

THE DOW CHEMICAL COMPANY

ROCKY FLATS PLANT  
P. O. BOX 2131  
DENVER COLORADO

August 17, 1960

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TRIP REPORT - BERYLLIUM SAMPLE EXCHANGE ANALYTICAL METHODS

The purpose of this trip to Cleveland, Ohio, on July 11 and 12, 1960 was to discuss the results of a beryllium metal sample exchange program and to consider analytical methods.

The meeting was held in the Cleveland Scientific and Engineering Center with Mr. E. S. Melick of the Brush Beryllium Corporation acting as host. Mr. Melick is in charge of the laboratories at the Elmira Ohio Plant and also he is responsible for the Quality Control of the chemical aspects of beryllium.

The following persons attended the meeting on July 11:

- E. S. Melick, Brush Beryllium Corporation
- A. Lavery, Wyman Gordon
- A. Szymanski, Beryllium Corporation
- G. B. Kriebel, Beryllium Corporation
- G. E. Simons, Beryllium Corporation
- G. W. Briscoe, U.C.N.C., Y-12, Oak Ridge\*
- S. R. Gille, U.C.N.C., Y-12, Oak Ridge\*
- H. C. Anderson, The Dow Chemical Company, Rocky Flats
- J. T. Byrns, The Dow Chemical Company, Rocky Flats
- L. C. Farrell, The Dow Chemical Company, Rocky Flats

\*Oak Ridge did not participate in the sample exchange program, however, they did analyze the exchange samples for BeO content. They are quite interested in the beryllium analytical methods and will participate in the next sample exchange program.

Results of the sample exchange program were discussed in the order which follows:

## 1. Assay for Total Beryllium

The only real discrepancy was the high results of Wyman Gordon which Mr. Lavery contended were in error and since had been rerun satisfactorily by his laboratory. Brush, Beryleo and Wyman Gordon are currently using the Empirical Volumetric Assay Method. Since the mean product level appears to be 98.4 %, it is their feeling that they are satisfactorily meeting Rocky Flats specifications. From the statistical study shown in Table I it appears that there are no significant differences between the assays reported by the respective vendors and those reported by Rocky Flats.

TABLE I

Comparison of Dow and Vendor Analyses on Beryllium - July 8, 1960

	<u>Average Assay (67 Samples)*</u>	<u>Average BeO (67 Samples)*</u>
Dow	98.44%	1.72%
Brush	98.42%	1.63%
	<u>Average Assay (62 Samples)*</u>	<u>Average BeO (30 Samples)*</u>
Dow	98.48%	1.85%
Beryleo	98.35%	1.75%

\*Includes all results subsequent to May, 1960 method revision (Requisition No. 6000) for Part Types 42, 47, 79 and 81.

Statistical information such as standard deviations, etc. for the volumetric method used by the vendors is, at best, very meager and not directly related to information which we have. It is believed that their standard deviation is approximately 0.4, a factor of two higher than our standard deviation for the gravimetric method.

## 2. BeO Determination

The BeO determination was discussed in detail. All installations have analyzed metal samples for BeO by the Bromine-Methanol Method, however, Brush contends that their experience indicates that the Bromine-Methanol Method usually gives higher results than does the HCl Volatilization Method which they prefer. It was pointed out by Mr. Melick that in either method of analysis any nonreactive beryllium compounds in addition to BeO would not be dissolved or

removed which could subsequently be determined as Fe tending toward erroneous results. Mr. Melick was concerned with the possibility that some compounds other than BeO remained after treatment with bromine-methanol. H. C. Anderson mentioned that electron diffraction examination of the residue remaining after bromine-methanol dissolution of a 4 gram metal sample showed only BeO to be present. He also pointed out that less than 5% of any other compound might be undetectable by this X-ray diffraction analysis.

In the sample exchange it was of interest to note that the Brush HCl volatilization and Rocky Flats bromine-methanol results were in close agreement in four out of five exchange samples. At Oak Ridge these samples were run by the KBrF<sub>4</sub> method for oxygen and the results agreed well with the HCl volatilization method.

The statistical study summarized in Table I shows Berylec reporting higher than Dow and Brush reporting lower on BeO. It was decided that in the next sample exchange both methods of analysis would be employed by Brush as in the past. Also, additional work would be attempted by Rocky Flats in an effort to identify any other materials which might be present in the BeO residue from the bromine dissolution.

### 3. Carbon Determination

The carbon determination was discussed briefly and it was indicated that all installations except Rocky Flats Plant employ the Leco combustion method. Rocky Flats reported ranges for the carbon determination for the five exchange samples, whereas, the other installations claimed that close agreement on carbon answers were obtained by their laboratories. This discrepancy might in part be reconciled by the fact that the other installations used crushed samples instead of chips such as Rocky Flats analyzed. Rocky Flats will soon have a Be mortar and pestle in operation and will be in the position to compare "as received" chips versus crushed chip samples.

### 4. Other Impurities

Other impurities were discussed briefly. Minor variations were noted on the reported values, however, no difficulty of major importance was noted. It was decided that in the future all installations would report values for elements customarily determined by their laboratories.

Wynan Gordon feels that they are off on their Fe value and they report nothing lower than 500 ppm for Mg. Brush thought that their values were low for Mg.

### 5. Sample Exchange

It was decided that the sample exchange program would be continued. Mr. Melick volunteered to prepare samples as follows:

One powder metal sample and four chip samples - each chip sample would be submitted as the original chip and as the pulverized chip for a total of nine samples. Assay and DeC determinations would be run on the powdered chips and metal powder. All liquidity determinations would be run on the crushed chips as well as the powder sample. Mr. Melick estimated that the samples should be ready to ship to Rocky Flats during the first week of August. When the samples are received by Rocky Flats they will be packaged and mailed to all participants without any additional treatment.

Brush will attempt to have sufficient samples so that any one of the samples could be used for any particular determination.

Brush will run DeC by HCl volatilization and bromine-ethanol dissolution. Oak Ridge will use the Leco oxygen analyzer as well as the ERP method. Rocky Flats will run DeC by vacuum fusion and the Bromine-ethanol Method.

### 6. Sampling

Both Brush and Wynn Gordon use a cyclone separator for taking samples from a jet. Wynn Gordon takes this sample from the second layer out.

Brylson's sample represents the inner part of a log.

### 7. Evaluation of Methods

The following methods were discussed to determine whether precision could be improved for many of the DeC and DeO.

Wynn's reviews:

Oxygen by neutron activation.

Brylson by improvement of the so-called commercial Brylsonator.

Both methods are being evaluated by the ERP AOC Group. E-12 is investigating the Brylsonator also.

Oxidation-reduction holds little promise.

Volometric Method (used by Brush).

Direct F<sup>-</sup> titration - being reviewed by Y-12.

8-OH Quinaldine - being reviewed by Y-12.

Arsenate titration used by Berylco.

BaBeF<sub>4</sub> - erratic results obtained at RPP. Y-12 values were high for this method.

### Tuesday, July 12

The Oak Ridge and Rocky Flats personnel were given a tour of Brush's Cleveland Plant. The Wyman Gordon and Berylco personnel were not invited on the tour since they are competitors of Brush. Mr. Donald F. Williams, in charge of Quality Control for the Cleveland Plant, was our contact at the Brush plant offices. Mr. Williams works very closely with his colleague, Mr. Melick, who is in charge of Quality Control at the Kinross Plant. In general, the major split is Mechanical Quality Control versus Chemical Quality Control.

We were given a tour through the Machine Shop area where some of Rocky Flats work was in progress. The only point of interest here is the fact that none of the lathes in use had hoods. Each lathe was equipped with a high velocity pickup tube (estimated air flow - 20 CFM) collecting the chips and powder at the tool contact point. In addition, it is of interest to note that no air sampling devices were in operation during the hour or more we were there. It is apparent that Brush has very little concern over beryllium contamination in the area. Mr. Paul A. Chupak, Foreman of the Machine Shop, showed us a shop sample collector which is a quart size cyclone separator type device. This sample collector is connected to the vacuum system and is placed at the tool point when in operation. He said the sample size to be collected had been increased ten times.

A tour of the Chemical Laboratories under Mr. A. James Stenhouse, Technical Manager of Chemical Laboratories, was of interest in that another opportunity was gained to observe the empirical volumetric assay. Again, all information obtained from Brush indicated that they are able to make this method work about twice as well as we can at Rocky Flats. Their daily factor data appeared quite favorable and they claimed to have very good success with the method. Statistical information, as available at Rocky Flats, was unavailable at Brush. It requires two hours per sample for duplicate analysis. The duplicates vary approximately 0.4%. Brush claims that B & A sodium fluoride must be used to get acceptable results.

We were later introduced to John G. Theodere, Technical Manager of the Materials Process Division. Of interest was the particle size determination performed by Brush. They have a Micromerograph and they have

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not found the particle size apparatus satisfactory for Be powders in as much as the column retains better than 50% of the material due to the fact that Be metal particles become electrically charged during the determination. The minimum particle size for this apparatus is in the 20-25 micron range. They also have had some experience with the Coulter counter, however, it is necessary to screen particles before selecting the orifice for a given particle size study.

Briefly, the density determination was observed. The air-water method was used employing a modified analytical balance for small items and a Model No. 3091 Toledo Speedweigh 50 pound capacity scale. No information was available on the sensitivity of the Toledo Speedweigh which is used for large pieces such as machined logs. This water was rust colored indicating a possible greater buoyant effect because of possible solids in solution.

Mr. Williams was very cordial and very helpful in introducing us to the people with whom we were to talk. He mentioned that in the event we had further questions in the future he would be most pleased to help us in any way.

H. G. Anderson

J. T. Myers

L. C. Farrell

HCA, JTB, LCF:fa