COEI-2-AZOTOT Determination of total nitrogen

1. Apparatus

1.1. The apparatus used for separating NH_3 is either a distillation apparatus with a rectifying column or a distillation apparatus under a current of steam (diagram) made up of:

- A 1 l flask **A** of borosilicate glass used as a boiler with a stopcock funnel for filling. It can be heated by a gas or electric furnace.
- An adapter **C** which gathers the spent liquid from the bubbler **B**.
- A bubbler **B** of 500 ml with an inclined neck; the supply tube must reach the lowest part of the flask. The out-going tube has an anti-entrainment ball that makes up the top part of the bubbler. A stop-cock funnel **E** allows to introduce the liquid to be treated and alkaline lye.
- A cooler 30 to 40 cm long, vertical, with a ball with fine dowel bush on the tip.
- A 250 ml conical flask for the distillate.

2. Mineralisation flask, 300 ml ovoid-shaped flask with a long neck.

2. Reagents

- Concentrated sulphuric acid (R).
- Mineralisation catalyser (R).
- Sodium hydroxide solution at 30% (m/m) (R).
- Boric acid solution at 4% (R).
- Hydrochloric acid solution 0.1 M.
- Mixed-based indicator with methyl red (R) and methylene blue.
- The boiler must contain acidulated water by 1 per 1 000 of sulphuric acid. It is advisable to boil this liquid before any operation, with the drain cock P open to let the CO2 escape.

3. Procedure

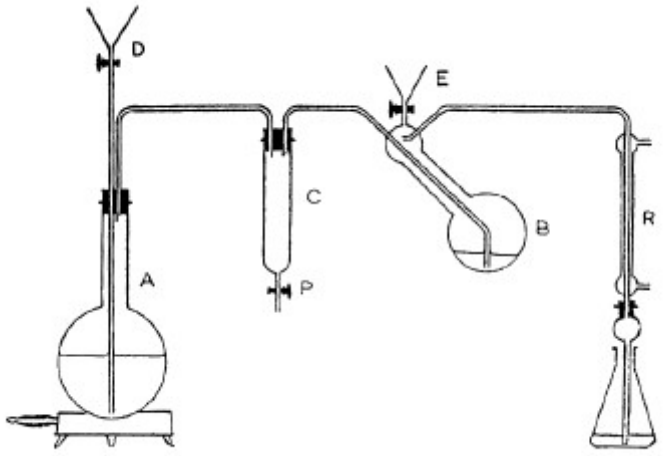
In the mineralisation flask, introduce the test sample containing 4 to 50 mg of nitrogen. Add 5 g of mineralisation catalyser (R) and 10 ml of concentrated sulphuric acid (R), if the quantity of dry organic matter to be mineralised is below 500 mg. Increase these quantities if a higher quantity of organic matter must be used.

Heat in an open flame under a hood. The neck of the flask is maintained inclined until the solution becomes colourless and the walls of the flask are clear of carbonised products.

After cooling, dilute with 50 ml of water and cool; introduce this liquid in the bubbler **B** with the funnel **E**, then add 40 to 50 ml of sodium hydroxide solution at 30% (R) in order to obtain frank alkalinisation of the liquid. Entrain the ammoniac with the vapour by gathering the distillate in 5 ml of boric acid solution (R) placed beforehand in a receiving conical flask with 10 ml of water, with the tip of the ampoule plunged into the liquid. Add 1 or 2 drops of mixed-based indicator and gather 70 to 100 ml of distillate.

Titrate the distillate with the hydrochloric acid solution 0.1 M until the indicator turns pink violet.

1 ml of 0.1 M hydrochloric acid solution corresponds to 1.4 mg of nitrogen.



Apparatus for the distillation of ammoniac
in a current of steam (PARNAS and WAGNER)
The cocks P and E can be replaced by a plastic
pipe fitting with a Mohr pinch-clamp cock.