DETERMINATION OF THE WATER CONTENT OF FOODS

III. THE DIRECT DETERMINATION OF TOTAL SOLIDS IN LIQUID SACCHARINE FOOD PRODUCTS AND IN MEAT-CURING BRINES, ACCORDING TO JOSSE-BUYZE*

by

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INTRODUCTION

A convenient method for direct determination of total solids in aqueous sugar solutions has been proposed as early as 1893 by the French chemist JOSSE[6]. In this method the solution is *absorbed by filter paper* and easily desiccated afterwards since no essential retardation of desiccation due to crust formation[10] occurs. Although this technique has been applied in some instances [11, 9, 3] it never became very popular, probably due to the inconsistency of the results obtained by some workers[12, 7, 5].

The JOSSE-method to our opinion was considerably improved by BUYZE[2] who proposed a special filter paper pile to prevent sticking of the paper. His pile consists of a central, folded cone, surrounded by a periferous, pleated cylinder; cf. Fig. 1. Any liquid, not absorbed by the pile, is caught by two normal analytical filters, which are present below the pile proper.

This paper reports experiments carried out in this laboratory:

i) with *model* substrata, to check the exactness of the method;

ii) with *actual* substrata, to study the suitability of the method.

MATERIALS

Model substrata

A *brine* was prepared from sodium chloride A. R. and peptone (Difco, for bacteriological purposes).

The sodium chloride was heated at 500° C in an electric furnace to expel any occluded water. The peptone was dried for 72 h at 40° C over P_2O_5 in vacuo; it did not loose any further weight on prolonged drying and therefore was considered as to be water-free.

The anhydrous materials were dissolved in distilled water to give a solution, containing about 20% of NaCl and 0.5% of peptone, which corresponds to the average of data found in this laboratory when analyzing meat-curing brines.

An artificial *saccharine sap* was prepared from dextrose A.R., levulose (Kahlbaum;

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"reinst. für analytische Zwecke"; 1942), potassium sulfate (desiccated at 500° C) and dehydrated Difco-peptone. The materials were dissolved in distilled water to give a solution, containing about 20% of invert sugar, 2% of potassium sulfate and 0.5% of peptone. Since experience shows that very few levulose preparations are really pure, the solution was analysed for total reducing sugars according to the titrimetric LUFF-SCHOORL copper-reduction method[13].

A liquid *malt extract* was prepared from Difco dehydrated malt extract. The preparation was desiccated for 3 days at 40° C over P_2O_5 in vacuo, after which it did not loose any further weight, and then dissolved in distilled water to give a solution of about 20%.

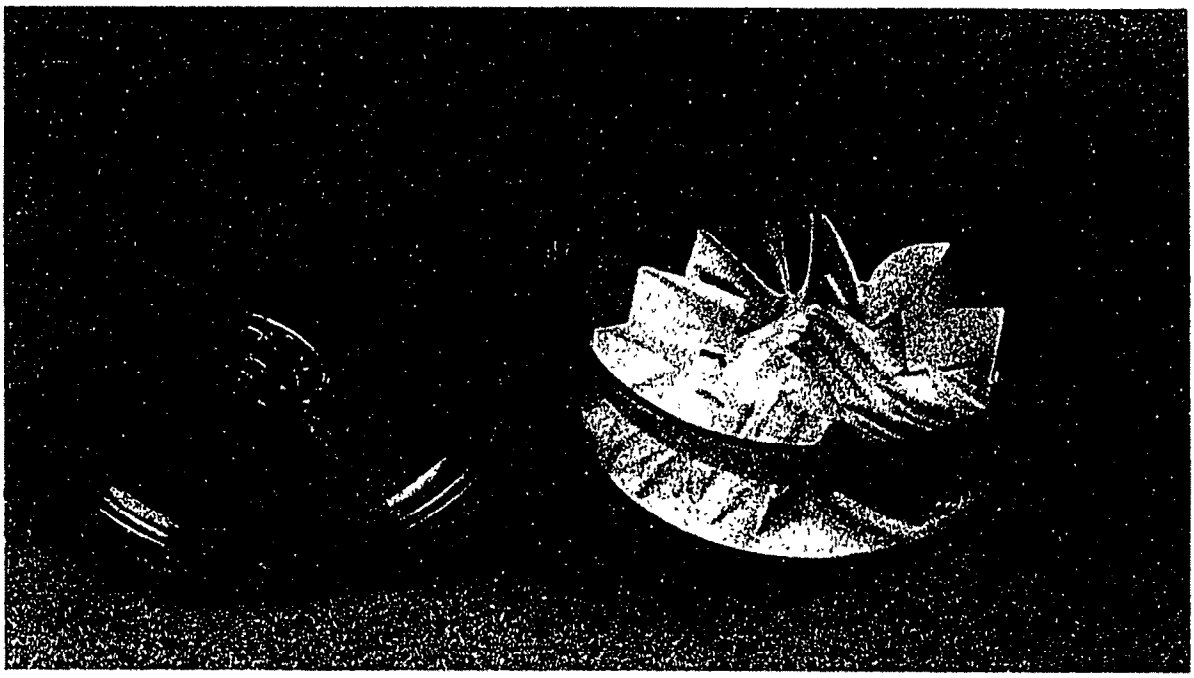

Fig. 1

Actual substrata

A *coffee extract*, containing about 16½% of solids, was prepared in the following way.

About 150 g of commercially ground, roasted Africa coffee was moistened with water and then repeatedly percolated with about 300 ml of water in a steam-heated laboratory percolator (50 × 4 cm). The concentration of the total solids in the extract finally obtained was determined by recalculation of the $d_4^{20} = 1.0660$ with a standard table[14].

A sample of commercial *grenadine syrup* ($n_D^{20} = 1.4418$, corresponding to 60% of solids) could not be desiccated as such, since its pH was 2.2, which should have induced serious caramellisation reactions on drying. It was therefore partially neutralized with 0.5 *N* sodium hydroxide, until its — electrometrically controlled — pH was 6.8. The solution obtained was then diluted to about 20% of solids; in the calculation the Na added and H replaced were accounted for.

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A sample of commercial *orange syrup* (n_D^{20} = 1.4364, solids = 58%; pH = 2.2) was treated analogously until its pH was 6.6 and it contained about 20% of solids.

Samples of *confectioned pears and cherries*, containing about 70% of total solids, pH = 4.2 and 3.7 resp., were homogenized in a Turmix-apparatus, diluted 1 to 4 under adjustment of their pH to about 6.7, and then treated as outlined above.

A sample of commercial *tomato puree* was analyzed for pH, reducing sugars, saccharose, levulose[15], NaCl[4], total solids (from n_D^{20} = 1.3620 according to BIGELOW[1]), and insoluble solids.

To determine the latter figure about 2 g of puree was diluted and homogenized with 100 ml of distilled water and then centrifuged for 10' at 2000 r/min. The fairly clear serum was filtered over a tared oven-desiccated folded filter (Ø = 15 cm) and the residue washed and centrifuged again until the liquid showed no Cl-reaction and no reduction when heated with the LUFF-SCHOORL-reagent[13]. Then the residues were transferred to the filters previously used for filtering the sera, and dried to constant weight in a well ventilated standard oven at 102.5° C. Insoluble solids were calculated from the residue by subtracting 0.7% for water bound by the macromolecular dry substance [8].

The data obtained are recorded in Table I.

TABLE I

ANALYTICAL DATA OF TOMATO PUREE USED

pH	Na Cl %	invert sugar %	surplus levulose %	saccharose %	total solids %	insol. solids %
3.6	1.3	9.8	0.7	1.6	20.4	2.6

The puree was adjusted to pH = 6.6 in the usual way and then diluted to about 10% of solids, since a more concentrated dispersion could not be distributed evenly over the filter paper pile.

METHOD

The BUYZE-*piles* were prepared from folded filter papers (Whatman No, 12 or Schleicher & Schüll No. 588) of Ø = 15 cm and current circular filter papers of the same type. The flasks, equipped with the piles, were dried in a standard electric oven (65 × 45 × 45 cm) at an average temperature of 102.5°C and an average absolute water vapour pressure of 10 mm Hg. They were closed in the oven when still hot and then transferred quickly to a desiccator in which calcium chloride was present. After cooling for exactly 30 min they were weighed to 0.2 mg. Drying was continued until the weight changes noticed in consecutive weighings did not surpass 1.0 mg.

Over the piles, dried in this way, about 2 ml (in the case of diluted tomato purée: 5 ml) of the substrata were *dispersed* within 1 min with the help of a 5 ml-pipette. Moistening the pile was effected by applying one drop of the substratum to each of the vertical edges of the periferous cylinder and distributing

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the rest evenly over the central cone. The flasks were then closed immediately and weighed quickly.

Drying was carried out as described for the tares. The time required for reaching "constant weight" (t_c) was noted in every determination and from these data the average value $\bar{t}_c$ (in hours) was calculated.

RESULTS

The results obtained, when the model substrata were analyzed in two series of duplicates, are recorded in Table II.

TABLE II

DETERMINATION OF TOTAL SOLIDS IN MODEL SUBSTRATA

Substratum	pH	Total solids *(real)* calculated	Total solids (Josse-Buyze) (%)					$\bar{t}_c$ (h)
			1	2	3	4	Av	
Meat-curing brine	6.6	18.2	18.2	18.2	18.2	18.2	18.2	3
Saccharine sap	6.0	18.6	18.5	18.6	18.5	18.5	18.5	4
Malt extract	4.6	18.3	18.2	18.1	18.1	18.1	18.1	4

The results obtained with the actual substrata are presented in Table III

TABLE III

DETERMINATION OF TOTAL SOLIDS IN ACTUAL SUBSTRATA

Substratum	pH	Total solids (from n_D^{20} or d_4^{20}) calculated (%)	Total solids (Josse-Buyze) (%)					$\bar{t}_c$ (h)
			1	2	3	4	Av	
Coffee extract	4.8	16.6	16.1	16.1	16.0	16.0	16.1	3
Grenadine syrup	6.8	60.0	60.0	60.0	60.0	60.0	60.0	4
Orange syrup	6.6	57.6	56.8	57.0	57.1	56.9	57.0	4
Pears, confectioned	6.7	69.5	69.2	69.1	—	—	69.2	2
Cherries, confectioned	6.8	70.7	69.7	69.8	—	—	69.8	2
Tomato puree	6.6	20.4	21.7	21.5	21.7	21.7	21.7	5

DISCUSSION

The replicates obtained by this technique are very satisfactory, thus confirming Buyze's claim.

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The correlation between calculated and found values for total solids is excellent as far as concerns the data obtained with *model substrata*. The correlation between calculated data and the values found in practice in *commodities*, is satisfactory for the products, where total solids can be calculated with a high degree of certainty from refractive indices, but, as can be expected, is poorer with substrata where the latter calculation is problematic, e.g. in tomato products.

SUMMARY

The filter-paper technique of JOSSE for determination of total solids in liquid food products poor in biocolloids, as recently modified by BUYZE, gives results of excellent consistency, when applied to synthetic mixtures and to fruit syrups of well-known composition. The time required for reaching constant weight of the residue varies from 2-5 (average 3½) h at 100° C.

RÉSUMÉ

La technique de JOSSE, à l'aide de papier filtre modifiée récemment par BUYZE, pour le dosage des solides dans les produits alimentaires liquides, pauvres en biocolloïdes donne d'excellents résultats dans le cas de mélanges synthétiques et de sirops de fruits de composition connue. Le temps nécessaire pour obtenir un poids constant du résidu varie de 2 à 5 heures (en moyenne 3½), à 100°C.

ZUSAMMENFASSUNG

Die, kürzlich von BUYZE abgeänderte, JOSSEsche Filtrierpapier-Methode zur Bestimmung des Gesamtgehaltes an festen Stoffen in flüssigen Nahrungsmitteln, die arm an Biokolloiden sind, gibt ausgezeichnet übereinstimmende Resultate, wenn man sie auf synthetische Gemische und Fruchtsyrupe von bekannter Zusammensetzung anwendet. Die zur Erhaltung eines Rückstandes von konstantem Gewicht benötigte Zeit beträgt 2-5 (durchschnittlich 3½) Stunden bei 100°C.

REFERENCES

1 W. D. BIGELOW *et al.*, Cited by: W. V. CRUESS, *Commercial Fruit and Vegetable Products*. 3rd Edition, New York, 1948. p. 448.
2 H. G. BUYZE, *Chem. Weekblad*, 46 (1950) 342.
3 J. W. EVANS AND W. R. FETZER, *Ind. Eng. Chem. Anal. Ed.*, 13 (1941) 855.
4 K. W. GERRITSMA, J. H. VAN DE KAMER AND J. WILLEMS, *Chem. Weekblad*, 46 (1950) 213.
5 J. J. GHIJSEN AND H. J. P. SUYKERBUYK, *Chem. Weekblad*, 46 (1950) 474.
6 M. A. JOSSE, *Bull. Assoc. Chim. Sucr. Distill.*, 10 (1893) 656.
7 T. J. MITCHELL, *J. Sci. Food and Agric.*, 1 (1950) 122.
8 D. A. A. MOSSEL, *Rec. Trav. Chim.*, 69 (1950) 932.
9 J. PIEN ET H. MEINRATH, *Ann. Fals. Fraudes*, 31 (1938) 282.
10 J. F. REITH, D. A. A. MOSSEL AND J. H. VAN DE KAMER, *Anal. Chim. Acta*, 2 (1948) 359.
11 G. W. ROLFE AND G. DEFREN, *J. Am. Chem. Soc.*, 18 (1896) 869.
12 G. W. ROLFE AND W. A. FAXON, *J. Am. Chem. Soc.*, 19 (1897) 698.
13 N. SCHOORL, *Z. Untersuch. Lebensm.*, 57 (1929) 566.
14 A. THIEL, R. STROHECKER UND H. PATZSCH, *Taschenbuch für die Lebensmittelchemie*. Berlin, 1947.
15 F. TH. VAN VOORST, *Anal. Chim. Acta*, 2 (1948) 813.

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