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Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Separation by column chromatography and determination of the different phosphate forms

Tripolyphosphate et pyrophosphate de sodium à usage industriel — Séparation par chromatographie sur colonne et dosage des différentes formes de phosphates

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FOREWORD

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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.

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It has been approved by the Member Bodies of the following countries:

Belgium	Hungary	South Africa, Rep. of
Bulgaria	India	Spain
Chile	Israel	Switzerland
Czechoslovakia	Italy	Thailand
Egypt, Arab Rep. of	Netherlands	Turkey
France	New Zealand	United Kingdom
Germany	Poland	Yugoslavia

No Member Body expressed disapproval of the document.

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Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Separation by column chromatography and determination of the different phosphate forms

1 SCOPE

This International Standard specifies a method for the separation and the determination of the different phosphate forms in sodium tripolyphosphate (pentasodium triphosphate) and sodium pyrophosphate (tetrasodium diphosphate) for industrial use.

2 FIELD OF APPLICATION

The procedure specified allows the selective determinations of orthophosphate (Na_2HPO_4), of pyrophosphate ($Na_4P_2O_7$), of tripolyphosphate ($Na_5P_3O_{10}$) and of trimetaphosphate $(NaPO_3)_3$ and also the evaluation of the total content of more condensed forms of phosphates, in the absence of tetrameta- and pentametaphosphates.

NOTES

1 In practice, tetrametaphosphates and pentametaphosphates are not usually present in commercial tripolyphosphates and pyrophosphates.

2 Although the procedure can separate orthophosphate, its determination is not possible if the content is lower than 0,1 %; in this case its determination shall be carried out by the method specified in ISO 2998.

3 REFERENCES

ISO 2998, *Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of orthophosphate — Photometric method using the reduced molybdophosphate.*

ISO 3357, *Sodium tripolyphosphate and sodium pyrophosphate for industrial use — Determination of total phosphorus(V) oxide content — Quinoline phosphomolybdate gravimetric method.*

4 PRINCIPLE

Absorption of the phosphate anions on an anionic ion-exchange resin. Elution with potassium chloride solutions of increasing concentrations. Determination of P_2O_5 in the different eluate volumes.

5 REAGENTS

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

5.1 Ion-exchange resin, strongly basic anionic type, in the chloride form, particle size between 0,07 and 0,16 mm.

NOTE — The resins commercially available as Biorad AG 1 X 8 or Dowex 1 X 8, purified and graded, meet these requirements and have given satisfactory results.

5.2 Hydrochloric acid, approximately 2 M solution.

5.3 Buffer solution, pH 4,3.

Dissolve 51 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) and 46 ml of glacial acetic acid (ρ approximately 1,05 g/ml) in water. Add several milligrams of phenylmercury(II) acetate ($C_6H_5HgOOCCH_3$) and dilute to 1 000 ml.

5.4 Potassium chloride, approximately 0,25 M solution.

5.5 Potassium chloride, approximately 0,50 M solution.

5.6 Potassium chloride, approximately 0,75 M solution.

Prepare solutions 5.4, 5.5, and 5.6 and then add to each solution 10 ml of the buffer solution (5.3) and several milligrams of phenylmercury(II) acetate per litre.

6 APPARATUS

Ordinary laboratory apparatus and

6.1 Ion-exchange column consisting of a glass tube 300 mm long and internal diameter 12 mm. The top of the tube is fitted with a ground glass socket enabling a 100 ml graduated cylindrical dropping funnel to be fitted (see figure 1). The bottom of the tube is extended by a narrower glass tube about 90 mm long and 8 mm diameter, fitted at its middle with a stopcock, the end of which is drawn out to a jet.

The bottom of the ion-exchange column is fitted with a silica wool pad about 10 mm thick or, preferably, a fritted glass disk of porosity P 100 (pore diameter between 40 and 100 μm).

NOTE — The solutions can also be introduced into the column using either a syphon, as illustrated in figure 2, or a suction device, as illustrated in figure 3.

7 PROCEDURE

7.1 Preparation of the ion-exchange column

Wash about 20 g of the resin (5.1) with a jet of water on a 63 μm sieve (see ISO 565) to remove fine particles. Transfer the residue on the sieve to a ground-glass filter, porosity P 40 (pore diameter between 16 and 40 μm) and remove most of the water by suction. After this preliminary preparation, allow the resin to stand for about 12 h in 100 ml of the hydrochloric acid solution (5.2); 18 g of the resin are required.

Introduce the resin into the ion-exchange column (6.1) — 18 g occupies a height of approximately 240 mm — and wash with water until the pH of the effluent is between 4,5 and 5.

During the analysis, maintain the liquid level at all times several millimetres above the top of the column of resin.

7.2 Test portion and preparation of the test solution

Place 0,500 g, weighed to the nearest 0,000 1 g, of the test sample in a 250 ml one-mark volumetric flask. Dissolve in water, add 5 ml of the buffer solution (5.3), dilute to the mark and mix. Filter the solution if it is turbid.

7.3 Preliminary test

7.3.1 *General*

The conditions under which the elution of phosphates is carried out depend on the particle size distribution of the ion-exchange resin. The separation can, moreover, be influenced by the presence of foreign salts in the product examined. For this reason it is recommended that a preliminary test be carried out on the test solution (7.2) to determine the conditions to be observed during the chromatographic separation. In addition, this test provides confirmation that the product is "normal" and that it can be analysed by the procedure specified in this International Standard.

7.3.2 *Determination of the elution conditions*

Immediately after preparation, place 10,0 ml of the test solution (7.2) in the upper part of the column, on top of the ion-exchange resin. Apply a slight air pressure, controlled by means of a pressure regulator, and open the stopcock so as to allow the solution to penetrate into the resin. Close the stopcock before the solution has completely run through so that several millimetres of solution remain above the resin surface.

Carefully wash the inner walls of the column above the resin surface with about 5 ml of water and re-open the stopcock to allow the washings to enter the resin. Close the stopcock before all the liquid has penetrated into the resin. Pass through the column, from the graduated cylindrical dropping funnel, about 110 ml of the potassium chloride solution (5.4), controlling the rate at a constant value of about 2,5 to 3 ml/min (obtained by application of pressure, controlled by the pressure regulator), and collect the eluate in fractions of 5 ml.

Determine the P_2O_5 content of each fraction (see 7.6 and annex A) so as to determine which contain orthophosphate and pyrophosphate respectively, eluted in that order.

Then carry out the same procedure passing about 80 ml of the potassium chloride solution (5.5) through the column to elute the tripolyphosphate.

Finally, carry out the same procedure with about 80 ml of the potassium chloride solution (5.6) to elute the trimetaphosphate. Draw a diagram (see figure 4 for a typical diagram) having the successive 5 ml fractions as abscissae and the corresponding contents of P_2O_5 as ordinates.

Determine from this diagram the minimum volumes of the potassium chloride solutions which should be used to effect the separation.

7.4 Regeneration of the column

At the end of the preliminary test, check that several millimetres of potassium chloride solution remain above the resin surface, pass about 200 ml of the hydrochloric acid solution (5.2) through the column and allow the column to stand under acid conditions for about 12 h. Wash the column, first by passing about 50 ml of the hydrochloric acid solution (5.2) and then by passing about 50 ml of water. Add more water so as to fill the column completely. Stopper the column and invert it to suspend the resin in the water. Return the column to the upright position, allow the resin to settle and pass water through the resin until the pH of the effluent is between 4,5 and 5.

7.5 Separation procedure

Proceed as in 7.3.2, using 10,0 ml of the test solution (7.2) prepared immediately beforehand and using the volumes of the different potassium chloride solutions determined as in 7.3.2. Collect separately, but in one portion each, the four eluate volumes corresponding to each of the four phosphate forms (ortho-, pyro-, tripoly-, and trimetaphosphate). Before changing from one eluate volume to the next, collect two or three 5 ml fractions to verify that the separation is satisfactory. (The intermediate fractions should contain only negligible quantities of P_2O_5. Otherwise, repeat the test.) After collection of the last fraction, regenerate the column, following the procedure specified in 7.4, retaining the eluate obtained by passing the 200 ml of the hydrochloric acid solution (5.2). This will contain any higher condensed forms of phosphate which may be present.

7.6 Determination of phosphorus(V) oxide in the eluates

7.6.1 For the analysis of the 5 ml fractions collected during the preliminary test, use the reduced molybdophosphate photometric method specified in annex A.

7.6.2 For the analysis of sodium tripolyphosphate :

– Determine the phosphorus(V) oxide in the eluate volumes corresponding to the ortho-, to the pyro- and to the trimetaphosphates using the reduced molybdophosphate photometric method specified in annex A. Use the same method to determine the phosphorus(V) oxide content in the eluate volume corresponding to higher condensed forms of phosphate after having previously evaporated the hydrochloric acid.

– Determine the phosphorus(V) oxide in the eluate volumes corresponding to tripolyphosphate by the gravimetric method specified in ISO 3357 as follows. Quantitatively transfer the eluate volume to a 600 ml beaker. Add 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, cover the beaker with a clock glass and boil the solution for 20 min. Cool to room temperature, add 100 ml of the citromolybdate reagent (3.2 of ISO 3357) and proceed as described in 5.3.2 of ISO 3357, starting from paragraph 2.

7.6.3 For the analysis of sodium pyrophosphate :

– Determine the phosphorus(V) oxide in the eluate volumes corresponding to the ortho-, to the tripoly- and to the trimetaphosphate using the reduced molybdophosphate photometric method specified in annex A. Use the same method to determine the phosphorus(V) oxide in the eluate volume corresponding to higher condensed phosphate forms after having previously evaporated the hydrochloric acid.

– Determine the phosphorus(V) oxide in the eluate volumes corresponding to pyrophosphate by the gravimetric method specified in ISO 3357 as follows. Quantitatively transfer the total quantity of the eluate volume to a 600 ml beaker. Add 10 ml of nitric acid solution, ρ approximately 1,40 g/ml, cover the beaker with a clock glass and boil the solution for 20 min. Cool to room temperature, add 100 ml of the citromolybdate reagent (3.2 of ISO 3357) and proceed as specified in 5.3.2 of ISO 3357, starting from paragraph 2.

7.6.4 The sum of the phosphorus(V) oxide contents obtained by the procedures specified in 7.6.2 and 7.6.3 should not differ by more than 1 % from the phosphorus(V) oxide content determined on 10,0 ml of the test solution (7.2) by the method specified in ISO 3357. Otherwise, repeat the test.

8 EXPRESSION OF RESULTS

Using the quantities, in milligrams, of the phosphorus(V) oxide found in each eluate volume, calculate the corresponding contents of phosphate and express them as percentages by mass, using the general formula

$$m_i \times F_i \times \frac{100}{m_o} \times \frac{250}{10} = \frac{2\,500}{m_o} \times m_i \times F_i$$

where

m_o is the mass, in milligrams, of the test portion (7.2);

m_i is the mass, in milligrams, of P_2O_5 found in the eluate volume concerned;

F_i is the conversion factor from P_2O_5 to the corresponding phosphate form, which takes the following values :

for the eluate volume corresponding to Na_2HPO_4,	$F_i = 2{,}000$
for the eluate volume corresponding to $Na_4P_2O_7$,	$F_i = 1{,}873$
for the eluate volume corresponding to $Na_5P_3O_{10}$,	$F_i = 1{,}728$
for the eluate volume corresponding to $(NaPO_3)_3$ and to higher condensed phosphates, expressed as $(NaPO_3)_n$,	$F_i = 1{,}437$

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard or in the International Standards to which reference is made or regarded as optional.

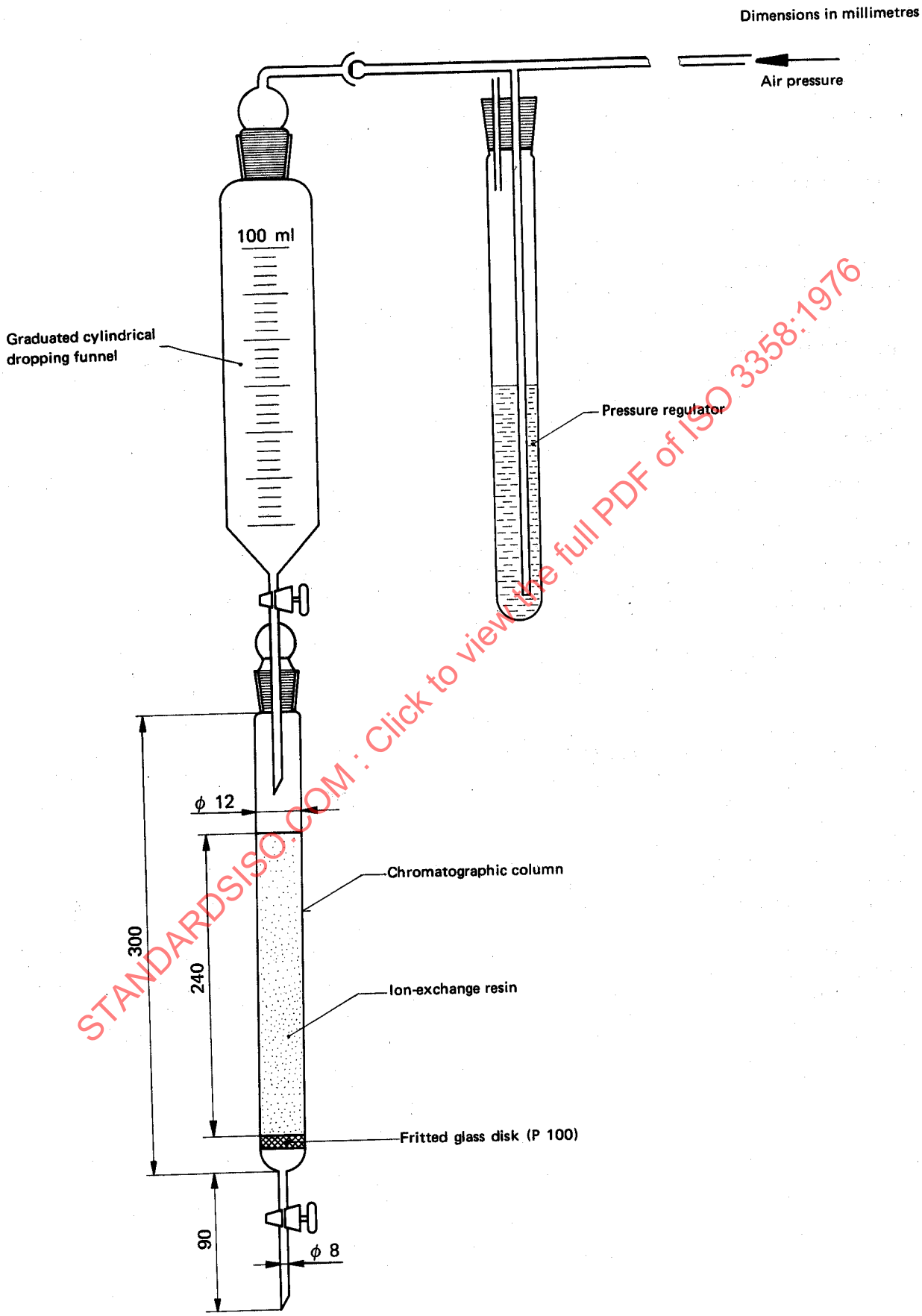

FIGURE 1 – Ion-exchange column

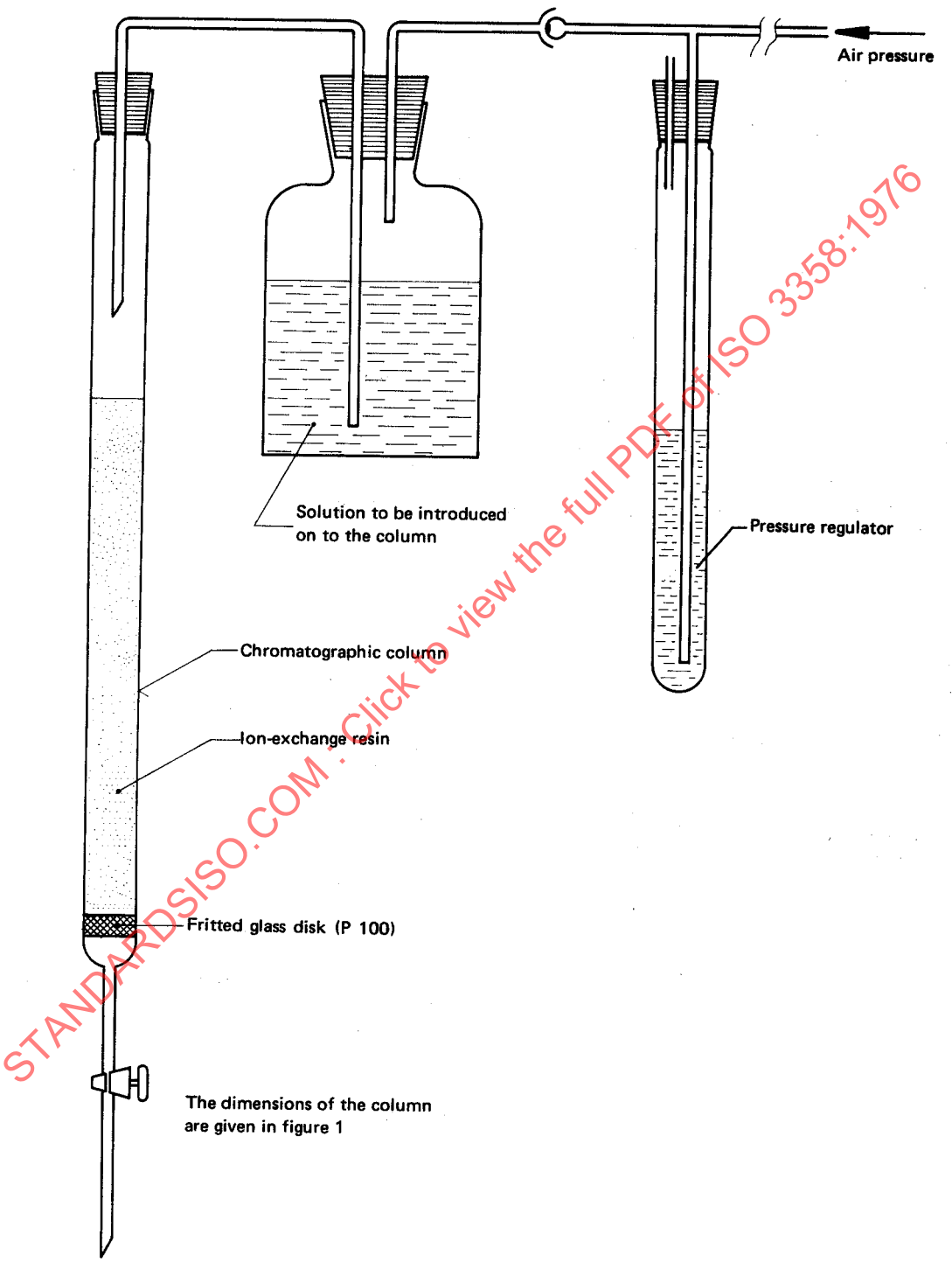

FIGURE 2 – **Typical pressure device**

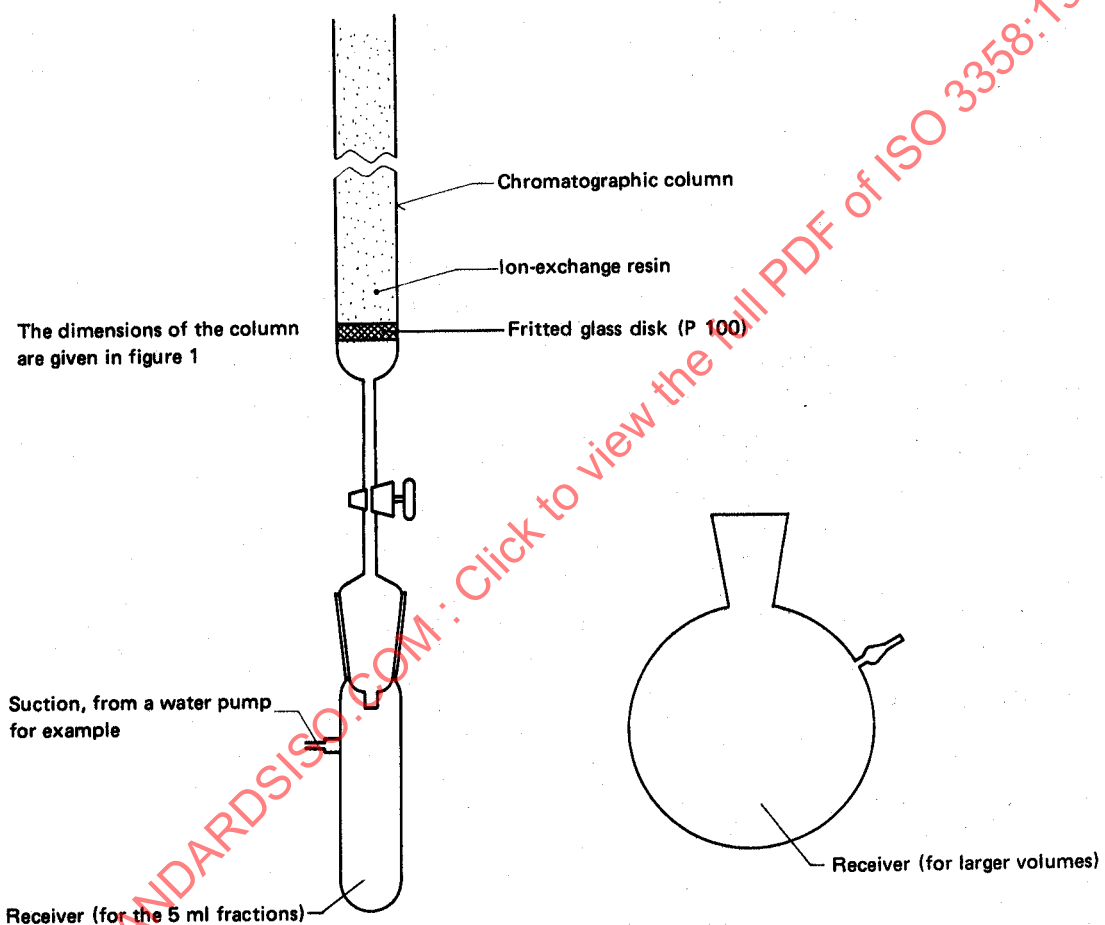

FIGURE 3 – **Typical suction device**

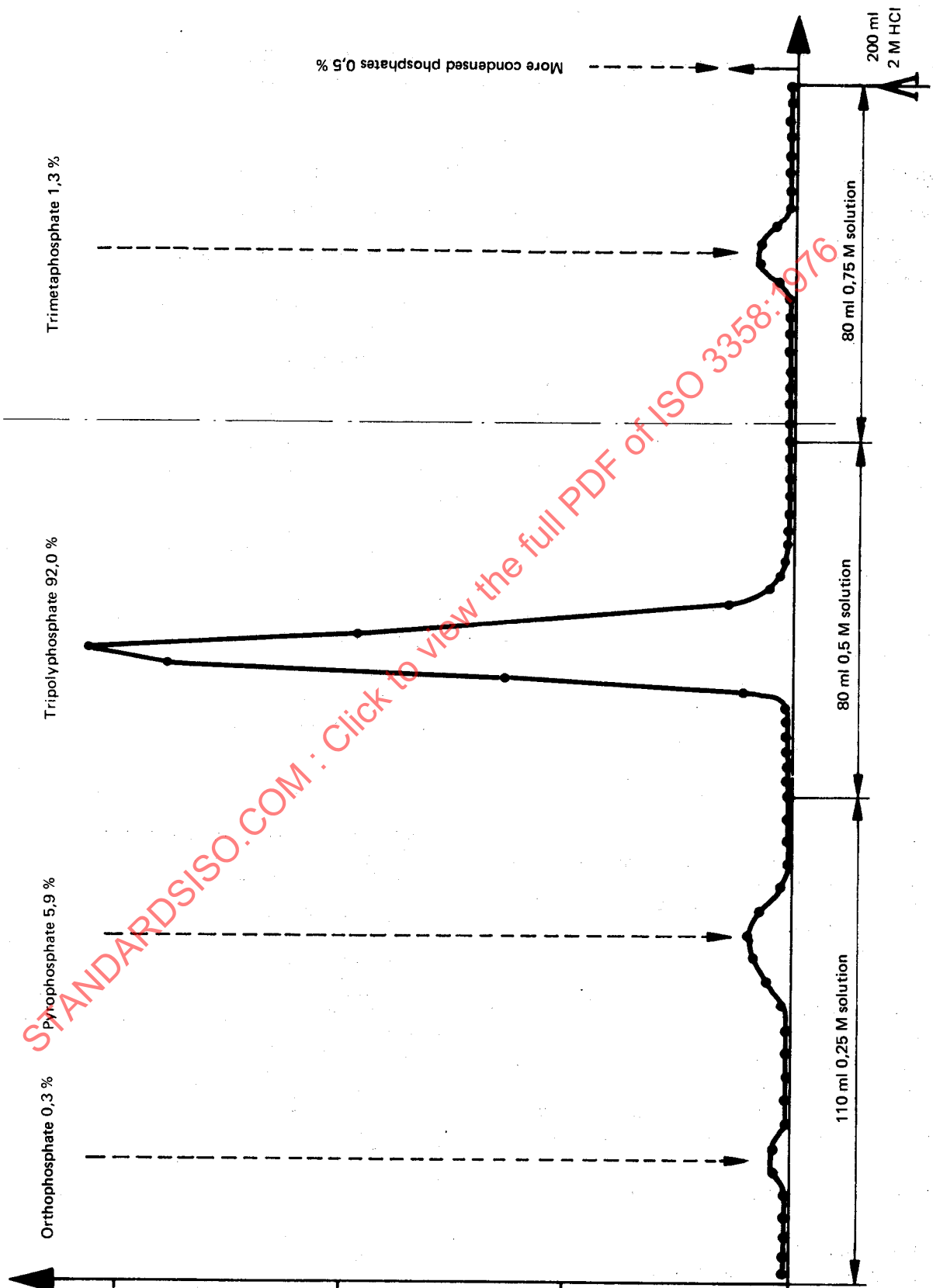

FIGURE 4 – Typical diagram obtained during determination of the elution conditions