Collaborative Study of Modified AOAC Moisture Method and Other Rapid Moisture Methods for Analysis of Meat and Meat Products

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Three new methods for determining moisture content of ground beef, frankfurters, and fresh pork sausage were tested collaboratively by 10 laboratories. Results were compared with those obtained by AOAC official methods. The new methods involved: 1) use of a gravity oven at 125°C in place of a mechanical convection oven, 2) exposure of samples to IR radiation for 40 min, and 3) azeotropic distillation for 30 min, using either cumene, *m*-xylene, or 2-octanol. Determinations of moisture content by the use of a gravity oven and IR radiation were in agreement with the official methods, but azeotropic distillation with any of the 3 solvents gave lower values for all 3 types of meat product. The expected standard deviation, s_e, averaged for the 3 types of meat product in per cent moisture was as follows: gravity oven, ±0.65; IR radiation, ±1.18; and azeotropic distillation, ±0.79, ±0.86, and ±0.98, using cumene, *m*-xylene, and 2-octanol, respectively. For the official methods, ±0.64% moisture was obtained for s_e. An amendment to the official final action method for the determination of moisture in meat and meat products, 24.003(b), was adopted as official first action to provide for the optional use of an air oven.

The official methods for moisture analysis of meat and meat products serve as standards for the meat industry but alternative, more rapid methods are needed for process and quality control. Many unofficial methods in use have not been evaluated collaboratively so that accuracy and precision of these methods have not been satisfactorily determined. Pettinati *et al.* (1) stated that the ideal rapid method should be simple to perform, inexpensive, and reasonably accurate. With the need for rapid methods in mind, several analytical techniques which were evaluated in our laboratory and reported in previous communications (2, 3) were tested collaboratively with 10 laboratories. As with the previous studies, data obtained with the new methods were compared with those obtained by official methods (4). Moisture analyses were obtained by the following methods: (*1*) **24.003(a)**; (*2*) **24.003(b)**; (*3*) drying samples 2 hr in a gravity oven at 125°C (similar to method *2* above, except that a large gravity oven which is more commonly found in laboratories was used in place of a mechanical convection oven); (*4*) exposure of samples to infrared radiation on the Ohaus moisture balance, or similar apparatus, for 40 min; (*5*, *6*, and *7*) azeotropic distillation for 30 min using cumene, *m*-xylene, and 2-octanol, respectively, to determine moisture content directly as volume of water condensate.

The solvents selected for azeotropic distillation were those reported to be the most suitable in the extensive study by Cohen and Kimmelman (3). Meat and meat products containing 35 to 62% moisture were evaluated in the initial studies (2, 3). In this collaborative study the moisture content range was 42 to 63%.

Collaborative Study

A 100 lb lot of commercial grade lean beef was cut into approximately 1″ cubes and mixed thoroughly. The entire quantity was then passed 3 times through a meat grinder, using a ⅛″ diameter cutting plate, and mixed thoroughly after each grind.

A 100 lb lot of commercial grade all meat frankfurter was passed twice through a meat grinder, using a ⅛″ diameter cutting plate, and mixed thoroughly after each grind.

A 100 lb lot of commercial grade, uncooked

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pork sausage meat was prepared the same as frankfurter above.

Samples were prepared in a constant temperature (7–8°C) processing room. Individual samples for collaborators were packed and vacuum sealed in polyethylene bags. The sample bags for each collaborator were placed in a styrofoam shipping container and stored in a walk-in freezer overnight. Crushed Dry Ice was then placed in each container with the samples and the containers were sealed and shipped by air to each laboratory. The laboratories received the frozen samples within 24 hr. Collaborators were instructed to (*1*) keep the container contents frozen until analyzed, (*2*) thaw samples overnight in a refrigerator or under running tap water, (*3*) mix the contents of each plastic bag thoroughly before sampling, (*4*) store the sample in a sealed container in the refrigerator when not in use, and (*5*) use the sample within 24 hr of complete thaw.

The 10 collaborators were requested to make 6 replicate determinations on each of the 3 samples. If unable to comply, they were either to describe the deviation from the procedure or to telephone the Associate Referee for further instruction before proceeding with the analyses. In methods 1–3, collaborators were asked to use only one shelf in the oven which had been preset for the required temperature at that shelf height.

Modification of 24.003(b)

Principle

The official procedure for drying samples specifies a mechanical convection oven which is more expensive than the more commonly available and less expensive gravity oven. Five g samples are dried in a gravity oven at the same temperature (125°C) as **24.003**(b) and for the same time (2 hr).

Apparatus

(a) *Oven.*—Gravity oven capable of maintaining ca 125°C on single shelf which is set at determined height within oven, preferably larger than 2½ cu. ft and with ≥2" of insulation.

(b) *Pans.*—Aluminum, drying, ≥50 mm diameter and ≤40 mm deep.

Method 1

Proceed as in **24.003**(a). Weigh ca 5 g samples to nearest 0.001 g. Dry samples in 100–102°C oven for 18 hr.

Method 2

Proceed as in **24.003**(b). Dry samples in mechanical convection oven 2 hr at 125°C.

Method 3

Using gravity oven, dry samples as in Method 2. If mechanical convection oven is the only type available, use it with blower off.

Infrared Radiation (Ohaus Moisture Balance)

Principle

The IR lamp (250 or 375 w) is positioned so that it provides 125°C at drying surface of sample flattened on 2–4" diameter pan of triple beam, single pan balance.

Apparatus

(a) *Moisture balance.*—Ohaus Model No. 770 (Ohaus Scale Corp., 29 Hanover Rd., Florham Park, N.J. 07932), or IR radiation lamp, 250 or 375 w and triple beam, single pan balance that can weigh ±0.1 g.

(b) *Pans.*—Aluminum, drying, 2–4" diameter.

Procedure

Use Ohaus moisture balance Model No. 770 or any IR lamp (250 or 375 w) and balance. Set height of lamp to produce 125°C at surface of pan placed either on asbestos pad or on pan of triple beam, single pan balance which can weigh to ±0.1 g. Flatten ca 10 g sample rapidly and evenly on 2–4" diameter aluminum pan. (The bottom of a beaker may be used to flatten sample.) Weigh to nearest 0.1 g and place weighed pan and contents under lamp. As sample dries, weigh residue and record weight at intervals of 10, 15, 20, 30, 40, and 45 min. Count heating time only when pan is in drying position and lamp is on. Calculate weight loss as per cent moisture.

Azeotropic Distillation

Principle

A solvent immiscible with water, or of low mutual solubility, is boiled in flask containing meat or meat product. Water-cooled condenser above flask cools azeotropic vapor distilled from flask which condenses and separates into 2 immiscible phases which drop into receiver tube. Solvent portion of condensate floats above water layer, overflows, and returns to boiling flask. Volume of water in calibrated tube is direct measure of moisture content of sample. For example, if 10.00 g sample is added to solvent in boiling flask and volume of water in receiver tube reads 5.00 ml, then moisture content of sample is 50%.

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

This report of the Associate Referee, E. H. Cohen, was presented at the 86th Annual Meeting of the AOAC, Oct. 9–12, 1972, at Washington, D.C.

Table 1. Collaborative moisture analysis results by method 24.003(a) for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %									
	Ground beef		Frankfurter		Pork sausage		Ranked results			
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	63.02	0.554	57.14	0.234	42.17	0.263	5	6	4	15
2	62.70	0.434	57.47	0.163	41.65	0.187	7	3	7	17
3	61.22	0.392	56.85	0.187	42.28	0.264	10	9	3	22
4	64.25	0.147	57.05	0.282	41.20	0.342	2	8	10	20
5	63.71	0.089	57.53	0.090	42.68	0.115	3	1	1	5
6	62.96	0.551	57.49	0.358	41.59	0.423	6	2	8	16
7	63.25	0.194	57.16	0.093	41.96	0.230	4	5	5	14
8	62.62	0.322	57.13	0.253	41.95	0.141	8	7	6	21
9	62.38	0.435	56.41	0.085	41.38	0.359	9	10	9	28
10	64.29	0.176	57.32	0.154	42.52	0.345	1	4	2	7

Final Results, Per Cent Moisture

Statistic	Beef	Franks	Pork	Average
Grand mean, $\bar{\bar{x}}$	63.04	57.15	41.94	—
Range	−2.44 to 1.42	−0.87 to 0.76	−1.18 to 0.93	—
s_d	0.918	0.338	0.486	0.581
s_r	0.366	0.209	0.283	0.287
s_b	0.906	0.327	0.472	0.569

Apparatus

(a) *Flask.*—Boiling, flat bottom, short single neck, pourout, 250 ml, ₸ 40/50 outer joint (SGA Scientific, Inc., 735 Broad St., Bloomfield, N.J. 07003, No. JF-2090, or equivalent).

(b) *Adapter.*—Straight, reducing, ₸ 24/40 outer joint at top and ₸ 40/50 inner at bottom (SGA Scientific, Inc., No. JA-1830, or equivalent).

(c) *Receiver.*—Distilling, modified Bidwell-Sterling, 10 ml, graduated in 0.1 ml, with solvent return tube, ₸ 24/40 outer joint at top and ₸ 24/40 inner joint at bottom (SGA Scientific, Inc., No. JD-7820, or equivalent).

(d) *Condenser.*—West, Ful-jak, with water-cooled ₸ 24/40 outer joint at top and water-cooled ₸ 24/40 inner joint having drip tip at bottom, 30 cm jacket length (SGA Scientific, Inc., No. JC-8650, or equivalent).

(e) *Heating mantle.*—For 250 ml flask, with full upper insulation around base of joint, 115 w (Electro-Thermal Sales Corp., 13 Landing Rd., Roslyn, N.Y. 11576, No. MG-7803, or equivalent).

(f) *Transformer.*—Variable, 1.75 amp, 120 v input, for heating mantle (Lapine Scientific Co., 6001 S. Knox Ave., Chicago, Ill. 60629, No. 126-78, or equivalent).

(g) *Asbestos cloth.*—For thermal insulation of flask joint, receiver tube arm, and base of condenser (Fisher Scientific Co., 191 S. Gulph Rd., King of Prussia, Pa. 19406, No. 1-474, or equivalent).

(h) *Solvents.*—Cumene, *m*-xylene, and 2-octanol, Baker grade purity (J. T. Baker Chemical Co., 222 Red School Lane, Phillipsburg, N.J. 08865).

Method 5

Weigh 10.00±0.01 g sample onto 2 × 2" square of aluminum foil and drop foil and contents into 250 ml boiling flask. Pour ca 100 ml cumene into flask. Place flask and contents into heating mantle. Connect reducing adapter to neck of flask, moisture receiver tube to adapter, and water-cooled condenser to receiver tube. Use asbestos cloth to thermally insulate neck of flask, receiver arm, and base of condenser. Connect heating mantle to variable transformer and heat at maximum rate until flask contents begin boiling. Use transformer to control boiling rate just below that which causes solvent-fat bubbles to rise into reducing adapter. Estimate water volume from the graduated markings on the receiver tube to nearest 0.02 ml after 5, 10, 15, 20, and 30 min and record readings.

Methods 6 and 7

Proceed as in Method 5, using *m*-xylene and 2-octanol, respectively, in place of cumene.

Statistical Treatment of Data

Analytical results obtained by the 10 collaborators were treated for error analysis following the procedures suggested by Youden (5–7) and the ASTM (8). Tables 1–7 list the mean values $\bar{x}$ of each collaborator's replicate analyses and the grand mean values $\bar{\bar{x}}$ determined for each sample. Adjacent to each column of means is listed the collaborator's random error s_r which was calculated from the differences between the replicate values and the mean value.

Table 2. Collaborative moisture analysis results by method 24.003(b) for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %						Ranked results			
	Ground beef		Frankfurter		Pork sausage					
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	61.58	0.454	55.83	0.324	42.02	0.264	10	10	5	25
2	63.05	0.414	57.33	0.103	41.75	0.295	4.5	4	9	17.5
3	63.05	0.288	57.08	0.306	42.37	0.308	4.5	6	3	13.5
4	63.04	0.321	57.75	0.237	42.90	0.266	6	1	1	8
5	64.41	0.398	57.62	0.114	42.32	0.337	1	2	4	7
6	62.44	0.339	57.08	0.198	41.42	0.309	9	7	10	26
7	63.39	0.245	57.27	0.151	41.86	0.398	2	5	7	14
8	62.94	0.421	57.02	0.103	41.79	0.336	7	8	8	23
9	62.52	0.054	56.66	0.221	41.96	0.429	8	9	6	23
10	63.09	0.078	57.43	0.188	42.54	0.380	3	3	2	8

Final Results, Per Cent Moisture

Statistic	Beef	Franks	Pork	Average
Grand mean, $\bar{\bar{x}}$	62.95	57.11	42.09	—
Range	−2.26 to 1.91	−1.71 to 1.06	−1.15 to 1.09	—
s_d	0.722	0.548	0.438	0.570
s_r	0.329	0.209	0.336	0.292
s_b	0.709	0.541	0.416	0.556

Table 3. Collaborative moisture analysis results by modified method 24.003(b) for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %						Ranked results			
	Ground beef		Frankfurter		Pork sausage					
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	63.11	0.320	56.88	0.565	42.04	0.309	6	8	6	20
2	59.33	1.908	55.95	0.718	40.57	1.127	10	10	10	30
3	63.60	0.253	56.93	0.216	42.42	0.256	4	7	3.5	14.5
4	64.94	0.684	57.88	0.281	42.15	0.396	2	1	5	8
5	65.23	0.235	57.54	0.097	42.56	0.153	1	2	2	5
6	63.31	0.215	57.45	0.306	41.92	0.326	5	4	8	17
7	64.36	0.321	57.22	0.201	42.01	0.307	3	6	7	16
8	62.66	0.272	57.30	0.240	42.42	0.438	8	5	3.5	16.5
9	62.23	0.599	56.79	0.407	41.90	0.243	9	9	9	27
10	63.00	0.225	57.51	0.274	42.86	0.447	7	3	1	11

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	63.18	57.15	42.08	—
Range	−6.38 to 2.61	−2.15 to 1.14	−2.88 to 1.20	—
s_d	1.663	0.540	0.617	0.940
s_r	0.704	0.374	0.476	0.519
s_b	1.638	0.518	0.586	0.914
Final Results,[a] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	63.60	57.28	42.25	—
Range	−2.32 to 2.19	−1.29 to 1.01	−0.94 to 1.03	—
s_d	1.029	0.359	0.330	0.573
s_r	0.383	0.314	0.332	0.344
s_b	1.017	0.336	0.301	0.552

[a] Data from Collaborator 2 omitted.

Table 4. Collaborative moisture analysis results by infrared radiation drying method for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %						Ranked results			
	Ground beef		Frankfurter		Pork sausage					
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	63.15	0.855	57.07	0.378	41.90	0.573	6	8	8	22
2	62.67	0.516	57.67	0.516	42.50	0.548	8	5	6	19
3	63.33	0.961	57.85	0.695	43.50	0.322	3	3	2	8
4	63.27	0.781	57.33	0.280	42.72	0.500	4	6	5	15
5	65.50	0.548	59.67	0.816	44.50	0.548	1	1	1	3
6	63.00	0.316	57.72	0.567	42.75	0.418	7	4	4	15
7	57.67	2.168	55.80	1.094	39.35	1.231	10	10	10	30
8	63.17	0.408	57.23	0.898	42.27	0.766	5	7	7	19
9	63.92	0.499	58.59	0.720	43.17	0.458	2	2	3	7
10	61.20	1.921	57.02	0.581	41.07	0.956	9	9	9	27

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.69	57.59	42.37	—
Range	−7.29 to 3.31	−3.29 to 3.41	−4.37 to 2.63	—
s_d	2.059	1.022	1.406	1.496
s_r	1.083	0.694	0.684	0.821
s_b	2.011	0.982	1.378	1.457
Final Results,[a] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	63.24	57.79	42.71	—
Range	−3.54 to 2.76	−1.79 to 3.21	−3.01 to 2.29	—
s_d	1.126	0.853	0.977	0.986
s_r	0.884	0.634	0.593	0.704
s_b	1.066	0.813	0.947	0.942

[a] Data from Collaborator 7 omitted.

Collaborators were ranked for each sample by assigning values 1 to 10 to the corresponding means arranged from highest to lowest result. Youden presented approximate 5% 2-tail limits for ranking scores (ref. 5, Table 7). For this study there was only a 5% chance that a collaborator would not score between 4 and 29, in the absence of systematic error. The calculated scores are listed for each of the moisture methods in Tables 1–7.

Total standard deviation s_d was calculated from the differences between each collaborator's mean and the grand mean. The random (within-laboratory) error s_r contribution by all laboratories for a given method was calculated from the collaborator's replicate differences from its mean, summed for all laboratories. Systematic (between-laboratory) error s_b was calculated by difference from the total standard deviation and random error as follows:

$$s_b^2 = s_d^2 - s_r^2/k$$

where k is the number of replicates. These summary statistics were listed twice in Tables 3–7. Intermediate results were calculated from all data and final results were calculated after omitting outlier data.

An important part of the error analysis consisted of judging outlier data in order to obtain results representative of a consensus of the collaborators. Inspection of the tabulated random error values and the collaborators' scores indicated the presence of outlier data. To confirm which datum was to be rejected, a statistical outlier test based on an assumed underlying normal distribution was selected from the methods recommended by the ASTM. The sample coefficient of kurtosis for 2-sided tests was applied at the 1% level of significance.

Since "true" moisture content of the samples was not known, linear regression was used to compare results on all 3 samples by each of methods 2–7 with those by method 1. An F-test was then performed by comparing the ratio of the sum of squares about each linear regression line constructed and the sum of squares about a line for exact agreement. A calculated value which exceeded the tabular F-value indicated that the

Table 5. Collaborative moisture analysis results by the azeotropic distillation method using cumene for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

Coll.	Ground beef $\bar{x}$	Ground beef s_r	Frankfurter $\bar{x}$	Frankfurter s_r	Pork sausage $\bar{x}$	Pork sausage s_r	Ranked results B	Ranked results F	Ranked results P	Coll. score
1(AR)	62.25	0.455	56.53	0.468	41.53	0.350	6	7	1.5	14.5
2	62.97	0.841	57.92	1.262	40.60	0.540	3	1	8	12
3	62.23	0.408	56.60	0.438	41.53	0.372	7	4.5	1.5	13
4	62.70	0.746	56.32	1.107	41.22	1.093	5	9	6	20
5	63.12	0.436	56.60	0.200	41.30	0.616	2	4.5	5	11.5
6	62.75	0.524	56.58	0.417	40.28	0.256	4	6	9	19
7	64.70[a]	0.616	56.35	0.609	41.37	0.665	1	8	4	13
8	61.87	0.501	56.97	0.294	40.87	0.432	9	2	7	18
9	62.07	0.139	56.90	0.194	41.37	0.152	8	3	3	14
10	56.10	4.025	54.85	0.771	39.60	1.313	10	10	10	30

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.08	56.56	40.97	—
Range	−11.08 to 2.92	−2.66 to 3.74	−2.97 to 1.83	—
s_d	2.246	0.757	0.634	1.213
s_r	1.388	0.673	0.676	0.913
s_b	2.159	0.705	0.571	1.145
Final Results,[b] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.74	56.75	41.12	—
Range	−1.54 to 2.26	−1.75 to 3.55	−1.52 to 1.68	—
s_d	0.847	0.487	0.439	0.592
s_r	0.551	0.662	0.562	0.592
s_b	0.811	0.405	0.375	0.531

[a] Collaborator 7 reported 5 replicate values.
[b] Data from Collaborator 10 omitted.

method being compared did not give the same analysis values.

Conclusions on the merit of each method were based in part on the expected standard error s_e, calculated from the error components obtained from all collaborative data as follows:

$$s_e^2 = s_b^2 + s_r^2/k$$

where k represents the number of replicate analyses to be performed. For these calculations, k was set equal to 1. The systematic and random error values were averaged for all 3 samples to calculate s_e for each method.

Results and Discussion

The numerical values of standard deviation, random error, and systematic error obtained for the 3 meat product samples were found to be disproportional to sample moisture level. Dissimilarities in the values of the error components among the 3 samples were more likely due to the fineness of grind of the samples. Frankfurters, the most finely comminuted product, generally yielded the smallest random and systematic errors, and the higher error usually associated with moisture analysis of ground beef is evident in the results for each method.

Method 1. **24.003(a)**.—Evaluation of results by this method served as the basis for comparison with the new methods. Table 1 presents the reduced data from 180 single determinations. The value of ±0.637% moisture was obtained for s_e.

Method 2. **24.003(b)**.—This method was found to be equivalent to Method 1 in all respects. Table 2 shows the reduced data for 180 single determinations. The low F-value, Table 8, also shows the equivalence of the 2 methods. The value of ±0.628% moisture for s_e agrees well with that of Method 1.

Method 3. Modified **24.003(b)**.—Data for this method in Table 3, reduced for 180 single determinations, indicated Laboratory 2 to be a probable outlier by the high random error and a score of 30. After we omitted data from Laboratory 2, the final results were equivalent to those of Methods 1 and 2. The low F-value from Table 8 indicates that this method determines the same per cent moisture as Method 1. The value of ±0.650% moisture for s_e agrees well with that of methods 1 and 2.

Table 6. Collaborative moisture analysis results by the azeotropic distillation method using *m*-xylene for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

Coll.	Ground beef $\bar{x}$	Ground beef s_r	Frankfurter $\bar{x}$	Frankfurter s_r	Pork sausage $\bar{x}$	Pork sausage s_r	Ranked results B	Ranked results F	Ranked results P	Coll. score
1(AR)	62.33	0.774	56.95	0.266	41.33	0.339	6	2	3	11
2	62.70	0.721	57.25	0.715	40.75	0.274	3	1	8	12
3	62.37	0.388	56.63	0.388	41.67	0.372	4	3	1	8
4	62.33	1.211	56.40	1.095	40.93	0.372	5	4	5.5	14.5
5	63.57	0.476	55.47	0.789	40.93	0.367	2	9	5.5	16.5
6	60.75	0.689	55.40	1.133	41.33	0.427	9	10	2	21
7	64.73	0.575	56.27	0.809	40.85	0.663	1	7	7	15
8	62.20	0.456	56.33	0.273	40.70	0.518	8	5	9	22
9	62.24	0.193	56.29	0.244	41.25	0.153	7	6	4	17
10	59.92	1.269	55.93	1.329	40.43	1.346	10	8	10	28

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.31	56.29	41.02	—
Range	−4.31 to 2.89	−2.29 to 2.31	−3.02 to 0.98	—
s_d	1.324	0.586	0.370	0.761
s_r	0.750	0.800	0.577	0.709
s_b	1.289	0.486	0.286	0.687
Final Results,[a] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.58	56.33	41.08	—
Range	−2.58 to 2.62	−2.33 to 2.27	−1.28 to 0.92	—
s_d	1.084	0.607	0.326	0.673
s_r	0.668	0.717	0.410	0.599
s_b	1.049	0.531	0.280	0.621

[a] Data from Collaborator 10 omitted.

Method 4. Infrared Radiation.—Table 4 shows the reduced data from 180 single determinations. A score of 30 and the consistently high random error by Laboratory 7 indicated the reasons those data were omitted to obtain the final results for this method. Whereas mean moisture values were estimated correctly by this method, as indicated by the F-value in Table 8 being nonsignificant, both systematic and random errors were about twice those of Methods 1–3. As a consequence, the value of ±1.176% moisture for s_e is large also.

Methods 5–7. Azeotropic Distillation with Cumene, m-Xylene, and 2-Octanol.—Tables 5 (cumene), 6 (*m*-xylene), and 7 (2-octanol) show the reduced data for the 3 methods, from 179, 180, and 168 single determinations, respectively. These 3 methods are discussed together because of their similarities in performance and in the results. Outlier data from one laboratory were omitted to obtain final results for all 3 methods. Table 8 shows significant F-values for the 3 methods. This indicated that azeotropic distillation did not give the same mean moisture values as the official methods. Data for the regression equations of these methods in Table 8 indicated that the source of deviation was due to the negative intercepts. Thus, the methods showed a bias, or a tendency to underestimate moisture determination by an average of 1%. The average estimates of errors are slightly higher than methods 1–3. The value of the expected standard error for each method was calculated to be: cumene, s_e = ±0.795% moisture; *m*-xylene, s_e = ±0.863% moisture; 2-octanol, s_e = ±0.977% moisture.

Comments by Collaborators

Method 2.—Collaborator 3 dried samples 4 hr with blower on instead of 2 hr, by mistake.

Method 3.—Collaborator 2 reported that the gravity oven on hand maintained 125±5°C. Collaborator 3 approved of this method as being equivalent to the official moisture method. Collaborators 4, 5, 7, and 8 used mechanical convection ovens with the blower off. Collaborator 5

Table 7. Collaborative moisture analysis results by the azeotropic distillation method using 2-octanol for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %						Ranked results			
	Ground beef		Frankfurter		Pork sausage					
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	61.83	0.258	56.27	0.547	40.60	0.363	6	3.5	6	15.5
2	61.55	0.505	55.83	0.493	40.00	0.548	8	6	9	23
3	62.10[a]	0.141	56.50[a]	0.424	41.40[a]	0.000	5	2	2	9
4	62.13	0.787	56.27	1.435	40.70	0.797	4	3.5	5	12.5
5	63.85	0.259	57.87	0.665	42.58	0.426	1	1	1	3
6	62.97	0.638	55.77	1.263	41.23	0.575	2	7	3	12
7	61.82	0.842	55.37	0.493	40.30	0.687	7	9	7	23
8	62.37	0.557	56.17	0.320	40.77	0.388	3	5	4	12
9	60.72	0.378	55.61	0.654	40.22	0.252	9	8	8	25
10	59.08	1.399	54.52	1.386	37.00	2.757	10	10	10	30

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	61.84	56.02	40.48	—
Range	−3.84 to 2.36	−3.52 to 2.78	−6.48 to 2.82	—
s_d	1.277	0.865	1.431	1.191
s_r	0.702	0.895	1.036	0.878
s_b	1.238	0.767	1.354	1.120
Final Results,[b] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.15	56.18	40.87	—
Range	−1.95 to 2.05	−1.98 to 2.62	−1.87 to 2.43	—
s_d	0.882	0.728	0.787	0.799
s_r	0.561	0.815	0.526	0.634
s_b	0.846	0.630	0.751	0.743

[a] Collaborator 3 reported duplicate values only.
[b] Data from Collaborator 10 omitted.

Table 8. Computations for comparison of moisture methods to method 24.003(a)

Moisture method number	Data omitted, coll. no.	Values from linear regression				Slope = 1 intercept = 0 sum of squares, SS_E	F
		Intercept	Slope	Sum of squares, SS_E	Corr. coeff.		
2	none	0.840	0.985	14.82	0.996	15.4	0.57
3	2	0.046	1.005	11.26	0.997	14.1	3.10
4	7	2.051	0.972	36.37	0.991	46.1	3.33
5	10	−1.761	1.025	11.86	0.997	18.2	6.62[a]
6	10	−1.395	1.014	19.84	0.995	31.3	7.26[a]
7	10	−1.357	1.008	14.54	0.996	36.8	19.22[a]

[a] Significant at the 1% level.

found that, with the blower off, the oven required 1¾ hr to recover 125°C after samples were placed on the shelf (this may indicate a very small oven or very thin-walled insulation).

Method 4.—Collaborator 3 used an Ultra-X infrared lamp, 250 w, supplied by Koch Equipment Co. and a Mettler Model P120N balance and felt that the procedure had significant value when the equipment is properly set up. Collaborator 6 indicated that the method showed good correlation with other methods for determining moisture and recommended that analyses be performed in a constant-temperature enclosure because laboratory temperature rise during the day caused the temperature at the pan to rise about 8°C. Collaborator 7 found that 2 days of on and off adjusting were required to find the distance that gave a fairly stable 125°C. (The Associate Referee found 4 hr to be sufficient.)

Methods 5, 6, and 7.—Collaborator 2 found that irregular menisci were obtained and the volume could not be read accurately. Water bubbles formed in the solvent layer adhered to the walls of the receiver tube, and would not drop to the water layer without physical assistance. Collaborator 3 used a hot plate successfully instead of a heating mantle and timed distillation from start of condensate formation. Collaborator 4 felt that azeotropic distillation is impractical due to erratic results that are obtained, toxicity of solvents, and problem of solvent disposal. Collaborator 5 found that 20 min was sufficient to determine moisture content of frankfurters and sausage, while ground beef required 30 min. Erlenmeyer flasks of 250 ml in place of round flasks and hot plates in place of heating mantles were used but a problem was encountered with foam above the boiling solvent which rose above the flask neck. However, the collaborator felt the rapid methods are worthy of consideration due to reasonable degree of accuracy, speed of analysis, and availability of required equipment. Collaborator 7 found that water droplets were trapped in the solvent layer of receiver. These were dislodged with a wire. The problem was more pronounced with pork samples than with the other 2 products. Collaborator 10 found that water droplets clung to the sides of the condenser and that the frankfurter sample received was insufficient to complete all analyses.

Conclusions

The unofficial guidelines for control work on both finished product and meats prior to processing were discussed by Cohen (2). The guidelines for all products were stated to be a desired accuracy of ±1% of the moisture content obtained by official analysis. A precision of ±1% of the determined value is desired for finished product and ±3% may be tolerated for meats prior to processing.

A comparison of the statistics determined in this collaborative study with the above guidelines shows that moisture determination by the modified official method gave results equivalent to those obtained by the existing official methods. The infrared radiation method estimated mean moisture content accurately but precision was too low for the method to be useful for finished product except where a rapid method without high precision is desired. Azeotropic distillation estimated moisture about 1% low and gave a standard error higher than the official methods. Cumene and *m*-xylene in azeotropic distillation yielded more accurate and more precise results than 2-octanol and therefore are the preferred solvents. As with infrared radiation, azeotropic distillation is not precise enough for finished products except where rapid analysis would be desired without high accuracy and precision.

Youden (6) stated that rarely will a new method excel an old method in every particular. However, for processors and control agencies, the rapid methods, i.e., moisture balance and azeotropic distillation, are recommended where high accuracy and precision are not mandatory.

Relative to the collaborative study, a comment is directed toward those who may initiate a similar type of study. While 60 results were reported for the most part for each product sample (6 replicates × 10 laboratories), each group of data led to error terms as follows: s_r was estimated with 50 degrees of freedom and s_b with 9 degrees of freedom. The error term s_b is important because a collaborative test attempts to estimate the population of all laboratories. Therefore, in a similar study s_b is determined more representatively by having 30 laboratories run duplicate determinations in order to estimate s_r with 30 degrees of freedom and s_b with 29. However, it is only conjecture that s_b in the present study is not representative and would be any different in a sampling of 30 laboratories since s_b is always the major error factor, being 2–3 times s_r in many cases.

Recommendations

Based on the results of this collaborative study, it is recommended—

(1) That a single shelf in a gravity oven be adopted as official first action as an alternative method of drying in 24.003(b), which presently specifies a mechanical convection oven.

(2) That infrared radiation drying for 40 min be adopted as official first action as an alternative, rapid screening method for analysis of meats prior to processing or for analysis of processed meat products when rapid analysis is more important than a high degree of accuracy and precision.

Recommendation (1) of the Associate Referee was approved by the General Referee and by Subcommittee C and was adopted by the Association; *see* (1973) *JAOAC* **56**, 399–400. The Association felt that a study of current screening methods should be conducted before additional screening methods are adopted.

(3) That azeotropic distillation for moisture determination in 30 min with *m*-xylene or cumene be adopted as official first action as an alternative, rapid screening method for analysis of meats prior to processing or for analysis of processed meat products when rapid analysis is more important than a high degree of accuracy and precision.

Acknowledgments

The Associate Referee expresses his appreciation to the following collaborators and their associates for their cooperation and participation in this study:

Joseph S. Conrey, U.S. Department of Agriculture, Washington, D.C.

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References

(1) Pettinati, J. D., Swift, C. E., & Cohen, E. H. (1973) *JAOAC* **56**, 544–561

(2) Cohen, E. H. (1971) *JAOAC* **54**, 1432–1435

(3) Cohen, E. H., & Kimmelman, C. P. (1972) *JAOAC* **55**, 574–577

(4) *Official Methods of Analysis* (1970) 11th Ed., AOAC, Washington, D.C.

(5) Youden, W. J. (1967) *Statistical Techniques for Collaborative Tests*, AOAC, Washington, D.C.

(6) Youden, W. J. (1951) *Statistical Methods for Chemists*, John Wiley & Sons, New York

(7) Youden, W. J. (1959) *Ind. Qual. Control* **15**, 24–26

(8) *ASTM Annual Book of Standards* (1970), Part 30, American Society for Testing and Materials, Philadelphia, sec. **E178–68**

Table 5. Collaborative moisture analysis results by the azeotropic distillation method using cumene for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %						Ranked results			
	Ground beef		Frankfurter		Pork sausage					
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	62.25	0.455	56.53	0.468	41.53	0.350	6	7	1.5	14.5
2	62.97	0.841	57.92	1.262	40.60	0.540	3	1	8	12
3	62.23	0.408	56.60	0.438	41.53	0.372	7	4.5	1.5	13
4	62.70	0.746	56.32	1.107	41.22	1.093	5	9	6	20
5	63.12	0.436	56.60	0.200	41.30	0.616	2	4.5	5	11.5
6	62.75	0.524	56.58	0.417	40.28	0.256	4	6	9	19
7	64.70[a]	0.616	56.35	0.609	41.37	0.665	1	8	4	13
8	61.87	0.501	56.97	0.294	40.87	0.432	9	2	7	18
9	62.07	0.139	56.90	0.194	41.37	0.152	8	3	3	14
10	56.10	4.025	54.85	0.771	39.60	1.313	10	10	10	30

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.08	56.56	40.97	—
Range	−11.08 to 2.92	−2.66 to 3.74	−2.97 to 1.83	—
s_d	2.246	0.757	0.634	1.213
s_r	1.388	0.673	0.676	0.913
s_b	2.159	0.705	0.571	1.145
Final Results,[b] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.74	56.75	41.12	—
Range	−1.54 to 2.26	−1.75 to 3.55	−1.52 to 1.68	—
s_d	0.847	0.487	0.439	0.592
s_r	0.551	0.662	0.562	0.592
s_b	0.811	0.405	0.375	0.531

[a] Collaborator 7 reported 5 replicate values.
[b] Data from Collaborator 10 omitted.

method being compared did not give the same analysis values.

Conclusions on the merit of each method were based in part on the expected standard error s_e, calculated from the error components obtained from all collaborative data as follows:

$$s_e^2 = s_b^2 + s_r^2/k$$

where k represents the number of replicate analyses to be performed. For these calculations, k was set equal to 1. The systematic and random error values were averaged for all 3 samples to calculate s_e for each method.

Results and Discussion

The numerical values of standard deviation, random error, and systematic error obtained for the 3 meat product samples were found to be disproportional to sample moisture level. Dissimilarities in the values of the error components among the 3 samples were more likely due to the fineness of grind of the samples. Frankfurters, the most finely comminuted product, generally yielded the smallest random and systematic errors, and the higher error usually associated with moisture analysis of ground beef is evident in the results for each method.

Method 1. **24.003(a)**.—Evaluation of results by this method served as the basis for comparison with the new methods. Table 1 presents the reduced data from 180 single determinations. The value of ±0.637% moisture was obtained for s_e.

Method 2. **24.003(b)**.—This method was found to be equivalent to Method 1 in all respects. Table 2 shows the reduced data for 180 single determinations. The low F-value, Table 8, also shows the equivalence of the 2 methods. The value of ±0.628% moisture for s_e agrees well with that of Method 1.

Method 3. Modified **24.003(b)**.—Data for this method in Table 3, reduced for 180 single determinations, indicated Laboratory 2 to be a probable outlier by the high random error and a score of 30. After we omitted data from Laboratory 2, the final results were equivalent to those of Methods 1 and 2. The low F-value from Table 8 indicates that this method determines the same per cent moisture as Method 1. The value of ±0.650% moisture for s_e agrees well with that of methods 1 and 2.

Table 6. Collaborative moisture analysis results by the azeotropic distillation method using *m*-xylene for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

	Moisture, %						Ranked results			
	Ground beef		Frankfurter		Pork sausage					
Coll.	$\bar{x}$	s_r	$\bar{x}$	s_r	$\bar{x}$	s_r	B	F	P	Coll. score
1(AR)	62.33	0.774	56.95	0.266	41.33	0.339	6	2	3	11
2	62.70	0.721	57.25	0.715	40.75	0.274	3	1	8	12
3	62.37	0.388	56.63	0.388	41.67	0.372	4	3	1	8
4	62.33	1.211	56.40	1.095	40.93	0.372	5	4	5.5	14.5
5	63.57	0.476	55.47	0.789	40.93	0.367	2	9	5.5	16.5
6	60.75	0.689	55.40	1.133	41.33	0.427	9	10	2	21
7	64.73	0.575	56.27	0.809	40.85	0.663	1	7	7	15
8	62.20	0.456	56.33	0.273	40.70	0.518	8	5	9	22
9	62.24	0.193	56.29	0.244	41.25	0.153	7	6	4	17
10	59.92	1.269	55.93	1.329	40.43	1.346	10	8	10	28

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.31	56.29	41.02	—
Range	−4.31 to 2.89	−2.29 to 2.31	−3.02 to 0.98	—
s_d	1.324	0.586	0.370	0.761
s_r	0.750	0.800	0.577	0.709
s_b	1.289	0.486	0.286	0.687
Final Results,[a] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.58	56.33	41.08	—
Range	−2.58 to 2.62	−2.33 to 2.27	−1.28 to 0.92	—
s_d	1.084	0.607	0.326	0.673
s_r	0.668	0.717	0.410	0.599
s_b	1.049	0.531	0.280	0.621

[a] Data from Collaborator 10 omitted.

Method 4. Infrared Radiation.—Table 4 shows the reduced data from 180 single determinations. A score of 30 and the consistently high random error by Laboratory 7 indicated the reasons those data were omitted to obtain the final results for this method. Whereas mean moisture values were estimated correctly by this method, as indicated by the F-value in Table 8 being nonsignificant, both systematic and random errors were about twice those of Methods 1–3. As a consequence, the value of ±1.176% moisture for s_e is large also.

Methods 5–7. Azeotropic Distillation with Cumene, m-Xylene, and 2-Octanol.—Tables 5 (cumene), 6 (*m*-xylene), and 7 (2-octanol) show the reduced data for the 3 methods, from 179, 180, and 168 single determinations, respectively. These 3 methods are discussed together because of their similarities in performance and in the results. Outlier data from one laboratory were omitted to obtain final results for all 3 methods. Table 8 shows significant F-values for the 3 methods. This indicated that azeotropic distillation did not give the same mean moisture values as the official methods. Data for the regression equations of these methods in Table 8 indicated that the source of deviation was due to the negative intercepts. Thus, the methods showed a bias, or a tendency to underestimate moisture determination by an average of 1%. The average estimates of errors are slightly higher than methods 1–3. The value of the expected standard error for each method was calculated to be: cumene, s_e = ±0.795% moisture; *m*-xylene, s_e = ±0.863% moisture; 2-octanol, s_e = ±0.977% moisture.

Comments by Collaborators

Method 2.—Collaborator 3 dried samples 4 hr with blower on instead of 2 hr, by mistake.

Method 3.—Collaborator 2 reported that the gravity oven on hand maintained 125±5°C. Collaborator 3 approved of this method as being equivalent to the official moisture method. Collaborators 4, 5, 7, and 8 used mechanical convection ovens with the blower off. Collaborator 5

Table 7. Collaborative moisture analysis results by the azeotropic distillation method using 2-octanol for meat product samples, ranked collaborator results, and summary of statistical analysis of the data

Coll.	Ground beef $\bar{x}$	Ground beef s_r	Frankfurter $\bar{x}$	Frankfurter s_r	Pork sausage $\bar{x}$	Pork sausage s_r	Ranked results B	Ranked results F	Ranked results P	Coll. score
1(AR)	61.83	0.258	56.27	0.547	40.60	0.363	6	3.5	6	15.5
2	61.55	0.505	55.83	0.493	40.00	0.548	8	6	9	23
3	62.10[a]	0.141	56.50[a]	0.424	41.40[a]	0.000	5	2	2	9
4	62.13	0.787	56.27	1.435	40.70	0.797	4	3.5	5	12.5
5	63.85	0.259	57.87	0.665	42.58	0.426	1	1	1	3
6	62.97	0.638	55.77	1.263	41.23	0.575	2	7	3	12
7	61.82	0.842	55.37	0.493	40.30	0.687	7	9	7	23
8	62.37	0.557	56.17	0.320	40.77	0.388	3	5	4	12
9	60.72	0.378	55.61	0.654	40.22	0.252	9	8	8	25
10	59.08	1.399	54.52	1.386	37.00	2.757	10	10	10	30

Statistic	Beef	Franks	Pork	Average
Intermediate Results, Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	61.84	56.02	40.48	—
Range	−3.84 to 2.36	−3.52 to 2.78	−6.48 to 2.82	—
s_d	1.277	0.865	1.431	1.191
s_r	0.702	0.895	1.036	0.878
s_b	1.238	0.767	1.354	1.120
Final Results,[b] Per Cent Moisture				
Grand mean, $\bar{\bar{x}}$	62.15	56.18	40.87	—
Range	−1.95 to 2.05	−1.98 to 2.62	−1.87 to 2.43	—
s_d	0.882	0.728	0.787	0.799
s_r	0.561	0.815	0.526	0.634
s_b	0.846	0.630	0.751	0.743

[a] Collaborator 3 reported duplicate values only.
[b] Data from Collaborator 10 omitted.

Table 8. Computations for comparison of moisture methods to method 24.003(a)

Moisture method number	Data omitted, coll. no.	Intercept	Slope	Sum of squares, SS_R	Corr. coeff.	Slope = 1 intercept = 0 sum of squares, SS_E	F
		Values from linear regression					
2	none	0.840	0.985	14.82	0.996	15.4	0.57
3	2	0.046	1.005	11.26	0.997	14.1	3.10
4	7	2.051	0.972	36.37	0.991	46.1	3.33
5	10	−1.761	1.025	11.86	0.997	18.2	6.62[a]
6	10	−1.395	1.014	19.84	0.995	31.3	7.26[a]
7	10	−1.357	1.008	14.54	0.996	36.8	19.22[a]

[a] Significant at the 1% level.

found that, with the blower off, the oven required 1¾ hr to recover 125°C after samples were placed on the shelf (this may indicate a very small oven or very thin-walled insulation).

Method 4.—Collaborator 3 used an Ultra-X infrared lamp, 250 w, supplied by Koch Equipment Co. and a Mettler Model P120N balance and felt that the procedure had significant value when the equipment is properly set up. Collaborator 6 indicated that the method showed good correlation with other methods for determining moisture and recommended that analyses be performed in a constant-temperature enclosure because laboratory temperature rise during the day caused the temperature at the pan to rise about 8°C. Collaborator 7 found that 2 days of on and off adjusting were required to find the distance that gave a fairly stable 125°C. (The Associate Referee found 4 hr to be sufficient.)

Methods 5, 6, and 7.—Collaborator 2 found that irregular menisci were obtained and the volume could not be read accurately. Water bubbles formed in the solvent layer adhered to the walls of the receiver tube, and would not drop to the water layer without physical assistance. Collaborator 3 used a hot plate successfully instead of a heating mantle and timed distillation from start of condensate formation. Collaborator 4 felt that azeotropic distillation is impractical due to erratic results that are obtained, toxicity of solvents, and problem of solvent disposal. Collaborator 5 found that 20 min was sufficient to determine moisture content of frankfurters and sausage, while ground beef required 30 min. Erlenmeyer flasks of 250 ml in place of round flasks and hot plates in place of heating mantles were used but a problem was encountered with foam above the boiling solvent which rose above the flask neck. However, the collaborator felt the rapid methods are worthy of consideration due to reasonable degree of accuracy, speed of analysis, and availability of required equipment. Collaborator 7 found that water droplets were trapped in the solvent layer of receiver. These were dislodged with a wire. The problem was more pronounced with pork samples than with the other 2 products. Collaborator 10 found that water droplets clung to the sides of the condenser and that the frankfurter sample received was insufficient to complete all analyses.

Conclusions

The unofficial guidelines for control work on both finished product and meats prior to processing were discussed by Cohen (2). The guidelines for all products were stated to be a desired accuracy of ±1% of the moisture content obtained by official analysis. A precision of ±1% of the determined value is desired for finished product and ±3% may be tolerated for meats prior to processing.

A comparison of the statistics determined in this collaborative study with the above guidelines shows that moisture determination by the modified official method gave results equivalent to those obtained by the existing official methods. The infrared radiation method estimated mean moisture content accurately but precision was too low for the method to be useful for finished product except where a rapid method without high precision is desired. Azeotropic distillation estimated moisture about 1% low and gave a standard error higher than the official methods. Cumene and *m*-xylene in azeotropic distillation yielded more accurate and more precise results than 2-octanol and therefore are the preferred solvents. As with infrared radiation, azeotropic distillation is not precise enough for finished products except where rapid analysis would be desired without high accuracy and precision.

Youden (6) stated that rarely will a new method excel an old method in every particular. However, for processors and control agencies, the rapid methods, i.e., moisture balance and azeotropic distillation, are recommended where high accuracy and precision are not mandatory.

Relative to the collaborative study, a comment is directed toward those who may initiate a similar type of study. While 60 results were reported for the most part for each product sample (6 replicates × 10 laboratories), each group of data led to error terms as follows: s_r was estimated with 50 degrees of freedom and s_b with 9 degrees of freedom. The error term s_b is important because a collaborative test attempts to estimate the population of all laboratories. Therefore, in a similar study s_b is determined more representatively by having 30 laboratories run duplicate determinations in order to estimate s_r with 30 degrees of freedom and s_b with 29. However, it is only conjecture that s_b in the present study is not representative and would be any different in a sampling of 30 laboratories since s_b is always the major error factor, being 2–3 times s_r in many cases.

Recommendations

Based on the results of this collaborative study, it is recommended—

(1) That a single shelf in a gravity oven be adopted as official first action as an alternative method of drying in 24.003(b), which presently specifies a mechanical convection oven.

(2) That infrared radiation drying for 40 min be adopted as official first action as an alternative, rapid screening method for analysis of meats prior to processing or for analysis of processed meat products when rapid analysis is more important than a high degree of accuracy and precision.

Recommendation (1) of the Associate Referee was approved by the General Referee and by Subcommittee C and was adopted by the Association; *see* (1973) *JAOAC* 56, 399–400. The Association felt that a study of current screening methods should be conducted before additional screening methods are adopted.

(3) That azeotropic distillation for moisture determination in 30 min with *m*-xylene or cumene be adopted as official first action as an alternative, rapid screening method for analysis of meats prior to processing or for analysis of processed meat products when rapid analysis is more important than a high degree of accuracy and precision.

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References

(1) Pettinati, J. D., Swift, C. E., & Cohen, E. H. (1973) *JAOAC* **56**, 544–561

(2) Cohen, E. H. (1971) *JAOAC* **54**, 1432–1435

(3) Cohen, E. H., & Kimmelman, C. P. (1972) *JAOAC* **55**, 574–577

(4) *Official Methods of Analysis* (1970) 11th Ed., AOAC, Washington, D.C.

(5) Youden, W. J. (1967) *Statistical Techniques for Collaborative Tests*, AOAC, Washington, D.C.

(6) Youden, W. J. (1951) *Statistical Methods for Chemists*, John Wiley & Sons, New York

(7) Youden, W. J. (1959) *Ind. Qual. Control* **15**, 24–26

(8) *ASTM Annual Book of Standards* (1970), Part 30, American Society for Testing and Materials, Philadelphia, sec. **E178–68**