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Fumeless Digestion of Nitrogen.

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ESTIMATION of nitrogen is perhaps the most important item of procedure in modern chemical analysis, over a million determinations being annually carried out by those engaged in scientific research alone. The estimations are usually carried out according to one or the other of the several modifications of the Kjeldahl method,[1] and involve prolonged digestion with concentrated sulphuric acid—an operation which is attended by emission of objectionable acid fumes. Some recent workers[2,3] have suggested that the residue after wet combustion of carbon can be distilled as such for nitrogen, but such a procedure, especially in the case of soils and other biological materials, leads invariably to retention of nitrogen in the digest, and consequently, low and inconsistent estimates being obtained.

A systematic enquiry into the various factors relating to the digestion has shown the following:—(*a*) the conversion of organic nitrogen into ammonia proceeds more rapidly in presence of small amounts of water combined with an oxidising agent (preferably chromic acid) than with concentrated sulphuric acid alone. In the case of soils, a mixture of sulphuric acid and water in the proportion of 2 to 1 yields the best results, the entire digestion being complete in 30 mins. During digestion, the proportion of acid to water has to be maintained constant, so it would be necessary to fit the digesting flask with an air- or water-cooled condenser. (*b*) The digesting mixture requires only a low flame and does not bump, so the long-necked (Kjeldahl) flask generally used for the purpose can be dispensed with. In fact, both the digestion and the distillation can be conducted in the same flask. (*c*) The minute quantities of nitrogen still retained in the digest can be easily released by addition of small amounts of zinc just prior to distillation with alkali. Metallic aluminium or Devarda's alloy can also be used for the purpose, but their action is a little too vigorous, and causes alkali spray to pass over into the distillate. (*d*) In the case of materials containing chlorides, it would be necessary to add a small quantity of mercuric or silver sulphate to the digesting mixture, for, otherwise, free chlorine will be formed and nitrogen will be lost in the elementary form. If the substance (*e.g.*, soil) contains nitrate, the latter should first be extracted with water preferably in presence of a suitable flocculant such as calcium sulphate. The residue is digested in the usual way and the digest, together with the extract containing nitrate, distilled with zinc and alkali, in the manner outlined above.

A simple method embodying the above principles has been developed and applied successfully to the estimation of nitrogen in soils. The procedure is also being extended to other biological materials and to nitrogenous substances in general. Attempts are also being made to combine the above method with that for the estimation of carbon[4] so that both the determinations can be carried out on the same sample.

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[1] Kjeldahl, *Z. anal. Chem.*, 1883, **22**, 366.

[2] Anderson and Schutte, *J. Biol. Chem.*, 1924, **61**, 57.

[3] Brown, *Ind. Eng. Chem.*, 1927, **19**, 629.

[4] Subrahmanyan, Narayanayya and Bhagvat, *J. Indian Inst. Sci.*, 1934, **17A**, 197.