



Chemical synthesis of battery-active manganese dioxide  
by Charles E Fahlgren

A THESIS Submitted to the Graduate Faculty in partial fulfillment of the requirements for the degree  
of Master of Science in Chemical Engineering  
Montana State University  
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A portion of the second year's work on a project designed to survey chemical syntheses of battery-active manganese dioxide is presented here. Adaptation of domestic ores to commercial production of battery-active manganese dioxide by inexpensive chemical syntheses is the ultimate objective of the project.

This report includes discussion of sample preparation and performance of manganese dioxide samples from eight reactions of chemically pure reagents. Also reported are chemical analyses of the specimens, consisting of values for total manganese, available oxygen, moisture content, and pH. Apparent densities, bobbin weights, and initial drain test performances are tabulated with the chemical analyses.

Two reactions have produced samples which have met initial drain specifications; the reactions are as follows: calcium hypochlorite oxidation of manganous chloride and calcium hypochlorite oxidation of manganous nitrate.

CHEMICAL SYNTHESIS OF BATTERY-ACTIVE  
MANGANESE DIOXIDE

by

CHARLES E. FAHLGREN

A THESIS

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in

partial fulfillment of the requirements

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Master of Science in Chemical Engineering

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Approved:



Head, Major Department



Chairman, Examining Committee



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Bozeman, Montana  
June, 1953

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ABSTRACT

A portion of the second year's work on a project designed to survey chemical syntheses of battery-active manganese dioxide is presented here. Adaptation of domestic ores to commercial production of battery-active manganese dioxide by inexpensive chemical syntheses is the ultimate objective of the project.

This report includes discussion of sample preparation and performance of manganese dioxide samples from eight reactions of chemically pure reagents. Also reported are chemical analyses of the specimens, consisting of values for total manganese, available oxygen, moisture content, and pH. Apparent densities, bobbin weights, and initial drain test performances are tabulated with the chemical analyses.

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## INTRODUCTION

Manganese dioxide, the depolarizing component in Leclanche type dry cells, has long been known to have battery-active qualities. As early as 1913 patents were obtained, according to Bolen and Weil (1), for chemical syntheses of manganese dioxide suitable for battery depolarization. By 1918 a synthesis of battery-active manganese dioxide by electrolysis of manganous sulfate had been developed.

Ore deposits on the African Gold Coast furnish the only good naturally occurring battery-active manganese dioxide. Gold Coast ore and synthetic ore produced by the electrolytic process today supply the manganese dioxide to United States manufacturers of dry cells. The Gold Coast supply has the inherent disadvantage of a long transportation route and the electrolytic product, while produced domestically, is necessarily expensive due to large consumption of electric power.

As a major user of the dry cells produced, the United States Army Signal Corps has stimulated and sponsored research and development studies on synthetic manganese dioxide. Such studies have been carried out by A. M. Magnuson (3), The Tennessee Corporation (9), and Western Electrochemical Company (7) and their reports show successful production of high quality manganese dioxide by electrolytic methods from domestic ores. Western Electrochemical Company now has a commercial operation producing electrolytic manganese dioxide at their plant at Henderson, Nevada.

The Signal Corps, thinking to overcome the disadvantages of distance in the case of the Gold Coast supply and of cost in respect to electrolytic production, has sponsored further research at Montana State College;

the end sought being: development of a cheap chemical synthesis of battery-active manganese dioxide from domestic ores. Investigations reported here are part of the work done in the second of two years devoted to the project designed to achieve the above goal.

Work done on the chemical synthesis project during its first year at Montana State College was reported in theses by J. A. Davidson (2) and R. L. Nickelson (5). Second year work is reported in this thesis and in theses by W. G. Moore (4) and R. Sadagopachari (6).

The effectiveness of manganese dioxide as a depolarizer in dry cells is best determined by actual testing of cells containing the specimen dioxide. A group of preliminary tests will, however, sometimes give indications of how the specimen will perform in a battery. These tests are as follows: chemical analysis, x-ray diffraction, electron diffraction, and electron microscopy. Of these tests, the x-ray diffraction seems to be the best means of determining the presence of the crystalline phase, called gamma, that supposedly gives optimum battery performance.

The extent of the second year investigations of the chemical synthesis project includes development of the more favorable reactions tried by Davidson (2) and Nickelson (5) and testing other reactions previously untried at Montana State College. In all, sixteen reactions were studied; eight are reported here and eight are reported by Moore (4).

## SPECIFICATIONS

The specifications following have been tentatively established by the Army Signal Corps as of November 17, 1949, for acceptable synthetic manganese dioxide depolarizers. These specifications were taken from Bulletin SCL-3117-A (8).

### Chemical Composition

Available oxygen as % MnO <sub>2</sub>	85.0% min
Total manganese as % Mn	58.0% min
Absorbed moisture as % H <sub>2</sub> O	3.0% max
Iron as % Fe (soluble in HCl)	0.2% max
Silicon as % SiO <sub>2</sub>	0.5% max
Other metallic impurities	0.1% max
pH range	4 to 8

### Crystalline Phase

From crystallographic and microstructural analyses predominance of the crystallized phase known as gamma manganese dioxide should be evident.

### Particle Morphology

The particle shall present irregular shapes with no evidence of cleavage (that is, with non-rectilinear profiles having nebulous rather than sharp, well-defined edges) when examined at direct magnification from 5,000 to 20,000 diameters.

### X-Ray Diffraction Pattern

Specimen diffraction pattern should be characterized by the diffuse-line pattern of gamma manganese dioxide. Significant lines,  $d$ , of gamma manganese dioxide and their respective relative intensities,  $I$ , for iron

radiation comprising this specification are as follows:

<u>d</u>	<u>I</u>
4.00	5
2.45	7
2.14	7
1.65	10
1.40	5

Figure 3, in the Appendix, shows the various diffraction patterns of the seven types of manganese dioxide.

Particle Size

Particles should be of such a size that 95 percent of the specimen shall pass through a U. S. Standard Sieve No. 325.

Apparent Density

The bulk density of the specimen as measured by a Scott volumeter should be not less than twenty grams per cubic inch.

Battery Life

The specifications for discharge of standard A size dry cells are as follows:

	<u>Low drain</u>	<u>High drain</u>
Type discharge	continuous	continuous
Discharge resistance	166.7 ohms	16.67 ohms
End voltage	1.13 volts	1.00 volts
Temperature	70°F	70°F
Relative humidity	50%	50%
Battery life	130 hours	5.5 hours

## MATERIALS AND METHODS

### Reagents

Although the goal of this project is to develop chemical syntheses for producing battery-active manganese dioxide from commercially available ores, only C. P. grade chemicals have been used in this study. The immediate problem was to produce specification grade product, leaving purity difficulties inherent in work with ores to be overcome during a later phase of the project. Data obtained in this study with pure reagents will be used as a guide in selecting processes suitable for study in adaptations of commercial ores.

### Syntheses

Methods of preparations of samples discussed in this report are shown in the Appendix in Table I. Important variables encountered were reaction temperature, concentration, and reaction time. Some tendencies were indicated with respect to correlation of specimen properties with reaction variables; these tendencies are noted in the discussion section. The wide scope of this investigation and time limitations have prevented the making of complete studies of reaction variables. Studies that have been made were on batchwise reactions, leaving continuous processing as a future development.

### Battery Fabrication and Testing

Standard A size dry cells were constructed in accordance with usual cell assembly techniques; Signal Corps specifications were followed in proportioning the various mixtures and solutions that were used. A mixture containing 80% by weight of the MnO<sub>2</sub> sample, 18% Shawinigan

carbon black, and 12%  $\text{NH}_4\text{Cl}$  were mixed in a pebble mill for 20 minutes to assure uniform consistency. This mixture was dampened with a wetting solution consisting of 5.8%  $\text{NH}_4\text{Cl}$  by weight, 8.6%  $\text{ZnCl}_2$ , and 85.6%  $\text{H}_2\text{O}$  until material acceptable for tamping was obtained.

From the mixture described above, bobbins were tamped in an apparatus as shown in the Appendix, Figure 1. Due to the corrosiveness of the mixture used in bobbin making, stellite and methacrylate plastic were utilized for the parts of the apparatus which would contact the mixture. Use of the stellite die also was advantageous since it produced smooth-sided bobbins.

Still in accord with Signal Corps specifications, the bobbins were wrapped in cotton gauze and set in zinc cans with a cold paste electrolyte containing a ratio of 50 ml of a solution consisting of 23.7%  $\text{NH}_4\text{Cl}$  by weight, 22.3%  $\text{ZnCl}_2$ , 0.1%  $\text{HgCl}_2$ , and 53.9%  $\text{H}_2\text{O}$  to 10.7 gm of a mixture containing 74.7% cornstarch and 25.3% flour. A paper washer was placed around the carbon rod, a brass cap was pressed on the rod, and then the bobbin was sealed in the can with wax.

After a period of four days the batteries were discharged through test resistances as partially diagrammed in the Appendix, Figure 2. The test rack consisted of eight 16.67 ohm resistances and twenty-four 166.7 ohm resistances. End voltages for the two drain tests were 1.13 volts for the 166.7 ohm drain, low drain, and 1.00 volts for the 16.67 ohm drain, called high drain.

#### Oxygen Analysis

Available oxygen determinations were made in accordance with Signal Corps methods as stated in Bulletin SCL-3117-A (8). The determination

was made as follows: To 0.2175 gm of dried  $MnO_2$  was added 50 ml of 0.20 N  $Fe(NH_4)_2(SO_4)_2$  solution containing 175 ml con  $H_2SO_4$  per liter. Another 50 ml of the  $Fe(NH_4)_2(SO_4)_2$  solution was pipetted into a second beaker to run as a control sample. Both samples were diluted to 200 ml with distilled water and digested on a steam plate. After digestion they were readjusted to the original 200 ml volume and titrated with 0.2 N  $K_2Cr_2O_7$  solution. Since a suitable indicator was not available, a potentiometric titration was run using a type K potentiometer, calomel cell, and platinum electrode. The available oxygen was computed as four times the difference in volume of dichromate solution used in the sample and blank and reported as percent manganese dioxide.

#### Total Manganese Analysis

Portions of the specimen weighing 0.10 gm were dissolved in 10 ml water and 5 ml con HCl and the solution was evaporated almost to dryness. To this mixture was added 25 ml con  $H_2SO_4$  and 10 ml con  $HNO_3$  and the contents were heated till all traces of brown fumes were expelled. After cooling the solution, 275 ml water and 3 gm  $NaBiO_3$  were added. Excess oxidant was removed with a sintered-glass funnel and the material was also washed free of permanganate with hot water. This filtrate was poured into 50 ml of an oxalate solution and the excess was titrated with 0.1 N  $KMnO_4$ .

#### pH Determination

A part of the manganese dioxide specimen weighing 0.75 to 1.00 gm was digested with 100 times its weight of distilled water for ten minutes at the boiling point. Volume was readjusted to the original and the solution was cooled. The pH of the supernatant liquid was then determined with a

Beckman pH meter.

Density

Densities were determined by running the specimen through a Scott volumeter into a cubic inch container. The apparent densities were then reported as grams per cubic inch.

Moisture Content

A sample weighing one to three grams was dried for sixteen hours at a temperature of 110°C. The loss in weight was taken as the moisture content.

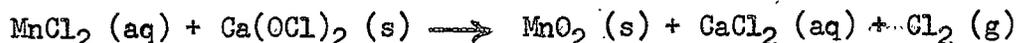
## DISCUSSION OF RESULTS

Methods of preparation and analytical and test data for the manganese dioxide specimens produced in this study are shown in the Appendix, in Table I and Table II. Some samples were sent to the Signal Corps Laboratory for confirmation of the data obtained at Montana State College; in general the agreement has been good, with the Signal Corps reporting slightly better battery life than was obtained here for the same samples.

This discussion is divided into eight parts according to the reactions studied. Reactions are formulated in the most probable manner as indicated by experimental observations, some thermodynamic calculations, and literature references such as Bolen and Weil (1). In some cases, however, the reaction by-products are of uncertain composition. Manganese dioxide was the only reaction product analyzed and evaluated.

A code was used to designate different samples, the first portion indicating the manganese source (N for manganese nitrate, S for sulfate, etc.) and the second indicating the treatment (CH for calcium hypochlorite, H for heating, etc.).

### Calcium Hypochlorite Oxidation of Manganous Chloride



Samples K-CH-10 and K-CH-11 were made with bleaching powder, calcium chloro-hypochlorite ( $\text{CaOCl}_2$ ), and they both have poor chemical characteristics and drain tests. Starting with K-CH-12 a pure calcium hypochlorite,  $\text{Ca}(\text{OCl})_2$ , was used in making the rest of the K-CH- series.

Samples K-CH-12 through K-CH-27 are generally within specifications for chemical analysis; the apparent densities of these samples are on the

average five to ten grams per cubic inch below specifications. Density does not seem to correlate well with other properties however, as evidenced by comparison of densities, bobbin weights, and drain tests of samples such as K-CH-17 (10.1 gm/in<sup>3</sup>, 10.2 gm bobbins, 3.4 hrs high drain, and 108 hrs low drain) and K-CH-25 (13.6 gm/in<sup>3</sup>, 8.9 gm bobbins, 5.7 hrs, and 149 hrs). Correlation between density and bobbin weight is of a doubtful nature; K-CH-17 (10.1 gm/in<sup>3</sup>) had an average bobbin weight of 10.2 gm while K-CH-20 (16.7 gm/in<sup>3</sup>) also had an average bobbin weight of 10.2 gm.

Bobbin weights, being proportional to the amount of material forced into the bobbin die, are more dependent on tamping qualities of the specimen than on its apparent density. A specimen having good tamping qualities usually tends to be easily wettable by the solution used to dampen the tamping mixture. Specimens that were pebble-milled for an hour or two needed less wetting solution than unmilled samples and on the average they produced a higher weight bobbin. Comparisons of K-CH-11, unmilled, with an apparent density of 9.7 gm/in<sup>3</sup> and bobbin weight of 8.7 gm and K-CH-20, pebble-milled, with 16.7 gm/in<sup>3</sup> as its density and 10.2 gm as its bobbin weight show the effect of pebble-milling on tamping qualities since 22.2 ml wetting solution seemed to give optimum dampness to the sample having 8.7 gm bobbins and on the other hand 9.5 ml on a similarly sized batch gave 10.2 gm bobbins. Since pebble-milling seemed advantageous with respect to bobbin weights, samples from K-CH-19 on were milled.

In an attempt to ascertain reaction conditions producing specimens that would give the best battery life, eight samples, K-CH-20 through K-CH-27, were prepared at four temperatures and two concentrations.

Figures 4 and 5 in the Appendix show plots of temperature versus battery life for the eight samples. From the array of data, although scant, some tendencies might be indicated for batch preparations with  $\text{Ca}(\text{OCl})_2$  additions over a 3.5 hr period. Better battery life was produced by the sample from the more concentrated solution within the 30 to 90°C temperature range. Temperature optimums are indicated at 50°C to 70°C for the concentrated solutions while the curves of the dilute reactions rise to the left with the exception of one erratic point at 50° on the high drain curve.

Need for further study at lower temperatures is indicated from the tendencies shown in the temperature versus battery life curves for dilute reactions.

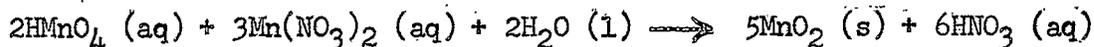
Reaction time for samples K-CH-20 to K-CH-27 was said to be 3.5 hours; this meant that the  $\text{Ca}(\text{OCl})_2$  was added to the reaction mixture over a period of 3.5 hours. Actual reaction is apparently almost instantaneous since immediate evolution of chlorine gas from the mixture upon addition of  $\text{Ca}(\text{OCl})_2$  has been noted.

Ten of the seventeen samples prepared by this type synthesis under various reaction conditions have met the initial drain specifications for low drain life, three have met high drain specifications, and two samples have met both initial drain specifications.

Commercial exploitation of this reaction would probably hinge on a favorable development of a recycle process that would regenerate the hypochlorite material. Density improvement can be achieved by pebble-milling, pH control by water washing is fairly easy, and specification grade product can be made under various conditions. Contingent upon

obtaining material of sufficient purity from ores and of sufficient cheapness through recycling, commercial production of battery-active  $MnO_2$  by this reaction is feasible.

Permanganic Acid Oxidation of Manganous Nitrate



Permanganic acid used in this synthesis was made by reacting barium permanganate and sulfuric acid and filtering off the barium sulfate precipitate. Due to the high cost of barium permanganate and the consequent expensiveness of the permanganic acid and the relatively poor drain tests of samples M=N-1, M=N-2, and M=N-3, work on this reaction was stopped after the three samples were made.

Battery life of M=N-2 and M=N-3 (3.6 hrs high drain, 104 hrs low drain, and 3.4 hrs and 104 hrs) is roughly comparable to natural Gold Coast ore performance. Both of these samples were pebble-milled and their bobbin weights were within the 9.5 to 10.5 gm specifications.

Control of pH to the 4 to 8 range called for in the specifications was not easily obtained by water washings. The acid produced by the reaction was removed only after extensive washings and in the case of M=N-2 the sample was given a dilute basic wash, which apparently did not hurt its battery life by comparison with the similar but water-washed M=N-3.

Further development of this reaction is needed to realize its potentialities. The reaction is attractive yieldwise; a high percentage of the starting materials emerge as usable product. Contingent upon finding a resin stable to  $HMnO_4$ , an ion exchange cycle to produce a cheaper per-

manganic acid may possibly be used.

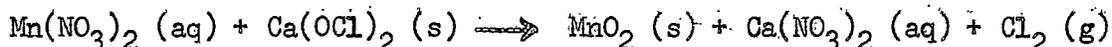
Permanganic Acid Oxidation of Manganous Hydroxide



This synthesis was part of the survey of reactions between permanganic acid and manganous salts; other reactions in this survey not included in this report are discussed in a thesis by Moore (4).

One of the two samples produced by this synthesis had a high drain battery life of 5.5 hours which met initial drain specifications. Both samples, M-OH-1 and M-OH-2, had low drain battery lives comparable to natural ore performance. Neither of the two samples was pebble-milled and their bobbin weights were 8.6 gm and 7.9 gm respectively.

Calcium Hypochlorite Oxidation of Manganous Nitrate



As in the K-CH- series the first few N-CH- samples were made with bleaching powder,  $\text{CaOCl}_2$ , and correspondingly poor analytical and test data were the result. Beginning with N-CH-10 the samples in this series were made with pure calcium hypochlorite. Starting with N-CH-12 all samples were pebble-milled before battery tests and analyses were made.

After seeing the performance of N-CH-10, 5.3 hrs high drain and 133 hrs low drain, which approaches specifications rather closely and after the subsequent good low drain tests of N-CH-11 and N-CH-12, 135 hrs and 143 hrs, a study of concentration, reaction temperature, and reaction time was planned. Samples N-CH-13 through N-CH-24 were produced under this plan. Two concentrations, 400 gm percent aqueous  $\text{Mn(NO}_3)_2$  per 2 liters water and 400 gm per 10 liters water, were used to give concentration

extremes. Three different temperatures were used--50, 70, and 90°C and two reaction times, two hours and six hours, gave contrast to that variable.

Curves were drawn through points plotted as battery life versus reaction temperature for the different concentrations and reaction times. These curves are shown in the Appendix, Figure 6 to Figure 9.

Tendencies indicated by the curves in Figure 6 seemed to be toward an optimum temperature around 70°C for the dilute reaction at two hours while no outstanding tendency is to be noted for the concentrated case. Better battery life at the lower reaction temperatures is indicated in Figure 7 in the six hour curves. Comparison of Figure 7 and Figure 4 shows a similarity between reaction temperature and high drain battery life for the K-CH- series and the N-CH- runs.

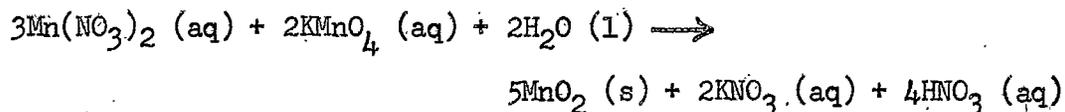
The difference between a two-hour reaction time and six hours is evident on comparison of Figure 8 and Figure 9. The curves in Figure 8 indicate better battery life on the low drain test from samples produced at the higher temperatures while the six hour curves in Figure 9 show the best performances from low temperature samples.

Tendencies of different reactions mentioned in the preceding paragraphs are not to be taken as inflexible conclusions. Attempts were made of course to hold all things equal in the series of preparations and to change only one variable at a time but some unobtrusive deviation in handling or in battery-making technique could have affected battery life.

Production problems inherent to this reaction are similar to those of the K-CH- method; ore purification and hypochlorite cost would be the

immediate deterrents to commercial production.

Potassium Permanganate Oxidation of Manganous Nitrate



The first of two samples produced by this survey reaction had good chemical characteristics, an average bobbin weight of 9.0 gm, and drain tests of 4.0 and 80 hours. The material is roughly comparable to Gold Coast ore.

The second sample, whose analysis shows it to be poorer than the first, has not been battery tested at the time of this writing. Both samples had a fairly high water content, indicating possibly the presence of a slightly deliquescent hydrate.

Fuller potentialities of this reaction may be realized by further study.

Sodium Hypochlorite Oxidation of Manganous Nitrate



Three of the four N-SH- samples tested have low drain battery lives of over the 130 hours specification for initial tests. Chemical properties of the samples were good, the pH was easily brought within specifications by water washes, and good bobbin weights were readily achieved.

This reaction has good potentiality of producing samples that will meet all specifications. If samples cannot be made that meet high drain standards, however, thought has been given to making a blend of an N-SH- sample with its excellent low drain capability and a sample giving good high drain life. No sample blends have been tried here as yet but a

common practice of battery companies is to upgrade their Gold Coast ore with the more expensive electrolytic product.

Thermal Decomposition of Manganous Oxalate



Fulfillment of the preceding reaction was attempted at various temperatures and by different means, i.e. by open dish heating in air and by heating under vacuum and letting in air onto the supposed MnO. The methods that were tried failed to produce manganese dioxide; the products probably were manganese sesquioxide,  $\text{Mn}_2\text{O}_3$ , or hausmannite,  $\text{Mn}_3\text{O}_4$ , depending on the temperature of decomposition. Thermodynamic calculations indicated formation of the lower oxides over the dioxide.

This reaction might be of possible value in making pure  $\text{Mn}_2\text{O}_3$  or pure  $\text{Mn}_3\text{O}_4$  to be later used in a leaching process that produces battery-active  $\text{MnO}_2$  as discussed by Moore (4). The sulfuric acid leach of OX-H-5 produced a sample with a good chemical analysis but the sample has not yet been battery tested.

Sodium Hypochlorite Oxidation of Manganous Sulfate



The sample produced by this survey reaction gave a battery life of 4.0 hours high drain and 99 hours low drain, putting it in the Gold Coast ore class. As with the other reactions in which only a few samples were made, the full capabilities of this synthesis can only be seen after further testing.

SUMMARY

1. Pebble-milling a sample for one to two hours seems to improve the tamping qualities and subsequent bobbin weights of the sample.
2. All reactions reported here, with the exception of thermal decomposition of manganous oxalate, have produced samples giving as good or better battery drain tests than natural Gold Coast ore.
3. Specification drain tests have been achieved by the following reactions:

High drain only--permanganic acid oxidation of manganous  
hydroxide

Low drain only--sodium hypochlorite oxidation of manganous  
nitrate

High drain and low drain--(1) calcium hypochlorite oxidation  
of manganous chloride and (2) calcium hypochlorite oxidation of manganous nitrate.

ACKNOWLEDGEMENT

The author acknowledges with thanks the support of the United States Army Signal Corps which sponsored this research.

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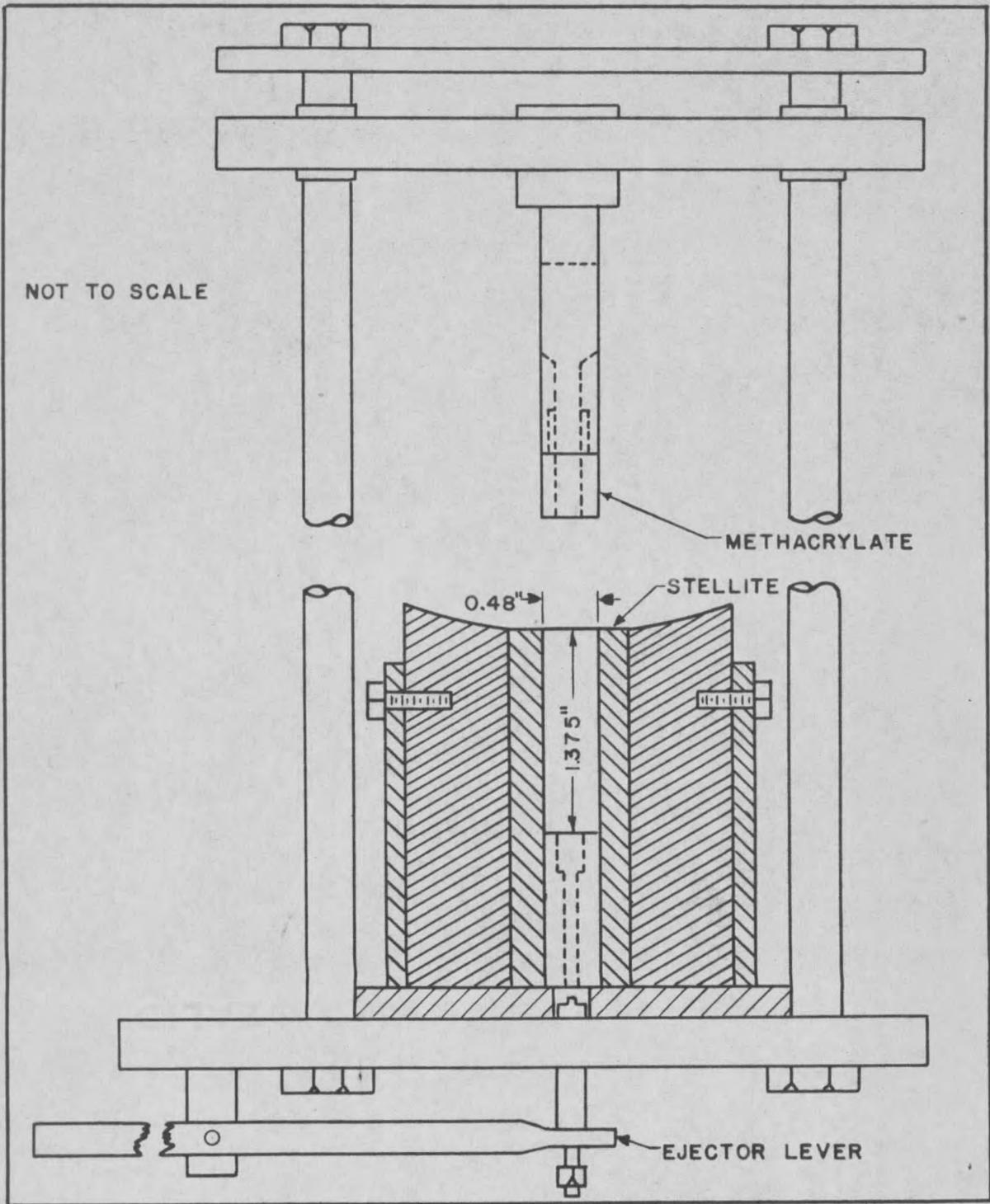


Figure 1. Bobbin Tamping Apparatus

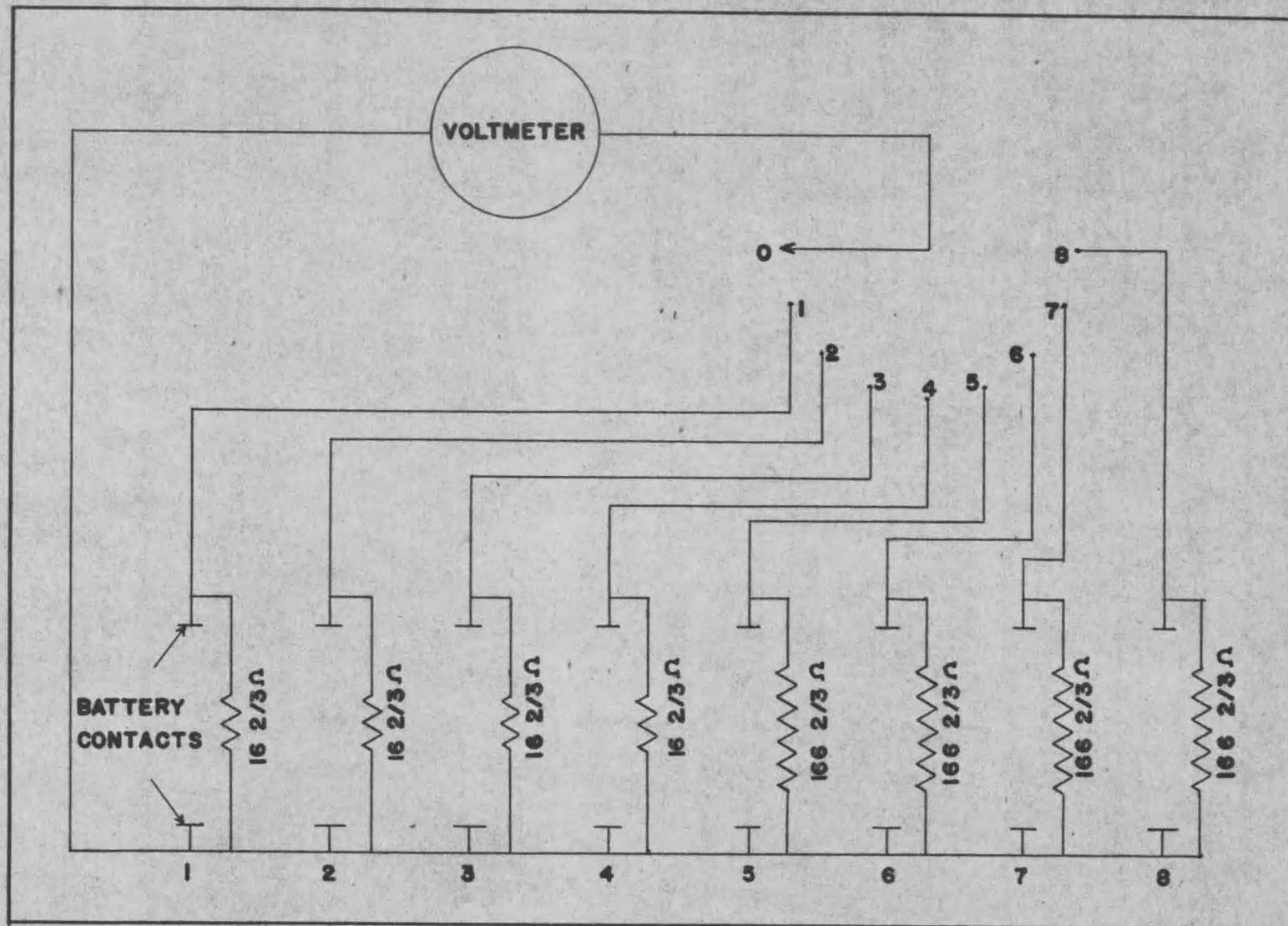


Figure 2. Schematic Diagram of Battery Test Rack





























