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In replying please address:

[redacted] 25X1

January 5, 1959

Dear Sir:

Enclosed is our proposal for an additional eight-month period of research on the development of a hydrogen generator (Task Order No. C). It is based on recent discussions with your technical representative.

In connection with the performance of the proposed research, it is our understanding that your technical representative will provide one balloon for use in the full-scale experiment and 120 pounds of sodium borohydride.

We are looking forward to working together on this proposed program, and would appreciate any efforts made to expedite consideration of this proposal, so as to minimize the lapse in the continuity of the activity in connection with this research. If you should have any questions with regard to our proposal, please let us know. Any inquiries of a contractual nature may be directed to [redacted] at Extension 159. 25X1

Very truly yours,

[redacted] 25X1

Vice President

[redacted] 25X1

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In Duplicate

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PROPOSAL

on

THE DEVELOPMENT OF A HYDROGEN GENERATOR

INTRODUCTION AND OBJECTIVES

On May 29, 1956, an effort under Task Order No. C was undertaken that was directed toward the development of an experimental hydrogen generator capable of generating enough hydrogen to provide 250 lb of lift at sea level (about 3,500 cubic feet of hydrogen) over a period of 45 to 60 minutes. Under Phase I of this research program, a literature search was performed; an analysis was conducted of various potentially applicable systems and a selection was made of the most desirable system compatible with the Sponsor's specifications for the generator of interest; a laboratory investigation was conducted on the characteristics of the selected hydrogen-generating reaction; and the preliminary design of an experimental full-scale generator was prepared. The system selected involved a reaction of cobaltous chloride hexahydrate (catalyst) with sodium borohydride. Laboratory experiments showed this reaction to be practicable for producing the required amount of hydrogen. The proposed design for the experimental full-scale generator consisted of a rubberized-fabric cylinder about 8 feet in diameter and 3 feet in height. The experimental unit was expected to contain about 560 gallons of solution corresponding to a depth of 1-1/2 feet; the additional height was to provide foaming space above the solution.

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Subsequently, under Phase II of this research program, the effort was directed toward the further development and evaluation of an experimental full-scale generator based on the preliminary design prepared under Phase I. The principal problem anticipated under Phase II was maintaining control of the total generation time in the course of scaling up from 1/100 scale (the size of the experiments involved in the laboratory studies) to full scale (as represented by the generator of interest). The cobaltous chloride - sodium borohydride hydrolysis is a heterogeneously catalyzed reaction. The chemical kinetics and, therefore, the total generation time of this reaction are strongly influenced by the distribution of the catalyst through the system. In view of the high cost of the chemicals involved, an experimental 1/5-scale generator was designed and prepared, to facilitate the investigation of the effects of catalyst distribution and rate of solution of the borohydride on the operation of an experimental full-scale generator. Based on the results of 1/10- and 1/5-scale experiments conducted in the experimental 1/5-scale unit, the design of the experimental full-scale generator was finalized. After this unit was prepared by a commercial fabricator, a handling technique and an operating procedure were developed for use in a full-scale generation experiment.

A full-scale experiment was carried out under field conditions on August 5, 1958; it was mutually considered to have been quite successful. This experiment also served as a demonstration of the one-man operation of the experimental unit, and involved balloon handling by your technical representatives. The proceedings were recorded on movie film; a detailed review and summary of the results of, and the conclusions drawn from, the proceedings were presented in a letter dated August 21, 1958, to your technical representative.

As a result of the full-scale demonstration, a review of the film, and several recent discussions, your technical representative has suggested that additional consideration be given to selected areas of study in an attempt to firm up further the operation of the unit. These areas are primarily concerned with the proper procedure to be used in operating the experimental full-scale unit at low temperatures, e.g., at about 35 F, and the adaptation of the procedure to operation on dry land. Also, there appears to be a need for certain modifications and/or additions to the experimental generator; these are directed toward minimizing the possibility of rupture as a result of the build up of excessive internal pressure. The program of research proposed herein and hereafter identified as Phase III describes an investigation directed toward achieving the above-indicated objectives.

DISCUSSION OF THE PROBLEM

Two general types of problems are anticipated with regard to the generation of hydrogen at near-freezing temperatures: those associated with the chemical reaction and those pertaining to the mechanical aspects of the experimental generator. The handling of the experimental unit on dry land and the prevention of rupture are problems which are associated mostly with the mechanical aspects of the experimental unit. The two categories of problems are discussed in some detail in the following.

The Chemical Problem

The Phase II studies were concluded with a full-scale experiment and demonstration of the entire hydrogen-generation procedure. The full-scale experiment was run at 82 F in a natural body of water. Based on data obtained previously, approximately 8 pounds of catalyst were used, and generation was successfully concluded in 26 minutes.

The chemical problem stems from the fact that excessively large quantities of catalyst are required in order to achieve total generation within 30 to 40 minutes, and possibly within 60 minutes, at low temperatures (i.e., at temperatures below 50 F); for example, based on the previously obtained data, it appears that at 37 F approximately 100 pounds of catalyst is required. Such a large catalyst requirement introduces three major problems: (1) in view of the limit of solubility of the catalyst in water, such an amount of catalyst is more than can be dissolved in water to provide approximately 5 gallons of catalyst solution; (2) the amount of catalyst solution needed would be too heavy to be handled by one operator as a single "package"; and (3) the initial surge of hydrogen that occurs when the catalyst solution mixes with the borohydride solution might be uncontrollable when such a large amount of catalyst is used.

Thus, it appears that the resolution of the chemical problem lies in decreasing the amount of catalyst required for cold-temperature operation (i.e., temperature < 50 F) while still generating the required amount of hydrogen within 60 minutes.

The Mechanical Problems

The mechanical problems of cold-temperature generation are concerned with (1) the rate of flow of the catalyst solution into the experimental generator, and (2) the pressure that is built up inside the experimental unit as a result of the substantially instantaneous reaction which occurs when the catalyst solution first contacts the borohydride solution.

The full-scale experiment, which was run at 32 F using a relatively small amount of catalyst, showed that the substantially instantaneous release or surge of hydrogen can cause noticeable pressure inside the experimental generator. Consequently, it is believed that the relatively large amount of catalyst which must be used at low temperatures may well increase the internal pressure beyond the strength capabilities of the experimental generator. The area of the top of the experimental full-scale unit is approximately 7,500 square inches, and the manufacturer has estimated that the unit can be expected to withstand pressures up to 1/2 psi satisfactorily. In order to prevent the build up of higher pressures, it appears that the flow of the catalyst solution into the experimental unit should be extended over a longer period of time. It is believed that this period of time can be reasonably reliably estimated on the basis of small-scale generation experiments. After the entry time was estimated, it would be necessary to investigate the size and number of entry holes needed in order to attain the desired rate of catalyst flow into the experimental full-scale generator.

Even with reasonable control of the initial surge of hydrogen, it is quite possible that excessive internal pressure might be built up as a result of blockage of the outlet tube, for example, as a result of twisting. Consequently, a relief valve could be used advantageously. A satisfactory valve for this purpose would have sufficient capacity to permit the passage of a large amount of gas at 1/2-psi maximum pressure. The accuracy of relief valves at such a low pressure, and particularly of those with large capacities, is questionable if it is necessary to keep the valve simple, as is the case here. However, it appears that a suitable valve might be prepared for the experimental generator rather simply - by providing for the pressure to act against the weight of a part rather than against a spring. Thus, such a valve might include a simple lid-type part the weight of which was designed to permit the relief of gas at about 1/2-psi maximum pressure. This type of relief valve might readily be incorporated in an appropriate connector (to connect the outlet of the experimental generator to the filler tube of the balloon), the design and preparation of which have been included in the consideration described herein, at the request of your technical representative.

The problems of handling the experimental generator in an operation conducted on dry land can only be estimated at this time. However, it currently appears that dry-land operation of the experimental full-scale generator is quite feasible.

PROPOSED METHOD OF PROCEDURE

Chemical Problem

From the chemical point of view, the Phase II studies can be summarized in terms of the relationship between amount of catalyst and temperature, particularly at the upper part of the probable operating-temperature range. To achieve satisfactory generation, all other factors being fixed, it is necessary for the operator to add the proper amount of catalyst; this is based on the temperature of the borohydride solution. This part of the proposed program would be directed toward establishing the relationship between amount of catalyst and temperature at near-freezing temperatures, so as to provide for generation within 60 minutes in low-temperature operations.

As currently contemplated, the proposed effort in connection with the chemical problem would be directed toward reducing the amount of catalyst required, so that it could be dissolved in water to provide 5 gallons or less of solution; and toward minimizing the surge which occurs at the start of generation. Five gallons of saturated cobaltous chloride hexahydrate - water solution, at about 32 F, contains approximately 40 pounds of catalyst; this is an estimate based on consideration of the available data. Emphasis in the proposed effort would be placed on attempting to develop a procedure for generating hydrogen in the experimental full-scale unit using this amount or less of catalyst.

1/10-Scale Experiments

As currently planned, two approaches would be used in investigating the chemical problem. In the first, the amount of catalyst would be reduced to the equivalent of 40 pounds per full-scale generation, and generation-time data would be obtained as a function of initial borohydride-solution temperature. If generation times of 60 minutes or less were obtained at low temperatures (such as 35 F), it probably would not be necessary to perform any additional research on the chemical problem. On the other hand, as we expect from our knowledge of this system, the low-temperature limit in the first approach (i.e., the lowest temperature at which a 60-minute generation time is obtained using 40 pounds of catalyst) may occur at about 40 F or slightly higher. Then, it would be necessary to attempt to increase the generation rate at the near-freezing temperatures in another manner.

In this second approach, the borohydride concentration would be increased by decreasing the amount of water used to prepare the borohydride solution. (In terms of the experimental full-scale unit, this means that less than 560 gallons of water would be used to dissolve the borohydride.) Thus, less catalyst would be required to perform the desired generation within 60 minutes.

Previously obtained data have shown that, at about 65 F, reducing the catalyst concentration by a factor of two increases the total generation time by about 50 per cent. If this relationship holds at low temperatures, then in order to perform the generation within 60 minutes using 40 pounds or less of catalyst, the borohydride-solution temperature probably cannot be below about 40 F. It is probable, therefore, that both approaches would merit investigation, in an attempt to adjust the operating procedure appropriately for application at low temperatures.

In the proposed study of these two approaches, it is currently anticipated that ten to twelve 1/10-scale generation experiments would be conducted; these would be aimed toward obtaining more data on the catalyst-temperature relationship for cold-weather generation. Five or six of these would be run in the approximate temperature range of 35 to 45 F using the "standard" borohydride concentration, namely, 100 pounds of borohydride per 560 gallons of water. The amount of catalyst used would be selected in an attempt to achieve a total generation time of 60 minutes or less. This part of the proposed program would provide data on (1) the catalyst-temperature relationship appropriate for cold-weather generation, and (2) the low-temperature limit for generation within 60 minutes at the "standard" borohydride concentration.

If, as expected, the low-temperature limit is found to be above 35 F, then five or six 1/10-scale runs would be carried out within the same temperature range (35 to 45 F) at an increased borohydride concentration. Examination of our available data indicates that a borohydride concentration requiring about 350 gallons of water for cold-weather full-scale generation would probably be appropriate. This concentration would lead to a total temperature rise of about 93 F in the course of the reaction, as compared to that of about 58 F in the present system involving the "standard" borohydride concentration. Under cold-weather conditions, this temperature rise would not be expected to present any problems. The results of this part of the proposed program would also provide information pertinent to the catalyst-temperature relationship for cold-weather operation.

In an attempt to obtain an adequate understanding of the surge problem, it is proposed to scale down the outlet on the experimental small-scale generator; this would facilitate observation of any pressure build up as a result of the surge during the small-scale generations. It is also currently planned to conduct a few laboratory-size experiments to examine the substantially instantaneous nature of the initial reaction between the catalyst and sodium borohydride at about 35 F. The purpose of these experiments would be to recheck the quantitative data obtained in earlier studies at higher temperatures. These experiments would also yield measurements of the heat of this reaction; these data would help in gaining a better understanding of the initial temperature rise associated with the reaction.

It is currently contemplated that, after the above-described research was conducted, sufficient information would be available to provide a basis for full-scale generation under low-temperature conditions. However, it is considered worth while to run one or two 1/10-scale experiments at an available field site before conducting the full-scale experiment at that site. These small-scale runs would serve to confirm the results obtained in the previously described experiments and also to increase the reliability of the conclusions drawn.

Full-Scale Study

Our previous studies have shown that the small-scale runs (i.e., the 1/10- and 1/5-scale experiments) can be used to establish the reliability of this system with a fair degree of accuracy. However, sufficient reliability with regard to full-scale operation can be established only on the basis of a full-scale experiment; the many parameters which enter into the scaling-up problem cannot be entirely accounted for except as they manifest themselves and/or interact in the course of a full-scale generation.

It is currently planned that one full-scale experiment would be conducted with the experimental unit on dry land, at a temperature of 35 to 40 F. Prior to this, any modifications that were investigated as described below and were considered mutually to be satisfactory would have been incorporated in the experimental full-scale unit, within the limits of the time and funds provided. The catalyst and the borohydride concentrations used would be selected on the basis of the results of the small-scale runs. As in our previous full-scale effort, provisions would be made to assist your technical representative in collecting the hydrogen in a balloon. However, to facilitate the performance of this experiment and for purposes of economy, it is currently planned that this run would be conducted in a manner that would not provide a one-man demonstration of the experimental unit; it appears expedient for us to plan to provide a fair amount of assistance in setting up the experiment.

Mechanical Problems

The proposed research pertaining to the anticipated mechanical problems would involve effort directed toward investigating several areas of interest; these include changes in the experimental 1/5-scale generator, design and preparation of an experimental combined connector and relief valve, control of the catalyst flow rate, modification of the experimental full-scale generator, and exploration of the handling characteristics of the experimental full-scale unit when operated on dry land. The proposed effort, as currently contemplated, in each of these areas is outlined below.

Changes in the Experimental 1/5-Scale Generator

During the effort under Phase II, the experimental small-scale generator developed some serious leaks. Under Phase III, these would be repaired so as to permit additional experiments, as described above, to be conducted. Further, in order to scale properly the outlet of this unit to that of the experimental large generator, some modification effort would be performed.

Also, there is a possibility that modification of the catalyst-ring holes may be necessary. It is not expected that excessive internal pressures would be built up in the experimental small generator during the course of the above-described experiments. However, if this should happen, then it is anticipated that modification of the catalyst ring would be investigated in an attempt to slow down the rate of catalyst entry. The general procedure that would be followed is outlined below under "Study of the Control of the Catalyst Flow Rate".

Consideration of the Experimental Connector With Relief Valve

During the previous full-scale experiment, the temporary crude connector that was prepared in the field failed and prevented proper filling of the balloon. Observations of the proceedings suggested that a satisfactory connector should probably be rigid and also capable of resisting the effects of moisture and of temperatures of about 150 F. Discussions of the proposed connector with your technical representative have resulted in a preliminary decision to investigate the preparation of this item from a plastic type of material. As currently envisioned, the proposed connector would be fastened to the experimental-generator outlet and would provide a horizontal opening to which the balloon feeder tube could be connected.

After some consideration of the problem of relieving excessive internal pressure in the experimental full-scale generator, it appears worth while to investigate the possible usefulness of a lid of predetermined weight as a relief valve. Thus, a vertical arm might be incorporated in the proposed connector, and a properly weighted lid might be placed at the top of the arm. Such an experimental connector, when attached, would look like a T placed sideways on the experimental generator; the balloon filler tube would be attached to the bottom end of the vertical arm so as to provide for passage of the gas under conditions of tolerable pressure.

The action of this type of relief valve represents one of the oldest and simplest methods for relieving pressure. It is particularly suitable when the critical pressure is low. The primary problem associated with this type of valve is the prevention of sticking or cocking of the lid. However, it is believed that a satisfactory experimental relief valve of this type could be prepared for this application.

In addition to the proposed design effort which is generally outlined above, it is anticipated that it would be necessary to investigate methods of preparing the experimental connector and relief valve. At present, it appears practicable to consider a method involving the preparation of a suitable form, coating of the form with a suitable plastic, and curing the plastic in situ. If an investigation of this technique does not provide satisfactory results, other methods of preparation would be considered.

Study of the Control of the Catalyst Flow Rate

The small-scale experiments described above are expected to provide a basis for estimating an appropriate catalyst-entry period of time for the experimental full-scale generator, so as to minimize difficulties associated with the initial surge of hydrogen. After this time interval is estimated, experiments would be conducted with the proposed amount of fluid and different-sized holes, in an attempt to establish a proper catalyst-solution flow rate. Most of these experiments would be run in some kind of piping in which appropriate holes were drilled. The amount of fluid flowing from the holes during different time periods of operation would be measured. On the basis of the results obtained, an estimate would be made of the appropriate size and number of holes needed in the catalyst-solution ring of the experimental full-scale generator.

Modification of the Experimental Full-Scale Generator

Subsequent to the above-outlined study of the catalyst-solution flow rate, the experimental full-scale generator would be modified to provide an appropriate size and number of holes in the catalyst-solution ring. Currently, the only sizeable opening in the experimental unit is the outlet. It is possible to manipulate the experimental generator so that each hole in the catalyst-solution ring would be accessible through the outlet opening; in this way, the different holes could be covered or reduced in size systematically. This procedure appears to be somewhat complicated; however, it is believed that it would be much more practical than would that of slitting the ring, adjusting the holes, and then repairing the slit.

Exploration of the Handling Characteristics of the Experimental Full-Scale Unit on Dry Land

Although the proposed full-scale experiment outlined above would not constitute a demonstration of a one-man operation of the experimental unit, it appears worth while, on the basis of previous experience, to provide for investigating the handling characteristics of the experimental generator in connection with dry-land operation. The manufacturer has assured us that the experimental full-scale generator can be placed on dry land and filled with the necessary amount of water, without rupture occurring. This estimate has been substantiated at least in part by our operation of the experimental

1/5-scale generator in this fashion, in connection with the previously conducted effort under Task Order No. C. It is estimated that, under these conditions, the forces acting on the experimental small unit were as great as would be those on the experimental full-scale generator; both have been prepared from the same materials and in the same manner.

This proposed exploration would involve conducting probably more than one run in the modified experimental full-scale unit on dry land, without the borohydride and cobaltous chloride being used. A cursory investigation would be made of a suitable method of filling the experimental full-scale unit with an appropriate amount of water (for the borohydride solution). No provision has been made herein for effort directed toward modifying the experimental full-scale generator in an attempt to alter its filling characteristics. It is expected that, in the course of the proposed effort, some feel for the difficulties involved, if any, would be obtained, and that a resolution of such difficulties would be fairly straightforward.

Of particular importance in the proposed "dry" runs would be the checking of the time interval required for the flow of an appropriate amount of simulated catalyst solution through the catalyst ring. If the rate of entry is not satisfactory, it would be necessary to modify the holes in the ring accordingly. Some need for modification of this sort is to be expected since the area of the holes in the flexible ring material is difficult to estimate, and to provide for.

Another aspect which would be investigated would be the problem of attaining the necessary amount of low-temperature water during the time of the year when the ambient air temperatures might be relatively warm. It is estimated that the full-scale experiment would be run at a time when the ambient temperatures would be considerably above 40 F. It is currently contemplated that the full-scale run would be conducted at a borohydride-solution temperature of 35 to 40 F. Consequently, it would be necessary to provide a relatively large amount of low-temperature water.

At the present time, we believe that the water could be cooled readily by using an auxiliary water tank and ice. A suitable tank, costing approximately $150, would consist of a 12-foot-diameter plastic liner supported by a wire fence. (The diameter of the experimental full-scale generator is 8 feet.) The proposed procedure would involve filling the tank with ice water, and then placing the experimental generator in the tank and filling it with ice water. It appears that it would be readily possible to maintain the experimental-generator water at low temperature while the necessary associated equipment was being set up. When the experiment was to be started, the water in the tank could be released, and the experimental generator would be left standing in the dry tank. The supporting fence of the water tank could then be removed, and the generation process could be carried on. If this method was found to be unsatisfactory, others would be investigated.

On the basis of recent discussions, it is our understanding that one balloon for use in the full-scale experiment and an additional

120 pounds of sodium borohydride would be furnished by your technical representative. We currently have on hand approximately 120 pounds of borohydride. The above-indicated additional amount is based on the requirements of the currently planned schedule of experiments; this includes approximately fourteen 1/10-scale experiments (about 10 pounds per experiment) and one full-scale experiment (about 100 pounds per experiment). It is expected that your technical representatives would be present during the full-scale experiment.

After the conclusion of the full-scale experiment, the experimental generator(s) would be provided to your technical representative for further evaluation.

REPORTS AND LIAISON

Your technical representative would be kept informed of the progress of the research program by informal monthly letter reports. These reports would be supplemented by meetings with your technical representative. At the conclusion of the proposed research period, a summary report would be prepared that would include a description of the experiments conducted and the results obtained.

Also, the operating procedure for the experimental full-scale generator that was prepared under Supplement No. 4 to Task Order No. C would be revised to reflect any appropriate changes resulting from the effort proposed herein.

DURATION AND ESTIMATED COSTS

It is proposed that the contract provide for an additional eight-month period of research, with an estimated additional appropriation of $15,317, including the additional fixed fee. A general breakdown of the estimated appropriation increase is attached.

THE CONTRACT

The proposed contract would be a period-basis research agreement, consistent with our current contractual arrangements and providing only for a fixed period of research leading toward the objectives outlined above.

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