vealed by x-ray and photomicrographic techniques, is related to yields of head rice, color, stability to oxidation, and nutritive value.

Acknowledgment

The authors are grateful to the X-Ray Department, General Electric Co., New Orleans, La., for making available an x-ray grain inspection unit and for technical assistance in its operation. The aid of R. W. Haines, photographer, Northern Utilization Research Branch, in the preparation of Figure 5 is gratefully acknowledged.

Literature Cited

(1) Carter, L. C., "Needs of the Rice Industry and Research Which Would Be Most Helpful," Report on Research Conference on Processing and Utilization of Rice, Southern Regional Research Laboratory, March 15 and 16, 1950.
(2) DuBois, D., *Northwest. Miller*, **248,** No. 11, 1a–18a (1952).
(3) Henderson, S. M., *Rice J.*, **57,** 16–18 (1954).
(4) Kik, M. C., Arkansas Agr. Expt. Sta., *Bull.* **508** (1951).
(5) Milner, M., Lee, M. R., and Katz, R., *Food Technol.*, **6,** 44–5 (1952).
(6) Milner, M., Lee, M. R., and Katz, R., *J. Econ. Entomol.*, **43,** 933–5 (1950).
(7) Milner, M., and Shellenberger, J. A., *Cereal Chem.*, **30,** 202–12 (1953).
(8) Milner, M., Shellenberger, J. A., Lee, M. R., and Katz, R., *Nature*, **170,** 533 (1952).
(9) Nichols, L., *Research*, **2,** 401–7 (1949).
(10) U. S. Agricultural Marketing Service and Regulatory Announcement No. **147** (rev. 1941).

Received for review August 2, 1954. Accepted October 21, 1954. The use of trade names in this article is for identification only and implies no endorsement of manufacturer or product.

Determination of Moisture in Chocolate

K. G. SLOMAN, EMANUEL BORKER, and M. D. REUSSNER
Central Laboratories, General Foods Corp., Hoboken, N. J.

A RAPID, precise, and accurate method was needed for determining moisture in chocolate. Oven-drying methods require too much elapsed time, and distillation methods are not satisfactory at moisture levels normally encountered in chocolate. Therefore, the the Karl Fischer titration was investigated.

Kentie and Barreveld concluded that Karl Fischer titration is the fastest and best method for following water content during processing (*1*), but presented no data on its accuracy.

The Karl Fischer reagent used in this laboratory is that suggested by Seaman *et al.* (*3*). The conventional Fischer reagent is split into two parts: Reagent A containing methanol, sulfur dioxide, and pyridine, and Reagent B containing iodine and methanol. Sodium tartrate dihydrate is used to standardize the reagent (*2*). In the work reported, both the Fisher Titrimeter and the Beckman Aquameter were used for the titration.

Water is extracted from the chocolate sample by heating with methanol just to boiling, cooling, adding Reagent A, and after 10 minutes titrating with Reagent B. When samples were extracted at room temperature with methanol or Reagent A, the values were low. A longer boiling time did not increase the amount of water extracted.

To show that added water could be recovered, several experiments were performed in which water was added to chocolate and determined by Karl Fischer titration. Some difficulty was encountered in quantitatively adding water to the chocolate. Melting the chocolate at 70° C., so that the water might be beaten into it, caused the loss of some water, even in a closed (though not hermetically sealed) system. However, gravimetric determinations before and after addition of water gave a good measure of the amount of water actually retained in the sample.

Five experiments using the gravimetric measure of water added showed that the recovery ranged from 90 to 117% of the total water present in the sample, when the sample was analyzed on the same day as prepared or the day following (Table I). The standard deviation for a single determination, calculated from the duplicate determinations represented in Table I, is ±4% of the moisture value.

Proper sampling of chocolate is also a consideration in determining moisture. One experiment showed a moisture gradient as follows in a bar 1 inch thick, but additional data are needed to establish the normal moisture gradient in bar chocolate.

	Water, %
Skin, 1/8 inch thick	1.13
Inner layer, 1/4 inch down	0.55
Center 1/4 inch	0.62

In order to eliminate the effect of surface moisture, it is advisable to remove the outer skin and chip the remainder of the bar to get a homogeneous sample. The best method of obtaining a representative sample is to melt the sample in an oven at 50° C., stir, and then sample, but this takes longer than shaving and chipping. No moisture was lost after several hours at 50° C. in a closed container.

As the results found in two laboratories in different sections of the country were in agreement, no correction for the effect of humidity seems necessary.

Table I. Recovery of Added Moisture in Chocolate

Expt.	H_2O in Original, %	Change in H_2O Found Grav., %	Total H_2O, % Calcd.[a]	Total H_2O, % Found K.F.	Recovery Based on Total H_2O in Sample, %
A	0.98	+0.33	1.31	1.40	107
		+0.76	1.74	2.04	117
B	0.65	+0.43	1.08	1.00	93
		+0.80	1.45	1.31	90
C	0.82	+0.40	1.22	1.22	100
		+0.86	1.68	1.71	102
D	0.52	+0.37	0.89	0.91	102
		+1.18	1.70	1.68	99
E	0.60	+0.41	1.01	1.02	101
		+0.94	1.54	1.53	99

[a] Calculated from original H_2O content by Karl Fischer and gravimetric change. All values are duplicate determinations obtained 1 day after moisture addition.

Literature Cited

(1) Kentie, A., and Barreveld, A., *Chem. Weekblad*, **46,** 833 (1950).
(2) Neuss, J. D., O'Brien, M. G., and Frediani, H. A., *Anal. Chem.*, **23,** 1332 (1951).
(3) Seaman, W., McComas, W. H., Jr., and Allen, G. A., *Ibid.*, **21,** 510 (1949).

Received for review April 21, 1954. Accepted October 8, 1954.