

NOTE

Method for Ash Determination by Conductivity¹K. M. FJELL,² W. SEIBEL,^{3,4} and P. GERSTENKORN³

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Ash determination is an important analytical tool in management of the extraction rate of wheat (*Triticum aestivum*) during the milling process. The ash content is lowest in the center of the kernel and increases towards the outer parts because the bran layer contains several times more minerals than does the pure endosperm. In discussing the cumulative ash curves, many authors have indicated that ash curves can be influenced by hardness, variety, whole grain ash, and milling system (Seibel 1974; Posner and Deyoe 1986; Li and Posner 1987, 1989).

In Europe, ICC Standard 104/1 (ICC 1993a) has been the most common ash method in use for a great many years. This method prescribes an ashing temperature of 900°C, using a great deal of time, labor and energy, as well as relatively expensive equipment (such as an electrically heated muffle furnace). In milling especially, a rapid feedback of ash values may be needed to make changes in the grist or other milling parameters. Therefore, a rapid and inexpensive test for ash determination has been needed by the milling industry for many years.

Such a method was described by Clements (1977) and has been in use in Switzerland for several years (W. Saurer, *personal communication*). ICC Working Group 3 (Ash) studied the accuracy of this method; the collaborative test results were reported by Fjell and Örsi (1993). This working group was asked by the ICC Executive Committee to develop a new ICC standard method (rapid method) for ash determination.

Importance of Ash Content

In many European countries, especially in countries of the European Union, wheat flour is classified on an ash basis. Germany (DIN 1992) has a traditional ash classification of six flours (range of ash content, dwb): W405 (maximum 0.50%), W550 (0.51–0.63%), W812 (0.64–0.90%), W1050 (0.91–1.20%), W1600 (1.21–1.80%). There is no ash limitation for whole wheat meal because whole wheat meal consists of all kernel parts, including the germ.

The wheat flour exported from the European Union is also based on ash content (EG 1993). We have six flour types, based on ash content, which correspond with the equivalent amounts of levy-free wheat imports. The most important classes are flour types 1, 2, and 3, with corresponding ash contents (dwb) of maximum 0.600, 0.601–0.900%, and 0.901–1.100%, respectively. These three flours (type 1, 2, and 3) have levy-free wheat import equivalencies of 137, 128, and 118 kg, respectively.

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MATERIALS AND METHODS

Mineral (ash) determination by conductivity is based on the principle that, like metals and gases, liquids conduct electricity. The specific conductivity is in proportion to the concentration of the electrolyte in the liquid. The determination of the conductivity is measured between an anode and a cathode submerged in the electrolyte and expressed with units of $\mu\text{S}/\text{cm}$.

Method

Flour (1 g) is extracted with 25 ml of deionized water for 60 min at 30°C. Extraction takes place directly in 50-ml centrifuge tubes, by stirring at 15-min intervals with a glass rod. After the extraction, the supernatant is cleared by centrifuging at 4,000 rpm for 3–5 min. The supernatant is then collected carefully and measured by a conductivity meter.

An empirically derived standardization procedure should be conducted using finely ground flours with ash contents in the range of 0.400–0.500%. Ash content is determined using ICC Standard 104/1. At the same time, conductivity values are also measured. The unknown values *A* and *B*, can then be solved in the model: $\log_{10}(\text{ash}) = A + B \log_{10}(\text{conductivity})$. The standard deviation (SD) and the correlation coefficient (*r*) can be calculated. Using a standardized KCl solution, the equipment and methods can be controlled over a period of time.

RESULTS AND DISCUSSION

Ring Tests

A collaborative testing of the method was coordinated by ICC Working Group 3 (ash determination). Six laboratories in different countries participated using a conductivity meter according to the OIML Recommendation 68 "Calibration Method of Conductivity Cells." In each laboratory, two people completed the analysis. Five samples of flour with different ash content were sent to the participants with a standard KCl solution.

Twelve researchers accomplished five repetitions on each flour sample. Intralaboratory repeatability error on conductivity, reported as a relative standard deviation, ranged from 1.48 to 2.12%. Interlaboratory reproducibility at 10.57–14.05% was much larger. Despite the large reproducibility error, the relative differences between the various samples were consistent from laboratory to laboratory.

Participants were also asked to determine the conductivity of KCl standard solution, both before and after testing the flours. From these values, it was possible to correct the conductivity levels among the laboratories. The individual values were multiplied by a factor (*f*), calculated for each laboratory, as: $f = \text{mean of all KCl determinations} + \text{mean of KCl determination by participants}$.

The statistical evaluation of the corrected data showed that: 1) relative standard deviation in the laboratories remained practically unchanged from 1.48–2.12% to a corrected range of 1.52–2.31%, and 2) relative standard deviation between laboratories was signif-

TABLE I
Comparison of Ash Content (% dm) of Five Wheat Flours with Ash Content Predicted from Flour Conductivity with ICC Standard Method

Method	Sample ^a				
	1	2	3	4	5
ICC 104/1	0.393	0.519	0.730	0.871	1.58
Conductivity	0.398	0.544	0.780	0.804	1.56

^a *n* = 60 (6 laboratories × 2 researchers × 5 repetitions).

ificantly reduced from 10.57–14.05% to a corrected range of 2.52–5.53%.

Each of the samples sent out for examination was also tested by 10 repetitions of the regular ash method (ICC 1993a). Combining these results with the conductivity measurements produced the equation: $\log(\text{ash}) = -3.746 + 1.4375 \times \log(\text{conductivity})$ ($r = 0.985$ and $SD = 0.0365$).

The mean values as shown in Table I, are the results of the comparison of the conductivity ring test with similar values determined by the ICC Standard 104/1 (ICC 1993a). Samples 1 and 5 came out with quite comparable results, while sample 4 showed a relatively large difference. Such variations are difficult to avoid and have also been detected in an international ring test of ICC Standard 104/1, which were also performed by the ICC Working Group 3 (Ash) (reproducibility: 0.015–0.079%).

Linear regression between the measured ash contents (ICC Standard 104/1) and the ash content predicted by conductivity showed a slope not significantly different from sample 1 and a y-intercept not significantly different from 0 at the 95% level of probability (ICC 1993b). This result means that there is a good agreement to the ash content according to ICC Standard 104/1 (Fjell and Örsi 1993).

The distribution of the 12 single values of sample 1 is shown in Figure 1. Only two laboratories reported figures outside the tolerance.

Gerstenkorn (1993) found that the ash content of 60 flours and wheat meal samples, as predicted by conductivity, had a standard deviation of 0.020%. The samples ranged in ash content from 0.394 to 1.879%. The flour samples ranged between 0.394 and 0.604% and showed a deviation of 0.017%, which is under the tolerance stated in the ICC Standard 104/1. Whole wheat meal gave a higher deviation (0.028%), which is still within the tolerance indicated in that method. The data supports the conclusion that the conductivity method is sufficiently accurate for most purposes.

The method discussed here is less time-consuming than the standard ash method, which requires 3–4 hr. Preparation time included, the analysis with the conductivity method will require 1.5 hr. In addition, the cost of these analyses is quite modest and does not require expensive equipment.

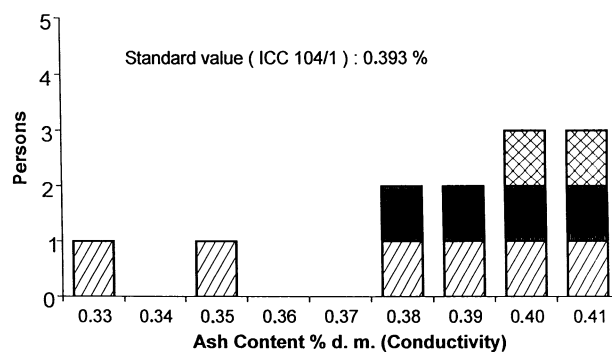


Fig. 1. Distribution of ash content for 12 single values of sample 1. Striped, shaded, and cross-hatched boxes represent results of one, two, and three laboratories, respectively.

CONCLUSION

Ash determination by conductivity is an alternative to the standard ash method. The presupposition is that method instructions are closely followed and that the equipment is carefully controlled. The conductivity meter should conform to the OIML Recommendation 68 "Calibration Method for Conductivity Cells." The ash conductivity method will be made into an ICC Standard Method. In June 1994, the ICC General Assembly, adapted it as ICC Draft Standard 157.

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