

Proposed Technique for the Determination of Both Moisture and Fat in the Same Sample of Meat or Meat Product

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The moisture content of ground meat, processed frankfurters, and uncooked pork sausage mixtures was determined within a 15–30 min time period and with a minimum of 95% recovery of moisture. Selective solvents, such as octane, nonane, toluene, xylene, ethylbenzene, and cumene were evaluated. After removal of the moisture, the residue in the flask was sampled for the fat content. A 20 ml aliquot of the cooled total solution was removed, transferred to a tared vessel, and evaporated at its boiling point under a stream of nitrogen. The oil residue was weighed and calculated as percent fat. Recoveries of fat ranged from 95 to 100% for all solvents and meat products tested except for the uncooked pork sausage mixture. The latter product yielded 80–91% recovery of fat for all solvents. To obtain a 95–100% recovery from the uncooked pork sausage mixture required an additional 15–30 min digestion. The time required for fat determinations was from 15–30 min, depending upon the boiling point of the solvent.

The importance of determining the moisture of food products is widely recognized. However, unofficial methods for moisture determination in meat (1–6), except in a few instances (7, 8), are not readily used and are not very rapid (under 1 hr). The recoveries of moisture are not always sufficiently high (above 95%) to justify continued use of these methods. In addition the analysis of meat for fat is of equal importance. The official fat analysis requires a period ranging from a minimum of 7 hr to a maximum of 22 hr. Although previous investigators have demonstrated rapid methods of fat analysis in meat, a shorter time for analysis was usually gained at the expense of accuracy or by the use of costly apparatus. The Associate Referee recently reported a reduction in the time of fat analysis from

7 to 1.5 hr (9), but an even shorter time for analysis was desired, preferably under 0.5 hr. Several investigators have proposed both moisture and fat analysis on the same sample of food product (10–14). Investigations on meat (4, 6, 15) have not reduced the time of analysis for moisture and fat on one sample below 2 hr, except when used in conjunction with the Steinlite LOS Fat Tester (7). This method required a minimum of 30 min total time to give a range of $\pm 3\%$ moisture and $\pm 3\%$ fat. The initial cost for the above method is relatively high and the recovery is low when compared to those obtained by the use of azeotropic distillation with selected solvents, followed by aliquot sampling for fat analysis as proposed in this report.

METHOD

Apparatus and Reagents

(a) *Distillation apparatus*.—Flat-bottom 250 ml flask with F 40/50 joint, 40/50 to 24/40 F reducing adapter, modified Bidwell-Sterling 10 ml distillation receiver, 30 cm full-jacketed West condenser, and heating mantle regulated by variable transformer and with full upper insulation for above flask. Adapter reduces interference from foaming and raises moisture receiver above heating mantle. Use asbestos cloth for further thermal insulation of exposed glassware up to condenser. For recovery of distilling solvent for re-use, use tared 125 ml Erlenmeyer flask with F 24/40 joint connected to small Soxhlet extractor modified with Teflon stopcock at base of thimble chamber (no thimble used) and standard Soxhlet condenser.

(b) *Reagents*.—Anhydrous commercial grade solvents listed in Table 1.

(c) *Samples used*.—Commercial ground beef, frankfurters, and uncooked sausage mixture.

Preparation of Sample

Grind 2 times (except once for ground beef) through $\frac{1}{8}$ " plate, then once through $\frac{3}{4}$ " plate at 3°C, using prechilled grinder.

Determination of Moisture

Place 10.00 ± 0.05 g meat product in boiling flask and quickly add 100.0 ± 0.5 ml solvent to prevent

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evaporation of water from sample. Assemble apparatus. Adjust heat to produce reflux level of 2" above tip of full-jacketed condenser. Time to reach boiling is ca 4-6 min, depending on bp of solvent. Lower voltage if foam, due to fat content, rises above adapter. Read volume of water distilled by reading at bottom of meniscus of solvent-water interface. Estimate volume to ± 0.02 ml. Take volume reading at time solvent first appears in receiver and then record volume at 15 and 30 min. For 10 g sample, % moisture = ml water $\times 10$. Conduct all distillation in a well ventilated hood ($> 150'$ /min velocity at a 1' opening), using great care with highly flammable and/or toxic solvents.

Determination of Fat

Transfer solvent remaining in distilling receiver with disposable pipet to Erlenmeyer flask. Cool flask and transfer 20 ml decanted solution with class A pipet to tared 100 ml tall-form beaker containing boiling chips. Evaporate solvent at its bp on hot plate (ca 200°C) with stream of nitrogen directed on solution until only fat remains (6-15 min, depending on bp of solvent.) Immediately remove beaker to prevent decomposition of fat. Cool beaker in pre-chilled aluminum chamber (16). Weigh beaker and calculate % fat = (g fat in aliquot $\times 5 \times 100$)/10. Alternatively, to determine fat and reclaim solvent for re-use, use modified Soxhlet apparatus. Transfer 40 ml fat-solvent mixture to tared ∇ 24/40 Erlenmeyer flask containing boiling chips. Connect to Soxhlet apparatus and distill until no further solvent condenses. Remove any remaining solvent in flask as in above procedure. Cool in prechilled desiccator, weigh, and calculate as above, making allowance for larger aliquot.

Results and Discussion

Results of the analysis for moisture and fat from a single sample of 2 lots of ground beef are shown in Table 1 and for frankfurter and sausage in Table 2. All samples were analyzed in duplicate for moisture and in quadruplicate for fat.

After analysis of ground beef lot A, the use of the following solvents was discontinued: pentane, hexane, heptane, cyclohexane, tetrachloroethylene, and 1,1,1-trichloroethane. The results obtained were too low (below 95% recovery) for moisture except for tetrachloroethylene, which required custom-made moisture receivers. The results obtained for fat were also too low in each of the above solvents except heptane.

The remainder of the experiment was conducted with octane, nonane, toluene, *m*-xylene, ethylbenzene, and cumene. For ground beef, the

solvents best suited for rapid analysis of moisture and fat from the same sample in less than 60 min total distillation, evaporation, and cooling time were octane, nonane, *m*-xylene, ethylbenzene, and cumene.

Processed frankfurters yielded good recoveries for moisture and fat with the use of octane, nonane, toluene, *m*-xylene, ethylbenzene, and cumene, except for fat recoveries using nonane. No explanation is possible at this time.

For uncooked sausage mixture the recoveries for moisture were good; however, the recoveries for fat were only 90.2-91.4%. The uncooked high fat emulsion required 45 min digestion time to attain 95.4% recovery of fat for xylene, 94.3% for cumene, and 95.7% for toluene. A 60 min digestion time yielded 97.8, 95.7, and 98.7% recoveries, respectively. Thus, for sausage an additional increase of 15-30 min in the total time is necessary for fat analysis.

Moisture receivers should be calibrated by the analyst with distilled water at a given temperature and the volume readings of the moisture distilled from samples should be made at the same temperature.

Recommendations

It is therefore recommended that the following solvents be considered for use when a rapid method for the analysis of moisture and fat is required: nonane in place of octane for higher recovery; *m*-xylene in place of toluene; and ethylbenzene or cumene, depending upon the meat products to be analyzed.

It is further recommended that this rapid technique be considered for collaborative study as an alternative method for general analysis and not for the purpose of superseding existing AOAC methods.

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Table 1. Comparison of various solvents for use in the rapid determination of moisture and fat in the same sample of ground beef

Solvent	Ground Beef, Lot A				Ground Beef, Lot B			
	Moisture, %		Fat, %		Moisture, %		Fat, %	
	Found, ± sd ^a	Rec. ^b	Found, ± sd	Rec.	Found, ± sd	Rec.	Found, ± sd	Rec.
Pentane	1.1±0.5	1.9	20.19±0.04	88.4				
Hexane	14.8±1.1	25.3	15.40±0.69	67.4				
Heptane	48.8±2.5	83.6	22.03±0.98	96.5				
Cyclohexane	22.3±1.8	38.2	19.83±3.60	86.8				
Octane	56.7±1.2	97.2	22.44±1.26	98.3	51.5±2.1	96.8	27.39±0.91	95.7
Nonane	58.0±0.2	99.4	22.03±0.37	96.5	53.5±0.0	100.5	27.82±0.69	97.2
Toluene	54.9±1.9	94.1	21.35±0.58	93.5	50.7±2.6	95.2	26.57±1.02	94.6
<i>m</i> -Xylene	57.7±0.9	98.9	21.91±0.09	95.9	53.5±0.7	100.5	27.66±0.53	96.6
Ethylbenzene	58.1±0.1	99.6	21.39±0.58	93.7	52.8±1.1	99.1	27.22±0.09	95.1
Cumene	58.4±0.5	100.1	22.12±0.38	96.8	53.5±0.7	100.5	27.58±0.54	96.3
Butyl ether	58.4±0.2	100.1	20.58±0.39	90.1				
Tetrachloroethylene	57.5±0.0	98.6	20.48±2.90	89.7				
1,1,1-Trichloroethane	37.4±0.5	63.7	21.22±1.00	92.9				

^a sd = Standard deviation; n = 2 for all moisture samples, n = 4 for all fat samples except nonane (n = 2); 20 ml aliquot for all samples except nonane (40 ml aliquot).

^b % Recovery = (% found by rapid method/% found by official method) × 100.

Table 2. Comparison of various solvents for use in the rapid determination of moisture and fat in the same sample of meat product

Solvent	Frankfurter				Sausage			
	Moisture, %		Fat, %		Moisture, %		Fat, %	
	Found, ± sd ^a	Rec. ^b	Found, ± sd	Rec.	Found, ± sd	Rec.	Found, ± sd	Rec.
Octane	53.7±1.2	98.4	28.02±0.05	98.3	44.5±1.4	97.9	36.57±0.15	89.4
Nonane	54.5±0.0	99.8	25.44±0.28	89.2	45.3±0.4	99.7	37.39±0.84	91.4
Toluene	52.3±0.4	95.8	27.60±0.70	96.8	43.0±2.1	94.6	32.72±2.58	80.2
<i>m</i> -Xylene	53.4±0.9	97.8	28.07±0.07	98.5	45.4±0.2	99.9	37.26±0.47	91.1
Ethylbenzene	54.6±0.6	100.0	27.96±0.47	98.1	45.3±0.4	99.7	36.30±0.45	88.7
Cumene	55.3±0.4	101.3	28.72±0.50	100.2	45.7±0.1	100.6	36.95±0.21	90.3

^{a, b} See footnotes, Table 1.

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