Photostat Price \$ 3.30
Microfilm Price \$ 2.40
Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

THIS DOCUMENT CONSISTS OF 18 PAGE(S)
NUMBER 11 OF 20 COPIES, SERIES a

A-2703

-1-
Summary Report

on

THE RECOVERY OF TUBALLOY FROM CARBON-PARTS

Submitted

on

November 5, 1945

under

Contract No. W-7405 eng-74

by

Purdue Research Foundation

and

Department of Chemistry, Purdue University

Personnel

Advisory and Directing Staff: T. DeVries, Physical Methods; H. Hunt
and M. G. Mellon, Analytical Chemistry; D. W. Pearce, Inorganic
Chemistry; and R. N. Shreve, Chemical Engineering.

Research Staff: A. A. Alberts, W. S. Barnhart, R. E. Burns, L. R. Evans,
E. S. Gantz, R. E. Hatton, B. W. Hotten, K. W. Krantz, M. Kruder,
R. Mezey, J. S. Newcomer, A. M. Ribley, B. F. Rider, G. M. Rothrock,
C. V. St. John, R. C. Schreyer, T. S. Taylor, P. E. Weimer, and
Z. D. Welch.

Service Staff: J. P. Bennett, B. L. Brown, W. H. Davis, W. E. Fish,
C. S. Silverthorn and E. L. VanDermay.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

2

Summary Report

on

Research at Purdue University

by

Purdue Research Foundation and Department of Chemistry

November 5, 1945

THE RECOVERY OF TUBALLOY FROM CARBON PARTS

Abstract

1. The recovery of tuballoy from carbon-parts has been investigated by (a) burning the carbon in the presence of a basic substance such as sodium carbonate which, at the same time, converts the tuballoy into a soluble form and (b) extracting the tuballoy with hot nitric acid.
2. Sodium peroxide, sodium hydroxide, calcium carbonate and sodium carbonate were investigated as aids in burning carbon parts. Sodium carbonate was found to be the most effective.
3. Mixtures of sodium carbonate and carbon-parts tend to sinter at high temperatures. Eschka's mixture, calcium oxide-sodium carbonate mixtures and calcium carbonate were found to be less effective than sodium carbonate but the mixtures did not sinter.
4. No appreciable loss of tuballoy was observed during the burning of carbon parts with sodium carbonate.
5. More than 90% of the tuballoy present in the carbon-parts was converted to a soluble form by heating a mixture of sodium carbonate and carbon-parts for 1 hour at about 750°C.
6. The addition of sodium silicate to a mixture of sodium carbonate and carbon-parts had no observed effect on the oxidation of the carbon.
7. The most effective weight ratio of sodium carbonate to ground carbon-parts for use in the recovery of tuballoy from the carbon-parts by burning the carbon was found to be 5:1.
8. At least 90% of the tuballoy present in the carbon-parts can be removed by digestion with nitric acid for one hour.
9. Concentrated nitric acid was found to be only slightly more effective than 10% nitric acid in extracting tuballoy from carbon-parts.

THE RECOVERY OF TUBALLOY FROM CARBON-PARTS

In the operation of a plant certain carbon-parts became coated and impregnated with tuballoy containing substances. Even though these carbon-parts and residues contain only about 0.5% tuballoy it is highly desirable to recover this material. Any carbon particles removed from wash solutions must be also treated to remove any tuballoy present.

Washing the carbon-parts with nitric acid and steam followed by electrostripping removes most of the tuballoy on the surface in a minimum of time. Breaking the carbon-parts into smaller pieces and burning them in an atmosphere of oxygen in a specially designed furnace creates two major problems: namely, loss of tuballoy in the carbon dust and retention of considerable tuballoy in the ash which, too, must be treated for the recovery of tuballoy.

In the present investigation, two general approaches to the problem of recovery of tuballoy from carbon-parts have been used. These are (1) burning the carbon in the presence of a basic material such as sodium carbonate which leaves the tuballoy in a soluble form, and (2) grinding and extracting most of the tuballoy with hot nitric acid after which treatment the residue is burned.

Carbons-parts were ground to approximately 100 mesh by breaking the pieces with a hammer, grinding the broken pieces with a gyratory crusher and finally grinding further with a rod mill. Unless otherwise specified, a portion of this ground carbon was used in the experiments described in this report.

Analytical procedures used for the determination of tuballoy in the extracts obtained in these researches are designated as Method A and Method B. Method A consists essentially of a cupferron separation of tuballoy followed by a dichromate titration and Method B consists of a diethyl ether extraction of tuballoy from a nitric acid solution followed by a colorimetric determination.

A detailed description of these methods as well as the method of preparation and treatment of the sample is described in Appendix A.

Burning Carbon Parts in the Presence of Basic Materials

The experiments were conducted in either of two ways. In one method, the ground carbon parts was thoroughly mixed with sodium carbonate or other basic material in a 100 ml. nickel crucible and the mixture heated in a muffle furnace. In the other method, the materials were mixed in a crucible and heated by a Meker burner with agitation which was supplied by a nickel stirrer. In a few cases air was introduced through a small nickel pipe placed in the mixture. Both stirring and introduction of air were used as a means of increasing the rate of burning.

Sodium Peroxide. In Experiment LE-14 it was found, that upon heating a mixture of 10 g. of ground carbon-parts and 20 g. of sodium peroxide it burst into flame. Attempts were made to control the reaction by heating a mixture of ground carbon-parts and sodium peroxide gradually with a free flame. But it was not found possible to burn an appreciable amount of the carbon before a vigorous reaction occurred.

Sodium Hydroxide. When a mixture of 10 g. of ground carbon-parts and 30 g. of sodium hydroxide was heated in a muffle furnace to 700°C. (Experiment LE-30C), the melt foamed over the sides of the crucible. In Experiment LE-31, the melt was stirred while being heated gradually with an open flame. As in the previous case, foaming could not be prevented. An attempt to reduce the foaming by diluting the sodium hydroxide with an equal weight of sodium carbonate was unsuccessful (Experiment LE-30D).

Sodium Carbonate. It was found that the carbon may be burned completely and the tuballoy rendered in a soluble form by heating ground carbon-parts in the presence of sodium carbonate. In a typical experiment, 10 g. of ground carbon parts was mixed with 50 g. of sodium carbonate in a 100 ml. nickel crucible. The crucible was placed in a muffle furnace and heated at 750°C. for 15 hours. The crucible was removed from the furnace, cooled and the mixture dissolved in nitric acid. The nitric acid solution was filtered to remove a small amount of a red-orange material which was found to be readily soluble in dilute hydrochloric acid. It was shown by a thiocyanate test that this material was an iron compound. The results of this experiment show that tuballoy can be rendered completely soluble by burning the carbon in the presence of sodium carbonate.

Comparison of Basic Materials. Three basic materials have been found which catalyze the burning of carbon and render the tuballoy in a soluble form. These materials are sodium carbonate, calcium carbonate and Eschka's mixture (2 parts magnesium oxide and 1 part sodium carbonate by weight). Ground carbon parts were burned in Eschka's mixture, in a calcium oxide-sodium carbonate mixture, in calcium carbonate and in sodium carbonate in an attempt to find a reaction mixture which could be stirred easily as well as one which would aid in burning carbon and in the recovery of tuballoy. When sodium carbonate was used, stirring became very difficult because of sintering. It was believed that stirring was advisable to increase the rate of burning of the carbon. Several experiments were carried out in which carbon-parts-flux mixture, contained in nickel crucibles was stirred and heat applied by a Meker burner. After the mixture had cooled, nitric acid was added, the solids were removed by filtration and the residue was washed. The filtrate was analyzed for tuballoy content by Method B. Data from these experiments are summarized in Table 1. Results from Experiment LE-23C are included in Table 1 for comparison between sodium carbonate and other materials. Data from Experiment LE-34 show 100% recovery of tuballoy from carbon-parts. These data indicate that Eschka's mixture, calcium carbonate and a calcium oxide-sodium carbonate mixture are not as effective as sodium carbonate for solubilizing the tuballoy. All of the ground carbon-parts reaction mixtures, except those obtained when sodium carbonate was used as a flux, were stirred

Table 1

Comparison of Basic Materials

Expt. No.	LE-29	LE-33	LE-35	LE-34	LE-23C
Carbon-parts, g.	10	10	10	10	10
Flux	Eschka's Mixture	CaO + Na ₂ CO ₃	CaCO ₃	Na ₂ CO ₃	Na ₂ CO ₃
quantity, g.	10	30	30	50	50
Time, hr.	1.5	3	1.5	20	1
Tuballoy recovery, mg.	6.5	0.7	11.2	15.6	15.2
%	41	5	72	100	96

very easily. The ease of stirring becomes an increasingly important factor as the scale of operation increases.

In a large scale experiment (RH-14) broken carbon-parts (about one cubic inch) was burned in the presence of Eschka's mixture. The experiment was conducted in a large nickel pot equipped with a heavy duty stirrer and four nickel pipes through which air was introduced continuously. The pot was set in a brick furnace and heated with four blast lamps. The temperature of the mixture was maintained at $600 \pm 25^\circ\text{C}$. during the entire experiment. One thousand five hundred grams of Eschka's mixture was introduced into the pot and heating started. After one hour, carbon parts was added intermittently in batches of 500-900 g. until a total of 3382 g. of carbon-parts had been added. Stirring was intermittent due to mechanical difficulties. Consequently the stirrer was in operation about one-half of the total heating period. After 51 hours of continuous heating the material was cooled, then mixed with nitric acid and the residue removed by filtration. The filtrate contained 8.484 g. of tuballoy (determined by Method B). The residue weighed 1760 g.; thus, about one-half of the carbon parts charged was burned.

The percentage of tuballoy originally present in the carbon had not been determined; hence, the percentage removed by this procedure cannot be calculated.

Loss of Tuballoy During Burning. In Experiment LE-20, 10 g. of ground carbon-parts was extracted with 250 ml. of 70% nitric acid at reflux temperatures for 17 hours. The residue was filtered off, mixed with 30 g. of sodium carbonate in a 100 ml. nickel crucible and heated at 750°C . for 16 hours. The reaction mixture was dissolved in dilute sulfuric acid and mixed with the nitric acid extract. The total amount of tuballoy recovered in this manner was 17 mg. (analysis by Method A).

In Experiment LE-21, 10 g. of ground carbon parts was mixed with 50 g. of sodium carbonate in a 100 ml. nickel crucible and heated at 750°C . for 18 hours. The mixture was dissolved in dilute sulfuric acid and analyzed by Method A. The amount of tuballoy recovered in the experiment was 18 mg.

It is shown in Table 5 that extraction of ground carbon-parts with nitric acid removes at least 90% of the tuballoy present. Therefore, if any tuballoy were lost during the burning operation, the recovery should be higher in Experiment LE-20 than in Experiment LE-21 because in the former experiment a part of the tuballoy was extracted before burning.

Effect of Time. A series of experiments was conducted in which aliquots of the same batch of ground carbon-parts were used to determine the effect of time of burning on the removal of tuballoy. In each of these experiments, 10 g. of ground carbon parts was mixed with 50 g. of sodium carbonate in a 100 ml. nickel crucible and heated at 750°C . The mixture was dissolved in dilute sulfuric acid, the residue removed by filtration and the sulfuric acid extract analyzed for tuballoy content by Method A. The results are summarized in Table 2.

Table 2

The Effect of Time of Burning on the Recovery of
Tuballoy from Ground Carbon-Parts

Expt. No.	LE-23C	LE-23B	LE-23A	LE-23G
Carbon-parts, g.	10	10	10	10
Na ₂ CO ₃ , g.	50	50	50	50
Temp., °C.	750	750	750	750
Time, hr.	1	3	10	24
Tuballoy in filtrate, mg.	15.2	14.5	14.5	15.7
Tuballoy removed, %	96	93	93	100

The conditions used in Experiment IE-23G were sufficient to burn all of the carbon and to render all of the tuballoy soluble. Therefore, the amount of tuballoy recovered in this experiment was used to determine the percentage recovery in the other experiments. The analytical results for Experiment IE-23C are probably high, but these data show that more than 90% of the tuballoy may be rendered soluble in one hour or less. They also indicate that some of the tuballoy, less than 10%, is difficult to solubilize and cannot be completely removed until the carbon has been burned away. It may be that the tuballoy is in the silicate bonding material of the carbon-parts.

Effect of Sodium Silicate on the Rate of Burning. Since silica is known to be a catalyst for the oxidation of carbon, a series of experiments was conducted to determine its effect on the rate of burning of carbon-parts and the recovery of tuballoy in the presence of sodium carbonate. Aliquots of the same ground carbon as used in the experiments summarized in Table 2 were used. In these experiments 10 g. of carbon-parts was mixed with 50 g. of sodium carbonate and 2 g. of sodium silicate in a nickel crucible and heated at 750°C. for the time specified. The mixture was dissolved in dilute sulfuric acid, the residue filtered off and the filtrate analyzed for tuballoy content by Method A. Data from these experiments are summarized in Table 3. A comparison of these data with those in Table 2 show that the addition of silica has not increased the recovery of tuballoy from ground carbon-parts under the conditions used. Perhaps there is sufficient silica in the bonding material used in the carbon-parts to catalyze the burning so that the quantity added in these experiments had no appreciable effect.

Effect of the Weight Ratio of Sodium Carbonate to Ground Carbon Parts. Experiment IE-25 was performed to determine if equal weights of sodium carbonate and ground carbon-parts are as effective as a 5:1 weight ratio of the same materials in burning the carbon and in recovering the tuballoy. This experiment was performed in an open crucible with a stirrer. Heat was applied with a Meker burner. The sodium carbonate sintered after about 20 minutes and the mixture could no longer be stirred. Nevertheless, heating was continued for one hour without stirring. The percentage of tuballoy recovered was but 10% of that obtained in similar experiments in which a 5:1 ratio was used.

Extraction of Tuballoy from Ground Carbon-Parts with Nitric Acid

Extractions of ground carbon-parts have been made with nitric acid. The procedure, in most cases, has been to mix ground carbon-parts with nitric acid in a flask equipped with a water-cooled condenser and to reflux the acid. In experiments in which refluxing nitric acid was used, agitation produced by boiling was considered adequate.

Experiments in which a temperature below that of refluxing nitric acid was desired, the mixture was stirred mechanically. In some cases, the extraction was made at room temperature by shaking the mixture in a separatory funnel or by rotating the mixture in a ball mill.

Table 3

The Effect of the Addition of Sodium Silicate on the
Recovery of Tuballoy from Ground Carbon-Parts

Expt. No.	LE-23D	LE-23E	LE-23F
Carbon-parts, g.	10	10	10
Na ₂ CO ₃ , g.	50	50	50
Na ₂ SiO ₃ , g.	2	2	2
Temperature, °C.	750	750	750
Time, hr.	1	3	10
Tuballoy in filtrate, mg.	11.9	13.2	14.4
Tuballoy removed, %	76	84	92

The ground carbon-parts used in this series of experiments was shown to contain 15.7 mg. tuballoy for 10 g. of carbon-parts.

Effect of Particle Size. A series of experiments was conducted to determine the effect of particle size upon the amount of tuballoy extracted. One kilogram of carbon-parts was extracted with one liter of 70% nitric acid at 50°C. for a period of 24 hours. The acid was removed after the first 12 hours and an additional liter of nitric acid was added. The amount of tuballoy extracted was determined by Method B. Data for this series of experiments are summarized in Table 4. These results indicate that tuballoy is more easily and completely extracted when the particle size is small. The amount of tuballoy in the carbon-parts varies for each piece and the samples used in Experiments TE-108 and BH-45 were not aliquots of the carbon parts that was used in Experiment TE-111. It is quite logical however, that the more finely ground material having a large surface area per unit weight should be more easily extracted.

Effects of Time and Acid Concentration. Experiments were conducted to show the effects of time and acid concentration on the completeness of extraction of tuballoy from ground carbon-parts with nitric acid. In each experiment, 10 g. of ground carbon-parts and 250 ml. of nitric acid were mixed and the acid refluxed for specific lengths of time. The residue was filtered off and the acid extract analyzed for tuballoy content by Method B. A portion of the original starting material was found to contain 15.7 mg. of tuballoy per 10 g. of carbon-parts. Data from these experiments are summarized in Table 5. These data show that 70% nitric acid is slightly more effective than 10% nitric acid and that refluxing for 1 hour removes as much tuballoy as refluxing for 4 hours. They also indicate that treatment with dilute acid for one hour is adequate to extract about 90% of the tuballoy present in the ground carbon-parts.

Another series of experiments (Experiments LE-44A, B, C, and D) was performed to verify the data in Table 5. In this series, more carefully controlled conditions were used. A weighed amount of carbon-parts was mixed with 100 ml. nitric acid and the acid refluxed for one hour. After one hour, the residue was filtered off and the nitric acid analyzed for tuballoy content by Method B. The residue was dried at 110°C. for 12 hours. The dried residue was mixed with 50 g. of sodium carbonate and the mixture heated for 16 hours at 750°C. When cool, the mixture was dissolved in nitric acid and the solution analyzed for tuballoy content by Method B. Experiment LE-44D was conducted by shaking nitric acid and ground carbon-parts in a separatory funnel for one hour. The resulting residue and acid extract were handled and analyzed in a manner similar to that just described. Data for these experiments are summarized in Table 6. These results confirm the previous conclusion that digestion of the carbon-parts with nitric acid for one hour is sufficient to remove at least 90% of the tuballoy from ground carbon-parts and that concentrations of nitric acid above 10% do not materially increase the completeness of the removal of tuballoy. They also demonstrate that agitating the mixture at room temperature for one hour is adequate to remove approximately 90% of the tuballoy originally present in the ground carbon parts.

Table 4
Effect of Particle Size upon the Extraction of
Carbon-Parts with Nitric Acid

Expt. No.	TE-111	TE-108	BH-45
Carbon-parts, size, mesh	1	10-50	100-200
quantity, g.	100	100	100
HNO ₃ , vol., ml.	200	200	200
conc., %	70	70	70
Temperature, °C.	50	50	50
Time, hr.	24	24	24
Tuballoy removed, g.	0.05	0.14	0.15

~~Table 5~~

The Effect of Time and Acid Concentration on the Extraction
of Tuballoy from Ground Carbon-Parts with Nitric Acid

Expt. No.	LE-37A	LE-37B	LE-37C	LE-37D
Time, hr.	1	1	4	4
HNO ₃ , conc., %	10	70	10	70
vol., ml.	250	250	250	250
Tuballoy extracted, mg.	14.0	14.8	14.0	14.5
%	90	95	90	93

The ground carbon-parts used in this series of experiments was shown to contain 15.7 mg. tuballoy for 10 g. of carbon-parts.

Table 6

Effect of Acid Concentration upon the Extraction of
Tuballoy from Ground Carbon-Parts with Nitric Acid

Expt. No.	LE-44A	LE-44B	LE-44C	LE-44D
Carbon-parts, g.	9.97	9.95	10.00	9.98
HNO ₃ , conc. %	70	40	10	50
vol., ml.	100	100	100	100
Time, hr.	1	1	1	1
Tuballoy in filtrate, mg.	13.6	13.6	14.0	12.9
Tuballoy in residue, mg.	1.2	1.2	1.1	1.8
Tuballoy extracted, %	92	92	93	88

Effect of Continuous Extraction. Ground carbon-parts has been extracted with nitric acid in a continuous extractor. The carbon-parts was held in a glass thimble which was stoppered at the bottom by a glass wool plug. It was thought that this method of extraction would be more effective than digestion. Data from an experiment (Experiment LE-19) conducted in this manner and continued for 170 hours, showed that only 80% of the tuballoy originally present in the carbon-parts was removed. A comparison of this value with tuballoy recoveries obtained by digesting the carbon-parts with nitric acid shows that tuballoy was more completely removed in a shorter period of time by digestion.

Effect of Simultaneous Extraction and Grinding. Experiments have been conducted in which simultaneous extraction and grinding were done. The ground carbon-parts and nitric acid were rotated in a porcelain ball mill. It was hoped that grinding in the presence of nitric acid might extract essentially all of the tuballoy. Data from these experiments are tabulated in Table 7. They show that grinding during extraction with nitric acid has no appreciable effect upon the percentage of tuballoy extracted.

In Experiment LE-50, the average particle size of the carbon-parts was determined before and after the experiment to determine if there was any reduction in particle size. Before extraction the average diameter of the carbon particles was found to be 3.8×10^{-4} cm. and after extraction the average particle size was 2.7×10^{-4} cm.

Acknowledgement

This work was done by Messrs. L. R. Evans, R. E. Hatton, B. W. Hotten, A. M. Ribley, B. F. Rider, G. M. Rothrock, and C. V. St. John.

Respectfully submitted,

E. T. McBee

E. T. McBee
Project Leader

T. DeVries

T. DeVries
Research Supervisor

L. R. Evans

L. R. Evans
Senior Research Chemist

15

Table 7

Effect of Simultaneous Grinding and Extraction of
Ground Carbon-Parts with Nitric Acid

Expt. No.	IE-46	IE-50
Carbon-parts, g.	261.1	123.4
HNO ₃ , conc. %	10	35
vol., ml.	1050	200
Time, hr.	3	41.5
Tuballoy extracted, mg.	755	153
Tuballoy extracted, mg.	59.6	18.1
Tuballoy extracted, %	93	89

ANALYTICAL PROCEDURE

Determination of Tuballoy from Carbon-Parts

Carbon-parts are decomposed and the tuballoy containing materials rendered soluble by heating with sodium carbonate. Such a treatment burns away the carbon with little or no dusting and leaves a residue which is soluble in acid. Both sulfuric acid and nitric acid have been used in this laboratory to dissolve the residue. When sulfuric acid was used, tuballoy determinations were made by a procedure involving a cupferron separation followed by titration. This procedure is described as Method A. Whenever nitric acid was used to dissolve the residue, the tuballoy was extracted with diethyl ether and the amount of tuballoy determined colorimetrically. This procedure is described as Method B.

Preparation and Treatment of the Sample. Weigh 10 g. of a uniform sample of carbon parts ground to at least 60 mesh. Heat with 50 g. of sodium carbonate in a 100 ml. nickel crucible for 12 hours at 750°C. Leach the mass with 250 ml. of hot water and cautiously acidify with dilute acid (approximately 50 ml. of 6 N acid). Boil the acidic solution 15 minutes and reserve for separation and determination of tuballoy. Usually some silicic acid separates when the acidic solution is boiled.

Method ACupferron Separation Followed by Titrimetric Determination of Tuballoy.

Sulfuric acid extracts were analyzed by Method A. Evaporate the solution to fumes of sulfur trioxide, cool, dilute to 50 ml. and adjust to 10% sulfuric acid by volume. Add a 2% potassium permanganate solution until a pink color is obtained. Cool to 20°C., add 10 ml. of a freshly prepared 5% aqueous solution of cupferron and extract in a separatory funnel with 50 ml. of chloroform. Add another 10 ml. of 5% cupferron solution and extract with 50 ml. portions of chloroform until the chloroform layer is colorless. Add 2 ml. of 30% hydrogen peroxide to the aqueous portion and evaporate to fumes of sulfur trioxide. Dilute to 25-50 ml. and adjust the acid concentration to 10% sulfuric acid by volume. Add a 2% potassium permanganate solution dropwise until the solution is pink. Treat with 5 ml. of 4% zinc amalgam for 10 minutes in a separatory funnel, drain off the amalgam and wash it with 25 ml. of 2% sulfuric acid. Add the washings to the solution to be titrated. Add 2 ml. of 85% phosphoric acid, 10 ml. of 0.1 N ferric alum and 3 drops of barium diphenylamine sulfonate indicator and titrate with 0.02 N potassium dichromate to the first permanent color change. Apply the correction factor obtained for the blank on all the reagents used and calculate the tuballoy content.

Method B

Diethyl Ether Extraction Followed by Colorimetric Determination. Nitric acid extractions were analyzed by Method B. Evaporate the nitric acid solution to dryness in a beaker or until salts begin to separate. Cool the concentrate, add 10 ml. of water and enough concentrated silver nitrate solution to precipitate any remaining chloride ions. Transfer the solution and precipitate to an ether extractor. Wash out the beaker with a 10% solution of nitric acid saturated with calcium nitrate (25°C.) directly into the ether extractor. Add enough ether to bring the liquid level to within 4 cm. of the sidearm. Add 50 ml. of water and 100 ml. of diethyl ether (alcohol free) to the side flask. Heat the side flask electrically so as to give a steady stream of ether from the condenser tip. Continue the extraction for 1.25 hours stirring every 10 minutes. After 0.5 hour substitute the side flask with another one containing fresh ether and water. When the extraction is complete, empty the flasks into a beaker and evaporate the ether by moderate heating in order to avoid fume off. Dissolve the residue from the ether extraction in water and dilute to 100 ml. in a volumetric flask. Pipette an aliquot containing 5-10 mg. of tuballoy into a 100 ml. volumetric flask. Add 20 ml. of a 20% sodium carbonate solution and dilute to the mark. Filter the solution through a fine grained paper to remove suspended material. Place a portion of this filtrate in the reference cell of the spectrophotometer. Add four drops of 30% hydrogen peroxide to the remaining filtrate and shake. Measure the transmittancy of the solution at 370_{mμ} in a 1 cm. cell against the reference cell in a Beckman or other suitable spectrophotometer. Determine the tuballoy concentration by means of a calibration curve.