Fapas® – Food Chemistry Proficiency Test Report 27206

Histamine in Canned Fish

August-September 2017

PARTICIPANT LABORATORY NUMBER

Participants can log in to Fapas® SecureWeb at any time to obtain their laboratory number for this proficiency test.

Laboratory numbers are displayed in SecureWeb next to the download link for this report.

REPORT INTEGRITY

Fapas® reports are distributed as digitally signed Adobe® PDF files. When these files are opened with Adobe® Reader v9 or later, a blue ribbon and information bar indicates that the certificate has been validated. This confirms that the author of the report is Fapas® and that the document has not been altered since it was signed [1, 2].

The integrity of hard copies of Fapas® reports cannot be assured in this way, i.e. printed copies are not controlled. A watermark stating this appears on every page when a Fapas® report is printed.

End users of Fapas® reports should ensure that either the PDF file they are viewing displays a valid Fapas® digital signature or that the content of their hard copy exactly matches the content of a PDF file that displays a valid Fapas® digital signature.

QUALITY SYSTEMS

Fapas® – Food Chemistry is accredited by UKAS as complying with the requirements of ISO/IEC 17043:2010 [3].

Fera is an ISO 9001 certified organisation.

UKAS PROFICIENCY TESTING
0009

Fera hereby excludes all liability for any claim, loss, demands or damages of any kind whatsoever (whether such claims, loss, demands or damages were foreseeable, known or otherwise) arising out of or in connection with the preparation of any technical or scientific report, including without limitation, indirect or consequential loss or damage; loss of actual or anticipated profits (including loss of profits on contracts); loss of revenue; loss of business; loss of opportunity; loss of anticipated savings; loss of goodwill; loss of reputation; loss or damage to or corruption of data; loss of use of money or otherwise, and whether or not advised of the possibility of such claim, loss demand or damages and whether arising in tort (including negligence), contract or otherwise. This statement does not affect your statutory rights.

Nothing in this disclaimer excludes or limits Fera liability for: (a) death or personal injury caused by Fera negligence (or that of its employees, agents or directors); or (b) the tort of deceit; [or (c) any breach of the obligations implied by Sale of Goods Act 1979 or Supply of Goods and Services Act 1982 (including those relating to the title, fitness for purpose and satisfactory quality of goods);] or (d) any liability which may not be limited or excluded by law (e) fraud or fraudulent misrepresentation.

The parties agree that any matters are governed by English law and irrevocably submit to the non-exclusive jurisdiction of the English courts.

SUMMARY

1. The test material for Fapas® – Food Chemistry proficiency test 27206 was dispatched in August 2017. Each participant received a canned fish test material to be analysed for histamine.

2. An assigned value (x_a) was determined for histamine and in conjunction with the standard deviation for proficiency (σ_p) was used to calculate a z-score for each result.

3. Results for this proficiency test are summarised as follows:

analyte	assigned value, x_a mg/kg	number of scores, |z| ≤2	total number of scores	% |z|≤2
Histamine	37.7	142	175	81

CONTENTS

1. INTRODUCTION

1.1. Proficiency Testing

Proficiency testing aims to provide an independent assessment of the competence of participating laboratories. Together with the use of validated methods, proficiency testing is an essential element of laboratory quality assurance.

Further details of the Fapas® – Food Chemistry proficiency testing scheme are available in our protocols [4, 5].

2. TEST MATERIAL

2.1. Preparation

Preparation of the samples for this proficiency test was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [3].

The test material was prepared from tuna steaks.

Histamine was spiked into the test material.

Samples were stored at an ambient temperature until dispatch.

2.2. Homogeneity

To test for homogeneity, randomly selected test materials were analysed in duplicate. Testing was sub-contracted to a laboratory meeting the quality requirements of the scheme's accreditation [3].

These data showed sufficient homogeneity and were not included in the subsequent calculation of the assigned value.

2.3. Dispatch

The start date was 10 August 2017. Test materials were sent to 182 participants.

3. RESULTS

The instructions for reporting results were as follows:

- Determine the level of **histamine** present in the test material, in **mg/kg, as received, corrected for recovery**.

Results were submitted by 175 participants (96%) before the closing date for this test, 28 September 2017.

Each participant was given a laboratory number, assigned in order of receipt of results. The reported analyte concentrations are given in Table 1.

Participants' comments are given in Table 2.

The analytical methods used by each participant are summarised in APPENDIX I.

4. STATISTICAL EVALUATION OF RESULTS

The results submitted by participants were statistically analysed in order to provide an assigned value for histamine. The assigned values were then used in combination with the standard deviation for proficiency, σ_p, to calculate a z-score [6] for each result. The procedure is detailed in the relevant protocols [4, 5].

Further background on the procedure followed can be found in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

4.1. Calculation of the Assigned Value, x_a

The assigned value, x_a, for histamine was derived from the consensus of the results submitted by participants.

The following results were excluded from the calculation of the assigned value:

i) non numerical results i.e. qualitative or semi-quantitative results,

ii) results reported as approximately 10, 100 or 1000 × greater or smaller than the majority of submitted results (as these were considered to be reporting errors),

iii) results uncorrected for recovery unless stating N/A.

For histamine, this procedure was straightforward and the robust mean was chosen as the assigned value.

The assigned value for histamine is shown in Table 3.

4.2. Standard Deviation for Proficiency, σ_p

The standard deviation for proficiency, σ_p, was set at a value that reflects best practice for the analyses in question.

For histamine, σ_p was derived from the appropriate form of the Horwitz equation [8].

The values for σ_p used to calculate z-scores from the reported results of this test are given in Table 3.

4.3. Individual z-Scores

Participants' z-scores were calculated as:

$$z = \frac{(x - x_a)}{\sigma_p}$$

where x = the participant's reported result,
x_a = the assigned value
and σ_p = the standard deviation for proficiency.

Participants' z-scores for histamine are given in Table 1 and shown as a histogram in Figure 1. It is possible for the z-scores published in this report to differ slightly from the z-score that can be calculated using the formula given above. These differences arise from the necessary rounding of the actual assigned values and standard deviations for proficiency prior to their publication in Table 3.

The number and percentage of z-scores in the range $-2 \leq z \leq 2$ for histamine are given in Table 4.

5. INTERPRETATION OF SCORES

In normal circumstances, over time, about 95% of z-scores will lie in the range $-2 \leq z \leq 2$. Occasional scores in the range $2 < |z| < 3$ are to be expected, at a rate of 1 in 20. Whether or not such scores are of importance can only be decided by considering them in the context of the other scores obtained by that laboratory.

Scores where $|z| > 3$ are to be expected at a rate of about 1 in 300. Given this rarity, such z-scores very strongly indicate that the result is not fit-for-purpose and almost certainly requires investigation.

The consideration of a set or sequence of z-scores over time provides more useful information than a single z-score. Examples of suitable methods of comparison are provided in the IUPAC International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories [7].

6. REFERENCES

1 Adobe Approved Trust List, https://helpx.adobe.com/acrobat/kb/approved-trust-list2.html#Whatisit accessed 01/06/2017.

2 GlobalSign PDF Signing Tool, https://www.globalsign.com/en/pdf-signing/ accessed 01/06/2017.

3 ISO/IEC 17043:2010, Conformity assessment – General requirements for proficiency testing.

4 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 6, April 2017, Part 1 – Common Principles.

5 Fapas®, 2017, Protocol for Proficiency Testing Schemes, Version 5, April 2017, Part 2 – Fapas® Food Chemistry scheme (FAPAS).

6 AMC Tech Brief No. 74, z-Scores and other scores in chemical proficiency testing – their meanings, and some common misconceptions, *Anal. Methods*, 2016, **8**, 5553.

7 Thompson, M., Ellison, S.L.R. and Wood, R., 2006, The International Harmonised Protocol for the Proficiency Testing of Analytical Chemistry Laboratories, *Pure Appl. Chem.*, **78**, No. 1, 145–196.

8 Thompson, M., 2000, Recent trends in inter-laboratory precision at ppb and sub-ppb concentrations in relation to fitness for purpose criteria in proficiency testing, *Analyst*, **125**, 385-386.

Table 1: Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
001	36.7	97.4	3	-0.3
002	38.169	98.5	5.0	0.1
003	39.9	not determined	10	0.6
004	33	98	2	-1.3
005	35.209	NA		-0.7
006	40.9	100	3	0.9
007	40	NA	50	0.7
008	40	na	10	0.7
009	33.16	98.16	10	-1.3
010	40.3	95.5	2.3	0.8
011	38.5	95.5	2.3	0.2
012	40.8	112.5	2	0.9
013	40	na	2.5	0.7
014	46.3	na	2.3	**2.5**
015	35.8	96	0.5	-0.5
016	51.930	121.3	2.5	**4.1**
017	46	na	5	**2.4**
018	37	93	4	-0.2
019	38.775	100	2	0.3
020	41.01	100	0.1	1.0
021	33.4	NA	2.5	-1.2
022	39	na		0.4
023	38.2	102.1	10	0.2
024	38	96	20	0.1
025	35.8	NA	2.5	-0.5

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
026	37.3	95.2	25	-0.1
027	28.09	97	0.5	**-2.7**
028	35	98	20	-0.8
029	35.69	na	10	-0.6
030	42.4	100	5	1.4
031	33.9	80	1	-1.1
032	40.62	105.8	NOT TESTED	0.8
033	39	100.6	5	0.4
034	41.0	80	20	1.0
035	37	NA	5	-0.2
036	38.87	na	0.2	0.3
037	39.10	89.75	7	0.4
038	43.4	101.6	1.9	1.6
039	37.5	n.a	10	0.0
040	55	N/A		**5.0**
041	34	none indicated	10	-1.1
042	33	82	6	-1.3
043	45.70	116	0.50	**2.3**
044	30.12	103.96	3	**-2.2**
045	36.56	na		-0.3
046	38.04	88.37	<5	0.1
047	36	na		-0.5
048	32.7	84.1	3.0	-1.4
049	34.8	na	na	-0.8
050	54	120	7	**4.7**

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
051	38.1	98	3	0.1
052	45.4	NA	10	**2.2**
053	31.11	84.77	0.000006	-1.9
054	37.30	101.79	3.66	-0.1
055	38.3	na""	2 mg/kg	0.2
056	34.53	101.99	3.66	-0.9
057	39.55	81.19	0.20	0.5
058	39.345	na	2.5	0.5
059	35.9	na	10.0	-0.5
060	39.1	85.2	2.3	0.4
061	42.57	-	-	1.4
062	35.94	95	0.01	-0.5
063	36.8	N/A	2.5	-0.2
064	37.37	na	1	-0.1
065	36.52	95.40	0.01	-0.3
066	37.48	91.55	0.01	-0.1
067	38.84	na	3.11	0.3
068	35.5	97	0.001	-0.6
069	36	na	5	-0.5
070	35	na	1	-0.8
071	26.7	109.8	1	**-3.1**
072	38.5	na	10	0.2
073	36	100	2	-0.5
074	27.84	67.5	0.0250391	**-2.8**
075	40.6	93.9	10	0.8

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
076	45.2	na		**2.2**
077	32.60	NA	< 2.5	-1.5
078	41.22	97.65		1.0
079	40.59	NA	NA	0.8
080	35.03	90	5.00	-0.8
081	34.0275	N/A	N/A	-1.0
082	35.1	97.6	2	-0.7
083	0.2664	100	0.0001	**-10.7**
084	35.10	107.13	2.82	-0.7
085	35.41	na	na	-0.6
086	36.88	na	na	-0.2
087	31.0	105	10	-1.9
088	39.23	na	5	0.4
089	20.1	84.1	0.5	**-5.0**
090	39	92.3	1	0.4
091	27.6	N/A	2.5	**-2.9**
092	40.44	nd	1.0	0.8
093	54	na	0	**4.7**
094	18.63	97 - 103	5	**-5.5**
095	42.48	85.00	na	1.4
096	38.5	na	na	0.2
097	44.05	na		1.8
098	37	NA	2.5	-0.2
099	39.1	na	25	0.4
100	29.70	99.95	na	**-2.3**

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
101	38.6	100	2.5	0.3
102	30.64	85.74	-	-2.0
103	39.6	100	10	0.6
104	43	NA	1	1.5
105	39.4	100.7		0.5
106	42.09	100	13	1.3
107	40	NA	1	0.7
108	38.55	94.60	-	0.3
109	34.2	103.5	4	-1.0
110	40	NA	2.5	0.7
111	49.7	92	5.8	**3.4**
112	37.64	82.88		0.0
113	46.5	-	1	**2.5**
114	38.47	97.5	5.0	0.2
115	36.9	na	na	-0.2
116	35.01	na	na	-0.8
117	31.4	30	2.5	-1.8
118	38.45	98.0	2.5	0.2
119	31.18	na		-1.9
120	50	na	5	**3.5**
121	35	na	2.5	-0.8
122	36.6	na	10	-0.3
123	36	96	6	-0.5
124	39.40	98.4	1.0	0.5
125	40.11	99.1	<5	0.7

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
126	39.2	na	10	0.4
127	45.92	92.0%	3.00	**2.4**
128	36.5	na	5	-0.3
129	35.63	102.3	<1	-0.6
130	36.10	101.5	<10	-0.4
131	40.0	105.0	6.3	0.7
132	36.50	87.98	1.20	-0.3
133	26.67	80.00	3.16	**-3.2**
134	35.7	100.9	0.1	-0.6
135	37	120	5	-0.2
136	40.3	98.1 (recovery of the method)	0.5	0.8
137	33.96	108.86	3.57	-1.1
138	35.30	96.63	0.01	-0.7
139	30	20	2.5	**-2.2**
140	40.36	91.92	6.59	0.8
141	33.72	98.2	0.3	-1.1
142	24.9	NA	10	**-3.7**
143	29.38	95	2	**-2.4**
144	30.22	-		**-2.1**
145	37.4	100	3.7	-0.1
146	44.15	85	5-50	1.9
147	140.0	89%	1.0	**29.3**
148	35.6	80	0.6	-0.6
149	42.18	na		1.3
150	39.59	na	5	0.6

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 1 (continued): Results and z-Scores

laboratory number	analyte			
	Histamine assigned value: 37.7 mg/kg			
	result	Recovery (%)	Limit of Detection mg/kg	z-score
151	30.6	75	0.2	-2.0
152	40	78.6	20	0.7
153	30.05	97.23 %	< 2.0 mg/kg	**-2.2**
154	40	na		0.7
155	397	100	0.1	**103.0**
156	89.57	85-105	2-100	**14.9**
157	36	na	1	-0.5
158	36.8	na		-0.2
159	33	na	2	-1.3
160	41	na	2	1.0
161	41.27	na		1.0
162	30.9	101	3	-1.9
163	41.47	na	8.0	1.1
164	47.2	na	2.5	**2.7**
165	35.9	na	2.5	-0.5
166	35.7	na	2.5	-0.6
167	32.9	na	2.5	-1.4
168	42.5	na	2.5	1.4
169	43	na	2.5	1.5
170	45	na	2.5	**2.1**
171	43.5	na	2.5	1.7
172	39.9	na	2.5	0.6
173	40	na	2.5	0.7
174	43	na	2.5	1.5
175	31	na		-1.9

z-scores outside |z| >2 are shown in **bold,** see Section 5

Table 2: Participants' Comments

laboratory number	comments
035	PT-UCA-37
041	mean value 34 mg/kg (single value 33mg/kg and 35mg/kg)
043	Result is corrected for recovery
046	Quantec Turner Fluorometer
049	AOAC performance tested liecense number 021402
060	Uncertainty: 5.8 mg/kg
074	Method used: AOAC Fluorometric Method, 1990
077	Veratox Quantitative Histamine Testkit
088	ST4B
093	we have an internal calibration curve up to 50 ppm
097	Histamine is analyzed spectrofluorimetrically following the procedure of Lerke and Bell (1976). the sample was received on 22 august.
105	- A.O.A.C. Official Method of Analysis.2016. Chapter 35 p. 17-19
136	result has not been corrected for recovery

comments are as submitted by participants

Table 3: Assigned Values and Standard Deviations for Proficiency

analyte	data points, *n*	assigned value, x_a mg/kg	uncertainty, *u*	standard deviation for proficiency, σ_p	
Histamine	166	37.7	0.4	Horwitz [8]	3.49

Table 4: Number and Percentage of z-Scores where $|z| \leq 2$

analyte	number of scores where $\|z\| \leq 2$	total number of scores	% $\|z\| \leq 2$
Histamine	142	175	81

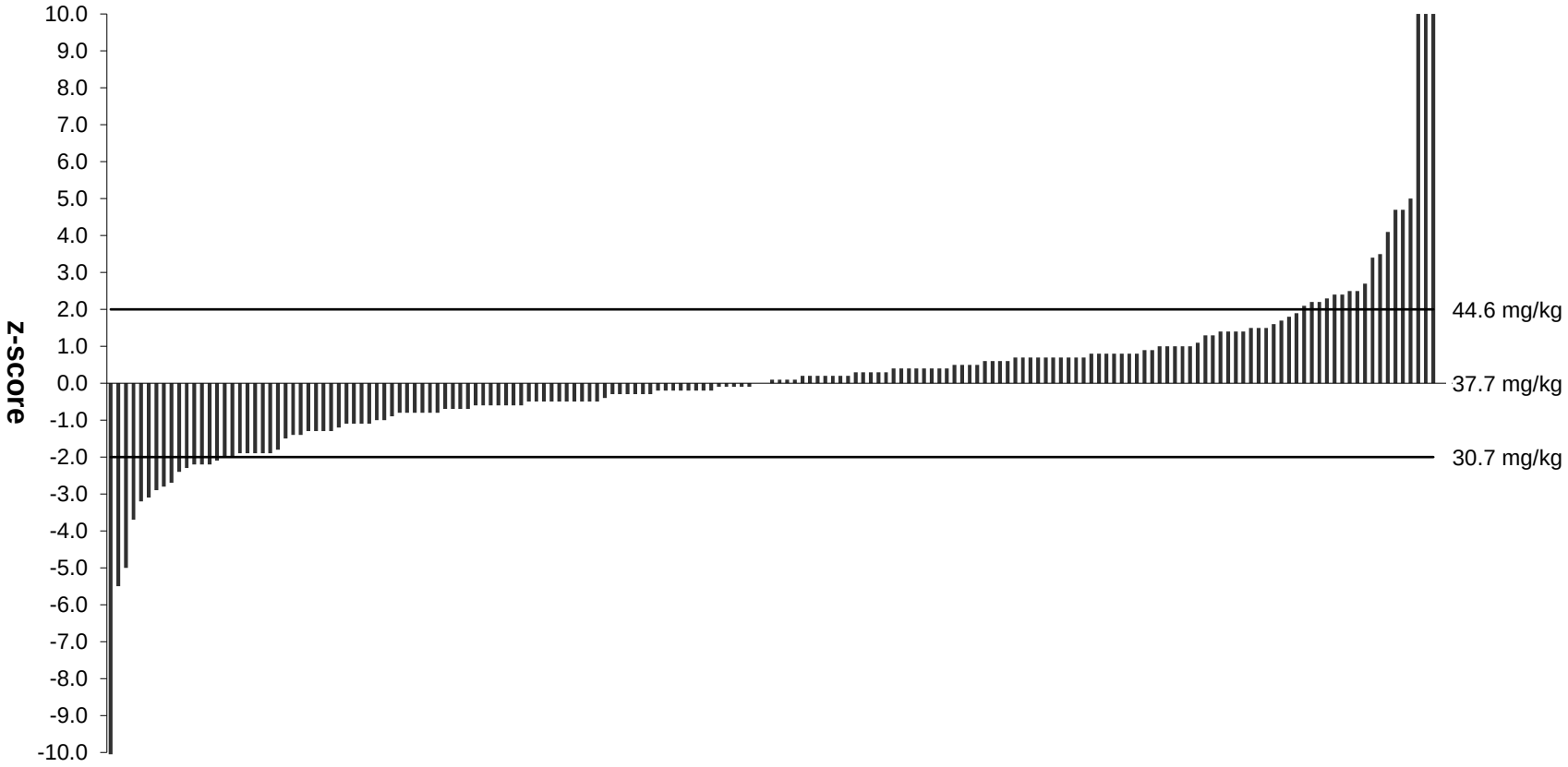

Figure 1: z-Scores for Histamine

APPENDIX I: Analytical Methods Used by Participants

Methods are tabulated according to the information supplied by participants, but some responses may have been combined or edited for clarity.

Histamine HPLC

Is the Method Used Accredited?	laboratory number
yes	001 003 006 009 027 028 029 031 034 036 037 039 042 048 054 059 060 069 071 072 084 089 092 096 098 099 103 123 126 129 136 137 145 146 149 157 163
no	004 008 012 014 015 021 033 055 064 070 077 078 079 088 094 100 105 107 114 122 130 131 141 151 153 155 156

Reference	laboratory number
1995	060
2005	103
2007	089
2009	084
2014	123
AFNOR 2015	006
AOAC Official Methods	031
AOAC Official Methods	100
AOAC Official Methods	054
AOAC Official Methods - A.O.A.C. Official Method of Analysis.2016. Chapter 35 p. 17-19	105
AOAC Official Methods 977.13	099
AOAC Official Methods 1990	074
AOAC Official Methods 1999	069
AOAC Official Methods 2012 977.13 Chapter 35 17-19	078
AOAC Official Methods 2012 Chapter 35 page 17-19	079
AOCS	004
CNS 2007	089
HPLC in Food Analysis	146
HPLC in Food Analysis	163
In house method	145

Reference (continued)	laboratory number
In-house	031
Internal procedure	029
ISO 2015	094
J. AOAC Int. 1996	114
J. AOAC Int. 1997 81/5 991-998	136
J. Assoc. Off. Anal. Chem. 1995 78 1045-1050	039
J. Assoc. Off. Anal. Chem. 1998 81 991-998	037
J. Chromatography A 2004 1032 79-85	009
Journal of food control 2005 16 465-472	141
LMBG Method	149
Ministry of Health and Walfare,Japan 2005	103
OJ (EU) Regulation	031
SNI-2354.10 2009	084
SOP for determination of Biogenic Amines, Torry Research Station, MAFF, Scotland	001
TFDA	071
Tracy, M., Pickering, M., Verhuls, T. Cation Exchange Analysis of foods and Beverages for Biogenic Amines. Food and Testing Analysis. 1995. 1995	060

Sample Amount Used for Analysis (g)	laboratory number
<1	131
≥1 - <2	088 126
≥2 - <5	029 034 071 089 096 141 163
≥5 - <10	004 006 009 012 031 033 037 039 059 069 070 072 078 079 084 094 098 105 114 122 123 129 149 153 155
≥10 - <15	003 027 036 042 060 099 103 136 145 146 151
≥15 - <20	074

Extraction Solvent Components	laboratory number
trichloroacetic acid	001 003 008 012 028 033 036 039 042 048 072 089 096 103
formic acid (methanoic acid)	155
methanol	031 059 060 070 078 079 099 105 155
perchloric acid	006 009 027 029 037 069 088 094 114 123 145 149 153 163
phosphate buffer	034
sodium chloride solution	141
water	001 003 004 029 031 036 092 126
F.A. in water	071
formic acid+ammonium formate	031
HCl	060
Trichloroacetic acid	084 122
trichloroacetic acid, Butyl methyl ether	146
water/perchloric acid	136

Extraction Procedure	laboratory number
add filter aid	123
blend / homogenise with solvent	003 008 012 027 029 033 034 036 037 060 070 078 079 088 099 103 129 136 151
hot water extraction	004 105
maceration/homogenisation	042 114
shake with solvent	012 028 039 042 071 072 096 145 146 155
shaking	059 089 126 163
sonicate/ultrasonic bath	048 122
Ultra Turrax	001 006 031 069 072 094 149
ultrasonic extraction	009
vortex mix	009 114 141 153
mix with solvent	084

Sample Work Up	laboratory number
back-extraction	126
Carrez I & II	027
centrifuge	006 009 012 028 031 034 042 048 060 069 071 084 089 103 114 123 129 141 145 146 153 155 163
defatted with hexane	088
dilute	033 048
Extrelut	099
filter	003 004 008 012 029 031 036 037 039 042 048 071 078 079 096 105 122 149 151
none	001
Ultra Turrax	031 059 094 136

Sample Clean-up Technique	laboratory number
carbon based column	129
extraction	123 141
Extrelut	099
filter	001 006 009 027 033 037 060 069 084 103 136 145 146
liquid/liquid extraction	126
silica column	105
solid phase extraction (SPE) (column/cartridge)	012 042
none	003 004 034 039 059 079 088 089 094 122 149 153 155
ion exchange	078
Ion Exchange - Amberlite	151

Ion Exchange Column Type	laboratory number
ALKION, K+ 4 X 150 mm Pickering	060
anion exchange resin	070
C18(2)150x2mm	129
Dowex (1X8)chloride form, 50-100 mesh	079
Packed Column	151
resin	078
Self Pack Amberlite CG 50	036 146
Spherisorb ODS2 Column, 80 Å, 5 um, 4,6 * 150 mm	037

SPE Sorbent Type	laboratory number
C18	037 146
MCX	042
SCX	012

Solvent Exchange Solvents Used	laboratory number
Butanol	141
HCl	151
Methanol	129
pH6 potasium phosphate/acetate buffer	060

HPLC Injection Volume (µl)	laboratory number
<5	033 048 084 103 122 129 141 155
≥5 - <10	027 031 036 060 088 123 126 151
≥10 - <25	001 003 006 009 012 028 029 034 039 042 059 069 070 071 072 089 096 114 145 146 149 153
≥25 - <50	037 094 136 163

HPLC Column Packing	laboratory number
C18	003 009 012 027 028 029 031 034 036 037 039 042 069 070 072 084 088 089 094 096 114 123 129 136 141 145 146 149 151 153 163
C8	048 126
endcapped	006
Ion Exchange	060
NH2	001
amide	033
Hilic	155
PFPP	122
silica	103

HPLC Column Temperature (°C)	laboratory number
ambient	003 009 028 069 084 096 114 129 149 151 153
>ambient - <50	001 006 008 012 027 029 034 036 037 039 042 048 059 060 070 071 072 088 089 094 103 122 123 126 136 141 146 155 163
≥50	033

Mobile Phase Components	laboratory number
acetate	001 027 029 059 089 149 163
ethanoic acid (acetic acid)	060 088
acetonitrile	001 003 006 009 027 028 029 037 039 042 048 059 069 071 084 088 089 096 103 114 123 126 149 153 155
formic acid (methanoic acid)	031 071 103 122 126 141 155
ion pair agent	027
methanol	001 009 012 036 042 059 070 072 088 103 122 151
phosphate	012 042 048 060 129
sodium hydroxide	145
water	001 003 012 027 028 029 031 036 059 060 071 072 114 126 145 155
1-Octanesulfonic Acid Sodium Salt	012
2-propanol	060
acetonitrile, methanol, acetic acid	146
acetonitrile/water	094
AcN 0.01% FA	008
ammonio formiato	034
ammonium acetate	103
ammonium formate	031 033
buffer/acetonitrile	136
Octane sulphonic	059
octonosulfonic acid sodium salt	149
Pic B 8	145

Buffer Used	laboratory number
acetate buffer	001 027 037 039 059 089 096 103 145 146 149 151
phosphate buffer	009 028 042 048 060 070 084 123 129 136 141 153
0.01% FA	008

Mobile Phase Flow Rate (ml/min)	laboratory number
<0.25	033 103 122
≥0.25 - <0.75	003 008 009 027 031 034 036 070 071 072 084 114 126 129 136 141 151 155
≥0.75 - <1.25	001 006 012 028 037 042 059 060 069 088 089 094 096 123 145 146 153
≥1.25 - <1.75	029 039 048 149

HPLC Pre Column Derivatisation	laboratory number
dansyl chloride	003 037 069 072 094 114 146
fluorescamine	123
OPA	036 048 084 149 151
none	012 034 039 070 088 089 103 129 141

HPLC Post Column Derivatisation	laboratory number
aqueous ammonia	037
none	034 070 084 089 103 123 129 141 146 149
OPA	012 027 029 039 059 096
OPA, Mercaptoethanol	145
OPA/mercaptoethanol	136
OPA/Thiofluor	060
o-phthaldialdehyde, mercaptoethanol	001

Source of Standards	laboratory number
Acros	037
Dr Ehrenstorfer	141
Fluka	008 089 114 149
Merck	048
Sigma/Aldrich	001 003 009 027 028 029 034 036 039 042 060 069 071 072 084 094 096 122 126 129 136 145 146 151 153 155
Wako	033 088 103
CDN isotopes	008
Kanto Kagaku	012

Detector	laboratory number
Diode Array Detector	009 028 042 163
fluorescence	001 012 027 029 036 039 048 059 060 070 079 084 096 136 145 146 149 151
MS	034 103 126 034 103 126
PAD	089 094
UV	003 006 069 072 088 114 123 129 153
MS - MS	141
MS, MS	122
MS/MS	031 033 071
MS-MS	155
PDA	037

Wavelength (absorbance)(nm)	laboratory number
-	151
210	028
214	009
214nm	129
215	042
218	163
254	003 006 037 069 072 088 094 114
254.4	089

Wavelength (excitation)(nm)	laboratory number
330	027 060 149
337	146
338	039
340	036 048 059 136 145
348	012
350	070 084 151
360	079
365	001
388	096

Wavelength (emission)(nm)	laboratory number
418	001
444	070
445	059 145
448	039 096
450	012 036 048 079 084 149 151
455	136
465	027 060
520	146

Histamine ELISA

Is the Method Used Accredited?	laboratory number
yes	002 010 011 016 019 021 089 092 098 110 118 123 131 156 157
no	006 014 049 055 082 107 121 158 162 164 165 166 167 168 169 170 171 172 173 174

ELISA Test Kit Manufacturer	laboratory number
ELISA Systems	131
Labor Diagnostika Nord	049
Neogen	007 010 011 019 021 055 082 092 098 107 110 118 121 157 158 164 165 166 167 168 169 170 171 172 173 174
R-Biopharm	016 156
Bioo Scientific	002
Novakits	014

ELISA Test Kit Name	laboratory number
Histamine ELISA	007 162
Ridascreen Histamin	016 131
Veratox for Histamine	010 011 019 021 055 082 092 098 107 110 118 121 157 158 164 165 166 167 168 169 170 171 172 173 174
Histamine Enzymatic Assay Kit Manual	002
Histasure	014
HistaSureTM Elisa	049
Ridascreen Histamine (enzymatic)	156

ELISA Product Code	laboratory number
9505	019 021 055 082 092 098
9506	010 011 110 118 121 157 158 164 165 166 167 168 169 170 171 172 173 174
R1601	016 131
1032-08	002
7FCE3600	014
FC E 3600	162
FC E-3600	049
R1605	156

ELISA Lot Number or Batch Number of Kit	laboratory number
11157	156
150799-1	016 131
161045	049
170285	014 162
242294	055 082
242795	010 011 110
244648	019
246349	157 158
248417	118
249404	098
Etalon, conjugué : 240875 / Sunstrat : 161201-01 / Solution d'arrêt : 161205-01	168 169 170 171 172 173 174
Etalon, Conjugué : N°241024 / Substrat : 161216-01 / Solution d'arrêt : 160630-01	121 164 165 166 167
TL58564	002

Assay Procedure Followed Exactly as per Kit Instructions?	laboratory number
yes	002 010 011 014 016 019 021 049 055 082 092 098 107 110 118 121 131 156 157 158 162 164 165 166 167 168 169 170 171 172 173 174

ELISA Sample Extraction (weight/volume, g/ml)	laboratory number
<0.5	010 011 016 110
≥1 - <2	131
≥2 - <5	002
≥5 - <10	055 082 092 098 107 118 156 158
≥10	014 019 021 049 121 157 162 164 165 166 167 168 169 170 171 172 173 174

ELISA Standards Used as Supplied (ready to use) or Dilutions Required?	laboratory number
as supplied	002 010 011 014 016 019 021 049 055 082 092 098 107 110 118 121 131 156 157 158 162 164 165 166 167 172 173
diluted	168 169 170 171 174

Standards Analysed in Duplicate?	laboratory number
yes	002 014 016 049 092 123 131 156
no	010 011 019 021 055 082 098 107 110 118 121 157 158 162 164 165 166 167 168 169 170 171 172 173 174

Negative Extraction Control Sample Run?	laboratory number
yes	002 014 016 049 082 092 098 110 121 123 157 158 164 165 166 167 168 169 170 171 172 173 174
no	010 011 019 021 055 107 118 131 156 162

Positive Quality control Sample Run?	laboratory number
yes	002 014 016 049 082 092 098 107 110 118 121 123 131 156 157 158 164 165 166 167 168 169 170 171 172 173 174
no	010 011 019 021 055 162

If 'yes' Was it Supplied by the Kit? If 'no' What Was Used?	laboratory number
supplied with kit	002 010 011 014 049 107 110 121 131 156 164 165 166 167 168 169 170 171 172 173 174

ELISA Calculation of Results	laboratory number
4 parameter	014
cubic spline	016
logit / log	010 011 019 021 055 082 092 098 110 118 121 157 158 162 164 165 166 167 168 169 170 171 172 173 174
Point to point	002 107
log/lin	049

Other Method (give brief outline)	laboratory number
AOAC performance license number 021402	049
Biosensor Biofish	118
Used Histamine standard from another supplier for positive QC. Also ran a QCS material.	098

Histamine Other Method

Is the Method Used Accredited?	laboratory number
yes	015 030 035 038 046 052 053 054 056 067 090 092 106 109 130 133 134 137 138 143 148 159 162 163
no	022 062 064 071 077 080 088 097 100 104 105 107 108 132 144

Reference	laboratory number
2009	133
AOAC Official Methods	132
AOAC Official Methods	030
AOAC Official Methods - A.O.A.C. Official Method of Analysis.2016 Chapter 35 p. 17-19	105
AOAC Official Methods 20th edition, 2016. Chapter 35, 35.1.32 p. 17-19.	067
AOAC Official Methods 1987	159
AOAC Official Methods 1987	062
AOAC Official Methods 1990 15 977.13	143
AOAC Official Methods 1990 15th Ed.	138
AOAC Official Methods 1990 977.13	046
AOAC Official Methods 1990 977.13 876-877	015
AOAC Official Methods 1995 16Ed	035
AOAC Official Methods 1995 16th Ed Procedure 35.1.32	052
AOAC Official Methods 2005	109
AOAC Official Methods 2005 18th	134
AOAC Official Methods 2005 18th edition,2005 35.1.32	064
AOAC Official Methods 2005 2/18 17-19	137
AOAC Official Methods 2012	090 148
AOAC Official Methods 2012 19th Edition 17-19	130
AOAC Official Methods 2012 19th Rdition	144
AOAC Official Methods 2012 20th Edition,2016 35	054 056
AOAC Official Methods 2012 977.13	100 108
AOAC Official Methods 2012 977.13 35.1.32	080
AOAC Official Methods 2012 II 17	022
AOAC Official Methods 2012 JAOAC 60,1125,1131 (1977) CAS-51-45-6 35.1.32	053
AOAC Official Methods 2014	162
AOAC Official Methods 2015 20 17	038
J. Food Science 1976 41 1282-1284	106
J. Food Science 1976 Vol 41 p 1282-1284	097
SNI 2354.10:2009 2009	133
Veratox Quantitatieve Histamine Testkit.	077

Other Method (give brief outline)	laboratory number
A.O.A.C. Official Method , 2005, 997.13	132
AOAC 977.13	056
BioFish 300	030
BIOLAN BIOFISH	104
Extracted with 75% MeOH. Passed through ion exchange column. OPT solution is added to eluate to form fluorescent histamine derivatives. Fluorescent intensity of derivatives is measured using fluorometer & histamine is quantified using external standards.	090
Extraction with Trichloracetic 10%. Purification on cationic resin amberlit CG 50. Elution with 0n2 N HCl. Complexation with OPA. Determinationn with spectrofluorometry (EX: 360nm, Em: 450)nm	106
Fluorometric method	038
Histamine in seafood Fluorometric method	137
Histamine is analyzed spectrofluorimetrically following the procedure of Lerke and Bell (1976)	097
Methanol extraction followed by filtration	143
Methanol extraction, column purification and fluorimetric detection	035
na	064
Spectrofluorometer	133
Spectrofluorometric method	100

APPENDIX II: Fapas® SecureWeb, Protocol and Contact Details

1. Fapas® SECUREWEB

Access to the secure area of our website is only available to participants in our proficiency tests. Please contact us if you require a UserID and Password. Fapas® SecureWeb allows participants to:

- Obtain their laboratory numbers for the proficiency tests in which they have participated.
- View the results they submitted in past and current proficiency tests.
- Submit their results and methods for current tests.
- Review future tests they have ordered.
- Order proficiency tests, reference materials and quality control materials.
- Freely download copies of reports (PDF file), of proficiency tests in which they have participated.
- View charts of their z-scores obtained in previous Fapas® – Food Chemistry proficiency tests.

2. PROTOCOL

The Protocols [4, 5] set out how Fapas® – Food Chemistry is organised. Copies can be downloaded from our website.

3. CONTACT DETAILS

This report was prepared and authorised on behalf of Fapas® by Dominic Anderson (Round Coordinator). Participants with any comments or concerns about this proficiency test should contact:

Fapas®

Fera Science Ltd (Fera)
National Agri-Food Innovation Campus
Sand Hutton
York
YO41 1LZ
UK

Tel: +44 (0)1904 462100
Fax: +44 (0)1904 500440

info@fapas.com

www.fapas.com