
INTERNATIONAL STANDARD



4285

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

Phosphoric acid for industrial use — Guide to sampling techniques

Acide phosphorique à usage industriel — Guide technique pour l'échantillonnage

First edition — 1977-12-01

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UDC 661.634 : 620.11

Ref. No. ISO 4285-1977 (E)

Descriptors : phosphoric acids, sampling, chemical 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 4285 was developed by Technical Committee ISO/TC 47, *Chemistry*, and was circulated to the member bodies in October 1975.

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

Australia	Germany	Poland
Austria	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Israel	Switzerland
Czechoslovakia	Italy	Turkey
Finland	Mexico	United Kingdom
France	Netherlands	U.S.S.R.

This International Standard has also been approved by the International Union of Pure and Applied Chemistry (IUPAC).

Phosphoric acid for industrial use – Guide to sampling techniques

1 SCOPE

This International Standard is a guide to sampling techniques concerning the procedure to be followed and the precautions to be observed for the taking, the preparation and the storage of samples from a delivery of phosphoric acid for industrial use, with a view to assessing its quality.

2 FIELD OF APPLICATION

This guide to sampling techniques applies to ortho-phosphoric and polyphosphoric acids prepared by thermal and by wet processes, as well as to crystallized phosphoric acids.

The sampling of wet-process acids is particularly difficult because of the frequent presence of a more or less copious precipitate that is not easy to disperse into a suspension.

3 REFERENCES

ISO 683/XIII, *Heat-treated steels, alloy steels and free-cutting steels – Part 13: Wrought stainless steels*.

ISO 3165, *Sampling of chemical products for industrial use – Safety in sampling*.

ISO ..., *Chemical products – Sampling techniques for mobile liquids and mobile liquids derived from solids*.¹⁾

4 MEANS OF DELIVERY

Phosphoric acids are generally delivered in large containers or mobile tanks (trucks, wagons, tankers) each of which may form a batch, and, more rarely, in smaller containers (for example, capacity less than 1 000 ml).

5 APPARATUS

5.1 For sampling

5.1.1 Sampler or weighted bottle, of stainless steel complying with the requirements of grade 20a of ISO 683/XIII (see ISO ...).

5.1.2 Containers, of plastics (polyethylene, polypropylene, polystyrene) when the acids are at a temperature below 50 °C but of stainless steel, complying with the requirements of grade 20a of ISO 683/XIII, when the acids are at a temperature above 50 °C.

The caps of plastics containers shall not contain metal inserts.

NOTE — The use of glass flasks for the taking and storage of samples is not recommended. They can cause the formation of films that adhere to the walls and form fluorosilicates by attack of the glass by the hydrofluoric acid present in phosphoric acids.

5.2 For mixing in the delivery container

Among the available means for mixing, those with the greatest chances of success are the following :

5.2.1 Mechanical stirrer (for example, propeller type), in the case of small delivery containers.

5.2.2 Stirring system by air bubbling or closed-circuit pumping, in the case of large containers and tanks.

NOTES

1 The air-bubbling system is the simplest and most practical and may, furthermore, be adapted to the shape of the reservoir by the use of spargers. It may, however, possibly result in the removal of hydrogen fluoride and fluorosilicate gas from the liquid, or vary the water content.

Closed-circuit pumping, provided that the output of the pump is high enough, is undoubtedly the most effective.

2 The mechanical stirrer and the air lance should be of acid-resistant stainless steel or be plastics coated. For assaying high strength acids, use dry air. If closed-circuit pumping is the method selected, it should be remembered that strong phosphoric acids crystallize at temperature of up to 28 °C; facilities should, therefore, be available to drain and wash out lines and pumps.

1) In preparation.

5.3 Other equipment (for crystallized acids)

5.3.1 Suitable heating system : immersion heaters, heating jackets, etc.

Take care when using immersion heaters, steam coils and jackets as a significant degree of corrosion always occurs if the surface temperature exceeds 80 °C. Furthermore, the temperature of significant attack is decreased considerably if halides are present. The use of hot water through acid-resistant steel coils is recommended.

6 PROCEDURE

6.1 Safety measures

WARNING — Carry out the different operations for the sampling of phosphoric acid with all safety precautions. In particular, the wearing of protective goggles and anti-acid gloves is mandatory. See also ISO 3165.

6.2 Sampling of thermal-process acids

As these acids are normally free from deposits, their sampling does not present any special difficulties. Follow the general techniques specified in ISO ...

6.3 Sampling of wet-process acids

These acids generally contain a precipitate which increases with time and which consists chiefly of calcium sulphate (gypsum), fluorosilicates and phosphates (of calcium, iron, aluminium, etc.). The sampling is difficult : it depends on the strength of the acid, on the storage time (if the acid is "old", the precipitate may be copious and more or less agglomerated), and on the size of the containers (stirring is more difficult in large containers).

The usual technique consists in stirring, in the most effective manner possible, the acid to be sampled, so as to place any precipitate completely into suspension (complete mixing may, in certain cases, require 8 h) and if possible, carrying out sampling while maintaining the stirring.

6.3.1 Case of large containers and mobile tanks

This is the most frequent case.

The batch or the consignment may consist of a single container.

Sampling may be carried out, by agreement between the interested parties, during either the filling or the emptying of each container.

6.3.1.1 SAMPLING

6.3.1.1.1 After filling or before emptying

Stir the contents of the container by the most effective means available (see 5.2) and maintain the stirring during the sampling.

Take the samples as provided for in the sampling plan¹⁾, using the sampler or the weighted bottle (see 5.1) and following the method specified in ISO ..., depending on the shape of the container.

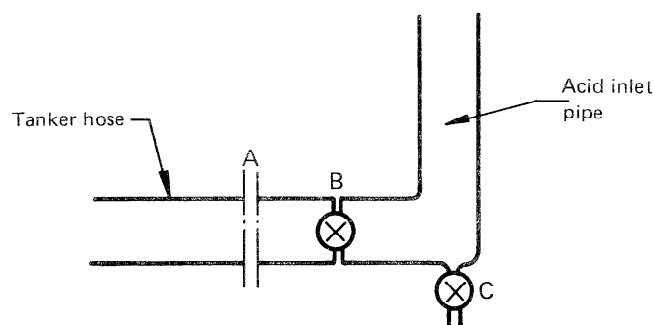
In general, depending on the size of the container, its shape and the locations of the openings, take one or more series of three primary samples — at the bottom, mid-height and surface — by the procedure specified in ISO ...

6.3.1.1.2 During transfer

It is particularly advantageous and useful to take the sample during the filling or emptying of the delivery container, because, by this means, a more representative sample is generally obtained.

Stir the product upstream of the transfer piping, that is in the storage tank during filling of the container, or in the container itself during emptying.

In general, take three primary samples, at the beginning, half-way through, and at the end of transfer. For this purpose, proceed as follows : Connect the tanker hose to the inlet valve at flange A. Close drain valve C and open inlet valve B. Start the tanker pump. When a sample is required, switch off the tanker pump, and quickly close valve B. Then open valve C (this valve is normally used to drain the inlet line) and take a sample of acid.



¹⁾ The sampling plan depends on the particular characteristics of the acid to be sampled, and should be drawn up on statistical bases, amongst others, in conformity with International Standards at present under study.

6.3.1.1.3 In all cases

Collect the various samples quantitatively (without loss of precipitate) in the plastics or stainless steel containers (5.1.2). If possible, avoid the storage of samples of any acid containing significant (greater than 100 mg/kg) levels of halide ions in stainless steel containers.

6.3.1.2 PREPARATION OF REPRESENTATIVE SAMPLES FROM THE CONTAINER (THE CONTAINER CONTENTS) AND FROM THE BULK SAMPLES

Proceed as indicated in ISO . . . , but take particular care to mix each primary sample as thoroughly as possible before combining them in suitable proportions. When samples have been taken during transfer, the masses mixed shall be proportional to the rate of flow.

NOTE — If the samples are to be analysed separately, do not mix them but store them individually as partial samples.

Prepare and store the samples in the plastics containers (5.1.2).

6.3.1.3 PREPARATION OF LABORATORY SAMPLES

Mix the bulk sample (which may be the container sample in the case of a single container) as thoroughly as possible and take the required number of portions of suitable volume, following the sampling plan, so that each laboratory concerned receives

- one assay sample, on which other determinations may also be carried out,
- one sample for other determinations,

and so that one or more additional samples may be kept in case of dispute. Treat all these samples in such a way as to give a solution in which any precipitate has been completely dissolved by a suitable method.

Store the samples in the plastics containers (5.1.2).

6.3.2 Case of smaller containers (for example of capacity less than 1 000 l)

Mix the contents of the containers to be sampled. If a precipitate is present that cannot be dispersed by stirring

(a particularly unfavourable possibility), it may be possible to determine the proportion of sediment and to sample the supernatant liquid and the sediment separately.

6.3.2.1 PRELIMINARY EXAMINATION OF THE CONTENTS OF THE CONTAINERS

Before proceeding to the sampling proper, carry out a preliminary examination of the contents of the containers to be sampled, as indicated in ISO . . .

From the observations made, two cases may arise :

- a) the presence of a precipitate that can be placed in suspension by stirring;
- b) the presence of a more or less copious precipitate that is dense and contains agglomerates, and that cannot be dispersed by stirring.

6.3.2.2 SAMPLING

In the first case, a), carry out the sampling as for large containers (see 6.3.1.1.1), while stirring or, if this is not possible, immediately after stirring.

NOTE — As phosphoric acid is quite viscous, the precipitate, if easily dispersable, may remain in suspension for a considerable time.

In the case of small containers (several hundred litres) in which stirring may be very effective, take a single sample per container.

In the second case, b), allow the contents of the containers to be sampled to settle, determine the proportion of liquid phase to sediment in at least some of the containers, and attempt to sample the two phases separately.

6.4 Sampling of crystallized acids

Crystallized phosphoric acids belong to the category of solids which may be made mobile by heating.

Follow the procedure specified in ISO . . .

7 SAMPLING REPORT

Draw up the report in conformity with ISO . . .

ANNEX

ISO PUBLICATIONS RELATING TO PHOSPHORIC ACID FOR INDUSTRIAL USE

ISO 848 — Determination of calcium content — Titrimetric method.

ISO 849 — Determination of iron content — 2,2'-Bipyridyl photometric method.

ISO 2997 — Determination of sulphate content — Method by reduction and titrimetry.

ISO 3359 — Determination of arsenic content — Silver diethyldithiocarbamate photometric method.

ISO 3360 — Determination of fluorine content — Alizarin complexone and lanthanum nitrate photometric method.*

ISO 3361 — Determination of soluble silica content — Reduced molybdosilicate spectrophotometric method.

ISO 3706 — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method.*

ISO 3707 — Determination of calcium content — Flame atomic absorption method.*

ISO 3708 — Determination of chloride content — Potentiometric method.*

ISO 3709 — Determination of oxides of nitrogen content — 3,4-Xylenol spectrophotometric method.*

ISO 4285 — Guide to sampling techniques.

* Also applicable to phosphoric acid for use in the foodstuffs industry.