AN ERROR IN THE PRESENT OFFICIAL METHOD FOR THE DETERMINATION OF ALBUMINOID NITROGEN, AND THE EFFECT OF THE PRESENCE OF METALS PRECIPITABLE BY POTASSIUM SULPHIDE IN THE DETERMINATION OF NITROGEN BY KJELDAHL'S METHOD.*

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In the determination of nitrogen by the Kjeldahl method directions are given for the addition of potassium sulphide to precipitate *all* mercury from the solution in order to prevent the formation of mercuro-ammonium compounds which are not completely decomposed by soda solution.

The determination of albuminoid nitrogen according to the present official method, requires the use of 0.7 to 0.8 grm. of copper hydroxide previous to the determination of the nitrogen by the Kjeldahl method.

The copper oxide is carried along in the determination, and is freely soluble in the sulphuric acid; no provision is made for its removal, and when the potassium sulphide is "added to precipitate *all* mercury in solution," copper sulphide is alike precipitable in the acid solution with the mercury. The copper present is more than enough to combine with all the potassium sulphide; the result is that neither metal is completely precipitated, and a large and variable portion of the mercury is left in the solution and all the conditions are favorable for the formation of mercuro-ammonium compounds. The work given to the potassium sulphide is doubled and the object for which it is added is not attained.

In order to determine to what extent the mercury so left in the solution would effect the results a number of determinations were started; to some the official "20 c.c. of potassium sulphide" were added, and to others a sufficient quantity of the same solution to

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precipitate *all* the copper as well as *all* mercury in solution. The large amount of precipitate formed in the latter case caused so much bumping that only one determination out of six could be completed.

In order to obviate this difficulty the operation was carried on exactly as required by the official method except that previous to distillation the acid solution was made up to 200 c.c. and divided into two 100 c.c. portions. One portion was distilled with the relative official amount of potassium sulphide, and the second with enough of the same solution to complete the reaction. This gives a more satisfactory basis for the comparison of results, since all the conditions previous to the distillation are the same as to the degree of oxidation and the amounts of mercury and copper in solution.

The following results were obtained :

	50 c.c. K_2S	30 c.c. K_2S.	Difference.
Ensilage No 1.....	1.28 per cent.	1.12 per cent.	0.16 per cent.
	1.25 “	1.14 “	0.11 “
No. 2.....	0.66 “	1.53 “	0.13 “
	0.66 “	1.60 “	0.06 “
No. 3.....	1.36 “	1.27 “	0.09 “
	1.20 “	1.15 “	0.05 “
No. 4.....	1.52 “	1.45 “	0.07 “
	1.54 “	1.45 “	0.09 “
No. 5.....	1.18 “	1.08 “	0.10 “
	1.23 “	1.12 “	0.11 “
	Average...............		0.096

Since all the conditions previous to distillation and addition of the potassium sulphide were the same in each portion as to the degree of oxidation, and amounts of mercury and copper in solution the constant larger proportion of nitrogen yielded with the 50 c.c. of potassium sulphide must be due to the incomplete precipitation of all the mercury in solution when only 20 c.c. is added. The average difference in the ten portions amount to nearly a tenth of one per cent.

Blank determinations with both 20 and 50 c.c. portions of potassium sulphide showed no quantity of nitrogen. A difference of a tenth of one per cent. of nitrogen when multiplied by the protein factor would make a difference of over six-tenths of one per cent. of protein, a difference which is too great to be overlooked.

Some change must therefore be made in combining Stutzir's and Kjeldahl's methods. The following proportions of substance and solutions will give satisfactory results. Start with 7 gms. of substance instead of 1 gram, take a quantity of copper hydroxide and glycerine solution equivalent to .5 to .6 grams of the hydroxide instead of .7 to .8, and finally use 30 c.c. of potassium sulphide instead of 20. Trials with these proportions on the committee sample of corn meal gave 1.56 per cent. and 1.55 per cent. of albuminoid nitrogen with the old proportions, with all of the copper and mercury precipitated, 1.56 per cent. of albuminoid nitrogen. (1)

In the determination of nitrates by the official method 2 grams of zinc dust are used but the previous addition of 2 grams of salicylic acid converts the zinc into an insoluble and crystalline form at the close of the digestion. This is shown to be true by the following results of the analysis of pure potassium nitrate, when the solution was divided as in the case of the determination of albuminoid nitrogen.

		20 c.c. K_2S	40 c.c. K_2S
1.	Nitrogen	14.02 per cent.	14.02 per cent.
2.	"	13.98 "	13.98 "

If in the above tests the salicylic acid is omitted the results obtained are not uniform, and fall short of the actual amount of nitrogen present. This is due to the presence of the zinc in solution, which causes the same trouble as the copper. Any other metal precipitable by potassium sulphide would be equally liable to produce the same effect, as all of the conditions would be equally favorable for the formation of mercuro-ammonium compounds.

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(1) These changes as given above have been adopted in the official method.