DETERMINATION OF THE WATER CONTENT OF FOODS

II. INDIRECT DETERMINATION OF WATER IN POTATO STARCH BY MEANS OF PHOSPHORUS PENTOXIDE AS A REFERENCE METHOD AND IN SERIAL ANALYSIS

by

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INTRODUCTION

In the preceding general article on the indirect determination of water content[4], the ultimate conclusion was that it is impossible to outline an indirect method suitable for the estimation of the water content of all raw materials. Judging each individual specimen on its own merits, therefore, the investigator applies that method which, on theoretical grounds, he considers best calculated to produce a value most nearly approximating the true water content. While this may have considerable value as a reference method, it is inexpedient for practical purposes.

The well-known PREGL method of drying over P_2O_5 in vacuo[3] has proved to be a very satisfactory reference method for many raw materials, provided the drying temperature be properly related to the nature of the material; in many instances it has, moreover, been found to be a useful routine method.

Since no detailed description of the method is to be found in the literature, the procedure, as provisionally standardized in the Netherlands for the estimation of the water content of potato starch[7], may conveniently be dealt with in this article.

The principle of this test is the elimination of water from the sample in an atmosphere virtually deprived of all moisture by the presence of P_2O_5. Desiccation is accelerated by working in vacuo.

DESCRIPTION OF THE METHOD

Apparatus

A balance, weighing with an accuracy of 1 mg;

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4 porcelain or aluminium weighing boats length about 7 cm, diameter 1.5 cm as shown in Fig. 1.

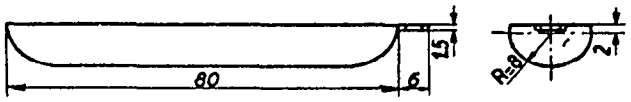

Fig. 1

2 thick-walled glass drying tubes with ground stopper; greased with refined petrolatum, Fig. 2;

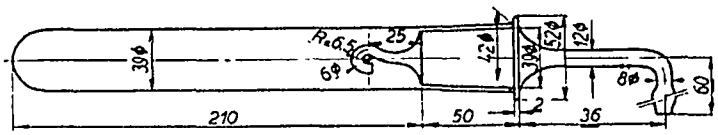

Fig. 2

1 vacuum pump (*e.g.*, water-jet vacuum pump);

1 drying oven adjusted at 75° C with openings and clips by means of which half the drying vessel can be kept outside the oven; see Fig. 3;

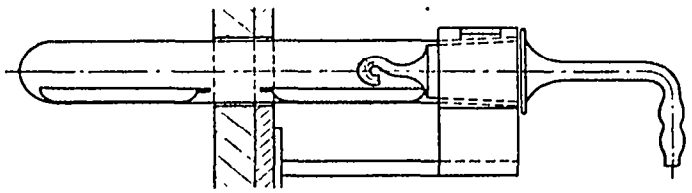

Fig. 3

1 piece of strong copper wire hooked at one end;

4 weighing vessels with ground stopper, Fig. 4.

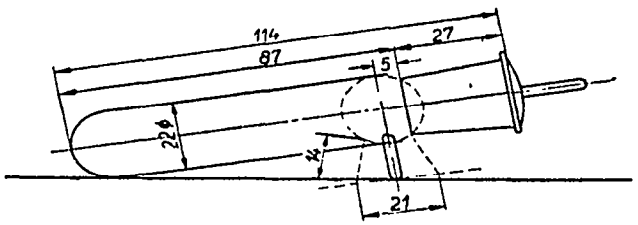

Fig. 4

Reagent

Phosphorus pentoxide, A.R.

Procedure

Place the empty porcelain weighing boat inside the weighing vessel, close it with a glass stopper and determine its weight (a grams). Then fill the boat with the substance to be dried, insert it into the weighing vessel, close it with the glass stopper and weigh again (b grams).

With the aid of the copper wire work the filled weighing boat to the hinder part

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of the drying tube and place a second porcelain boat filled with P_2O_5 in front of it. Close the drying tube and push it halfway into the drying oven so that the boat containing the substrate shall be heated, whereas the P_2O_5 container remains outside the oven. Evacuate to a pressure of 10 to 20 mm Hg. Leave the drying tube in the oven for some hours and then supply fresh P_2O_5. After a drying period of about 16 hours take the tube out of the oven and let it cool down. Then release the vacuum by admitting air slowly, remove the boat containing P_2O_5 and transfer the other boat to the weighing vessel as quickly as possible, close it immediately and weigh (c grams).

Repeat this drying process for 6 hours (c' grams).

If $c-c' < 1.5$ mg, desiccation is considered to be complete. If $c-c' > 1.5$ mg, repeat the operation until two successive weighings differ by less than 1.5 mg.

Calculation

The percentage water content is equal to: $W = \frac{b-c'}{b-a} \cdot 100$

Remarks

1. Care must be taken to prevent the grease on the tube-grounds from contaminating the dishes when slipped into and out of the tube. It is therefore advisable to cover the greased parts with a strip of paper before passing the boats over them.

2. When releasing the vacuum, air must be admitted *slowly*, otherwise the dried substrate may be blown out of the boat. It appeared to be unnecessary to dry the air first, as any moisture it contains is extracted from it by the P_2O_5 before it reaches the substrate.

EXACTNESS AND ACCURACY

In order to verify the exactness and accuracy of this method, we determined the water content of $BaCl_2.2H_2O$, since MOSSEL[2] had shown that this hydrate — originally introduced by SCHMIDT[5] — has many advantages as a reference substance for water content determinations. The results are given in Table I.

TABLE I

THE WATER CONTENT OF $BaCl_2.2H_2O$ (14.74 % OF H_2O) BY THE STANDARD METHOD AT 75° C

Hydrate (g)	Loss of Water in % after varying drying periods (days)				
	2½	3½	4	5	6
3.2466	14.74	14.70	14.73	—	—
3.2452	14.74	14.70	14.73	—	—
4.4238	—	—	14.75	14.73	14.73
3.4650	—	—	14.71	14.67	14.72
3.6545	—	—	14.76	14.74	14.74

These figures demonstrate the exactness and accuracy of the method.

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APPLICATION OF THE METHOD TO POTATO STARCH

For the following experiments three 2.5–3 g samples of potato starch were weighed out. After establishment of constant weight at 75° C, the temperature was raised to 100° C; when at this temperature, too, the weight remained constant, the temperature was increased further, first to 120° C and then to 140° C.

The results of these experiments are summarized in Table II.

TABLE II

THE WATER CONTENT OF POTATO STARCH BY DRYING BESIDE P_2O_5 IN VACUO AT INCREASING TEMPERATURE

Time Drying	Temp. (° C)	Water Content in %		
		No. 1	No. 2	No. 3
17 hours	75	18.81	18.84	18.89
24 ,,	75	18.81	18.84	18.86
41 ,,	100	18.83	18.87	18.92
48 ,,	100	18.83	18.86	18.90
65 ,,	120	18.88	18.87	18.91
72 ,,	120	18.91	18.89	—
89 ,,	120	18.84	18.91	—
96 ,,	140	18.89	18.94	—
120 ,,	140	18.93	18.91	—
144 ,,	140	18.88	18.92	—

The P_2O_5 was renewed after 2 hours and after 17 hours of drying. After the sixth weighing, a pale yellow discoloration was clearly visible in all starch samples.

A separate drying test was carried out at 20° C and, at the same time, a triplicate determination at 75° C for checking purposes. The results are given in Table III.

TABLE III

THE WATER CONTENT OF POTATO STARCH DETERMINED BY DRYING BESIDE P_2O_5 IN VACUO AT 20° AND 75° C

Drying Time	Water Content in %				
	20° C		75° C		
	No. 1	No. 2	No. 1	No. 2	No. 3
17 hours	—	—	18.85	18.87	18.84
24 ,,	—	—	18.85	18.87	18.85
41 ,,	—	—	18.84	18.86	18.85
3 days	18.50	18.45	—	—	—
4 ,,	18.53	18.63	—	—	—
5 ,,	18.58	18.69	—	—	—
7 ,,	18.74	18.74	—	—	—
8 ,,	18.71	18.76	—	—	—
10 ,,	18.70	18.78	—	—	—
14 ,,	18.81	18.82	—	—	—
17 ,,	18.83	18.86	—	—	—
19 ,,	18.85	18.84	—	—	—

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The P_2O_5 was renewed after 2 and after 20 hours of drying. From these experiments the following conclusions may be drawn:

The experiments carried out at 20° C—the lowest drying temperature—show "constant weight" after 17 days; hence the values 18.83, 18.85% and 18.86, 18.84% may be claimed as the nearest approximation to the "true" water content of the sample of potato starch.

Seeing that the results obtained at 75° C do not differ significantly from those found at 20° C, it may safely be assumed that the true water content values are obtained at 75° C.

The fact that values recorded at higher temperatures show a tendency to increase above the level found at 75° C, and the pale yellow discoloration of the desiccated potato starch indicate incipient decomposition above 75° C.

Hence, drying beside P_2O_5 at 75° C in vacuo is the most suitable reference methode for potato starch.

THE USE OF THE REFERENCE METHOD AS A ROUTINE METHOD FOR THE DETERMINATION OF THE WATER CONTENT OF POTATO STARCH

Owing to variations in the relative humidity of the surrounding air, differences of 1.7% at 75° C, 0.7% at 100° C and 0.1% at 128° C are liable to be found in the water content of starch[7]. Hence no commercially useful water content of potato starch can be determined below 128° C in a *freely ventilated* oven; indeed, even at 128° C the deviation from the true water content may be as much as 0.1%. It is therefore necessary to make the water test in a moisture-free atmosphere, or to dry the material at a temperature above 128° C. In actual practice 175° C has been found to meet the case[1]. The drawback to this latter temperature is, however, that some low grade potato starches suffer appreciable decomposition and the recorded result of the water content test is thus too high. The only alternative, then, is to dry in a moisture-free atmosphere at the highest admissible temperature, to accelerate drying. Here, too, our method can be employed to good purpose.

Our first care was to ascertain the necessary drying time at 130° C to attain "constant weight" (apart from the slight decomposition) and also whether the material of the boat, *viz.*, aluminium or porcelain, affected the result in any way.

The results of these experiments are given in Table. IV. The results show that drying at 130° C is complete in one hour and that there is no noticeable difference in outcome between a porcelain and an aluminium boat, though it may be well to point out that porcelain takes a little longer to cool down.

As 3 × 1 g of P_2O_5 are required for complete drying over P_2O_5, we next tried to *economize* 2 × 1 g of P_2O_5 by previously drying the material in a freely ventilated oven. To do this we left the boat of weighed potato starch in the weighing

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TABLE IV

DETERMINATION OF THE TIME NEEDED TO DRY POTATO STARCH AT 130° C TO CONSTANT WEIGHT IN PORCELAIN AND IN ALUMINIUM BOATS

Drying time at 130° C (h)	Water Content in %	
	Porcelain Boat	Aluminium Boat
0.5	18.77	18.81
1	18.84	18.93
2	18.85	18.88
3	18.92	18.90

The P_2O_5 was renewed after 15 min and again after 30 min.

vessel, placing the latter, open, in a freely ventilated oven of the required temperature. After the proper drying time the vessel was removed from the oven, the boat was carefully withdrawn from the vessel and placed in the vacuum drying tube. The procedure is then as described on page 2. The results of this experiment are given in Table V.

TABLE V

THE WATER CONTENT OF POTATO STARCH DETERMINED BY DRYING BESIDE P_2O_5 IN VACUO AT 130° C AFTER PRELIMINARY DRYING IN A FREELY VENTILATED OVEN AT 100° C AND 130° C

Preliminary Drying Time at 100° C in min	Preliminary Drying Time at 130° C in min	Water expelled by Preliminary Drying in %	Drying Time over P_2O_5 in vacuo at 130° C in min	Water Content in %	
15	—	4.3	45		18.64
30	—	5.7	30		18.86
45	—	11.2	15		18.83
—	15	9.9	45	18.37	18.64
—	30	14.4	30		18.81
—	45	16.9	15		18.89
—	30	—	20	18.75	18.87
—	30	—	30	18.90	18.89
—	30	—	40	18.86	18.91

It is evident from these figures that P_2O_5 can be economized without disadvantage by preliminary drying; nor does it complicate the procedure, since all that is required is to transfer the samples from the oven to the vacuum tube instead of renewing the P_2O_5. The material may conveniently be dried preliminarily in the same tube as that which contains the boat of potato starch when it is weighed.

It is clear from the results that the final value is reached after the material has been dried preliminarily for 30 minutes at 130° C and then for 30 minutes beside P_2O_5 in vacuo, likewise at 130° C.

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The method thus applied produces quick, satisfactory results and at the same time minimizes the consumption of P_2O_5.

Finally, steps were taken to verify the reproducibility of the reference and the routine method.

All the following series of tests were carried out by one of us, each series on a different day. The results are given in Table VI.

TABLE VI

THE WATER CONTENT OF POTATO STARCH ACCORDING TO THE REFERENCE METHOD AND THE ROUTINE METHOD

Method	Reference Method (Drying beside P_2O_5 in vacuo at 75° C)		Routine Method (Preliminary Drying at 130° C, followed by Drying beside P_2O_5 in vacuo at 130° C)	
Series	I	II	I	II
Percentage Water Content	18.85 18.86 18.87 18.87 18.87 18.88 18.90 18.91	18.81 18.81 18.81 18.84 18.85 18.85 18.86 18.87	18.78 18.82 18.84 18.80 18.83 18.84 18.80 18.83 18.85 18.81 18.83 18.87 18.81 18.83 18.89 18.81 18.83 18.91 18.82 18.83 18.91	18.80 18.83 18.88 18.80 18.83 18.88 18.80 18.84 18.88 18.81 18.85 18.88 18.82 18.86 18.89 18.82 18.87 18.90 18.82 18.87 18.91
Number of Determinations . . .	8	8	21	21
Extreme Values	18.85–18.91	18.81–18.91	18.78–18.91	18.80–18.91
Arithmetic Mean	18.88	18.84	18.84	18.85
Standard Deviation σ $\sqrt{\frac{(x-x')^2}{n-1}}$	0.02	0.02	0.03	0.04

SUMMARY

A description is given of the method of PREGL for the determination of the water content of potato starch with the aid of P_2O_5 in vacuo at 75° C. Its exactness and accuracy are verified with $BaCl_2.2H_2O$ as reference substance.

Results obtained with this method do not show a significant deviation from "true" water values: hence, the method may be generally used as a reference method.

An outline is then given of the procedure by which the method may be utilized as a routine

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method, *viz.*, by combining preliminary drying in a freely ventilated oven at 130° C for half an hour with desiccation beside P_2O_5 in vacuo at 130°, again for half an hour.

Finally, both the reference method and the routine method are put to the test of reproducibility and are found to produce satisfactory check values.

RÉSUMÉ

On donne une description de la méthode de PREGL, pour le dosage de l'eau dans l'amidon de pomme de terre, à l'aide de P_2O_5, dans le vide, à 75° C. Son exactitude et sa précision sont vérifiées par $BaCl_2.2H_2O$ comme substance de référence.

Les résultats obtenus sont satisfaisants et permettent d'utiliser ce procédé comme méthode de référence.

On propose un procédé pour des analyses en série, combinant le séchage dans un four ventilé à 130° pendant 30 minutes avec un séchage sur P_2O_5 dans le vide à 130°, également pendant 30 minutes.

Enfin, la méthode de référence ainsi que la méthode pour les analyses en série ont été soumises à un essai de reproductibilité et ont donné des résultats satisfaisants.

ZUSAMMENFASSUNG

Die PREGL'sche Methode zur Bestimmung des Wassergehaltes von Kartoffelstärke mit Hilfe von P_2O_5 im Vakuum bei 75° wurde beschrieben und ihre Genauigkeit mit Hilfe von $BaCl_2.2H_2O$ als Vergleichssubstanz geprüft.

Die mit dieser Methode erzielten Ergebnisse zeigen keine bedeutende Abweichung von den "wahren" Wasserwerten: die Methode kann daher allgemein als Vergleichsmethode angewendet werden.

Eine Arbeitsvorschrift für Reihenanalysen wird umrissen; man trocknet zuerst in einem ventilierten Ofen bei 130° C und dann in Gegenwart von P_2O_5 im Vakuum bei 130° C und zwar je eine halbe Stunde.

Die Vergleichsmethode und die Routineprobe für Reihenanalysen geben gut reproduzierbare Werte.

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