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SUMMARY REPORT

ON

TASK ORDER NO. C

(PHASE II)

December 31, 1958

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INTRODUCTION

This report describes the underlying research, and the design and method of operation of a generator for hydrogen gas that was developed under Task Order No. C to meet specified operational requirements and conditions. The research period covered by this report was from June 28, 1957, through December 31, 1958.

Two reports describing the effort performed under Task Order No. C were issued previously. A topical report, titled "An Analysis of the Proposed Hydrogen-Generator Development", dated September 7, 1956, discussed various means of meeting the specified requirements for a hydrogen generator. It was necessary for the generator to produce 3,500 cu ft of hydrogen gas (sea level) at an isolated site by a controlled reaction in less than 1 hr. Since only surface water would be available at the point of generation, all chemicals and equipment would have to be transported, assembled, and/or handled by a single operator; therefore, the total weight would have to be less than 500 lb, separable into units of no more than 50 lb each. It was concluded in the analysis described in the above-mentioned report that a solution of sodium borohydride could be hydrolyzed to produce the required amount of

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hydrogen, and that a generator could be designed to meet the requirements.

The second report, "Summary Report on Task Order No. C", dated January 28, 1957, described the results of small-scale laboratory generation studies, which constituted Phase I of this research program. The largest of the small-scale tests run produced 1/100 of the hydrogen required for the desired full-scale generator. These experiments determined the rate of hydrogen evolution at various temperatures and involved several different concentrations of sodium borohydride and catalyst. A number of different catalysts for controlling the hydrogen evolution were considered, and cobaltous chloride hexahydrate was studied extensively after it was selected as the preferred catalyst. The experimental results from that phase of the study established a preferred method of setting up and carrying through the hydrogen-generating reaction, to meet the specified operating time of less than 1 hr over a range of temperatures. It was then possible to describe a proposed design for a full-scale generator.

It was next necessary to study hydrogen generation on a larger scale, as described herein, because of other operating variables introduced as the scale of operation necessarily was enlarged. As anticipated, an appreciable period of time was required at the start of the large-scale reaction for adding the large volume of catalyst solution and for mixing it with the reaction solution. The rate of loss of reaction heat to the surroundings was also different in the large generator; this

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changed the pattern of temperature rise in the reaction solution, and affected the total time of generation.

The experiments described in this report, and identified as Phase II of this research program, were concluded with one final full-scale operating demonstration in a prototype generator. This demonstration was preceded by a number of preparatory 1/10- and 1/5-scale tests in a 1/5-scale generator. These preparatory tests were made at less than full scale, to permit reduction of the over-all cost of the chemicals used in this phase of the Task Order No. C research program.

SUMMARY AND CONCLUSIONS

The generation of hydrogen in the full-scale demonstration experiment was completed 26 min after the addition of catalyst solution to the reaction pool. This was well within the specified maximum limit of 60 min for hydrogen generation.

Preparations for hydrogen generation were successfully accomplished by one man, as specified. It appears that it would be desirable to shorten the time required to prepare the generator for operation. In particular, the natural flow of water into the generator through ports below the water line requires about 55 min. Consideration might well be given to shortening the filling period by providing for and utilizing a more positive method of filling the unit with water.

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The full-scale demonstration was conducted with surface water at 82 F, which is at the upper end of the anticipated temperature range. Operation at the lower end of the temperature range near 32 F would present an additional problem of handling the increased amount of catalyst necessary to compensate for the retarding effect of low temperature. The qualitative principles of modified procedures for operating at low temperature to meet the specifications are understood. Several small-scale tests and another full-scale demonstration at low temperature are recommended to establish quantitatively the effects of the procedural modifications for operation at low temperatures.

DESIGN OF PROTOTYPE FULL-SCALE
GENERATOR

The hydrogen generator was constructed from neoprene-coated nylon fabric in the form of a cylinder about 8 ft in diameter and 30 in. high. The generator was supported by 16 vertical stays inserted in pockets in the cylinder wall. The stays were held in their vertical position by an inflated double-walled section of the upper half of the cylinder wall.

Figure 1 is a photograph of the inflated generator standing on its stays on dry ground. The generator was designed to operate also while floating in a pool of water. Under this condition, the inflated air chamber supported the generator on the water surface, and the stays served to keep the wall section

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below the chamber extended in its cylindrical form while the generator was being filled with water and during the period of gas evolution.

The vertical dimensions of the generator and its air chamber were so chosen that the amount of water required by the generator entered through ports below the water line while the generator was floating free in the water. The two water-entry ports with open-end fabric sleeves permanently attached are seen in the foreground of Figure 1. Not visible in the photograph is a loop at the center of the fabric bottom to which was attached a weight which pulled the bottom downward during the filling operation. Also not visible is the valve through which the air chamber was inflated by a hand pump. Extending above the edge of the air chamber on the top of the generator was a short open-end tube, stiffened by a flanged upper edge; this provided access to the interior of the generator above the water line. This opening shows in the left foreground of Figure 1 and served several purposes. After the water entered and was isolated by tying off the sleeves on the water-entry ports, the solid chemicals required to make up the generator solution were dumped through the top opening. The same opening could serve for inspection of the solution to permit determining whether the added solids were completely dissolved. The solution could be inspected by dipping out a small sample and inspecting it for suspended solids, but this precaution was not usually essential. It was critically important, however, to measure the solution temperature accurately

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by inserting a thermometer through the opening, because the temperature determines how much catalyst must be added to generate the hydrogen in 1 hr or less. Finally, the flanged top opening served to attach the balloon to the generator. The end of a flexible tube leading to the balloon was pulled over and tied below the flanged edge of the open tube before the catalyst solution was added to the generator.

The catalyst was added as a solution from the catalyst reservoir, most of which shows in Figure 1 as a collapsed doughnut-shaped ring of fabric cemented in a concentric position on the top cover of the generator. The capacity of the reservoir was about 5 gal, which was sufficient to contain all of the catalyst solution under most operating conditions. The common wall between the ring-shaped reservoir and the generator top was pierced by 20 equally spaced 1/8-in.-diameter holes, through which the catalyst solution flowed and was distributed onto the surface of the reaction pool. At the right side of Figure 1 is a 3/4-in.-diameter pressure-equalizing tube bent into a semicircle and connecting the free space above the reaction pool to the catalyst reservoir. This acted to prevent the initial pressure, built up by the generated hydrogen, from retarding the flow of catalyst solution from the reservoir into the reaction pool.

Lying diagonally across the top of the generator in Figure 1 is a fabric filling funnel whose flexible spout was permanently attached to the catalyst reservoir at a point in the foreground which is concealed by the projecting air chamber.

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The upper open-end body of the funnel was large enough to be pulled downward over the top of a container holding the catalyst solution. The funnel fabric would be gathered tightly about the waist of the container, which would then be inverted so as to pour the catalyst solution through the spout into the catalyst reservoir. After transfer of the catalyst solution was completed, the spout was sufficiently long so that it could be tied off to prevent the escape of generated hydrogen gas.

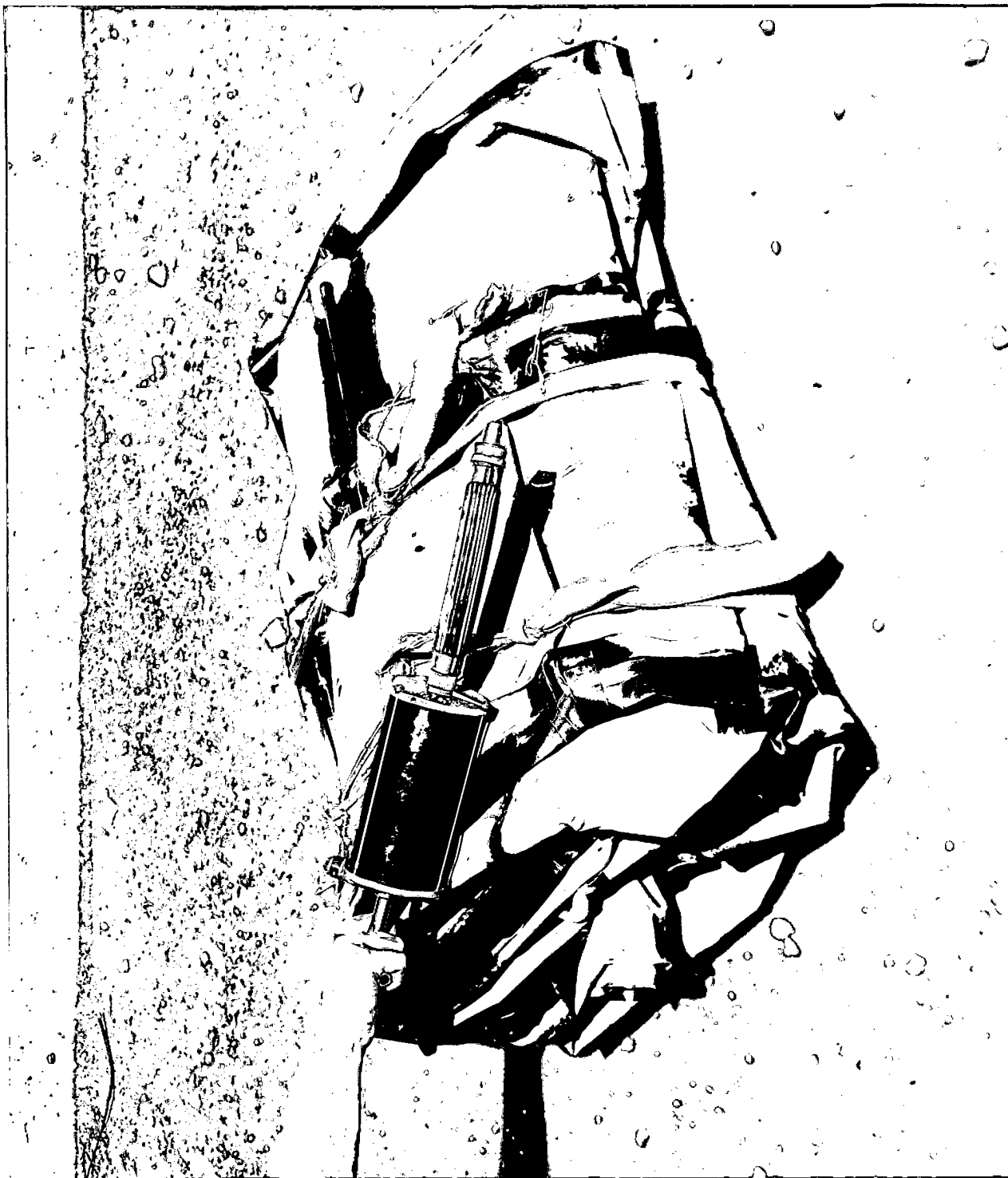
Careful inspection of the photograph shows four fabric loops spaced at 90-degree intervals around the top edge of the generator. These were provided for convenient attachment of ropes to the generator. The ropes might be necessary in order to anchor the balloon while it was being filled, or to prevent the floating generator from drifting while the operator was manipulating the balloon.

Figure 2 is a photograph of the deflated generator packed for transport. The package measures 15 x 24 x 35 in. and weighs 38 lb. The inflating pump is shown held above the generator to indicate the relative size of these two items.

A drawing of the full-scale generator is included in this report as Appendix I.

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FIGURE 2. DEFLATED HYDROGEN GENERATOR PACKED FOR TRANSPORT AND INFLATING PUMP

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MATERIAL REQUIREMENTS FOR HYDROGEN GENERATION

The materials and equipment indicated below are required to generate approximately 3,500 cu ft of hydrogen in the Task Order No. C generator at an isolated site, as specified, at which there would be only a pool of surface water sufficiently large to float the generator. The items of equipment and materials are listed in the sequence in which they would be needed in order to carry through the generating procedure.

Materials ListFor Generation of Hydrogen

- (1) Hydrogen generator
- (2) Air pump, capacity about 70 cu. in. per stroke
- (3) Fabric bag for weights, with drawstring, about 300-cu.-in. capacity
- (4) Weights, 15 lb, as rocks or sand, probably at site
- (5) Rope, 1/4 in., about 40 ft
- (6) Tools: knife, pliers, dipper, and funnel
- (7) Solution container, 5-gal plastic narrow-mouth bottle with stopper
- (8) Sodium hydroxide, 2 lb, in moisture-proof, unbreakable, nonmetallic package
- (9) Rubber gloves, shoulder length

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- (10) Sodium borohydride, 100-lb of crystals in five 20-lb moisture-proof, unbreakable, nonmetallic packages
- (11) Thermometer, all-metal dial type, 32 to 120 F/1 F
- (12) Chart showing weight of catalyst required at various temperatures of sodium borohydride solution
- (13) Cobaltous chloride hexahydrate catalyst, 46 lb of crystals; in individual moisture-proof, unbreakable, nonmetallic packages, 3 of 10 lb each, 2 of 5 lb each, 5 of 1 lb each, and 2 of 1/2 lb each.

For Collection of Hydrogen

- (1) Balloon and gas connection tube
- (2) Tarpaulin
- (3) Sand bags
- (4) Ropes
- (5) Adapter and clamp for connecting balloon to generator.

OPERATING INSTRUCTIONS

Procedure

The following stepwise procedure was carried through by one man in one full-scale demonstration run, and is recommended for field operations:

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- (1) Select a site for operation that provides a water-pool depth of about 4 ft near the bank.
- (2) Unpack the hydrogen generator close to the water edge. Unfold the generator pack in a position to favor easy inflation, with the air valve accessible.
- (3) Attach the air pump to the air valve. Inflate the air chamber (with about 700 strokes of the pump) until the generator stands firmly on its supporting stays, as shown in Figure 1.
- (4) Detach the air pump and close the air valve.
- (5) Select about 15 lb of rocks or sand at the site. Place in the fabric weight bag and tie the mouth of the bag. The proper weight of rocks may be estimated by comparison with an equal weight of packaged catalyst crystals.
- (6) Tip the generator and tie the weight bag to the center of the bottom with the straps provided.
- (7) Attach a rope to the generator loop so that the floating generator does not drift. Anchor the other end of the rope on shore.
- (8) Slide the generator into the water, and push it away from the bank until it floats freely.
- (9) Push both of the water-inlet tubes into the generator and lay them along the inside of the generator wall.

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- (10) Tilt and resettle the generator in the water several times to dislodge any air bubbles trapped beneath the bottom of the generator. Make certain that the weight bag is hanging free from the bottom. Recheck the position of the water-inlet tubes along the inside wall of the generator as necessary during the filling period of about 55 min.
- (11) While the water is running into the generator, spread the tarpaulin on the ground, unpack the balloon, and lay it out on the tarpaulin; be careful to avoid damage to the balloon fabric. Prepare the balloon auxiliary equipment for use during balloon inflation.
- (12) After the generator has filled to the proper level, with the bottom fully extended by the attached weight, pull out both of the water-inlet tubes and knot each to shut off the water inlets.
- (13) Open the 2-lb package of sodium hydroxide flakes and pour them into about 1 gal of water in the 5-gal plastic container. Shake briefly to dissolve, and pour the solution slowly through the open gas exit into the generator pool. Induce some mixing by tipping the generator a few times.
- (14) Add the sodium borohydride crystals to the generator pool by opening and emptying one package at a time into the open gas exit.

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- (15) Check the pool for undissolved crystals by dipping out a small sample from the center bottom and inspecting. Stir with the dipper or tilt the generator to speed up the mixing and solution, if necessary.
- (16) Measure the temperature of the solution by inserting the thermometer into generator through the open gas exit.
- (17) Determine weight of the catalyst required by referring to chart (Table 1).
- (18) Make up the catalyst solution in the 5-gal plastic container. First, fill the container about one-third full of water, add some of the catalyst, and shake to dissolve. Add more water (corresponding to about one third the volume of the 5-gal container) and the remaining catalyst, and shake again. Fill the container to the top with more water. Insert the container stopper, and shake or roll the container to mix the solution. Set the container with the solution upright near the generator at the water edge.
- (19) Attach the balloon to the gas exit of the generator and arrange the connecting tube so that the balloon will start to fill without attention while the catalyst solution is being added to generator.

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TABLE 1. AMOUNT OF CATALYST REQUIRED FOR
HYDROGEN GENERATION AT TEMPERA-
TURES ABOVE 50 F

Weight of Sodium Borohydride: 100 lb

Weight of Sodium Hydroxide: 2 lb

Volume of Catalyst Solution: 5 gal

(made up on basis of values
given below)

Volume of Generator Solution: 560 gal

Generation Time: Approximately 40 min

Initial Temperature of Solution, F	Weight of Catalyst, lb
90	6
88	6-1/2
85	7-1/2
82	8-1/2
80	9-1/2
78	10-1/2
76	11-1/2
74	13
72	14-1/2
70	16
68	17-1/2
66	19-1/2
64	21-1/2
62	24
60	27
58	30
56	33-1/2
54	37
52	41-1/2
50	46

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- (20) Support the full container of catalyst solution upright on the top edge of the floating generator, remove the stopper, and pull the open end of the filling funnel over the neck of the container and down around the body of the container.
- (21) Tip the container and pour the catalyst solution through the funnel into the catalyst reservoir on the top of the generator.
- (22) Hydrogen generation will start as soon as the catalyst solution starts to run into the generator pool from the reservoir. Quickly tie off the flexible spout of the filling funnel to prevent any loss of hydrogen.
- (23) Attend the balloon during the filling operation. No further control of hydrogen generation is needed.

Discussion of Operating Procedure

The foregoing procedure was developed from the experimental results of 19 experiments which measured the time necessary to generate 1/10 or 1/5 of the required amount of hydrogen in a 1/5-scale generator. One full-scale test was run in the prototype full-scale generator. With the initial temperature of the generator pool at 82 F, the addition of 8-1/2 lb of catalyst in 5 gal of solution resulted in a generation time of 26 min. This was

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regarded as a good agreement with the generation time of about 40 min predicted from the tests on a smaller scale, inasmuch as a minimum generation time was desired.

The amounts of catalyst required are given only for temperatures of 50 to 90 F in the description of the operating procedure, because the larger amounts which are apparently required for lower temperatures down to 36 F exceed the solubility of the solid catalyst in 5 gal of catalyst solution. Also, a larger volume of catalyst solution would be difficult for one operator to handle. There are several possibilities for an acceptable modified operating procedure at low temperatures that would yield the desired generation time of less than 1 hr. It appears that the best modification can be selected only after some additional experimental study is conducted at 1/10 scale in preparation for a second full-scale experiment.

The type of modifications needed to provide for low-temperature operation is described later, following a summary of the technical principles of the hydrogen-generating reaction in the next section. It may be advisable to include some discussion of the technical principles in an operating manual for the generator. Therefore, the principles are described in the following primarily in a qualitative sense. The quantitative and mathematical discussion included here could be deleted for purposes of the operator's manual with only minor changes in the text.

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TECHNICAL PRINCIPLES OF HYDROGEN GENERATIONPrimary Variables of the Reaction

Hydrogen gas is released from a sodium borohydride (NaBH_4) solution by a chemical reaction between the borohydride ion (BH_4^-) and the water. This reaction does not occur unless there is a catalyst present in the solution. After the catalyst is uniformly mixed in the solution, the rate at which hydrogen is generated depends primarily on three variables:

- (1) the temperature of the solution
- (2) the concentration of the catalyst
- (3) the concentration of the borohydride.

It is the combined effect of these variables that determines the speed of the reaction at any moment, and thus the time required for the reaction to be completed.

The three variables listed above differ in their individual effects on the rate of hydrogen generation. The generation rate increases with rising temperature, and doubles for a rise of about 15 F. The generation rate also increases in proportion to an increase in the catalyst concentration, so that doubling the concentration of the catalyst would double the generation rate, if both the temperature and the borohydride concentration remained the same. Finally, the borohydride concentration has the least effect on the generation rate. Increasing the borohydride concentration by almost 50 per cent reduces the total generation time by less than 10 per cent.

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During the time that hydrogen is being generated, the temperature and the borohydride concentration are continuously changing, and the hydrogen generation rate changes also. The reaction which produces hydrogen releases heat and consumes the borohydride. Most of the heat remains in the reacting solution and serves to raise its temperature, although some heat is lost continually to the surrounding water and to the air outside the generator.

The total reaction time is thus greatly affected by the initial temperature of the solution and by the amount of water in the generator. Both influence strongly the range of temperature that is covered during the reaction, and must be carefully determined in order to control the reaction time. If the amount of water added to the generator is intentionally reduced, then a much shorter generation time is possible. The concentrations of borohydride and catalyst are increased by dissolving fixed weights of these solids in less water; also, the temperature rise occurring during the generation reaction is larger in proportion to the reduction in the volume of the solution. This occurs because, regardless of the amount of water used, approximately the same amount of heat is released in the complete reaction, and it heats the smaller amount of solution to a higher temperature. For example, the maximum temperature rise for 100 lb of borohydride in 560 gal of water is 58 F above the initial temperature of the solution. Using only 350 gal of water would lead to a temperature rise of about 93 F above the initial temperature.

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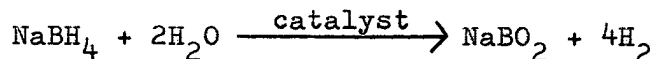
Mathematical Expression for the Rate of Reaction

According to the conventional methods of expressing reaction rates, the following rate equation was derived from the experimental data obtained in isothermal bench-scale tests, and was used to calculate the qualitative comparisons in the preceding section:

$$-\frac{d}{dt}(\text{NaBH}_4) = 1.45 \times 10^{13} (\text{CoCl}_2)(\text{NaBH}_4)^{0.39} e^{-17,500/RT}$$

where:

$$-\frac{d}{dt}(\text{NaBH}_4) = \text{the differential rate of consumption of NaBH}_4 \text{ in moles per (liter)(second) at time } t \text{ in the assumed reaction:}$$



(NaBH_4) and (CoCl_2) = the concentrations, in moles per liter, of the compounds in the reaction solution at time t

1.45×10^{13} = the value of the rate constant* for the catalyzed reaction

* The rate constant is conventionally designated as k in reaction-rate equations. The value given here was calculated from the bench-scale tests. The numerical value of k appears, however, to vary with the scale of the test. It is probable that certain secondary variables affect the over-all reaction rate as the scale of the reaction is changed from bench scale to 1/5 scale to full scale. It was found that more catalyst than would be predicted from this equation was needed in order to attain the desired reaction time in the larger scale tests. If the results of these tests are used to calculate the rate constant, its value for the full-scale reaction is estimated as 3.6×10^{12} .

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$e^{-17,500/RT}$ = the dimensionless Arrhenius constant, $e^{-\frac{\Delta E}{RT}}$, which expresses the variation of the reaction rate with temperature. When the activation energy, ΔE , is expressed as 17,500 calories, the gas constant, R , is 2.00 calories per degree K, the temperature T is in degrees Kelvin, and e is the Napierian logarithm base 2.718.

The rate equation is based on the usual assumptions that temperature and the concentrations of the reactants are uniform throughout the reacting solution. To calculate the total reaction time, the integrated form of the equation is first used to calculate the approximately constant rate over a short time interval of arbitrary length, such as 1 or 5 min. The calculated rate of consumption of NaBH_4 and the length of the time interval are used to calculate the amount of NaBH_4 reacting during the interval (i.e., rate x time = amount). The corresponding temperature rise is then calculated from the heat liberated by the amount reacted. The temperature rise and NaBH_4 consumption as determined give new values for the solution temperature and NaBH_4 concentration at the end of the time interval. These are substituted into the rate equation for the calculation of the rate during the next interval. When this stepwise procedure shows that the NaBH_4 has been exhausted, a summation of the time intervals gives an approximate value for the total reaction time.

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Secondary Variables Affecting Total Reaction Time

When a large reactor is used to generate the considerable quantities of hydrogen required, an appreciable time elapses before a uniformly reacting system is established that corresponds approximately to the ideal system discussed above. The quantitative details of the events occurring at the beginning of the reaction are not completely understood. However, two secondary variables are believed to have a major influence on the total reaction time. These are (1) the time required for the formation of the active catalyst after the addition of the cobalt chloride solution to the reactor pool, and (2) the time required for the dispersion of the catalyst throughout the reactor pool so that it can initiate hydrogen evolution.

It is known that the active catalyst is a solid product of a preliminary reaction of the cobalt chloride catalyst solution as it is added to the sodium borohydride solution. The preliminary reaction occurs rapidly, with the evolution of heat and hydrogen and with the consumption of some of the sodium borohydride. When larger amounts of cobalt chloride catalyst are added to counteract the slowness of the catalyzed reaction at low temperatures, the preliminary reaction may consume an appreciable part of the sodium borohydride. In this case, although the total evolution of hydrogen is affected only slightly, the initial concentrations in the system and the corresponding reaction time may be significantly changed.

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The effective concentration of solid catalyst, which controls the rate of hydrogen evolution, may be affected not only by the total weight of cobalt chloride added, but also by the average particle size and the solid surface area per unit weight. The properties of the solid active-catalyst particles may be strongly influenced by the temperature at which precipitation occurs, by the concentration of the cobalt chloride in the solution, and by the rate of addition.

The time required to disperse the active catalyst uniformly throughout the reaction pool after it is precipitated may be a significant part of the total reaction time. The active-catalyst particles formed at the top of the reactor pool fall toward the bottom by gravity, but the rate of fall is slow for these small particles. The downward motion is opposed by the rising bubbles of hydrogen, that are evolved exclusively at the solid surfaces, and also by the upward currents of the solution heated by the reaction.

The catalyst solution is distributed over the surface of the borohydride-solution pool from multiple holes in the catalyst reservoir so that the horizontal distance to the center and edges is minimized. Agitation by the hydrogen bubbles would tend to cause circulation and horizontal dispersal of the particles after some of them reached the bottom.

Although the qualitative aspects of the complex variables which affect the length of the initial time period are deduced from a few experiments, times for large-scale reactions cannot be predicted

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precisely by extrapolation. The only alternative is to conduct experiments over the total range of conditions which may be met in field operations. Therefore, an additional program of tests at low temperatures has been recommended, as discussed later in this report.

EXPERIMENTAL PROGRAM

Plan of the Experiments

It was pointed out previously that the rate of the catalyzed hydrolysis of NaBH_4 was originally studied in laboratory-scale tests (Phase I). The time required for the complete reaction to occur can be adjusted by adding a selected amount of catalyst. A reaction rate equation was deduced from these experiments that was presented earlier in this report. The equation, as written, describes the reaction rate at a fixed temperature and at fixed concentrations of reactants. However, the form of the equation shows that, to maintain constant total reaction time at different temperatures, the logarithm of the weight of the catalyst must be varied directly as the reciprocal of the initial temperature of the reacting solution; it is also evident that all other reaction conditions must be the same.

In connection with this type of problem, the ideal of exact similarity cannot be reached even in replicate experiments. A change in the scale of the experiment introduces larger changes in the uncontrolled secondary variables, such as the time for the

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formation and mixing of the active catalyst in the reaction pool, and these affect the total reaction time. The quantitative effect of these uncontrolled variables had to be measured experimentally, and this was the general objective of the experiments described in this report. It was also the purpose of these larger scale experiments, to provide bases for developing a procedure which would lead to reproducible results in the field, and for examining the generator design for possible shortcomings which could be eliminated in a full-scale prototype unit.

It was impossible to investigate each operating variable individually, because this would have required an excessively large number of experimental runs. Therefore, the first group of runs was designed to explore the combined effects, on the reaction time and completeness of reaction, of the concentration of the reactants, temperature, method of catalyst addition, volume of reaction solution, and generator design. Another group of runs studied specifically the effect of the amount of catalyst on the reaction time, and a third, the effect of the initial temperature on the reaction time. Last, the possible effect of natural surface water on the reaction time was measured in preparation for the final full-scale demonstration run. The full-scale generator was also tested in the field to determine the best procedure for filling it with water and for preparing the reaction pool.

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Materials

Sodium borohydride was supplied by the Sponsor, and was a product of Metal Hydrides, Inc., of Beverly, Massachusetts. The manufacturer's specifications indicated 98 per cent purity for the material provided. Analyses of three samples gave assays of 93, 95, and 98 per cent NaBH_4 . The cobalt chloride catalyst was cobaltous chloride hexahydrate (purified, crystals) obtained from Matheson, Coleman, and Bell. The sodium hydroxide used was Baker & Adamson chip sodium hydroxide (97 per cent min). River water was generally used in making up the solutions, (unless indicated otherwise).

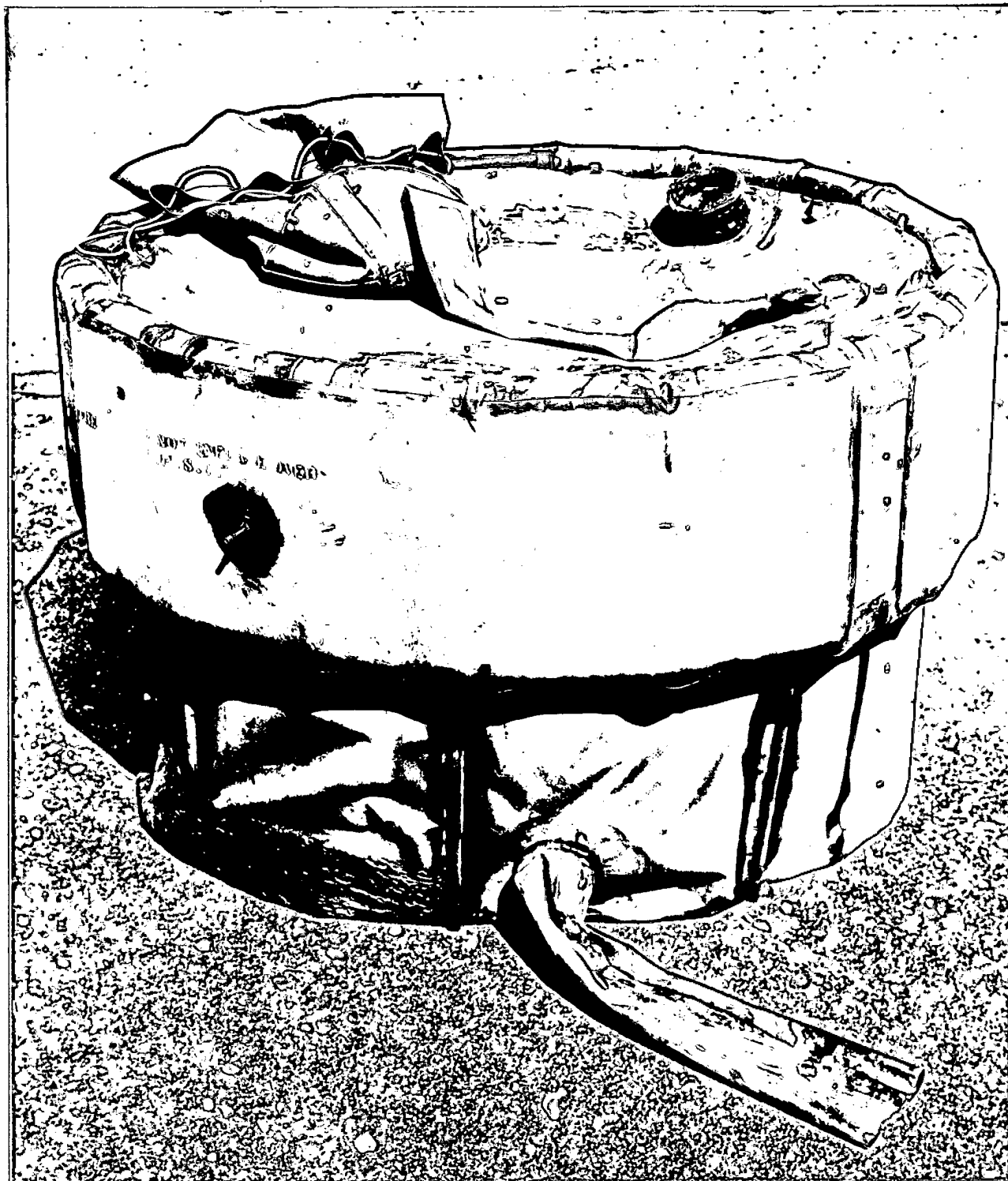
Apparatus

Figure 3 shows the experimental 1/5-scale generator as finally modified during the course of these experiments; a drawing of this unit is included in this report as Appendix II. The 1/5-scale generator was essentially the same as the full-scale generator already described, except that the diameter was smaller, so as to provide 1/5 the volume of reacting solution with the same solution-pool depth. The ring-shaped catalyst reservoir was cemented on the inside of the top; thus, it does not show clearly in Figure 3. The same experimental generator was used for the 1/10-scale tests by filling it with solution only to one-half the normal depth (normal depth, 18 in.).

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FIGURE 3. EXPERIMENTAL 1/5-SCALE HYDROGEN GENERATOR

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This experimental generator was approximately 43 in. in diameter and 33 in. high, and was made from neoprene-coated nylon fabric approximately 0.020 in. thick. The unit was supported by wooden stays and by an inflated ring of 3-in. annular thickness that was attached to the top and was inflated with the use of a hand pump. Access ports for the addition of the catalyst solution, for the entry of the water, and for inspection purposes were provided by three gas-tight brass zippers. This experimental generator was designed to withstand 0.4 psig and a maximum temperature of 165 F.

The gas outlet was fitted with a brass collar holding two thermocouple wells and an exit tube for the generated hydrogen. In the course of the experiments, the evolved gases were passed through a meter before being exhausted to the atmosphere by a specially constructed exhaust system, which minimized the fire and explosion hazards. The temperature of the reacting solution was measured by single-junction iron-Constantan thermocouples inserted into the generator through the gas outlet. Barometric pressures were read from either an aneroid or a mercurial barometer.

During the initial series of tests, leakage occurred around the zippers, which were difficult to operate and eventually were broken. It was concluded that the zippers would be unreliable for field operation of such a unit.

The zippered water inlet at the bottom edge, and the zippered catalyst-solution entry port in the top were patched and sealed. A 4-in.-diameter neoprene-coated fabric water-inlet tube

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and drain was subsequently fabricated and assembled to the catalyst-solution ring. A neoprene-coated fabric bag was attached to the end of the catalyst entry tube. This bag was large enough so that it could be pulled over a 5-gal bucket; thus, to make the catalyst-solution addition, the bucket could be up-ended so as to simulate the contemplated procedure of pouring the catalyst solution into the full-scale generator. This experimental generator, so modified, was used beginning with Run 9 and the modifications were found to be quite satisfactory.

Procedure

In a standard 1/5-scale experiment, the inflated 1/5-scale generator was filled with 110 gal of water pumped from two 55-gal drums, and a weighed amount of NaBH_4 (approximately 20 lb) was dissolved in the water. The temperature of the pool was read from a thermometer dipped in the solution. An appropriate weight of catalyst, determined on the basis of the measured temperature and the desired total generation time, was dissolved in 2 gal or less of water and added to the generator pool. At first, the catalyst weight to be used was read from a chart of catalyst weights and temperatures given in the earlier report on the Phase I effort. The results obtained from the early experiments described herein quickly showed that these catalyst weights were insufficient to produce complete reaction within the 60-min limit

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for large-scale tests; consequently, revised values for catalyst weight were deduced from the experimental results and utilized. (The revised catalyst weights were given in Table 1.)

At times, the standard amounts of water and borohydride were varied somewhat to accommodate the objectives of individual experiments. Also, the catalyst solution was, on occasion, added through a funnel with a long curved spout, which could discharge the solution at a selected location near the edge or at the center of the pool, instead of distributing it over the top of the pool from the perforated ring.

It had been shown in the earliest tests that the measured volume of hydrogen corresponded consistently to almost complete reaction, and that the course of the reaction could be followed by noting the rise in the temperature of the reaction pool. Therefore, in the later tests, the gas meter was by-passed, and the time when the temperature of the reaction pool stopped rising, was measured as indicating complete reaction.

Experimental Results

Table 2 gives the experimental results of the 1/10- and 1/5-scale runs in the 1/5-scale generator in air; Table 3 lists similar information for the runs in which the reaction pool within the experimental generator was surrounded by water on the outside. For each table, Column (1) gives the run numbers, which were assigned in chronological order, and Column (2) indicates the

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TABLE 2. HYDROGEN GENERATION WITH EXPERIMENTAL GENERATOR IN AIR

Run No. (1)	Vol of Hydrogen (Dry, STP), cu ft		Temperature, F		Catalyst Added (CoCl ₂ ·6H ₂ O)		Total Reaction Time, min (8)	Remarks (9)
	Theore- tical (2)	Experi- mental (3)	Initial (4)	Rise (5)	Amount, lb (6)	Method (7)		
1	360	--	53	52	0.92	From ring	65	Preliminary trial run
2	360	--	62	--	0.71	From ring	70	Preliminary trial run
3	720	686	62	63	1.37	From ring	85	Slow rate
4	360	--	61	53	0.96	At center	58	H ₂ leak
5	648	599	54	46	1.62	At edge	150	Reduced NaBH ₄ concentra- tion
6	772	720	46	61	2.02	At center	125	--
7	772	744	64	74	1.83	At center	70	Increased NaBH ₄ concentra- tion
8	772	764	55	47	2.02	At center	125	Reduced NaBH ₄ concentra- tion
9	720	686	66	46	1.67	From ring	64	--
10	720	--	63	57	2.66	From ring	59	--

Note: The numbers in parentheses, located under the last line of each of the column headings, are provided to facilitate the presentation and comprehension of the discussion of the research in the text.

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TABLE 3. HYDROGEN GENERATION WITH EXPERIMENTAL GENERATOR IN WATER

Run No. (1)	Vol of Hydrogen (Dry, STP), cu ft		Temperature, F		Catalyst Added (CoCl ₂ .6H ₂ O)		Total Reaction Time, min (8)	Remarks (9)
	Theore- tical (2)	Experi- mental (3)	Initial* (4)	Rise (5)	Amount, lb (6)	Method (7)		
11	360	364	47	37	5.58	From ring	34	--
12	360	373	47	29	4.18	From ring	59	--
13	360	354	47	33	5.15	From ring	41	--
14	360	--	66	35	1.98	From ring	42	--
15	360	--	65	29	2.17	From ring	35	--
16	720	--	66	46	4.41	From ring	23	--
17	720	--	55	57	11.70	From ring	~10	--
18	370	--	85	23	0.91	From ring	30	Pond water used
19	370	--	84	33	0.78	From ring	37	Pond water used
20**	3,700	--	82	55	8.60	From ring	26	Pond water used

Note: The numbers in parentheses, located under the last line of each of the column headings, are provided to facilitate the presentation and comprehension of the discussion of the research in the text.

* The surrounding water was also at this temperature.

** This was the full-scale demonstration run in the prototype generator.

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theoretical amount of hydrogen corresponding to the weight of NaBH_4 used in each run. In the calculation of the values in the table from the weight of NaBH_4 used, it was assumed in each case that the NaBH_4 was 95 per cent pure. Some runs show target volumes of hydrogen of 720 cu ft and others, 772 cu ft, but these are all nominally 1/5-scale runs. These differences arose from slightly different assumptions as to the NaBH_4 purity, in the calculation of the amount to be used in the experimental runs. It should be recognized that these differences had no significant effect on the results obtained and the conclusions drawn. The same explanation applies to the nominally 1/10-scale runs, which, as described in the tables, show target hydrogen volumes of 360 and 370 cu ft.

Column (3) shows the standardized volume of hydrogen generated experimentally for those runs in which it was measured. The values given were calculated from the measured volume, temperature, and pressure of the metered gas, assuming that the gas was saturated with water vapor and was all at the measured temperature.

The gas temperature rose continuously in the course of each run, so that the temperature and water-vapor corrections had to be calculated for incremental volumes in order to obtain by summation the total correction. Considering also that the actual purity of the NaBH_4 undoubtedly varied from run to run, the agreement between Columns (2) and (3) is considered to be remarkably good. The ratio of the values in Columns (3) and (2), representing the completeness of the reaction, varied between 93 and 103 per cent. Following the performance of Run 13, the volume of hydrogen

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evolved was not measured, since it was then known that the reaction went to substantial completeness.

Columns (4) and (5) give the initial temperature of the reaction pool and the temperature rise noted at the end of the reaction. In the runs described in Table 3, the experimental generator wall was cooled by the surrounding water which was at the initial temperature; the temperature of the surrounding water did not change appreciably during any of the runs.

The temperature rise for this reaction using the standard concentrations* would be 58 F without heat losses through the walls of the generator or in the exit gas and vapor. In most of the runs, the temperature rise generally followed a qualitatively appropriate pattern. Runs 5 and 8, at reduced concentration, showed a temperature rise below normal, and Run 7, with a more concentrated solution, yielded a higher than normal temperature rise. In Run 5, the solution volume was normal and less NaBH_4 was used. Run 8 involved a reduced concentration which was obtained by adding 15 extra gal of water to the pool. In Run 7, there was a deficiency of 10 gal of water.

The water-cooled runs in Table 3 generally showed, as expected, less of a temperature rise than did those described in Table 2 that were air cooled. Within Table 3, the data on temperature rise showed the expected correlation with the duration of

* For 1/5 scale, these are 110 cu ft of water and approximately 20 lb of NaBH_4 .

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the run [in Column (8)]; the shorter the total reaction time, the less was the opportunity for heat losses to occur through the generator walls, and consequently, the greater was the increase in temperature.

Column (6) indicates the weight of solid catalyst added in water solution to the generator in each case, and Column (7), the method of addition of the catalyst solution. Column (8) lists the time interval between the start of the catalyst-solution addition and the end of hydrogen evolution, i.e., the total time for the hydrogen-generation reaction.

DISCUSSION OF EXPERIMENTAL RESULTS

Figure 4 plots for each experimental run the catalyst weight against the initial temperature. The weight scale is logarithmic; the temperature scale is linear with the reciprocal of the absolute temperature. Catalyzed reactions requiring the same reaction time at different temperatures should theoretically fall on a straight line when the catalyst weight and the initial temperature are plotted on such scales, if all other reaction conditions are comparable.

Two straight lines are shown on Figure 4. The upper line is discussed later. The lower line was constructed from the experimental results of the previously conducted 1/100-scale tests, (described in the "Summary Report on Task Order No. C" dated January 28, 1957), and represents the amount of catalyst

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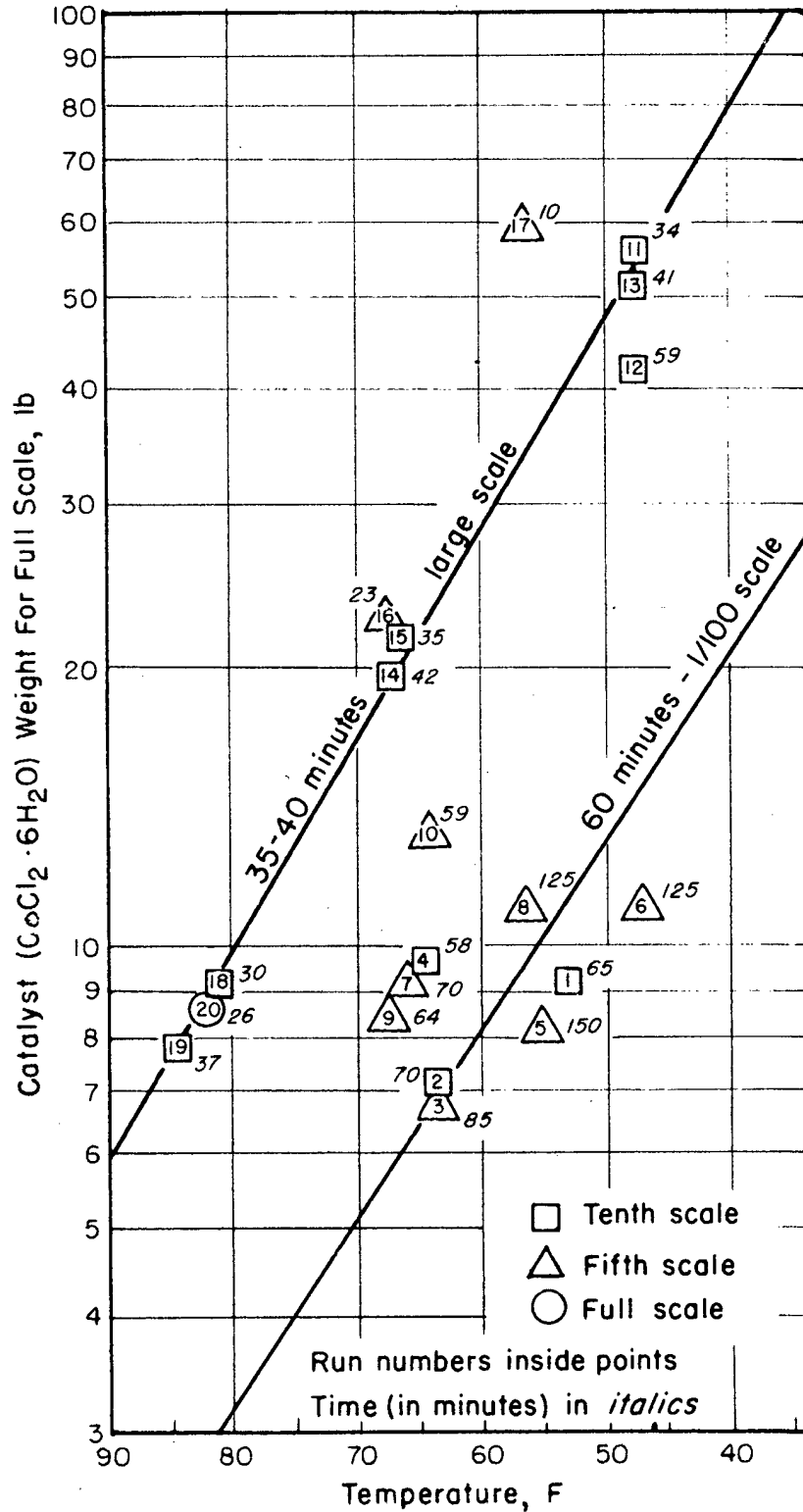


FIGURE 4. LARGE-SCALE GENERATION OF HYDROGEN

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necessary to bring about a complete reaction in 60 min at these temperatures in the laboratory-scale runs. However, all of the experimental points shown on Figure 4 are for large-scale runs, described in this report; the run number is inserted inside each point, and the time in minutes for complete reaction is shown in italics at the side.

In the interpretation of the significance of the experimental results, the individual experimental points in Figure 4 are considered in terms of the corresponding experimental time periods as shown. If reference is made to the first series of 10 runs (Table 2), it should be noted that all of the corresponding points would have fallen directly on the lower line of Figure 4 if the catalyst weights had been so chosen, at the option of the experimenter. Only Runs 2 and 3 used exactly the weight of catalyst called for by the 60-min line for the 1/100-scale tests. Further, in these two runs, the times for complete reaction were appreciably longer than 60 min. It thus appeared that the total reaction time was longer in the large-scale runs because of reaction variables which differed from those prevailing in the laboratory-scale reactions. The remainder of the series are grouped around the 60-min line, and represent departures from the standard conditions as outlined specifically in Table 1. The three points which fall below the lower line represented runs in which considerably less than the standard amounts of catalyst* were used; this in itself

* It should be emphasized that any discussion of the amount of catalyst used involves directly a consideration of the initial temperature of the reaction pool. Thus, in any comparative review of the data in Tables 2 and 3, and particularly the data on the amount of catalyst used, it should be noted that the initial temperatures varied considerably.

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would be expected to retard the reaction and thus increase the time. In Run 5, the concentration of NaBH_4 was lower, and the catalyst solution was added at the outside edge of the pool at one location. Either of these two variations alone would also be expected to increase the reaction time. All of these factors in combination caused a large increase to 150 min in Run 5. Run 6 involved a less critical addition of catalyst at the center of the pool, and the 125-min reaction time obtained was slightly less unfavorable.

In Run 7, the retarding effect of pouring the catalyst solution into the center was partially compensated for by increasing the amount of catalyst and increasing the concentration of NaBH_4 ; but, the resulting reaction time of 70 min was still above the value predicted from the laboratory-scale tests. It was concluded, however, that the retarding effect of the center pour could not account entirely for the relatively long 70-min reaction; and that unidentified scaling effects were also involved. Run 8 confirmed qualitatively the retarding effects of low NaBH_4 concentration and center pour, partially counteracted by an increased amount of catalyst.

Runs 9 and 10 completed the first series of runs; these involved successively increased amounts of catalyst with other conditions maintained standard. Run 10, with a total reaction time of 59 min, fell barely within the 60-min maximum; the amount of catalyst used was about twice that shown by the lower line in Figure 4.

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It thus became apparent that there was a definite effect encountered in scaling the reaction to a larger size and this increased the total reaction time considerably. Although this scaling effect is not completely understood and was referred to earlier as "unidentified", the evidence points to inefficient mixing of the catalyst solution vertically in the generator (1/5 scale) unit. This retarding effect might be overcome by injecting the catalyst solution at the bottom of the reaction pool, so that natural convection induced by the temperature rise and by the vigorous gas evolution would cause more positive mixing, and would reduce or eliminate the probability of a non-reacting bottom layer during the early part of each run. A redesign of the system to provide for this method of catalyst-solution addition, and a corresponding study of a new associated procedure did not appear as advisable, over all, as the simpler alternative of increasing the amount of catalyst used, in order to counteract the slower reaction characteristic of the larger scale generation operation.

Therefore, a second series of runs, Nos. 11 through 18, was carried out to establish a new reference curve for catalyst requirements. It was known qualitatively that the cooling effect of water surrounding the generator would also retard the reaction, so, in this series, the generator was floated in water. It was estimated on the basis of detailed calculations that a reduction in reaction time by about one-third would probably be attained by doubling the catalyst weight, under otherwise equivalent

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conditions. Also, it was decided that it would be worth while to work toward a target time of about 35 to 40 min, to insure completion of the generation operation well within the specified time limit. It was felt that a 40-min target time would provide a safe margin for possible variations in the scaling effect, which was not completely understood and so could not be positively controlled. Also, the shortest possible generation time was desirable in itself.

On the basis of these considerations, Runs 11, 12, and 13 were made at 47 F, and these successfully bracketed the target time of 40 min, as anticipated. Another group of three runs at about 66 F again bracketed the target time, and provided the basis for the construction of the upper line of Figure 4, which shows the amount of catalyst required to complete large-scale generations in 35 to 40 min. Run 17 was partially successful in establishing a safe lower time limit; the completion of the reaction was timed at about 10 min. A large increase in the amount of catalyst used, in excess of the corresponding amount indicated by the new reference line (the upper line of Figure 4), showed that the reaction could operate predictably and fairly smoothly in much shorter times. However, in Run 17, the capacity of the gas exit was exceeded, and the experimental fabric generator was damaged slightly.

The 1/5-scale generator was subsequently repaired and moved to a pond of surface water, where it was used in conducting Runs 18 and 19, to examine the possible effects of higher initial temperature and of the impurities of the natural water on the reaction time. As expected, no significant effect of water impurities

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was found. However, there appeared to be some trend toward shorter reaction times at higher temperatures.

The experimental program was concluded with a full-scale demonstration, Run 20, in the prototype full-scale generator, following the procedure outlined in detail in the early sections of this report. The reaction was successfully completed in 26 min without damage to the generator. A failure in an "on the spot" improvised fitting connecting the generator gas outlet to the balloon tube prevented complete filling of the test balloon; however, this was not attributable to the generator design.

A trend exists at high initial temperatures toward generation times which are shorter than those predicted by the upper line of Figure 4. This is satisfactory and even desirable. The same trend implies an undesirably large increase in the generation time at the extreme low end of the anticipated-service temperature range; it appears that some adjustment in operating procedure will have to be made to counteract this trend.

The trend to longer generation times at the low temperatures is also indicated by the greater slope of the upper line in Figure 4 as compared to the lower one. Thus, the upper line shows proportionately larger catalyst requirements at the low temperatures. It is believed that this trend is qualitatively distinct from the scaling effect attributed to stratification and inefficient mixing, which have previously been discussed as the reason for a portion*

* The remainder of the upward displacement was attributable to the decision to reduce the target total generation time from 60 min to 35-40 min.

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of the upward displacement of the large-scale reference line in Figure 4. It seems probable that there exists a catalyst concentration effect that has not been accounted for in the basic reaction equation. Because the active catalyst is a solid formed from the cobalt chloride solution, uniform mixing would produce a cloud of active-catalyst particles in the reaction pool, and each particle would be reacting within its own sphere of surrounding solution. The greater the number of active-catalyst particles, the smaller the sphere of reaction of each, or the greater the tendency for a "blanketing" or interference of each particle with its neighbors. Thus, with increasing amounts of catalyst used, and this is the case at the lower temperatures, the activity of the catalyst per unit weight would be reduced, with the resulting trend toward increasing generation time at the low temperatures.

It is not possible to increase the amount of catalyst sufficiently to counteract the increase in total generation time at the low temperatures, because of the limited solubility of the cobalt chloride catalyst in water at such temperatures. The present procedure calls for dissolving the catalyst required for a full-scale run in 5 gal of water; this represents about the maximum volume and weight of catalyst solution which can be handled by one man when the solution is in a single container. At temperatures of less than about 45 to 50 F, the solubility of the cobalt chloride catalyst in 5 gal of water is about 45 lb per 5 gal of solution, or less than the catalyst requirements shown on the upper line of Figure 4 for the temperature range below 50 F.

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In addition to the limitation on cobalt chloride solubility at the low temperatures, it may be undesirable to add excessively large amounts of catalyst because of the rapid evolution of hydrogen during the preliminary reaction which forms the solid active-catalyst particles from the cobalt chloride solution. Based on calculations made early in this program, 10.6 cu ft of hydrogen are released in the initial surge for each pound of catalyst added. The initial surge of gas might reach dangerous proportions, although it could probably be controlled by reducing the rate of flow of the cobalt chloride solution into the pool.

To provide for generation at the low temperatures, three variations in the procedure to overcome solubility limitations might be made without necessitating a major change in the full-scale generator design:

- (1) The amount of catalyst might be reduced to give a longer generation time of about 55 min.
- (2) The amount of cobalt chloride that could be dissolved might be increased by heating the catalyst solution, by means of auxiliary equipment which might be provided within the over-all total weight limitation.
- (3) The volume of water in the reaction pool might be reduced for low-temperature generation, without changing the quantity of NaBH_4 used; thus, the heat of reaction would raise the reaction pool to a higher temperature, which would accelerate the reaction, thereby shortening the generation time or reducing the amount of catalyst required.

These variations might be utilized singly or in combination.

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Experimental runs would be necessary in order to measure the quantitative effects of these three variations. The general objective of such experiments would be to develop the simplest procedure that would permit satisfying the reaction specifications at low temperatures.

Although it would be preferable not to make any change in the full-scale generator design, the following suggestions requiring both a design and procedural change are presented in order to complete this discussion; both changes would be directed toward achieving more efficient mixing of the catalyst solution in the reaction pool.

It is deduced, from comparison of the effects of pouring the catalyst solution in through the ring and at the center of the pool, that lateral distribution occurs more readily than was originally expected. It, therefore, seems probable that more efficient mixing could be attained by adding the catalyst solution through a tube to the bottom center of the reaction pool. This should induce a circulation pattern upward at the center and downward around the generator wall and should rapidly spread the catalyst to all parts of the pool. However, some positive method of forcing the catalyst solution through such a tube would probably be required to counteract the back pressure which would build up as soon as the reaction started.

The other change which might be applicable would be to make the catalyst addition by dumping pelletized solid anhydrous cobalt chloride into the pool. The pellets would presumably fall

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by gravity to the bottom before they dissolved. There would be no problem of ultimate solubility in the whole pool, as there is for a limited amount of catalyst solution made up before addition to the pool. Material handling would be easier with the pellets, as the weight and volume of catalyst required would be much lower than the total weight and volume of 5 gal of solution. The maximum weight of catalyst might be about 25 lb for the lowest temperature, and this amount, in the form of pellets, would occupy a volume of perhaps 2 gal or less.

However, the addition of solid catalyst would require:

(1) a different type of catalyst reservoir which could be filled with the pelletized catalyst, closed, and then manipulated to permit dumping the solid into the generator pool; (2) formulation of a catalyst pellet that would sink to the bottom and then disintegrate to a fine powder so as to facilitate rapid dissolution; and (3) a series of experimental runs to confirm these predictions and establish the details of a revised procedure.

RECOMMENDATIONS FOR FUTURE WORK

It is recommended that additional work be done to provide for hydrogen generation in the prototype full-scale generator at low temperatures. It is suggested that such future work should be directed toward a study of the reduction in catalyst requirements at low temperatures by working to a target value of 55-60 min for

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the total generation time, and by reducing the amount of water used in the reaction pool so as to increase the associated temperature rise when the hydrogen-generation reaction occurs.

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APPENDIX I

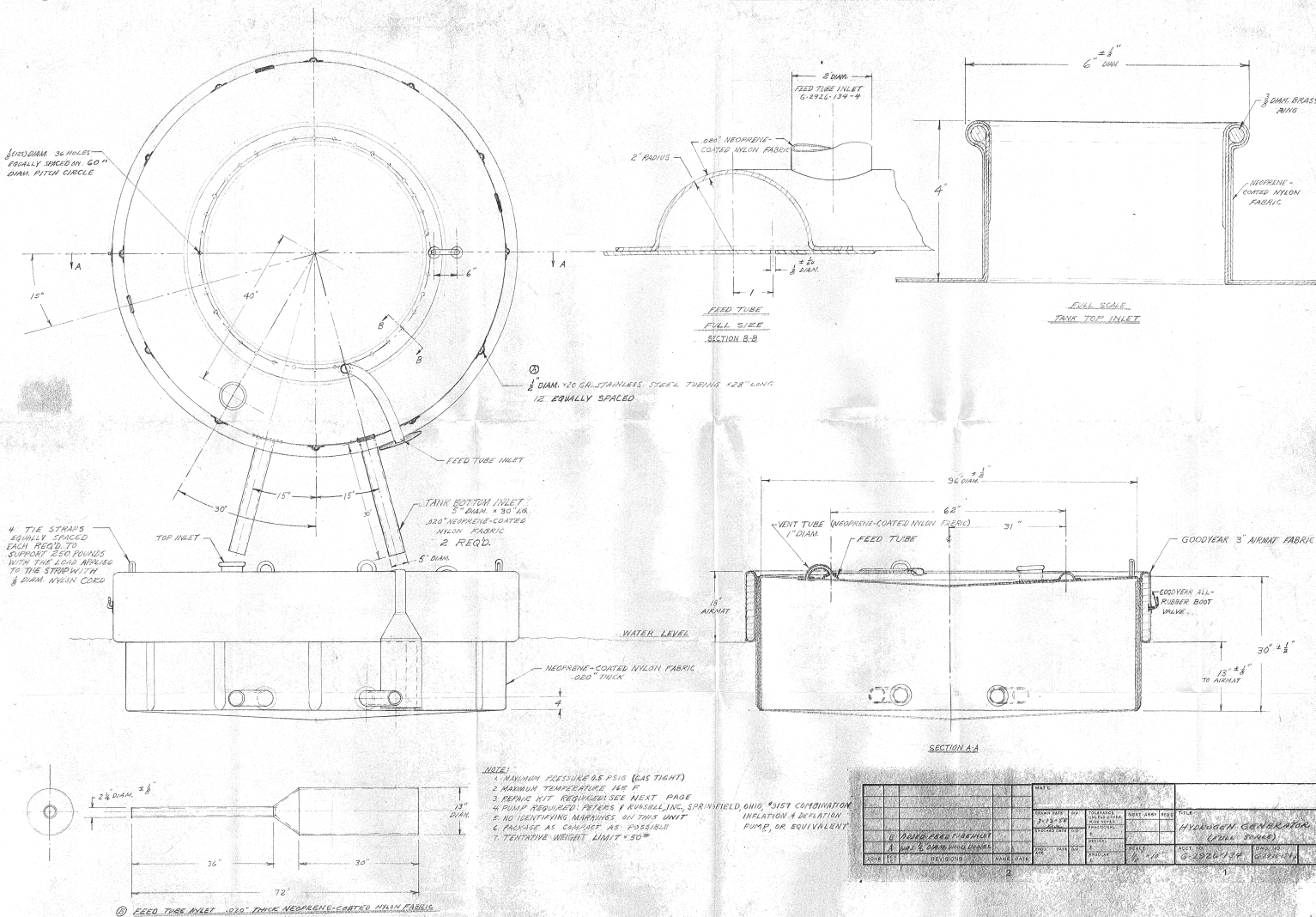
Drawing of Full-Scale Generator

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Declassified and Approved for Release 2012/10/25 : CIA-RDP79-03638A001200110001-9

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NO.	REV.	DATE	BY	CHKD.	DESCRIPTION
1					HYDROGEN GENERATOR (FULL SCALE)
2					
3					
4					
5					
6					
7					
8					
9					
10					

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Declassified and Approved for Release 2012/10/25 : CIA-RDP79-03638A001200110001-9

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GOODYEAR "SWIMMING TANK" REPAIR KIT

The repair kit provided by the manufacturer of the experimental generators consisted of the following:

<u>Quantity</u>	<u>Material</u>	<u>Goodyear No.</u>
4	1/2-pint cans of patching cement	1451-C
4	8-cc bottles of cement accelerator	983-C
1	1-inch cement brush	--
1	2-1/4 x 2-1/4-inch roller	--
24	1-inch-diameter patches	--
1	Linear yard of neoprene-coated nylon fabric	--
1	Instruction booklet	--

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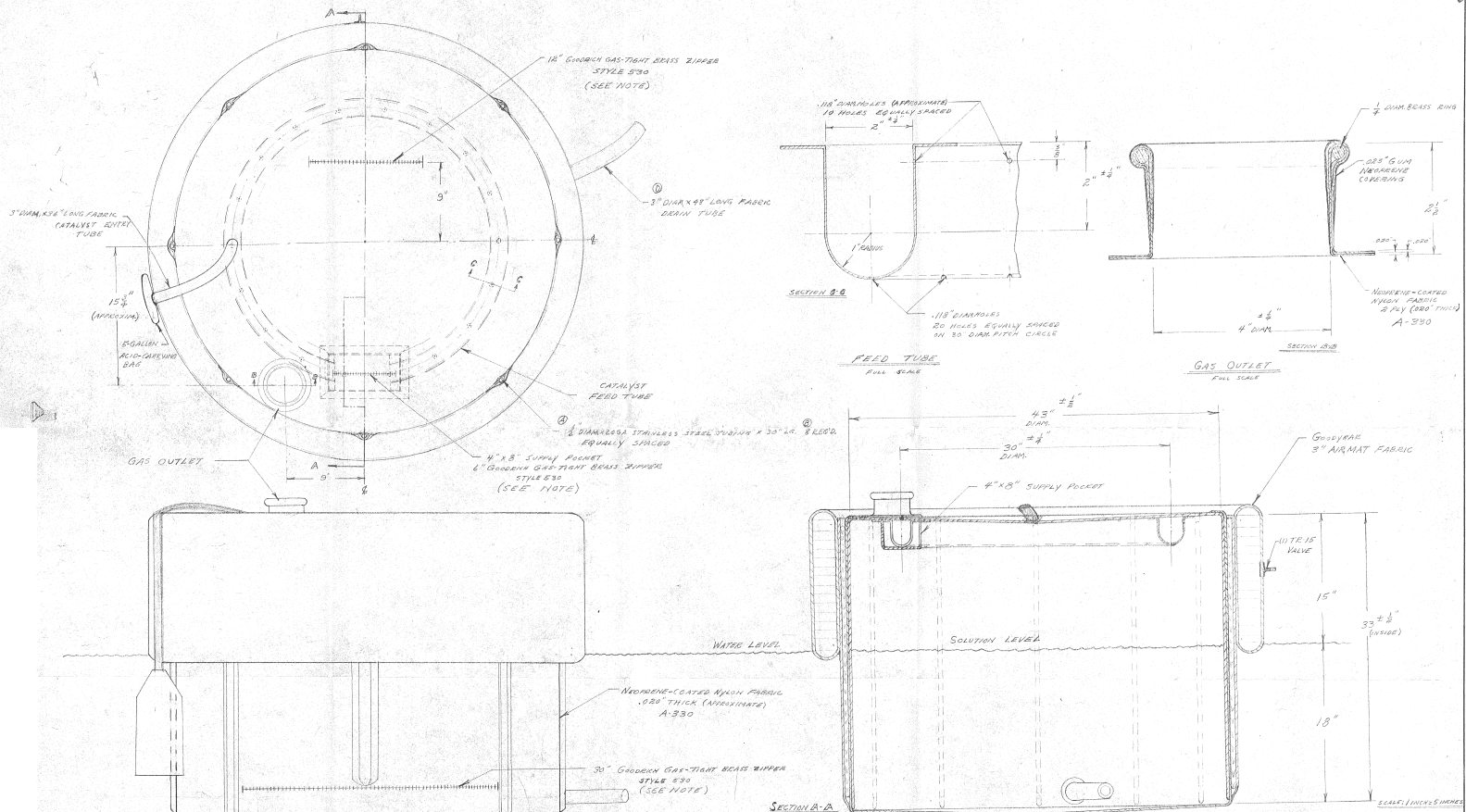
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APPENDIX II

Drawing of Experimental 1/5-Scale Generator

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NOTE:
 ALL ZIPPER WERE FINISHED WITH FABRIC & COVERED WITH NEOPRENE-COATED NYLON FABRIC

REV.	REVISIONS	DATE	NAME	NO. REQS.	DATE	ENGR.	FRANCOIS DIMENSIONS
1	DRAIN TUBE ADDED			20		KWJ	4
2	30" DIAM. HOLES FOR BEAD RINGS						
3	30" DIAM. HOLES FOR BEAD RINGS						
4	30" DIAM. HOLES FOR BEAD RINGS						
5	30" DIAM. HOLES FOR BEAD RINGS						
6	30" DIAM. HOLES FOR BEAD RINGS						
7	30" DIAM. HOLES FOR BEAD RINGS						
8	30" DIAM. HOLES FOR BEAD RINGS						
9	30" DIAM. HOLES FOR BEAD RINGS						
10	30" DIAM. HOLES FOR BEAD RINGS						
11	30" DIAM. HOLES FOR BEAD RINGS						
12	30" DIAM. HOLES FOR BEAD RINGS						
13	30" DIAM. HOLES FOR BEAD RINGS						
14	30" DIAM. HOLES FOR BEAD RINGS						
15	30" DIAM. HOLES FOR BEAD RINGS						
16	30" DIAM. HOLES FOR BEAD RINGS						
17	30" DIAM. HOLES FOR BEAD RINGS						
18	30" DIAM. HOLES FOR BEAD RINGS						
19	30" DIAM. HOLES FOR BEAD RINGS						
20	30" DIAM. HOLES FOR BEAD RINGS						

PROJECT NO. HYDROGEN GENERATOR
 DRAWING NO. G-926-134
 G-926-134-1