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NATO STANDARD

AOP- 4679

ENERGETIC MATERIALS SPECIFICATION FOR RED PHOSPHORUS, AMORPHOUS, MICROENCAPSULATED (FOR USE IN PYROTECHNICS)

**Edition A Version 1
MAY 2018**



**NORTH ATLANTIC TREATY ORGANIZATION
ALLIED ORDNANCE PUBLICATION**

Published by the
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NATO LETTER OF PROMULGATION

25 May 2018

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AOP-4679

TABLE OF CONTENTS

CHAPTER 1	INTRODUCTION	1-1
1.1.	AIM	1-1
1.2	WARNING	1-1
1.3	AGREEMENT	1-1
ANNEX A	PHYSICAL AND CHEMICAL PROPERTIES	A-1
A.1.	DESCRIPTION	A-1
A.2.	REQUIREMENTS	A-1/A2
ANNEX B	TEST PROCEDURES.....	B-1
B.1	Determination of Free Phosphorus	B-1
B.1.1	Reagents	B-1
B.1.2	Procedure	B-1
B.1.3	Colorimetric determination	B-2
B.1.4	Calculation	B-2
B.2	Determination of White Phosphorus	B-2
B.2.1	Reagents	B-2
B.2.2	Procedure	B-3
B.2.3	Colorimetric determination	B-3
B.2.4	Calculation	B-4
B.3	Determination of Volatile Matter	B-4
B.3.1	Procedure	B-4
B.3.2	Calculation	B-4
B.4	Determination of Acidity	B-4
B.4.1	Procedure	B-4
B.4.2	Calculation	B-5
B.5	Determination of Matter Insoluble in Aqua Regia	B-5
B.5.1	Apparatus	B-5
B.5.2	Reagent	B-6
B.5.3	Procedure	B-6
B.5.4	Calculation	B-6
B.6	Determination of Metallic Impurities	B-6
B.6.1	Apparatus	B-6
B.6.2	Procedure	B-7
B.6.3	Calculation	B-7
B.7	Determination of Particle Size Distribution	B-7

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AOP-4679

B.8 Propensity for Phosphine Formation	B-7
B.8.1 Apparatus	B-7
B.8.2 Reagents	B-8
B.8.3 Procedure	B-8

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AOP-4679

<p>CHAPTER 1 INTRODUCTION</p>

1.1. AIM

1. The aim of this agreement is to establish common chemical requirements and test procedures for red phosphorus, amorphous, microencapsulated, and suitable for use in smoke and countermeasure munitions.

1.2 WARNING

1. This STANAG calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and in no way absolves the user from the statutory obligations relating to health and safety at any stage during use. Particular hazards are as follows: Red phosphorus is a flammable material, sensitive to friction and may pose a moderate explosion hazard if in contact with reactive materials. Phosphine gas is highly toxic and may be present in sealed containers of red phosphorus which should always be opened in a well ventilated area. Carbon disulphide is highly flammable and toxic.

1.3 AGREEMENT

1. Participating Nations agree to adopt the chemical requirements described in Annex A and the test procedures described in Annex B for red phosphorus, amorphous, microencapsulated.
2. This AOP will be considered implemented when a nation has issued the necessary orders and instruction putting the contents of this agreement into effect.

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**Annex A to
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ANNEX A PHYSICAL AND CHEMICAL PROPERTIES
--

A.1. DESCRIPTION

The material shall consist essentially of amorphous red phosphorus in the form of a dark red powder. It may also be supplied as a water containing suspension. If water is added for safety reasons, the material shall be dried before testing. The red phosphorus shall be microencapsulated with a suitable polymeric coating and may also contain stabilizer and dust suppressor additives to minimize formation of phosphine and phosphoric acid during shipment, processing and particularly, storage.

A.2. REQUIREMENTS

PROPERTY	REQUIREMENT	TEST METHOD (ANNEX B)
Free phosphorus, %	90.0 minimum	B.1
White phosphorus, %	0.02 maximum	B.2
Volatile matter, %	0.5 maximum	B.3
Acidity	acidity to methyl orange, nil	B.4
Matter insoluble in aqua regia and retained on a 250µm sieve	< 0.1 %	B.5
Metallic impurities Iron %	0.1 maximum	B.6
Copper %	0.01 maximum	
Particle size	To be specified by the purchaser	B.7
Phosphine formation	10 ppm PH ₃ maximum evolved after 24 hrs at 40°C (100% RH) 10 ppm PH ₃ maximum evolved after 48 hrs at 25°C (75% RH)	B.8

Table 1: Requirements

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PROPERTY	REQUIREMENT	TEST METHOD
Microencapsulant	Maximum 10% in total	To be provided by the supplier

Table 2: Requirements

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ANNEX B TEST PROCEDURES

Unless otherwise instructed, the following shall be used:

Chemicals of analytical reagent quality

B.1 Determination of Free Phosphorus

B.1.1 Reagents

Nitric acid, concentrated (density = 1.40 g cm⁻³)

Perchloric acid, concentrated (60%)

Nitric acid, diluted 1:2 parts in water

Ammonium vanadate solution: Dissolve 2.5 g of ammonium vanadate in 500 cm³ of hot distilled water. Cool, add 20 cm³ of concentrated nitric acid and dilute to 1000 cm³ with water. Filter if necessary to obtain a clear solution.

Ammonium molybdate solution: Dissolve 50 g of ammonium molybdate, tetrahydrate in warm distilled water. Cool and dilute with water to 1000 cm³, filtering if necessary.

Standard phosphate solution: Dissolve 1.0982 g potassium di-hydrogen phosphate, previously dried in an oven at 105°C, in 250 cm³ of distilled water in a volumetric flask. Dilute 50 cm³ of this solution to 1000 cm³ with water in a volumetric flask. (1 cm³ ≡ 0.05 mg P)

B.1.2 Procedure

Weigh accurately, (m_1), between 0.040 g - 0.042 g of sample and transfer to a 400 cm³ beaker. Add 20 cm³ of water and 30 cm³ of nitric acid ($d = 1.40\text{g cm}^{-3}$) and cover with a clock glass. Heat the beaker slowly to allow the reaction to proceed and then more strongly until the solution is of a syrupy consistency. Add carefully, dropwise, 10 drops of perchloric acid and continue heating until white fumes appear. Remove the beaker from the heat, cool and transfer quantitatively to a 1000 cm³ volumetric flask, washing out with water. Transfer by pipette 20 cm³ of the solution into a 100 cm³ volumetric flask adding:

10 cm³ of 1:2 dilute nitric acid

10 cm³ of ammonium vanadate solution

10 cm³ of ammonium molybdate solution

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Annex B to AOP-4679

Shake the flask well to mix the solutions and then make up to the mark with water. Allow to rest for at least 30 minutes in the dark. Prepare a blank solution in the same way but omitting the phosphorus extract.

B.1.3 Colorimetric determination

Prepare a calibration solution of phosphorus as follows:

Use the Standard phosphate solution, $1 \text{ cm}^3 \equiv 0.05 \text{ mg P}$ (paragraph B.1.1). Into a series of 100 cm^3 graduated flasks, add 5, 10, 15, 20 and 30 cm^3 of this solution (equivalent to 0.25, 0.5, 0.75, 1.0 and 1.5 mg of phosphorus) and then add to each flask the nitric acid, ammonium vanadate and ammonium molybdate solutions as used for the test sample.

Allow the colour to develop and measure the absorbance of the solutions at 465 nm using a suitable UV-VIS spectrometer.

Plot a calibration graph for the five standard solutions and use this to determine the concentration of phosphorus in the test sample.

B.1.4 Calculation

$$\text{Free Phosphorus (\%)} = \frac{\text{mg P (determined from graph)} \times 5000}{m_1 \text{ (in mg)}}$$

B.2 Determination of White Phosphorus

B.2.1 Reagents

Carbon disulphide (analytical grade) See Note 1
Nitric acid, concentrated (density = 1.40 g cm^{-3})
Iodine (purified by re-sublimation)
Perchloric acid, concentrated (60%)

Nitric acid, diluted 1:2 parts in water

Ammonium vanadate solution: Dissolve 2.5 g of ammonium vanadate in 500 cm^3 of hot distilled water. Cool, add 20 cm^3 of concentrated nitric acid and dilute to 1000 cm^3 with water. Filter if necessary to obtain a clear solution.

Ammonium molybdate solution: Dissolve 50 g of ammonium molybdate tetrahydrate in warm distilled water. Cool and dilute with water to 1000 cm^3 , filtering if necessary.

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**Annex B to
AOP-4679**

Standard phosphate solution, ($1 \text{ cm}^3 \equiv 0.05 \text{ mg P}$) (see paragraph B.1.1)

Note 1: carbon tetrachloride may be used as an alternative to carbon disulphide in this procedure.

B.2.2 Procedure

Transfer 25 g ($\pm 0.1\text{g}$) of sample into an Erlenmeyer flask with a stopper. Add 50 cm^3 of carbon disulphide, stopper the flask and shake well. Allow to rest in the dark for at least 10 hours. Using a pipette, transfer 20 cm^3 of the solution to a filter funnel lined with a Whatman No 2 paper (or equivalent), cover with a watch glass to prevent evaporation and collect the filtrate in a 100 cm^3 beaker. Add to the beaker approximately 10 g of concentrated nitric acid and 0.5 g ($\pm 0.05 \text{ g}$) of iodine. Transfer the beaker to a hot water bath and heat until the carbon disulphide evaporates completely and the beaker is coated in sublimed iodine scales. Then transfer the beaker to a hot plate and bring the solution to a syrupy consistency without allowing it to dry out. When the solution is almost dry, add cautiously dropwise, approximately 1 cm^3 of concentrated perchloric acid and continue heating until white fumes appear. Remove from the hotplate, cool and transfer quantitatively to a 100 cm^3 graduated flask. Dilute to the mark with water.

Using a pipette, transfer 50 cm^3 of the extract into another 100 cm^3 graduated flask and then add:

- 10 cm^3 of dilute nitric acid (1:2)
- 10 cm^3 of ammonium vanadate solution
- 10 cm^3 of ammonium molybdate solution

Shake the flask well to mix the solutions and then make up to the mark with water. Allow to rest for at least 30 minutes in the dark. Prepare a blank solution in the same way but omitting the phosphorus extract.

B.2.3 Colorimetric determination

Prepare a calibration solution of phosphorus as follows:

Use the Standard phosphate solution, $1 \text{ cm}^3 \equiv 0.05 \text{ mg P}$ (Paragraph B.1.1): Into a series of 100 cm^3 graduated flasks, add 2, 10, 20, 30, 40 and 50 cm^3 of this solution (equivalent to 0.1, 0.5, 1.0, 1.5, 2.0 and 2.5 mg of phosphorus) and then add to each flask the nitric acid, ammonium vanadate and ammonium molybdate solutions as used for the test sample.

Allow the colour to develop and measure the absorbance of the solutions at 465 nm using a suitable UV-VIS spectrometer.

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Plot a calibration graph for the six standard solutions and use this to determine the concentration of phosphorus in the test sample.

B.2.4 Calculation

White Phosphorus (%) = mg P (determined from graph) $\times 10^{-2}$

B.3 Determination of Volatile Matter

B.3.1 Procedure

Weigh a clean, dry, aluminium dish with a loosely fitting lid (m_1). Add 5 g (± 0.05 g) of the sample to the dish, replace the lid and re-weigh (m_2). Carefully remove the lid and place the dish, with the lid alongside, in a vacuum desiccator over freshly activated silica gel. Evacuate the desiccator and leave for 24 hours, during which time the pressure in the desiccator must not exceed 10 mm mercury (1333 Pa). Release the vacuum carefully, passing the incoming air through a suitable drying vessel filled with activated silica gel. Replace the lid on the dish and reweigh (m_3).

Note 2: It is important not to disturb the very light amorphous powder when releasing the vacuum.

B.3.2 Calculation

$$\text{Volatile matter (\%)} = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

B.4 Determination of Acidity

B.4.1 Procedure

Prepare a Büchner funnel by fitting a double thickness of close texture filter paper (Whatman No 42 or equivalent). Fit the prepared funnel to a clean, dry, filter-flask, capacity 500 cm³ to 1000 cm³ and apply suction. Weigh 15 g (± 0.1 g) of sample into a clean, dry, 250 cm³ beaker and add by pipette 100 cm³ of freshly boiled and cooled water. Stir the mixture for 10 minutes and then allow it to settle for 5 minutes (See Note 3).

Decant the supernatant liquid through the prepared funnel and add by pipette a second 100 cm³ portion of freshly boiled and cooled water to the beaker. Stir for 5 minutes and allow to settle for 5 minutes. Decant the supernatant liquid through the funnel and then add a third portion of 100 cm³ freshly boiled and cooled water to the beaker. Repeat the 5 minute stirring and 5 minute settling and then decant the whole of the liquid into the funnel. Do not wash the beaker or funnel. Suck dry. Thoroughly mix

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Annex B to AOP-4679

the contents of the filter flask and transfer to a suitable measuring cylinder. The volume should be $300 \text{ cm}^3 \pm 3 \text{ cm}^3$.

Note 3: The stipulated times should be adhered to so that oxidation of phosphorus is minimized.

Using a pipette, transfer 100 cm^3 of the water extract to a 250 cm^3 Erlenmeyer flask and add a few drops of phenolphthalein indicator solution. Titrate the solution using standard sodium hydroxide solution (0.1M) until the red violet end-point (V_1). Carry out a blank determination using 100 cm^3 of freshly boiled and cooled water (V_4).

Transfer a second 100 cm^3 portion of the water extract to another 250 cm^3 Erlenmeyer flask and add a few drops of screened methyl orange indicator solution.

When an acid reaction is indicated, titrate the solution with the standard sodium hydroxide solution (V_2)

When an alkaline reaction is indicated, titrate with standard 0.1 M hydrochloric acid (V_3).

Carry out a blank determination using 100 cm^3 of freshly boiled and cooled water (V_5).

B.4.2 Calculation

Acidity to phenolphthalein (as H_2SO_4) % = $(V_1 - V_4) \times 0.098$

Acidity to methyl orange (as H_2SO_4) % = $(V_2 - V_5) \times 0.098$

Alkalinity to methyl orange (as Na_2CO_3) % = $(V_3 - V_5) \times 0.106$

B.5 Determination of Matter Insoluble in Aqua Regia

B.5.1 Apparatus

Sieve: (ISO 3310-1; BS410) 50 mm diameter and of the following aperture size: 250 μm

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**Annex B to
AOP-4679**

B.5.2 Reagent

aqua regia: Mix together carefully, 1 volume of nitric acid (density = 1.42 g cm⁻³) and 3 volumes of hydrochloric acid (density = 1.18 g cm⁻³)

B.5.3 Procedure

Transfer 5 g (\pm 0.05g) of sample (m_1) to a 600 cm³ beaker and add 50 cm³ water and 20 cm³ of aqua regia. Cover the beaker with a clock glass. Warm to initiate the reaction, then remove from the heat and add a further 20 cm³ of aqua regia. Maintain the reaction by the application of heat and drop wise addition of the aqua regia until a total of 120 cm³ of the reagent has been added. When the vigorous reaction has subsided, heat the solution to boiling and boil for 5 minutes. Cool to ambient temperature and dilute the solution to approximately 300 cm³ with water.

Filter the solution through a No 42 filter paper and transfer any residue in the beaker quantitatively to the paper. Wash the filter paper thoroughly with water and finally with acetone. Place the filter paper in a previously ignited, cooled and weighed porcelain crucible (m_2) and dry in an oven at $103 \pm 2^\circ\text{C}$. Ignite the crucible and contents at 700°C until all of the carbon has been burnt off, cool and reweigh (m_3).

Transfer the residue in the crucible to a 250 μm sieve and brush with a small soft brush made of natural bristle until no more material passes the sieve. Weigh any material retained on the sieve (m_4).

B.5.4 Calculation

$$\text{Total material insoluble in aqua regia (\%)} = \frac{m_3 - m_2}{m_1} \times 100$$

$$\text{Matter insoluble in aqua regia retained on a 250}\mu\text{m sieve (\%)} = \frac{m_4}{m_1} \times 100$$

B.6 Determination of Metallic Impurities

Determine the content of copper and iron impurities by ICP spectroscopy or any other suitable method.

B.6.1 Apparatus

For ICP measurement, ensure all glassware is free from metals. Soak glassware in 5% dilute nitric acid, rinse with distilled water, soak in 5% dilute hydrochloric acid and again rinse in water. Run the ICP spectrometer in accordance with the manufacturer's

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Annex B to AOP-4679

specification using 223.008 nm for copper and 238.204 nm for iron. Calibrate the spectrometer using standard iron and copper solutions up to 5 ppm concentration.

B.6.2 Procedure

Weigh accurately, 0.2 g (± 0.05 g) of the red phosphorus into a 400 cm³ beaker. Add 50 cm³ of distilled water, 25 cm³ of concentrated nitric acid and 25 cm³ of concentrated hydrochloric acid. Heat the sample slowly until the volume is approximately 30 cm³. Remove the sample from the source of heat and cover with a watch glass if the reaction becomes violent. If particulates are still present, add a further 10 cm³ of nitric acid and wash down the inside of the beaker with distilled water. Evaporate the contents to approximately 30 cm³, cool and filter (using a fine texture paper) into a 100 cm³ volumetric flask. Dilute to volume with water.

Run the prepared solution in the ICP and compare with calibration solutions.

B.6.3 Calculation

$$\% \text{ Iron (or copper)} = \frac{A}{W} \times 0.01$$

Where A is the concentration of the metal in mg l⁻¹ and W the weight of sample in grammes.

B.7 Determination of Particle Size Distribution

Prepare a suitable dispersion of the sample in water (or water/alcohol) and determine the particle size distribution using a laser light scattering technique according to the manufacturer's operating instructions.

B.8 Propensity for Phosphine Formation

B.8.1 Apparatus

The test assembly is comprised of a gas-tight glass vessel of known volume V_{App} linked via a re-circulatory gas pump to a phosphine specific gas detector. A schematic diagram is given in Figure 1. The design shall allow a sample of red phosphorus to be suspended approximately centrally in the vessel and a glass dish with a reagent for establishing a certain relative humidity in the base of the vessel. The gas pump and detector shall be linked to the glass apparatus by suitable tubing. The tube material and any tap fittings shall be gas tight and inert to phosphine over the time period of the test.

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Annex B to AOP-4679

Note 4: A suitable vessel may be fabricated from laboratory glassware such as a bell jar or multi-necked flask.

Note 5: It is desirable to control the temperature of the test vessel assembly to the test temperature $T \pm 1^\circ\text{C}$.

Note 6: Any commercial phosphine detector may be used. A typical detection range will be 0-1000 ppm at 1 ppm resolution. The detector shall be calibrated as recommended by the manufacturer using a standard phosphine gas source. Some examples of commercially available detectors are listed in Table 4.

Note 7: It is desirable to control the temperature of the test vessel assembly to $25 \pm 1^\circ\text{C}$

B.8.2 Reagents

a. Sodium Chloride solution: Prepare a saturated solution in hot water, cool and add excess solid sodium chloride to form a slurry. The atmosphere above this solution in the test apparatus will be approximately 75% relative humidity at 25°C .

b. Water. The atmosphere above the water will be 100% relative humidity

B.8.3 Procedure

Place a sample of red phosphorus into the test apparatus together with a glass dish of reagent for establishing a certain relative humidity. Close the gas taps and maintain the assembly at test temperature for the test duration. Reagents, relative humidities, test temperatures and test durations are given in Table 3. At the end of the test period after opening the taps the atmosphere is pumped over the gas detector in order to measure the phosphine concentration.

The volume of phosphine generated from 1 g of phosphorous at the chosen test conditions is given by:

$$V^{(\text{PH}_3)} = X * V^{(\text{App})} / m^{(\text{P})}$$

$V^{(\text{PH}_3)}$ = evolved volume of phosphine [$\mu\text{l}^{(\text{PH}_3)}/\text{g}^{(\text{P})}$]

X = phosphine concentration [ppm]

$V^{(\text{App})}$ = volume of test apparatus [L]

$m^{(\text{P})}$ = phosphorus sample mass [g]

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PROCEDURE	REAGENT	R.H.	TEST TEMPERATURE	TEST DURATION	REQUIREMENT (Maximum)
A	A	75%	25 ± 1 °C	48 hrs.	10 µL (PH ₃)/g (P)
B	B	100%	40 ± 1 °C	24 hrs.	10 µL (PH ₃)/g (P)

Table 3: Test temperatures and durations

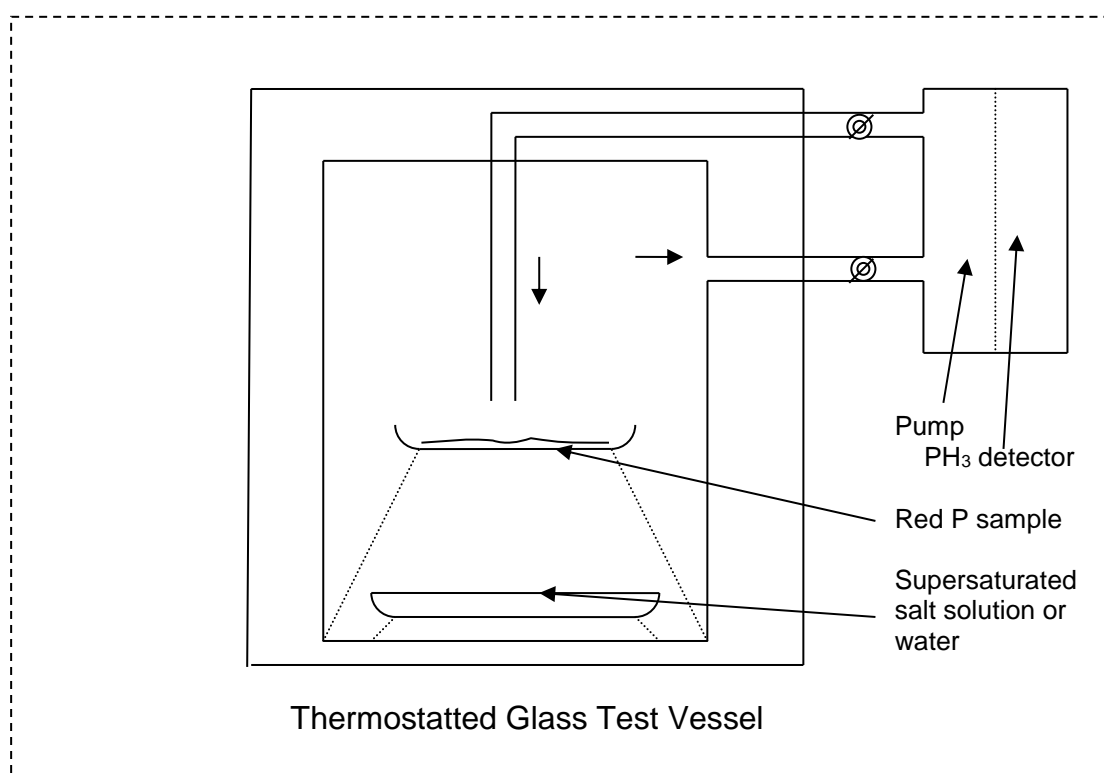


Figure 1: Schematic of gas apparatus

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Supplier	Model	Range/ppm	Comments
Dräger Multi-national outlets	PAC 7000	0-20ppm/0.01ppm	Various configurations available
Honeywell Multi-national outlets	ToxiPro	0-20ppm/0.01ppm	Electrochemical sensor
Uniphos Envirotronic PVT Ltd Mumbai, India	350 PH3	0-20ppm/0.01ppm	Electrochemical sensor
Sensidyne- Kitegawa Multi-national outlets	121U 146S 121SG	0.05-2ppm, 0.1-20ppm, 5-150ppm	Detection tubes
Honeywell Multi-national outlets	PHD6	0-20ppm	Electrochemical sensor No 54 54 13

Table 4: Examples of commercially available phosphine detectors

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