

DATE 2/19/54 INIT M.C.G.



Mr. E. C. McCarthy

February 15, 1954

MOUND LABORATORY-MONSANTO
Central File No. 54-2-80

RE: CONTROL-ANALYTICAL PHASE OF CERAMIC PROGRAM

A survey has been completed of the preparation, fabrication, recovery, and development phases of the ceramic project to determine the instrumentation needed for adequate control and material analysis. Estimates of the number of samples involved are presented in Table I, however these estimates may change considerably as the actual process evolves. Some of the instruments have been purchased. Plans are presented for integration of the new instruments with existing facilities and the needs of the program.

It is expected that the thermal column and electrolysis development areas will begin to require analytical data on hydrogen-deuterium gas mixtures about April, 1954. None of these samples will contain tracer quantities of tritium until sometime in 1955. Since present schedules call for a selection of final recovery process by October, 1955, this source of samples should be discontinued by that date.

The salt preparation, fabrication and recovery areas should be installed and operations started in these areas in October, 1954. There will be considerable increase in the number of samples at this time. Furthermore, the samples, in general, will be mixtures of the three isotopes. While the total quantity of material processed in the first few months may be relatively small, there will be an extensive sampling of material until some pattern of operation develops. Thus the control instrumentation must be ready for a moderately heavy load as soon as preparation lines and recovery lines are placed in operation.

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Re: Control-Analytical Phase of Ceramic Program Cont'd. 2/15/54

If a low temperature distillation facility is installed, it will not require any analytical services before about March, 1955. By October, 1955 extensive work on all parts (except possibly one) of the recovery development work should be concluded. If development work on one method is continued temporarily, it should be concluded by January, 1956. Closing of the development phases will materially decrease the load on the analytical instrumentation.

Three analytical methods have been considered for analyzing hydrogen-deuterium-tritium mixtures - mass spectrometry, nuclear magnetic resonance, and emission spectroscopy. In addition other techniques such as thermal conductivity and calorimetry may be of value for special analyses.

Considerable development work was done at KAPL and the National Bureau of Standards in developing an emission spectrographic method for mixtures of two hydrogen isotopes. The major difficulty encountered was in developing a light source which exhibited sufficient reproducibility and low memory effect to give reliable data. The results of these projects were transmitted to Hanford where the emission method was considered in addition to a mass spectrographic method. A decision to use the mass spectrographic method at Hanford was reached after some difficulty had been encountered with the emission source and the mass spectrograph had demonstrated its ability to analyze mixtures of hydrogen and tritium. Mass spectrographs have been used

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Re: Control-Analytical Phase of Ceramic Program Cont'd. 2/15/54

successfully for some time at Los Alamos to analyze mixtures of hydrogen and tritium. Project Whitney at Livermore has purchased a nuclear magnetic resonance instrument from Varian Associates and are investigating its use for hydrogen-deuterium-tritium analysis. Preliminary data (January, 1954) indicated that it would not be usable with gas samples, solid hydride samples gave broad responses which led to low sensitivity, and the detectability for deuterium was not high. The instrument showed promise for the analysis of liquid samples for hydrogen and tritium content. It appears that at no A.E.C. site is there any existing experience in the analysis of mixtures of the three isotopes, the nearest approach being at Los Alamos and Livermore where this problem is now being attacked.

In talking with B. B. McInteer, of Los Alamos, about the analytical problem it appeared that while no method has yet been developed, a mass spectrographic method appeared feasible. Such a method was being worked upon at Los Alamos and would probably depend upon a reproducible equilibration of the sample and calibration with known mixtures. Analysis of tritium-hydrogen mixtures was routine using a modified version of Model 21-201 Consolidated-Nier mass spectrometer.

Faced with this situation the decision was made to ⁽¹⁾ purchase two Model 21-201 Consolidated-Nier mass spectrometers, (2) budget money for a nuclear magnetic resonance instrument in the thermal diffusion production facility, (3) supplement existing calorimetry equipment, and (4) rely on existing equipment for any emission spectrographic work required. The nuclear magnetic resonance equipment may

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Re: Control-Analytical Phase of Ceramic Program Cont'd. 2/15/54

be ordered later if experience at Livermore and program developments at Mound indicate that it would be justified. Since our spectrographic laboratory has experience in analyzing mixtures of hydrogen and deuterium, this experience and equipment can be relied upon for analysis of samples of this type.

One of the mass spectrometers will be shipped from Pasadena, California, February 18, 1954 and the second should follow in about two weeks. The instrument will be installed in R-114. Mr. I. E. McCaffery, an engineer from Consolidated's Chicago office will assemble and place the instruments in operating condition. This will take about two or three weeks for each instrument. Mr. McCaffery will instruct Mound personnel in the use of the instruments. Following installation, both of the instruments will be modified according to plans originated at Los Alamos. This modification will be performed by the Technical Services Section while the instruments are in R-114 and will permit the instrument to sequentially scan and record the signal at each of six preselected mass points. The mass spectrometers will stay in R-114 and be used in conjunction with the development work until the construction in the operating areas is completed. One instrument will then be moved to the salt preparation area in the T Building and the second will be moved adjacent to the recovery column in the SW Building.

A survey has been made of the origin and frequency of samples originating in the preparation, fabrication, recovery and development areas and the types of analyses required. These data are summarized

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Re: Control-Analytical Phase of Ceramic Program Cont'd. 2/15/54

in Table I. The maximum number of samples is based on full-scale operation for one shift. In the event that two shift operation of any part of the project becomes necessary to handle quantities of material, the capacity for proper analytical control exists and it is assumed that the analytical work will all be done in one shift or additional shifts will be added to complete the work.

The T Building mass spectrometer will be used for checking purity of deuterium and tritium received, the purity of hydride material, analysis of scrap hydride before reforming, and any other gas analysis problems arising in conjunction with preparation or fabrication phases. The SW mass spectrometer will analyze gases liberated by amalgamation or electrolysis and will serve as a control on column feed, raffinate, and product. Since the column will operate on a three shift basis the latter mass spectrometer will be used during the same period. Because of the deleterious effects of repeatedly shutting off the instruments and the relatively long delays in reaching equilibrium after a shutdown, the instruments will be kept in standby operating condition when not being used to analyze samples. This will also apply to the non-operating shift periods for the mass spectrometer in the T Building.

The samples from the development phases requiring mass analysis will be taken as discrete samples in small gas carriers which can be attached directly to the manifolds of either of the mass spectrographs. From the estimated number of samples it appears that development samples

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Re: Control-Analytical Phase of Ceramic Program Cont'd. 2/15/54

can be handled in the regular day shift or certainly completed in the second shift.

Present planning is to place calorimeters for the ceramic project in the present calorimetry room in the T Building and take samples to that room. These samples will be lumped batches of tritium which have just been received, small analytical samples of hydride, and individual finished parts. All of these samples will be in doubly-sealed containers. New calorimeters capable of handling the lumped-batch containers will have to be constructed. Existing calorimeter #51 will be used for measurement of analytical samples. Two calorimeters now exist (54 and 58) which may be used for measurement of finished parts. Additional calorimeters will be required by about September, 1955.

The lithium metal received will be analyzed spectrographically to determine that it meets specifications. This analysis will be done using instrumentation available in the Research Division. A minimum of one month's supply of satisfactory lithium should be kept on hand at all times.

DLF:mg

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TABLE I

ANTICIPATED ANALYTICAL-CONTROL SAMPLES CERAMIC PROGRAM THROUGH 1956

<u>Mass Spectrometry</u>	<u>Samples/Shift</u>		<u>Total</u>	
	<u>Min.</u>	<u>Max.</u>	<u>Min.</u>	<u>Max.</u>
<u>T-Building Mass Spectrometer</u>				
1. - Verify purity of D ₂ received	1	2		
2. - Verify purity of T ₂ received	2	6		
3. - Analyze gas from hydride sample	1	2		
4. - Analyze scrap hydride prior to reforming	2	4	6	14
<u>SW-Building Mass Spectrometer</u>				
1. - Gases from electrolysis	1	8		
2. - Sample electrolysis gas storage	1	8		
3. - Gas from amalgamation	1	1		
4. - Column feed	1	2		
5. - Column raffinate	1	2		
6. - Column product	6	8	11	29
<u>Calorimetry</u>				
1. - Inventory of lumped batch (U traps)	2	4		
2. - Analytical sample hydride	1	2		
3. - Individual part assay	1	15	4	21
<u>Spectrographic</u>				
1. - Lithium purity	1	3	1	3
<u>Development Program (samples for Mass Spectrographs)</u>				
1. - Thermal columns	8	16		
2. - Electrolysis	8	16		
3. - Low temperature distillation	4	10	20	42