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A PICNOMETER FOR THE DETERMINATION OF DENSITY OF MOLASSES

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A PICNOMETER FOR THE DETERMINATION OF DENSITY OF MOLASSES

By W. B. Newkirk

The difficulty of obtaining accurate results on the density determinations of viscous liquids has long been recognized. During the past few years there has been a great increase in the quantity and value of all grades of molasses and sirups. In the determination of the value of these products for commercial purposes the density is necessarily one of the important factors. Its accurate determination in all viscous liquids is likewise of importance for scientific purposes. It has, therefore, become essential that a method be developed to determine this quantity with all possible accuracy, and it is to this end that the present work was undertaken.

The difficulties met in the accurate determination of the density of molasses are due to (1) high viscosity, (2) included gases, and (3) dissolved gases.

As a result of the high viscosity, the use of hydrometers is prevented, and the thorough mixing or intermingling of the various components of the batch or sample of molasses is made difficult. Obviously, in materials of this character a representative sample can not be obtained without vigorous agitation, which causes the entrainment or inclusion of large amounts of air. Finally, the viscosity retards the escape of the entrained or included air.

The effects of included air upon the density of the liquor are too evident to need discussing. The removal of the dissolved gases before taking the density may in some cases be essential and in others not.

It was decided to use the picnometer or specific-gravity bottle as a starting point in the development of an improved procedure. The picnometer of Johnson and Adams [1] was accepted as the most accurate type to control the volume. In this bottle the volume is fixed by an optically flat disk, seating on an optically

[1] Jour. of Am. Chem. Soc., 34, p. 566; 1912.

flat plane ground on an enlarged rim at the top of the bottle. The fact that it is not necessary to use a lubricant on this joint, the absence of errors caused by dirt lodging in the customary ground stopper, and the greater ease with which the surfaces can be brought into contact when using a viscous material, permit great accuracy in fixing the volume. Johnson and Adams found that the volume could be reproduced to within 0.2 mg of water, and the author found similar accuracy.

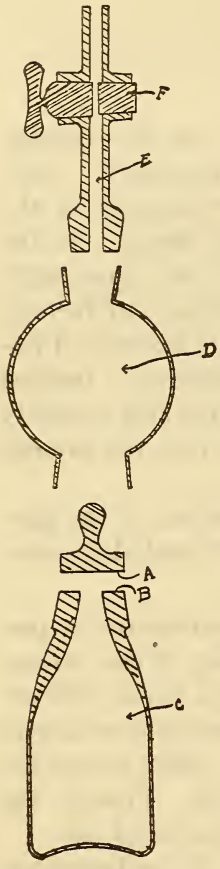

FIG. 1.—*Form of picnometer used*

Any known method of removing the included gases causes considerable foaming. This necessitates the removal of the air before filling or the accommodation of this foam in the picnometer design. In an effort to meet this difficulty a large expansion chamber, equipped with a ground joint and fitted over the top of the bottle, has been described by an anonymous writer.[1] This permitted the molasses to expand into the chamber while deaerating, and then as the gas rose to the surface the air-free molasses filled the bottle. When the bottle was full, the volume of molasses could be fixed.

Investigations at this Bureau established the fact that the removal of gases was best effected by the use of a vacuum. In order to permit this to be done as readily and effectively as possible, an improved picnometer, shown in Fig. 1, was designed.

It consists of a bottle, *C*, fitted with an enlargement at the top, *B*, ground optically flat and closed off by another optical flat, *A*. An expansion chamber, *D*, is ground to the bottle and fitted with a vacuum connection, *E*. To avoid loss of water due to evaporation under reduced pressure, the connecting tube is fitted with a stopcock, *F*, so that when the proper vacuum has been reached the apparatus can be closed off from the vacuum source. With this provision the volume to be filled with water vapor is very small and the amount of water evaporated will be negligible. The bottle is so shaped as to have a smooth gradual slope to the top, so that the bubbles will rise with the least effort to the expan-

[1] Anonymous—International Sugar Jour., 15, p. 489; 1913.

sion chamber. It has thick walls over the neck, so that it can be readily handled without changing the temperature or volume by heat transmitted to the flask by the fingers. The joints of the expansion chamber, vacuum connection, and stopcock are ground to an accurate fit. Since it is unnecessary to employ a vacuum with a pressure lower than the vapor pressure of molasses at room temperature, it is entirely practicable to utilize the sample under test to lubricate and seal all the ground joints. Needless to say, this is an important advantage.

In using the picnometer, the expansion chamber, after lubrication of all joints with molasses, is placed on the bottle. The molasses to be analyzed is flowed into the bottle and into the expansion chamber until the latter is about one-third full. The vacuum line is then connected and the pressure reduced until the gas expands to visible bubbles. The apparatus is immediately closed off by turning the stopcock, *F*, and the whole placed in the thermostat for accurate work or in the balance case for control work. When all the bubbles have collected in the expansion chamber and the temperature has reached equilibrium, the volume is fixed with the plate after removing the expansion chamber. It is then wiped and weighed.

The densities are determined by correcting the weights to vacuo and comparing to the weight of an equal volume of water at 4° C in vacuo. They are reported in degrees Brix,[1] according to Circular 44 of this Bureau.

In the present investigation, two methods for de-aerating were studied:

1. Heating.—This reduces the viscosity and expands the gases, causing the separation to take place more quickly.

2. The Application of Vacuum.—This expands the gases, causing a rapid separation to take place.

The use of heat was found to cause considerable decomposition of an unknown character, as will be seen from the following experiments:

Experiment 1.—The density was determined after filling. Brix found, at 20° C, 76.41°.

Experiment 2.—After experiment No. 1 the picnometer was allowed to stand without refilling, the expansion chamber in place and a third full, for 24 hours. In removing the expansion cham-

[1] Degrees Brix is the density of a pure-sugar solution, expressed as the percentage, by weight, of sugar present in the solution. The comparison of density and degrees Brix is tabulated in Table 31, Circular 44, of the Bureau of Standards.

ber care was taken to save the extra molasses in the chamber for the next experiment. After standing 24 hours the density was determined. Brix found, at 20° C, 76.91°.

EXPERIMENT 3.—The picnometer was subjected to vacuum without refilling and with the same molasses in the expansion chamber. The vacuum applied was equal to 200 mm mercury and was held for 12 hours, after which the expansion chamber was removed with the same care as in experiment No. 2. The density was again determined. Brix found, at 20° C, 76.96°.

EXPERIMENT 4.—The picnometer again, with the same molasses and without refilling, was, with the expansion chamber and its same molasses in place, immersed in a water bath held at 80° C for a period of two hours. It was brought to 20° C, and the density was again determined and was found considerably lower than experiment No. 3. Brix found, at 20° C, 76.66°.

Experiment 5.—The picnometer was refilled with the original molasses and, with the expansion chamber in place, was subjected to the same conditions as No. 3, namely, 200 mm vacuum at room temperature for 12 hours. The density found checked satisfactorily with that found in No. 3. Brix found, at 20° C, 77.04°. The data are summarized in Table 1.

It is seen that in experiment No. 4, in which heat was used to aid the deaerating, the density is considerably lower than those found in experiments 2, 3, or 5, and is not in agreement with them. It is evident that the use of heat should be avoided.

TABLE 1

Experiment No.	Time standing	Temperature	Vacuum	Degrees Brix at 20° C
	Hours	°C	Millimeters	
1	(a)	24	(a)	76.41
2	24	24	(a)	76.91
3	12	22	200	76.96
4	2	70	(a)	76.66
5	12	20	200	77.04

[a] None.

The reproducibility obtained is shown by the data given in Table 2. The experiments were conducted as outlined in describing the use of the picnometer. The joints were lubricated with molasses and the bottle filled and the expansion chamber one-third filled with the molasses, a vacuum of 200 mm was applied and the picnometer sealed off. After standing 12 hours at room

temperature, it was placed in a thermostat held at 20° C until equilibrium was established. The volume was then fixed and the weighing immediately made.

TABLE 2.—Density Determinations of Molasses

Material	Duplicate 1	Duplicate 2
	Degrees Brix	Degrees Brix
Black-strap molasses A	76.96	77.04
Black-strap molasses B	76.91	76.91
Beet molasses	81.40	81.37
Black-strap molasses C	76.77	76.76
Black-strap molasses D	77.41	77.41
Black-strap molasses E	68.70	68.71

We may, then, say that the individual accidental error is within one-tenth of 1° Brix, and therefore considerably better than hitherto attainable. The general run of determinations will check to within a few hundredths of 1° Brix. In summarizing, it may be stated that a new picnometer design has been developed which permits greater accuracy in density determinations of molasses than has been possible heretofore. A method of removing the entrained gases is proposed which does not have the bad features of heating or dilution. Heating is shown to have a deleterious effect in density determinations. The accidental error is shown to be small and probably smaller than the accuracy with which a representative sample of a given molasses can be obtained.

WASHINGTON, December 18, 1919.