144. A MICRO-KJELDAHL APPARATUS

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THE apparatus described below is suitable for the estimation of amounts of N as small as $50\mu\text{g}$. In order to avoid any great increase in the volume of the reacting solutions during the steam distillation the usual apparatus was modified as follows.

The distillation apparatus (see Fig. 1) consists of two communicating bulbs. The upper one (*A*) is fitted with a trap which is continuous with the distillation tube, which in turn is surrounded by a condenser. The lower bulb (*B*) has a

Fig. 1.

slightly widened bent tube (*C*) on one side to receive one of the solutions, and a small tube (*D*) on the other side to serve as a connexion with the vapour source. To assure continuous regular boiling a vapour trap (*E*) is inserted. The whole apparatus, including the condenser, is made in one piece of pyrex glass and can be tilted round a horizontal axis perpendicular to the plane *A, B, C, D*.

The incineration of the substance, containing some $50\text{--}150\mu\text{g}$ N, is carried out by means of $0.1\text{ ml. H}_2\text{SO}_4$, with CuSO_4 as a catalyst and finally 1 drop of H_2O_2 . The colourless clear liquid is transferred into the widened part (*C*) of the bent tube of the lower bulb by means of a capillary pipette and the tube is then closed with a glass stopper (*F*) inserted into a rubber tube. Into the lower bulb 1 ml. 33% KOH has been introduced previously through (*D*) with a drop of liquid paraffin to avoid foaming. The lower bulb is then connected to a steam generator by rubber tubing. When bubbles begin to pass through the KOH solution the apparatus is tilted so that the solution of incinerated substances runs into the bulb (*B*). During the distillation the bulb (*C*) should be cooled by the external application of a wad of cotton-wool soaked in cold water. When a small amount of water has condensed in (*C*) the apparatus is again tilted so that this water runs back into *B*; this washes the remains of the solution out of (*C*). It is advisable to heat the upper part of the apparatus with a free flame in order to prevent condensation. The distillation should be continued for 6 min., the last 3 min. serving

to wash out the tube. The distillate is collected in a known amount of $N/100$ HCl and after adjustment to a definite volume, e.g. 6 ml., the excess of HCl is determined iodometrically in the usual manner. The accuracy of the method is $\pm 3\%$, using 0.2–0.3 mg. of substances containing about 10–30% N.

Example: 1.24 mg. cytosine were dissolved in 1 ml. water. 0.192 ml. of the solution containing 0.238 mg. cytosine was incinerated and the N content estimated.

N content calculated: $89\mu\text{g.} = 37.6\%$. Found: (1) $92.5\mu\text{g.} = 38.8\%$; (2) $88.2\mu\text{g.} = 37.0\%$.

I wish to express my indebtedness to Mr Frank Goulden for constructing the first model of this apparatus.