31 Nitrogen—Total

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31-1 INTRODUCTION

The determination of total N in soils and other complex heterogeneous materials containing several forms of N presents many difficulties. Total N analysis of soils is further complicated by the inadequacy of knowledge concerning the forms of N present and by the low N content of the material under analysis. The total N content of soils ranges from <0.02% in subsoils to >2.5% in peats; the surface layer of most cultivated soils contains between 0.06 and 0.5% N.

Two methods have gained general acceptance for determination of total N: the Kjeldahl (1883) method, which is essentially a wet oxidation procedure, and the Dumas (1831) method, which is fundamentally a dry oxidation (i.e., combustion) technique.

In the Kjeldahl method, organic N in the sample under analysis is converted to NH₄⁺N by digestion with concentrated H₂SO₄ containing substances that promote this conversion, and the NH₄⁺N is determined from the amount of NH₃ liberated by distillation of the digest with alkali. In the original Kjeldahl procedure, H₂SO₄ alone was employed for digestion, KMnO₄ being used to complete the oxidation of organic matter. However, numerous investigations have shown that both the speed and the completeness of conversion of organic N to NH₄⁺N by digestion with H₂SO₄ can be increased by adding salts to raise the temperature of digestion or by adding catalysts to promote oxidation of organic matter. In practically all Kjeldahl methods now employed, K₂SO₄ or Na₂SO₄ is used to raise the temperature of digestion, and catalysts such as Se, Hg, or Cu are used to promote oxidation of organic matter.

In the classical Dumas method of determining N, the sample is heated with CuO at a high temperature (usually above 600°C) in a stream of purified CO₂, and the gases liberated are led over hot Cu to reduce nitrogen oxides (mainly N₂O) to N₂, and then over CuO to convert CO to CO₂. The N₂-CO₂ mixture thus obtained is collected in a nitrometer containing...