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Study of Energy Conversion Devices
Report No. 1

Contract DA-36-039-SC-78955 *new*
Task No. 3-99-09-401

First Quarterly Progress Report
July 1, 1959 to Sept. 30, 1959

U. S. Army Signal Research and Development Laboratory
Fort Monmouth, New Jersey

October 31, 1959

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MSA Research Corporation

Subsidiary of Mine Safety Appliances Company

Callery, Pennsylvania

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STUDY OF ENERGY CONVERSION DEVICES

Report No. 1

The purpose of this contract is to develop a cyclic or continuous thermally regenerative galvanic cell.

First Quarterly Progress Report

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Task No. 3-99-09-401

U. S. Army Signal Research & Development Laboratory
Fort Monmouth, New Jersey

October 31 1959

This report prepared by:

Approved by:

R. E. Shearer

R. E. Shearer

C. B. Jackson

C. B. Jackson

Vice President, Director of Research

MSA RESEARCH CORPORATION

Callery, Pennsylvania

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I PURPOSE

It is the purpose of the research and development work under this contract to develop a practical electrochemical device which converts thermal energy into electrical energy by means of formation of suitable compounds followed by the electrochemical recombination of the decomposition products on a cyclic and continuous basis. Special consideration is to be given to the ionic hydrides.

As was pointed out in MSA Research Technical Proposal TP-1138, submitted to the U. S. Army Signal Research and Development Laboratory preliminary to award of the subject contract, MSA Research has developed a thermally regenerative galvanic cell based on the electrochemical formation of the ionic hydrides on a batch basis⁵. In general, hydrides of the alkali and alkaline earth metals are formed in fused halides from a metal electrode and a hydrogen electrode. Reactants have been regenerated on a batch basis by heating the product. This cell will be the basis of part, at least, of a program directed to a cyclic system.

II ABSTRACT




A closed cycle system previously built ~~with MSAR funds~~ was operated regeneratively. An emf of 30 mv was obtained regeneratively although regenerator temperature was held to about 790°C and although an emf of only 0.27 V was obtained in the cell just prior to regeneration with a pressure of one atmosphere of hydrogen. On the basis of experience on the first unit, an improved system was designed and built.

A batch-type cell was put into operation with the eutectic of KCl-LiCl as solvent and with a potassium electrode. A peak emf of 0.33 V was obtained just before accidental overheating caused a structural failure.

Differential thermal analysis during temperature changes shows promise of a lower melting eutectic for halides of lithium with addition of LiI to the former eutectic of LiF-LiCl.

Background data is presented in this report to show the basis of experimental approaches taken in the subject contract.



III CONFERENCES

On August 13, 1959 Messrs. C. B. Jackson, J. W. Mausteller, R. E. Shearer and R. C. Werner visited the U. S. Army Signal Research and Development Laboratory to discuss the proposed work plan under the subject contract. Messrs. C. Clark, A. Daniel, H. Hunger and J. Lyons represented the Signal Corps. It was agreed that operation of a closed cycle system should be attempted on the basis of the limited available present knowledge, that studies of alternate solvents and anode material should be made, and that studies of single electrode potentials should be considered, especially when lower temperature operation is possible.

IV FACTUAL DATA

A. Background Data

Considerable background information has been developed both from MSAR-supported work and from the literature prior to the experimental program under the subject contract.

Decomposition Pressures. While there is considerable deviation from Raoult's Law at lower temperatures with the lithium hydride-lithium system as is shown in Fig 1¹, such deviations may not exist at the higher regeneration temperatures. The deviation is associated with the plateaus in the composition-pressure isotherms as is explained by Gibb and Messer¹:

"The dissociation pressure of the hypothetical completely pure stoichiometric lithium hydride is very high. The removal of small amounts of hydrogen produces a large decrease in pressure, the liberated lithium metal going into solution in the hydride, which remains a single phase. When enough hydrogen has been withdrawn to saturate the lithium hydride with lithium metal, the pressure becomes independent of further change of composition, a second phase, lithium rich, separating out. The constant pressure in this two-phase region of varying composition is the so-called "plateau" pressure. When enough hydrogen has been withdrawn, the hydride phase disappears, and further hydrogen loss produces a decrease in pressure due to loss of hydrogen from solution in lithium metal in the remaining single phase. The pressure finally diminishes to zero at zero hydrogen content (pure lithium)."

According to Heumann and Salmon², lithium hydride and lithium are completely miscible at 997°C so that the plateau will not appear and Raoult's Law should apply more closely. At a slightly lower temperature than this, Gibb and Messer¹ show pure hydride to have a dissociation pressure of 10,000 mm. Application of Raoult's Law for a solution of 5 wt% LiH in Li would suggest a dissociation pressure of about 500 mm.

Data are not available on decomposition pressures of lithium hydride dissolved in halide eutectics; but Gibb and Messer¹ cite earlier work showing pressure of 85 mm, 345 mm, and 2477 mm at 800, 900 and 1000°C respectively for a mixture of 85.4 mol % of LiH and 14.6 of LiF. This is considerably lower than would be obtained by application of Raoult's Law and suggests one possible advantage to regenerating from the metal phase. Since there is no information available on the distribution coefficients of ionic hydrides between liquid metal phase and halide solvent phase, compositions of these phases under actual operating conditions remain unknown and can be learned only experimentally. With compositions unknown, decomposition studies can best be carried out in the closed cycle system directly and can be measured by emf's developed as well as by sampling of the gas stream.

Alternate Hydrides. In event that excessively high decomposition temperatures are required for lithium hydride in metal or halide solution, alternates might be considered in hydrides having smaller standard free energies of formation than has lithium hydride.

Potassium hydride is such a compound; and a low melting halide eutectic, that of the chlorides of lithium and potassium, show promise as the solvent. It has a melting point of 352°C^3 and a lower potassium ion concentration (59 mol %) than solvents having the potassium ion as the only cation. An unknown factor in this system is solubility of metallic potassium in the solvent. This might prove a serious problem if there is a parallel situation in KX-K binary systems, where X is F, Cl, Br and I. Here there is complete miscibility as close as +47, +48, -7 and +22 degrees, respectively, to the melting points of the salts⁴. An experimental approach is needed since there appear to be no data on the proposed system in the literature.

Other hydrides of possible use are shown in Table 1 which also includes some physical properties.

Table 1 - Representative Thermally Regenerative Ionic
Hydride Electrochemical Systems

Hydride	Temperature ¹ (°C)	E° from -ΔF° at 298°K (volts)	Solvent	
			Composition (mol %)	M. P. (°C)
BaH ₂	730	0.70	7 BaF ₂ -93 BaI ₂	680
NaH	425	0.36	34.5 NaF-65.5 NaCl	685
KH	432	0.28	40 KF-60 KBr	580
SrH ₂	840	0.71	11 SrF ₂ -89 SrCl ₂	740
LiH	850	0.72	89 LiBr-11 LiF	450
CaH ₂	1000	0.78	81 CaCl ₂ -19 CaF ₂	644

¹ Temperature for dissociation pressure of 1 atmosphere for the pure hydride.

Alternate Solvents. Since thermodynamic considerations show the advantage of a low temperature cell, lower melting solvents are attractive for improved cell performance. Unanswered questions in this field include:

1. Eutectic compositions having lower melting points.
2. Effect of these lower melting eutectics on cell voltage and stability of the metal electrode from the standpoint of solubility in the melt.
3. Similar stability of the metal electrode in the solution of product hydride in the melt.

The purpose of one phase of the experimental program is to find answers to questions 2 and 3 with respect to the eutectic of LiBH_4 - KBH_4 which is reported⁶ to melt at 102°C and to contain about 46 wt % KBH_4 and with respect to the low melting eutectic of the chlorides of K, Rb, Cs and Li which is reported⁷ to melt at 270°C . As to the borohydride eutectic, it is thermally unstable, requiring that regeneration be done from the liquid metal phase. As to stability of a lithium electrode in the quaternary eutectic, thermodynamic considerations are reassuring as is shown by the following standard free energies of formation in KCal/mole at 1000°K as calculated from Quill⁸:

LiCl - 80.2
KCl - 81.6

RbCl - 82.4
CsCl - 83.2

With respect to the hydride of potassium, however, it is unstable in contact with lithium metal as is indicated by standard free energies of formation at 298°K which are -16.7 KCal/mole for LiH^9 and -7.46 for KH^9 . An experimental approach will indicate whether there is stability of KH in solution.

Single Electrode Potentials. Information on single electrode potentials is of value in determining the degree of reversibility of individual electrodes and which electrode needs the greatest degree of optimization. Considerable work has been done in the field of electrode potentials in fused salts using a variety of reference electrodes. Delimarskii and Koltii¹⁰ used a glass-tin-sodium reference electrode in fused salts containing $10^{-4}M$ or up to $600^{\circ}C$. Liu discusses this and other reference electrodes in his 1957 thesis¹¹. The glass-tin-sodium electrode and asbestos diaphragms show promise of stability in the media in question. As lower melting solvents are used, irreversible behavior of electrodes may create problems and require attention. In event problems arise in electrode behavior, it is planned to make a glass-tin-sodium electrode according to Delimarskii and Kolotti and use it as reference against the hydrogen electrode in a sodium hydride cell. Platanex III platinum plating solution has been obtained for possible use in studying hydrogen electrode materials.

Materials of Construction. Unstressed types 304 and 316 stainless steel have been used satisfactorily in earlier work on the lithium hydride cell over a period of many months. The reducing atmosphere associated with hydride cell operation eliminates corrosive lithium oxide, presumably minimizing corrosion by this factor. Where stresses are introduced, such as by bending, such stresses should be eliminated by annealing and quenching, or operation should be conducted outside the range where grain-boundary precipitation of carbon occurs. Cell operation below 480°C and regenerator operation at 980°C is thus possible for types 304 and 316 stainless steel for reasonable periods of time. For operation in the range which is critical for types 304 and 316, stabilized types 321 and 347, and the low-carbon type 304L are more suitable. Scaling is a problem in the high temperature operation of ordinary carbon steel.

As for electrical insulating materials, cell design has in the past provided for insulation to be accomplished in regions remote from the hot liquids in the cell. This has permitted relatively low temperature operation of fiber and neoprene o-rings in liquid level probe-type assemblies for insulating electrodes. These probe assemblies permit adjustment of the depth of immersion of the electrodes while providing electrical insulation. Where higher temperature operation of insulation is desired, porcelain spark plug components may be used in conjunction with a bellows-type adjustment for depth of immersion of the electrodes.

Mass transfer has been a phenomenon encountered in liquid lithium systems giving rise to rapid corrosion of stainless steel when there is a temperature differential and the lithium is flowing. This phenomenon is due to dissolution of nickel in high temperature regions and deposition of this metal in low temperature regions. This calls for use of other liquid metals with stainless steel, of carbon steel with moving liquid lithium with temperature gradients, or of regenerating from the solution of hydride in fused salt solvent. In the case of the hydride cell, the reducing atmosphere present in the cell eliminates the corrosive nitride and oxide of lithium, possibly permitting use of flowing lithium with even stainless steel.

B. Experimental Data. The experimental program has consisted of:

1. operation on a closed cycle basis, a unit previously built with MSAR funds
2. construction of a second unit designed on the basis of experience with the older one
3. operation of a batch-type cell with a metal electrode of potassium and as solvent the eutectic of the chlorides of potassium and lithium
4. differential thermal analysis of a ternary solution of halides of lithium
5. design and construction of two new batch-type cells.

Each of these phases will be discussed in turn.

1. Differential Thermal Analysis. One phase of the experimental program has as its purpose the determination of the composition of lower melting eutectics. This will be accomplished first by screening a number of possible systems for low melting points by thermal differential analysis, then analyzing the eutectic mixture by chemical analysis. Thermal differential analysis, in differentials between sample and inert reference as they are heated or cooled. Liquid residue remaining just before complete freezing is the eutectic, which can be analyzed chemically. As an example, the ternary eutectic involving halides of lithium might be determined by adding lithium iodide to the binary eutectic of the fluoride and chloride of lithium.

The apparatus used is shown in Fig. 2. It is sealed off to permit vacuum drying of the salts at elevated temperatures and to prevent absorption of moisture from the air. In one series of tests, sand was placed in the reference well and a mixture of 10% by weight of lithium iodide and 90% eutectic of the fluoride and the chloride of lithium. Complete reproducibility was not obtained, possibly because of segregation of components; but in a number of runs a break was shown in the range of about 395 to 430°C as is shown in the typical curves given in Figs. 3 and 4, the latter being 20% LiI. In effort to sharpen the break, sand will be eliminated as the reference to improve thermal conductivity to the reference thermocouple; and in effort to obtain better reproducibility, provision will be made to stir the sample while freezing.

2. Batch-type Cell. In order to determine the feasibility of a potassium hydride cell with the eutectic of the chlorides of lithium and potassium (41 mole % LiCl, 59 mole % KCl) melting at 352°C, a container made earlier with MSAR funds and shown in Fig. 5 was charged with the above eutectic and a potassium electrode holding 20 ml of liquid potassium. The usual spurious emf was found and persisted for a period of about 50 hours, including 17 hours with a hydrogen atmosphere and 5 hours with the terminals short-circuited through a Model 931 Weston Ammeter to reduce or electrochemically react out metal oxides probably causing spurious voltages. During this time, the micrometallic filter element on the bottom of the gas electrode had become clogged and was opened only by raising the cell temperature from 370°C to 420°C and the gas pressure raised to a value of the order of 30 psi. Later it was possible to operate at 377°C. After this interval, the emf with argon was down to 0.01 V as measured on a Model 931 Weston Voltmeter. When hydrogen was introduced into the gas electrode, the emf rose to 0.23 V in 12 minutes, then declined to 0.00 V in two hours. It was suspected that the first charge of potassium had been consumed; and a new charge of 22 ml was added. The emf promptly rose to 0.20 V, then to 0.26 V after about 90 minutes when argon was substituted for the hydrogen overnight. Apparently adding the fresh charge of potassium introduced a new spurious emf; for the following morning an emf of 0.21 V was measured with argon in the gas electrode. When hydrogen was introduced, the emf started to rise, reaching a value of 0.33 V after six hours. At this point, a load was put on the terminals taking only 55 A/ft² of gas electrode area at 0.06 V. On the following morning it was found that the cell had opened up because of excessive temperature arising from a malfunctioning controller.

In order to test in batch operation systems involving more expensive salts such as the borohydrides and the halides of cesium and rubidium, a more compact batch-type cell was designed and constructed.

3. Closed Cycle Operation. To determine operational characteristics of a closed cycle system designed and constructed with MSAR funds on the basis of data already collected prior to this contract in our work, operation was undertaken of the system shown in Fig. 6. Not shown are additional external calrod heaters and 3 in. thick Unibestos pipe insulation on all heated cells and pipes. Two 1500 watt calrod heaters were wrapped around the cell, and one 1000 watt heater around the bottom of the regenerator. One 1000 watt calrod was strapped to the top connecting pipe and two similar ones to the bottom connecting pipe. Container material was carbon steel as were initial connecting pipe material and electrode cup, which was packed with stainless steel mesh. The drawing shows 1/2 in. connecting pipes, which are of stainless steel and which replaced the initial 1/4 in. carbon steel pipes.

On charging the cell with solvent, eutectic proportions of the fluoride and the chloride of lithium (20 mole % Li F - 80% Li Cl), it was necessary to vacuum dry, then melt two charges before the liquid salt level came to the desired height.

After charging with lithium, initial operation was conducted with the regenerator at room temperature and only the cell at operating temperature of 480°C. A small spurious emf of opposite sign to the hydride emf was observed; but after passage of hydrogen for two hours a positive emf was obtained and reached a peak value of 0.55 V in another three hours with hydrogen pressure at one atmosphere. At this point, the voltage fell suddenly to zero and a vacuum developed in the cell. On cooling the cell, it was found that lithium had dropped from the top of the cell onto the hydrogen electrode and shorted it out. It was apparent that some lithium had held on the entry tube on charging and spread over the top of the cell. Subsequent charges of lithium were made by inserting the delivery tube further into the cell. On washing out the cell, it was found that the salt phase had cracked during the rapid cooling, permitting flow of lithium under the hydrogen electrode. One inch hexagonal blocks of steel 6 inches long were consequently placed below the weir to prevent such flow subsequently.

After recharging and heating, the 1/4 in. carbon steel connecting pipes between the cell and the regenerator developed leaks through overheating and were replaced with more heat resistant 1/2 in. stainless steel pipes. Lithium hydride was added to both the regenerator and the cell to give a hydride concentration of 5 mole % to the solvent in both sides of the system. This was done so as to obtain regeneration data as quickly as possible from the system which appeared to be deteriorating rapidly under operating temperatures.

Upon operation again, spurious voltages as high as one volt were observed and required over nine hours to be eliminated with passage of hydrogen and drawing of electrical energy. When the true hydride emf had developed, there was a fluctuation in emf, the speed of which varied with rate of flow of hydrogen. It was further noted a vacuum developed unless a minimum flow of hydrogen was maintained, indicating chemical consumption of hydrogen by the lithium. During this operation, a peak value of only 0.27 volt was obtained with one atmosphere of hydrogen.

It was then decided to attempt to run the cell regeneratively. Argon gas purged all free hydrogen, the system was closed off and the regenerator temperature raised. With argon purge gas flowing, the emf was less than 0.1 mv. The cell was run for seven days regeneratively: and voltages generally in the range of 5 to 7 mv were observed, on one occasion as high as 30 mv. At such small voltages, current densities were considered to be of no practical interest and therefore were not measured. Cell temperatures were maintained generally around 495 to 505°C and regeneration temperatures 705 to 760°C. On one occasion, the cell was evacuated with a Welch Duo Seal vacuum pump to see if the longer mean free path of the hydrogen evolved from the regenerator would help improve the emf by permitting faster transfer from the regenerator to the cell. No significant improvement was observed.

During regenerative operation, two samples of gas were withdrawn from the regenerator while voltage readings of 2.4 mv and 5.0 mv were being obtained. Hydrogen content of the argon cover gas was determined to be 0.008% and 0.009% respectively by use of a Consolidated Engineering Corporation Model 21-620 Mass Spectrometer.

After the above-mentioned period of closed cycle operation, a leak developed in a bend in the stainless steel connecting pipe, and operation had to be discontinued. Metallographic examination showed the defect to be due probably to a combination of stresses in a bend with operation at a temperature of 480°C, causing precipitation of carbon at grain boundaries. As was discussed under Materials of Construction (p8) design to permit operation outside this critical temperature causing carbon precipitation will relieve this situation in stressed regions.

4. Redesign of Closed Cycle System. As a result of experience with the first closed cycle system, a new unit was designed and constructed. This is shown in Fig. 7.

Changes in design include use of stainless steel in all high temperature regions, a decrease in size of the larger components, use of a larger weir between the gas electrode and the metal electrode to minimize possibility of passage of liquid metal through cracks in cooled salt, use of a Kanthal external heater for the regenerator to obtain higher temperatures, sealing the top of the weir to the top flange to permit maintenance of an inert atmosphere over the lithium, and use of 1/2 in. stainless steel connecting pipe instead of 1/4 in. pipe to improve liquid circulation.

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VI. CONCLUSIONS

A lithium hydride cell has been made to operate regeneratively.

A potassium hydride cell has been made to operate with a peak open circuit voltage of 0.33 V having been obtained in a first attempt.

Differential thermal analysis has shown promise of obtaining a lower melting lithium halide eutectic by addition of LiI to the LiCl-LiF binary eutectic.

A regenerative system will be designed so that regeneration from the metal phase can be studied.

VII. PROGRAM FOR NEXT QUARTER

An attempt will be made to operate a hydride cell on the low-melting eutectic of the borohydrides of lithium and potassium as solvent.

The quaternary eutectic of the chlorides of rubidium, cesium, potassium and lithium will be used as solvent in a lithium hydride cell.

The newly constructed unit will be operated regeneratively at higher temperatures to obtain operational and life data using at first the lithium hydride system.

Work will continue to screen lower - melting eutectics, first by thermal differential analysis, then by chemical analysis.

Further studies on alternate anode materials will be made, with sodium and with potassium in other solvents such as halides of the metal constituting the anode material.

DISTRIBUTION OF HOURS

T. A. Ciarlariello	47 hours
J. C. King	315 hours
J. W. Mausteller	21 hours
R. E. Shearer	209 hours
R. C. Werner	14 hours
Drafting and Hourly	<u>421 hours</u>
	1027 total hours

IDENTIFICATION OF PERSONNEL

Dr. R. C. Werner -- Associate Director of Research

Received his B. S. in Chemical Engineering and Engineering Math from University of Michigan in 1938. He received his M. S. and Ph. D. in Chemical Engineering from the University of Michigan in 1939 and 1943. He was employed by the Blaw Knox Company from 1942 to 1944. Dr. Werner joined Mine Safety Appliances Company in 1944 as the Project Supervisor. and later was made Supervisor of Development Engineering. In 1957 he was appointed Operations Manager and Associate Director of Research. Dr. Werner is responsible for the original concept of a thermal'y regenerative galvanic cell and will be monitoring all phases of the contract.

Dr. J. W. Mausteller -- Research Manager

Dr. Mausteller received his B. S. degree in Chemical Engineering from Bucknell in 1944, his M. S. and Ph. D. degrees were obtained from Penn State University in Physical Chemistry in 1949 and 1951 respectively. He was first employed as a Chemist by E. I. duPont De Nemours and Co. , 1944 to 1947; then as a Research Assistant in the fluorine labs of Penn State University from 1947 to 1950. In 1950 Dr. Mausteller came to Callery Chemical Co. as a Research Chemist and then as an Engineer in 1951. He was a Project Engineer from 1952 to 1955. From 1955 to 1957 he was a supervisor for Mine Safety Appliances Co. Since 1957 Dr. Mausteller has been Research Manager for MSA Research.

Robert E. Shearer -- Research Chemist

Mr. Shearer received his A. B. degree (cum laude) from Marietta College in 1937. He then did graduate work at John Hopkins University, 1938 to 1939, and at West Virginia University, 1939 to 1941. In 1954 he attended the Oak Ridge Institute of Nuclear Studies. He was employed as a Research Chemist and later as Supervisor at Union Switch and Signal from 1941 to 1957. Since 1957 Mr. Shearer has been employed as a Research Chemist by MSA Research Corporation. He is co-inventor with Dr. Werner of the thermally regenerative galvanic system.

Thomas A. Ciarlariello -- Chemical Engineer

Mr. Ciarlariello received his B. S. degree in Chemical Engineering from the University of Texas in 1951. He graduated from ORSORT in 1957. From 1951 to 1953 he was a Design Engineer for NACA. He was then employed as a Shift Foreman for B. F. Goodrich Chemical Co. in 1953. He came to MSA Research Corp. in 1953 and is presently employed as Development Engineer.

John C. King -- Associate Chemist

Mr. King received his B. S. degree in Education at Clarion State Teachers College in 1952. He received a G. E. Science Fellowship at Union College in 1953 and was a Science Teacher from 1953 to 1955. He has been an Associate Chemist with MSA Research Corp. since 1955. He has done much of the assembly work and testing leading to reduction to practice of the thermally regenerative galvanic system.

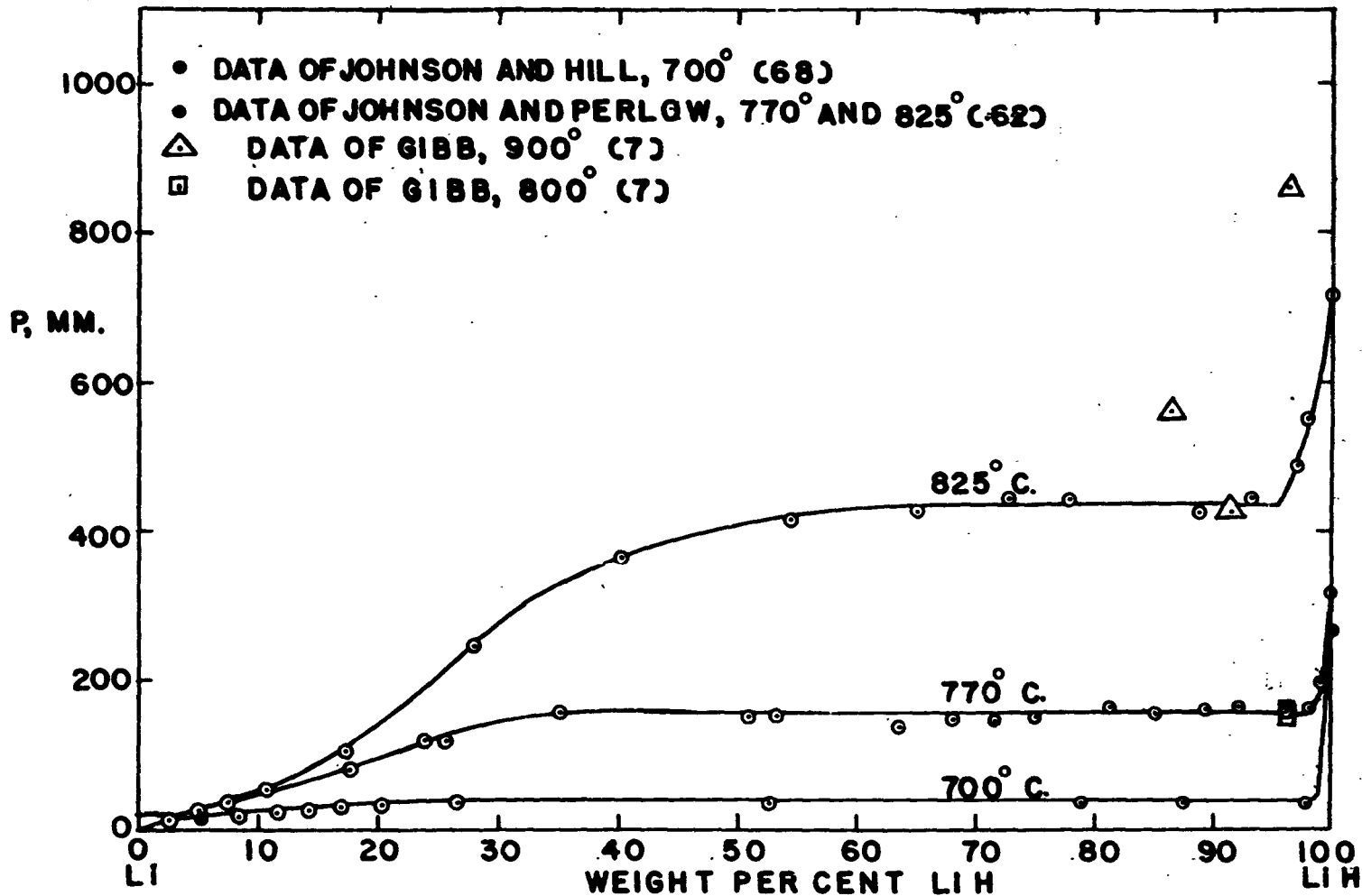


Fig. 1 - Dissociation Pressures of Lithium-Lithium Hydride System

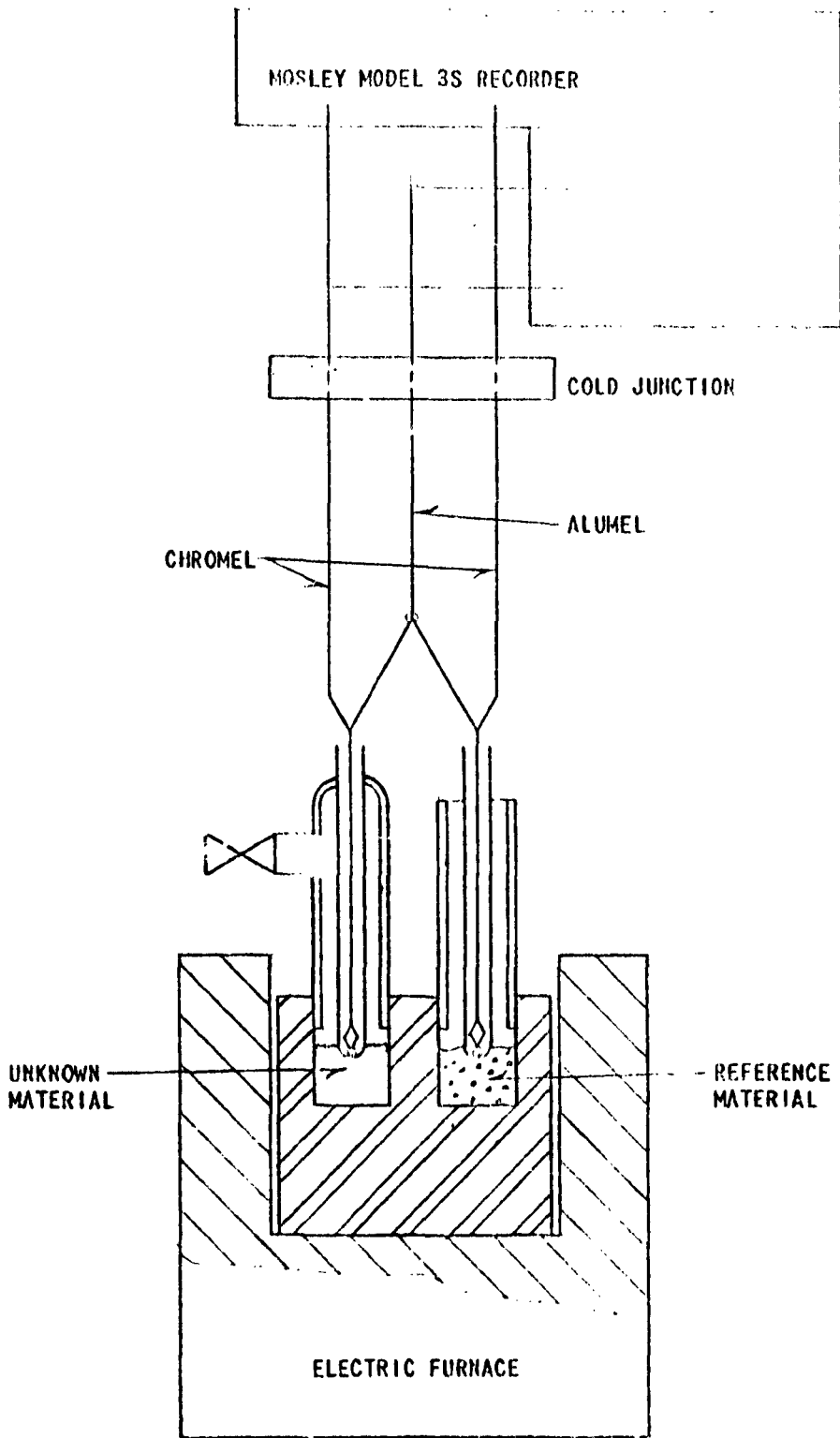


Fig. 2 - Thermal Differential Analysis Arrangement

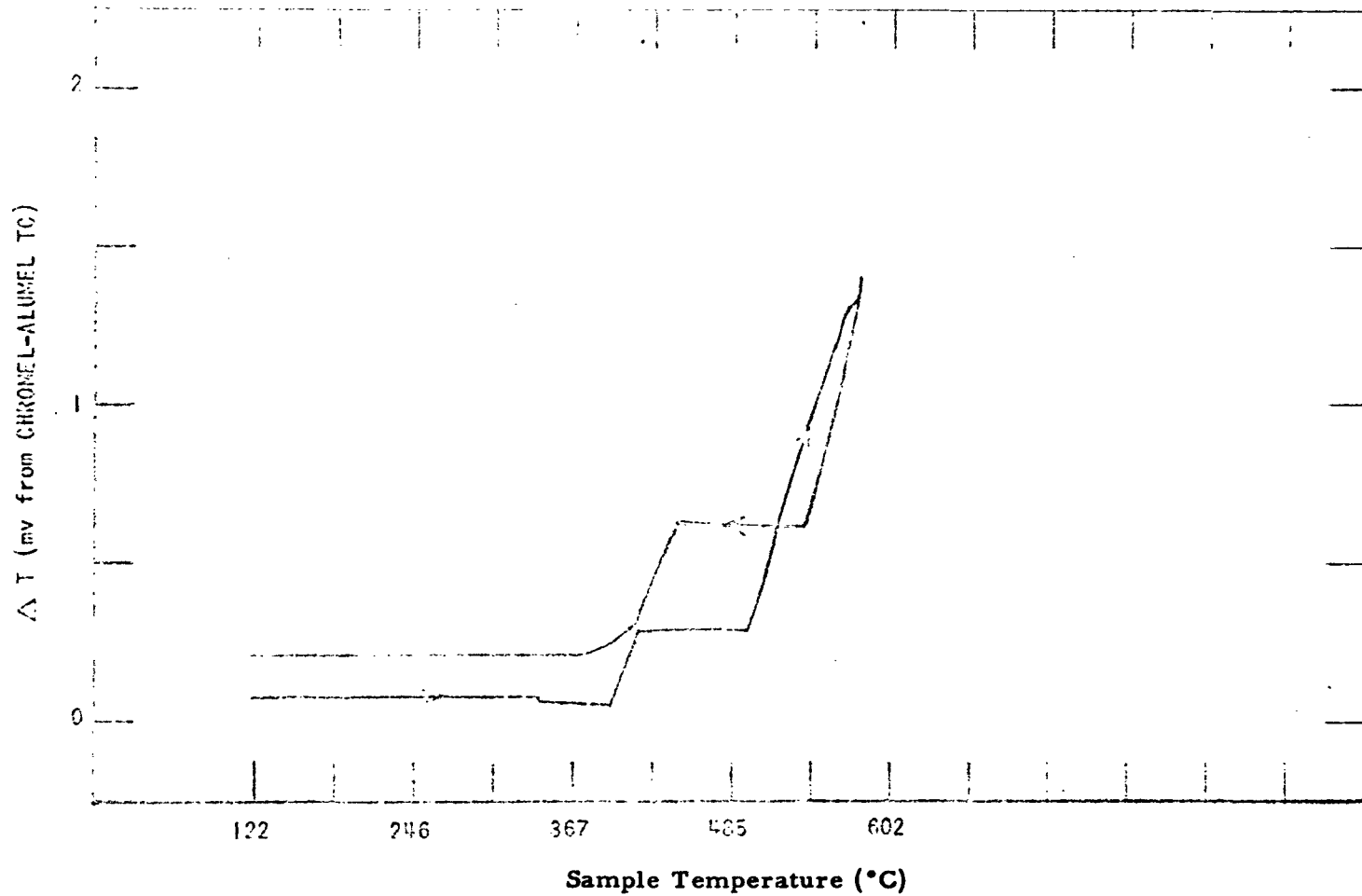


Fig. 3 - Sample Temperature vs Temperature Differential Between Reference and Sample of 10 wt. % LiI and 90% Eutectic of LiF%LiCl

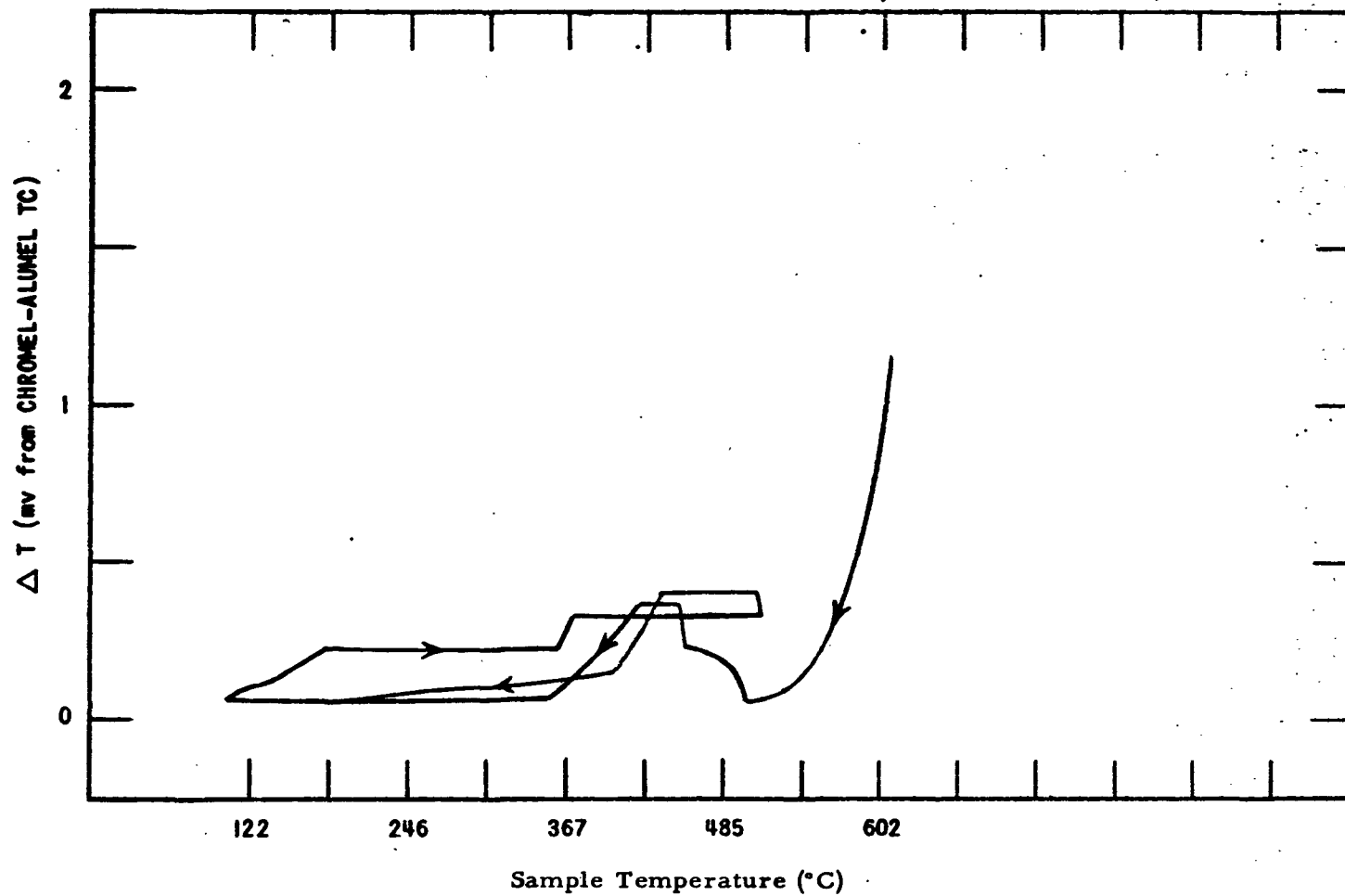


Fig. 4 - Sample Temperature vs Temperature Differential Between Reference and Sample of 20 wt. % LiI and 80% Eutectic of LiF-LiCl

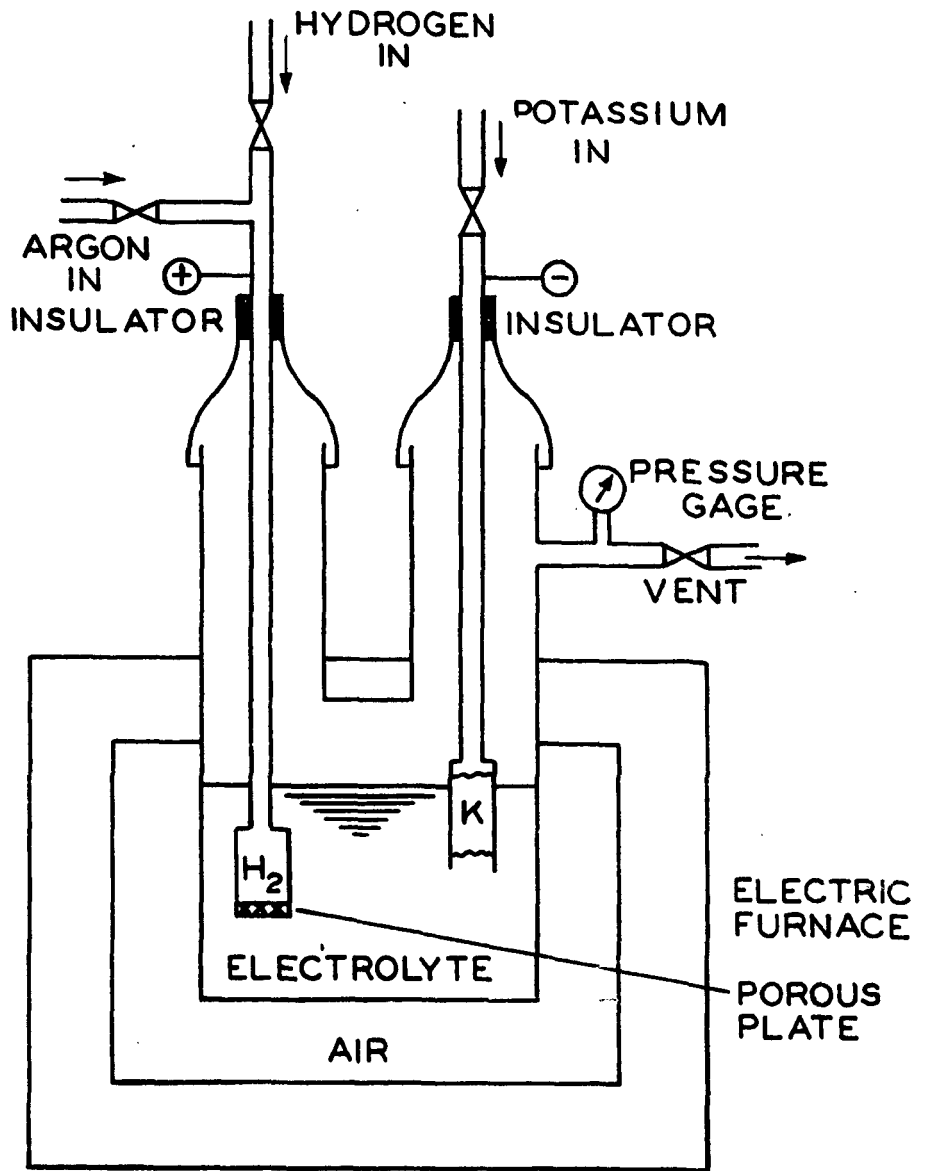


Fig. 5 - Sketch of Potassium Hydride Cell

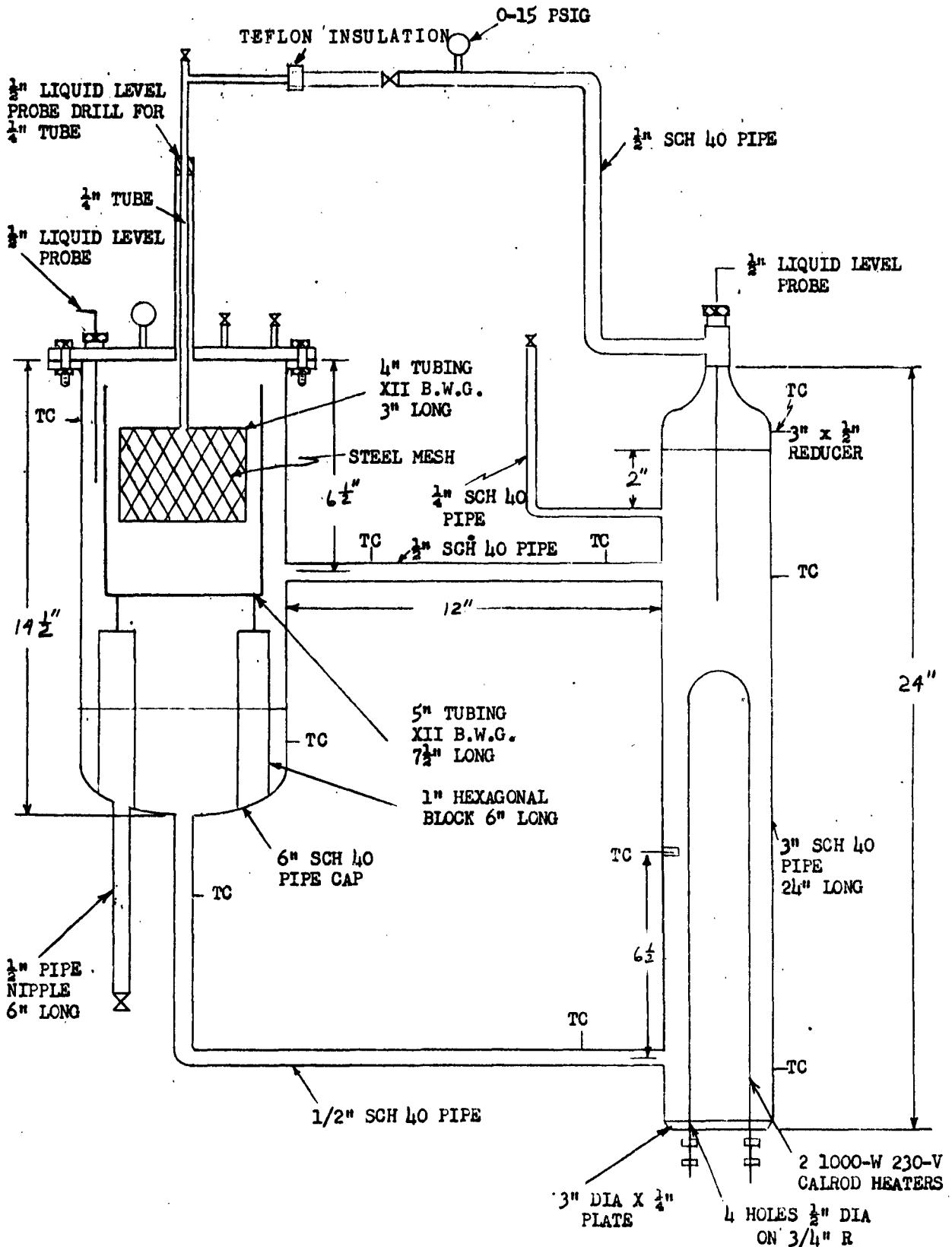


Fig. 6 - Regenerating Fuel Cell

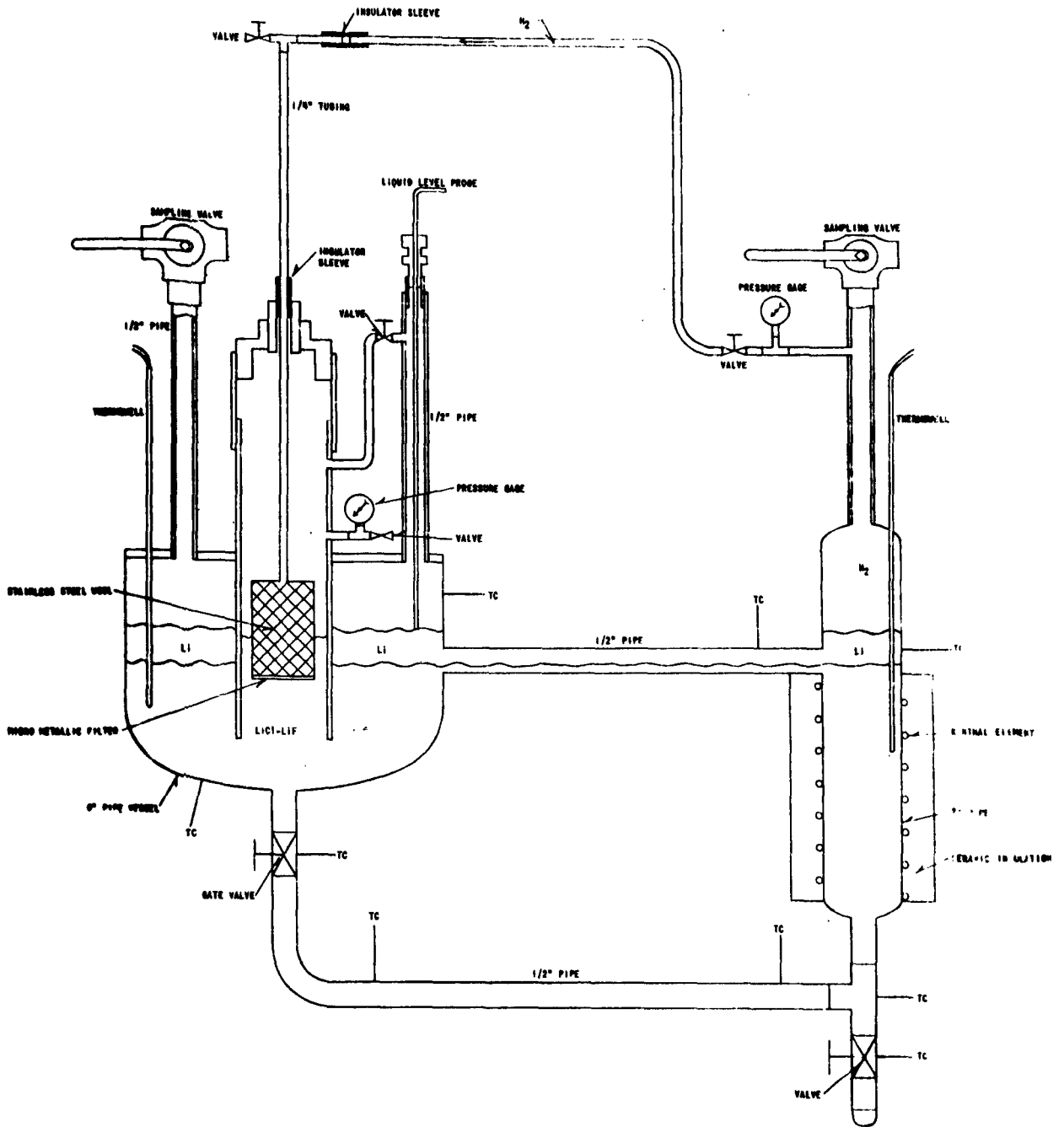


Fig. 7 - Regenerating Fuel Cell (Revised)