27.3.06

AOAC Official Method 935.29
Loss on Drying (Moisture) in Malt

Gravimetric Method
First Action 1935
Final Action

A. Apparatus

(**a**) *Weighing dish.*—Use glass bottle or Al dish, with tight-fitting cover, ca 40 mm diameter for 5 g test portion, or 55 mm for 10 g test portion.

(**b**) *Oven.*—With automatic control holding temperature within 0.5 C, and large enough to hold all tests on 1 shelf in such manner that no test dish is outside area indicated by test to give comparable results in duplicate tests. Standardize oven as follows: Place weighed duplicate test portions in oven at 103 –104 C and dry 3 h. Weigh, and redry 1 h longer. If loss of moisture is 0.1%, raise temperature 1 C and again test with new duplicate test portions. Take, as standard, lowest temperature <106 C giving moisture content that, after 3 h of drying, is within 0.1% of value attainable at same temperature within 4 h. Keep ventilators of oven open during entire drying period, and do not open door during the 3 h of drying.

B. Preparation of Test Sample

(**a**) *If extract determination is to be made.*—Grind test sample as in 935.30D (*see* 27.3.07), and transfer in one continuous operation. When many test samples are to be analyzed, grind first test sample, remove beaker, and grind second test sample while adjusting weight of first test sample. Remove second test sample, insert third test sample, and repeat operation.

(**b**) *If extract determination is not to be made.*— Have test sample of same fineness as finely ground malt used to determine extract. Weigh ca 5 g whole malt (or 10 g if 55 mm diameter weighing bottle is used) and grind through clean dry mill directly into weighing bottle. Brush all malt from mill into weighing bottle and cover immediately.

C. Determination

Weigh test portion to 1 mg and place in oven previously heated to standard temperature. Remove cover of weighing bottle and heat exactly 3 h at standard temperature. Replace cover, transfer to desiccator, cool to room temperature, and weigh to 1 mg. Report moisture to nearest 0.1%.

AOAC Official Method 935.30
Extract of Malt

First Action 1935
Final Action 1960

A. Reagent

Iodine standard solutions.—(**a**) *0.01M.*—Dissolve 0.63 g I_2 and 1.25 g KI in H_2O, and dilute to 500 mL. (**b**) *0.02M.*—Dissolve 1.27 g I_2 and 2.50 g KI in H_2O, and dilute to 500 mL. Prepare fresh solutions monthly and store in dark. For daily use, keep portion of solution in small brown dropper bottle.

B. Apparatus

(**a**) *Mills.*—Miag-Seck (available from Buhler-Miag, Inc., PO Box 9497, Minneapolis, MN 55440, USA; Buhler-Miag Ltd., D-3300 Braunschweig, Germany). For fine grinding use cone-type, 300 rpm, and for coarse grinding, roller-type, 150 rpm.

(**b**) *Sieves.*—Half-height, 8 in. (20.3 cm) standard sieve No. 30 (with pan and cover). For classification of laboratory and brewery grindings use additional standard sieves Nos. 10, 14, 18, 60, and 100.

(**c**) *Mash beakers and counter weights.*—Made of either pure Ni, stainless steel, or brass, not Cu, and of such dimensions as to ensure tight connection between beakers and Miag-Seck mill while grinding.

If counter weights are used for the mash beakers, check tare weights frequently.

(**d**) *Mashing apparatus*.—Use beakers, stirrers, and solder made of same metal. Provide each stirrer with blade that in operation has clearance of ca 2 mm from bottom and 5 mm from wall of mash beaker. Blade is ca 8 mm wide, and each side has 45 pitch, arranged as in a propeller, to force mash upward. Speed of mash stirrer must be 80–100 rpm, each stirrer of each beaker having same speed. Mechanically stir water in bath thoroughly to assure uniformity of temperature and have level of H_2O above maximum mash level.

(**e**) *Gypsum plate.*—Thoroughly mix 100 mL H_2O with 135 g plaster of Paris. Pour mixture while still free-flowing, into suitable flat molds (cigar boxes, etc.). Porcelain plate for color reactions, Coors No. 550, size 00, may be used.

(**f**) *Filter paper.*—Use Schleicher and Schuell 32 cm fluted paper No. 560 (or No. 597, 32 cm, fluted by analyst) or Alston Filtration, (PO Box A, Mount Holly Springs, PA 17065, USA), 32 cm fluted paper No. 509 (replaced by 5090-3200) (or No. 609 [replaced by No. 6090], 32 cm, fluted by analyst).

(**g**) *Funnels.*—Use short-stem glass funnels ca 20 cm diameter and do not let paper project above rim. Stem must extend 3–5 cm into receiving flask.

(**h**) *Flasks.*—Use dry 500 mL Erlenmeyers marked at 100 mL level.

(**i**) *Pycnometers.*—Use any suitable pycnometer, but preferably Reischauer or Boot (vacuum) type. Reischauer type is ca 15 cm high with neck ca 9 cm long and 2.5–3.5 mm id. Fine, well-defined mark is found 55–70 mm below upper rim of neck. When filled with H_2O at 20 C its capacity must be 48–50 g. Use ca 15 mL glass funnels to fill pycnometers.

Boot type is cylindrical and holds ca 50 g H_2O at 20 C. Vacuum seal must be well rounded off and not pointed. Pycnometer opening is wide enough to permit easy filling and emptying, and stopper has fine capillary opening. Walls of bottle meet stopper in rising acute angle of ca 45 so that no depression or groove retaining moisture is formed at this point. (Available from Rascher and Betzold, Inc., 5410 N. Damon Ave, Chicago, IL 60625, USA.)

(**j**) *Emptying device for Reischauer pycnometer.*— Bend piece of metal capillary tubing (brass, stainless steel), <2 mm od, to ca 45 angle. End to be inserted into pycnometer must reach within 2–3 mm of bottom. Connect other end either to rubber aspirator bulb or to compressed air supply 5 psi.

(**k**) *Water bath.*—Automatically controlled. If automatic control is not available, use following apparatus. Have water level of bath (5–15 L) reach above neck marks of pycnometer, keep water bath temperature at 20 0.05 C, and read on accurate thermometer, calibrated to 0.1 C. Maintain temperature of water bath by very slow but continuous flow of ice-water from container (2–4 L, containing ice and water). Regulate flow of ice-water by hand. Stir water in bath mechanically and continuously without splashing.

C. Standardization

(**a**) *Setting of mill.*—Use malt of characteristics shown in Table **935.30**.

Fine grinding.—Weigh 50 g specified malt into mash beaker, grind, and collect in same beaker. Mill must not be in motion when test sample is introduced. Transfer to No. 30 sieve placed on receiving pan and shake in horizontal plane on flat surface 3 min, pausing every 15 s long enough to give screen and pan 2 sharp taps on surface over which it is sliding. Transfer and weigh particles remaining on and adhering to screen. Consider mill as having standardized setting when weight of ground malt remaining on No. 30 sieve is between 4.5 and 5.5 g (9–11%). Standardize mill at least twice yearly. Suitable mechanical shaking device, giving equivalent results, may be used to standardize mill.

Coarse grinding.—Proceed as for *fine grinding.* Consider mill as having standardized setting when portion of ground malt remaining on No. 30 sieve is between 37 and 38 g (74–76%).

(**b**) *Reischauer type pycnometer.*—Clean interior and exterior of pycnometer with chromic acid solution, discharge carefully with air, and wash several times with H_2O, then alcohol, and finally ether. To remove last traces of ether vapor and to replace with laboratory air, connect dry metal capillary tubing to vacuum and insert into

Table 935.30. Characteristics of malt

Variety: Malt made from 6-rowed Midwestern variety of barley	
Moisture	4.2–4.8%
Extract in finely ground malt, dry basis	74.0–77.0%
Color of laboratory wort, 972.13A or **B** (*see* 27.3.10)	1.8
Diastatic power, dry basis	100 C
Ratio soluble protein to total protein	36–42
Mealiness: Glassy	5%
Mealy	90%
Acrospire development: 0–2 grown	5%
¾ to full grown	80%
Overgrown	5%
Assortment: From malt meeting above specifications, take that portion passing through 7/64 in. (2.8 mm) screen and remaining on 6/64 in. (2.4 mm) screen for actual standardizing operation.	

pycnometer 1–2 min. Carefully wipe pycnometer, let stand few min, and determine weight to 0.2 mg.

Fill with freshly distilled water and place in water bath held at 20 ± 0.05 C. Tap gently to force out air bubbles. After 25 min, remove liquid above mark with capillary pipet provided with small rubber bulb. To make final adjustment of meniscus, absorb last portion of liquid with thin strips of blotting paper; also remove any liquid adhering to inner surface of neck. Adjust H_2O level so that lower part of meniscus rests on mark. Make all adjustments of liquid level within pycnometer neck while holding by neck, without touching body of pycnometer with hands. Keep body of pycnometer submerged during entire period of meniscus adjustment.

Raise pycnometer to room temperature by insertion into water bath kept at exactly that temperature, and hold 10 min. Remove pycnometer, carefully dry exterior, and weigh to 0.2 mg. Subtract weight empty pycnometer. Difference in weights represents H_2O capacity of pycnometer at 20 C. Redetermine tare weight and H_2O capacity at frequent intervals.

(**c**) *Boot type pycnometer.*—Clean pycnometer and determine its weight in same way as for Reischauer type. Cool H_2O in ice bath to temperature slightly <20 C. Rinse pycnometer once with the cool H_2O, fill, stopper, and dry exterior. Remove stopper, insert thermometer adjusted to 20 C in H_2O, and note temperature, which should be 20 0.1 C. If it is not, choose different "filling" temperature, which may vary with analyst and season from 19.4 to 19.7 C or more. Make subsequent determinations, using predetermined filling temperature. Place cap over stopper and weigh.

Redetermine weight, H_2O capacity, and filling temperature at least weekly.

D. Determination

Fine grinding.—Weigh ca 55 g test portion (at room temperature) into tared mash beaker and grind through mill set for standardized fineness of grind. Collect finely ground malt in same mash beaker, carefully brushing malt particles remaining in mill into mash beaker. Mix, and without delay, place mash beaker with contents on balance accurate to within 0.05 g under 750 g load and adjust weight malt to 50 0.05 g by removing excess into tared dish for moisture determination.

Coarse grinding.—Weigh 50.5 g test portion (at room temperature) into tared mash beaker and grind through mill set for standardized coarseness of grind. Collect coarsely ground malt in same mash beaker, carefully brushing particles remaining in mill into mash beaker. Without delay, place mash beaker with contents on balance accurate to within 0.05 g under 750 g load and adjust weight malt to 50 0.05 g by removing excess.

(**a**) *Mashing procedure*.—"Mash" in ground malt with 200 mL H_2O at 46 C and mix well with glass rod to prevent formation of lumps. Carefully rinse glass rod and wall of beaker with small amount H_2O. Note odor of mash and report as aromatic, slightly aromatic, musty, green, stale, etc. Promptly place mash beakers in mashing apparatus containing H_2O previously heated to 46 C, and set stirrers in motion. Place thermometer in each mash beaker. Keep temperature at 45 C exactly 30 min from time beakers were placed in mashing apparatus. Raise mash temperature 1 /min to 70 C. Add 100 mL H_2O, previously heated to 70 –71 C, and hold mash 60 min at 70 C. (Temperature deviations during mashing procedures should not exceed 0.5 C.)

(**b**) *Conversion.*—Transfer drop of mash with thin glass rod (ca 3 mm diameter) onto absorbent gypsum plate, **B**(**e**), or into one cavity of porcelain plate, and test with drop of 0.01M I_2 solution on gypsum plate, or with drop of 0.02M I_2 solution, **A**(**b**), on porcelain plate. Make tests 5, 7, and 10 min after 70 C is reached, and thereafter if necessary, at 5 min intervals. Conversion is complete when test drop and I_2 solution produce only yellow stain on gypsum or porcelain plate. Report time of conversion in periods: <5 min, 5–7 min, etc. Time of conversion is not determined on coarsely ground malt.

(**c**) *Cooling and filtration.*—After 60 min, cool mash promptly (within 10–15 min) to prevailing room temperature. Stop stirrers. Remove thermometers after adhering mash particles are rinsed into beaker with H_2O. Remove each beaker with its stirrer from mashing apparatus. Rinse mash particles adhering to stirrer into beaker with H_2O. Dry outside of each beaker, taking care to remove moisture adhering to rim. Without delay, adjust weight of contents of mash beaker to 450.0 0.05 g by adding H_2O.

Stir mash thoroughly with glass rod, once when removing beakers from balance pan and again immediately before pouring mash onto filter. (Stirrings must be 5 min but <15 min apart.) While stirring cooled mash, take care to prevent splashing or spilling. Mix drops adhering to beaker wall into mash by rotary stirring with glass rod.

Pour entire contents of beaker into funnel provided with specified filter paper. Cover funnel with ca 20 cm diameter watch glass during entire filtration. Return first 100 mL filtrate to filter. When no more liquid is present above filter cake, discontinue filtration and remove receiving flask containing wort for later observations and tests. In case of slow running worts, stop filtration after 2 h. In case of coarse ground malt mash, collect exactly 200 2 mL wort. When filtration is complete, mix wort in receiving flask thoroughly by rotary motion. Speed of filtration is normal if filtration is complete (as defined above) within 1 h after returning the 100 mL filtrate to filter bed; slow, if filtration takes longer. Observe degree of clarity and report as clear, slightly hazy, or hazy.

Remove ca 100 mL wort for determination of color. (Color is not determined on wort from coarsely ground malt.)

(**d**) *Specific gravity.*—Rinse empty pycnometer twice with ca 10 mL wort, and if Reischauer pycnometer is used, remove rinses each time with emptying device. Fill with wort, place in water bath, and proceed as in **C**(**b**) or (**c**). Weigh filled pycnometer within 3 h of completed filtration. Difference between this weight and that of empty pycnometer represents wort capacity of pycnometer at 20 C. Calculate specific gravity of wort to fifth decimal place, rounding off to 0.00005 or 0.00010, by dividing weight wort by weight H_2O.

No calculation is made of specific gravity in vacuo. If duplicate determinations made by same analyst in different beakers differ by >2 units in fourth decimal place, repeat entire determination.

(**e**) *Extract.*—Determine extract yield of wort by reference to specific gravity values given in **970.90** (*see* Appendix C), and calculate extract yield of malt by following formulas:

$$\text{Extract as-is basis, \%} = \frac{P(800 \quad M)}{(100 \quad P)}$$

where P = g extract in 100 g wort [Plato, **970.90** (*see* Appendix C)]; and M = % H_2O in the malt.

$$\text{Extract dry basis, \%} = \frac{E \quad 100}{100 \quad M}$$

where E = extract as-is basis; and M = % H_2O in the malt.

Report extract as-is basis and dry basis to nearest 0.1%.

27.4.03

AOAC Official Method 945.15
Loss on Drying (Moisture) in Cereal Adjuncts

Air Oven Method (103 –104 C)
First Action 1945
Final Action

A. Apparatus

See 935.29A (*see* 27.3.06).

B. Determination

Grind as in 935.30D (*see* 27.3.07), fine grinding, and proceed as in 935.29C (*see* 27.3.06).