Comparison of the Official AOAC Method with Rapid Methods for the Analysis of Moisture in Meats

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Moisture studies were conducted on 4 types of meat. A method specifying 2 hr at 125–150°C in a gravity convection oven gave similar results to 24.003. Five rapid methods were also evaluated: the IR automatic moisture analyzer, the IR moisture determination balance, toluene extraction, specific gravity change, and hot plate methods. The methods were evaluated for precision and recovery of moisture as compared with the official AOAC method and for their practicability, ease of handling, and rapidity of analysis.

Miller (1) states that the accuracy of the oven moisture procedure depends on the validity of 2 assumptions: (*a*) the attainment of constant weight implies complete removal of water, and (*b*) the entire weight loss is due to the volatilization of water. No mention of the expected accuracy and precision of the AOAC oven method is found in the literature. The ISO subcommittee for meat and meat products (2) has established that the difference between duplicates should not be greater than 0.5% moisture. An unofficial guideline for control work on the finished product has been "not to exceed ±1% of the value of the moisture content obtained by the official analysis" (3). For rapid methods that may be of value to both processors and control agencies in the field, the precision should be within ±1% of the determined value. For analysis of raw material, however, ±3% may be tolerated.

The official AOAC procedure (**24.003**) for analysis of moisture in meat and meat products (4) presents 2 options: One is to dry a sample representing 2 g dry weight for 16-18 hr at 100–102°C in an air oven (mechanical convection preferred); the other option is to dry a similar sample for 2–4 hr, depending on the product, to constant weight in a mechnical convection oven at about 125°C. Windham (5) compared the older methods of vacuum oven drying at 70 and 100°C and 100 mm Hg with the 2 official oven methods which involved heating at 100 and 125°C. The vacuum oven methods were found to be either too long (17 hr at 70°C) or too inaccurate for high fat-containing samples (6 hr at 100°C).

Azeotropic distillation of moisture in meat products was extensively investigated by Bidwell and Sterling (6), resulting in the most widely used apparatus for azeotropic distillation. An excellent review by Fetzer (7) covers a period of 50 years of use. More recent papers by Kaufmann and Keller (8) using trichloroethylene, Phillips and Enas (9) using tetrachloroethane, Lórant and Pollak (10) using acetyl chloride, and Wyler (11) recommending tetrachloroethylene have not shown any advantages in shortening time of distillation over the classical use of toluene. The solubility of toluene, so frequently referred to as the reason for changing to a different azeotropic solvent, is approximately 0.002 ml/5 ml H_2O. This value is not readable on the commonly used Bidwell-Sterling apparatus (0.01 ml by estimation). According to Hoffman (12) many of the chlorinated compounds mentioned above are too toxic for use in routine processing laboratories by inexperienced technicians. In addition, some of the high boiling compounds, such as tetrachloroethane (bp 146°C), may decompose products to give false readings. Methods employing capryl alcohol (13) and *n*-butyl ether (14) and the SI-MO-FAT method of Davis *et al.* (15) have not provided sufficient improvement in accuracy or time of extraction to justify supplanting the conventional usage of toluene. Other rapid methods have been reported for moisture analysis of meat, such as the high-heat oven method of Perrin and Ferguson (16), the high-heat hot plate method of Krol and Meester (17), the use of the Ultra-X apparatus by Bartels and Gerigk (18), the near infrared (IR) method of Ben-Gera and Norris (19), the nuclear magnetic resonance (NMR) analyzers (Newport of North America Inc. and Varian Corp.), radiofrequency (RF) analyzers (Moisture Register Co.), and the IR automatic spectrophotometric analyzers (Anacon Inc.).

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We investigated a number of methods which we thought might be of practical use, particularly for raw material analysis and control work during processing. As many laboratories do not have mechanical convection ovens, we decided to test AOAC method **24.003(b)** with a gravity convection air oven at temperatures from 100±2°C to 200±5°C and with drying times from 1.0 to 16 hr, using ground beef and ground frankfurters. In addition, more rapid unofficial methods were compared, using samples of ground round beef, ground beef (hamburger), frankfurters, and pork sausage. The rapid methods were the IR automatic moisture analyzer, an IR moisture determination balance, an azeotropic distillation, a specific gravity method, and a hot plate method. The low cost of apparatus was a factor in the selection.

METHODS

Sample Preparation

Fresh ground round beef, ground hamburger beef, all meat frankfurters, and pork sausage were purchased from local supermarkets. These meat products were selected to cover a wide moisture range. Samples were ground 3 times through ⅛″ plate, mixing thoroughly each time, and stored in a moisture-proof container at 3°C until analyzed within 24 hr.

Official AOAC Method

Use **24.003(a)** as official method. Dry 5 g samples 16 hr in gravity convection oven at 102±2°C.

Variations of AOAC Method

Carry out all tests in same oven as above. Dry 5 g samples at temperatures ranging from 100 to 200°C for 1 to 5 hr. Use only 1 shelf and measure temperature at that shelf height.

Other Rapid Moisture Methods

(a) Analyze in IR automatic moisture analyzer (Dynatronic Industrial Corp.) 12 g samples at 100±10°C for 0.75 to 1.00 hr.

(b) Use Ohaus Model 770 moisture determination balance. Dry 20 g samples at 105±5°C for 0.75 to 1.00 hr.

(c) For azeotropic distillation, use Bidwell-Sterling type receiver with ₮ 24/40 joints, and heat 10 g sample and 100 ml toluene on 425°C hot plate until no additional water collects after 10 min of further heating or for 60 min. (*Note:* Fat foams up receiver above 480°C.)

(d) For specific gravity method, homogenize 25–100 g ground sample 30 sec with 50 g cold 100% ethyl alcohol, and then centrifuge. Weigh fat-free supernatant in tared 10 ml volumetric flask at 20°C. % Moisture = [(50/sp gr alcohol solution) − 50] × f × 100 (f in calculation for samples weighing 25, 50, 75, or 100 g was 4, 2, 4/3, and 1, respectively).

(e) For hot plate method, use hot plate capable of maintaining 200°C as measured by dial-type bimetallic thermometer. Weigh ca 10 g sample to nearest 0.01 g into tared aluminum pan or cup, 50 mm in diameter and 40 mm high placed on sheet of 150 × 150 mm aluminum foil (tared with cup) to prevent loss from splattering. Heat 45 min at 200°C, cool 10 min in desiccator, and weigh for moisture loss.

Results

Table 1 shows the effects of variations in the temperature of the gravity convection oven and the time of sample heating on the moisture values as compared with the AOAC official method, **24.003(a)** (16 hr at 102°C), for both ground beef and processed frankfurters. These data indicate that heating a shorter time at a higher temperature (2 hr at 125°C) in a gravity convection oven yields results comparable to **24.003(a)**. Heating at higher temperatures for longer periods of time produced sample charring and erroneously high results.

Table 2 shows data obtained with the official method and with the 5 rapid methods tested at this laboratory; 3 of these methods, the IR oven, IR balance, and toluene distillation, are occasionally used in the meat industry. A good rapid method should be faster than the official method without too great a difference in accuracy and precision. An acceptable difference must be defined in terms of the processor's individual needs. As one meat product may differ in analysis from another if rapid procedures are used but not if longer approved procedures are used, correction factors may be acceptable for individual processors to compensate for differences of each product, if the per cent recovery by the rapid methods is constant. If not, then a rapid method would be useless. Results should not deviate more than ±3% of the absolute value obtained with the official method. An example of this would be: AOAC method = 50.0 moisture (absolute value), rapid method = 50.0±1.5%. If the rapid method retained good precision (0.5% between duplicates) but was constantly biased in one direction, then a correction factor could safely be used.

For ground round beef, use of the IR oven, IR

Reference to brand or firm name does not constitute an endorsement by the U.S. Department of Agriculture over others of a similar nature not mentioned.

Table 1. Effect of time and temperature in a gravity convection oven on the determination of per cent moisture in ground beef and ground processed frankfurters[a]

Sample	Oven Temp., °C	Time in Oven, hr				
		1	2	3	4	16[b]
Ground beef	102	38.6±1.6	53.6±2.5	58.4±0.7	59.0±0.4	59.2±0.4
	125	53.1±4.2	59.6±0.2	59.4±0.5	59.7±0.3	
	150	57.5±0.5	59.2±0.2	59.6±0.2	60.0±0.3	
Ground processed frankfurters	102	41.1±0	49.3±1.2	50.7±0.7	49.6±1.0	52.5±0.3
	125	45.7±2.8	52.2±0.6	53.2±0.3	52.9±0.2	
	150	51.3±0.7	53.5±0.1	53.8±0.2	54.2±0.2	
	175	53.7±0.3	54.7±0.1	54.6±0.2	55.0±0.5	
	200	54.7±0.2	55.4±0.4	56.2±0.4	56.9±0.6	

[a] Triplicate samples.
[b] Official AOAC method, sec. 24.003(a).

balance, and hot plate methods produced results comparable with those from the AOAC method. For beef hamburger, the IR oven, IR balance, and specific gravity methods were best. For frankfurters and pork sausage, the results with the IR oven and IR balance were better than those by the other 3 methods.

Practical considerations, such as the time involved, the expense of a centrifuge, homogenizer, and analytical balance, and the ability of the analyst, are factors that show that the specific gravity method is hardly suitable for routine rapid analysis.

The toluene extraction method is rapid and relatively inexpensive and does not require special training, but its precision and recovery are low. This method is widely used in small plants. As several investigators (1, 20, 21) have reported both higher and lower results by toluene distillation for determination of moisture, this method warrants a further systematic study to try to improve the precision and recovery to an acceptable level.

The 2-octanol method proposed by Everson *et al.* (13) is being tested, together with toluene distillation, using equipment modified slightly from that used in this report. Preliminary results showed that toluene started to distill more quickly, but after a 15 min distillation, the results obtained with the 2 methods agreed. Distilling for an additional 10–15 min, however, caused the moisture values with toluene to increase 1.5–2.3%, while those with 2-octanol increased only 0–1%. The results with 2-octanol were consistently lower than those with toluene by at least 1%.

The hot plate method described here was developed during this study. Krol and Meester (17) reported another hot plate method. In that study, 6 types of meats were heated at 150–170°C for 10–40 min and differences from the official methods ranged from −0.4 to +0.8%. For the types of meat tested with our method, we required heating 40–45 min at 200°C to achieve 100% recovery. We found that placement of a 150 × 150 mm aluminum sheet beneath the pan to prevent loss of fat due to splattering improved the precision of the method.

Other rapid moisture analyzers include NMR, near-IR wavelength, and radiofrequency devices. Prices ranged from $500 for RF to $11,000 for NMR. No opportunity was available to evaluate these apparatus.

In conclusion, several rapid methods exist for the analysis of moisture in meat products with a reasonable degree of accuracy and precision.

The IR balance is recommended in preference to the IR oven on the basis of economy ($100 vs. $600) and speed (45 min vs. 1 hr). Both methods produced results that were in reasonable agreement with the AOAC method for all 4 materials tested. Preliminary data indicate that a series of IR lamps, each adjusted to give the correct temperature, and a single balance can be used for multiple analyses, instead of a series of IR balances. The hot plate method is worthy of consideration because the equipment is available in most laboratories and because the results for all samples except pork sausage were within the ±3% absolute limit.

In view of the interest of the meat industry in rapid, accurate, and low-cost moisture methods, further work on a collaborative scale seems warranted.

Table 2. Comparison of moisture determination by rapid methods and the official AOAC method

Sample	Per Cent Moisture[a]					
	AOAC **24.003(a)**	IR Oven[b]	IR Balance[c]	Toluene[d]	Spec. Gravity	Hot Plate[e]
Beef round	62.80±0.10	63.50±0.57	62.95±0.58	59.83±1.86	67.73±1.04	62.65±0.21
Rec., %[f]	100	101.1	100.2	95.3	107.8	99.8
Beef hamburger	58.37±0.35	58.20±0.62	58.55±1.22	55.77±0.71	57.98±0.95	59.22±0.93
Rec., %	100	99.7	100.3	95.5	99.3	101.5
All meat frankfurters	57.52±0.28	57.93±0.29	58.08±0.66	55.75±0.69	58.93±1.52	58.37±0.32
Rec., %	100	100.7	101.0	96.9	102.5	101.5
Pork sausage	35.70±0.56	35.23±0.58	36.53±0.54	34.53±1.13	44.10±3.65	37.11±0.23
Rec., %	100	98.7	102.3	96.7	123.5	103.9

[a] For all samples N = 6 sets, except AOAC (N = 2) and specific gravity for pork sausage (N = 3); values are in per cent ± standard deviation.
[b] IR oven, 60 min at 100±10°C for all samples.
[c] IR balance, 45 min at 100°C for all samples, except frankfurters (60 min).
[d] Toluene method, 60 min at 425°C hot plate temperature.
[e] Hot plate, 45 min at 200°C.
[f] % Recovery = (rapid method/**24.003(a)**) × 100.

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