REPORT FILE

INTERNAL REPORT

ANALYTICAL PROCEDURES

THORIUM PROCESS

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INTRODUCTION

The following report contains the analytical procedures used in connection with the production of thorium and its salts at Horizons Incorporated.

The types of samples on which these procedures were used include thorium metal, pure salts such as the nitrate and chloride, experimental salts of unknown composition, and thorium scrap material. In the metal, carbon, nitrogen, and oxygen were usually determined. 1-1 HCl will, after long boiling, dissolve everything but ThO_2 according to some authorities. Thus, the HCl insoluble procedure may be used as an indirect determination of oxygen. To obtain complete solution of the metal, fluoride ion must be added in very small amounts to catalyze the reaction. Fluoride may be added in the form of hydrofluoric acid or sodium fluosilicate to hydrochloric acid (1:1) or nitric acid (1:1).

In the case of samples of the metal or the pure salts, thorium may be determined by precipitation as the hydroxide and ignition to the oxide.

In all other materials where other cations may be present, thorium can be precipitated as the oxalate, although this is a more time consuming procedure. Precipitation must be made in a solution containing not more than 4% mineral acid. The oxalate precipitation will serve to separate thorium from everything except zirconium and the rare earths.

In unknown materials and thorium scrap, there is frequently

a large amount of insoluble material which may be largely ThO_2 . This must be filtered off before any further analysis is made. ThO_2 is very finely divided and may run through the finest filter paper. To prevent this, filtration is done in a 5% HCl solution. If the material still goes through the filter a small amount of aluminum sulfate may be added to the solution. Thorium determination must then be made by the oxalate method.

Thorium may be detected qualitatively and determined colorimetrically in small amounts by the reagent thoron - 1 - (o - Arsenophenyl a 20)-2. Naphtol 3,6 Disulfonic acid. We have obtained this reagent and are experimenting with its use.

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I. DETERMINATION OF THORIUM AS OXALATE

Procedure

1. Weigh out sample exactly (0.5 to 5.0 g. depending on the per cent expected). Place in a 400 cc. beaker and heat with about 200 cc. water until boiling.
2. Add 20-30 cc. of boiling saturated oxalic acid solution with stirring. This will start precipitation of thorium oxalate.
3. Allow system to stand at room temperature for 2 to 3 hours.
4. Filter off the precipitate using Whatman No. 42 paper.
5. Wash the precipitate on the filter paper with 0.5% HCl containing a small amount of oxalic acid.
6. Transfer filter paper with precipitate to a weighed porcelain crucible and ignite over the hot flame of a Meaker burner. Cool in desiccator and weigh. Repeat ignition to constant weight.
7. Calculate the per cent thorium as follows:

$$\% \text{ Th} = \frac{\text{Wt. precipitate} \times 0.8788 \times 100}{\text{Wt. sample}}$$

This procedure will separate thorium from almost all other metals. Precipitation of thorium in the presence of interfering cations should be made in 4% HCl solution.

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II. DETERMINATION OF THORIUM BY PRECIPITATION AS THE HYDROXIDE

Thorium may be determined by precipitation of the hydroxide and ignition to the oxide, in a pure salt or one containing no other elements whose hydroxides would precipitate.

Procedure

Weigh out a sample containing about 0.1 gm. of Th and place in a 400 cc. beaker. Dilute to 200 cc. and add an excess of ammonium hydroxide while stirring. Heat gently until the precipitate coagulates. Filter through Whatman No. 42 paper and wash the precipitate with water. Transfer the filter paper with precipitate to a porcelain crucible and ignite over the flame of a Meaker burner. Cool in desiccator and weigh.

Calculation:

$$\frac{\text{Wt. precipitate} \times 0.8788 \times 100}{\text{Wt. sample}} = \% \text{ Th}$$

III. NITROGEN

Apparatus

Micro Kjeldahl apparatus, catalog No. M-3074.

Scientific Glass Apparatus Co., Bloomfield, New Jersey

Reagents

Boric Acid - 2% aqueous solution

Boric Acid - 0.85 M sol. (52.5 g/liter)

Hydrofluoric Acid

Sodium Hydroxide - 50% wt. solution/N/100 HCl

Preparation

This procedure may be used to determine ammonia in soluble salts or nitrogen in thorium metal. Soluble salts may be dissolved in water, diluted to a definite volume and aliquot portions taken for analysis. For thorium metal, weigh 3-1.0 g. samples and place in platinum dishes. Add about 10 cc. 1-1 HCl and a very small amount of sodium fluosilicate. Warm if necessary to obtain complete solution. Add 10 cc. 0.85 M boric acid to complex any free fluoride and prevent attack on glassware during distillation.

Procedure

Fill steam generating flask with H₂O containing 5 cc. of H₂SO₄ per liter. Set variac at 53 and allow apparatus to heat up. Distill for one hour. Rinse apparatus. Pour sample through funnel. Add 10 cc. of 50% NaOH and wash funnel with H₂O. Attach receiving flask, containing

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5 ml - 2% H_3BO_3 and 3 drops each of methyl red and bromocresol green, to delivery tube. After first drop of distillate comes over, continue distillation for 10 minutes. Remove receiving flask, washing down tip of delivery tube. Titrate with 0.01 N HCl until pink. Rinse apparatus.

Blanks

Before any samples are run several blanks on the same reagents used for samples must be run until a consistent low (< 0.1 cc. N/100 HCl) titration value is obtained.

Calculation:

$$\frac{(\text{cc } 0.01 \text{ N HCl [sample]} - \text{cc } 0.01 \text{ N HCl [blank]}) \cdot 0.014}{\text{Wt. sample}} = \% \text{ N}$$

$$\frac{(\text{cc } 0.01 \text{ N HCl [sample]} - \text{cc } 0.01 \text{ N HCl [blank]}) \cdot 0.017}{\text{Wt. sample}} = \% \text{ NH}_3$$

IV. DETERMINATION OF HCl INSOLUBLES IN THORIUM METAL

Weigh a 1.0 gm. sample into a 400 cc. beaker. Add 300 cc. 6 N HCl. Place on hot plate over low heat and boil sample gently for at least 4 hours. If the solution is yellow, boiling is continued until the yellow color disappears. More 6 N HCl is added as needed to keep the volume approximately constant. Cool, filter through a weighed Gooch crucible, and wash with dilute HCl (1-10). Dry for 1 hour at 105°C . Cool in a desiccator and weigh.

$$\frac{\text{Wt. Insolubles} \cdot 100}{\text{Wt. Sample}} = \% \text{ Insolubles}$$

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V. DETERMINATION OF HNO₃ HF INSOLUBLES IN THORIUM METAL

Weigh a 1.0 gm. sample into a 400 cc. beaker. Add 50 cc. of H₂O, 50 cc. concentrated HNO₃, and 1 cc. of HF (42 cc. 48% HF per 1000 cc. H₂O). Place on hot plate over low heat and boil gently until sample is all dissolved (about 1 hour). Filter through a weighed Gooch crucible, wash with H₂O and dry at 105°C for 1 hour. Cool in desiccator and weigh.

Calculation:

$$\frac{\text{Weight Insolubles} \times 100}{\text{Wt. Sample}} = \% \text{ Insolubles}$$

VI. CARBON DETERMINATION USING THE LECO SEMI-AUTOMATIC UNIT

Solutions Required

1. KOH - 900 g. C.P. material in 1800 ml. distilled H₂O.
2. Leveling Solution - Add few crystals of Me Orange to 750 ml. distilled H₂O. Filter if necessary. Add 30 ml. conc. H₂SO₄ and 30 g. of Na₂SO₄; add small amount of a good wetting agent.

Apparatus

1. Carbon burette -- pear-shaped bulb with calibrated stem.
2. Absorption pipette - large vessel in rear with float valve and inner bulb. Fill with KOH solution.
3. Leveling Bottle - tall jacketed bottle set in recess in base.

Fill with leveling solution.

4. Header Stopcock - large 3-way stopcock above the apparatus.

Always operate in a clockwise direction.

Intake position - handle horizontal and to the left.

Transfer position - handle vertical and pointing up.

Exhaust position - handle vertical and pointing down.

Lock position - handle horizontal and to the right.

5. Pressure Valve - on base of instrument - left side.
6. Exhaust Valve - on base of instrument - right side.
7. Drying Train - incorporates H_2SO_4 wash bottle, anhydrite and $CaCl_2$ tower and rotometer flow meter.
8. Combustion Tube Furnace - built in pyrometer and current controls. Connected through sulfur trap to carbon burette intake position.

Procedure

1. Weigh out desired sample and accelerator if desired, place in boat, cover with shield, and place in cool zone of combustion tube. Set oxygen flow at about 1.5 liters/min.

Sample -- 1.000 g. - 0.00 - 1.50% Scale

0.2500 g. - 0.00 - 6.00% Scale

2. Adjust leveling solution to zero mark on burette, then fill burette with leveling solution by applying pressure with pressure valve for about 30 seconds with header at "exhaust".

3. Turn off oxygen. Turn on furnace and heat to desired temperature (2150-2250° F average materials, up to 2400° F. for high alloy steels and refractory materials). Sample into hot zone. Heat from 15-40 seconds. If no shield used, 15-20 seconds is sufficient on usual carbon steels, mild alloys and gray or hard irons. If a shield is used, a longer preheat time is required up to 60 seconds.
4. Turn header to "intake". Close pressure valve. Open exhaust and turn on oxygen after sufficient preheat. A pronounced hesitation should be noted in the rate of drop of leveling fluid near the upper part of the burette bulb. If this does not occur, it indicates improper combustion of the sample, or too rapid flow of oxygen. When leveling fluid reaches the blue mark on the burette, turn off oxygen and unplug oxygen baffle in combustion tube. The leveling fluid will drop swiftly to about an inch above the zero mark and then settle gently to zero. If the liquid level rises and falls, hose connections are not steady. Close exhaust valve. Remove plug from absorption pipette.
5. With fluid at zero, turn header to "transfer", open pressure valve and transfer gas to absorption vessel.
6. After burette float valve seats, close pressure valve and open exhaust after a few seconds to draw the gas less the CO₂ back into the burette.

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7. After the pipette float valve has seated, turn header to "lock" position. Bring the burette liquid level to same level as that in the auxiliary leveling tube alongside using pressure valve to raise it, exhaust to lower it.
8. Take reading at the bottom of meniscus. The result is then multiplied by a factor obtained from the factor chart depending on current temperature and barometric pressure.
9. The burette then may be filled and gases exhausted as before and unit is ready for the next determination.

VII. DETERMINATION OF WATER IN THORIUM SALTS -- Karl Fischer Method

Apparatus

Two automatic 50 cc. burettes with connecting burette reservoirs. Both top and side openings must be protected with calcium chloride drying tubes.

Reagents

Karl Fischer Reagent:

May be purchased as a single stable solution. If this is too concentrated for the size samples to be titrated it may be diluted with pyridene or alcohol.

Methanol - Should be ACS reagent grade - as dry as possible.

Standardization of Reagent

Karl Fischer reagent must be standardized every day or whenever determinations are made. Sodium tartrate dihydrate may be used as a primary standard for this purpose.

Procedure

Weigh out duplicate samples containing 50-100 milligrams of water, using closed weighing bottles which have previously been washed in acetone and dried in the oven overnight. Transfer the sample as quickly as possible to a 250 cc. volumetric flask (with a ground glass stopper). The flask should have been washed and dried in the same manner as the weighing bottles. Add 25 cc. methanol from the automatic burette. Since most thorium salts are extremely hygroscopic it is sometimes difficult to transfer all the sample from the weighing bottle to the flask. In this case, use all or part of the 25 cc. of methanol to wash the sample into the flask. Stopper the flask and allow to stand with occasional shaking for one-half hour. Prepare two blanks containing 25 cc. of methanol each and two standards using 0.5 gms. sodium tartrate in 25 cc. methanol. After one-half hour, titrate the samples with Karl Fischer Reagent from the automatic burette. Be sure the burette tip is placed within the elongated neck of the volumetric flask so that as little air reaches it as possible. Add the reagent slowly while shaking the flask. As the endpoint nears, the color changes from canary yellow (chromate yellow) to a reddish-brown iodine color which is the endpoint. Titrate the blanks and standards first and then match the color to these when titrating subsequent samples. The endpoint should be stable for at

least 15-20 minutes. If it is not, continue titrating until a stable endpoint is obtained. Some thorium salts will disperse throughout the alcohol making it difficult to see the endpoint. In this case titrate slowly, stopping after each addition of reagent. Allow the salt to settle to the bottom and observe the color of the supernatant liquid to obtain the endpoint.

Calculation:

Standard

$$\frac{\text{Wt. in milligrams} \times 0.1566}{\text{cc. reagent for sample} - \text{cc. reagent for blank}} = \frac{\text{mg H}_2\text{O per cc KF reagent}}$$

Sample:

$$\frac{(\text{cc reagent for sample} - \text{cc reagent for blank}) \times \text{titer factor}}{10 \times \text{sample weight in grams}} = \% \text{ H}_2\text{O}$$

VIII. CHLORIDE - VOLUMETRIC - MOHR

This method is applicable to soluble chlorides, in neutral solutions.

Reagents

0.1 N AgNO₃ - 17.1 g. AgNO₃ per liter

Standardized against NaCl

Saturated solution K₂CrO₄

Procedure

A 0.2 to 1.0 g. sample is weighed out into a 250 ml. Erlenmeyer flask. If the sample contains HCl it should be neutralized to a methyl

orange end point with NH_4OH . Several drops of K_2CrO_4 are added to the neutral solution. Standard AgNO_3 is added until a faint blood-red tinge is produced.

Calculation:

1 ml. 0.1 N AgNO_3 = 0.003546 g. Cl or

$$\frac{\text{vol. AgNO}_3 \times \text{N} \times .03546.100}{\text{wt. sample}} = \% \text{ Cl}$$

IX. CHLORINE - GRAVIMETRIC METHOD

Weigh out 2-0.5 g. samples. Dissolve in 100 ml H_2O + 2 ml dilute HNO_3 . Add 0.1 N AgNO_3 slowly while stirring until no more precipitate forms with the reagent (30 ml 0.1 AgNO_3 will precipitate 0.1 g Cl). Then add about 20% AgNO_3 in excess. Heat the solution to boiling, occasionally stirring or shaking until the precipitate coagulates and the solution is clear.

Filter through a weighed Gooch crucible. Wash with 1% HNO_3 . Dry at 105°C , cool and weigh.

Calculation:

$$\frac{\text{wt. AgCl} \times .2474 \times .100}{\text{wt. sample}} = \% \text{ Cl}$$

X. DETERMINATION OF NO_3

Solutions

KMnO_4 0.1N may be purchased or diluted from 1N and standardized against sodium oxalate.

FeSO₄ app. 0.2N must be standardized every time it is used.

55 g. FeSO₄ }
20 g. NaCl } Heat to dissolve in 100 cc. H₂O

Dilute to 1L with 50% H₂SO₄. (Gets very hot-must be cooled and done slowly)

Sample Size

Difficult to determine if liquid usually 5 ml in 100 ml. Then take 5, 10, 25 ml aliquots.

Procedure

Take duplicate aliquots and place in 250 ml. Erlenmeyer. Add 25 cc. FeSO₄. (Accurately from burette.) Add, slowly, 20 cc. con. H₂SO₄ to flask. Brown color indicates nitrate. If sample remains yellow add more sample. If it becomes dark brown and then orange, add more FeSO₄ or take smaller sample.

Boil sample about 3 minutes, or until the brown color has disappeared and the sample is yellow. Remove from hot plate and cool under running H₂O. Add 50 ml. of H₂O and cool.

Pour into 1 L. beaker of cold H₂O. Titrate with KMnO₄ until a faint pink persists for 30 seconds.

With each set of samples run duplicate titrations on 10 ml. samples of FeSO₄. Run through same procedure as above.

Calculation:

$$\frac{((\text{ml. FeSO}_4 \times \text{normality}) - (\text{ml. KMnO}_4 \cdot N)) 2.067}{\text{wt. sample}} = \% \text{ NO}_3$$

XI. COLORIMETRIC METHOD FOR IRON

For small amounts of Iron

Reagents

20% ammonium thiocyanate, ethylene glycol monobutyl ether

Procedure

Transfer a sample containing 0.1 - 1.0 mg. Fe to a 100 ml. volumetric flask. The solution should be adjusted so that it contains the equivalent of about 5 ml. concentrated HCl or H₂SO₄. A blank with the same acidity and dilution should be run with the sample. 10 ml. of 20% ammonium thiocyanate are added to sample and blank. In order to stabilize the color for one hour, 2 ml. of ethylene glycol monobutyl ether are added and the sample and blank diluted to mark. Readings are obtained from the colorimeter using a green filter.

A standard iron solution is run with each set of samples to check the curve.

Calculation:

Reading from colorimeter is located on curve to give gm/ml Iron.

Then

$$\frac{\text{gm/ml. Iron} \cdot 100}{\text{gm/ml. sample}} = \% \text{ Fe}$$

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Sr. Research Associate

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APPENDIX

Thorium Analysis - Horizons Incorporated

January, 1955

Personnel: 1 Supervisor, 4 Chemists.

Space: Total area, 460 sq. ft. Fume hoods, 9 linear ft. Laboratory bench occupied by fixed equipment, 25 linear ft. Laboratory bench for general operations, 27 linear ft.

Unusual Items of Equipment

1. Leco No. 4000 Semi-automatic Carbon Determinator
2. Burrel High Temperature Electric Furnace - Model T-2-9
3. Micro Kjeldahl Apparatus No. M-3074
Scientific Glass Apparatus Co., Bloomfield, New Jersey
4. Klett - Summerson Photoelectric Colorimeter, No. 1578.
5. Beckman pH Meter - Line Model H-2
6. Centrifuge - Precision - Model H-8, Catalogue No. 67342

XII. DETERMINATION OF INSOLUBLES - CENTRIFUGE METHOD

Apparatus - Precision Centrifuge - Model H-8, Catalogue No. 67342

2 sets 100 ml centrifuge tubes -

one with tube graduated to 1.5 cc

one with tube graduated to 0.2 cc

Procedure

Weigh out a 5.0 g. sample into a 400 cc beaker. Add about 50 cc of water and 5 cc of concentrated HCl. Heat gently until salts are in solution. Pour into centrifuge tube, washing out all material from the beaker into the tube. Use the small (0.2 ml) tubes for samples with 25% or less insoluble material and the large tubes (1.5 ml) for samples likely to have more than 25%. Cool and dilute with water to the 100 ml mark. Place tubes in centrifuge, making sure that the weight of samples and tubes is balanced on both sides within a few grams. Set centrifuge on speed No. 1 for 30 seconds, then speed No. 2 for 30 seconds, and finally speed No. 3 for 3 minutes. After the centrifuge has stopped, remove the tubes and read the number of cc insolubles present in each. From the standard curve for the particular type sample being run, read off the number of grams insolubles corresponding to the number of cc.

Calculation:

$$\frac{\text{grams insolubles} \times 100}{\text{wt. sample}} = \% \text{ insoluble}$$

Standardization:

Since the kind of insoluble material will vary with different types of samples, a standard curve must be established for each type of insoluble material. Weigh out duplicate samples. Put both samples in solution with water and HCl according to the above procedure. Centrifuge one sample to obtain the number of cc insoluble. Filter the other one through a weighed Gooch crucible, dry, and weigh to obtain the number of grams insoluble. Run a series of samples in this manner. Plot the number of cc against the number of grams.

The accompanying table and graphs give an example of the above procedure. The insoluble material in this case was probably a hydrous oxide of thorium

Table I.

Determination of Insolubles - Large Centrifuge Tubes

Sample No.	Wt. Sample in gms.	No. of cc Insoluble	No. of gms Insoluble
1a	2.0 g	0.20	0.6230
1b	3.0	0.25	0.9313
1c	5.0	0.40	1.5543
1d	10.0	0.70	3.1120
2	15.0	1.10	4.5136
3	10.0	0.80	3.3992
4	10.0	0.58	2.4874
5	5.0	0.25	1.0470

Table II.

Determination of Insolubles - Small Centrifuge Tubes

Sample No.	Wt. Sample in gms.	No. of cc Insoluble	No. of gms Insoluble
6	5.0	0.17	0.8279
7	5.0	0.155	0.7070
8	5.0	0.12	0.5522
9	5.0	0.10	0.4213
10	5.0	0.07	0.2524
11	5.0	0.045	0.1420

ADDITIONAL LABORATORY ANALYTICAL PROCEDURES

DETERMINATION OF WATER IN TITANIUM SALTS -- Karl

Fischer Method

Apparatus

Two automatic 50 cc. burettes with connecting burette reservoirs. Both top and side openings must be protected with calcium chloride drying tubes.

Reagents

Karl Fischer Reagent:

May be purchased as a single stable solution. If this is too concentrated for the size samples to be titrated it may be diluted with pyridine or alcohol.

Methanol - Should be ACS reagent grade - as dry as possible.

Standardization of Reagent

Karl Fischer reagent must be standardized every day or whenever determinations are made. Sodium tartrate dihydrate may be used as a primary standard for this purpose.

Procedure

Weigh out duplicate samples containing 50-100 milligrams of water, using closed weighing bottles which have previously been washed in acetone and dried in the oven overnight. Transfer the sample as quickly as possible to a 250 cc. volumetric flask (with a ground glass stopper). The flask should have been washed and dried in the same manner as the weighing bottles. Add 25 cc. methanol from the automatic burette. It is sometimes difficult to transfer all the sample from the weighing bottle to the flask. In this case, use all or part of the 25 cc. of methanol to wash the sample into the flask. Stopper the flask and allow to stand with occasional shaking for one-half hour.

Prepare two blanks containing 25 cc. of methanol each and two standards using 0.5 gms. sodium tartrate in 25 cc. methanol. After one-half hour, titrate the samples with Karl Fischer Reagent from the automatic burette. Be sure the burette tip is placed within the elongated neck of the volumetric flask so that as little air reaches it as possible. Add the reagent slowly while shaking the flask. As the endpoint nears, the color changes from canary yellow (chromate yellow) to a reddish-brown iodine color which is the endpoint. Titrate the blanks and standards first and then match the color to these when titrating subsequent samples. The endpoint should be stable for at least 15-20 minutes. If it is not, continue titrating until a stable endpoint is obtained. Some thorium salts will disperse throughout the alcohol making it difficult to see the endpoint. In this case titrate slowly, stopping after each addition of reagent. Allow the salt to settle to the bottom and observe the color of the supernatant liquid to obtain the endpoint.

Calculation:

Standard

$$\frac{\text{Wt. in milligrams} \times 0.1566}{\text{cc. reagent for sample} - \text{cc. reagent for blank}} = \frac{\text{mg H}_2\text{O per}}{\text{cc KF reagent}}$$

Sample:

$$\frac{(\text{cc reagent for sample} - \text{cc reagent for blank}) \times \text{titer factor}}{10 \times \text{sample weight in grams}} = \% \text{ H}_2\text{O}$$

IV. DETERMINATION OF HCl INSOLUBLES IN THORIUM METAL*

Principle

Thorium oxide which has been heated to a temperature of 600°C or greater is insoluble in conc. HCl. Weighing the undissolved portion is a measure of the concentration of this compound.

Procedure for Metallic Thorium

If a large supply of metal is available, Method II can be used. If sample is limited it may be necessary to run other analyses on the sample. In this case use Method I.

1) Clean the surface of the metal by dipping it into a pickling solution of 40 ml. conc. HNO_3 and 2 ml of hydrofluorosilicic acid diluted to 100 ml.

2) Weigh a sample of 0.5-1.0 gm. into the pot of a reflux condenser.

3) Add 50 ml. of conc. HCl and reflux for at least four hours.

I.

4a) Transfer the solution to a 100 ml. flask and make to volume with conc. HCl.

4b) Shake well and pour rapidly about 25 ml. into a 100 ml. graduate. (read the exact volume).

4c) Add 40 ml. of water and filter through No. 42 filter paper.

* Method put into use 4/29/55.

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II.

4a) Add 50 ml. of water to the sample and filter through No. 42 filter paper.

Both Methods

- 5) Wash the filter paper with 1-1 HCl.
- 6) Ignite in a weighed platinum crucible at 900°C for two hours.
- 7) Weigh and reheat to constant weight.

Calculations

Let A = sample weight, gm.
 B = empty crucible weight, gm.
 C = weight of ignited crucible, gm.
 D = aliquot volume, ml.

$$\text{Method I} \quad \% \text{ HCl Insoluble, } \% \text{ ThO}_2 = \frac{(C-B)100}{D \cdot A} \cdot 100$$

$$\text{Method II} \quad \% \text{ HCl Insoluble, } \% \text{ ThO}_2 = \frac{(C-B)}{A} \cdot 100$$

Procedure for ThCl₄ Samples

- 1) Weigh duplicate samples of 0.5 - 1.0 gm. into a 100 ml. beaker.
- 2) Add 30 ml. conc. HCl.
- 3) Stir with a magnetic stirrer for twenty minutes.
- 4) Add 40 ml. of water and filter through No. 42 filter paper.
(Conc. HCl will destroy the paper.)

- 5) Wash the filter paper with 1-1 HCl.
- 6) Ignite at 900°C in a furnace in a weighed platinum crucible for two hours.
- 7) Weigh and reheat to constant weight.

Calculations:

Let A = sample weight, gm.
 B = empty crucible wt., gm.
 C = crucible wt. after ignition, gm.

% HCl Insoluble, % $\text{ThO}_2 = \frac{C-B}{A} 100.$

Set - of - 8 - Cycle

When running this procedure for a large number of samples weigh out material for eight runs at a time (a "set"). Run three sets at a time (A, B, and C) as indicated below. The description starts at the point where Set A is ready to come off reflux; the empty crucibles have been weighed and are ready to receive the residues of Set A. Set B has been weighed out into flasks.

1. Take Set A off reflux. Place Set B on reflux.
2. Set A: Filter. Burn. Start igniting.*
3. Set B: Clean crucibles in preparation for Set B.
4. Set C: Weigh out.
5. Set B: Weigh empty crucibles in preparation.
6. Set A: Weigh up cooled crucibles. Calculate.
7. Repeat cycles : Sets B, C, D

* Ignite 900-1000°C for 2 hrs. When ignition is complete interrupt other work and remove crucibles from furnace.

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