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The committee responsible for this document is Technical Committee UNBS/TC 2, Food and agriculture, Subcommittee SC 20, Agrochemicals and veterinary drugs.

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AFRICAN STANDARD

Agricultural liming materials — Specification



Reference No. ARS 1492:2020(E) ICS 65.080

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Introduction

At a global scale, soils are increasingly being degraded and becoming marginal for agricultural production driven by e.g. salinization, erosion and acidification (FAO, 2015). Soil acidification is one of the most prevalent problems in production of food and fibre because at least 40%, and by some estimates as much as 70%, of the world's arable land is affected (Rengel, 2003). With increased pressure to produce more food for the expanding population of this planet and with urbanization claiming large chunks of arable land, agriculture is pushed more and more into marginal land plagued with edaphic and other stresses, including acidity. The major processes leading to soil acidification include (1) net H^+ excretion by plant roots due to excess uptake of cations over anions; (2) removal of alkalinity in farm biomass products such as grain, hay, meat, and wool which increases as yields increase; (3) accumulation of organic anions in the form of soil organic matter; (4) mineralization of organic matter, nitrification of ammonium, and subsequent leaching of nitrate, and (5) input of acidifying substances such as NH_4^+ -based fertilizers and urea, elemental S fertilizer and the growth of legumes (Rengel, 2003; Goulding, 2016; Holland *et al.*, 2019).

Crop growth is often limited in tropical acid soils through a number of factors, including low pH per se, toxicities of aluminium (AI) and manganese (Mn), and deficiencies of phosphorus (P), calcium (Ca), magnesium (Mg) or molybdenum (Mo) (Blamey et al., 1989). At 8.1 %, aluminium is the most abundant metal and the third most common element in the earth's crust after oxygen (46.6 %) and silicon (27.7 %). At mildly acidic or neutral soil pH values, AI is primarily in the form of insoluble aluminosilicates or oxides. The principal adverse effects of acidity on soil fertility occur at soil pH values below 5.5 due to acid dissolution of aluminium (AI) and the onset of AI phytotoxicity that affect root (and plant) growth (Wong et al., 2003). Thus, Al toxicity is the major growth-limiting factor for crop cultivation on acid soils. The initial and most dramatic symptom of AI toxicity is inhibition of root elongation, which can occur within 1-2 hrs after exposure to AI. This results in reduced volume of soil explored by the root system and direct interference with uptake of ions such as calcium and phosphate across the cell membrane of damaged roots (Kochian, 1995). These phytotoxic effects are unimportant in moderately acidic soils with pH values of 5.5 to 6.5 when the concentration of toxic forms of AI is normally negligible. Manganese toxicity and deficiencies of phosphorus, calcium, and magnesium are common in acidic soils. Soil nutrient deficiencies exacerbate the problem of inefficient nutrient uptake due to restricted root growth and root damage (Sumner *et al.* 1991). In drier environments, poor water use due to poor root development is considered to be another adverse effect of AI phytotoxicity.

Liming is a common agricultural practice worldwide, used for increasing productivity in acid agricultural soils (Bolan *et al.*, 2003; Fageria *et al.*, 2011; Paradelo *et al.*, 2015). Lime requirements of crops grown on acid soils are determined by the quality of liming material, status of soil fertility, crop species and cultivar within species, crop management practices, and economic considerations. Soil pH, base saturation, and aluminium saturation are important acidity indices that are used to determine liming. Liming improves soil pH, Ca, and Mg contents and reduces AI concentrations in the soil solution. In addition, liming improves beneficial microbe populations in the soil. Furthermore, liming improves P concentration in the soil solution by reducing P immobilization by Fe and AI in acid soils (Fageria *et al.*, 2008). All these beneficial effects of liming improve the root growth of crop plants. Liming has also been shown to increase the soil organic carbon (SOC) with resultant increase in crop yields (Paradelo et al., 2015) while at the same time immobilizing cadmium in the soil, thus reducing its uptake by crops and improving the safety and quality of food crops (Shi *et al.*, 2019). Liming enhances the physical, chemical and biological properties of soil through its direct effect on the amelioration of soil acidity and its indirect effect on the mobilization of plant nutrients, immobilization of toxic heavy metals, and improvements in soil structure and hydraulic conductivity (K_s) (Bolan *et al.*, 2003).

A range of liming materials are available, which vary in their ability to neutralize the acidity. These include calcitic limestone (CaCO₃), burnt lime (quicklime) (CaO), slaked/hydrated lime (Ca(OH)₂), dolomitic limestone (CaMg(CO₃)₂), slag (CaSiO₃), marl/ marlstone, shells, magnesian limestone, marine limestone, chalk and other materials (Bolan *et al.*, 2003). The amount of liming material required to rectify soil acidity depends on the neutralizing value of the liming material and pH buffering capacity of the soil.

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Agricultural liming materials — Specification

1 Scope

This African Standard specifies the requirements and methods of sampling and tests for agricultural liming materials.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 12048, Solid fertilizers and liming materials — Determination of moisture content — Gravimetric method by drying at 105 \pm 2 °C

ISO 7409, Fertilizers — Marking — Presentation and declarations

ISO 8157, Fertilizers and soil conditioners — Vocabulary

ISO 8397, Solid fertilizers and soil conditioners — Test sieving

ISO 11047, Soil quality — Determination of cadmium, chromium, cobalt, copper, lead, manganese, nickel and zinc — Flame and electrothermal atomic absorption spectrometric methods

ISO 11465, Soil quality — Determination of dry matter and water content on a mass basis — Gravimetric method

ISO 14820-1, Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling

ISO 14820-2, Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation

ISO 17318, Fertilizers and soil conditioners — Determination of arsenic, cadmium, chromium, lead and mercury contents

3 Terms and definitions

For the purpose of this standard, the terms and definitions in ISO 8157 and the following apply.

3.1

agricultural liming material

a product whose calcium and magnesium compounds are capable of neutralizing soil acidity

3.2

blast-furnace slag

the non-metallic product, consisting essentially of silicates and aluminosilicates of calcium and other bases, that is developed in a molten condition simultaneously with iron in a blast furnace

3.3

blast-furnace slag, air-cooled

the material resulting from solidification of molten blast-furnace slag under atmospheric conditions; subsequent cooling may be accelerated by application of water to the solidified surface

3.4

blast-furnace slag, granulated

the glassy, granular material formed when molten blast-furnace slag is rapidly chilled, as by immersion in water

3.5

burnt lime (quick lime)

a material, made from limestone which consists essentially of calcium oxide or combination of calcium oxide with magnesium oxide

3.6

calcium carbonate equivalent (C.C.E.)

the acid neutralizing capacity (of an agricultural liming material) of the material expressed as weight percent of calcium carbonate

3.7

fineness

the percentage by weight of the material which will pass sieves of specified sizes

3.8

hydrated lime

a material, made from lime, which consists essentially of calcium hydroxide or a combination of calcium hydroxide with magnesium oxide and/or magnesium hydroxide

3.9

limestone

a material consisting essentially of calcium carbonate or a combination of calcium carbonate with magnesium carbonate capable of neutralizing soil acidity

4 Requirements

4.1 Chemical composition

Agricultural liming materials shall be classified in terms of calcium carbonate equivalent (C.C.E) as shown in Table 1.

| No. | Material | minimum Calcium carbonate equivalent (CCE), % | Test method |
|------|---------------|--|-------------|
| i) | Quicklime | 140 | Annex A |
| ii) | Hydrated lime | 110 | |
| iii) | Limestone | 80 | |
| iv) | Slag | 80 | |
| v) | Shells | 80 | |

Table 1 — Agricultural liming materials requirements

NOTE Marl and some by-product liming materials are used for neutralizing soil acidity, but due to their varying composition, their chemical limits are not included. In some economic circumstances limestone, slag, and shells of less than 80 % C.C.E. may be used.

4.2 Requirements for agricultural limestone

4.2.1 Sieve analysis classifications for agricultural limestone

The sieve analysis shall be done in accordance with ISO 8397. Agricultural limestone shall be classified according to the minimum percentage passing the no. 8(2.36-mm) and no. 60(0.25mm) as shown in Table 2.

| SI/no | Class | Passing no. 8(2.36mm) sieve, | Passing no. 60(0.25mm) |
|-------|-------------|------------------------------|------------------------|
| | designation | min per cent | sieve, min, per cent |
| i | S | 100 | 100 |
| ii | Т | 99 | 75 |
| iii | 0 | 95 | 55 |
| iv | N | 90 | 40 |
| V | E | 80 | 25 |

Table 2 — Sieve analysis classification for agricultural limestone

NOTE 1 These classifications apply where the agricultural limestone is obtained by the normal crushing procedure and the product contains the fines of fracture. In some economic circumstances, coarser products are used. The No. 60 (0.25mm) sieve was selected because research has shown that this sieve gives a more accurate representation of the particle size distribution of most agricultural limestones presently produced than a finer or coarser sieve. The No. 8 (2.36-mm) sieve is used to control the upper limit on the amount of coarse limestone particles that may be in the product.

NOTE 2 The relationship between increasing limestone fineness and reactivity is caused by having a larger surface area exposed to chemical reaction. The surface area of liming material increases proportionately with decreasing particle size, for example, the exposed area of a cube of 1 cm is 6 cm²: decreasing the particle size to 2 mm (10 mesh), or 0.05 mm (300 mesh) increases the exposed area to 30 and 1200 cm², respectively.

4.2.2 Neutralizing requirements for calcitic limestone

Calcite shall comply with the neutralizing requirement in Table 3

| | Characteristics | Grade I | Grade II | Method of test | | |
|------|---|---------|----------|----------------|--|--|
| i) | Calcium Carbonate Equivalent (CCE), %, min | 70 | 50 | Annex A | | |
| ii) | Active lime (total available as CaCO3 + MgCO ₃), %, min | 50 | 40 | Annex C | | |
| iii) | Magnesium, (as MgO) %, max | 10 | 10 | Annex D | | |
| iv) | Moisture content, %, max | 10 | 12 | Annex B | | |

Table 3 — Requirements for calcitic limestone

NOTE Another factor which influences the efficiency of a limestone is its neutralizing capacity. This is determined by reacting limestone with strong acids, either hydrochloric or sulphuric acid. The limestone's neutralizing capacity is expressed as a percentage value of pure calcium carbonate, which has a neutralizing value of 100%. Commercial limestones may have neutralizing capacities greater or lesser than pure CaCO3. Dolomitic limestone (a mixture of magnesium and calcium carbonates) has a theoretical neutralizing value of 108%. Generally, limestones have neutralizing values below 100%. Impurities such as clays in limestone reduce their relative neutralizing capacities.

To determine the agricultural value (A.V.) of limestones, the particle size efficiency rating (E.R.) has to be adjusted by the neutralizing value (N.V.) (synonymous with neutralizing capacity), plus an adjustment for limestone moisture content (M.C.). For example, a limestone having an efficiency rating (E.R.) of 85%, a neutralizing value (N.V.) of 95%, and a moisture content of (M.C.) 13%, has an agricultural value of:

AV = 85% (E.R.) X 0.95 (N.V.) X [1.00 – 0.13 M.C.]

AV = 70.25%

4.3 Sieve analysis classifications for agricultural slag

4.3.1 Air-cooled blast-furnace slag

Air-cooled blast-furnace slag shall be classified the same as agricultural limestone as in Clause 4.2.

4.3.2 Granulated blast-furnace slag

Granulated blast-furnace slag shall be classified in accordance with the minimum percentages passing the No. 8 (2.36-mm) and the No. 60 (0.25mm) sieves.

4.4 Requirement for hydrated lime and burnt lime

4.4.1 Fineness

Hydrated lime and quicklime for agricultural use shall be classified in accordance with the minimum percentages passing the No. 8 (2.36-mm) and No. 60 (0.25mm) sieves, as follows:

| Table 4 — Particle size requirement | nts for hydrated lime and quicklime |
|-------------------------------------|-------------------------------------|
|-------------------------------------|-------------------------------------|

| Passing sieve No. | Min. % | | Test method |
|-------------------|---------------|------------|-------------|
| | Hydrated lime | Quick lime | |
| 8(2.36 mm) | 100 | 95 | ISO 8397 |
| 60(0.25mm) | 97 | 35 | ISO 8397 |

4.4.2 Neutralizing requirements of hydrated lime and quicklime

Hydrated lime and burnt lime shall comply with the requirement in Table 5.

Table 5 — Requirements for hydrated lime and burnt lime as soil conditioners

| S/N | Characteristics | Grade I | Grade II | Method of test |
|------|---|---------|----------|----------------|
| i) | Calcium Carbonate Equivalent (CCE), %, min | 85 | 70 | Annex A |
| ii) | Active lime (available as , Ca(HO) ₂ , %, m/m, min | 60 | 50 | Annex C |
| iii) | Moisture content, %, m/m, max | 10 | 12 | Annex B |

4.4.3 Neutralizing requirements for dolomite

Dolomite shall comply with the requirements in Table 6.

Table 6 — Requirements for dolomitic limestone

| S/N | Characteristics | Grade I | Grade II | Method of test |
|------|--|---------|----------|----------------|
| i) | Calcium Carbonate Equivalent (CCE), %, min | 100 | 70 | Annex A |
| ii) | Total magnesia lime (MgCO ₃), %, min | 35 | 10 | To be provided |
| iii) | Moisture content, %, max | 10 | 12 | Annex B |

4.5 Heavy metal contaminants

The heavy metal contaminants, if present, shall not exceed the limits stipulated in Table 7.

| S/N | Properties | Maximum limits (mg/kg, dry weight) | Test method |
|-------|---------------|---------------------------------------|----------------------|
| i) | Arsenic (As) | 10 | ISO 17318 |
| ii) | Zinc (Zn) | 5 | ISO 11047 |
| iii) | Lead (Pb) | 30 | ISO 17318/ISO 11047 |
| iv) | Copper (Cu) | 300 | ISO 11047 |
| V) | Chromium (Cr) | 150 | ISO 17318/ ISO 11047 |
| vi) | Nickel (Ni) | 50 | ISO 11047 |
| vii) | Mercury (Hg) | 2 | ISO 17318 |
| viii) | Cadmium (Cd) | 5 | ISO 17318/ ISO 11047 |

Table 7— Limits for heavy metals contaminants for agricultural liming materials

5 Sampling

Sampling and sample preparation for inspection and testing shall be carried out in accordance with ISO 14820 parts 1 and 2.

6 Compliance

The lot shall be deemed to comply with the standard if after inspection and testing it complies with the requirements of this standard.

7 Packaging and labelling

7.1 Packaging

The agricultural liming materials shall be packed in clean, non-defective and strong packages. The material for which the package is made shall be such as to protect the contents from moisture and also not lead to easy rupture during handling, transportation and storage.

7.2 Labelling

7.2.1 Each package of the agricultural liming material shall be legibly and indelibly marked with the following particulars:

- a) Name of product as agricultural liming materials,
- b) **Types** (such as Calcite, Dolomite, Hydrated lime or Burnt lime.)
- c) Grade: such as Grade I or Grade II
- d) Elemental calcium and magnesium percentage by weight
- e) Calcium Carbonate Equivalent (CCE)
- f) net weight
- g) Effective Neutralizing Value (ENV)
- h) Lot number or batch number
- i) Name and physical address of the manufacturer
- j) Instructions for use and handing

k) Storage conditions

- I) date of manufacture and best before or expiry date
- m) Country of origin

7.2.2 Bulk containers

Where the agricultural liming materials is distributed in bulk, the marking information shall accompany the delivery note to the purchaser.

8 Material safety

Each Consignment shall be accompanied by a Material Safety Data Sheet (MSDS) and a Certificate of Analysis (COA).

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Annex A

(normative)

Determination of calcium carbonate equivalent (neutralizing value)

A.1 Coverage

The calcium carbonate equivalence (CCE) test is used to determine the neutralizing capability of a calcareous material and to report this value in terms of percent calcium carbonate equivalents (% CaCO₃).

A.2 Significance and use

Calcareous materials such as crushed limestone, hydrated lime and pulverized slags (from the production of steel) have been used extensively as soil modifiers or agricultural liming materials. A measure of their neutralizing capability can be determined through the use of this method of test. Not all neutralizing components of a calcareous material may be beneficial, therefore, the chemical analysis is suggested. ENDRA

A.3 **Apparatus**

- A.3.1 pH Metre.
- A.3.2 Mechanical Stirrer.
- A.3.3 Sieve, No. 60 (250-µm).
- A.2 Reagents
- A.2.1 Standard hydrochloric acid - 0.5 N.
- A.2.2 Standard Sodium Hydroxide Solution - 0.5 N.

Phenolphthalein Indicator Solution — Dissolve 0.1 g of phenolphthalein in 60 ml of rectified A.2.3 spirit and dilute with water to 100 ml.

A.3 **Procedure**

Weigh accurately about 0.5 g of the previously ground sample to pass through 250µm sieve, in A.3.1 a 250-ml stoppered conical flask. Add 40 ml of standard hydrochloric acid, with swirling. Heat to gentle boiling, agitating continuously. Boil for 5 minutes and then cool to room temperature. Titrate against standard sodium hydroxide solution using 2 to 3 drops of phenolphthalein indicator.

A.3.1.1 Carry out a blank test using the same quantities of all reagents but without adding the sample.

A.3.2 Calculation

Neutralizing value (as CaCO₃), percent by mass = $\frac{5(B-A) \times N}{M}$

where

- B = volume in ml of standard sodium hydroxide solution used in the blank determination,
- A = volume in ml of standard sodium hydroxide solution used with the sample.
- N = normality of standard sodium hydroxide solution, and
- M = mass in g of the sample taken for the test.

Annex B

(normative)

Determination of moisture content

B.1 Procedure

Weigh accurately 2 g of the powdered sample in a platinum or silica dish. Place It in an Oven maintained at $105^{\circ} \pm 2^{\circ}$ C, until on cooling in a desiccator and weighing, constant mass (± 2 mg) is obtained. Calculate the percent moisture m the sample.

B.2 Calculation

Moisture, percent by mass = $\frac{100(M_2 - M_2)}{M_2 - M}$

where

 M_1 = mass III g of the moisture dish with the material before drying,

 M_2 = mass in g of the moisture dish with the material after drying, and

M = mass in g of the empty moisture dish.

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Annex C

(normative)

Determination of available lime index

C.1 Scope

The available lime index of high-calcium quicklime and hydrated lime designates those constituents that enter into the reaction under the conditions of this specified test method, otherwise known as the "rapid sugar test method." The interpretation of results obtained by this test method shall be restricted by this definition.

C.2 Summary of Test Method

The sample is slaked and dispersed with water. The lime is solubilized by reaction with sugar to form calcium sucrate which is then determined by titration against standard acid using phenolphthalein as the indicator.

C.3 Reagents

C.3.1 Hydrochloric Acid, Standard (1.000 N) — Prepare a solution by diluting 83 mL of HCl to 1 L with CO_2 -free water.

C.3.2 Phenolphthalein Indicator (4 %) — Dissolve 4 g of dry phenolphthalein in 100 mL of 95 % alcohol.

C.3.3 Sucrose Solution — (40 g of pure cane sugar in solid form may be used per sample in place of a sugar solution.) Prepare a 40 % solution (w/v) using pure cane sugar and CO_2 -free water in a large beaker and stir until dissolved. Add several drops of phenolphthalein indicator solution. Add 0.1 N NaOH solution dropwise with stirring until a faint pink colour persists. Stock solution of sugar may be made for convenience; however, it should not be stored for more than two days. As an alternative the acidity of each lot of sugar can be determined, and a correction applied to the titration.

C.4 Procedure

C.4.1 Procedure for quicklime

C.4.1.1 The sample as received at the laboratory shall be thoroughly mixed and a representative sample with minimum weight of 100 g shall be taken and pulverized to pass a No. 50 sieve for analysis. Weigh rapidly 2.804 g of the finely pulverized sample, brush carefully into a 500-mL Erlenmeyer flask containing about 40 mL of CO_2 -free water, and immediately stopper the flask.

NOTE Water should not be added to the sample because, especially with quicklime, there is a tendency for the material to cake and form lumps difficult to completely dissolve in the sugar solution later. On the other hand, if the lime is added to a little water, a better dispersion of the fine particles occurs, leading to a more rapid dissolution of the sample. It is possible that in the case of quicklime, some slaking action occurs to facilitate the dispersion and solution.

C.4.1.2 Remove the stopper. Place the flask on a hot plate and immediately add 50 mL of boiling CO₂-free water to the flask. Swirl the flask and boil actively 1 min for complete slaking. Remove from the hot plate, stopper the flask loosely, and place in a cold-water bath to cool to room temperature.

C.4.1.3 Add 100 mL of the neutralized sugar solution (or approximately 40 g of pure cane sugar). Stopper the flask, swirl, and let stand for 15 min to react. (Reaction time should not be less than 10 min nor more than 20 min.) Swirl at 5-min intervals during reaction. Remove stopper, add 4 to 5 drops of 4 % phenolphthalein indicator solution and wash down the stopper and sides of the flask with CO₂-free water.

C.4.1.4 When titrating, first add about 90 % of the acid requirement from a 100-mL buret. Finish the titration, more carefully at approximately one drop per second, to the first disappearance of the pink colour, which persists for 3 s. Note the endpoint and ignore any further return of colour.

NOTE A mechanical stirrer may be used during the titration if desired. Put a clean magnetic stirrer bar into the flask and place the flask on the magnetic stirrer. Adjust to stir as rapidly as possible without incurring any loss by spattering. Unless the operator is familiar with previous analyses of the lime under test, and in cases where the available lime content varies to extremes, it is good practice to run a preliminary test by slow titration to determine the proper amount of acid required to neutralize the sample.

C.4.2 Procedure for hydrated lime

The procedure for determining $Ca(OH)_2$ is the same as for CaO with the exception that cold CO₂-free water is used and the boiling and cooling steps are omitted.

C.5 Calculation

C.5.1 Calculate for CaO as follows:

Available lime CaO, % =
$$\frac{N \times V \times 2.804}{W}$$

where:

- *N* = normality of acid solution,
- V = standard HCl used (1.000 N), mL,
- weight of sample, g, and 2.804 = CaO, g, equivalent to 1 mL of standard acid × 100 or 1 mL of standard HCl = 1 % CaO if exactly 2.804 g of sample is used.

W

C.5.2 Calculate for Ca(OH)₂ as follows:

Available lime [Ca(OH)₂ % =
$$\frac{N \times V \times 3.704}{N \times V \times 3.704}$$

where:

- V = standard HCI (1.000 N), mL,
- W = weight of sample, g, and
- 3.704 = Ca(OH)₂, g, equivalent to 1 mL of standard acid × 100 or 1 mL of standard HCl = 1.32 % Ca(OH)₂ when exactly 2.804 g of sample is used.

Annex D

(normative)

Determination of magnesium oxide content

D.1 Scope

Magnesium oxide in lime and limestone may vary from a few tenths to 2 % for high-calcium limestone to as much as 22 % for dolomitic limestone. The pyrophosphate gravimetric method has been used successfully throughout the industry to determine magnesium within this wide range.

D.2 Summary of test method

In this test method, magnesium is doubly precipitated as magnesium ammonium phosphate from the filtrate after removal of calcium. The precipitate is ignited and weighed as magnesium pyrophosphate $(Mg_2P_2O_7)$. The MgO equivalent is then calculated.

D.3 Reagents

D.3.1 Ammonium Phosphate, Dibasic Solution (250 g/L) — Dissolve 250 g of dibasic ammonium phosphate ($(NH_4)_2HPO_4$) in 1 L of water.

D.3.2 Ammonium Hydroxide Wash Solution (5 + 95) — Dilute 50 mL of NH₄OH with 950 mL of water and add 1 or 2 mL of HNO₃.

D.4 Procedure

D.4.1 Add 2 drops of methyl red indicator to the combined filtrates from the determination of calcium, acidify with HCl, and concentrate to about 250 mL. Add to this solution about 10 mL of the $(NH_4)_2HPO_4$ solution, 250 g/L, and cool the solution to room temperature. Add NH_4OH slowly while stirring constantly until the solution is alkaline or the crystalline magnesium ammonium phosphate begins to form; then add about 15 to 20 mL of NH_4OH in excess and continue stirring for several more minutes. Allow the beaker and precipitate to stand in a cool place overnight. Filter and wash with cold dilute ammonium hydroxide wash solution (5 + 95).

D.4.2 Dissolve the precipitate with hot diluted HCI (1 + 9) and wash the filter paper well with hot diluted HCI (1 + 99). Dilute the solution to 100 mL, cool to room temperature, and add 1 mL of the 20 % solution of (NH₄)2HPO₄. Precipitate the magnesium ammonium phosphate as before and allow to stand for about 2 h in a cool place.

D.4.3 Filter the precipitate on paper or in a tared Gooch crucible, washing with diluted NH₄OH (5 + 95). If filtered through a Gooch, place directly in a muffle at 400 °C and raise heat to 1100 °C. If filtration was through paper, place paper and precipitate in a weighed platinum or porcelain crucible. Slowly char the paper without inflaming and carefully burn off the resulting carbon (**Warning!** Extreme caution should be exercised during this ignition. Reduction of the phosphate precipitate can result if carbon is in contact with it at high temperatures. There is also a danger of occluding carbon in the precipitate if ignition is too rapid.). Ignite at 1100 °C for $\frac{1}{2}$ h, cool in desiccator, and weigh as Mg₂P₂O₇.

 $\label{eq:NOTE} \mbox{For research purposes or in the most exacting types of work, the manganese content of the pyrophosphate residue should be determined and deducted as $Mn_2P_2O_7$.}$

18.5 Calculation

Calculate the percentage of MgO to the nearest 0.01 % as follows:

MgO, % =
$$\frac{A \times 36.2}{B}$$

where:

| Α | = Mg ₂ P ₂ O ₇ , | g, |
|---|---|----|
| | | |

B = sample, g, and

36.2 = molecular ratio of 2MgO to $Mg_2P_2O_7 \times 100$.

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