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G. B. Stange

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WEEKLY ABSTRACTS

October 17, 1946

Section C-II

File Chemistry and General Chemistry

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Week Ending October 16, 1946

By Authority of [Signature]

HIGH TEMPERATURE PILE PROGRAM

(C. A. Boyd)

By [Signature] 4-2-05

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Reaction of Steam and BeO (M. G. Berkman)

Additional experiments were carried out to determine the effect of varying water vapor pressure and steam flow rate on the reaction between steam and BeO. The water used was thoroughly degassed before each run.

Temp. of BeO Pellet (°C)	Rate of Steam Condensation (ml/min)	Water Vapor Pressure (mm Hg)	Weight of BeO Lost (%)
45	0.1	40	0.32
46	0.02	20	0.26
47	0.07	200	0.25
48	0.07	130	0.26

As in previous experiments, the time for each run was 2 1/2 hours. In the present experiments, most of the water collected distilled during the beginning of the run and only a small fraction during the rest of the period.

Volatilization Tests on Impregnated Graphite (G. Mason, J. Wain)

Graphite sampler heated in a mullite tube (Al₂O₃, SiO₂) in helium at 1450°C became coated with large amounts of material which by preliminary spectrographic analysis consists chiefly of Si and Fe. Obviously, heating tests cannot be continued in this apparatus at this temperature. Consequently, it is planned to use induction heating in a quartz apparatus, the quartz being cooled by water or an air blast. It is hoped that the construction and preliminary testing of this apparatus will be completed this week.

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Graphite Impregnation (M. A. Kanter, R. W. Phillips, A. T. King, D. Schultz)

It was found in the previous weeks work that the gain in weights after impregnating was less from water solutions than from organic solvents. To check if this might be due to a reaction between the graphite and the water, the impregnations were repeated using pure water only. Weight losses of the order of 0.03% were obtained which would not account for the difference.

Work was begun on the stability of hexone solutions of uranyl nitrate hexahydrate. The samples were kept for periods of time at the refluxing temperature. A lemon-yellow precipitate, whose identity is still being investigated, was formed in all cases. Variation in the concentration of U₂N₆ gave varying amounts of precipitate. The effect of acid concentration was investigated and showed that increase in acid concentration shortened the time before precipitation occurred. This work will be continued.

Preparations were made to start impregnation of fuel tubes but this is being held up until a firing furnace can be set up.

In order to determine if any graphite is lost during firing after impregnation, tests were made in which the exit gases from the furnace were passed over hot BeO to oxidize any CO given off to CO₂, and then passed through a known amount of Ba(OH)₂ to absorb all the CO₂. It was shown that the amount of carbon coming off is proportional to the amount of uranyl nitrate absorbed. The magnitude of the loss varied from 5 to 8 milligrams on samples of approximately 2.5 grams. Plans are being made to run these same samples through a second impregnation.

Radiation of Beryllia (J. R. Gilbreath, S. R. Gaarder, J. L. Weeks, D. H. Rich, P. Boykins)

There are no new data to report. Report 9-3 (120-day irradiation) has arrived from H.E.W., and work has begun on determinations of physical changes on the exposed BeO and mixed oxide pellets and prisms.

Preparation of Crystalline BeO (M. P. Walling)

A program has been started to investigate possible methods of growing macro-crystals of BeO.

Two methods have been considered: the reaction of BeO with H₂O at elevated temperatures, and fusion and slow recrystallization.

An experiment employing the first of these methods is in preparation now. BeO powder will be sealed into a platinum tube in an atmosphere of water vapor. The tube will then be heated to ca. 1200°C and a temperature gradient of ca. 50°C (i.e., 1200°C to 1150°C) will be established along the length of the tube. It is hoped that this treatment will result in volatilization of the BeO-H₂O reaction product at the hot end of the tube and deposition of BeO at the cold end. By properly controlling the conditions, it should be possible to grow BeO crystals in the cold end of the tube. No work has been done on the fusion method as yet.

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ANALYTICAL PROBLEMS

(F. S. Tomkins).

Spectrographic Laboratory (F. Tomkins, J. A. Brody, C. Patterson, J. Paris, M. Walsh)

A. Service Analysis:

During the week the following analyses were completed:

1. Two samples of air filter paper quantitatively for Be.
2. One sample U_3O_8 for Fe
3. Three samples, qualitatively for major constituents, Al, Be, Mg, Cu, Th.
4. Two samples of PbO quantitatively for B and other impurities.

B. Research:

1. Microphotometric method. Recent experience analyzing Be metal has shown that appreciable B contamination results when pyrex glass is used. Since the most recent experimental standards have been made by impregnating pure BeO with solutions containing small known amounts of impurities, it was thought to be advisable to repeat the solution using quartz wherever possible. This has been done and the first set of standards have been arced and their spectra photographed. We should soon be able to obtain working curves covering the range from .1 ppm to 100 ppm densitometrically.

2. D-C Arc in a Controlled Atmosphere. The two rotameters which had been on order for some time arrived and were mounted on a panel placed back of the optical bench. Copper tubing was fitted to the rotameter with brass couplings. The connections to the oxygen and helium tanks cannot be made until some special needle valves have been ordered. Until these can be obtained, the rotameters may be put into operation using rubber connections to the tanks.

Wet Chemical Analysis (R. Bene, R. Hospelhorn, K. Jensen, R. Telford)

A. Services:

1. Two samples of graphite impregnated with uranium were analyzed for uranium.
2. One uranium oxide sample was analyzed for iron.
3. One thorium - copper sample was analyzed for thorium and copper.
4. Work is continuing on the determination of fluorine in beryllium metal.
5. Alloys of uranium, columbium, and zirconium are being analyzed for columbium and zirconium.
6. A uranium graphite ignition residue is being analyzed for silicon, iron, and uranium.

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B. Research:

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Another run was made in the BeCl_2 volatilization apparatus. The beryllium metal was incompletely volatilized after six hours of passing dry HCl over the sample. It is believed that this difficulty can be overcome by a modification of the quartz boat. The boat is being prepared and further work will be done on the analysis.

SPECIAL PROBLEMS

(O. C. Simpson)

Quantitative Determination of Hydrogen in Na-K alloy. F. L. Belletire, E. C. Andrews, L. Reberak)

Work is being continued by the glass shop on equipment for the purification of Na-K alloy and on the reaction chamber. Purification trains have been completed for argon and oxygen. Considerable time and effort is being spent planning and executing the necessary housekeeping and safety steps required by the hazardous nature of the material being handled.

Construction (W. Litt, E. Hageman, E. C. Andrews)

Room 12-A, the hot storage area, is practically complete, including light and heat. An order has been issued to install a hoist and rail to facilitate loading and handling lead pots. Ryerson shops continue to work on the storage vaults.

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