at **3.10.1**).

Note 6: For samples containing the analytes at low concentration, near to the detection limit, measurement at the extreme of the calibration curve may not give sufficient accuracy. In this situation it is possible to reinject the sample extract and quantify the analyte concentrations by peak height response after also injecting a suitable standard. The wavelength of 220 nm is chosen to allow the simultaneous determination of both analytes. However, at low

concentrations, and where interferences from aspartame breakdown products are present, sensitivity and accuracy can be improved by measuring each compound at the wavelength of maximum absorption.

Figure 1.
Liquid chromatogram of acesulfame-K (5.73 min) and saccharin (9.96 min) in pre-trial sample extract. HPLC conditions as 4.20.



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Annual Report of the Council of the Association of Public Analysts for 1995

presented by the Honorary Secretary, Dr. Peter Clare
at the 43rd Annual General Meeting of the Association
at the Strand Palace Hotel, London on the 1st June 1996

This report is concerned with the activities of the Association of Public Analysts (APA) for the year ending December 31st 1995 and discusses events that influence the development of the Association and its members. Public Analysts are qualified scientists who are officially appointed by Local Government Authorities to protect the public in relation to food and other materials. The Council of the Association has met on five occasions during 1995. The composition of the Council is set out in the Articles of Association of the APA and sixteen Public Analysts sit on this central body, all members being subject to election at the Annual General Meeting. Council is responsible for the co-ordination of Activities of the Association including the provision of representatives to outside bodies.

Public Analysts' Laboratories

31 Public Analysts' Laboratories are situated throughout the United Kingdom and are placed in both the private sector and the public sector of the British economy, a characteristic paralleled in many other professions. In addition members' laboratories are placed overseas in Eire, Australia, the Isle of Man and the Channel Isles. The laboratories are concerned with the scientific analysis and microbiological examination of foods, waters, environmental matters and many other topics. The laboratories in the UK have a common feature in that the most senior analytical scientists of each laboratory possess the statutory qualification Mastership in Chemical Analysis (MChemA). This is an award by examination of the Royal Society of Chemistry (RSC) and is recognised in Council Directive 89/48/EEC which is concerned with higher educational diplomas relating to professional activities. Typically a Public Analysts' laboratory has a staff of between 15 and 40 and is well equipped to deal with not only the predictable but also the unexpected.

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Food Authenticity

The Food Industry in the United Kingdom is a massive £90 billion business taking in farming, horticulture, manufacturing, catering and retailing. Protecting the safety and quality of food for the welfare of the consumer and the benefit of the fair trader has been the prime objective of Public Analysts for well over a century through the analysis and examination of foodstuffs and the subsequent interpretation of the results of this activity.

A regular feature during recent years has been a co-ordinated sampling and analysis programme of foods throughout the EC. In the UK this is organised by LACOTS, the Local Authorities Co-ordinating Body on Food and Trading Standards. In 1995 two foods, honey and instant coffee, have received this special scrutiny. Public Analysts' laboratories have been able to provide the analytical expertise necessary to carry out the investigations into the authenticity of the claimed geographic and botanical identities concerned with the description of honey and of the adulteration of instant coffees with other plant soluble components such as extracts of stalks and even glucose compounds. One sample of "instant coffee was found to consist for example of a mixture 40% of instant coffee and 60% of glucose compounds the deficiency in caffeine naturally associated with instant coffee being adjusted back to the level normally expected in an instant coffee by further addition of caffeine". These studies have recognised and made use of expertise specific to individual laboratories and consequently samples have been "passed on" within the terms of section 30 of the Food Safety Act 1990 to appropriately experienced Public Analysts. This administrative procedure has also been employed for the detection of unlabelled and yet irradiated foods such as herbs, spices and prawns.

During the year as a part of their statutory duties under the Food Safety Act 1990 Public Analysts have reported

a) the use of homogenised chicken skin with added water and soya protein being sold as shaped chicken breast.

b) the addition of excess rind emulsions to comminuted meat products such as burgers to "boost" the meat content of the product.

c) the incorporation of water into scampi flesh, a study that itself has resulted in the matter being raised at the Ministry of Agriculture, Fisheries and Food, (MAFF), consumer

panel group and the Royal Society of Chemistry initiating a MAFF supported study on the composition of scampi flesh.

d) the addition of excessive quantities of synthentic colouring agents to Ethnic foods such as Tandoori chicken.

e) the appearance of "novel" foods into the market

f) the presence of potentially dangerous levels of the drug ephedrine in fitness drinks.

European Issues

In February the European Commission Foodstuffs Inspection Team evaluated and subsequently reported on the Food Control System of the United Kingdom pursuant to Article 5 of Directive 93/99/EEC. The central objective of the assessment was the monitoring and evaluation of the equivalence and effectiveness of official food control systems operated by member states. Such monitoring is of crucial importance for protection of all consumers. In a full programme the team were able to visit the Colchester Environmental Health Department and the Essex Trading Standards Department and to hear presentations from officials representing MAFF, the Scottish Office, and the Department of Health. The President of the Association, Mr.Malvern Barnett was invited to explain the role of Public Analysts and Official Laboratories. In their report the Inspection Team cited the World Health Organisation (WHO) recommended sampling rate of 2.5 per thousand head of population and commented that "The Public Analyst is central in the frame of food control".

During the year the Ministry requested our views on standards appropriate for official food control laboratories and methods of analysis and examination. In a detailed paper the concepts of performance assessment and accreditation of laboratories were set out, the latter being based both on a combination of generic scientific principles and on a specific and detailed analytical methods. It is essential that an official laboratory has a comprehensive ability in order to deal competently with the wide range of problems it is likely to encounter and also have sufficient ability to recognise and pursue developments in food technology, often as yet unknown to enforcement scientists.

The Association has also commented on proposals for a code of practice which will specify the qualification requirements for officers authorised by the Food Safety Act for food inspection. Whatever the final content of this code of practice it is essential that the inspectors and scientists both

act and are seen to act as a team in order that the strengths of all available disciplines are available for this important aspect of public protection.

Deregulation and food standards

A Government initiative has during the year been centred on the concept of deregulation of food standards. In fact deregulation of standards has been occurring not only in 1995 but also in previous years. Potted meat, many canned meat products, mustard flour, tomato sauce and ketchup and aspects of the composition of soft drinks have all had statutory minimum standards removed. During December soft drinks, aspects of flour and bread and certain types of cheese had minimum compositional standards removed.

Where deregulation leads to an increase in informed consumer choice this can only be regarded as of benefit to a purchaser. However many names of foods that are regarded as implying a traditional quality and composition for food are likely to come under pressure. Since the removal of a standard for potted meat for example the meat content of the product has tumbled from over 95% to around 85%. Today shepherds pie on retail sale has been found containing less than 10% meat. Many soft drinks, such as lemonade, have the same composition as low calorie soft drinks being totally free from natural sugar sweeteners. The only difference between the two soft drinks is that the one labelled low calorie also declares that it can aid slimming only as a part of a calorie controlled diet. It is therefore quite likely that the names of foods that are traditional will become associated with foods that are of a modern construction and so of a different composition to that which may be expected from the name. For example many breadcrumb coated fish products now have a centre which is a product made from fish in combination with one or more other ingredients such as water, cereal, emulsifiers, and phosphates and yet they are described as coated fish. Meat that has been merely cooked at a low temperature and then flavoured is described as roast, even though the meat itself has not been roasted. The anticipated quantitative declaration of ingredients may provide some protection here, however the Association is firmly of the view that a traditional name has a meaning which must be protected. For example the name cod means 100% fish - it cannot be modified by a quantitaive declaration that the food described as cod only contains 75% of fish.

Training

The latest in the series of Training Guides prepared by the Association's training committee was published during the year. This publication was entitled "Certificate Writing" and it is a document that deals with the development, through case law, of the Public Analyst's Certificate, and provides worked examples chosen to illustrate the full range of situations to be expected from the variety of foodstuffs analysed and examined in laboratories and the necessary and appropriate interpretations of these scientific results. The guide includes certificates typical of those issued as a result of laboratory work on foods containing foreign bodies or otherwise contaminated, foods that are putrid, foods that are compositionally deficient and foods that are incorrectly labelled. This publication represents a major source of information and application. It will be seen as a worthy successor to previous papers on the subject written by former Public Analysts Hamence and Moir.

Training seminars were organised by the Training Committee of the Association on "Food Additives" in March and on "Plastic Materials and Articles in contact with Food" in December, and the Annual Training School of the Association was held at Reading University in April .

The UK section of the European Food Law Association held a meeting in March on the subject of "Deregulation and Quantitative Declaration of Ingredients (QUID) as applied to Food Law".

Annual Conference and Exhibition 1995

The Annual Conference and Exhibition was held in April at the Stakis Hotel, Northampton and took the theme of "Aspects of Control in the Modern Food Enforcement System".

Protecting the safety and quality of food for the welfare of the consumer and for the benefit of the fair trader have been prime objectives of Public Analysts for well over a century and UK legislation in the form of the Food Safety Act 1990, and the European Food Control and Additional Measures Directives (EEC 89/397 and EEC 93/99) reinforce the scientific basis for food enforcement throughout the UK. The conference looked at the various disciplines involved in food enforcement. Professor Burke, Chairman of the Advisory Committee on Novel Foods and Processes explained the procedures for the initial screening and assessment of new foods including irradiated foods and genetically modified foods. Diane McCrea of the Consumers' Association related the

complexity of modern day food production with a requirement for increased protection of the consumer. Charles Cockbill of the the Ministry of Agriculture Fisheries and Food and Nick Cull, Chairman of the LACOTS Quality Standards Panel, explained the obligations imposed on official laboratories and the official inspectorate necessary to achieve this protection. They emphasised the need for a co-ordinated approach to enforcement by local authorities. Peter Farnell of the Leatherhead Food Research Association presented a paper which illustrated the science including interpretative skills necessary to explore food authenticity topically taking the fraught problem of purity of orange juice as a worked example.

Anthony Harrison OBE, Past President of the Association spoke on the role of FLEP, the Food Law Enforcement Practitioners and the systems of food control activities in other EC countries. The facts presented made for an interesting comparison. Within the European Union the number of laboratories per million population varies from the UK's 0.7 to Finland's 12.6. Not only was it reported that the UK had the lowest number of laboratories it also had an exceptionally low number of samples being taken for analysis and examination.

Guest of Honour at the Annual Dinner was the Lord Dainton, who together with Councillor Bill Mackay, former Chairman of LACOTS, were appointed Honorary Vice Presidents of the Association. In addition Alan Turner OBE, Honorary Member of the Association, and three former Presidents of the Association, Anthony Harrison OBE, Wilfred Cassidy MBE and Phillip Hall, were presented with medals in the form of miniatures of the President's badge of office to commemorate their service to the APA.

The annual dinner itself was centered on guinea fowl which was cooked with wild thyme and shallots on a port wine reduction

Environmental Issues

Public Analysts continue to provide scientific expertise by monitoring of air, water and land for pollution and contamination.

As an initiative from the Department of the Environment a strategy for air quality management was introduced. This strategy requires local authorities to

a) review air quality within their areas,

b) establish air quality management areas where air quality targets are not being met,

c) prepare plans for remedial action.

In this strategy the priority pollutants were identified as ozone, benzene, butadiene, sulphur dioxide, carbon monoxide, nitrogen dioxide, particulates under 10 microns in size, lead and polyaromatic hydrocarbons (PAHs).

In 1972 the Department of the Environment established the Standing Committee of Analysts whose responsibility was for the preparation of booklets in the series "Methods for the Examination of Waters and Associated Materials". This continuous exercise has been managed by the Drinking Water Inspectorate. In April 1996 the new Environment Agency will be taking over the management of this committee and its remit will be extended to embrace the analysis and examination of air and land as well as water.

During 1995 this Standing Committee published "*Methods for the Isolation and Identification of Human Enteric Viruses from Waters and Associated Materials*" and in preparation for publication is a method which is concerned with the use of Inductively Coupled Plasma Atomic Emission Spectroscopy in water analysis. The Vice President of the Association, Norman Harrison, represents both the Royal Society of Chemistry and the APA on the main committee, this being supported by nine working sub groups which deal with the different areas of water analysis and examination.

Public Relations

In recent years the Association has published a number of brochures which have explained the role of Public Analysts. Issue 3 of Public Protection Science, entitled "1994 - From behind the Scenes", was published in the middle of 1995 and it received a wide circulation. Aspects of food composition and several of the more curious findings from Public Analysts Laboratories were reported. Perhaps the most unusual finding reported concerned cider being delivered to a domestic house through the mains tap as a result of a problem at a neighbouring cider factory.

The Public Relations Officer of the Association, Mr. Paul Lenartowicz has established a procedure for publicising the Association's view on topical issues and activities. Proactive and reactive issues are recognised

in the procedure and the mechanism for utilising the procedure established.

The Association exhibited at the annual conferences of the Institute of Trading Standards Administration (ITSA) which was held at Scarborough, North Yorkshire, and that of the Chartered Institute of Environmental Health (CIEH) at Bournemouth.

Representation on Outside Bodies

The Association continues to provide representation to a wide range of outside bodies which include LACOTS panels, MAFF Food Authenticity Groups and Food Analysis Performance Assessment Scheme (FAPAS) Steering Group, The Royal Society (RSC), Food Law Enforcement Practitioners (FLEP), Council for European Standards, (CEN), the Department of the Environment and The British Standards Institute, (BSI). During 1995 the President of the Association was invited to join The Food Advisory Committee of MAFF.

The Analytical Methods Committee of the RSC has traditionally played the lead role in determining the composition, including the Nitrogen content of flesh. The data so generated is used for the determination of the meat contents of meat products and is accepted by courts. In recent years data concerning the composition of beef and pork has been derived and published in the Analyst, the international analytical journal of the RSC. This year the exercise has been completed for sheep meat and publication is expected during 1996.

FLEP is an international group of representatives from EU member states and European Free Trade Association, (EFTA), countries. To date this organisation has relied on the financial support of the Dutch Government. In 1995 meetings have been held in Bilbao, Spain and Holland. FLEP supplements the work of national bodies by contributing views to the Commission from food law practioners' point of view. The chairman of the FLEP sub-group responsible for identifying and collating items is Anthony Harrison. Guidelines for the interpretation of EN45000 for the use of Official Food Control Laboratories, Proposals for the Co-ordinated sampling programme 1996, the Community Inspection Service and Food Frauds (milk and milk products, meat and meat products and fish and fish products have have been discussed at this forum.

The Scientific Affairs Committee

The Scientific Affairs Committee (SAC) of the Association has met on four occasions during 1995 and has responsibilities which include

a) the development of the validated enforcement methods.

b) addressing enquiries from members of the Association on topics within the areas of expertise of the sub committee structure of this committee.

c) responding, on behalf of the Association, to requests from MAFF, the Department of Health, the Department of Trade and Industry, and the Department of the Environment. These requests can be concerned with proposals for new amended legislation connected with the work of Public Analysts.

d) the generation and revision of food standards. This area of work, under the Standards and Guide Levels Sub-committee, has most recently reviewed the fat content of minced beef and published the results of this review in the Journal of the Association of Public Analysts.

Other activities have included the matter of uncertainty of scientific measurement and the production of a policy document on this subject. The members of the SAC are additionally active representing the Association on the outside bodies referred to above.

Conclusion

In recent years public interest in the composition and safety of foods has been demonstrated by media awareness of the presence of diethylene glycol (so called antifreeze) in Austrian wine, the presence of methanol (a toxic alcohol) in Italian wine, the presence of salmonella and listeria organisms in poultry and cheese products respectively, added water in meat products and the alleged incidence of food contamination from herds of cattle contaminated with bovine spongiform encephalopathy, (BSE). Hand in hand with these has been awareness of the appreciation of the value of scientific results in the courts where criminal investigations have been at issue.

In the field of consumer protection the contribution to the maintenance of food safety and quality from scientists is vital in that properly qualified scientists working in a regulated laboratory are capable of detailed analytical results and interpretations of these results. This system is

capable of being undermined by the diversion of funds into local authority so called "screen testing" laboratories and by short term decisions by some local authorities concerning Public Analyst appointments. The results of such may be quite disastrous not only for public protection but also for the maintenance of standards in the food industry because inadequate funds and inadequate periods of appointment will be available to ensure the necessary development of scientific expertise appropriate to modern food technology.

Food safety and composition remains the subject of criminal law within the UK and international law within the European Union.

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Annual statistics for the Public Analyst service 1995

Supplementary to the Annual Report of Council of the Association

Presented by Mr. P. Lenartowicz

Introduction

The following report presents data related to samples submitted to Public Analysts' laboratories by local authorities during the calendar year 1995.

As usual the statistics presented are purely in terms of numbers of samples and are not a direct measure of the total amount of actual work undertaken by laboratories. Some samples may only require minimal analysis and interpretation whilst others can result in a virtual research project in order to certify with confidence that they are or are not satisfactory - a difference that is rarely evident if the sample is viewed as a simple commodity. The recognition of need and matching of analytical response and interpretation with attendant quality assurance, always to a standard acceptable to a Court of Law, is one of the fundamental differences between the Public Analyst service and mere test houses.

Data

Two Public Analysts' laboratories in England closed during the year, both having been owned by large commercial organisations which decided to withdraw from providing a Public Analyst service. As a consequence statistical returns were not made by those laboratories.

In addition data was not returned by four other Laboratories in the United Kingdom, one declining to submit data as a matter of political policy and three having been unable to meet the final deadline for return. The submitted data has been calculated for the full population of the UK by interpolation, and detailed figures broken down by sample type appear in the Appendix.

Again this year the figures for Central and South Wales (covering the bulk of the population of the country) have been separated from the English statistics in order to give an indication of the relative position in Wales. The northernmost counties of Wales submit their samples to a laboratory that also serves a number of English authorities, therefore complete separation of Welsh statistics has not been possible and any references to England or Wales in the following discussion must be viewed accordingly.

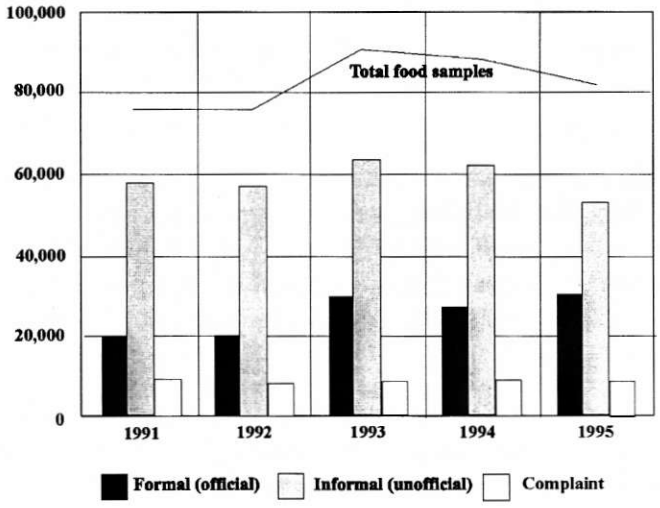
Data received from the two Channel Islands' laboratories has been recorded for comparative purposes, but has not been included in calculations of United Kingdom statistics

Food Work

Chart 1 shows the rates of submission of food samples for analysis in 1995 compared with the previous four years. The 3% fall in sampling levels for food noted in 1994 compared with 1993 worsened to a 7.5% fall from 1994 to 1995, although levels did remain a little higher than those of the preceding two years.

It was postulated in 1993 that the increase recorded for that year may have been a consequence of pressure on local authorities to achieve the World Health Organisation's recommended minimum sampling rate of 2.5 samples per 1000 population. If that was indeed the reason the effect was rather short-lived, with 1995 nearly down to the level prior to the increase. There does, however, appear to have been a general swing towards official (formal) sampling in preference to unofficial, from 25% of planned samples in 1991 to 36% in 1995.

Chart 1:
Food Samples by year

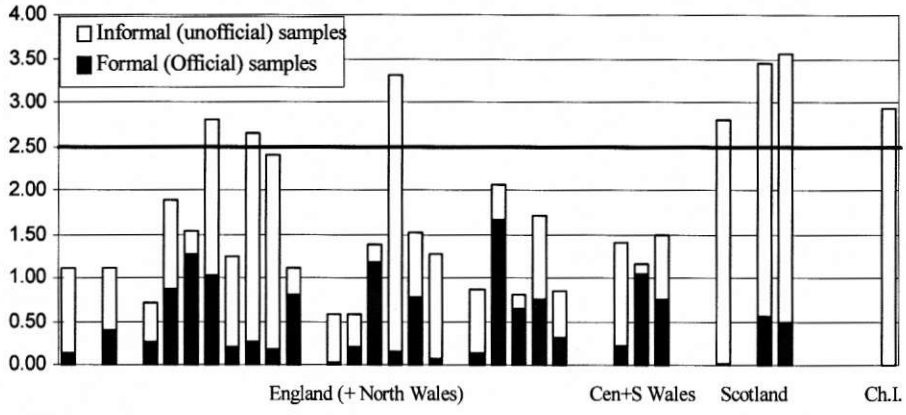


The mean number of planned food samples for analysis per 1000 population in the UK in 1995 was 1.47, against 1.59 in 1994 and 1.63 in 1993. (Total foods including complaints were 1.62, 1.74 and 1.81 respectively for 1995, 1994 and 1993.) These averages, however, mask a

great variation between individual authorities. Chart 2 illustrates the spread of planned samples on a laboratory-by-laboratory basis.

The authorities served by the Scottish laboratories have once again managed to meet World Health Organisation targets, achieving an average of 3.4 samples per 1000 population, while only three areas served by English or Welsh laboratories exceeded 2.5, the average in England and Wales being only 1.27 - just half the recommended minimum, with one laboratory's area sampling a mere 0.57 (less than last year's lowest of 0.72)

Chart 2: Total planned food samples per 1000 population



In addition to official samples, being those which have been taken under the Food Safety Act 1990 and upon which official action may be taken, the above totals for planned sampling include "informal" samples. Informal samples are those that have not been taken in the prescribed manner but which have been submitted to the official Public Analyst's laboratory. The formal sampling of food is a time consuming and highly skilled operation unlike the mass purchase of informal samples, and is therefore often used by local authorities for routine survey work as it saves substantially on the costs of sampling. It is notable that the areas sampling at the highest levels tend to be those with a relatively low proportion of formal samples. Chart 2 also indicates the relative proportions of official and unofficial samples.

Unsatisfactory Food Samples

Again this year information was sought in relation to unsatisfactory samples, which indicated that an average of 21% of routine food samples - one sample in five - had been found to be in some way unsatisfactory, and that 55% of complaints were upheld - figures which are virtually identical to the previous year (21% and 56% respectively).

A little over half of the unsatisfactory samples - 12%, or nearly one sample in eight - failed to comply with labelling requirements. There is sometimes a tendency to dismiss these as being 'only labelling errors', yet although some may have been relatively minor infringements this category includes very serious problems such as misleading or missing information (which in certain instances could prove hazardous to health, for example failure to declare ingredients to which a consumer might be allergic), failure to indicate the minimum durability (again with potentially dangerous results), and fraudulent claims, in addition to failure to make correct statutory declarations etc. The majority of labelling faults can only be detected by detailed analysis, and are not evident solely from a scrutiny of the label.

Food Factory Inspection

A total of 1231 days were spent on food factory inspection during the year, very similar to the previous year's 1262. Again it is clear from individual laboratories' returns that the use of the Public Analyst in factory inspections has only been adopted to any significant degree by a very small number of authorities, despite his potential value as an expert to assess sampling and analytical records as well as other technical aspects of food production. One laboratory accounts for well over three quarters of the time spent on factory inspection while over half of the Association's laboratories had no involvement at all.

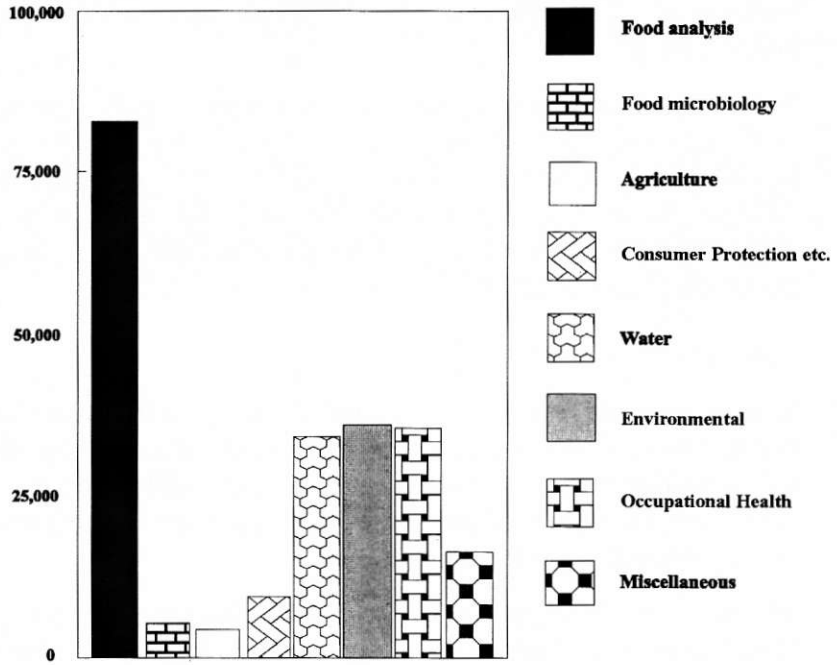
Non-food Work

There were 186 call-outs to emergency incidents involving chemicals (ranging from fires to chemical spillages), utilising the 24-hour-a-day, 365-day-per-year back-up service provided to the emergency services to advise and/or provide analytical facilities to help deal with any incident that may be a threat to the public or the environment. On average this equates to one call for assistance somewhere in the UK every 47 hours, a 33% reduction from the previous year, which may be indicative of a lower incidence of such emergencies.

The major areas other than food in which Public Analysts' laboratories provided analysis to local authorities during 1995 are as follows, in decreasing order of numbers of samples:

- Environmental investigations (soil, water, tip leachates, atmospheric samples etc.)
- Occupational Health (Asbestos identification and air testing, COSHH monitoring, etc)
- Water analysis (Mainly potable waters, but also swimming pools, etc)
- Consumer Products (Toys, cosmetics, household chemicals, etc. for safety and to assess Trade Descriptions)
- Miscellaneous (Radiation monitoring, toxicology etc)
- Agricultural samples (Fertilisers and Animal Feeds)

All samples analysed in 1995



During the year 620 days (19% greater than 1994) were spent assisting with the inspection of non-food factories, providing scientific expertise to interpret and assess the increasingly scientific (and pseudo scientific) aspects of manufacturing control.

Proficiency testing schemes

One increasingly significant aspect of Public Analysts' work that is not reflected in the statistics collected on samples analysed is the time spent on proficiency testing. For example, participation in the Ministry of Agriculture, Fisheries and Food's *Food Analysis Performance Assessment Scheme*, FAPAS, effectively involves up to one circulation every seven working days, yet is necessary if a laboratory is to demonstrate competence in food analysis as required by the European 'Additional Measures Directive'.

These samples all require full set-up, calibration and quality assurance procedures to be instigated, procedures that can be extremely time consuming for some circulations. If, as may often be the case, the circulations cannot be tied in with "real" samples for similar analysis these procedures still have to be carried out in full, imposing a substantial burden of work for no return. The analytical cost for participation in the full range of FAPAS has been estimated to be up to £40,000 per annum.

The observations apply to the many other proficiency testing schemes necessary or desirable in certain areas of work, for example the Regular Interlaboratory Counting Exchange (RICE) for asbestos, Workplace Analysis Scheme for Proficiency (WASP) for atmospheric analysis, 'AQUACHECK' for water, etc., although none have the same vast scope applicable to food analysis.

Observations

With respect to the sampling of food for analysis it is worrying to note that the position has worsened since last year, with a further decrease in the sampling rate to an average of 1.47 per 1000 head of population, which is little more than half of the minimum rate of 2.5 recommended by the World Health Organisation.

The reduction in sampling is of particular concern when set against the backdrop of a constant level of 21% unsatisfactory foods, and even more so given the moves towards deregulation of industry.

The high rate of unsatisfactory samples detected clearly demonstrates the effectiveness of the Public Analyst service in applying quality analysis backed by solid science, skill, experience, development and professional co-operation. However, the long term ability to maintain an effective service is dependant upon continued investment in both the seen and the unseen

aspects of public protection science, which may be irreparably damaged by diversion or dilution of limited resources for short term gain.

Analysis for the purposes of law enforcement and public and environmental protection is ever increasing in complexity as both manufacturing and analytical technology advance. This, combined with the increasing burdens of accreditation and proficiency testing, makes it important for maximum use to be made of the resources available to local authorities within their Public Analysts' laboratories in order spread the necessarily substantial standing costs of maintaining a comprehensive and effective service over as wide a base as possible.

The continuing use of the Public Analyst by local authorities for non food analysis is therefore welcomed, although it is evident from individual returns that the degree of use by Trading Standards and Environmental Health departments in respect of such work varies tremendously. The same is true of the use of Public Analysts for factory inspection, where their skills may be invaluable in interpreting analytical and technical data.

It is hoped that when the food law enforcement system comes to be reviewed the issues raised in this and previous reports will be addressed, and that due emphasis will be placed on cost effectiveness rather than merely on cost, the latter appearing to have been an increasing trend in recent years to the potential detriment of the long term ability to be effective.

APPENDIX

Data returned by laboratories for local authority work, 1995

	UK returned data	Interpolated to full UK pop.	Channel Isles
Population (millions)	48	56	0.14
Foods - all formal	25451	29900	0
Foods - all informal	43935	52619	235
Foods - Home Authority (if identified)	1801	1981	0
Foods - Port Health	1689	1936	0
Foods - Complaints	7161	8289	38
Foods - Bacteriological	4291	5170	62
Milks	5651	6662	235
Mineral & other bottled waters	454	529	0
Drinking waters	23485	27586	2412
Swimming Pool Waters	5447	6423	10
Pollution water, effluents, tip leachates	12909	15086	2057
Atmospheric samples	16203	18381	926
Soils	1716	1996	29
Workplace monitoring (excl. asbestos)	7225	8380	0
Asbestos (bulk & airborne)	22910	26587	48
Feeding Stuffs - Agriculture Act	2480	2831	0
Feeding Stuffs - Medicines Act	274	312	0
Fertilisers	668	777	0
Toys (Safety) Regulations	3646	4176	0
Cosmetics (Safety) Regulations	1028	1166	0
Other Consumer Prot. Act work	2660	3019	0
Trade Descriptions samples	985	1171	0

Building Materials	328	378	0
Coroners/toxicology	1157	1321	0
Radiation monitoring	7647	8770	0
Other miscellaneous	5031	5744	183
TOTAL SAMPLES	197090	230611	6000
Total foods for analysis	76547	90808	273
Total planned foods for analysis	69386	82519	235
Total foods bacteriological	4291	5170	62
Total Agriculture	3422	3919	0
Total Consumer Prot. etc (incl TDA)	8317	9533	0
Total waters (excl. environmental)	28933	34009	2422
Total environmental samples	30828	35463	3012
Total workplace samples	30135	34967	48
Total other samples	14163	16213	183
Emergency Callouts	154	186	
Factory Insp - Food	1211	1231	
Factory Insp - Non food	620	620	