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Oxidative Digestion of Organic Nitrogen.

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Oxidative Digestion of Organic Nitrogen.

It was shown in a previous communication[1] that nitrogen in soils and biological materials can be estimated by a process of oxidative digestion. The present note relates to (*a*) the nature of the products formed under such conditions and (*b*) an improved procedure for their inclusion in the estimate of total nitrogen.

The observations may be summarised as follows :—(1) When an aqueous suspension (or solution) of soil or other biological material is heated slowly with dichromate (or chromic anhydride) and sulphuric acid as in the usual procedure for the wet combustion of carbon, there is invariably some loss of nitrogen, the extent of such loss being inversely related to the rate of heating. The mechanism of this loss is still not clear though there is some evidence to show that ammonium dichromate, which is formed as an intermediate product, may undergo slight decomposition, yielding elemental nitrogen. The loss can be avoided, however, by first heating the material to be digested with water and sulphuric acid and then adding the oxidising agent to the boiling (or nearly boiling) mixture. (2) Small quantities of nitric acid are formed during the digestion. This is supported by the independent observations of Narasimhacharya.[2] The quantities are ordinarily too small to appreciably affect the estimate of total nitrogen even if the digest is boiled without condensing the ensuing vapours. On the other hand, the original material itself may contain useful amounts of nitrate which may be lost if proper precautions are not taken. The use of water or air-cooled condenser, as suggested by Narayanayya and Subrahmanyan (*loc. cit.*) helps to retain all the nitrogen in this form. Subsequent reduction of the digest with zinc or reduced iron in acid medium or Devarda's alloy in alkaline medium helps to include all the nitrate in the estimate of total nitrogen. More recently evidence has been adduced to show that even added nitrates can be retained in the digest and accurately estimated by adopting the above procedure. (3) It has already been shown (Narayanayya and Subrahmanyan, *loc. cit.*) that a part of the nitrogen is always retained in the digest and is not released by mere distillation with alkali. Further evidence has been obtained to show that the major part of this nitrogen is present in combination with the chromium in solution and is released on treating the digest with such reagents as are usually employed for reducing dichromate. The nature of the related compound (or compounds) is still obscure. (4) No information is available regarding the mode of association between nitrogen and the insoluble precipitate formed—especially in the case of soil—during digestion. It may nevertheless be mentioned that any nitrogen which may be present in that form is also included in the estimate of total nitrogen by treatment with reducing agents followed by distillation with alkali.

It was suggested by Narayanayya and Subrahmanyan (*loc. cit.*) that the total nitrogen in the digest can be estimated by boiling with zinc followed by distillation with excess of alkali. Unfortunately, all preparations of zinc (including some of the purest specimens) contain nitrogen. In some cases, the nitrogen content may be as high as that of the material to be digested. Reduced iron also contains nitrogen, though the latter is partly removed by pre-treatment with alkali. Since the major part of the zinc or iron is required for the reduction of unused chromic acid, it was considered desirable to use other reducing agents for that purpose. Among the various re-agents that were tried, alkali sulphites were found to be the most effective.

Based on the foregoing and other observations, an improved method of oxidative digestion has been developed and will be described elsewhere.

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[1] Narayanayya and Subrahmanyan, *Curr. Sci.*, 1935, **3.**

[2] Narasimhacharya, *J.S.C.I.*, 1935, **54,** 596 (Abstract).