sium nitrate before ashing (Fraps). In all these cases the gases still contain sulphur.

(5) By far the greater part of the loss of sulphur occurs during the preliminary charring, a much smaller part during the burning of the charred mass to ash.

(6) Combustion in a stream of oxygen, with absorption of the sulphur-containing products of charring and combustion either in heated sodium carbonate in the combustion tube or in a special apparatus, gives, under proper conditions, absolute values for the total sulphur. Such results are, however, exceedingly difficult, if not quite impossible, to attain by either the original Berthelot method or the Sauer method. The writer considers that he has ascertained and described the arrangement of apparatus and the details of manipulation which render possible the attainment of such accurate results with ease and certainty. It is essential to burn the escaping gases completely with an excess of oxygen, introduced laterally at a certain point in the combustion tube, before absorbing the sulphuric acid from them.

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[CONTRIBUTION FROM THE HAVEMEYER LABORATORIES, COLUMBIA UNIVERSITY, NO. 91.]

THE DETERMINATION OF NITROGEN IN FOOD MATERIALS AND PHYSIOLOGICAL PRODUCTS.

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Received January 12, 1904.

FOR THE determination of nitrogen in ordinary animal and vegetable substances where it exists mainly as proteids or related compounds, some modification of the Kjeldahl method is now almost always employed. The various modifications differ mainly in that different substances are used to facilitate the decomposition of the organic matter by the boiling sulphuric acid.[1] Those which appear to be most extensively used are mercury (with or without potassium permanganate), potassium sulphate, and copper sulphate. The combination of two or more of these reagents has frequently been suggested and has recently been adopted

[1] Since the literature of the subject is quite extended and quite familiar to those likely to be interested in the present work, specific references are omitted and the processes tested are indicated without detailed description.

to a considerable extent, especially in the agricultural laboratories of Germany.

Probably most laboratories in this country use one of the two processes adopted by the Association of Official Agricultural Chemists.[1] In the first, the sample is treated with 20 cc. concentrated sulphuric acid, and 0.7 gram mercury, boiled until colorless or of a pale straw color, and the solution is then treated with potassium permanganate to complete the decomposition of any organic compounds possibly remaining.[2] In the scond (Gunning) method, the sample is treated with 20 cc. of concentrated sulphuric acid and 10 grams of potassium sulphate, and the solution boiled until colorless or nearly so.

Various observers have noted that boiling until colorless is not always sufficient to insure the complete transformation of all the nitrogen into ammonium sulphate, and that, unless the digestion is prolonged beyond this point, the results are liable to be too low. So far as we are aware, however, this source of error has not been systematically studied. That it is not very generally appreciated in this country is sufficiently shown by the fact that the "official methods" have not been corrected in this respect. In the work here described we have studied this point as well as the use of different combinations of reagents, with a view to the selection of a simple and rapid procedure for the complete transformation of the nitrogen of food materials and physiological products.

ANALYTICAL DATA.

First Series.

(DETERMINATIONS MADE BY E. O. AND H. C. S.)

Six methods of digestion were tried:

(1) Sample + 20 cc. concentrated sulphuric acid + 0.7 to 1 gram mercury, boiled till colorless.

(2) The same, boiled one hour after colorless.

(3) The same, boiled three hours after colorless.

(4) The same, boiled until colorless and then treated with potassium permanganate.

(5) Sample + 20 cc. concentrated sulphuric acid + 0.7 to 1

[1] Bull. 46 (Revised) Bureau of Chemistry, U. S. Department of Agriculture.

[2] In many of the laboratories where this method is employed, the use of potassium permanganate is considered unnecessary and is omitted in routine work.

gram mercury + 1 gram copper sulphate, boiled same length of time as in (1).

(6) Treated as in (5), but boiled one hour longer.

The results of this series of determinations are shown in Table A.

TABLE A.—PERCENTAGES OF NITROGEN FOUND IN FIRST SERIES OF DETERMINATIONS.

Substance.	Method of digestion.					
	(1)	(2)	(3)	(4)	(5)	(6)
Lean meat	13.05	13.09	13.12	12.95	13.08	13.16
Dried curd	11.69	11.82	11.80	11.63	11.65	11.79
White of egg	12.58	12.67	12.68	12.64	12.63	12.64
Yolk of egg	11.36	11.53	11.52	11.48	11.38	11.51
Beans	3.49	3.53	3.59	3.53	3.56	3.53
Wheat bran	2.54	2.57	2.55	2.48	2.52	2.58
Average.....	9.12	9 20	9.21	9.12	9.14	9.20

Second Series.

(DETERMINATIONS MADE BY C. B. McL).

In this series the following methods of digestion were compared:

(1) Sample + 20 cc. concentrated sulphuric acid + 0.7 to 1 gram mercury, boiled until colorless.

(2) The same, boiled two hours longer.

(3) Sample + 20 cc. concentrated sulphuric acid + 10 grams potassium sulphate, boiled until colorless.

(4) The same, boiled two hours longer.

(5) Sample + 20 cc. concentrated sulphuric acid + 0.7 to 1 gram mercury, heated until frothing subsided, when 10 grams potassium sulphate were added and the mixture boiled until colorless.

(6) The same, boiled two hours longer.

(7) The same, except that 1 gram copper sulphate was added with the mercury, boiling continued about as long as in (5).

(8) The same, boiled two hours longer than (7).

The results obtained are shown in Table B.

TABLE B.—PERCENTAGES OF NITROGEN FOUND IN SECOND SERIES OF DETERMINATIONS.

Substance.	Method of digestion.							
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
Hide powder ..	15.49	15.74	15.58	15.72	15.69	15.90	15.65	15.90
Lean meat	12.75	13.08	12.93	13.18	13.00	13.22	12.88	13.21
Peptone	12.38	12.58	12.83	12.91	12.66	12.90	12.55	12.96
Dried curd.....	11.48	11.73	11.69	11.71	11.73	11.70	11.70	11.82
Average ...	13.03	13.28	13.26	13.38	13.27	13.43	13.20	13.47

Third Series.

(DETERMINATIONS MADE BY E. O.)

In this series only sulphuric acid, mercury and potassium sulphate were used. The methods of digestion were:

(1) Sample + 20 cc. concentrated sulphuric acid + 0.7 to 1 gram mercury, boiled until colorless.

(2) The same, boiled two hours longer.

(3) Sample + 20 cc. concentrated sulphuric acid + 10 grams potassium sulphate, boiled until colorless.

(4) The same, boiled two hours longer.

(5) Sample + 20 cc. concentrated sulphuric acid + 0.7 to 1 gram mercury + 10 grams potassium sulphate (added after frothing had subsided), boiled until colorless.

(6) The same, boiled one-half hour longer.

(7) The same, boiled two hours longer than (5).

The results obtained are given in Table C.

TABLE C.—PERCENTAGES OF NITROGEN FOUND IN THIRD SERIES OF DETERMINATIONS.

Substance.	Method of digestion.						
	(1)	(2)	(3)	(4)	(5)	(6)	(7)
Beans	3.59	3.61	3.58	3.64	3.61	3.69	3.68
Peas	3.62	3.65	3.61	3.66	3.65	3.65	3.69
Gluten flour	2.73	2.74	2.67	2.70	2.74	2.77	2.79
Gluten biscuit	4.68	4.78	4.74	4.80	4.79	4.77	4.78
Linseed meal	7.31	7.37	7.37	7.44	7.35	7.46	7.51
Cottonseed meal	7.34	7.42	7.31	7.44	7.50	7.50	7.54
Cocoa	2.97	3.07	3.09	3.11	3.07	3.15	3.15
Pepper	2.12	2.13	2.13	2.13	2.16	2.23	2.31
Globulin	16.63	16.73	16.70	16.81	16.59	16.81	16.91
Gelatin	15.50	15.69	15.47	15.64	15.71	15.75	15.83
Dried curd	11.55	11.86	11.55	11.76	11.82	11.84	11.86
White of egg	12.54	12.63	12.48	12.58	12.54	12.68	12.70
Yolk of egg	11.49	11.61	11.41	11.58	11.60	11.59	11.63
Lean meat	12.97	13.31	12.84	13.17	13.25	13.38	13.37
Peptone	12.68	12.81	12.68	12.89	12.56	12.88	12.91
Hide powder	15.37	15.39	15.35	15.39	15.66	15.69	15.87
Human feces	2.58	2.59	2.57	2.62	2.64	2.66	2.69
Uric acid*	32.81	33.21	32.88	33.06	32.94	33.12	33.18
Average	9.92	10.03	9.91	10.02	10.01	10.09	10.13

* The uric acid was weighed air-dry and the results are not corrected for the small amount of hygroscopic moisture present.

SUMMARY OF RESULTS.

Seventeen substances were tested with from six to twelve modi-

fications of the method of digestion. In several cases similar substances were tested by different analysts or at different times.

Whether the sample was decomposed by digestion with sulphuric acid and mercury or sulphuric acid and potassium sulphate, the transformation of nitrogen into ammonium sulphate was seldom, if ever, complete when the solution became colorless or a permanent faint straw color.

By continuing the digestion for two hours longer, higher results were obtained. The extent of this discrepancy varies both with the nature of the sample and with the judgment of the analyst as to the point at which the solution should be considered "colorless." The average difference found by us was about 1 per cent. of the amount of nitrogen present.

When both mercury and potassium sulphate were used in the digestion, it was still necessary to continue the boiling beyond the point at which the solution became colorless, but the time required was greatly reduced and the results were slightly higher than those obtained by the use of either reagent alone.

In the few cases tested, neither the time required nor the result obtained was appreciably affected by the use of copper sulphate or potassium permanganate in addition to the reagents already mentioned.

These results indicate that for the determination of nitrogen existing in the form of proteids and related compounds, the following procedure is advantageous, both as to accuracy and speed: Treat the sample with 20 cc. of concentrated sulphuric acid and 0.7 to 1 gram of mercury, heat gently until frothing subsides, then add 10 to 15 grams of potassium sulphate and boil. Usually the solution becomes colorless in less than thirty minutes, and the transformation of nitrogen into ammonium sulphate is complete within an hour. In a few cases appreciably higher results were obtained by boiling for two hours.

Having in view a similar study with organic compounds of known structure, we shall postpone any attempt to explain the discrepancies above described.

A large part of the analytical work was performed in the laboratory of Wesleyan University, for the privileges of which we are indebted to the courtesy of Professor W. O. Atwater.

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COLUMBIA UNIVERSITY.
January, 1904.