

SCHEDULE 2

METHODS OF ANALYSIS

3a.

DETERMINATION OF NITRIC AND AMMONIACAL NITROGEN — ULSCH METHOD

Determination

7.2 Place in the receiving flask an exactly measured quantity of standard sulphuric acid (4.2) as indicated in the Table of Method 2 (variant (a)) and add the appropriate quantity of indicator solution (4.7.1 or 4.7.2).

The end of the extension tube of the condenser must be below, the surface of the standard acid in the receiving flask.

Using a pipette, transfer an aliquot part of the clear solution as indicated in the Table of Method 2 (variant (a)) and place it in the distilling flask of the apparatus. Add 350 ml water, 20 ml 30% sulphuric acid solution (4.4), stir, and add 5 g of reduced iron (4.5). Wash the neck of the flask with several ml water, and place in the neck of the flask a small, long stemmed funnel. Heat in a boiling water bath for an hour and then wash the stem of the funnel with a few ml water.

Taking care to avoid any loss of ammonia, add to the contents of the distilling flask 50 ml concentrated sodium hydroxide solution (4.6), or in the cases where 20 ml hydrochloric acid (4.1) has been used to dissolve the sample, add 60 ml of concentrated sodium hydroxide solution (4.6). Assemble the distillation apparatus. Distil the ammonia according to the procedure given in Method 2. Titrate the excess acid with the standard solution of sodium or potassium hydroxide (4.3).