and also by the presence of citric acid in the solution where the "white precipitate" is formed, the trouble is prevented.

H. Neubauer[1] has shown that magnesium ammonium phosphate formed in solutions containing an excess of both ammonia and magnesium salt, which is the condition of precipitation in the method under discussion, contains more than a normal amount of magnesium. The writer has had magnesium of the magnesia mixture to deposit as hydroxide and contaminate precipitates where very large excesses of the magnesia mixture were used in strongly alkaline solution and on long standing, though little or no error is thought to be introduced by this latter cause where the prescribed amount of magnesia mixture is used and too long standing not allowed. It is to the excess of magnesium in the magnesium ammonium phosphate, we consider, that the high results by the molybdate-magnesia method are due.

The results upon which the foregoing discussion is based will appear in the proceedings of the A. O. A. C. in the "Report on Phosphoric Acid" made by the writer to that association at its recent meeting.

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ON THE GUNNING METHOD FOR TOTAL NITROGEN IN FERTILIZERS.

BY W. E. GARRIGUES.

Received September 28, 1894.

COMPARED to the Scovell-Kjeldahl nitrogen method, that of Gunning, modified to include nitrates, does not seem to have met with the favor that its cleanliness and simplicity would warrant. It is believed that this is, in the main, due to the lower results consequent upon the use of the latter process; hence the following record of an investigation of its merits.

The work was commenced by running parallel analyses on fertilizers of unknown composition, containing sodium nitrate, by both of the methods named. In each case the manipulation, adopted by the Association of Official Agricultural Chemists,

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[1] Abstract *J. Chem. Soc.*, of London, October *1893*, 489.

was rigidly adhered to. The appended figures, being the results obtained, speak for themselves:

Kjeldahl with zinc dust. Per cent. nitrogen.	Gunning with sodium thiosulphate. Per cent. nitrogen.		
3.01	2.40	2.40	
4.98	4.30	4.35	4.55
3.53	3.00	3.10	3.24
4.02	3.50	....	....
4.67	4.05	4.21	4.24

A sample was then prepared from pure sodium nitrate and tankage. The latter was valued by the plain Kjeldahl and Gunning methods and by soda-lime combustion, with the following results:

Kjeldahl.	Gunning.	Soda-lime.
7.05	7.05	7.08

The calculated content of the mixture was 8.97 per cent. nitrogen, about one-half of which was in the inorganic form. On submitting this to analysis there were obtained:

Kjeldahl.	Gunning.
8.90	7.17
....	7.31
....	7.46

It was very evident that in the writer's hands the Gunning method, as adopted by the Association, did not convert all the nitrogen present into ammonia. After numerous and varied experiments, which, as is usual when viewed in the light of foregone events, now seem to have been largely unnecessary; successful results were finally obtained by using the same reagents, both kind and quantity, but varying the mode and order of their addition.

For convenience of reference the official directions from Bulletin,38, U. S. Dept. of Agriculture, are here given:

"In a digestion flask holding from 250 to 500 cc. place from 0.7 to 2.8 grains of the substance to be analyzed according to the amount of nitrogen present. Ten grams of potassium sulphate, five grams of sodium thiosulphate, and thirty to thirty-five cc. of the salicylic acid mixture, *i. e.*, thirty cc. of sulphuric acid to one gram of salicylic acid, are then added and the whole digested at a low temperature until frothing ceases."

The above unsatisfactory, or rather useless, results were obtained in this manner. The chief fault is in the addition of potassium sulphate at such an early stage in the process; the large quantity of dry powder forming an effectual blanket over the sample, that for some time resists the permeation of the viscid acid solution.

The best procedure was found to be: Adding the salicylic acid mixture directly to the sample in the flask, which is then shaken until the liquid has thoroughly wetted the dry organic matter, allow to stand about fifteen minutes, and add the thiosulphate with constant shaking. This should be done in four or five portions, following with a fresh crystal only when the one previously added has completely dissolved. The potassium sulphate is then put in and the whole again well agitated. Digest as usual.

Handled in this manner the known mixture above referred to, gave:

8.85
8.87
8.90
8.92

Another sample of dry bone tankage and sodium nitrate was prepared and passed several times through a sixty mesh. sieve. The tankage was valued as before, and showed:

Kjeldahl.	Gunning.	Soda-lime.
4.32	4.32	4.39
4.40	4.42	....

The calculated content of the mixture was 6.49, and the analyses returned:

Kjeldahl.	Gunning.
6.45	6.43
....	6.45
....	6.45
....	6.46

Neither method gave results sufficiently accurate for the valuation of sodium nitrate alone; a carefully prepared sample of that salt showing on analysis:

Kjeldahl.	Gunning.
16.05	15.80
16.35	16.00
....	16.15
....	16.22
....	16.38
Theory 16.49	

It will be noticed that without a single exception the returns of analyses were below the amount actually present. No means of avoiding this loss were found. It would, therefore, seem justifiable to report the highest figure in closely agreeing duplicates instead of the customary average.

Several blanks run through to determine the freedom from nitrogen of the reagents employed, gave from nil to 0.05 per cent. No corrections were made for this.

During the distillation in the Gunning process the solution frequently foams badly; so much so that unless the flame is turned very low, the froth rises into the connecting bulb. The addition of paraffin wax does not always overcome this difficulty, and to avoid it, the use of an ordinary thirty-two-ounce wash-bottle, in place of the 500 to 600 cc. distillation flask is recommended.

There is little doubt that the seat of responsibility for many minor discrepancies is imperfect prepartion of the sample. Attendant upon the use of the official twenty-five mesh sieve, variations in the hands of the same manipulator frequently reach 0.15 per cent., even when exceptional care is exercised. It has, therefore, been found advisable in nitrated goods to quarter down the twenty-five mesh sample to about two ounces and pass that through a sixty mesh sieve; when this is done, the difference in duplicate analyses rarely exceeds 0.05 per cent., and decinormal acid and alkali often fail to show any.

Occasionally it becomes necessary to dry the sample before it can be thus finely powdered; in that case making the moisture determinations of Stillwell and Gladding (Bull. 38, p. 19, U. S. Dept. of Agr.) is the best procedure, *i. e.*, weigh out a portion of about ten grams from the original package when first opened, and determine the total moisture in a water-oven, dry the remainder of the sample to dustiness, and prepare it as above; then weigh out two grams of the fine sample simultaneously with the portion to be used for nitrogen, and ascertain the remaining moisture. A simple calculation brings the nitrogen figure back to the original condition of the goods.

Usually, however, nitrated fertilizers contain only a few per cent. of moisture, as indeed they should, lest the free acid of the phosphate in the presence of water, act on the easily decomposable salt and liberate nitric acid. This theoretic action has been found a sad reality by manufacturers who incorporated nitrate salts with a wet "super," the loss of plant food being accompanied by a destruction of the fiber of the bagging containing the goods.

FALLACIES OF POST-MORTEM TESTS FOR MORPHINE.[1]

BY DAVID L. DAVOLL, JR.

Received August 9, 1894.

IN the qualitative investigations upon morphine in a former series of experiments, the tissue was allowed to putrefy in the open air, a condition which is not present in the case of a buried body, for in the unopened cadaver, the bacterial products are very different in their deportment toward certain reagents, from those formed by aerobic germs and are the interferences which would be met with in chemico-legal work and which give some of the tests for morphine.

The writer, during the spring of 1893, in a series of determinations to ascertain the minimum of loss entailed in the recovery of a weighed amount of commercial morphine sulphate—the form generally employed in instances of poisoning—from a given amount of putrefied tissue, obtained the following results by the method given in detail below:

A. From sixty grams of stale bread, containing 0.2387 gram of morphine sulphate, 0.0844 gram of anhydrous alkaloid were recovered, the equivalent of 0.2245 gram of morphine sulphate; this shows a loss of 0.0142 gram or 5.94 per cent. By qualitative tests upon $\frac{1}{500}$ part of this recovered amount, very decided affirmative results were obtained.

B. From 100 grams of fresh liver, containing 0.3455 gram of morphine sulphate, 0.1163 gram of anhydrous alkaloid were recovered, the equivalent of 0.3093 gram of morphine sulphate, a loss of 0.0362 gram.

C. From putrid liver (after standing ten days in the open air and in a warm place) charged, when fresh, with 0.442 gram of morphine sulphate, 0.111 gram of anhydrous alkaloid were

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[1] Read at the Brooklyn Meeting of the American Association for the Advancement of Science, August, 1894.