THE USE OF UNIDIRECTIONAL PERMEABLE ION EXCHANGE MEMBRANES

FOR SALINE REDUCTION OF ALKALINE FARM WATER SUPPLIES

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PREFACE

The experimental results described herein were obtained as a part
of the Oklahoma Experiment Station Project No. 798, entitled "Farm
Water Supply Development". One of the objects set forth in this pro-
ject is to "Develop A Satisfactory Water Treating Plant for Farm Use".

This particular phase of the research project deals with the
removal of mineral hardness of natural underground waters. To date no
continuous process, other than distillation, is known to exist for
removal of mineral hardness. Present information indicates this method
is unfeasible for farm water supply use.

Of some thirty-three potential separation processes the electro-
ion-migration process appears most promising for treatment of rural
alkaline water supplies. This has been made possible through the
recent development of permselective membranes.

Membranes used in this study, which are ion exchange resins in
film form, have been made available from Rhom and Haas Company,
Philadelphia, Pennsylvania.

The author is especially indebted to Quintin B. Graves, Professor
of Civil Engineering, for wise guidance and helpful criticism in
acquiring and developing research equipment. His co-operation is also
gratefully acknowledged in the correlation of experimental results.

Many thanks are due Messrs. Lawrence Paxton, Frank Smith, Frank
Hedges, Samuel Miller, Edward Burd, Darrel Hobson, and Charles Sarkany,

operators of the water plant, without whose co-operation these experiments could not have been effected.

Helpful contributions were also made by Messrs. James E. Garton, Professor of Agricultural Engineering, Dr. V. G. Heller, Professor of Agricultural Chemistry, and Erwin W. Schroeder, Professor and Head, Agricultural Engineering.

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CHAPTER I

INTRODUCTION

A most important problem confronting the farm electrification engineer today is that of providing a satisfactory water supply for domestic use. Rural electrification has made possible the modernization of farm living. This has increased water usage from 4 to 6 gallons per day per person when pumped by hand as compared to 35 to 50 gallons per person per day with an automatic water system.

As a result, many farm wells are no longer dependable which, in the past, have yielded a sufficient supply of water for home use.

Much of Oklahoma underground water is heavily laden with alkaline salts and other undesirable minerals which render it unfit for household, irrigation, or farm purposes. Reduction of mineral content to useable portions by distillation is expensive. The development, in recent years, of granular ion exchange resin beads offer a feasible solution. Regenerating these resins involves stopping the demineralizing process at regular intervals and using concentrated chemicals to recharge the resin beads. Technical skill is also needed in this process.

Permeable ion exchange membranes, which have more recently been developed, are 90 percent or more unidirectional. The cation or negative membrane will permit the flow of 90 percent or more of the cations or negative charges and will restrict the flow of 90 percent or more of the anions or positive charges. Conversely, the positive membrane will permit the flow of the anions and restrict the cations.

Underground shale and clay are known to posses selective ion permeability. Living processes are dependent on permselectivity of plant and animal membranes. Thus it is possible that these membranes, using low voltage direct current as a form of energy, may offer a practical, economical method of rendering suitable highly mineralized waters for household use.

Ion-exchange is the name given to a reversible chemical reaction that takes place between an insoluble substance and a solution. Ions were first named by Faraday from a Greek word for "traveler". Ions composed of atom groups who have lost one or more electrons are positively charged and are known as cations. The other group, which has gained extra electrons, is known as Anions and are negatively charged.

Cation exchangers, if placed in a solution containing salt, will exchange some of the salt for the positive ions of the exchanger. The anion exchanger in the same solution will exchange anions with other anions in the solution. Thus it is possible to use an ion exchanger to chemically remove an unwanted substance from a solution.

CHAPTER II

REVIEW OF PREVIOUS LITERATURE

Little has been written concerning the use of ion permeable membranes using electric current for mineral reduction of potable water.

L. Michaelis[1], as early as 1925, noted that well dried collodion membranes (low porosity) showed a differential permeability for cations and anions. The cations penetrated more rapidly than the anions. He further states that differential ion permeability gives rise to what is known as anomalous osmosis. That is, under certain conditions, water will flow from a concentrated to a dilute solution when the two solutions are separated by a porous membrane. Osmosis or the movement of a solvent through a membrane into a more concentrated solution on the other side of the membrane is perhaps the earliest accounting of the action of permeable membranes. Living cells have such membranes and depend on osmosis for much of their activity.

Erbe[2] speaks of a method for measuring the pressure required to replace a liquid in a membrane by another immiscible liquid. He defines the forces necessary as 1.8 dynes per sq. cm. at 25 degrees.

Wyllie and Patnode[3] in an article published in the Journal of Physical Chemistry, Volume 54, page 204, describe a number of studies on ion permeable membranes. The following information is taken from this article.

Experiments carried out with shales have led to the development of artifical membrane electrodes based on the assumed electro-chemical structure of shales.

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At the beginning, a finely ground Woodford shale was mixed with an inert insulating bonding resin. Best results were obtained by using a mixture of 30 percent by weight of shale of the weight of methyl methacrylate resin. Later experiments included Wyoming Bentonite, a specimen of illite from Fithian, Illinois and fullers earth. Of these three materials, Bentonite appeared to be the best.

The principal difficulty associated with the use of Bentonite was the time lag for a state of equilibrium. Also the swelling of the clay particles caused rupture of the membrane. These experiments were later followed by membranes prepared from two commercial cation-exchange materials. The first resin was a phenol-formaldehyde type made by Rohm and Haas Company of Philadelphia. The second resin was a sodioum Zeokarb made by Permutit Company of New Jersey. A pressure of 3,000 pounds per square inch with a temperature of 150° was used in forming the membranes.

Results in using these membranes indicated that the total quantity of cation-exchange material in a membrane had no effect on its electro-chemical properties.

Sollner[4], in his work with permselective membranes, shows that the electrical resistance of such membranes varies according to the solutions.

Science News[5] states that cation exchange resins are now being used to remove salt from the human body in dropsy cases and later this may prove to be an accepted practice. They further state that Anion-exchange resins are now being used in treating stomach ulcers. This is accomplished by "binding" the acid in the stomach.

Agricultural Research[6] states that certain chemically modified cotton fabrics, including phosphorylated and aminized cottons, have ion-exchange properties similar to those of ion-exchange resins. Such fabrics are useful as both filters and ion-exchangers. They can physically remove particles suspended in a solution, and at the same time chemically displace another substance through ion exchange. These fabrics make possible moving-belt ion-exchange systems, with continuous regeneration as part of the process, that work faster than conventional systems using resins.

Hoffpauir and Guthrie[7] relate their experiences in preparing protein from oil seed meal. Protein from meal is usually prepared by dispersing the protein at a PH more basic than its isoelectric point, separating the undispersed material, and precipitating the protein by lowering the PH to about the isoelectric point. Increasing and decreasing the PH is ordinarily accomplished by the addition of an acid and a base. The work reported is summarized as follows:

Protein preparations, low in ash and phosphorus content, are made by the use of anion exchange materials to increase the PH of oil seed meal suspensions and cation exchange materials to reduce the PH of the protein dispersion to the isoelectric range. Preparations very low in ash and phosphorus may be obtained by the alternate use of aminized cotton fabric and phosphorylated cotton fabric until deionization is almost complete prior to separation of the protein curd. The method is also useful for the purification of protein preparations made by the usual methods. While commercial ion exchange resins may be used, the results with the fabrics have been better. The fabrics are also more convenient and are readily recovered and regenerated.

Phosphorylated cotton, a typical cation exchange cotton was originated by Ford and Hall[8] for the purpose of obtaining a flame resistant cotton fabric. The aminized cotton fabric, a typical anion exchange fabric was first recognized for its dyeing properties.

Phosphorylated cotton fabric, the cation exchange material, may be prepared by wetting cotton fabric with a solution containing 49.6 percent urea, 18.4 percent phosphoric acid, and 32 percent water, squeezing out the excess solution, heating the fabric for 30 minutes at 150°, and washing thoroughly. The aminized cotton fabric, the anion exchanger, may be prepared by wetting cotton fabric with a solution containing 25 percent sodium hydroxide, 10 percent 2-aminoethylsulfuric acid, and 65 percent water, heating in a drying oven for 40 minutes at 100°, and washed thoroughly.

Osborn, Collings, and Brake[9] describe a method of softening water by electrical treatment. The water to be treated is passed through a tube supporting an electrode assembly consisting of a series of superposed annular knife-edge elements, insulated from one another, and so arranged that the knife edges are the only part of the electrodes presenting a bare metal surface to the water. The first and last elements are connecting to a source of unidirectional current at 0.0001-0.001 watts dissipation per element.

Spiegler, Juda, and Carron[10] reporting pilot plant data on partial demineralization by ion exchange indicate that this method should prove most economical for waters containing less than 2,000 parts per million dissolved solids. They further state that the cost of demineralization by this method is estimated to be 60 cents per 1,000 gallons treated with 1,000 parts per million solids (as Na Cl) removed.

Howe[11] reporting on work conducted at the University of California on the desalting of sea water describes three different methods. The first, called diesel-waste heat plant, has low first cost, low operating cost and uses conventional equipment. It is limited to localities using diesel plants and could furnish on the average, not more than one-seventh of the average water demand.

The second, called the temperature-difference plant, was not described. The author states that this unit needs much development.

The third unit, described as the solar-difference plant, is described as the most promising of all. No significant information on costs and operational problems is as yet available.

The author further states that the membrane method seems attractive in some ways. A decision about its promise can be made when additional information is available.

Pike[12] reports that the Navy, in co-operation with Massachusetts Institute of Technology, has in operation an experimental plant for desalting of sea water. The process is described as being continuous and only electrical and mechanical means are involved in the purification. No moving parts are used and about two-thirds of the water that flows into the unit is purified. Electrical consumption is, at present, some 20 KWH per 1,000 gallons. No detailed information is given. The United States Department of Interior[13] reports on a saline water research program as authorized by the 82nd Congress, Public Law 448. The object of this program is to coordinate and stimulate research and development of economically feasible processes for saline reduction of alkaline waters. This law authorizes the expenditure of $400,000 annually for five years.

The report further states that present irrigation water rates range from a few cents per acre foot to as much as $39.00 per acre foot. The majority of private sources range from $1.50 to $6.00 per acre foot.

Municipal water rates range from a few dollars per acre foot to more than $115.00 with the average between $50.00 and $75.00.

Aultman[14] reports on desalting of sea water and states that, for operation costs only, the resinous ion exchange bead method will amount to $25.00 per 1,000 gallons. He further states that operation costs for the evaporative method will range from $2.60 to $1.07 per 1,000 gallons. Operation costs for the electrolytic method is given as $0.90 per 1,000 gallons.

Chemical and Engineering News[15] reports on the use of a perm-selective membrane developed by Ionics, Inc., that has an electrical conductivity of the order of electrolytic solutions. It was stated that sea water would require some 20 KWH for desalting.

The second annual report of the Secretary of Interior[16] describes a permeable membrane electrolysis cell containing 101 membrane compartments. This cell employs cation and anion membranes manufactured by Rohm and Haas Company.

A Saline Water Research and Development Summary[17] dated January, 1954, lists some 65 organizations engaged in research and development of saline water reduction. Some ten of these are foreign. Seven of these separation processes employ ion permeable membranes with electric current.

Dr. Juda[18] describes a six months contract of Ionics, Inc., with the Department of Interior. This contract began January, 1954 and

dealt with demineralizing saline water with electric current and ion permeable membranes. The objective of the contract was to make a preliminary type engineering cost estimate for producing ten acre-feet of saline free water per hour. Plans are to install pilot plants in South Dakota, Arizona, and Texas. These plans are predicated upon an estimated installation cost of $1,700,000 to $21,000,000 per plant. Plant costs vary due to the saline content of each water. Brackish water analysis at each proposed site indicates the South Dakota water to contain 885 P.P.M., and the water of Arizona to contain 4,635 with the Texas water to contain some 10,000 P.P.M. salts.

There are some 33 potential separation processes and phenomena as listed by the Department of Interior[19]. The Department also lists 16 potential energy sources. The separation processes and energy sources are as follows:

POTENTIAL SEPARATION PROCESSES AND PHENOMENA

I. Physical Processes and Phenomena

    A. Vaporization

        1. Vapor-compression distillation.
        2. Single and multiple-effect evaporation.
        3. Flash evaporation.
        4. Flash-type multiple-effect evaporation.
        5. Combination compression distillation and multiple-effect evaporation.
        6. Critical pressure devices.
        7. Superheated steam.
        8. Underwater combustion.
        9. Temperature differences.

    B. Crystallization

        1. Freezing of water.
        2. Crystallization of salts.

    C. Sublimation

    D. Adsorption

       1. Adsorption of water.
       2. Adsorption of ions.

    E. Diffusion Effects

       1. Thermal diffusion.
       2. Gravitational diffusion.

    F. Ultrasonics

    G. Osmosis

       1. Molecular oil films.
       2. Biological membranes.

    H. Immiscible Liquids

II. Chemical Processes and Phenomena

    A. Ion-exchange

    B. Hydration

    C. Precipitation

III. Electrical Processes and Phenomena

    A. Electro-ion-migration

       1. Electrolysis - 2 and 3-compartment cells.
       2. Perm-selective membranes.
       3. Electro-gravitational methods.

    B. Streaming Potential

    C. Electostatic Effects

       1. Migration in electrostatic field.
       2. Dielectric effects.

    D. Electromagnetic Effects

       1. Migration in electromagnetic field.
       2. Magnetic susceptibility.

    E. Ultra High-frequency Currents

POTENTIAL ENERGY SOURCES FOR DEMINERALIZATION

I. Combustion of Fuels

    A. Conventional

    B. Non-conventional

II. Natural Hydraulic Potential (falling water)

III. Utilization of Waste Heat

    A. Waste Heat From Industrial Processes

    B. Waste Heat From Nuclear Fission

IV. Nuclear Fission

V. Solar Energy

VI. Marine Energy

    A. Thermal

    B. Wave

    C. Tidal

    D. Electromotive Force

VII. Wind Power

VIII. Chemical Energy

IX. Atmospheric Heat

X. Geothermal Energy

DISCUSSION OF POTENTIAL SEPARATION PROCESSES AND PHENOMENA

I. Electrical Processes and Phenomena

    A. Electro-ion Migration

        The theoretical minimum energy required to cause separation of the salt from normal sea water, as reported by the Department of Interior[19], is approximately 3 kilowatt hours per thousand gallons. Practical electro-ion processes employing

relatively high rates of flow are needed.

The compendium also states that basic research is needed to determine the probable lower limit of energy needed which has been estimated at from fifty times to as low as five times the theoretical minimum.

Production of valuable compounds or minerals can reasonably be expected as a by-product of saline reduction of alkaline waters.

Perm-selective membranes using electric current as a source of energy, for saline reduction is a recent advancement, so the report states. If, in an electrolytic cell, the space between the electrodes is divided into a large number of compartments by alternately using cation and anion membranes a method of demineralization is obtained. The flow from these alternate compartments will consist respectively of water of reduced mineral content and an enriched brine. The amount of the current needed is reduced as the number of compartments is increased between the electrodes. Hence, the power required is a function of the number of compartments, the resistance of the unit, and the rate of hydraulic flow. The power for this process is not independent of the amount of salts present; however, it is not directly proportional to the number of equivalent parts per million present in the solution. The cost of such a process, so states the report, has been estimated at from 30 to 60 KWH per 1,000 gallons.

CHAPTER III

OBJECTIVES

The purpose of this study was to develop information on the feasibility of using ion permeable membranes for mineral reduction of domestic water supplies for rural areas.

Specific objectives were:

1. To determine optimum voltage across membranes in an alkaline solution for most effective results, using the minimum of electrical energy.

2. To determine the rate of mineral reduction of alkaline water solutions under varying conditions.

3. To measure electrical energy used for six of the most common mineral compounds found in rural household water supplies.

4. To measure the time rate of electrical energy in KWH/1000 gallons of an alkaline water supply.

5. To correlate these readings with completed theoretical electrical energy needed and determine the efficiency of permeable membranes in mineral reduction of alkaline waters.

6. To reduce the mineral content of a known water supply. Then compare the reduction of each of the six chemicals in this solution to the reduction obtained separately as in item 3.

7. To determine the optimum current per unit area of membrane surface to be used in mineral reduction of alkaline rural water supplies.

CHAPTER IV

THEORY OF PERMSELECTIVE MEMBRANES

When using ion permeable membranes for mineral reduction the action or process occuring at the anode and cathode are oxidation and reduction respectively, which is the removal or addition of electrons.

The ion velocity in a solution is proportional to the applied potential divided by the distance between the electrodes. Salts, when dissolved in water, split up or disassociate in such a way as to furnish two kinds of ions, metal and non-metal. The metallic ion (electro-positive) is often known as the cation while the non-metallic ion (electro-negative) is called the anion. This is the basis of the electrolytic theory of disassociation. Micheal Farady, in 1833, obtained results in this field which are now recognized as laws. They are as follows: (1) The amount of chemical change produced by an electric current, that is, the amount of any substance deposited or dissolved, is proportional to the quantity of electricity passed. (2) The amounts of different substances deposited or dissolved by the same quantity of electricity are proportional to their equivalent weights. This may be written as $W = \dfrac{I\,E\,T}{F}$. Transposing; $F = \dfrac{ITE}{W}$ where

    M = weight of substance in grams
    I = current strength in amperes
    E = equivalent weight
    F = quantity of electricity necessary to dissolve one gram
        equivalent of any substance
    T = time in seconds

For silver the equation reads $F = \dfrac{107.88}{0.00118} = 96{,}500$ ampere seconds which

is known as 96,500 coulombs or one faraday. This makes possible the determination of solubilities by conductance measurements and is a common laboratory procedure on solutions undergoing simple disassociation into ions of known conductance. Electricity may flow through a material or substance electronically, similar to the transfer of electric energy into the home by the electric utility service. In this case the energy is transferred by electron movement without the physical movement of the wires carrying the current. In electrolytic conduction, the ions are actually transferred through an aqueous solution. The ampere efficiency of such an electrochemical reaction may be computed by dividing the theoretical number of faradays required by the number actually used to complete this reaction.

The membranes used in this study are a recently developed product of the Rohm and Haas Company of Philadelphia, Pennsylvania. At moderate and high concentrations, the membrane conductance is less than that of the solution and at low solution concentrations, the membrane conductance approaches and may exceed that of the solution. These characteristics are desirable for mineral reduction of alkaline waters. Until these membranes were developed, granular cation and anion resinous beads were used in such processes. This required batch operation. Between steps the granular resinous beads were regenerated with an acid and a base. The new membranes, mentioned above, make possible continuous operation and eliminates the need for regeneration. In addition, these membranes have high permselectivity. The cation membrane allows passage of approximately 90 percent of the cations and retains approximately 90 percent of the anions. Conversely, the anion membrane will pass 90 percent or more of anions and retains 90 percent

or more of the cations.

These membranes, due to their excellent mechanical durability, their resistance to concentrated solutions of acids, bases, and salts, and to their high ion selectivity, appear to offer excellent possibilities in the saline reduction of alkaline water for farm water supplies.

CHAPTER V

APPARATUS AND METHOD OF PROCEDURE

Sample membranes, four inches square in size, were first obtained in February, 1953, from Rohm and Haas Company.

These membranes were used in a three compartment cell connected to a 6 volt automobile battery, as shown in Figures 1 and 2. Preliminary tests were conducted with known solutions of single compounds such as calcium sulfate, calcium chloride, magnesium sulfate. Observations were made on rate of current flow with regard to position of electrodes, rate of demineralization, deposition of minerals in end cells and erosion of electrodes. Results obtained in these preliminary runs indicated that, for all practical purposes, the electrodes should be located toward the outer ends of the cell. The conductivity in the outside cells increased during the current flow. The limiting factor to flow of electric current was the conductance of the membranes and the conductivity of the solution in the center compartment which decreased as the test continued. This was due to the removal of solids in the center compartment and the deposition of these solids in the outer compartments.

The first cell shown in Figure 1a was constructed from 1/8 inch plexiglass sheet and cylinder material, held together with four 1/8 inch brass rods placed equidistant around and parallel lengthwise to the cylinder. Extreme difficulty was encountered in obtaining a water-tight seal around the membranes. This was finally accomplished by
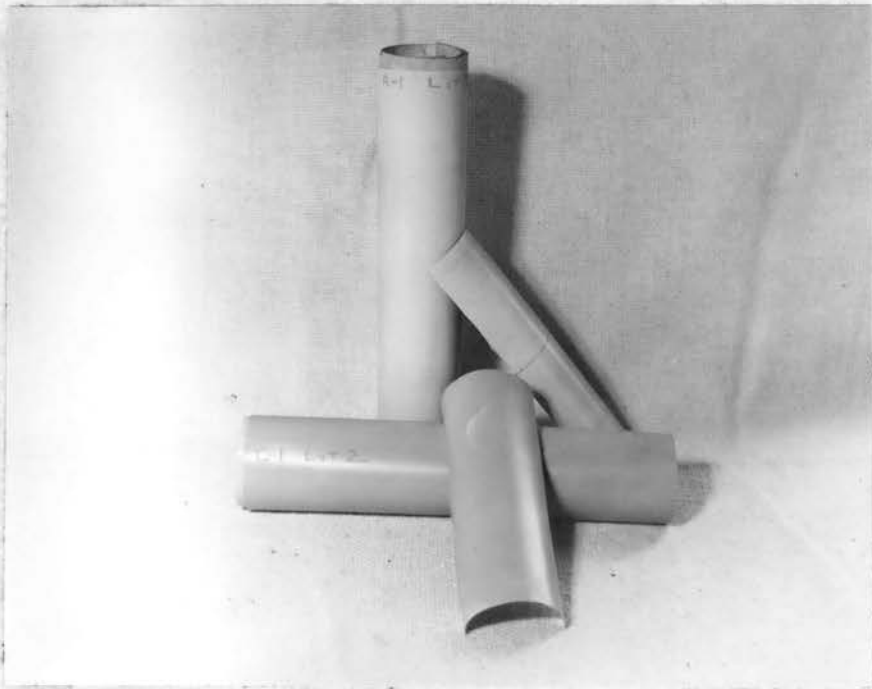
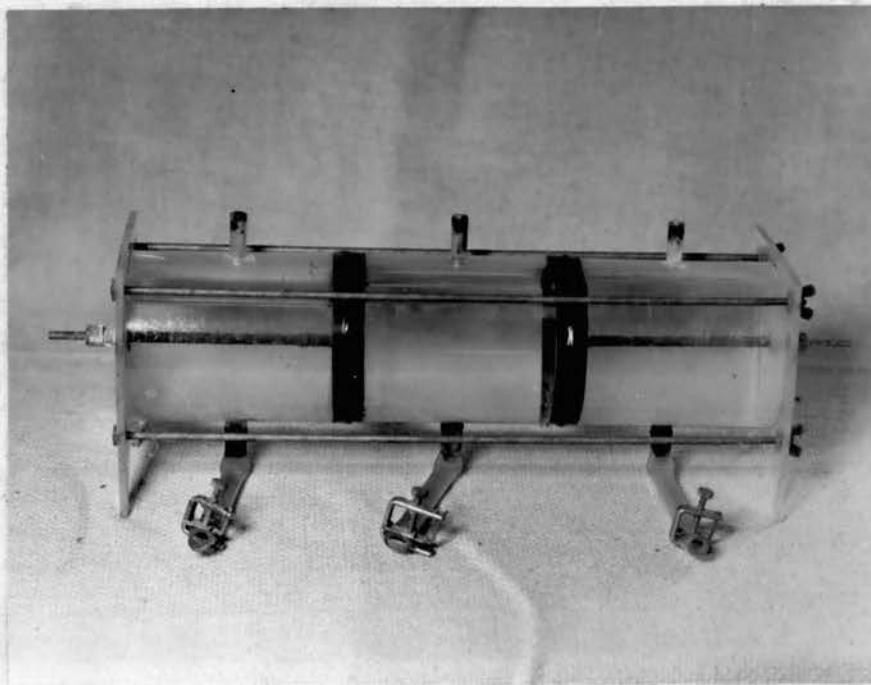Fig. 1 – Permselective membranes used in saline
reduction of alkaline waters.

Fig. 1a – Enclosed primary electro-dialyzer cell,
three compartment, of plexiglass.

BEFORE

DURING

AFTER
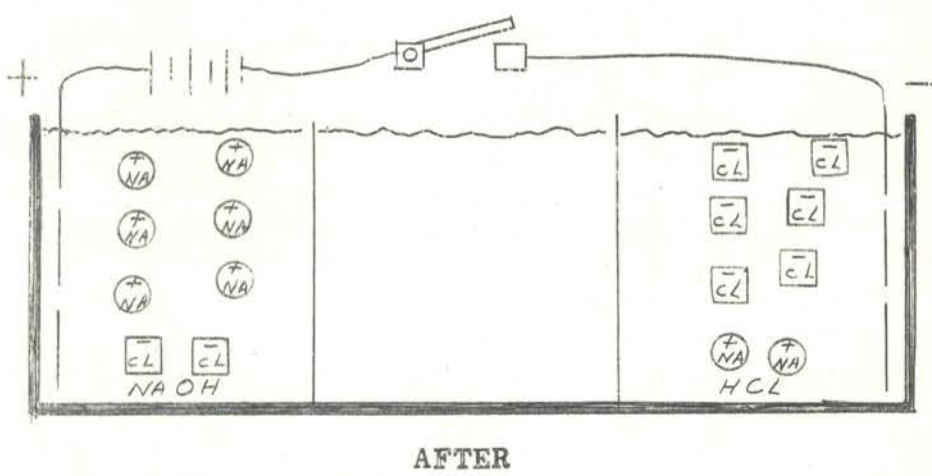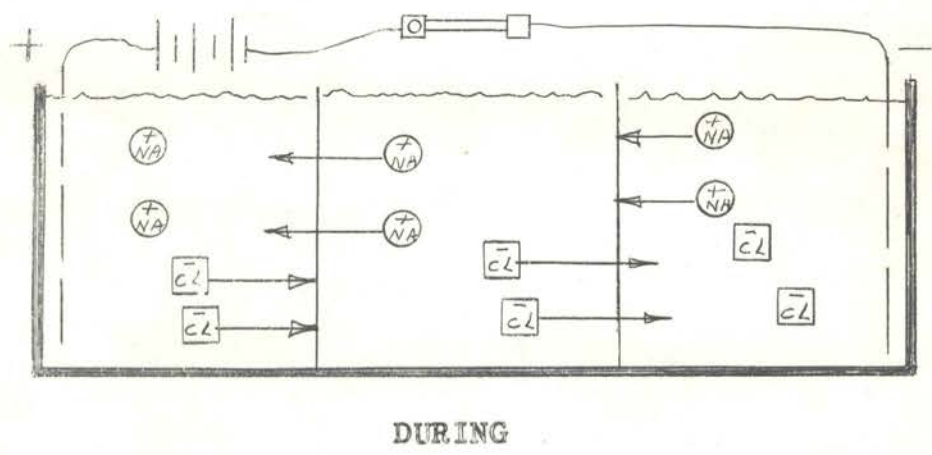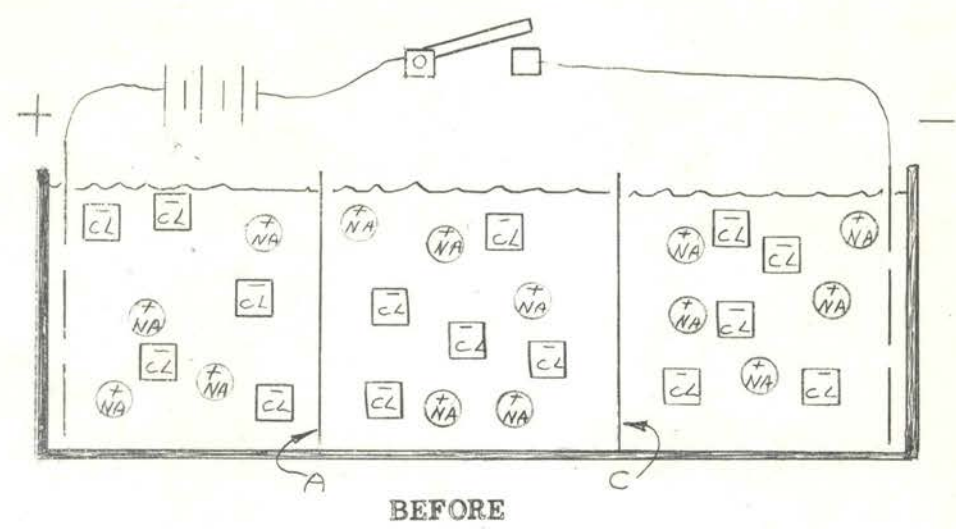
THREE COMPARTMENT CELL

Mineral reduction – sodium chloride solution

Figure 2

TABLE I

PERMSELECTIVE MEMBRANES - PRELIMINARY TESTS

Run 1		Run 2		Run 3	
Sample No.	PPM - $CaCl_2$	Sample No.	PPM - NaCl	Sample No.	PPM - NaCl
1	2748	1	5100	1	6600
2	2751	2	5000	2	6520
3	2682	3	4760	3	2860
4	2640	4	4660	4	1260
5	2445	5	2060	5	160
6	2298	6	540		
7	1944				
8	996				

trimming the membranes to within 1/8 inch of the cylinder on the outside. Thermoplastic electrican's tape was then heated to some 80° F. and wrapped around the cylinder, over the edges of the membranes. The solution was then allowed to flow as needed into or out of the compartments through the openings as shown. It was found that the membrane has a high coefficient of expansion when wet. For this reason the membrane was soaked before inserting and then kept in a solution to prevent rupture. Trouble was also encountered in attempting to insert the dry membrane into the cell as the solution would cause the membrane to buckle upon swelling. The capacity of each compartment would then change depending upon the hydrostatic pressure in each cell as compared to the one adjacent.

For cathode and anode material, a 3 inch circular lead sheet was soldered to 1/8 inch brass rod. These electrodes were anchored in place by using a 1/8 inch half union brass tube fitting with union nut and packing. The plexiglass sheeting was bored and tapped and the half union fitting supporting the rod was screwed directly into the plexiglass sheeting.

The electrodes were first placed near the membranes. In this position, as the mineral in the end cells became more concentrated, there was a tendency for the mineral solids to pile up on the outer or electrode side of the membrane instead of staying in solution. This difficulty was eliminated when the electrodes were placed near the outside of the cells.

In the first experiment 5,000 PPM solution was used and the current allowed to flow for some 80 hours. No visible action was noticed at the end of this period. The electrolytic action reduced the mineral

content during this period to less than 200 PPM. (Table I)

A three compartment cell was chosen, rather than a multicompartment cell, because of its simplicity and the need for only two membranes. The action of the cell is shown graphically in Figure 2, the arrows indicating direction of travel of the calcium and chloride ions. Arrows extending through the membrane indicate passage of ions while arrows ending at the membrane indicate the stoppage of the ions. The action shown results in the removal of the solids from the center compartment and concentration of solids in the outer compartments.

Preliminary tests were favorable and plans were then made to set up a series of similar cells under conditions favorable to accurate measurements. Results of these tests are shown in Tables II, III, IV, and V. An open rectangular box was first constructed of tempered 1/8 inch masonite, as shown in Figure 3. However, a satisfactory seal between the membrane and box was difficult to obtain. This method of construction was finally discarded for a similar box made of 1/2 inch white pine (Figure 4). The pine was waterproofed with several applications of glyptol paint manufactured by Du Pont De Muers Company. Before inserting the membranes in the box, the adjacent edges of each compartment were freshly painted. Three additional coats of paint were then applied to the outside of the box next to the membranes. No trouble was encountered with leakage of the solution afterwards.

Studies were again continued using similar solutions. Runs were made using voltages of 6, 12, 24, 36, 60 and 120 volts direct current. The 6, 12, and 24 volt direct current sources were obtained from batteries. The higher voltages were taken from a motor driven AC-DC convertor. After several trials, 24 volts was selected as the optimum

electrical pressure under the existing conditions. It was noted that, at the higher voltages, the solids in the solution collected along the outside surface of the membranes, thus hindering their action. At 24 volts and less, this trouble was not encountered.

Carbon, lead, silver, and iron were used for electrodes in these studies. Lead electrodes proved to be the most satisfactory and were used during the remainder of the tests. Lead sheets were bent and looped over the ends, as shown in Figure 4 for simplicity in attaching battery leads and maintaining a liquid seal.

Ammeters and voltmeters were placed in the circuit and readings were taken at regular intervals during each run. (Note Tables II, III, IV, V, VI, and VII appendix.) At the time each reading was taken, a 10 milliliter sample of solution was removed from the center compartment. (See Figure 5.) These samples were analyzed by titrating to determine rate of demineralization. Before sampling, the solution in this compartment was stirred in order to obtain a representative sample. The 110 ampere hour, 24 volt battery used to supply the electrical energy was recharged between runs. The ampere flow was low and consequently little variation occurred in voltage during each run.

As a second source of power alternating current was passed through a half wave rectifier of the selenium cell type. Results from this pulsating, direct current were similar to that obtained by using battery current.

Near the conclusion of this study a sample of Cimarron River water was taken near Perkins, Oklahoma. The same day the U.S.G.S., Division of U.S.D.I., obtained a sample near this same spot. Data for the two samples are given in Table IIB and Table VIII. U.S.G.S. states that
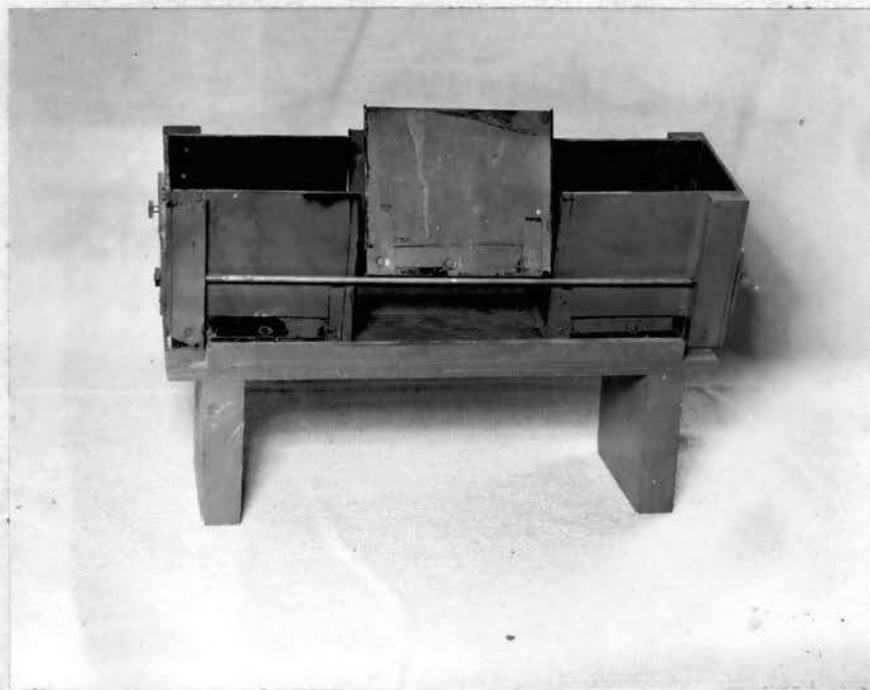
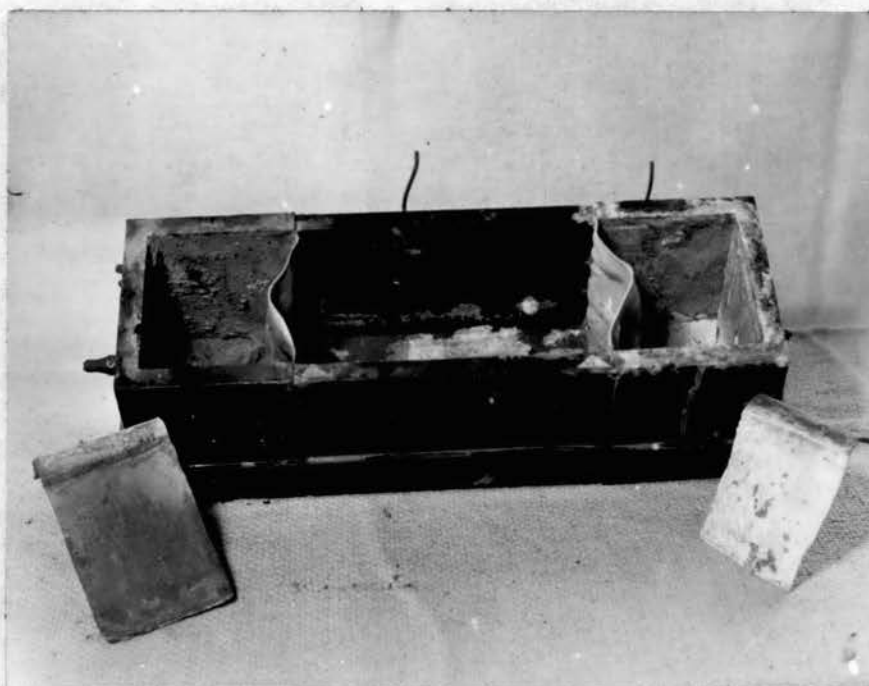Fig. 3 - Open electro-dialyzer cell.

Fig. 4 - Open electro-dialyzer cell with membranes
in place showing lead sheet electrodes.

TABLE II

PERMSELECTIVE MEMBRANES --- POWER REQUIREMENTS AND RATE OF SALINE REDUCTION

A Solution - Na(HCO$_3$)

Unit 2, Run 1 --- 1685 cc.

B Solution - Cimarron River Water

Unit 1, Run 1 --- 1820 cc.

HR	E	I	W	PPM	WH	WH$_{ac}$	HR	E	I	W	PPM	WH	WH$_{ac}$
2200	26.6	2.00	53.2	3220			1200	27.0	0.92	24.8	13,600		
0200	27.0	1.50	40.5	2360	162.0	162.0	1800	24.8	0.95	23.6	11,800	142	142
0600	26.2	0.10	02.6	1500	10.4	172.4	0200	25.2	0.41	10.3	9,280	82	224
1000	26.6	0.08	02.0	1160	8.4	180.8	0600	26.4	0.30	7.9		32	256
1400	26.6	0.04	01.1	730	4.4	185.2	1400	26.0	0.19	4.9	1,100	39	295
2300	26.6	0.02	00.5	390	4.5	189.7	1800	26.0	0.15	3.9		16	311
0200	26.7	0.02	00.5	250	1.5	191.2	2000	26.1	0.13	3.4	470	07	318
0600	26.6	0.01	00.3	200	1.2	192.4	0600	26.1	0.15	3.9		31	349
1400	26.6	0.01	00.3	150	2.4	194.8	1000	26.1	0.07	1.8	350	07	356
2200	26.8		00.3	100	2.4	197.2	1400	26.1	0.06	1.6		06	362
0600	27.0	0.01	00.3	70	2.4	199.6	1800	26.1	0.05	1.3	40	05	367

TABLE III

PERMSELECTIVE MEMBRANES —— POWER REQUIREMENTS AND RATE OF SALINE REDUCTION

A Solution - $MgSO_4$

Unit 2, Run 1 —— 1685 cc.

B Solution - $MgCl_2$

Unit 1, Run 2 —— 1820 cc.

HR	E	I	W	PPM	WH	WH_{ac}	HR	E	I	W	PPM	WH	WH_{ac}
1700	26.0	2.10	54.5	3520			1800	21.0	0.50	10.5	5240		
1800	26.0	0.25	6.5	2980	6.5	6.5	2200	22.0	0.46	10.3	4050	1.2	41.2
0600	27.0	0.24	6.5	1980	78.0	84.5	2400	22.0	0.40	8.8	3000	17.6	58.8
1000	25.2	0.28	7.1	1550	28.4	112.9	0200	21.0	0.35	7.4	1800	14.8	73.6
1400	25.6	0.27	6.9	1050	27.6	140.5	0400	22.0	0.28	6.2		12.4	86.0
1800	25.6	0.26	6.8	1020	27.2	167.7	0600	22.0	0.22	4.9	900	9.8	95.8
0600	25.6	0.19	4.9	570	58.8	226.5	1000	21.0	0.18	3.8		15.2	111.0
1400	26.6	0.18	4.8	300	38.4	264.9	0200	21.0	0.13	2.7	440	10.8	121.8
1800	25.6	0.15	3.8	260	15.2	280.1	1000	22.0	0.10	2.2	420	17.6	139.4
2200	25.6	0.17	4.4	270	17.6	297.7	0200	22.0	0.08	1.8	340	7.2	146.6
0200	25.6	0.18	4.6	150	18.4	316.1	0600	22.0	0.06	1.3	480	5.2	151.8
							1000	22.0	0.05	1.1	430	4.4	156.2
							1400	22.0	0.05	1.1	210	4.4	160.6

TABLE IV

PERMSELECTIVE MEMBRANES — POWER REQUIREMENTS AND RATE OF SALINE REDUCTION

A Solution - $CaSO_4$

Unit 2, Run 4 — 1685 cc.

HR	E	I	W	PPM	WH	WH_{ac}
1000	20.0	0.110	2.20	2180		
1400	20.0	0.140	2.80	1840	11.2	11.2
1800	20.0	0.130	2.60	1780	10.4	21.6
2200	20.0	0.170	3.40	1210	13.6	35.2
0200	20.0	0.160	3.20	960	12.8	48.0
0600	20.0	0.150	3.00	940	12.0	60.0
1000	20.0	0.010	2.00	780	8.0	68.0
1400	20.0	0.010	2.00	620	8.0	76.0
1800	20.0	0.010	2.00	570	8.0	84.0
2200	20.0	0.010	2.00	420	8.0	92.0
0200	20.0	0.015	3.00	310	12.0	104.0
0600	20.0	0.013	2.60	260	10.4	114.4
1000	20.0	0.006	0.12	210	00.5	114.9

B Solution - $CaCl_2$

Unit 2, Run 3 — 1685 cc.

HR	E	I	W	PPM	WH	WH_{ac}
2200	11.9	0.22	2.62	4400		
0200	11.9	0.11	1.30	4100	5.2	5.2
0600	11.9	0.10	1.19	3300	4.8	10.0
1800	12.0	0.08	0.96	2100	12.0	22.0
2200	12.0	0.08	0.96	1850	3.8	25.8
0200	11.9	0.06	0.71	1430	2.8	28.6
0600	11.9	0.06	0.71	1000	2.8	31.4
1000	11.8	0.05	0.59	1110	2.4	33.8
1400	11.9	0.05	0.60	1090	2.4	34.0
1800	11.8	0.04	0.47	900	1.9	35.9

TABLE V

PERMSELECTIVE MEMBRANES — POWER REQUIREMENTS AND RATE OF SALINE REDUCTION

A Solution - MgCl

Unit 1, Run 6 — 1820 cc.

B Solution - MgCl

Unit 2, Run 6 — 1685 cc.

HR	E	I	W	PPM	WH	WH_{ac}	HR	E	I	W	PPM	WH	WH_{ac}
1800	6.7	.05	.34	1900			1800	6.7	.05	.33	2260		
0600	6.6	.05	.33	2100	3.96	3.96	0600	6.6	.05	.33	2000	3.96	3.96
1400	6.6	.042	.28	1850	2.24	6.20	1400	6.6	.06	.39	2000	3.12	7.08
1800	6.6	.042	.28	1380	1.12	7.32	1800	6.6	.05	.33	1910	1.32	8.40
0700	6.6	.040	.26	1280	3.38	10.70	0700	6.6	.05	.33	1760	4.29	12.69
1800	6.5	.030	.20	900	2.20	12.90	1800	6.5	.042	.28	1750	3.10	15.79
0600	6.6	.020	.13	890	1.56	14.46	0600	6.6	.042	.28	1900	3.36	19.15
1000	6.6	.020	.13	800	.78	15.24	1000	6.6	.038	.25	1790	1.00	20.15
1800	6.6	.020	.13	610	1.04	16.28	1800	6.6	.035	.23	1600	1.84	21.99
2200	6.7	.020	.13	590	.52	16.80	2200	6.7	.035	.23	2050	0.92	22.91
0600	6.7	.010	.07	500	.56	17.36	0600	6.7	.025	.17	2010	1.36	24.27
1000	6.7	.012	.08	420	.32	17.68	1000	6.7	.025	.17	1700	.68	24.95

TABLE VI

PERMSELECTIVE MEMBRANES — POWER REQUIREMENTS AND RATE OF SALINE REDUCTION

A Solution - CaSO$_4$

Unit 1, Run 5 — 1820 cc.

HR	E	I	W	PPM	WH	WH$_{ac}$
1400	7	0.04	.28	1740		
1800	7.8	.04	.31	1510	1.24	1.24
2200	7.8	.035	.27	1470	1.08	2.32
600	7.8	.035	.27	1326	2.16	4.48
1000	7.8	.030	.23	1430	0.92	5.40
1400	6.7	.030	.20	1410	0.8	6.20
2200	6.7	.030	.20	1520	1.6	7.80
600	6.7	.030	.20	1310	1.6	9.40
1000	6.7	.029	.19	1550	.8	10.20
1400	6.7	.027	.18	1220	.7	10.90
1800	6.6	.026	.17	1150	.7	11.60

B Solution - CaSO$_4$

Unit 2, Run 5 — 1685 cc.

HR	E	I	W	PPM	WH	WH$_{ac}$
1400	7	0.04	.28	1980		
1800	7.8	.04	.31	1600	1.24	1.24
2200	7.8	.035	.27	1450	1.08	2.32
600	7.8	.04	.31	1310	2.48	4.80
1000	7.8	.033	.26	1240	1.04	5.84
1400	6.7	.034	.22	1300	0.88	6.72
2200	6.7	.033	.22	1000	1.76	8.48
600	6.7	.033	.22	950	1.76	10.24
1000	6.7	.033	.22	910	0.88	11.12
1400	6.7	.031	.21	800	0.80	11.92
1800	6.6	.03	.20	640	0.80	12.72
2200	6.6	.030	.20	500	0.80	13.52
1000	6.6	.031	.20	450	2.40	15.92
1400	6.6	.028	.18	220	0.72	16.64

TABLE VII

PERMSELECTIVE MEMBRANES —— POWER REQUIREMENTS AND RATE OF SALINE REDUCTION

A Solution – NaCl

Unit 1, Run 1 —— 1820 cc.

B Solution – NaCl

Unit 2, Run 1 —— 1685 cc.

HR	E	I	W	PPM	WH	WH_{ac}	HR	E	I	W	PPM	WH	WH_{ac}
1600	12.8	1.05	13.5	8640			1600	12.8	1.0	12.8	7600		
2400	13.1	.50	6.6	7910	52.8	52.8	2400	13.1	.5	6.6	7510	52.8	52.8
800	13.1	0.38	5.0	2740	40.0	92.8	800	13.1	0.3	3.9	2610	31.2	84.0
1600	13.1	0.30	3.9	2610	31.2	124.0	1600	13.1	0.28	3.67	2490	29.4	113.4
800	13.1	0.22	2.88	1460	46.0	170.0	800	13.1	0.28	3.67	1360	58.8	172.2
1600	13.1	0.20	2.6	1570	20.8	190.8	1600	13.1	0.28	3.67	1150	29.4	201.6
800	13.0	0.17	2.2	890	35.2	226.0	800	13.0	0.28	3.6	950	57.6	259.2
1600	13.0	0.14	1.8	780	14.4	240.4	1600	13.0	0.28	3.64	750	29.1	288.3
2400	12.9	0.08	1.03	550	8.2	248.6	2400	12.9	0.28	3.61	810	29.0	317.3
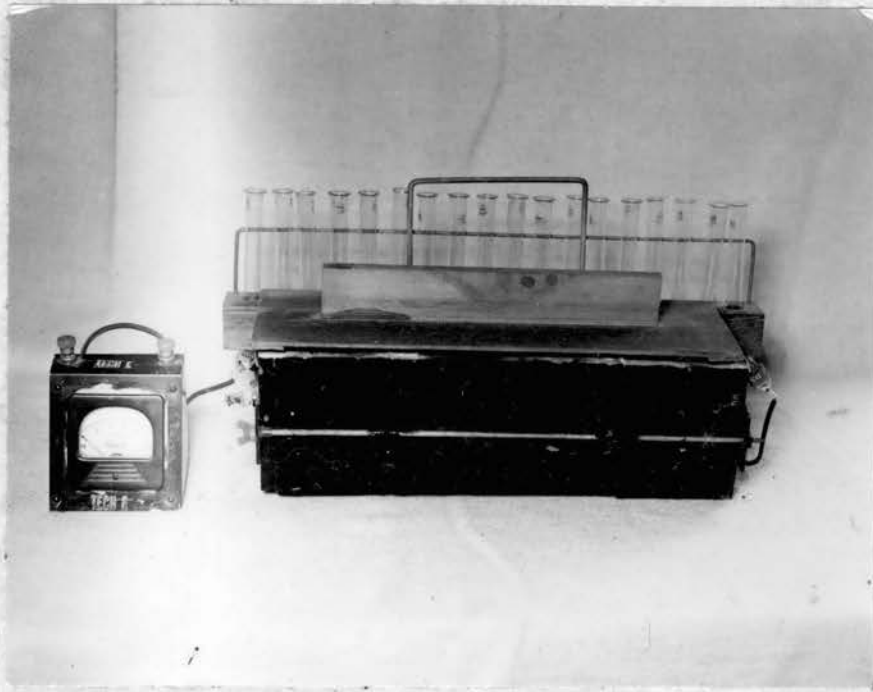
Fig.5 - Open electro-dialyzer cell in operation.
Note test tubes for water samples.

Fig. 6 - Battery of electro-dialyzer cells in operation.

water obtained from this sampling station often varies from day to day over a broad range. They also report that the Cimarron river is the most alkaline of all Oklahoma streams sampled and the Perkins sampling station yields the highest alkaline content.

Standard titrating equipment, as shown in figure 8, was used in determining mineral hardness of water. A simpler, less complicated method for hardness determination would have been the use of a conductance meter. This equipment was not available.

Fig. 7 - Electrode materials. Note reduction of
negative electrodes.

Fig. 8 - Standard titrating equipment used in
determining hardness of water.

TABLE VIII

CIMARRON RIVER WATER

Sample Taken By U.S.G.S. at Perkins, April 15, 1954

		PPM			PPM
Silica	$Si\,O_2$		Bicarbonate	HCO_3	264
Iron	Fe		Sulfate	SO_4	433
Calcium	Ca	209	Chloride	Cl	4530
Magnesium	Mg	65	Fluoride	F	
Sodium	Na	2940	Nitrate	NO_3	
Potassium	K		Carbonate	CO_3	

Total solids = 8630 PPM

Conductance (Micromhos) 14,300

CHAPTER VI

ANALYSIS OF DATA

In the final tests the capacity of the center chamber containing the treated water in the four demineralizing units was computed by measuring the volume in the solution before and after each run. The average of several runs was then taken to determine the volume of the treated water. Ammeter and voltmeter readings were taken at the time the water samples were removed. WH (watt hours) were computed for each of these readings.

No allowance was made for evaporation since a lid covered each cell (Figure 5) which reduced evaporation to a minimum. This information was then used to compute the actual faradays which determined the points along the ordinate. For corresponding points on the abcissa PPM (parts per million) were computed from each of the ten milliter solutions removed from center chamber.

Sample Calculations (Table II, Cimarron River water, Unit 1, run 1)

Capacity of center chamber = 1820 cc.

Hardness removed = 13,600 initial - 300 final = 13,300 PPM final.

Energy consumed during this period = 472.1 WH (watt hours) or 65,400 ampere seconds or .679 faradays.

Energy /1000 gal. = $\frac{3.785 \text{ cc. per gal. x } 1000 \text{ x } 472.1 \text{ WH}}{1,820 \text{ cc.} \qquad 1000}$ =

982 KWH /1000 gal.

Final computations of mineral saline hardness were influenced by the following variables. In the final runs for each of the chemicals

tested milammeter readings and ten milliter samples were scheduled to be taken every four hours, around the clock, for some 96 hours.

The water plant operating personnel took the samples and recorded the meter readings. Some six shifts were scheduled per 24 hours. Occasionally emergencies arose when sampling and readings occurred at odd hours or not at all as indicated in Tables II to VIII, listed on pages 25 to 30. Apparently stratification occurred in center chamber of demineralizing cell. Should the operator fail to stir this solution before sampling a representative sample was not obtained.

Standard titrating apparatus, using 100 cc. samples, as shown in Figure 10, was used in analyzing for PPM hardness. To obtain a reading the ten milliter solution was diluted with 90 cc. distilled water. The ten milliter samples were accumulated in test tubes racked adjacent to cell as shown in Figure 5. Evaporation from these vials varied with temperature and humidity. These variables should partly account for variable results obtained in titrating samples for hardness.

Ten to fourteen samples were removed during a run. The center chamber capacities varied in volume from 1685 cc. to 1920 cc. This variance in volume from start to finish of run would also introduce an error in accurancy of computation. All these factors would influence the deviation of points about a line on the graphs shown in Figures 9 to 20.

Sample Computation of Efficiency

Compound - Magnesium sulfate, Table 3a, unit 2, run 1.

PPM removed per 1,685 cc. solution;

Initial = 4,410 PPM   ---- 17.05 ampere hours required
Final   =   150 PPM
Removed = 4,260 PPM

Equivalent weight removed;

$$\frac{1,685 \text{ gms. } \times 4.260 \text{ PPM}}{1,000,000 \times 60.18 \text{ (E.W.)}} = .119 \text{ E.W.}$$

Efficiency = $\frac{\text{Theoretical faradays required}}{\text{Actual faradays used}}$

$$\frac{17.05 \text{ ampere hours } \times 3,600}{.119 \text{ E.W.}} = 515,000 \text{ ampere seconds used / E.W.}$$

$$\frac{96,500 \text{ amp. sec. (one faraday)}}{515,000 \text{ amp. sec.}} = 18.7 \text{ percent efficiency}$$

Energy required per 1,000 gallons water

$$\frac{3,785,000 \text{ gms. (1,000 gallons) } \times 443.4 \text{ WH}}{1,685 \text{ gms. } \times 1,000 \text{ W } \times 4.26 \text{ PPM}} =$$

$$993 \text{ KWH / 1,000 gal. / 1,000 PPM}$$

Electrode pencils of one-fourth inch carbon were compared for conductivity with three inch square lead sheets. No differences in cell conductivity were apparent. This would be expected as the salinity of electrode compartments increase as the solution in the center compartment of a primary cell is reduced in chemical content. No attempt was made to check treated water for lead content.

Specific membrane conductance was determined by first measuring cell resistance, containing a saline solution, with membranes in place. The membranes were then removed and resistance of solution again measured. Care was taken to use the same volume of solution with the same electrode spacing.

$$\text{Resistance (in ohms) of membrane / cm}^2 = \frac{\text{(resistance of cell)} - \text{(resistance of cell)}}{\text{(with membrane)}} \frac{\text{(without membrane)}}{\text{number of membranes}}$$

$$= \frac{200 \text{ ohms } - 150 \text{ ohms}}{(2.54 \text{ cm}^2/ \text{ in } \times 16 \text{ in}^2 \times 2 \text{ membranes}} =$$

$$.634 \text{ ohms/ cm}^2$$

An examination of the graphs of results, as shown in Figures 9 to 20, indicate the lower voltages will yield more efficient results. PPM / K (1000) gallons are used as points on the absicca as hardness of farm water supplies is indicated commercially in these units rather than EPM (equivalent parts per million) / 1000 gallons.

In the six volt group, calcium sulphate, Figure 20, requires 12.5 KWH / 1000 gal. / 1000 PPM. Figure 14, sodium chloride, of the 12 volt group requires 49.4 KWH / 1000 gal. / 1000 PPM and in the 24 volt group, Figure 7, 130 KWH / 1000 gal. / 1000 PPM are used. Theoretically, one faraday is needed to remove one EPM of any chemical. Using the same voltage calcium sulphate with an EPM of 68.07 would require slightly less power per gram than sodium chloride whose EPM is 58.45.

In comparing various current flows Figure 17 would indicate that a six volt source would be most desirable for optimum results. While the cells were operating it was observed that chemicals would pile up in the cell on the surface of the membrane next to the electrodes at the higher voltages. Polarity of electrodes was reversed as shown in Figure 10 and Figure 11 to check the permselectivity of the membranes. An examination of these curves indicates excellent selectivity as little or no demineralization took place during this time.
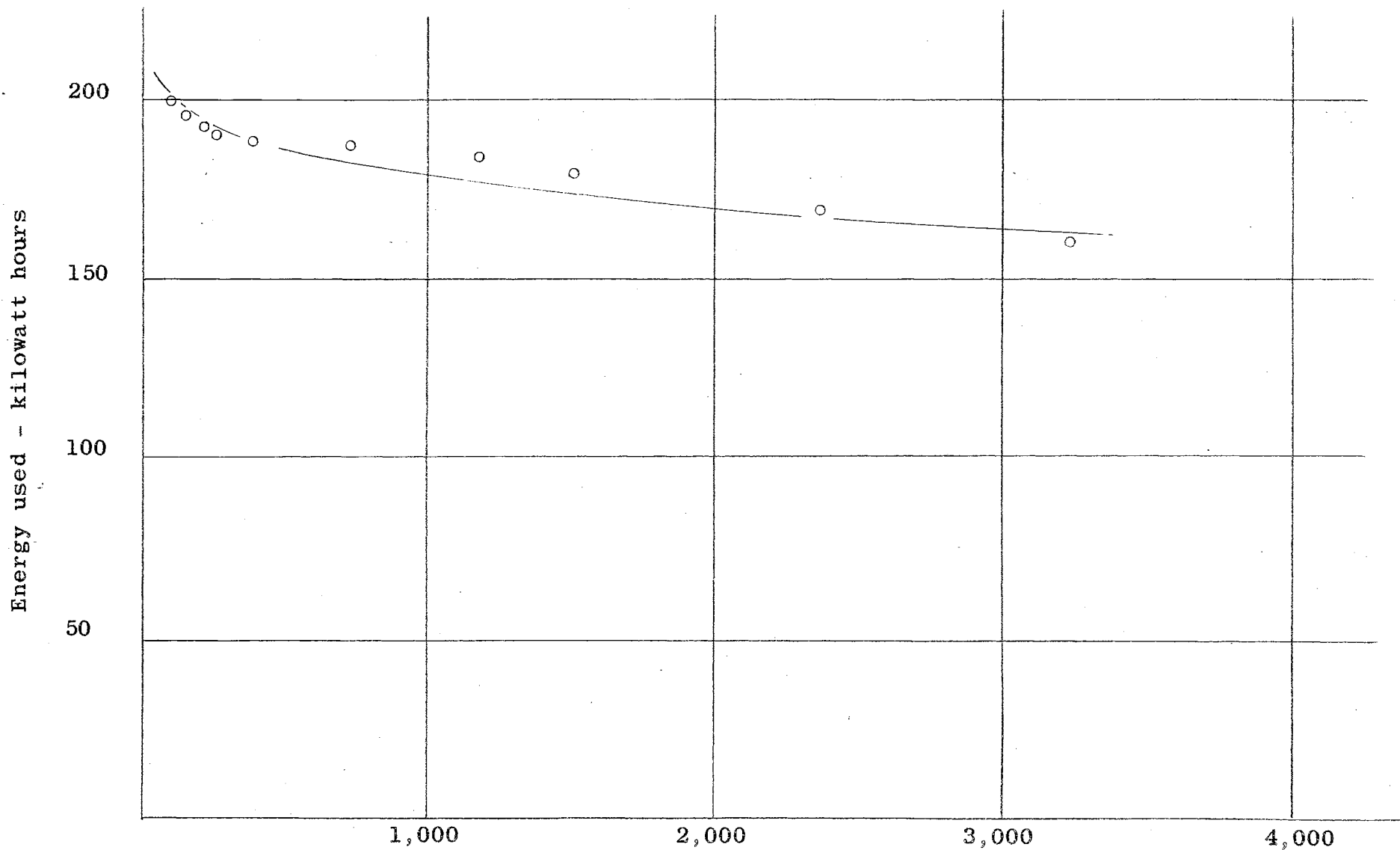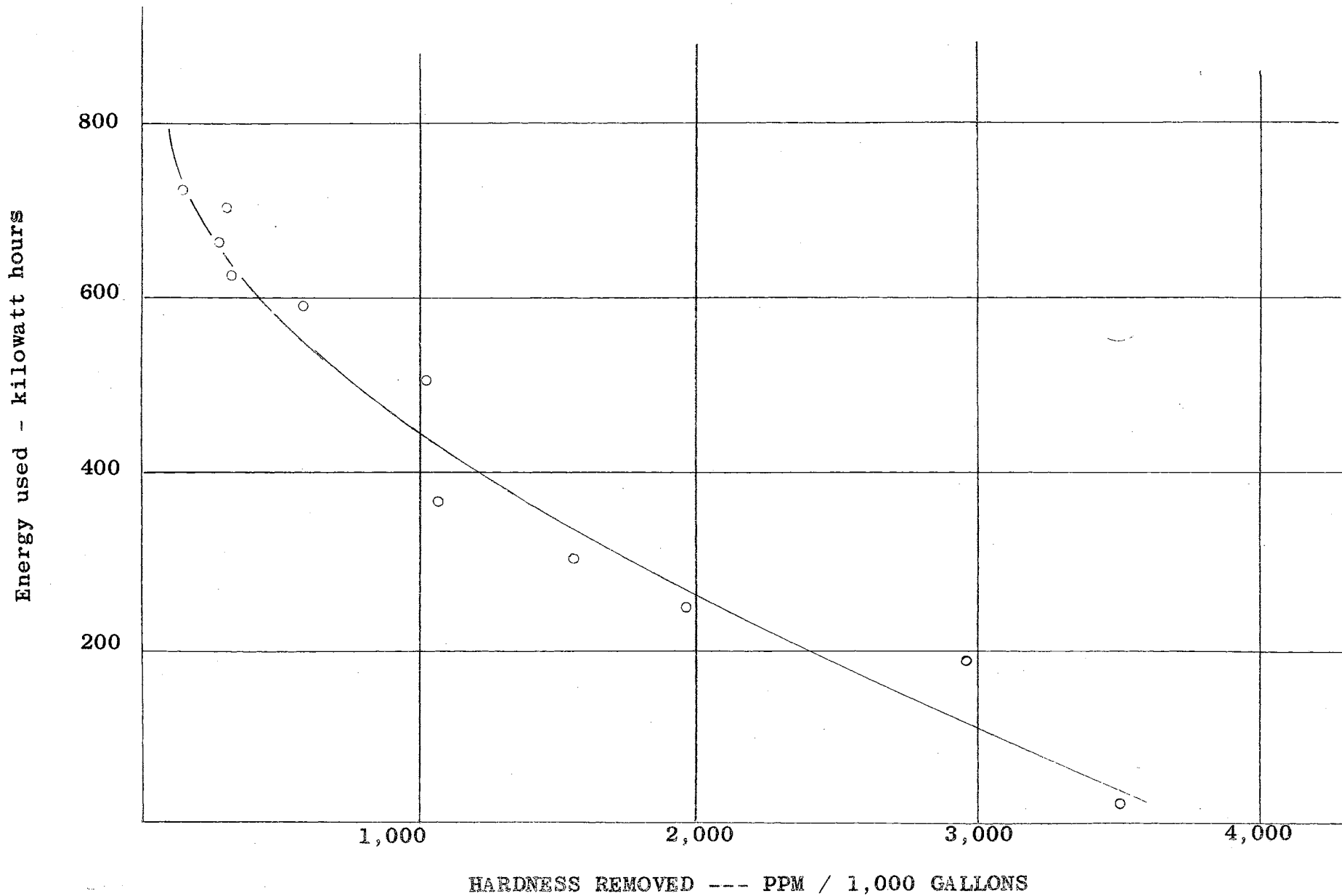
Figure 9   HARDNESS REMOVED --- PPM /1,000 GALLONS

Mineral reduction of Sodium Bicarbonate solution --- 24 volts (table 2A)
Electro-ion-migration using perm-selective membranes

Figure 10   HARDNESS REMOVED —— PPM / 1,000 GALLONS
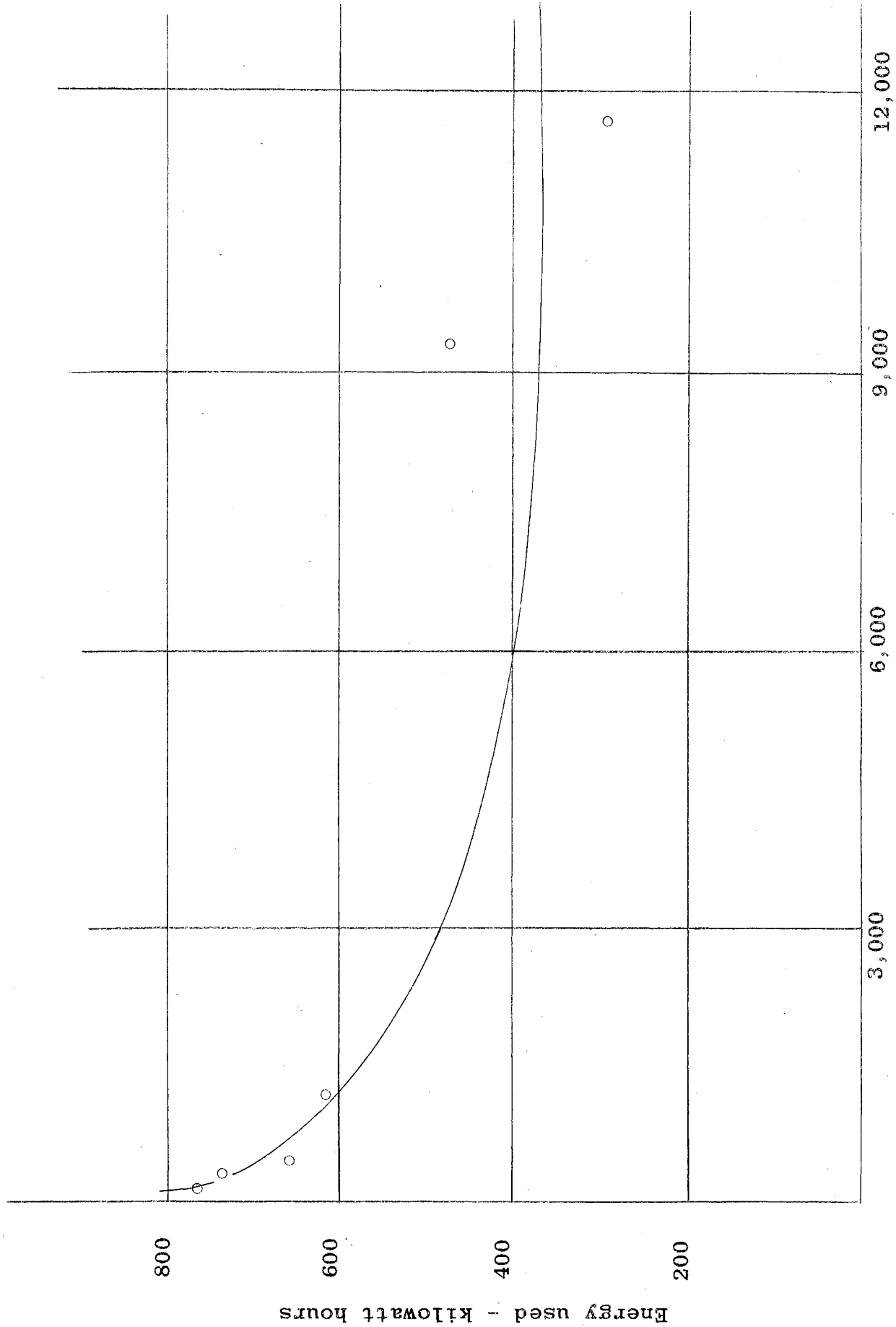
Mineral reduction of Cimarron river water —— 24 volts (table 2B)

Figure 11   Mineral reduction of magnesium sulphate solution --- 24 volts (table 3A)
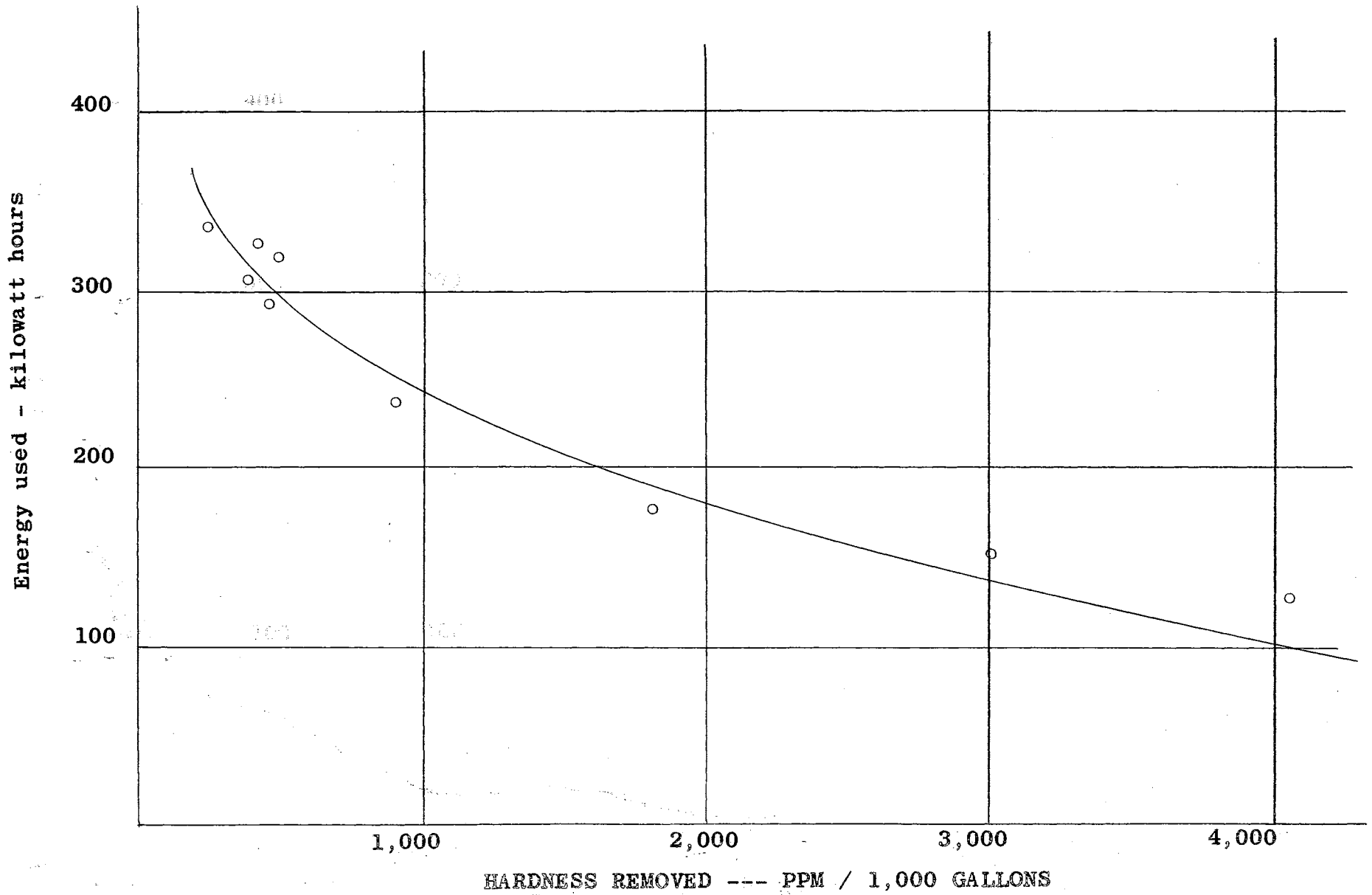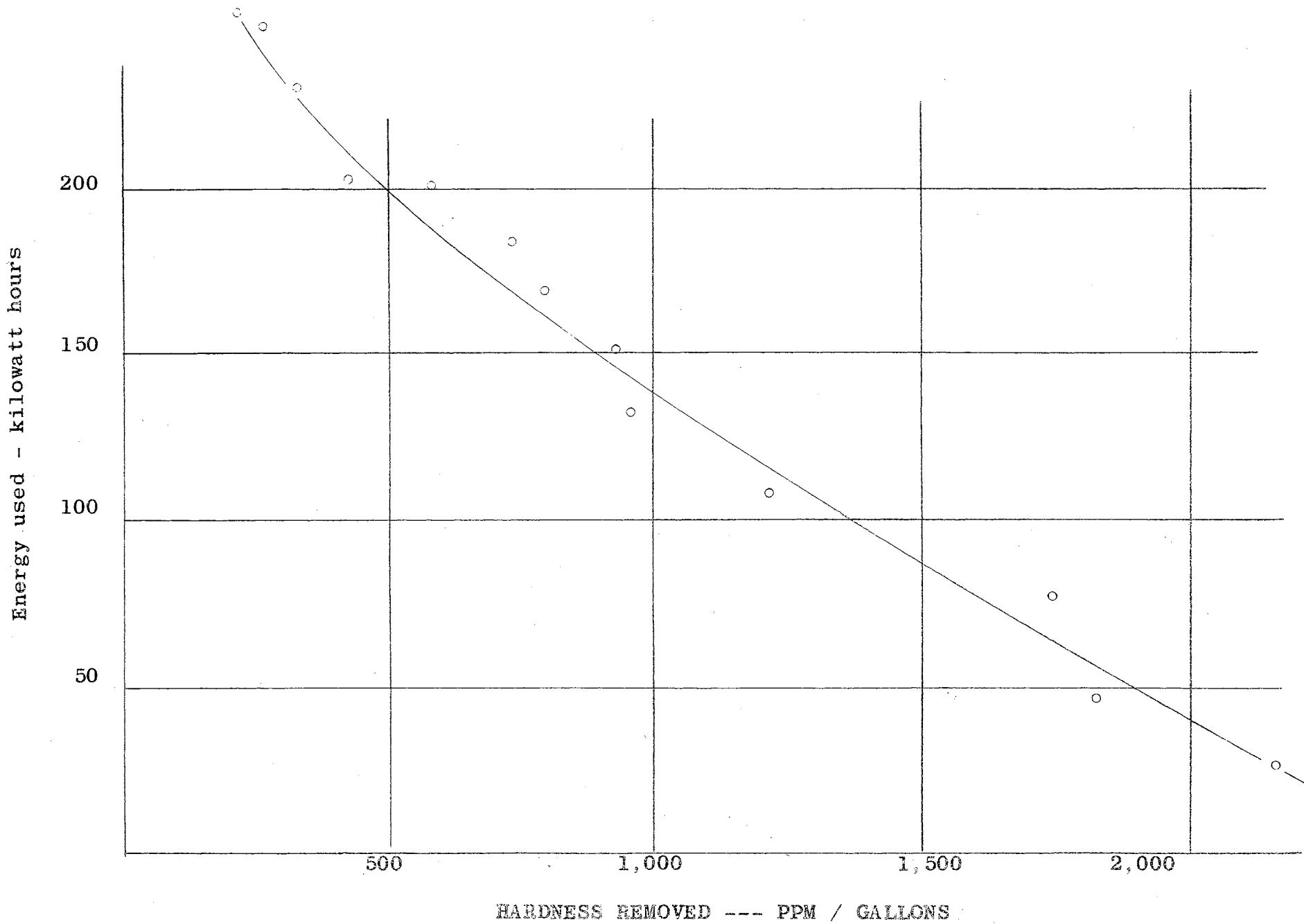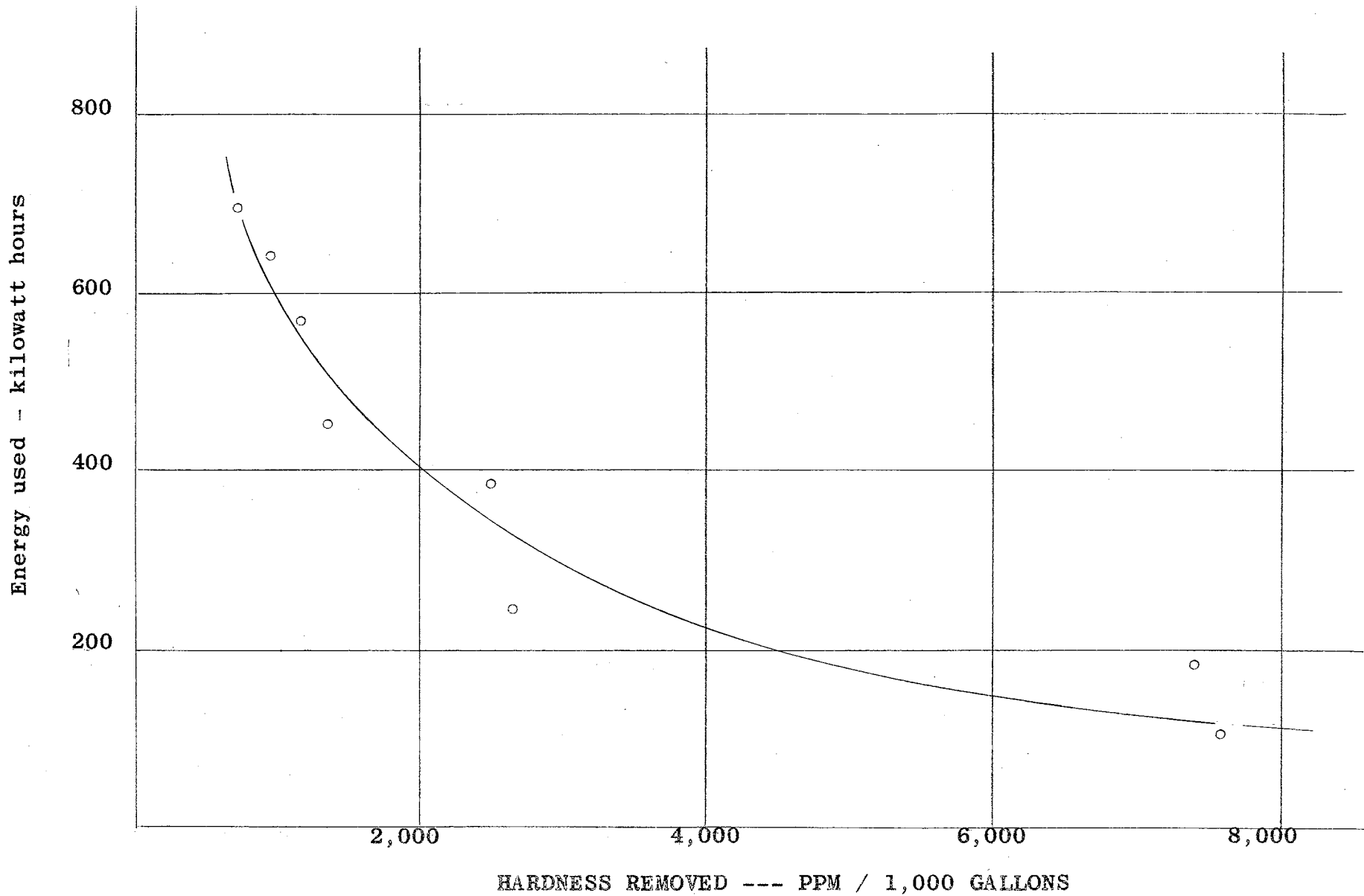   Electro-ion-migration using perm-selective membranes
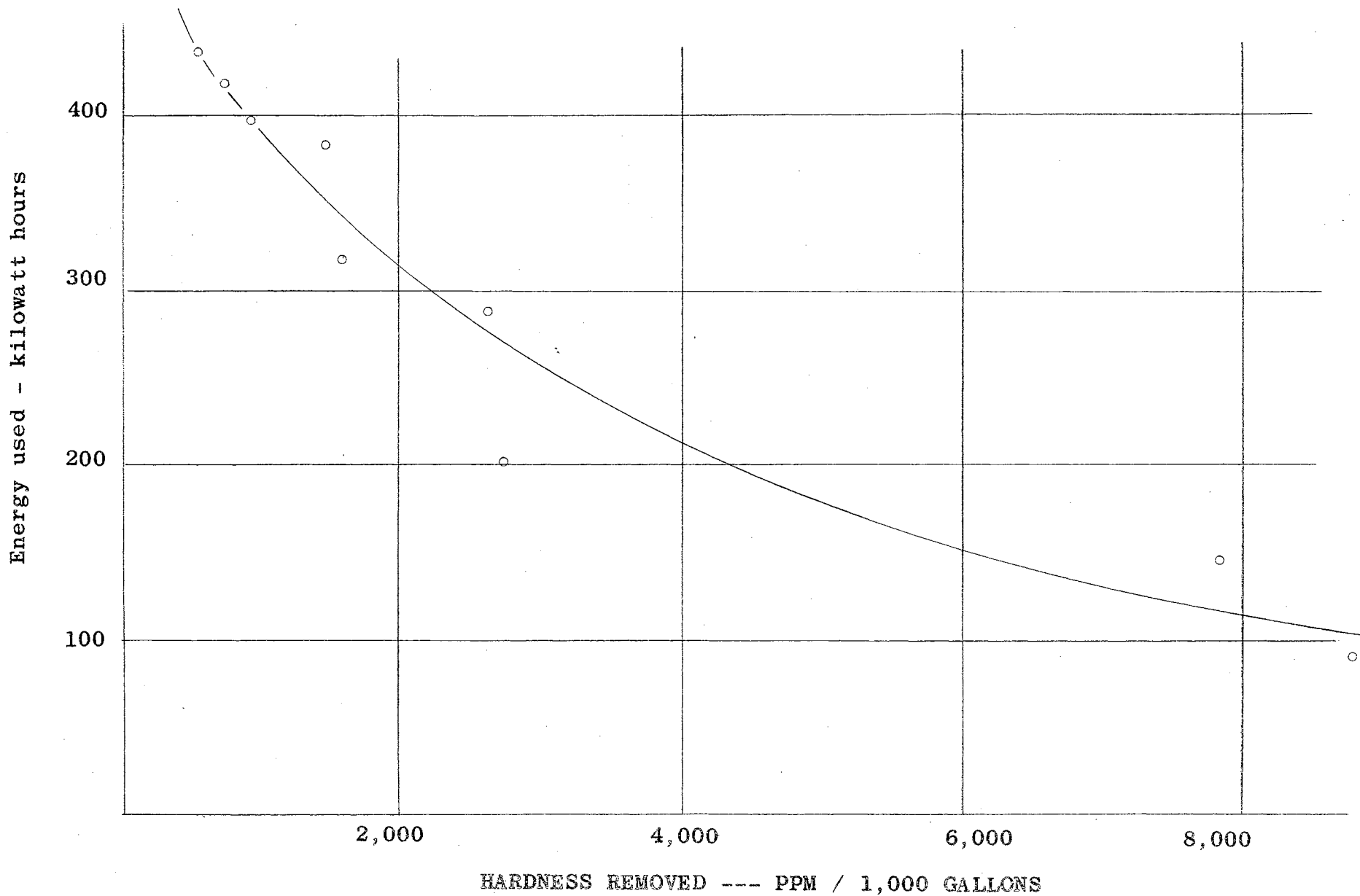
Figure 12   Mineral reduction of magnesium chloride solution --- 24 volts (table 3B)
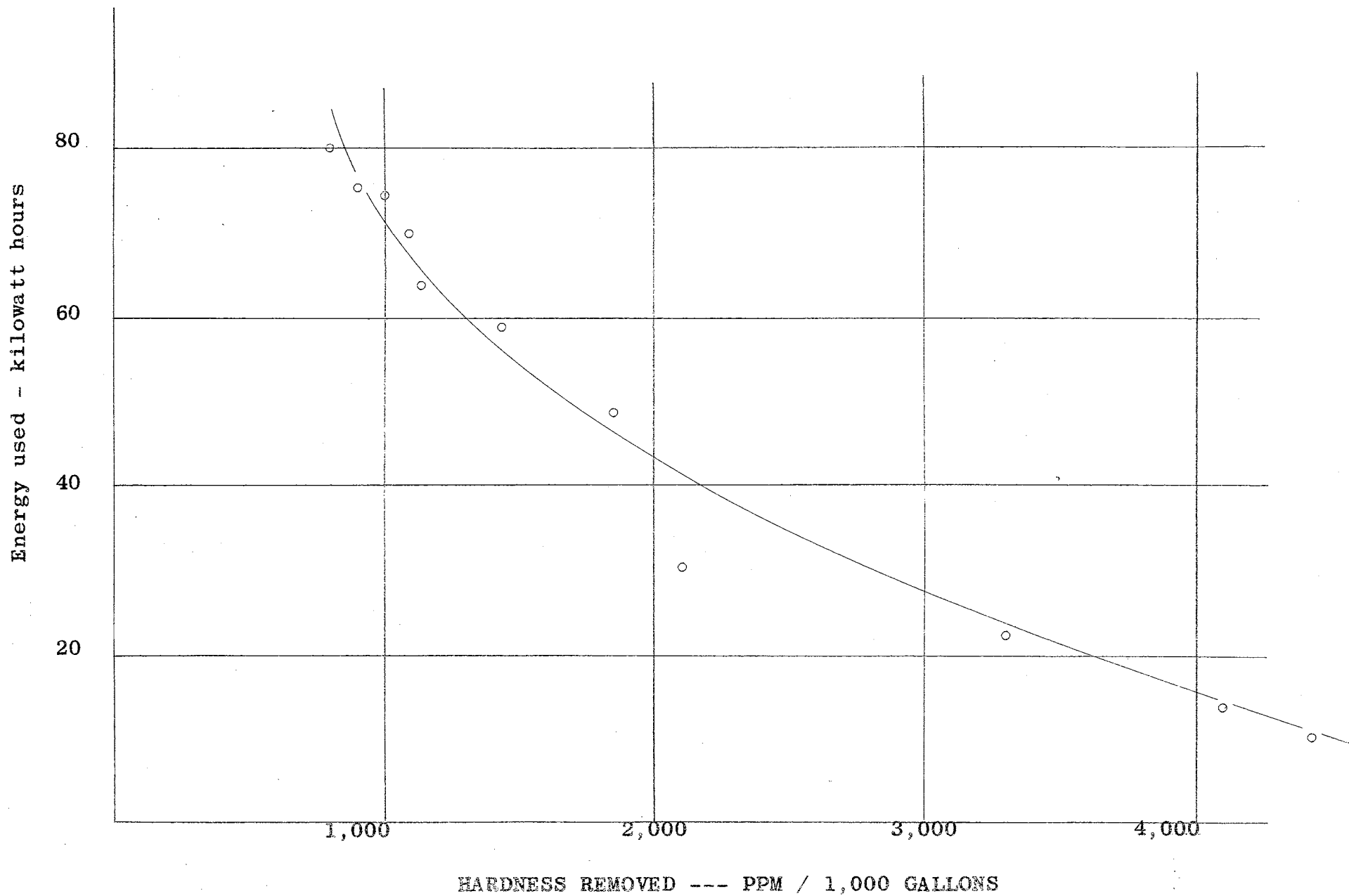Electro-ion-migration using perm-selective membranes

Figure 13   Mineral reduction of calcium sulphate solution --- 24 volts (table 4A)
              Electro-ion-migration using perm-selective membranes

Figure 14   Mineral reduction of sodium chloride solution --- 12 volts (table 7A)
Electro-ion-migration using perm-selective membranes

Figure 15 Mineral reduction of sodium chloride solution --- 12 volts (table 7B)
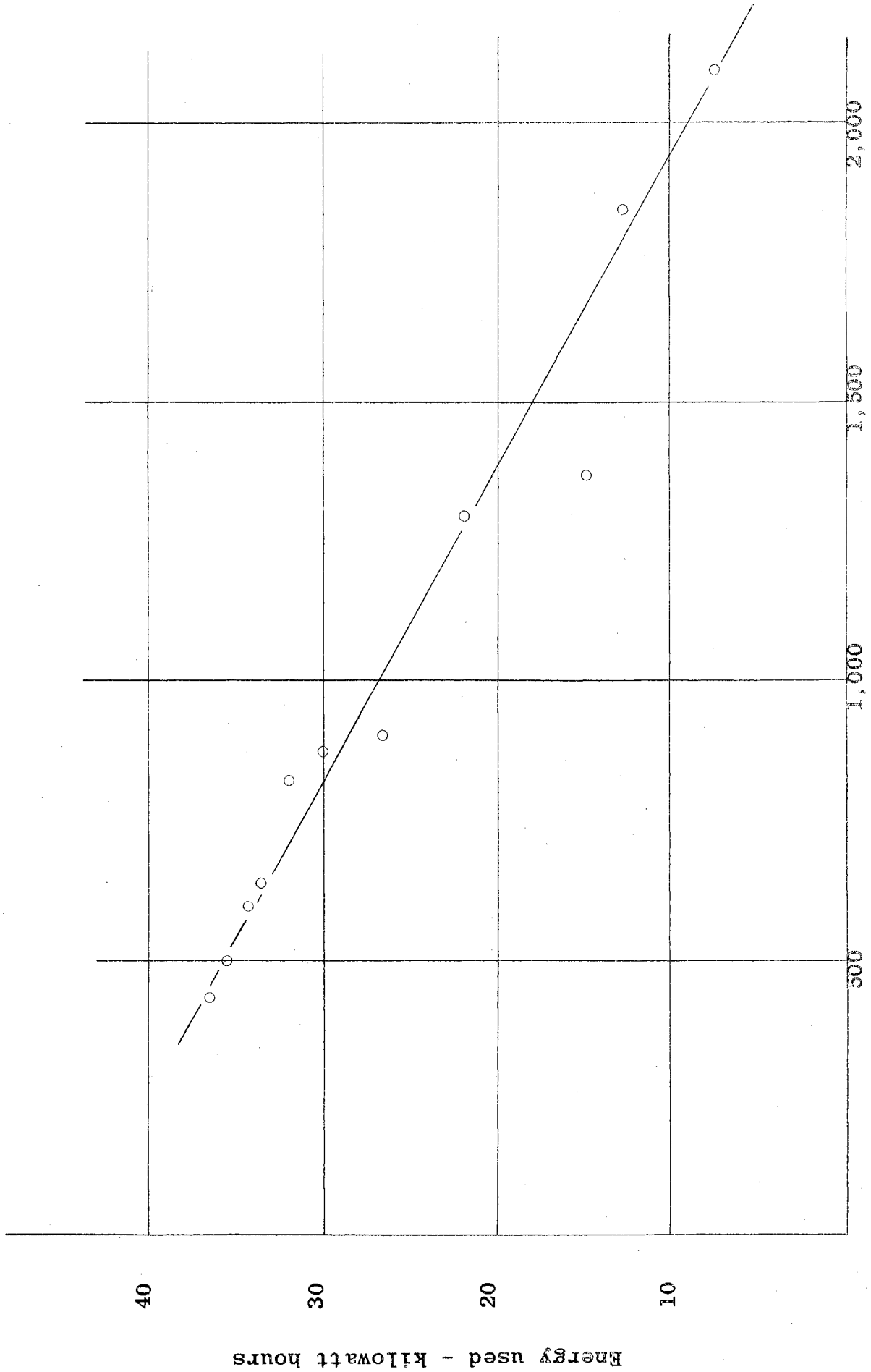   Electro-ion-migration using perm-selective membranes

Figure 16   Mineral reduction of calcium chloride solution --- 12 volts (table 4B)

HARDNESS REMOVED --- PPM / 1,000 GALLONS

Figure 17 Mineral reduction of magnesium chloride solution --- 6 volts (table 5A)
Electro-ion-migration using perm-selective membranes
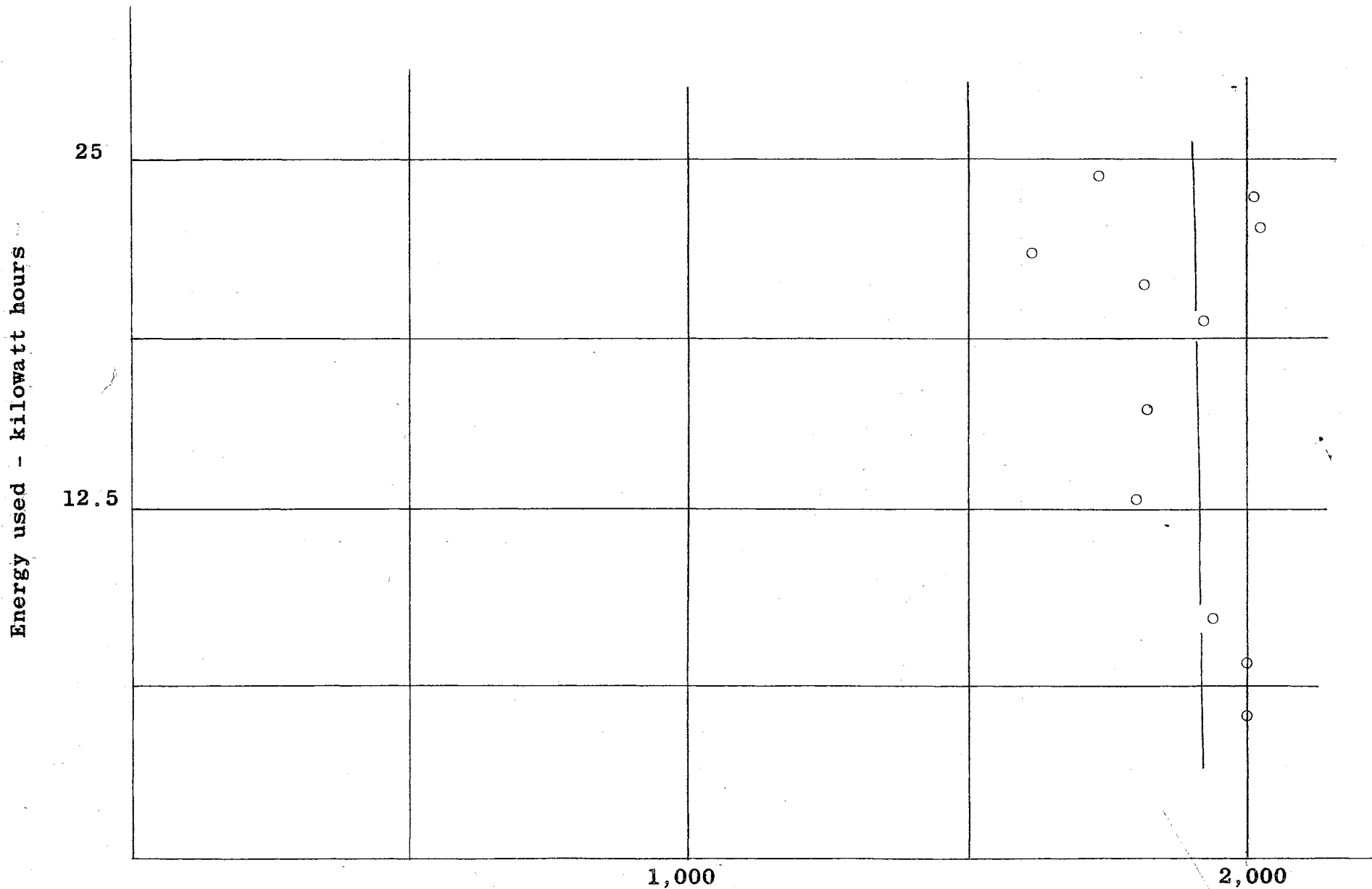
47

Figure 18   Magnesium chloride solution --- 6 volts (table 5B) polarity reversed
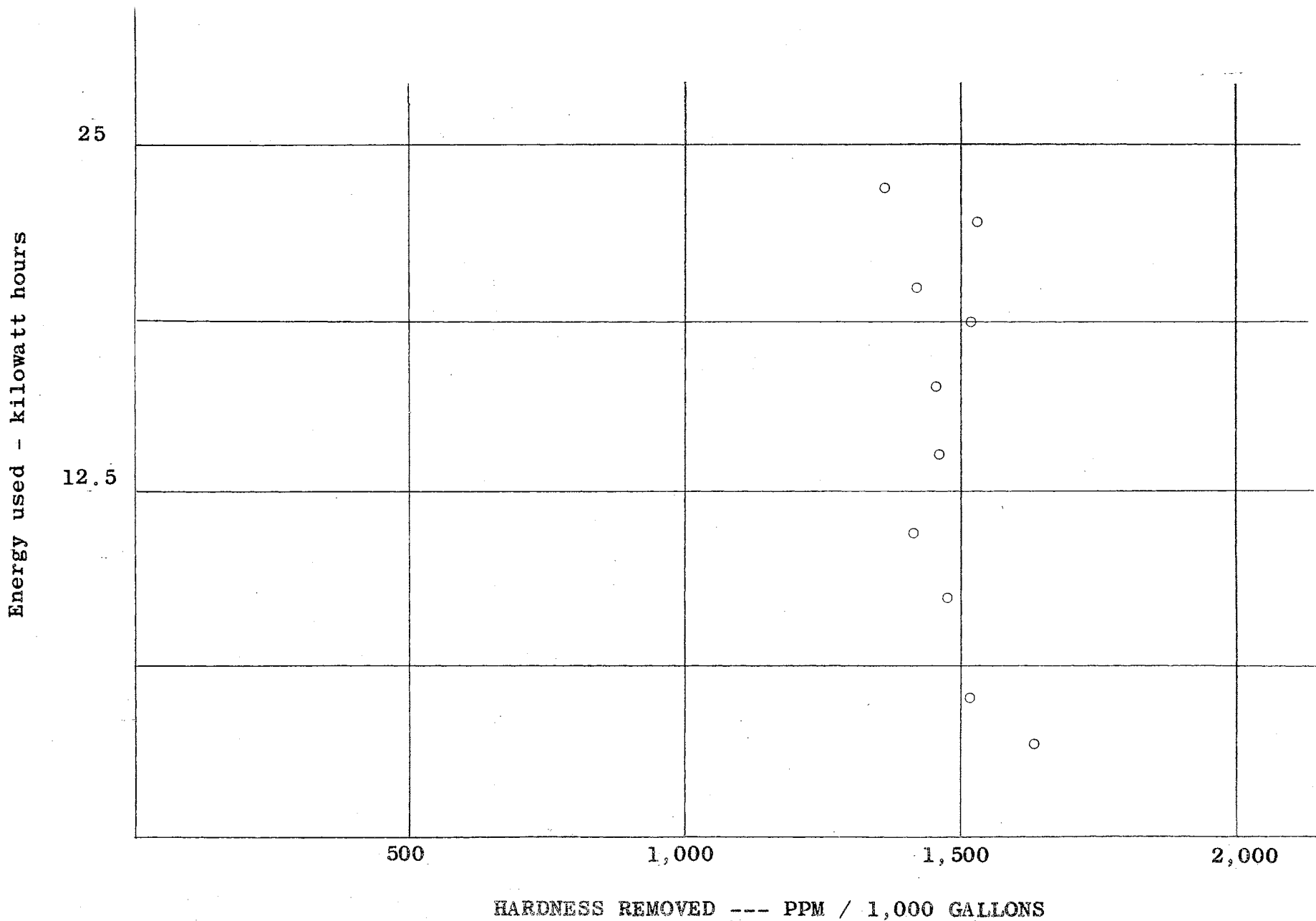Electro-ion-migration using perm-selective membranes - indicates

Figure 19   Calcium sulphate solution ---- 6 volts (table 6A) Polarity reversed
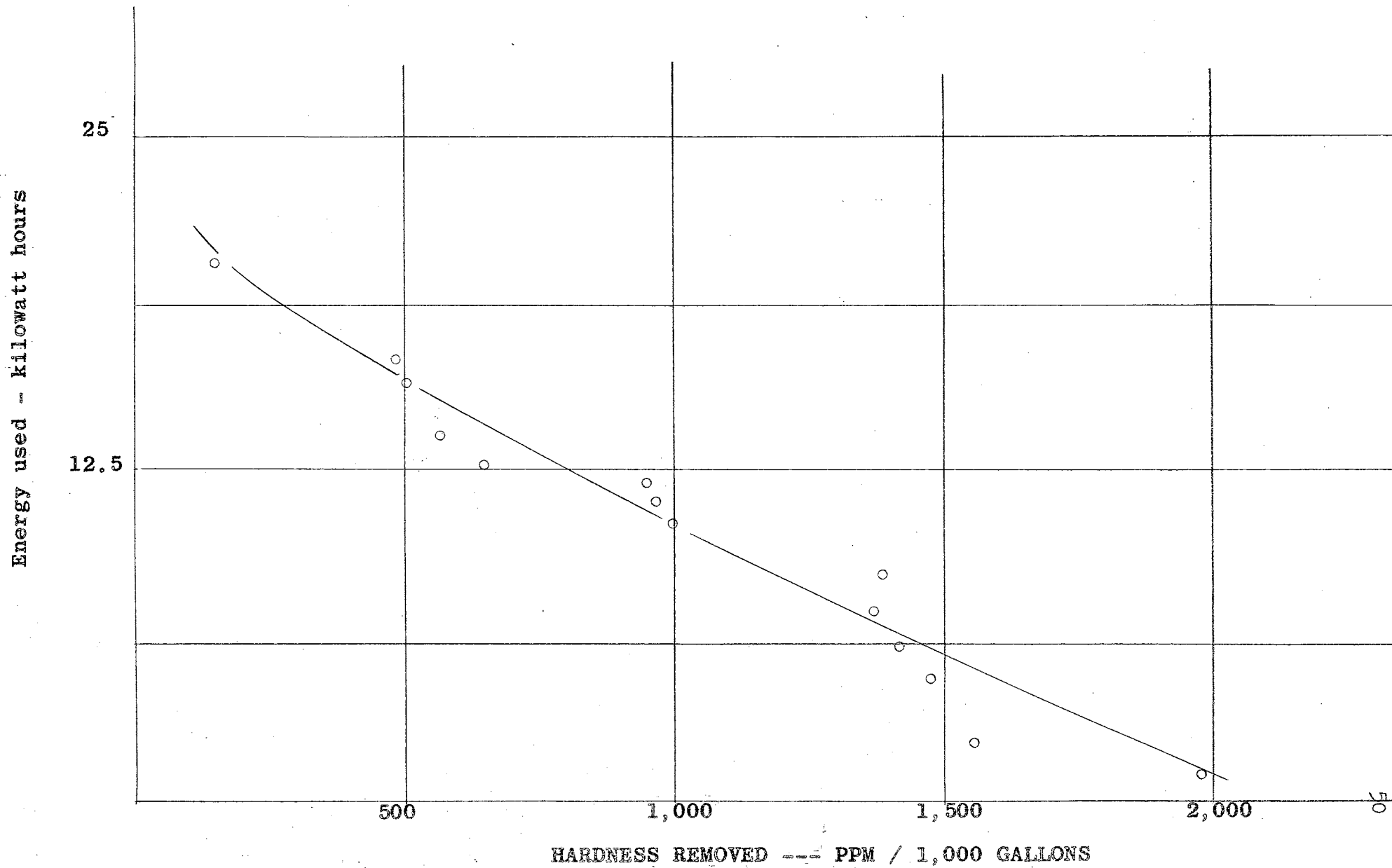   Electro ion migration using perm-selective membranes - indicates

Figure 20   Mineral reduction of calcium sulphate solution --- 6 volts (table 6B)
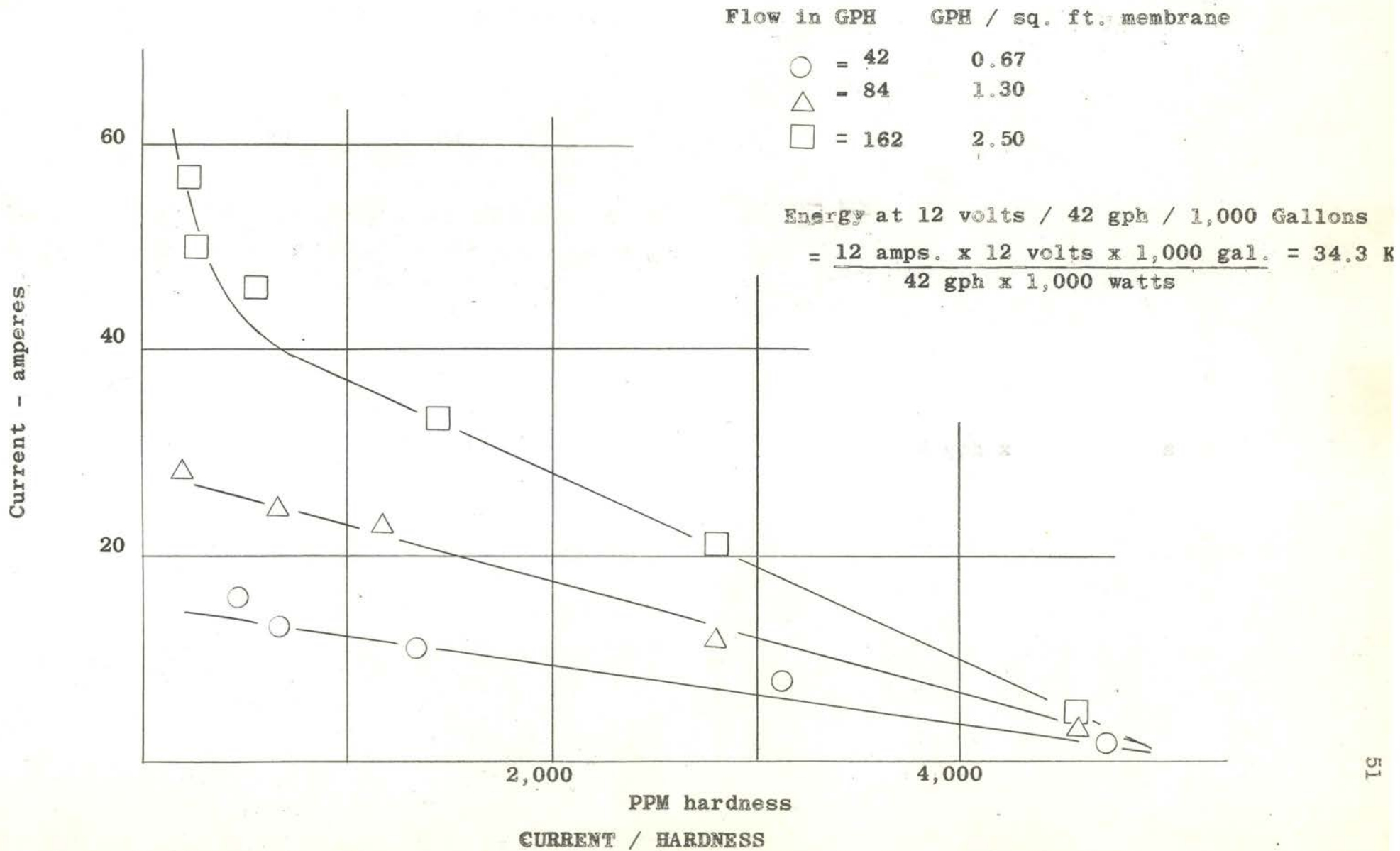Electro-ion-migration using perm-selective membranes

Figure 21 --- U. S. D. I. contract No. 14-01-001-52

CHAPTER VII

SUMMARY AND CONCLUSIONS

Results obtained from these studies as shown in Figure 17 indicate this process offers one of the most promising methods for saline reduction of water for rural supplies.

1. Optimum voltage is apparently the lowest voltage necessary to continue ion-migration. For similar saline reductions, Figure 13 indicates that approximately six times as much power is needed at 24 volts as is needed at 6 volts, Figure 19. Pulsating direct current obtained from a selenium-cell half wave rectifier gave results similar to direct current of same voltage obtained from a battery.

2. Results obtained indicate the rate of mineral reduction varies in a constant ratio with the applied voltage, except in the very low solution concentrations. At this point, the membrane resistance is less than that of the solution. More electrical energy is required with low solution concentrations. The United States Health Service states that an optimum household water should contain not more than 350 PPM hardness. However, in the interest of economy, it is questionable whether or not it is necessary to reduce mineral content to this extent for farm water supply purposes. Dr. V. G. Heller, Professor of Agricultural Chemistry, Oklahoma A. and M. College, who has supervised the testing of several thousand samples since 1924, states that water reduced to 1,000 PPM total solids, containing small amounts of sodium, is usable for household purposes. Samples tested in his laboratory

have ranged in hardness from less than 500 PPM to more than 150,000 PPM. The mean of 23 samples taken at random, from 8,000 samples is 6,312 PPM.

3. Electrical energy used for each of the chemicals commonly found in farm water supplies varies as the equivalent weights of these chemicals.

4. The time rate of energy per unit weight of mineral removed varied in a direct ratio, in concentrations above 400 PPM, from six volts to approximately thirty-six volts. At higher voltages the mineral passing through the membrane adhered to the electrode side of the membrane. The mineral appeared not to go into solution fast enough to avoid adhering to the membrane. A larger electrode or an agitation of electrode solution could possibly prevent this. In a continuous flow unit this waste material could be drained off fast enough to maintain a low concentration of minerals.

5. Efficiency of mineral reduction of saline solution appears to vary directly with the increase in current flow except in solutions of low mineral concentrations.

6. Energy required for reduction of mineral content of a known water supply appeared not to vary, in general, from the sum of the energies required to remove an equal sum of each chemical separately as in item 3.

7. Sufficient date was not obtained to determine the optimum current flow per unit area of membrane. Data available indicates that less than one ampere per square foot area of membrane is the desired electrical flow.

8. The information reported, herein, is compiled from data taken on batch apparatus. Future studies should include energy consumption and efficiency studies of continuous flow apparatus.

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