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0 Summary Report

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on

DETERMINATION OF NON-VOLATILE MATTER IN TUBALLOY TETRACHLORIDE

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- 1. Summary Report

on

Research at Purdue University

by

Purdue Research Foundation and Department of Chemistry

December 14, 1945

DETERMINATION OF NON-VOLATILE MATTER IN TUBALLOY TETRACHLORIDE

Abstract

In order to evaluate tuballoy tetrachloride prepared in this laboratory with respect to volatility methods for the determination of non-volatile matter were investigated. As a result of this investigation a procedure was devised which is both rapid and accurate. The apparatus used in this procedure differs from previously described equipment particularly in design and in materials of construction.

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DETERMINATION OF NON-VOLATILE MATTER IN TUBALLOY TETRACHLORIDE

A method is described for the determination of the amount of non-volatile matter in tuballoy tetrachloride. The method is applicable to the problem of this laboratory in determining if a sample of tuballoy tetrachloride will meet the required volatility specification, i.e., whether or not the sample contains less than 1% non-volatile matter. The method described differs somewhat from the procedures described by Smith and Muller in Report R.L. 4.6.911 and by Kraus in Report M-2184, particularly in the design and in materials of construction of the sublimation apparatus.

A complete determination of non-volatile matter can be made in about 1.5 hours. All operations involving the transfer of tuballoy tetrachloride are made in a dry box using magnesium perchlorate as a drying agent. Little or no moisture is taken up during the process inasmuch as in three successive experiments the sublimate was resublimed and found to contain only 0.09% non-volatile matter.

Apparatus

Diagrams of the sublimation apparatus and of the vacuum system are shown by Figures 1 and 2, respectively.

Sublimation Apparatus. The apparatus was made by sealing a Pyrex test tube about 25 cm. long and 30 mm. in diameter to a 35/45 tapered ground glass joint. A cold finger condenser was attached to the other part of the ground glass joint. The condenser extends into the tube so that the end is about 17 cm. from the end of the tube.

The tuballoy tetrachloride sublimate is collected on this cold finger which extends into the hot zone. This arrangement permits a short vapor path and also serves as a convenient means of preventing the deposition of any sublimate on the walls of the apparatus. Before using, this apparatus is washed with nitric acid, followed with water, rinsed twice with acetone and dried at 110°C.

Heater. A 12 inch multiple unit furnace is used to heat the sublimation apparatus. The temperature was measured by means of a chromel-alumel thermocouple and is controlled with an adjustable voltage transformer.

Sample Tube. The sample is introduced into the apparatus in a 6-inch Pyrex tube fitted with a glass wool plug. The glass wool plug is used to prevent particles from being carried out of the tube.

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SUBLIMATION APPARATUS

THERMOCOUPLE

ELECTRIC FURNACE

TO VACUUM SYSTEM

WATER OUTLET

WATER INLET

FIG. 2

FIG. 1

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DIAGRAM OF VACUUM SYSTEM FOR SUBLIMATION APPARATUS

FIG. 2

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Preparation of Sample

The sample of tuballoy tetrachloride is ground so that all particles pass through a 40 mesh sieve. The material is thoroughly mixed and stored in a sample bottle until used.

Volatilization

The sublimation apparatus, thoroughly dried, is mounted into place and evacuated. Dry air is then admitted into the apparatus through the sulfuric acid trap, and dry air is continuously blown through the apparatus as it is opened for the introduction of the tuballoy tetrachloride sample. The sample tube containing the tuballoy tetrachloride is shaken to spread the sample uniformly over the surface. (Dow-Corning silicone lubricant is used to lubricate and to seal the ground glass joint.) The system is evacuated and heating started. Heating is rapid to 300°C. However, if a considerable quantity of gaseous products are present in the sample, heating must be somewhat slower in order to prevent a sudden ejection of the glass wool plug.

The temperature is increased from 300°C. to 600°C. in about one hour. The pressure should be below 0.15 mm. at the end of the run as measured by a McLeod gage.

When volatilization is complete the residue is reddish-brown in color; a tinge of green in the residue indicates that volatilization is incomplete and heating should be continued at 600°C. If the residue is black, air has entered the system during the determination and the results should be discarded.

When sublimation is complete, the apparatus is removed from the furnace and cooled to room temperature in air.

Dry air is admitted into the apparatus before the removal of the sample tube. A Wire hook is used to remove the sample tube. This hook is devised to keep the lip of the tube away from the sides of the apparatus. Before weighing, the tube is wiped first with a damp cloth and then with a dry cloth.

Results

In three successive experiments the sublimate was resublimed to determine whether or not moisture was taken up during the sublimation operation. The final sublimate was found to contain 0.09% non-volatile matter. Hence, This method is applicable for determining whether or not the tuballoy tetrachloride sample meets volatility specifications.

Several experiments were conducted in which samples from the same tuballoy tetrachloride product were sublimed. Data from these experiments are summarized in Table 1. They show an average of 2.43% non-volatile material and an average mean deviation of 0.14%.

Data obtained from a typical experiment are shown in Table 2.

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Table 1

Non-Volatile Matter in TCI_4

<u>Expt. No.</u>	<u>TCI_4, g</u>	<u>Time, min.</u>	<u>Temp. Range, °C.</u>	<u>Pressure, mm. Hg.</u>	<u>Residue, g</u>	<u>Non-Volatile, %</u>	<u>Mean Deviation, %</u>
82	4.9228	65	140-600	0.31-0.09	0.1054	2.14	0.29
83	4.1969	60	80-820	0.19-0.10	0.0947	2.26	0.17
84	4.5058	60	25-600	0.14-0.10	0.1084	2.41	0.02
85	3.3914	60	25-600	0.21-0.10	0.0859	2.53	0.10
86	4.5491	70	25-600	0.55-0.12	0.1168	2.57	0.14
87	4.2144	700	25-610	0.26-0.09	0.1104	2.62	0.19
88	4.0096	60	25-600	0.24-0.10	0.1004	2.50	0.07
						Avg. 2.43	Avg. 0.14

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Table 1

Non-Volatilized Matter in Soil

Expt. No.	Soil	Temp. Range, °C.	Residue, %	Non-Volatilized, %	Mean Deviation, %
65	1-2328	110-120	0.31-0.02	0.1001	0.22
66	1-2328	110-120	0.12-0.10	0.0301	0.17
67	1-2328	110-120	0.11-0.10	0.0600	0.08
68	1-2328	110-120	0.21-0.10	0.0602	0.10
69	1-2328	110-120	0.12-0.12	0.1102	0.16
70	1-2328	110-120	0.22-0.03	0.1104	0.10
71	1-2328	110-120	0.21-0.10	0.1001	0.07
Avg. 0.14					

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Table 2

Determination of Non-Volatile Matter in TiCl_4

Experiment 86

(a) Wt. test tube + glass wool + stopper + TiCl_4 , g.	27.8691
(b) Wt. test tube + glass wool + stopper + residue, g.	23.4368
(c) Wt. test tube + glass wool + stopper, g.	23.3200
(d) Wt. residue, g.	0.1168
(e) Wt. TiCl_4 used, g.	4.5491
(f) Non-volatile matter, %	2.57

<u>Total Time,</u> <u>min.</u>	<u>Temp.,</u> <u>°C.</u>	<u>Pressure,</u> <u>mm. Hg</u>
0	25	0.35
5	315	0.55
40	575	0.12
70	610	0.12

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Respectfully submitted,

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