OFFICIAL, STANDARDISED AND RECOMMENDED METHODS OF ANALYSIS

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INTRODUCTION

When organic matter is to be destroyed as a preliminary to the determination of metallic traces, the choice of method will depend (*a*) on the nature of the organic material and of any inorganic constituent, and (*b*) on the metal that is subsequently to be determined and the method to be used for its determination.

A number of recommended methods of decomposition, both wet and dry are described, with a statement of the type or types of organic material to which each may be applied. Any discrimination on account of the metal to be determined is considered here only in rather general terms, except that special methods are necessarily given for mercury.

The influence of the metal and its method of determination on the choice of decomposition procedure are described more specifically in the section describing the determination of each metal, and each method will refer to this section for methods of decomposition.

WET DECOMPOSITION

The wet decomposition of organic matter by the action of various acids is of almost universal application, and the conditions can be adjusted so as to prevent loss of the more volatile elements, *e.g.*, arsenic, antimony and mercury. Some guidance on the choice of method is given under 'Applicability' and 'Disadvantages' at the beginning of the description of each method, but it must be emphasised that unfamiliar materials must always undergo a trial treatment on a small scale before the method to be used is selected. Only in this way can untoward incidents be avoided.

The amounts of acid specified will generally be satisfactory, but workers will learn by experience what are the optimum amounts to use for any particular material.

APPARATUS

When organic matter is heated with a mixture of concentrated acids in a Kjeldahl flask, experience has shown that decomposition takes place most efficiently when some means for partial reflux of the boiling mixture is provided, as by an extension to the neck of the flask (see Fig. 1). When many oxidations are in progress at the same time, it is advisable to trap the fumes, dilute them with water and dispose of them down the drains rather than into the atmosphere. The following description of suitable apparatus is given for guidance only.

Kjeldahl flasks (Fig. 1). These should be made of borosilicate glass or silica (100- to 250-ml nominal capacity) fitted with an extension to the neck by means of a standard ground joint. The extension serves to condense fumes into an acid-fume condenser, and carries a tap funnel through which the reagents are introduced.

Each flask should be supported in a circular hole in an asbestos sheet, and the hole should be of diameter such that the flask receives no direct heat from the burner above the level of the acid. Gas heating is preferable.

Kjeldahl digestion rack and acid-fume condenser (Fig. 2). A current of water is kept flowing through the condenser, and the removal of acid fumes can also be assisted by connecting the upper outlet to a water pump. The flasks, not being rigidly clamped, are easily handled by means

*Taken from reports of the Metallic Impurities in Organic Matter Sub-Committee to the Analytical Methods Committee (*Analyst*, 1960, **85**, 643; 1965, **90**, 515).

of tongs or asbestos finger-guards when it is necessary to deal with vigorous reactions or excessive frothing.

It is a convenience when handling flasks and test-tubes containing hot concentrated acids to have at hand a pair of the nickel tongs made for this purpose. The action of these tongs is improved by binding each jaw with asbestos string.

All glass and silica apparatus must be thoroughly cleaned with concentrated sulphuric and nitric acids and then thoroughly washed with distilled water immediately before use to ensure that it does not yield traces of metals under the conditions of test.

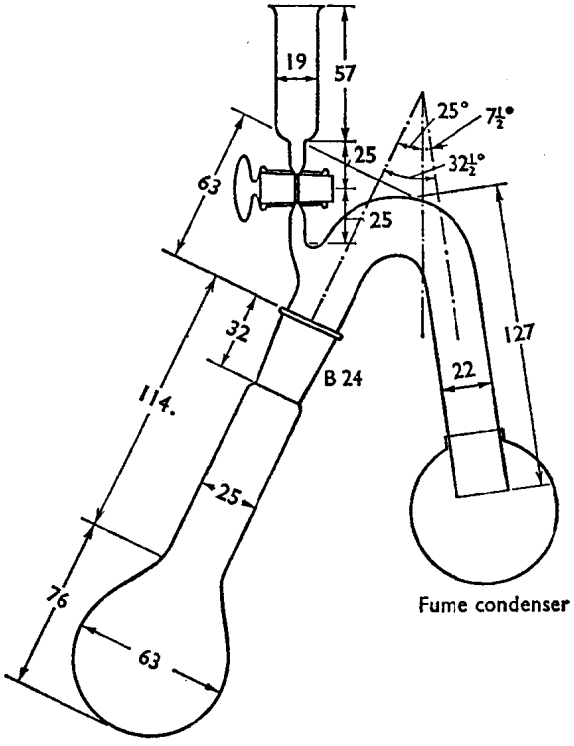

Fig. 1. Modified Kjeldahl flask (open type).
Dimensions, in mm, are for a flask of 150-ml capacity

REAGENTS

It is essential to use reagents and water of suitably low metal content, taking into consideration that the concentrated mineral acids are generally used in amounts several times that of the sample. Some of the more commonly used reagents are now available in grades containing very low concentrations of metallic impurities and are labelled as suitable for foodstuffs analysis. They should not be transferred from the lead-free-glass bottles in which they are supplied.

REAGENT BLANKS

Even when these low-metal reagents are used, reagent-blank determinations will be necessary. Separate instructions are not given for reagent blanks, since the procedures to be followed are similar to those for the determination proper with obvious modifications, but since the blanks must be prepared with the same quantities of reagents as are used in the tests, the measurement and recording of these quantities must not be overlooked.