

DETERMINATION OF MOISTURE AND FATTY SUBSTANCES CONTENTS OF SOLID ADJUNCTS

SUBMITTED BY MAX BENARD AND BRUCE JOHNSON ON BEHALF OF THE EBC AND IOB ANALYSIS COMMITTEES

Two new methods—moisture at 130–133°C and fatty substances extracted by petroleum ether—were collaboratively tested by 14 laboratories (EBC and IOB Analysis Committee members and other laboratories). For moisture the following values were obtained in the range 12 to 15% m/m, repeatability=0.13% m/m and reproducibility=0.60% m/m. For fatty substances in the range 0.3 to 0.9% m/m the following values were obtained: repeatability=(0.06 m+0.02)% m/m and reproducibility=(0.14 m+0.09)% m/m. Both methods were accepted and will replace methods 6.1 and 6.4 respectively in Analytica EBC. The method for Fatty Substances will replace IOB methods 4.1.3 and 4.2.3. These modifications will become operative on 1 September, 1996.

Key Words: *Analysis method, collaborative trial, fatty substances, moisture, precision, solid adjuncts.*

INTRODUCTION

The EBC Analysis Committee decided to test new methods for the determination of moisture and fatty substances in solid adjuncts. The objective was to harmonise EBC methods with international methods and:

(i) to replace the current EBC 6.1 method for moisture (at 106°C) by ISO method 6540 (at 130–133°C).

(ii) to replace the current EBC 6.4 method for fatty substances (extraction by carbon tetrachloride) by a method using petroleum ether (PE) as solvent, because carbon tetrachloride will be banned in a short time for health and environmental reasons. Moreover, ASBC (Cereals, 4)¹ and EEC methods² are already using PE as solvent.

EXPERIMENTAL

The same general procedure as for malt and laboratory wort, described in a preceding paper³ was followed. Four samples of maize grits were sent to 15 laboratories, which included 8 EBC Analysis Committee members, 2 IOB Analysis Committee members and 4 French laboratories regularly analysing this type of product. Results were received from 14 laboratories and statistically evaluated according to the ISO 5725 standard⁴.

RESULTS AND DISCUSSION

Moisture (Table I): Both methods ISO 6540 and EBC 6.1 give similar precision values (repeatability and reproducibility) and no relationship between precision values and the measured value could be observed. The repeatability value obtained (0.13% m/m) is in line with the value of 0.15% m/m reported in the ISO 6540 method.

As anticipated, the ISO method 6540 which recommends a longer drying time at a higher temperature gives slightly higher moisture values than those by the EBC method 6.1. The difference is fairly constant (0.55 to 0.74% m/m, mean 0.66% m/m) over the range 12 to 15% m/m.

Fatty substances (Table II): It seems that many laboratories have already banned carbon tetrachloride, as only 4 results using the EBC method 6.4 were received, which is not enough for a statistical evaluation. Values from a preceding test⁵ were: repeatability=0.09% m/m and reproducibility=0.19% m/m over the range 0.6 to 1.2% m/m.

The alternative method proposed by the Institute of Brewing Analysis Committee is based on a Soxhlet extraction with petroleum ether as solvent.

TABLE I. Moisture determinations (%)

Sample	A	B	C	D
<i>EBC method 6.1 (106°C, 3 h)</i>				
Number of results	13	14	14	14
Mean	13.82	11.33	12.51	12.44
S _r	0.066	0.081	0.063	0.037
CV _r %	0.48	0.72	0.51	0.30
Repeatability r ₉₅	0.184	0.228	0.177	0.104
S _R	0.262	0.169	0.161	0.161
CV _R %	1.89	1.49	1.28	1.29
Reproducibility R ₉₅	0.733	0.474	0.450	0.451
<i>ISO method 6540 (130°C, 4 h)</i>				
Number of results	12	12	11	12
Mean	14.54	12.07	13.06	13.07
S _r	0.042	0.033	0.053	0.063
CV _r %	0.29	0.27	0.40	0.48
Repeatability r ₉₅	0.117	0.092	0.148	0.175
S _R	0.279	0.255	0.154	0.164
CV _R %	1.92	2.11	1.18	1.25
Reproducibility R ₉₅	0.781	0.713	0.432	0.459

TABLE II. Fatty substances determinations (proposed IOB method) % as is

Sample	A	B	C	D
Number of results	10	10	11	10
Mean	0.286	0.690	0.870	0.669
S _r	0.014	0.024	0.027	0.020
CV _r %	5.00	3.47	3.11	3.04
Repeatability r ₉₅	0.040	0.067	0.076	0.057
S _R	0.046	0.071	0.080	0.054
CV _R %	16.2	10.4	9.2	8.0
Reproducibility R ₉₅	0.130	0.200	0.224	0.150

Repeatability and reproducibility are dependent on the measured value, and precision is similar to the current EBC method 6.4. Results obtained using both methods do not differ significantly.

CONCLUSIONS

The EBC Analysis Committee decided to accept, and recommend the proposed methods:

For moisture, method 6.1.1 will replace method 6.1 which will be archived.

For fatty substances, method 6.4.1 will replace method 6.4 which will be archived.

These modifications will become operative on 1st September, 1996.

TABLE III. Moisture and Fatty Substances in Solid Adjuncts. Summary of Precision Data

		Number of results	Range	Repeatability r_{95}	Reproducibility R_{95}
Moisture, EBC 6.1	%	13-14	11-14	0.17	0.53
Moisture, ISO 6540	%	11-12	12-15	0.13	0.60
Fatty Subst. EBC 6.4 (1992 test)	% as is	10	0.6-1.2	0.09	0.19
Fatty Subst. IoB	% as is	10-11	0.3-0.9	0.06 m+0.02	0.14 m+0.09

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