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Edition 1

Any reference to SABS ISO 622 is deemed
to be a reference to this standard
(Government Notice No. 1373 of 8 November 2002)

SOUTH AFRICAN NATIONAL STANDARD

Solid mineral fuels — Determination of phosphorus content — Reduced molybdophosphate photometric method

This national standard is the identical implementation of ISO 622:1981 and is adopted with the permission of the International Organization for Standardization.

SANS 52:2006

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Table of changes

Change No.	Date	Scope
Nat. amdt 1	2006	Amended to change the designation from SABS to SANS, with no technical changes.

National foreword

This South African standard was approved by National Committee SABS SC 27B, *Solid mineral fuels – Test methods*, in accordance with procedures of the SABS Standards Division, in compliance with annex 3 of the WTO/TBT agreement.

This SANS edition is technically identical to ISO 622:1981.

**Reaffirmed and reprinted in April 2012.
This standard will be reviewed every five years
and be reaffirmed, amended, revised or
withdrawn.**

International Standard



622

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Solid mineral fuels — Determination of phosphorus content — Reduced molybdophosphate photometric method

Combustibles minéraux solides — Dosage du phosphore — Méthode photométrique au molybdophosphate réduit

First edition — 1981-08-01

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Ref. No. ISO 622-1981 (E)

Descriptors : solid fuels, coal, coke, lignite, chemical analysis, determination of content, phosphorus, spectroscopic analysis.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 622 was developed by Technical Committee ISO/TC 27, *Solid mineral fuels*, and was circulated to the member bodies in May 1980.

It has been approved by the member bodies of the following countries :

Australia	Hungary	South Africa, Rep. of
Austria	India	Spain
Belgium	Ireland	Turkey
Brazil	Japan	United Kingdom
Canada	Korea, Rep. of	USA
Egypt, Arab Rep. of	Netherlands	USSR
France	Poland	Yugoslavia
Germany, F. R.	Romania	

No member body expressed disapproval of the document.

This International Standard cancels and replaces ISO Recommendation R 622-1967, of which it constitutes a technical revision.

Solid mineral fuels — Determination of phosphorus content — Reduced molybdophosphate photometric method

1 Scope and field of application

This International Standard specifies a reduced molybdophosphate photometric method for the determination of the total phosphorus content of hard coal, lignites and coke. Two methods for taking the phosphorus into solution are specified, namely extraction from the coal or coke ash with acid or by repeated oxidation of the coal or coke, by acid, to remove carbonaceous matter.

2 References

ISO 383, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 565, *Test sieves — Woven metal wire cloth and perforated plate — Nominal sizes of apertures.*

ISO 1171, *Solid mineral fuels — Determination of ash.*

ISO 1988, *Hard coal — Sampling.*

ISO 2309, *Coke — Sampling.*

3 Principle

3.1 Extraction

Method 1 : Removal of carbonaceous material by ashing in a muffle furnace under specified conditions, and extraction of phosphorus by treatment with hydrofluoric and sulphuric acids.

Method 2 : Removal of carbonaceous material by repeated oxidation with nitric acid in the presence of sulphuric acid.

3.2 Determination

Addition of ammonium molybdate and ascorbic acid solution to the acid solution. Measurement of the absorbance of the resulting blue solution by a suitable optical instrument.

4 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1 Hydrofluoric acid, approximately 400 g/l solution.

WARNING — Aqueous hydrofluoric acid is a highly corrosive liquid which attacks glass; the vapour is irritant and toxic. Its action on the skin and eyes is strongly corrosive, producing severe and painful burns which may not be immediately evident and which respond slowly to treatment.

The solution should be handled only inside a well-ventilated fume cupboard.

In the event of contact or suspected contact, flood with water and seek immediate medical attention. The manufacturer's literature should be consulted for further information.

4.2 Sulphuric acid, approximately 490 g/l solution.

4.3 Sulphuric acid concentrated, ρ 1,84 g/ml, approximately 98 % (m/m) solution.

4.4 Nitric acid concentrated, ρ 1,42 g/ml, approximately 70 % (m/m) solution.

4.5 Ammonium molybdate, 60 g/l solution.

4.6 Ascorbic acid, 50 g/l solution.

Prepare the solution fresh daily.

4.7 Antimony potassium tartrate ($\text{KSbO}_3 \cdot \text{C}_4\text{H}_4\text{O}_6$), 1,36 g/l solution.

4.8 Reagent solution.

Mix 25 ml of the sulphuric acid solution (4.2), 10 ml of the ammonium molybdate solution (4.5), 10 ml of the ascorbic acid solution (4.6) and 5 ml of the antimony potassium tartrate solution (4.7). Prepare fresh immediately before use.

4.9 Phosphorus, standard solution corresponding to 0,100 g of P per litre.

Weigh, to the nearest 0,000 1 g, 0,439 2 g of potassium dihydrogen monophosphate (KH_2PO_4) (dried at 110 °C for 1 h), and dissolve in water. Transfer the solution quantitatively to a 1 000 ml one-mark volumetric flask, dilute to the mark and mix.

1 ml of this standard solution contains 0,100 mg of P.