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Report

THE ANALYSIS OF URANIUM PHOSPHIDES

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THE ANALYSIS OF URANIUM PHOSPHIDES

by

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ABSTRACT

Methods are described for the chemical analysis of uranium monophosphide (UP) and a higher phosphide (U_3P_4) produced as an intermediate in the preparation of the monophosphide. Sintering with a 3 : 1 mixture of sodium peroxide and sodium carbonate in a nickel crucible at 350°C for 1 hour is employed to dissolve samples for the determination of the phosphorus content. After extracting the sinter with water and adjusting to between 0.5 and 0.8 M in HNO_3, the solution is passed through a column of cation exchanger (Zeocarb 225) to remove uranium. The phosphorus content is then determined by precipitation as ammonium phosphomolybdate, followed by filtration, drying at 280°C and weighing. A slight correction is necessary to allow for a negative bias of 0.1% in the method. The precision of a determination is <0.2% (coefficient of variation). For the determination of uranium, a separate sample is taken and dissolved in HNO_3 + HCl. After adjusting to pH 4.5, the uranium is precipitated as uranyl ammonium phosphate by adding ammonium hydrogen phosphate and boiling. The precipitate is filtered, ignited to uranyl pyrophosphate, and weighed. Results are quantitative for this determination with a coefficient of variation of 0.24%.

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1. Introduction

Uranium phosphides are being investigated at A.E.R.E. in a general programme for the development of improved fuels for nuclear reactors. Uranium forms the following three compounds with phosphorus - a monophosphide UP, and the higher phosphides U_3P_4 and UP_2. Uranium monophosphide is of greatest interest for the above requirements, but U_3P_4 is formed as an intermediate in the preparation of UP. An accurate knowledge of the phosphorus and uranium contents of these phosphides is required in research with these materials to establish the compositions of various products and also to assist in fixing optimum experimental conditions for their preparation.

2. Preliminary Considerations

An important requirement in this investigation was to develop methods giving accurate results for UP and U_3P_4 samples using readily-available apparatus. An amperometric titration(1) has been used for the determination of the phosphate content of solutions strongly acidic with respect to sulphuric and perchloric acids, after the removal of uranium by cation exchange. Absorptiometric methods are suitable for small amounts of phosphorus in various materials,(2) but the extension of these methods to differential measurements for macro amounts has not been reported.

Gravimetric methods appeared to offer the most favourable approach for the phosphorus contents of phosphides, after oxidation to orthophosphate. The orthophosphate can be precipitated as ammonium phosphomolybdate, which is filtered and weighed. This method is very attractive due to the favourable gravimetric factor. An alternative approach is based on the dissolution of the phosphomolybdate precipitate in ammonia, followed by precipitation of magnesium ammonium phosphate and ignition to the pyrophosphate. The direct weighing of ammonium phosphomolybdate was chosen for the analysis of phosphides, after dissolution and oxidation of the phosphorus to orthophosphate. Conditions reported by Stockdale(5) were used for the production of stoichiometric ammonium phosphomolybdate. Stockdale found that large amounts of iron caused a positive bias in his method, and, in view of this, uranium may be expected to cause difficulties. The determination of the uranium content of these materials was not expected to present many problems.

3. Experimental

3.1 Determination of Phosphorus

In preliminary experiments to test Stockdale's conditions for precipitation, a solution of potassium dihydrogen phosphate was prepared from Analar grade material and standardised gravimetrically by the magnesium pyrophosphate method (Phosphorus content 22.76 ± 0.02%). Stockdale's method was applied to suitable aliquots of this solution. After filtration and drying at 280°C, the precipitate was weighed as $(NH_4)_3[P(Mo_3O_{10})_4]$. The method gave recoveries of 99.9% of theoretical on pure phosphate solutions, but for synthetic solutions with uranium to phosphorus ratios (atomic) of 1 : 1, a positive bias of about 2% resulted. In the analysis of uranium phosphide samples, therefore the separation of uranium was found to be necessary before the precipitation of ammonium phosphomolybdate.

An obvious method for the separation of uranium from orthophosphate involves the use of a suitable cation exchange resin. Strelow and co-workers(6) have shown that hexavalent uranium has a high Kd for cation exchange resins from 0.1 M HNO_3 solutions, and that the value decreases rapidly with increase in the acidity. Calculations showed that a column of Zeocarb 225 (50-100 mesh) measuring 25 cm x 1.1 cm would have the capacity to retain the uranium from a 100 mg sample of UP. It was also estimated that the volume of solution required to remove the orthophosphate from the column would be convenient for the subsequent direct precipitation of ammonium phosphomolybdate. Experiments were carried out to test this procedure on a synthetic solution prepared such that 50 ml was equivalent to 100 mg UP. A suitable aliquot (50 ml) was passed through a column at 1.5 ml per minute, followed by small equal volumes of water for washing. Each volume of wash solution was tested for phosphate with ammonium molybdate reagent, and as washing approached completion the test was made more sensitive by developing the reduced phosphomolybdenum blue colour. It was found that 100 ml of water was sufficient to wash the column free of phosphate, and this was conveniently done with 4 x 25 ml portions. The total volume of effluent from the column was 150 ml.

Experiments on the dissolution of a UP specimen resulted in solutions with acidities much greater than 0.1 M, which is the optimum acidity for the absorption of uranyl ions onto Zeocarb 225. On attempting to reduce the acidity to 0.1 M by the addition of ammonium hydroxide, precipitation of uranyl ammonium phosphate occurred. In sample analysis, therefore, acidities greater than 0.1 M would be more convenient for the separation procedure, provided that the performance of the ion exchange column was satisfactory. Experiments were carried out with solutions in HCl and HNO_3 containing 90 mg amounts of uranium and varying amounts of acid. It was found that a column of the chosen dimensions was satisfactory for the retention of 90 mg amounts of uranium from solutions up to 1.0 M in HCl and 1.5 M in HNO_3 respectively. In the analysis of phosphides there is the need to oxidise the phosphorus to orthophosphate and so nitric acid was selected as the most suitable medium. Also the amount of nitric acid was selected so that its concentration in solution was in the range 0.5-0.8 M.

The phosphorus method was tested on a synthetic mixture simulating a uranium monophosphide sample. A suitable weight (1.3043 g) of freshly-ignited Specpure U_3O_8 was taken and treated with 10 ml HCl (sp.gr. 1.18) + 2.5 ml HNO_3 (sp.gr. 1.42) + 12.5 ml H_2O, followed by digestion on a hot plate to give complete solution. On cooling, the solution was transferred to a 250 ml graduated flask. A weighed amount (0.6321 g) of Analar potassium dihydrogen phosphate (dried at 120°C for 1 hour) was dissolved in distilled water and added to the uranium in the 250 ml flask. The solution was made up to volume with water and then thoroughly mixed. A 20 ml aliquot (≡ 100 mg UP) was taken and ammonium hydroxide was added carefully until a permanent precipitate was formed. Then 2 ml HNO_3 (sp.gr. 1.42) was added and the solution boiled for 2 minutes. After diluting to 50 ml with water, the solution was passed down the resin column at 1.25 ml/min. The beaker was washed with 2 x 12.5 ml of water which were poured down the column, followed by 3 x 25 ml aliquots of water. The total column effluent amounting to ~150 ml was collected in a 400 ml beaker, and the phosphomolybdate was precipitated, dried and weighed as described in Appendix I.

On 6 determinations a mean recovery of 99.9% was obtained with a coefficient of variation of 0.06%. This agreed with recoveries on a pure solution of potassium dihydrogen phosphate. The amount of phosphate precipitated in each determination was 11 mg, as compared with the optimum amount of 7 mg recommended by Stockdale. However, the ratio of MoO_3 to phosphate in solution was still within the limits given for the accurate precipitation of ammonium phosphomolybdate(5).

3.2 Determination of Uranium

An obvious approach to the determination of this constituent involved the recovery of uranium from the cation exchange column, followed by the determination of its concentration with an accepted volumetric or gravimetric procedure. The passage of 6 M HCl was found to be suitable for the recovery process, but it resulted in a total volume of 300 ml of solution for each sample. The following methods were examined for the determination of the uranium concentration:-

(a) Titration with ceric sulphate after reduction of uranium to the tetravalent state.

(b) Precipitation as ammonium diuranate and ignition to U_3O_8.

(c) Precipitation as uranyl ammonium phosphate and ignition to uranyl pyrophosphate.

Erratic results were obtained by all methods, and a positive bias was generally produced. The reason for this was thought to be due to traces of organic materials eluted from the resin column along with the uranium. Evaporation of eluates followed by destruction of organic matter with nitric acid resulted in slight improvements only. Moreover, the time taken for this procedure was fairly long as the 6 M acid solutions were very susceptible to superheating.

Due to the above difficulties, it was decided to determine the uranium content on a separate part of the sample, and the most direct method under these circumstances involved the precipitation of uranium as uranyl ammonium phosphate. Milner and Edwards(7) employed this precipitation in the presence of EDTA for the separation of uranium from other elements. A slightly modified form of this method was applied to the present problem, followed by ignition of the precipitate to uranyl pyrophosphate for weighing. The method was tested on 20 ml aliquots (≡ 100 mg UP) of synthetic solution, using the conditions as described(7), except for the addition of EDTA. A Whatman filter accelerator was added to each solution, before the precipitation of uranyl ammonium phosphate, to collect the fine precipitate and prevent it from passing through the filter paper. In addition, each solution was allowed to boil gently for 30 minutes after precipitation. The results of 6 determinations on the synthetic solution gave a mean recovery of 100.0% with a coefficient of variation of 0.24%. The uranium content of the U_3O_8 used in the preparation of the synthetic sample solution was checked volumetrically, after dissolution in HCl with the addition of hydrogen peroxide. The uranium in suitable aliquots was reduced to the tetravalent state with high-purity aluminium metal before titration with standardised ceric sulphate(8).

3.3 The Dissolution and Analysis of Uranium Phosphide Samples

A problem in the dissolution of metallic phosphides in mineral acid is the avoidance of losses of phosphorus as phosphine. A mixture of HNO_3 and HCl is recommended for dissolving phosphor bronze samples and HNO_3 plus bromine water for alkali metal phosphides(9). Initial experiments on dissolving a UP sample in HNO_3 and HCl resulted in the evolution of phosphine, especially on allowing the temperature to get too high. However, when the sample was covered with dilute HNO_3 and placed on a warm hot plate (~60°C), the attack was slow and gas bubbles leaving the solid were absorbed in the solution. To effect complete solution, it was necessary to add HCl in small increments and to digest the solution after each addition. 25 ml 20% $^{v}/v$ HNO_3 and up to 3 ml HCl (sp.gr. 1.18) were required for the complete dissolution of 100 mg amounts of uranium monophosphide. Removal of most of the excess acid by evaporation proved to be necessary before conditioning for the ion exchange separation. The alternative procedure of neutralising the acid with ammonium hydroxide was found to be unsatisfactory, since the ammonium ions in solution decreased the capacity of the ion-exchange column for uranium.

The higher phosphide (U_3P_4) proved to be more resistant to acid attack than the monophosphide, and required prolonged refluxing with HNO_3 and HCl to attain complete solution. An oxidising alkaline fusion or sinter is an alternative to an oxidising acid attack. A satisfactory procedure for some difficultly-soluble materials involves sintering the finely-ground sample with sodium peroxide in a platinum or nickel crucible at temperatures up to 500°C. Experiments showed that reaction between sodium peroxide and uranium phosphide was initiated at a temperature just below 300°C. It was a vigorous reaction causing liquefaction and high rates of attack on the nickel crucible. The reaction needed controlling, and sodium carbonate was chosen as a diluent because of its high melting point and low packing density.

Experiments were made initially with a 1 : 1 mixture by weight of sodium peroxide and sodium carbonate, 1 g being used for each 100 mg of phosphide sample. About $^{2}/3$rds of the sinter material was mixed intimately with the sample in a 4 ml nickel crucible and the remainder was used to cover the mix. The crucible was then covered with a lid and placed in an electrically-heated muffle furnace at a set temperature for a definite time. Each sinter was leached with water to determine the extent of the attack. Sinters prepared at 450°C for 1 hour gave complete solution of the sample. However, attack on the crucible varied from 2.5 mg to 12.5 mg of nickel removed for each sinter. In order to minimise this attack, experiments were carried out by varying the ratio of Na_2O_2 to Na_2CO_3 in the flux in an attempt to achieve a satisfactory sinter at a lower temperature. A sintering temperature of 350°C was considered to be desirable in view of the possible sublimation of P_2O_5 at temperatures higher than 369°C. The best results were obtained with a ratio of 75 : 25 for Na_2O_2 : Na_2CO_3 on sintering for 1 hour at 350°C. Complete solution of the sample resulted and the amount of nickel removed from the crucible was <1 mg. The method was applicable to both U_3P_4 and UP samples without any difficulty, and, by using it on monophosphides instead of mineral acids, the risk of losing phosphorus in the dissolution process should be reduced.

A minor disadvantage of the sinter method could come from the high concentration of sodium salts in solution, since their effects on the phosphorus determination were not known. An obvious approach to this problem was to remove most of the sodium ions from solution by increasing the size of the ion exchange column. The dimensions were increased to 17.5 cm in length and 2 cm in diameter. This choice represented a compromise caused by the need to wash the phosphate through the column with less than 250 ml of solution whilst removing as much sodium as possible. Determinations of sodium concentrations were carried out on a number of column effluents and they were found to range from 15 to 76 µg per ml. At this level and even at concentrations up to 760 µg per ml, no interference was detected in the precipitation of the ammonium phosphomolybdate, and the precision and accuracy of the determination was not affected.

In the analysis of phosphides (UP and U_3P_4), solution of the samples was obtained by sintering 0.1 g with 1 g of a 75 : 25 mixture of Na_2O_2 and Na_2CO_3 in a nickel crucible for 1 hour at 350°C. After leaching with water, the solution was acidified with HNO_3 and boiled. Then a few drops of HCl (sp.gr. 1.18) were added to dissolve any nickel compounds. The solution was adjusted to be 0.5 to 0.8 M in HNO_3 in a volume of 50 ml, and then passed down a column of Zeocarb 225 (50-100 mesh), 17.5 cm x 2 cm, at a rate not exceeding 2.5 ml per minute. The phosphorus content of the effluent was then determined by the method described in Appendix I. A separate portion of sample (0.1 g) was taken for uranium. This was dissolved in HNO_3 + HCl before completing the determination gravimetrically as described in Appendix II.

4. Results

Two samples of uranium monophosphide, prepared from high-purity uranium, were analysed for all constituents. Results for phosphorus were obtained after dissolving samples both by the mineral acid and by the sinter techniques. In the acid attack the dissolution was allowed to proceed very slowly and care was exercised so as to avoid any possible losses of phosphorus as phosphine. The good agreement for the phosphorus contents in Table 1 suggests that there is little to choose between the two methods of dissolution. In general application, however, the sinter method is the one least likely to produce losses of phosphorus. Results for oxygen were obtained by the inert-gas fusion method of analysis, and other impurities by emission spectroscopy. The satisfactory totals for the complete analysis confirm the suitability of the methods employed.

Two samples of the higher phosphide (U_3P_4) were also subjected to a complete analysis with the results shown in Table 1. The determination of phosphorus after dissolution of the sample directly in mineral acids was applied to sample No. 3 only. The slightly lower results, compared with those after dissolution by the sinter technique, confirmed the possibility of losing phosphorus during the prolonged attack with acid required for this type of material. In view of this the peroxide-carbonate sinter method should be employed for effecting the solution of phosphide samples before carrying out the determination of the phosphorus content. The satisfactory values for the total compositions of samples 3 and 4 in Table 1 support the suitability of the methods employed for these samples.

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TABLE 1

Results for the Analysis of Uranium Phosphide Samples

Sample / Constituent	Uranium Monophosphide		Uranium Phosphide (U_3P_4)	
	1	2	3	4
Phosphorus* (Acid)	11.37 ± 0.02	11.49 ± 0.01	14.40 (2 results)	-
Phosphorus* (Sinter)	11.37 ± 0.02	11.47 ± 0.02	14.46 ± 0.03	14.55 ± 0.02
Uranium	88.3 ± 0.2	87.8 ± 0.2	85.0 ± 0.2	84.8 ± 0.3
Oxygen	0.2	0.1	0.24	0.27
Other Metals	0.04	0.04	0.04	0.04
Total Composition (%)	99.91	99.43	99.74	99.66

*Results for phosphorus corrected for 99.9% recovery

APPENDIX I

Procedure for the Determination of Phosphorus in Uranium Phosphides

1. Outline

The sample is mixed with sodium peroxide-sodium carbonate sinter mixture in a nickel crucible and heated at 350°C for one hour. The sinter is leached with water, acidified with nitric acid, and boiled. After adjustment of free nitric acid content the uranium is absorbed on a column of Zeocarb 225 cation exchange resin. The phosphate is washed through the column with water and precipitated with ammonium molybdate reagent as the ammonium phosphomolybdate. This is collected on a sintered filter, dried at 280°C and weighed.

2. Applicability

The method is applicable to phosphides of uranium in the range $UP_{1.0}$ to $UP_{1.33}$.

3. Reagents

Peroxide-Carbonate mixture	Thoroughly mix 3 parts by weight of sodium peroxide 'Analar' grade with 1 part of sodium carbonate (anhydrous) 'Analar' grade. Prepare fresh for each batch of analyses and keep in tightly closed container.
Ammonium Molybdate reagent I.	Dissolve 500 g ammonium nitrate ('Analar' grade) in 500 ml distilled water in a 2 litre flask. Add slowly 700 ml nitric acid sp.gr. 1.42.
II.	Dissolve 50 g ammonium para-molybdate ('Analar' grade) in 300 ml distilled water.
	Slowly add solution II to solution I shaking the flask well during the addition. Dilute to 2 litres. Add 1 mg potassium dihydrogen phosphate dissolved in 10 mls distilled water. Digest in a water bath at 60°C for six hours and stand overnight. Filter through a Whatman No. 42 paper, but do not wash the paper.
Ammonium Nitrate 2% w/v	Dissolve 20 g ammonium nitrate ('Analar' grade) in distilled water, filter and make to 1 litre.

Ammonia solution 1:20	Add 10 ml ammonia solution (sp.gr. 0.88) to 200 ml distilled water.
Nitric Acid sp.g. 1.42	'Analar' grade.
Hydrochloric Acid sp.g. 1.18	'Analar' grade.
Ion Exchange Resin	Leach 100 g Zeocarb 225 cation exchange resin (50-100 mesh) overnight in 500 ml 2M sodium hydroxide solution. Wash well with water by decantation. Leach for one hour with three successive portions of 6M hydrochloric acid. Wash several times by decantation with water to remove fines.

Apparatus

Ion exchange columns	Construct a column as shown in figure I. Half fill the tube with water and expel air bubbles from the outlet tube. Mix the prepared resin with water and, with the help of a wash bottle, transfer the resin until a column 17.5 cm deep is obtained. Pass 100 ml 6M hydrochloric acid through the column followed by 300 ml distilled water. With 25 ml of water above the resin bed adjust the pinch cock to pass 2.5 ml water a minute. The column is then ready for use.

Procedure

Weigh 0.10 g to 0.12 g sample into a 4 ml nickel crucible. (Note 1). Take 1 g of the peroxide-carbonate mixture and mix the sample with about two-thirds of this. Use the remainder to cover the sample-reagent mix. Cover the crucible. Heat for one hour in an electric muffle furnace maintained at 350°C. Remove from the furnace and cool.

Note 1 Sample weight 0.12 g for UP, 0.10 g for U_3P_4.

Place the crucible and lid in a 125 ml conical beaker and extract with water. Wash crucible and lid well. Acidify the solution with nitric acid, and boil to remove carbon dioxide. Continue to boil to reduce the volume of solution. Add a few drops hydrochloric acid (sp.g. 1.18) and reduce to incipient crystallisation (Note 2).

<u>Note 2</u> Nickel attacked in the sinter stage appears as a black precipitate which requires the addition of hydrochloric acid to effect solution.

Cool, dilute with about 25 ml water, add ammonia solution dropwise until a definite permanent precipitate is formed. Add 2 ml nitric acid (sp.g. 1.42) and boil for two minutes. Cool and dilute to 50 ml.

Pass the solution down the column of cation exchange resin at a rate of 2.5 ml per minute. Collect the effluent in a 250 ml graduated flask. Wash the beaker with 2 x 12.5 ml portions of water and pass down the column. Wash the column free of phosphate with 6 x 25 ml portions of water, collecting all in the graduated flask. Make to volume.

Pipette 100 ml aliquots of the phosphate solution into 400 ml beakers. Dilute to 150 ml and place in a water bath at 60°C. When at bath temperature, add 100 ml molybdate reagent also at 60°C and stir thoroughly (Note 3). Keep in the water bath for one hour, stirring thoroughly every 15 mins. Remove from the water bath and stand overnight.

<u>Note 3</u> Only warm as much molybdate reagent as required for the immediate determination and for the same time as the samples.

Decant the supernate through a sintered silica filter crucible, porosity 4. Transfer the precipitate to the filter using 2 x 20 ml portions of 2% ammonium nitrate solution freeing any adhering precipitate from the sides

<u>Note 4</u> The precipitate has a slight solubility in the wash liquid and the wash volume should be strictly adhered to.

of the beaker with a rubber "policeman". Wash the precipitate with 5 x 10 ml portions of 2% ammonium nitrate solution (Note 4).

Dry the precipitate in an electric oven at 280°C to constant weight (Note 5). Dissolve the precipitate from the filter with 1:20 ammonia solution and dry the filter crucible for one hour at 280°C. Cool and weigh.

Note 5 As the precipitate is hygroscopic, a constant time in the dessicator should be adhered to. 2 filter crucibles in a 6 inch desiccator for 30 mins using fresh silica gel as dessicant has proved satisfactory.

The phosphorus content is given by

$$\%P = \frac{W_2 \times 2.5 \times 0.01651 \times 100}{W_1}$$

where W_2 = weight of precipitate dissolved from the filter in g.

W_1 = weight of sample taken in g.

and 0.01651 is the weight of phosphorus in 1 g of ammonium phosphomolybdate.

Regeneration of Resin column

Elute cations from the column by passing 300 ml 6M hydrochloric acid at not more than 5 ml/minute. Wash column with 300 ml water and the column is ready for use. If the column is allowed to stand for any length of time condition with 100 ml 6M hydrochloric acid, followed by 300 ml water.

From time to time it is advantageous to backwash the resin with water and allow the bed to resettle while the column is filled with water.

APPENDIX II

Procedure for the determination of Uranium in Uranium Phosphides

Outline

The sample is dissolved in nitric acid - hydrochloric acid mixture. The solution is adjusted and buffered to pH 4.5. Ammonium hydrogen phosphate is added and the solution boiled. The precipitate is filtered off and washed with ammonium acetate solution. It is dried, ignited and weighed as uranyl pyrophosphate.

Applicability

The method is applicable to phosphides in the range $UP_{1.0}$ to $UP_{1.33}$.

Reagents

Ammonium Acetate - Acetic acid buffer	Dissolve 7.7 g ammonium acetate 'Analar' quality in distilled water. Add 6.0 ml glacial acetic acid and dilute to 1 litre.
Ammonium Acetate Wash solution 1% $^w/v$	Dissolve 10 g 'Analar' quality reagent in distilled water and make to 1 litre.
Nitric acid 20% $^v/v$	Add 200 ml nitric acid (sp.g. 1.42) to distilled water, cool and dilute to 1 litre.
Ammonia Solution (sp.g. 0.88)	All 'Analar' quality.
Di-ammonium hydrogen phosphate	
Hydrochloric acid (sp.g. 1.18)	

Method

Weigh out 0.100 g sample into a 125 ml conical beaker. Add 25 ml 20% $^v/v$ nitric acid and cover with a watch glass. Digest on a warm hot-plate until reaction ceases. Then add hydrochloric acid (sp.g. 1.18) in small portions until solution is completed. (Note 1). Allow to cool.

Note 1 3 ml HCl (sp.g. 1.18) is normally sufficient. If $^P/U > 1$ additional nitric acid may be needed.

Transfer to a 400 ml beaker and add one Whatman accelerator dispersed in a little water. Add water to a final volume of 150 ml. Adjust pH to ~ 6.5 by adding ammonia solution dropwise (Note 2).

Note 2 Use pH meter or add 4 drops of methyl red and adjust to just alkaline.

Then add sufficient of the acetic acid - ammonium acetate buffer to restore the pH to 4.5 (Note 3).

Stir in 2 g di-ammonium hydrogen phosphate and bring to the boil. Allow to simmer gently for 30 mins. Cool (Note 4) and filter through a Whatman No. 42 filter paper. Transfer the precipitate to the paper with 1% ammonium acetate wash, and wash three times with this reagent.

Transfer to a weighed porcelain crucible, dry in an air oven, and transfer to an electric muffle furnace at 300°C. Raise the temperature slowly to char off carbonaceous matter and then increase the temperature to 1000°C. After 30 mins remove the crucible from furnace, cool in a desiccator and weigh. Repeat to constant weight (Note 5). Subtract the weight of the filter paper ash.

Then Uranium content is given by

$$U(\%) = \frac{W_2 \times 0.6668 \times 100}{W_1}$$

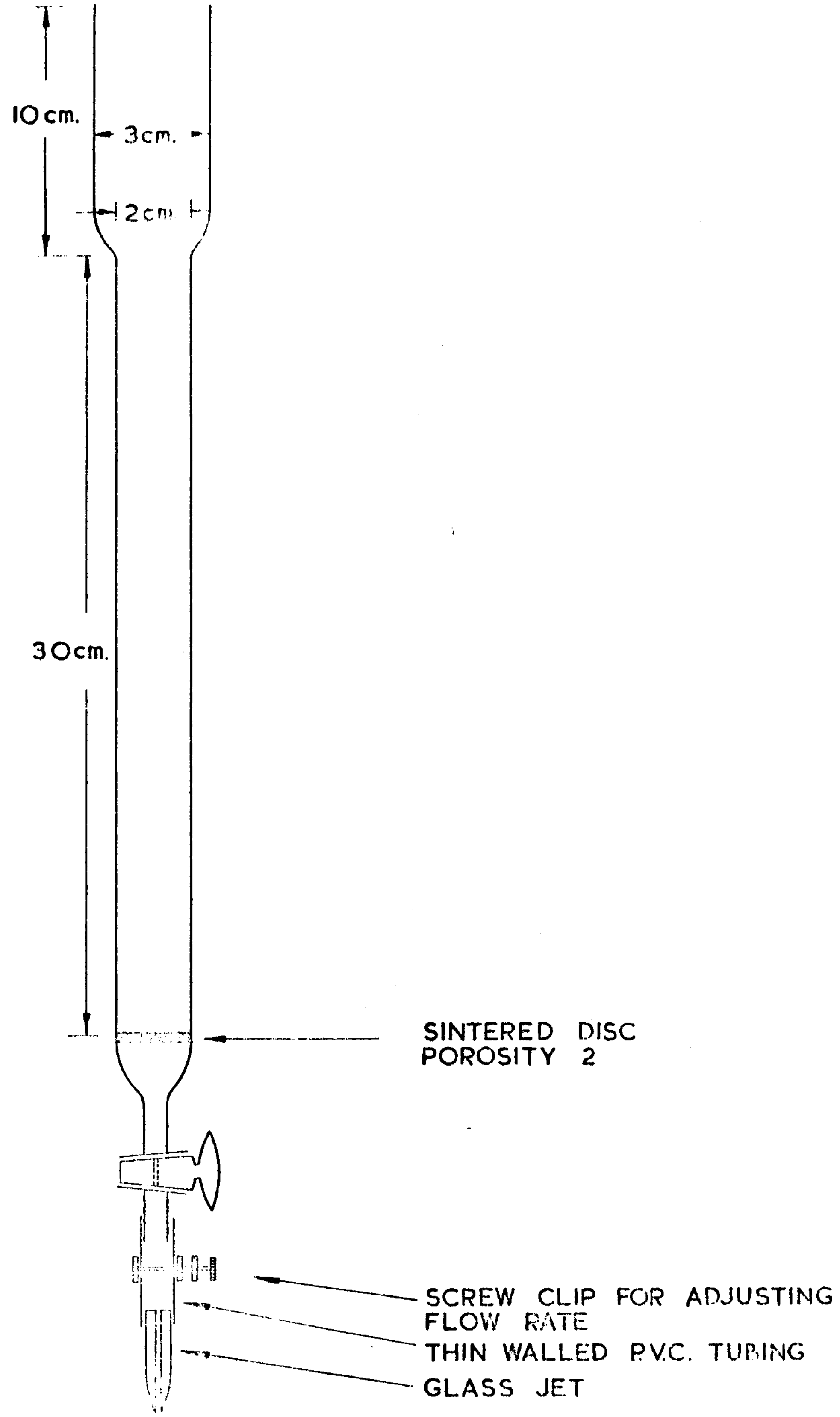
where W_1 = wt sample in grams

W_2 = final weight of precipitate in grams and 0.6668 is the weight of uranium in 1 g of uranium pyrophosphate.

<u>Note 3</u> 25 ml is normally sufficient.

<u>Note 4</u> Preferably stand overnight.

<u>Note 5</u> 30 mins at 1000°C is normally sufficient.



A.E.R.E. R.4906. FIG.1. ION-EXCHANGE COLUMN