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BRACKISH WATER DESALTER PACKET

M. E. Seigel, et al

AAI Corporation

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BRACKISH WATER DESALTER PACKET

Final Report
Contract No. DAAD05-69-C-0320

By
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October 1971

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13. ABSTRACT

An investigation was conducted into the desalting of brackish water by means of precipitants and ion exchange resins. Limited tests indicate that the desired level of desalination can be achieved in less than one hour by mixing approximately 9.0 grams of precipitation chemicals or 24.0 to 30.0 grams of mixed-bed ion exchange resins in one quart of a synthetic form of brackish water. The precipitants were obtained as a proprietary blend from Ionac Chemical Co., while the ion exchange resins were supplied by two manufacturers, Diamond Shamrock Chemical Co. and Dow Chemical Co.

The bulk of our development effort was concentrated on the precipitation method, which - because of its more advanced state-of-the-art - appeared to offer a greater chance of success within the limited time and funds available. Consequently, final delivery to the U. S. Army Land Warfare Laboratory consisted of precipitation chemicals sealed in 100 plastic packets, each of which contained two sections of 4.5 grams each. Thus, each packet is capable of desalting 2 one-pint or 1 one-quart quantities of brackish water in accordance with the instructions provided thereon.

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ABSTRACT

An investigation was conducted into the desalting of brackish water by means of precipitants and ion exchange resins. Limited tests indicate that the desired level of desalination can be achieved in less than one hour by mixing approximately 9.0 grams of precipitation chemicals or 24.0 to 30.0 grams of mixed-bed ion exchange resins in one quart of a synthetic form of brackish water. The precipitants were obtained as a proprietary blend from Ionac Chemical Co., while the ion exchange resins were supplied by two manufacturers, Diamond Shamrock Chemical Co. and Dow Chemical Co.

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Although both desalting methods offer considerable promise, we tend to favor the use of ion exchange resins for the following reasons:

1. They are more effective in both sulfate and chloride ion removal.
2. They present no toxicity problem.
3. They are easily removed by filtering.
4. They are less expensive.

Since there are numerous problem areas still associated with the two systems, we recommended that additional effort be funded to investigate these problems, particularly in regard to reducing material weight and volume.

FOREWORD

The work described in this report was conducted for the Research Analysis Branch, U. S. Army Land Warfare Laboratory, Aberdeen Proving Ground, Maryland, as Work Assignment No. 18, entitled "Brackish Water Desalter Packets (Task 04-S-71)," under Contract No. DAAD05-69-C-0320. The work reported on herein represents development of a concept originated by Mr. Joseph L Carney, LWL Project Officer.

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I. INTRODUCTION

This engineering letter report presents the results of a limited investigation into the development of brackish water desalter packets conducted by AAI Corporation during the period 1 April 1971 through 31 August 1971.

A. Background

The U.S. Army Land Warfare Laboratory is the development agency for the Individual Aid and Survival Kit used by the U.S. Army Special Forces. This small, lightweight kit, which contains many items for survival in a hostile environment, is periodically scrutinized for deficiencies. As a result, it has been determined that the kits currently in use do not provide a means for desalting drinking water should an evadee be in an environment where brackish water is the only source of water.

B. Objectives

The following objectives applied to this work assignment:

1. Develop and test packets of different configurations containing mixed-bed ion-exchange resins for desalting brackish water.

2. Develop and test packets containing precipitants for desalting brackish water.

3. Fabricate and deliver a quantity of desalter packets.

C. Services to be Performed

The contractor (AAI Corporation) was to exert his best efforts to complete the following services in two phases:

Phase I - Investigation

1. The contractor shall design, develop, and test packets of different configurations (e.g., tea bag) containing mixed-bed ion-exchange resins for reducing total dissolved solids in brackish water in survival situations to acceptable concentrations.

2. The contractor shall design, develop, and test packets of different configurations containing precipitants for reducing total dissolved solids in brackish water in survival situations to acceptable concentrations.

3. Each packet will contain the proper amount of resin or precipitants to desalt one quart of the test water to the acceptable concentrations. It is anticipated that enough packets, namely twelve, to desalt twelve quarts of brackish water will be included as a survival item in the individual survival kit. The combined bulk of the twelve packets should not exceed three cubic inches and not exceed three ounces in weight. The method of desalting the brackish water must be a simple one; e.g., introducing the packet into a canteen of water and shaking for a few minutes.

4. The contractor will investigate the feasibility of incorporating in the desalter packets a chemical which will mask, or preferably eliminate, any objectionable odor or taste which may be in the natural brackish water. This added chemical must be included in the total bulk and weight limitations stated above.

5. The following synthetic water to simulate natural brackish water will be used for testing:

Turbidity	- 65 JTU[a]
pH	- 7.8
Sodium bicarbonate, NaHCO	- 379 mg/1[b]
Sodium chloride, NaCl	- 2,500 mg/1
Magnesium sulfate, $MgSO_4$	- 545 mg/1
Calcium sulfate, $CaSO_4$	- 612 mg/1
Sodium sulfate, Na_2SO_4	- 115 mg/1
Conductivity	- 5,270 micromhos/centimeter
Total dissolved solids, TDS	- 4,150 mg/1

6. Each packet must reduce the total dissolved solids of a quart of this test water to 2,000 mg/1, including reducing the sulfate ion to 500 mg/1.

Phase II - Test Quantities

1. Approval of the type(s) of desalter packet by US Army Land Warfare Laboratory is required before fabrication of the test quantities.

2. The contractor shall fabricate 400 desalter packets.

(a) JTU = Jackson Turbidity Unit

(b) mg/1 = milligrams per liter, equivalent to parts per million or ppm.

II. DESCRIPTION OF INVESTIGATION

A. General

As specified in the objectives for this work assignment (Section I.B.), our investigative effort leading to the development of survival-type brackish-water desalter packets was divided into two general areas: precipitants and ion exchange resins. Detailed narrative accounts of these dual investigations are subsequently presented in Sections II.B. and II.C., with tabulated summaries of all test data and discussions thereof appearing in Section III.

To enable us to determine the various concentrations stipulated in Section I.C., it was necessary to purchase - as a part of the contract - two basic pieces of equipment. These consisted of the following items, both of which are manufactured by Delta Scientific Corp., Lindenhurst, N. Y.:

1. Model 1114 Portable Conductivity Meter
2. Model 50 Portable Laboratory with Photometric Test Meter (For Performing 21 Tests With Water and Wastewater).

Descriptive literature for both units is presented in Figures 1 and 2. In addition, the manufacturer's operating procedures are reproduced in Appendixes A and B.

For measuring pH, we utilized an instrument already on hand in AAI's chemistry laboratory. This was a Model 72 pH meter, manufactured by Beckman Instruments, Inc., Fullerton, California. It is a temperature-compensated, direct-reading device with a millivolt range of 0 to $\pm$ 500

BULLETIN 1114

MODEL 1114

Portable Conductivity Meter (Total Dissolved Solids)

FEATURES

- Pocket-sized, proven instrument for laboratory, plant and field use
- All solid state for ruggedness and reliability
- Broadest coverage in single instrument, 0.3 to 10,000 micromhos, in 8 overlapping, selectable ranges
- Dual overlapping meter scales (1 to 10 and 3 to 30) avoid crowding. Color coded scales and controls preclude reading errors
- Unbreakable plastic conductivity cell
- Lightweight and very compact
- Permanent, rechargeable battery
- High Accuracy

The Delta Model 1114 Portable Conductivity Meter for measuring total dissolved solids (TDS) is an instrument with all solid state circuitry housed in a sturdy moisture-proof, corrosion resistant case. It is powered by an internal, rechargeable battery which provides 48 hours continuous duty and can be fully recharged overnight. The electrode is a rugged, unbreakable plastic cell.

Readout is in micromhos. Dual, switchable scales — 1 to 10 and 3 to 30 — are used to preclude compressing dial divisions and to provide maximum overlapping in 8 selectable ranges, thus assuring greatest possible reading accuracy.

The Model 1114 is very easy to operate. The cell is merely immersed in the sample and the range selector switch rotated until an on-scale reading is obtained. It is widely used wherever purity of water or the concentration of dissolved solids must be measured or controlled. Typical uses include checking the conductivity of water in such applications as:

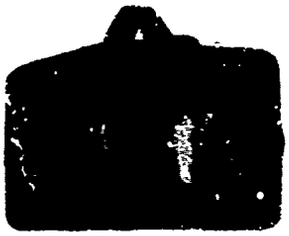
Chemical Plants • Cooling Tower Water • Coolant for Large Diesel Engines • Steam Condensate • Laboratory Stills • Boiler Feed Water • Beverage Bottling Plants • Checking Demineralizer Effectiveness • Paper Making • Textile and Yarn Processing • Food Processing • Municipal Potable Water and Waste Treatment Plants • Metal Degreasing, Anodizing, Pickling, etc. Wash Waters • Refining — and a host of others.

Figure 1. Advertising Literature for Delta Scientific Corp. Model 1114 Portable Conductivity Meter

SPECIFICATIONS

Accuracy	±2% or better for any range
Measurement Range	0.3 to 10,000 micromhos in 8 overlapping steps: 0.3 to 3, 1 to 10, 3 to 30, 10 to 100, 30 to 300, 100 to 1000, 300 to 3000 and 1000 to 10,000
Size, Instrument	6½ x 3½ x 3 inches
Weight of Instrument	2 lbs., 2 oz.
Weight of Electrode	1 oz.
Duty cycle	40 hours continuous, recharge in 14 hours
Cell Constant	1.00 (others on request)
Calibration	External knob sets full deflection

OPTIONS AND ACCESSORIES



Carrying Case

Carrying case of unbreakable ABS plastic, foam padded interior. Holds meter, and conductivity cell. Weight, approximately 1 lb.

Battery charger. Available for 110 volt, 60 Hz or 220 volt 50 or 60 Hz. Fully recharges battery overnight.

Platinizing device for replatinizing conductivity cell.

Platinizing solution, for use in conjunction with platinizing device.

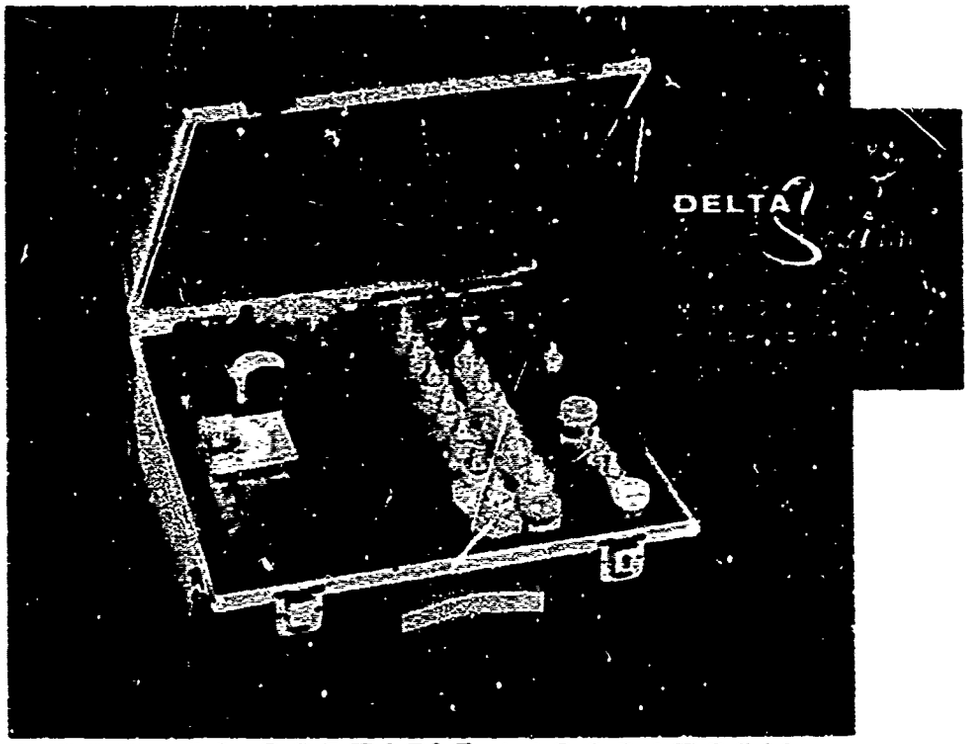
Optional cells for other than 1.00 constant. Specify requirement.

Delta Scientific Model 1114-1 Conductivity (Total Dissolved Solids) Meter complete with conductivity cell (1.00 constant) and 110 volt, 60 Hz battery charger .....

Carrying case .....

Printed in U.S.A.

Figure 1. Advertising Literature for Delta Scientific Corp. Model 1114 Portable Conductivity Meter (Continued from Preceding Page)



MODEL 50 PORTABLE LABORATORY

For Water, Wastes, Industrial Water Quality Control and Process Streams — Ideal for Government Agencies • Engineers and Consultants • Schools • Industry • Field Surveys

The Delta Scientific Model 50 Portable Laboratory is completely equipped to perform the 21 most widely required water and waste tests. The range of tests it covers and its compactness make it an ideal general purpose laboratory for health inspectors, consultants, engineers, stream survey teams and other users who find its portability and quick and easy to 1 procedures particularly attractive. While packaged for field use in a rugged case, the Model 50 also serves well in a plant or laboratory where water or waste tests are needed.

A Delta scientific Photometric Test Meter is built into the Model 50 Portable Laboratory. All of the necessary color filters, labware and reagents are included. Complete instructions for performing each test are given in the Model 50 Procedures Manual supplied with each unit. For the convenience of the inexperienced operator, all reagents bottles and powder packages are numbered individually and these simple numbers, rather than just the chemical names, are used in the direct use.

THE MODEL 50 PERFORMS ALL THESE TESTS.

Acidity	Chromate	Nitrate
Alkalinity	Copper	pH
Bromine	Hardness	Phosphate Ortho
Calcium Hardness	Hydrogen Sulfide	Phosphate Total
Carbon Dioxide	Iron	Sulfate
Chloride	Manganese	Sulfite
Chlorine, OTA & Total	Nitrite	Turbidity

Figure 2. Advertising Literature for Delta Scientific Corp. Model 50 Portable Laboratory

DESCRIPTION

The Model 50 Portable Laboratory consists of a Delta Scientific Test Meter, labware, all necessary reagents and other items for the 21 water and wastewater tests which can be performed with it. Liquid and dry powder reagents to accomplish all the tests are packed as part of the Model 50. The bottles are of two special types. One has a top that dispenses one drop at a time and is so designed that it is not contaminated if laid down, nor spilled if dropped. A second type incorporates an accurate measuring pipette as an integral part of its cap. Powder reagents are supplied in packages with miniature, accurately calibrated measuring "spoons".

Each bottle and cap and each dry reagent package is individually numbered. The test instructions refer to the reagents by their number for simplicity. The reagent bottles, powder packages and labware are nested in urethane foam for added protection.

Also supplied as part of the Model 50 Portable Laboratory are test cells and the color filters used in the Test Meter. The filters are permanently fadeproof, made of borosilicate glass in unbreakable plastic holders.

The built-in meter is powered by a self-contained, standard alkaline type "C" battery, which in normal use will last a year. Replacements are inexpensive and can be purchased in hardware or radio stores. The photometer lamp is operated well below its rating to give years of use in normal service. A self-focusing feature precludes the necessity of re-calibration when the lamp is changed. Calibration is permanent. All elements are rugged and are mounted in a metal housing within the case for additional protection. The case itself is a tough plastic composition, resistant to rough use. Weight 11 lbs. Size 6 x 12 x 12 inches

PRINCIPLE OF OPERATION

Over the years, chemical tests have been developed which cause a color reaction to occur when a water sample is treated with a reagent. These reagents are specific for the chemical determination being made. Such tests fall into two distinct categories, both of which are used in the Model 50 Portable Laboratory.

Colorimetric, in which the intensity of the color, for a measured amount of reagent, is directly related to the quantity of the substance present in a given volume of a sample. The most accurate way to measure this color intensity is with a photometric device known as a "photometer" or "colorimeter". A photometer consists of a light source falling on a photocell which is connected to a meter. Interposed in the light beam are a color filter and a cell or tube containing the sample. The amount of light admitted by the sample is displayed as a change in meter reading. Use of the color filter increases sensitivity to give results of far greater accuracy than those obtainable with equipment dependent upon the human eye. A battery operated photometer is built into the Model 50 Portable Laboratory.

Titrometric or Volumetric, in which the color change occurs abruptly as the reagent is added to the sample in carefully measured increments. Some concentration of the reagent is known, measuring the amount of it necessary to cause the color change in a given volume of sample gives a direct indication of the amount of the substance present in the sample. In the Model 50, a unique, simple "drop count" titration method is used. This avoids the use of fragile burettes, and is therefore ideal for tests in the field, and by inexperienced personnel in the plant.

TEST PROCEDURES

No special skill or special training is required to use the Model 50 Portable Laboratory for testing water or wastewater. Even an inexperienced person can make accurate measurements. Testing is simple and quick. No color judgment is required of the operator, as is necessary with comparators.

With the Model 50, colorimetric tests are made with a built-in photometric test meter. The Delta Scientific photometer is a foolproof instrument, simple to use and calibrated to give accurate results.

To perform tests with the photometer the operator merely inserts the color filter called out in the directions, fills one of the sample tubes supplied with a sample of the water being tested, depresses a button and adjusts the meter to read "0". This adjustment compensates for any color or turbidity naturally present in the water being tested.

Reagent is added to the sample in the other tube in accordance with the explicit, step-by-step instructions in the Procedures Manual. Substituting the tube with treated sample results in a specific reading on the meter that is directly related to the color intensity. This value is compared with the chart in the Procedures Manual and the result in parts per million is read off easily and accurately without any computations.

Titration tests, using the Model 50, are made by a simple dropcount method. The liquid reagents are supplied in bottles so designed that they release one drop at a time. To perform a titration test, one of the sample tubes is filled with the water being tested. The reagents are added in accordance with the instructions and the point at which the color change occurs is noted and the number of drops added to cause the sharp change in color are counted. This number is directly proportionate to the quantity of substance present and the result in parts per million is thus known.

ORDERING INFORMATION

Delta Scientific Model 50 Portable Laboratory

Includes built-in test meter, color filters, labware, reagents, etc. to perform 21 water and wastewater tests listed. Specify Delta Model 50 $248

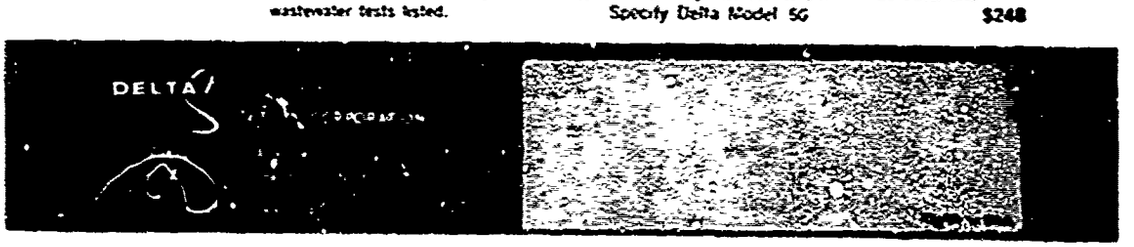


Figure 2. Advertising Literature for Delta Scientific Corp. Model 50 Portable Laboratory (Continued from Preceding Page)

(or ± 1000 by shifting zero position to either end of the scale) and relative accuracy of ±0.1 pH and ±6.0 millivolts; its repeatability is ± 0.05 pH.

In measuring the conductivity of a liquid containing more than one type of salt, it is necessary to group all the various ions of these salts together so that their concentrations may be expressed as one common standard. According to standard water analysis methods, salt content (usually referred to as "hardness") is normally considered in terms of equivalent calcium carbonate ($CaCO_3$) concentrations. This is the method used by the Delta Model 1114 conductivity meter for converting specific conductance (in micromhos per centimeter) to total dissolved solids (as ppm or parts per million). The significance of using this type of standard to convert from conductivity to total dissolved solids (TDS) is explained in the discussion which follows.

The conversion table accompanying the instructions for Delta Scientific's conductivity meter (see Appendix A) shows total dissolved solids in milligrams/liter (same as parts per million) as $CaCO_3$ for various specific conductances. Plotted, this data appears as Figure 3. Consequently, to use this table when working with dissolved solids other than calcium carbonate - as in our case - it is necessary to change the dissolved weights of such materials to their respective weights as $CaCO_3$; this is done by applying conversion factors to the individual ions (cations and anions) comprising each material. The various ions theoretically formed by the five constituents of the synthetic brackish water referred to in Section I.C. are shown in Table I.

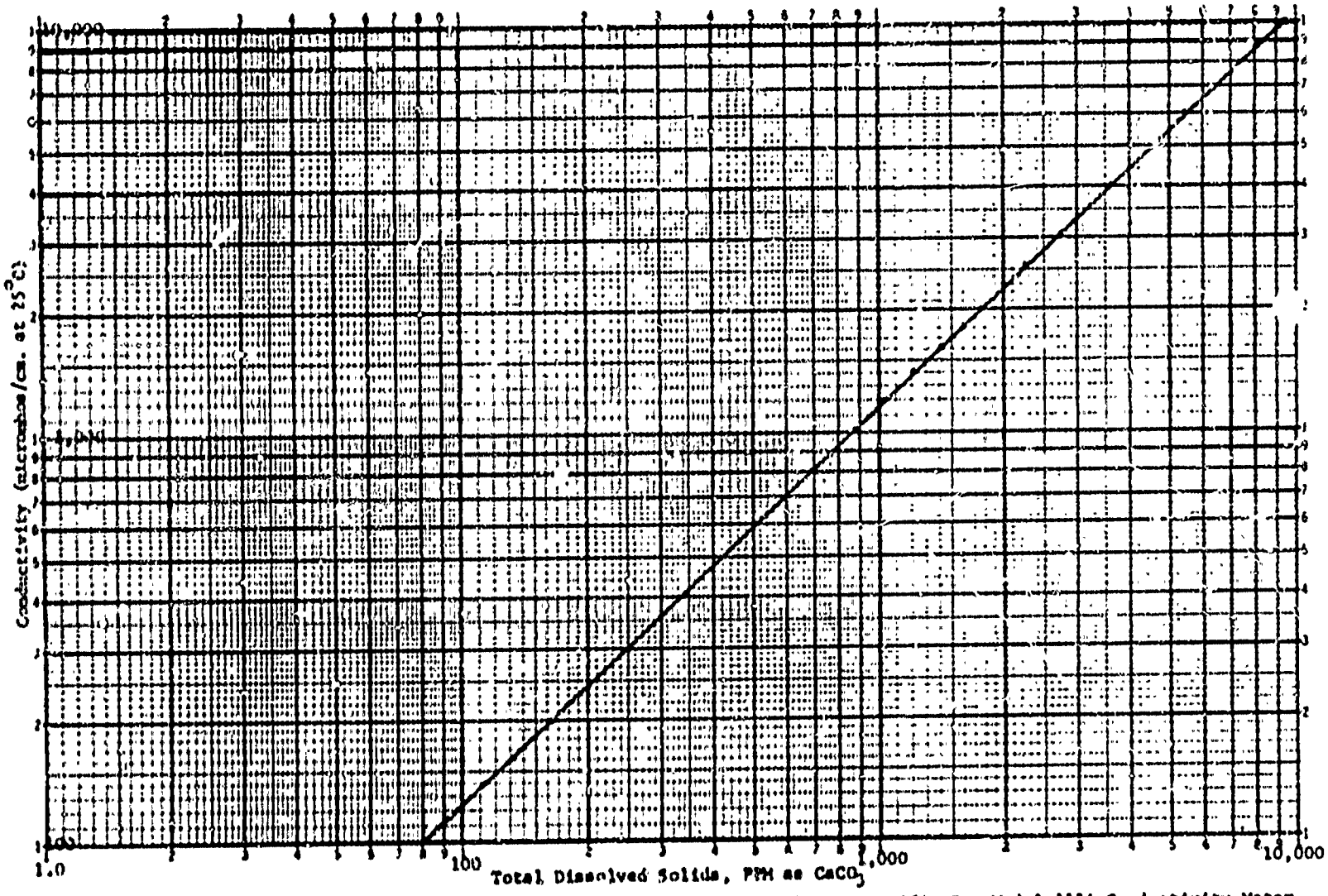


Figure 3. Plot of Conductivity vs. Total Dissolved Solids Data for Delta Scientific Co. Model 1114 Conductivity Meter

COMPOUNDS	IONS	
	CATIONS	ANIONS
$NaHCO_3$	Na^+	HCO_3^-
NaCl	Na^+	Cl^-
$MgSO_4$	Mg^{++}	$SO_4^=$
$CaSO_4$	Ca^{++}	$SO_4^=$
Na_2SO_4	$2Na^+$	$SO_4^=$

TABLE I

COMPOUNDS AND IONS FORMED

The factors to be used, therefore, in converting the six different ions (3 cations and 3 anions) listed above from concentration (in ppm) as ion to concentration (in ppm) as $CaCO_3$ are indicated in Table II.

ION	FACTOR TO CONVERT FROM PPM AS ION TO PPM AS $CaCO_3$ [a]
Cations	
Na^+	2.18
Mg^{++}	4.10
Ca^{++}	2.49
Anions	
HCO_3^-	0.82
Cl^-	1.41
$SO_4^=$	1.04

TABLE II

CONVERSION FACTORS FOR IONS

(a) Ref. 7. See page 115 for list of references.

Thus, to determine the dissolved content of each material as $CaCO_3$ in the synthetic brackish water, the following sequence of calculations should be made, using sodium chloride as an example:

1. Determine ions into which compound dissociates (see Table I).

$$NaCl \rightarrow Na^+ + Cl^-$$

2. Determine ratio of atomic weight of ion to molecular weight of compound for each ion.

$$\frac{\text{at. wt. } Na^+}{\text{mol. wt. NaCl}} = \frac{23.0}{58.45} = .395$$

$$\frac{\text{at. wt. } Cl^-}{\text{mol. wt. NaCl}} = \frac{35.5}{58.45} = .605$$

3. Determine concentration of each ion in solution <u>as ion</u> by multiplying ratio from step <u>2</u> by known concentration of compound in solution (2,500 ppm NaCl in synthetic brackish water).

$$\text{Conc. } Na^+ = .395 \times 2{,}500 = 985 \text{ ppm}$$

$$\text{Conc. } Cl^- = .605 \times 2{,}500 = 1{,}515 \text{ ppm}$$

4. Determine concentration of each ion in solution <u>as $CaCO_3$</u> by multiplying result from step <u>3</u> by factor to convert from ppm as ion to ppm as $CaCO_3$ (Table II).

$$Na^+ \text{ as } CaCO_3 = 985 \text{ ppm} \times 2.18$$
$$= 2{,}147 \text{ ppm}$$

$$Cl^- \text{ as } CaCO_3 = 1515 \text{ ppm} \times 1.41$$
$$= 2{,}136 \text{ ppm}$$

5. Determine concentration of compound as $CaCO_3$ by adding concentrations of each ion as $CaCO_3$.

	2147 ppm Na^+ as $CaCO_3$
	2156 ppm Cl^- as $CaCO_3$
Total	4293 ppm NaCl as $CaCO_3$

By conducting the preceding procedure for each of the five constituents of the synthetic brackish water and adding together the results of step 5, the total concentration of these components as $CaCO_3$ - rather than as ions - may be found. This total figure amounts to 6698 ppm (as compared to a sum of 4151 ppm as ions). The various figures produced by each calculation are presented in Table III.

Therefore, once the total dissolved solids in a solution are known in terms of $CaCO_3$, it is a simple matter to determine the conductivity of that solution - or vice versa - by referring to the conversion data or curve supplied by the meter manufacturer. A curve, based on data furnished by Delta Scientific Co. - see Appendix A - was depicted in Figure 3.[a] According to this curve, the conductivity for a TDS value of 6698 ppm (rounded off to 6700 ppm) is approximately 7200 micromhos/cm.

The preceding procedure represents the method by which all conductivity and/or TDS values quoted throughout this report were generated. The conductivity value of 5,270 micromhos/cm stated in the specification is

(a) This curve, it will be noted from the data, has been tailored to cover only that portion of the TDS range of interest to this program; namely, from approximately 100 to 10,000 ppm as $CaCO_3$.

(1) COMPOUND (a)	(2) MOL. WT.	(3) IONS FORMED (b)	(4) ATOMIC WT. OF ION	(5) RATIO OF ATOMIC WT. OF ION TO MOL. WT. OF COMPOUND (c)	(6) CONCENTRATION OF COMPOUND AS IONS (PPM) (a)	(7) CONCENTRATION OF ION AS ION (PPM) (d)	(8) CONCENTRATION OF ION AS $CaCO_3$ (PPM) (e)	(9) CONCENTRATION OF COMPOUND AS $CaCO_3$ (PPM) (f)
$NaHCO_3$	84.02	Na^+	23.0	.274	379	104	227	453
		HCO_3^-	61.0	.726		275	226	
NaCl	58.45	Na^+	23.0	.395	2,500	985	2,147	4,283
		Cl^-	35.5	.605		1,515	2,136	
$MgSO_4$	120.38	Mg^{++}	24.3	.202	545	110	451	903
		$SO_4^=$	96.1	.798		435	452	
$CaSO_4$	136.14	Ca^{++}	40.1	.294	612	180	448	897
		$SO_4^=$	96.1	.706		432	449	
Na_2SO_4	142.05	Na^+	23.0(X2)	.324	515	37	81	162
		$SO_4^=$	96.1	.676		38	81	
TOTALS		Na^+			4,151	1,126	2,455	6,698
		Mg^{++}				110	451	
		Ca^{++}				180	448	
		HCO_3^-				275	226	
		Cl^-				1,515	2,136	
		$SO_4^=$				545	982	

NOTES:

(a) Compounds (Column (1)) and their concentrations as ions (Column (6)) are as specified for synthetic brackish water by contractual scope of work.

(b) See Table I.

(c) Column (5) = Column (4) ÷ Column (2)

(d) Column (7) = Column (5) x Column (6)

(e) Column (8) = Column (7) x Conversion Factor. See Table II.

(f) Column (9) = Sum of Ion Concentrations in Column (8)

CONVERSION OF COMPOUNDS FROM CONCENTRATION AS IONS TO CONCENTRATION AS $CaCO_3$

TABLE III

inappropriate for our instrument, since - as it was learned later - it was not based on $CaCO_3$ as the standard but on NaCl, using another type of conductivity meter.[a] We elected to continue using the Delta Scientific meter with its manufacturer-generated conductivity/TDS conversion data based on $CaCO_3$, rather than attempt to generate another - and perhaps unreliable - table based on some other standard.

The synthetic brackish water utilized throughout this program was prepared in one-liter (1.057 quarts) quantities in accordance with the procedure outlined in Appendix C.

(a) This was the Model 532T1 DS Meter, manufactured by Myron L Company, Encinitas, California.

B. Desalting by Precipitation

1. Introduction

A brief literature survey was made in regard to the desalination of water by means of precipitation. Although several methods were mentioned, for the most part they were fraught with problems of a chemical or physiological nature, or they did not lend themselves to miniaturization in a survival kit. However, two techniques did appear to offer possibilities relevant to our application; these were:

a. Use of silver oxide to precipitate chlorides and neutralization with citric acid. (U.S. Patent No. 2,322,689)

b. The Permutit Process, used in marine survival kits for desalting sea water. (U.S. Patents 2,512,053; 2,600,719; 2,689,829; etc.)

Descriptions of these processes and the work conducted therewith are presented on the following pages.

2. Precipitation with Silver Oxide

In this method, the production of potable water is achieved by adding silver oxide, Ag_2O, to salt water to precipitate silver chloride - practically the only chloride insoluble in water - followed by neutralization of the resulting sodium hydroxide with citric acid, $H_3(C_6H_5O_7)$, to form harmless sodium citrate.

The reactions are as follows:

$$Ag_2O + 2\ NaCl + H_2O \rightarrow 2\ AgCl\downarrow + 2\ NaOH$$

$$3\ NaOH + H_3(C_6H_5O_7) \rightarrow Na_3(C_6H_5O_7) + 3H_2O$$

For removing sulfates, barium hydrate, $Ba(OH)_2 \cdot 8H_2O$ (or BaH, the shortened version), may be added to precipitate out as barium sulfate, $BaSO_4$, in the following manner:

$$BaH + \left\{ \begin{matrix} MgSO_4 \\ CaSO_4 \\ Na_2SO_4 \end{matrix} \right\} \longrightarrow BaSO_4 \downarrow + 8H_2O + \left\{ \begin{matrix} Mg(OH)_2 \downarrow \\ Ca(OH)_2 \downarrow \\ 2NaOH \end{matrix} \right\}$$

The products $Mg(OH)_2$ and $Ca(OH)_2$ are only slightly soluble in water, while the NaOH is easily neutralized by adding an excess of citric acid.

An attempt was made in the AAI laboratory to desalt one pint of synthetic brackish water using the procedure outlined above. The step-by-step process which we planned to follow and the calculations of the chemical reactants required are given in Appendix D.

The results of this test (#P-1) - as well as all subsequent tests - are summarized in tabular form in Section III. A brief discussion of this test, which cannot be termed successful, and pertinent observations follow.

Upon measuring the conductivity of the synthetic brackish water prior to treating, it was found to register approximately 6000 micromhos/cm instead of 7200 micromhos/cm as predicted. According to the conversion curve, this would indicate a TDS of 5500 ppm as $CaCO_3$ (6700 ppm predicted). There are several possible reasons for this variation:

1. Incomplete ionization of chemical constituents (apparently only 82% dissociated).

2. Nonconductive impurities in chemical constituents (1-2% at the most, according to analyses on container labels).

3. Errors in weighing constituents and measuring conductivity due to operator and/or accuracy of equipment. (Accuracy of analytical balance and conductivity meter were $\pm$ 2.0% and $\pm$ 0.4%, respectively.)

Although actually conducted later in the program, an attempt should have been made at this time to investigate this phenomenon and to compensate for it by increasing the TDS to the predicted value (6700 ppm), since the final weight of the desalting chemical required would be affected.

In any case, when the calculated quantity (2.35 gms) of silver oxide was first added to the solution, the conductivity inexplicably rose instead of going down. Additional increments of silver oxide (up to 2 grams) caused the conductivity to continue increasing (up to 7750 micromhos/cm). The test was stopped at this point since there was no ready explanation as to what was happening, and - in view of the time available - we were anxious to investigate other means of precipitation. This silver oxide/citric acid/barium hydrate procedure, therefore, should serve as an interesting area for future investigation.

3. Precipitation by Permutit Process

During World War II, Permutit Company, Ltd. of England developed a chemical method for desalting sea water by fliers forced down at sea. In brief, the chemical process consists of the following basic reactions:

a. $$Ag_2Z + \left\{\begin{matrix} 2\ NaCl \\ CaCl_2 \\ MgCl_2 \end{matrix}\right\} \longrightarrow \left\{\begin{matrix} Na_2Z \\ CaZ \\ MgZ \end{matrix}\right\} + 2\ AgCl\downarrow$$

Silver aluminosilicate (or silver zeolite)

b. $$Ag_2O + 2NaCl + H_2O \longrightarrow 2\ AgCl\downarrow + 2\ NaOH$$

Silver oxide included for supplemental chloride reduction.

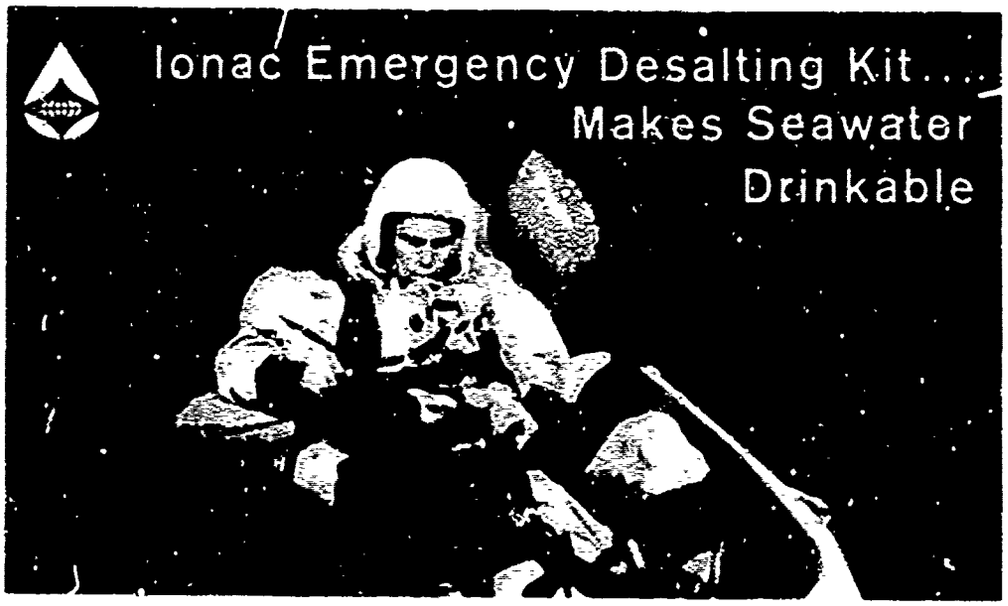
Quantity of silver oxide carefully controlled to limit formation of NaOH and resulting alkalinity.

c. $$Ba(OH)_2 \cdot 8H_2O + \left\{\begin{matrix} Na_2SO_4 \\ CaSO_4 \\ MgSO_4 \end{matrix}\right\} \longrightarrow BaSO_4\downarrow + \left\{\begin{matrix} 2\ NaOH \\ Ca(OH)_2\downarrow \\ Mg(OH)_2\downarrow \end{matrix}\right\}$$

Barium hydrate added to precipitate sulfates. Its water of hydration is apparently taken up by large excess of dry silver zeolite during blending.

These basic chemicals - silver zeolite, silver oxide, and barium hydrate - are compounded together into briquette form and supplied as components in a kit for use in emergency situations for desalting sea water. Such desalter kits are currently marketed by Ionac Chemical Co., Birmingham, New Jersey[(a)] in accordance with Military Specification MIL-D-5531. (See reproduction of advertisement in Figure 4.) Other constituents of the briquette are activated carbon (about 0.2 gram) for taste and odor improvement, stearic

(a) One other known source of similar desalter kits is Van Brode Milling Co., Inc., Clinton, Mass.; however, this company was not very cooperative in giving (or selling) sample quantities.



This compact kit can prevent the torture of thirst and the danger of body dehydration in accidents at sea. Available in two sizes, the eight hanpet kit is packed in a container, having a volume of only about one pint, yet provides more than seven pints of drinking water from seawater.

Developed by Ionac during World War II and still standard equipment on U.S. Navy and Air Force planes, also used by commercial, and private aircraft for over water flights. Now approved by the U.S. Coast Guard as a replacement for a certain portion of canned water carried on board merchant vessels, life boats and life rafts. Also used by all types of pleasure crafts.

Because of its emergency use and the fact that it may be called upon to save lives, the kit is manufactured under rigid controls. More than 20 tests are made before acceptance.

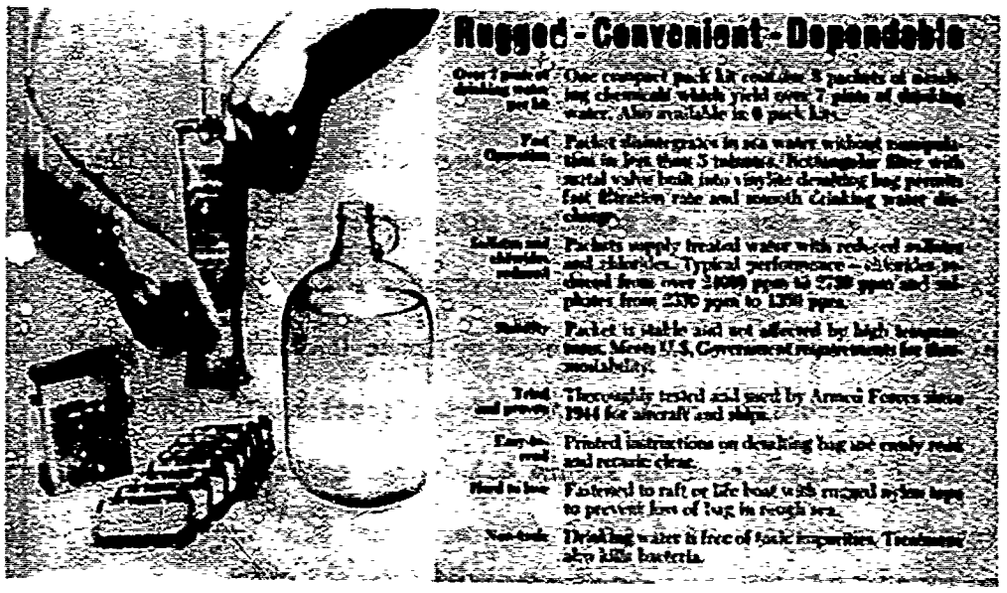


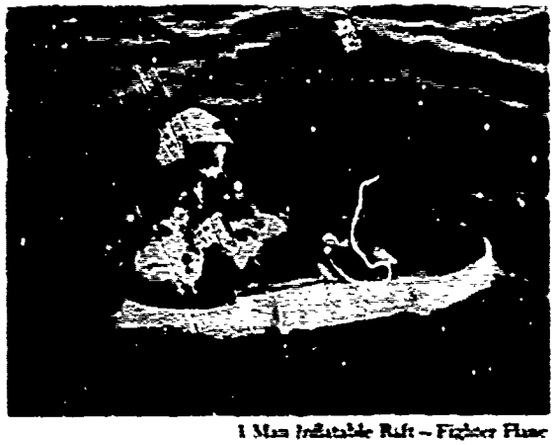
Figure 4. Advertising Literature by Ionac Chemical Co. for Emergency Desalting Kit

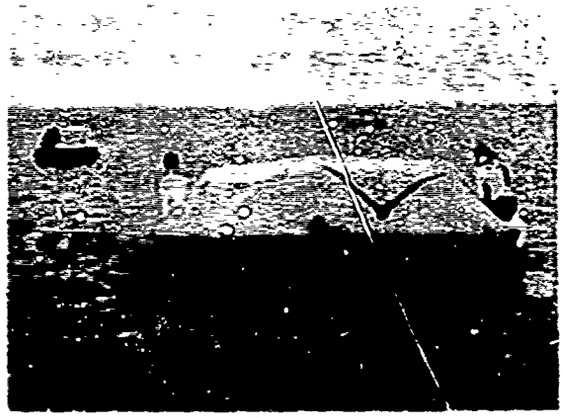
Desalter Kit Specifications

	[illegible]	[illegible]
Size Overall	[illegible]	[illegible]
Weight (Max.)	[illegible]	[illegible]
No. of Packets	[illegible]	[illegible]
Volume of Drinking Water per Packet	[illegible]	[illegible]
Total Drinking Water per Kit	[illegible]	[illegible]

Container – Electrolytic tin plate. Vertical lock seams sealed with spar varnish. Double bottom seams. Close-fitting hinged top which can be closed tightly each time can is opened. Baked enamel finish with directions lithographed on outside.

Processing Bag – Constructed of rugged translucent, waterproof plastic. Snap and strap top closure. Built-in, mildew proof filter with stainless steel drain valve. Instructions and filling line printed on bag.





1 Man Inflatable Raft – Fighter Plane or 25 Man Inflatable Raft – Merchant Marine

IONAC CHEMICAL COMPANY

A Division of Pfaudler Permutit Inc.

Birmingham, N. J.

Pd. U.S.A. Copyright: Ionac Chemical Company 1963 XXXX-7/63-3M

Figure 4. Advertising Literature by Ionac Chemical Co. for Emergency Desalting Kit (Continued from Preceding Page)

acid as a lubricant during briqueting, and bentonite clay as a disruptor to disintegrate the briquette when immersed in water. After treatment for a prescribed period, the resulting precipitants are removed by filtration and the desalted water is then ready for drinking.

At the suggestion of Mr. Carney of LWL - who supplied the initial briquettes for our evaluation - we investigated the application of this material to desalting brackish water. It was assumed that the quantity of chemical needed could be proportionately scaled down to remove the much lower concentration of total dissolved solids from brackish water (nearly 1/9 that of sea water). For this purpose, the following simple calculations were made:

a. Weight of briquette = 67 grams

b. One briquette reduces TDS of 1 pint of sea water from 35,000 ppm as ions to 2,750 ppm, a difference of 32,250 ppm (according to information supplied by Ionac Chemical Co.).

c. We want to reduce TDS of brackish water from 4,150 ppm to 2,000 ppm, a difference of 2,150 ppm as ions (or 2,800 ppm as $CaCO_3$ [a]).

(a) It is assumed removal of ions will occur in a manner similar to that described earlier for silver oxide/citric acid/barium hydrate method; that is, ion concentrations to be precipitated out will be approximately as follows:

	ppm as Ion	ppm as $CaCO_3$
Cl^-	1515	2136
$SO_4^=$	636	661
Total	2151	2797
Rounded Off	2150	2800

d. Therefore, the weight of chemical (X) needed to desalt 1 pint of brackish water is found by setting up a direct proportion and solving for X:

$$\frac{X}{2150 \text{ ppm}} = \frac{67 \text{ gms}}{32{,}250 \text{ ppm}}$$

$$X = \frac{67 \times 2150}{32{,}250} = 4.46 \text{ gms}$$

Thus, to desalt one pint of brackish water, 4.5 grams (rounded off) of chemical should be required, or 9.0 grams for one quart.

To check out this approach, we conducted a desalting test using one pint of synthetic brackish water and 4.5 grams of chemical in a loose or powder form. The chemical was converted to powder form by breaking up a briquette with mortar and pestle. Figure 5 shows before and after versions of such a briquette. Thus, the stearic acid and bentonite clay constituents remained in the powder although they took no part in the desalting process which ensued. In this test (#P-2), after about 55 minutes the TDS was reduced from an initial reading of 5300 ppm as $CaCO_3$ (equivalent to conductivity of 5700 micromhos/cm) to 3500 ppm, a difference of only 1800 ppm but significantly below the maximum allowable concentration of 3,900 ppm (i.e., 2,000 ppm as ion).

According to the separate analytical determinations made per MIL-D-5531 of the sulfate and chloride before-and-after concentrations, these ions were reduced by 540 ppm (940 to 400) and 490 ppm (1280 to 790), respectively, as ions. These values are equivalent to 562 ppm and 691 ppm, respectively, as $CaCO_3$, for a total reduction of 1253 ppm. Since the conductivity method showed a reduction of approximately 1800 ppm (as $CaCO_3$),

FIGURE 5

Precipitation Chemicals in Ionac Emergency Desalter Kit
In Briquette Form (Left) and Powder Form (Right)

it would appear - based on this limited data - that the desalting chemical, in addition to removing sulfate and chloride ions, also removes other ions to the extent of about 547 ppm (i.e., 1800 - 1253). These other ions, judging from the chemical reactions outlined earlier, were most probably calcium and magnesium, of which there was a theoretical total of 899 ppm as $CaCO_3$ (448 ppm Ca^{++} and 451 ppm Mg^{++}) initially present in the solution.

It is significant to note, however, from test #P-2 that (a) the remaining sulfate concentration (400 ppm as ion) was less than the allowed maximum of 500 ppm, and (b) the chloride content was diminished by only 38% ($\frac{490}{1280} \times 100$), instead of 100% as assumed.

In a subsequent test (#P-3), the quantities of liquid and chemical were doubled to one quart and 9.0 grams, respectively, to observe the effect of scaling up. This time, the TDS was reduced by 2,050 ppm (from 5,500 to 3,450 ppm) in just 35 minutes. Sulfate and chloride concentrations were decreased by 600 ppm and 650 ppm, respectively, as ions. Evidently, larger quantities of the chemical react considerably more quickly and more efficiently than smaller amounts. Of particular interest here are the finding of a greater concentration of sulfate ions than calculated (1100 ppm actual as compared to 945 ppm calculated) - which conceivably raises a question as to the validity of this method of analysis - and (b) the removal of 49% of the chloride ions.

The next test (#P-4), identical to #P-3, was conducted to check reproducibility. Here, the results were generally similar in that the TDS was lowered by 2,000 ppm as $CaCO_3$ (from 5200 to 3200 ppm), and the

concentrations of sulfate and chloride ions removed, as ions, were 575 ppm and 610 ppm, respectively.

At this point, based on the relatively small amount of data thus far collected, it appeared that 9.0 grams of the desalting chemical were sufficient to adequately desalt one quart of synthetic brackish water in 30 minutes. To verify this procedure, we conducted a desalting operation as it would be performed in the field; namely, add the desalting chemical (9.0 grams) to a canteenful of brackish water and agitate for 30 minutes. In this test (#P-5), the TDS was reduced from 5000 ppm (as $CaCO_3$) to 3150 ppm, a difference of 1,850 ppm. This result compared favorably to the earlier tests, in addition to demonstrating that desalination can be accomplished in the field in a relatively short period of time.

Since a tea-bag configuration was suggested as a possible means of packaging and using the chemical, we briefly looked into this concept. In test #P-6 a commercial type tea-bag was emptied of its normal contents and filled with 9.0 grams of desalting chemical. The resulting attempt at desalination did not prove highly successful, however, in that the TDS was reduced by only 600 ppm (as $CaCO_3$) after 30 minutes - less than one-third the reduction experienced in loose form - and 700 ppm after one hour. In fact, it was necessary to allow the treated water to sit overnight with agitation (by a magnetic stirrer) for a total of 17-1/2 hours in order to decrease the TDS below the desired maximum of 4300 ppm (as $CaCO_3$). Thus, it is apparent that teabag-type packaging is not advisable for this application since it does not permit the intimate contact necessary between the reacting ions to accomplish precipitation within a reasonable length of time.

The elimination of objectionable odor and taste is another desirable feature of the desalting system. To evaluate the effectiveness of Ionac's desalting chemical in this regard - in view of the presence of activated carbon - we added 9.0 grams of the material to a sample of foul-smelling liquid - containing little or no dissolved solids - supplied by LWL. After 30 minutes, the odor, although still detectable, was significantly diminished. Although this result was encouraging, to really determine the adequacy of activated carbon (or other materials), it is essential to conduct extensive additional tests with a wide variety of naturally occurring odors and tastes. Such tests, it is felt, should be carried out under close medical supervision.

As indicated earlier, the precipitation method described here depends upon silver and barium compounds to remove chloride and sulfate ions. Should there be a low level of salinity - such as in slightly brackish or fresh water - the silver and barium ions thus introduced exceed the chloride and sulfate ions available and - because they have nothing to react with - remain free in solution. In such a situation, the non-filterable silver and barium ions are taken internally when the treated water is consumed. This is potentially dangerous due to the physiological effects[a] of these chemicals on the body.

(a) The repeated ingestion of silver produces argyria, an incurable gray skin pigmentation; therefore, the U.S. Public Health Service limits its presence in normal drinking water to .05 ppm. Barium, due to the seriousness of its toxic effects, is limited to 1.0 ppm.

In the regular life-raft desalter kit, which is designed for ocean use, there is no problem in this regard due to the high concentration of total dissolved solids (approximately 35,000 ppm) in the water.[a] With brackish water, however, the user - in a survival situation - has no way of knowing the salinity of the water he intends to drink. Therefore, it is important that some simple means of detecting or measuring chloride and sulfate content be provided to enable the user to determine whether or not to use a desalter kit and to what extent (i.e., what quantity of chemical, if any, to use). No activity was conducted in this regard at AAI since it was considered to be beyond the contractual scope of work.

Upon being advised of the foregoing situation, Mr. Carney contacted Ionac Chemical Co. and arranged for us to procure a quantity of desalting chemical <u>without</u> barium hydrate.[b] Such material obviously would not be capable of removing sulfate ions. The presence of sulfates in drinking water, depending on the concentration, acts as a cathartic to produce diarrhea and other intestinal ailments. It is limited to 250 ppm by the USPHS.

(a) Major constituents, in ppm, of sea water are as follows:

Na^+	- 10,561	Cl^-	- 18,980
Mg^{++}	- 1,272	SO_4	- 2,649
Ca^{++}	- 400	HCO_3^-	- 142
K^+	- 380	Br^-	- 65
		Others	- 34
		Total	- 34,483

(b) The exact quantity of barium hydrate - or other constituents - per briquette is unknown since Ionac Chemical Co. considers this information proprietary.

A 10-pound lot of this special material, identified as lot no. SA-516, was received in powder form; since no briquettes were to be formed, the lubricant (stearic acid) and disruptor (bentonite clay) constituents were also omitted. This lot of material was intended for use later in filling the desalter packets to be delivered to LWL (see Section II.D.).

Desalting tests (#P-8, P-9 and P-10) were subsequently conducted with the new material to evaluate its reactivity. If certain assumptions are correct as to the TDS removable by the new material - as indicated in Table IV - then the residual ion concentration should drop to only about 2300 ppm (as ion), which is above the required maximum level of 2000 ppm. The continued presence of the sulfate ions, of course, accounts for this condition.

In the first test of this series (#P-8), 9.0 grams of lot SA-516 chemical were added to one quart of synthetic brackish water and agitated. It took just 15 minutes for the TDS to reach the 3600 ppm level (as $CaCO_3$) - representing, in this case, a difference of about 2000 ppm - and 60 minutes to stabilize at 2900 ppm. Since the barium hydrate, lubricant, and disruptor were absent from the new material, it contained more silver compounds per unit weight than the old chemical and therefore should reduce the chloride concentration to a greater extent. This was reflected to some degree in that a greater percentage of chloride ions (67%) was removed than in four previous comparable tests (#P-2 thru P-5), which averaged 49% removal.

TABLE IV

ASSUMED VALUES OF TOTAL DISSOLVED SOLIDS
REMOVED BY LOT SA-516 DESALTING CHEMICAL

ION	PPM AS ION			PPM AS $CaCO_3$		
	Before	After	Removed	Before	After	Removed
Cl^-	1515	0	1515	2136	0	2136
$SO_4^=$	945	945	0	982	982	0
Ca^{++}	180	0	180	448	0	448
Mg^{++}	110	0	110	451	0	451
Na^+	1126	1126	0	2455	2455	0
HCO_3^-	275	275	0	226	226	0
Total	4151	2346	1805	6698	3663	3035

On the other hand, as might be expected, the sulfate ion concentration was reduced by a slight 15% (150 ppm as ion), whereas earlier tests produced removals in excess of 50%. The phenomenon by which even 15% was removed, considering the absence of barium, cannot be explained at this time.

Since some discussion occurred at this point concerning desalination in quantities of one pint instead of one quart, the next two tests (#P-9 and P-10) each consisted of adding 4.5 grams of new material to one pint of brackish water. In both cases, it took approximately 10 minutes for the TDS level to drop below 3600 ppm (as $CaCO_3$) and 35 minutes to

stabilize at 2750 and 2300 ppm, respectively. The sulfate and chloride levels were not determined.

In view of these results, the lot SA-516 precipitating chemical was packaged as described in Section II.D. for delivery to LWL.

C. Desalting by Ion Exchange

1. Introduction

Basically, the ion-exchange method of desalination makes use of porous resin materials to exchange their ions with other ions contacted in water containing dissolved solids. In this process, two groups of opposite charges may be combined together to form "mixed-bed" resins, which are then used to draw off certain positive-charged cations and negative-charged anions to produce relatively fresh water suitable for drinking purposes.

Ion exchange operations may be carried out as either a column or batch technique. In column operations the ion exchange resin is placed in a vertical column to form a bed. The solution to be treated is flowed through this column, with the exchange reaction taking place until a target endpoint is reached. At this point the resin, if exhausted, may be regenerated for use in another cycle; that is, have its activity rejuvenated by replacing those ions absorbed from the solution with ions identical to those originally in the resin.

In batch operations the ion exchange resin is simply agitated in a vessel together with a given quantity of the solution to be treated. When the target endpoint is reached, the treated solution is removed by decanting or filtering, and the resin regenerated or discarded. Since batch application can be more easily and quickly handled in the field, we limited our investigation to this particular technique.

To illustrate how the ion exchange process works, following are the basic reactions for removing sodium and chloride ions from salt water (R = non-active portion of ion-exchange resin):

a. $RH^+ + Na^+ + Cl^- \longrightarrow RNa^+ + H^+ + Cl^-$

b. $ROH^- + H^+ + Cl^- \longrightarrow RCl^- + H_2O$

c. $ROH^- + Na^+ + Cl^- \longrightarrow RCl^- + Na^+ + OH^-$

d. $RH^+ + Na^+ + OH^- \longrightarrow RNa^+ + H_2O$

At the start of our investigation, we were presented with several candidate ion exchange materials by Mr. Carney, who had obtained them as representative samples from their respective manufacturers. These materials were as follows:

ITEM	MANUFACTURER
Amberlite MB-3 Amberlite IRN-150	Rohm & Haas Co.
Dowex 44 Dowex 50WX8 Dowex 1X8 Mixture of Dowex 50WX8 and 1X8	Dow Chemical Co.

In addition, we contacted various companies active in ion-exchange work and/or water treatment to obtain state-of-the-art information on brackish water desalination by ion exchange methods. The following nine companies were solicited in this regard:

COMPANY	PERSONS CONTACTED
Allied Chemical Corp., Specialty Chemicals Div., Morristown, N.J.	R. B. Jackson
Diamond Shamrock Chemical Co., Redwood City, California	D. A. Javes C. S. Parks (N.Y. Office)
Dow Chemical Co., Midland, Michigan	L. J. Lefevre D. Crane (Saddlebrook, N.J. Office)
W. R. Grace & Co., Polymers & Chemicals Div., Cambridge, Mass.	G. J. Power
Graver Water Conditioning Co., Union, N. J.	J. Levandusky N. J. Nitti
Ionics, Inc., Bridgeville, Pa.	Ron Schmidt
National Lead Co., Baroid Div., Houston, Texas	R. E. Offeman
Rohm & Haas Co., Ion Exchange Dept., Philadelphia, Pa.	R. W. Percival
Union Carbide Corp., Molecular Sieve Dept., Moorestown, N. J.	W. Wolf

In essence, we learned that because of the expense of the resins and their regeneration, the process of brackish water desalination by ion exchange has received very limited commercial attention to date. Fortunately, regeneration is of no importance in our application because of the one-time, throw-away use associated with survival type operations.

As a result of the various ion exchange resins considered, we selected two systems for further - though limited - study during this program. These materials are identified as follows:

MANUFACTURER	CATION	ANION
Diamond Shamrock Chemical Co.	Duolite C20X10	Duolite A7
Dow Chemical Co.	DOWEX 50WX8	DOWEX 1X8 (Preblended)

Their selection was based primarily upon the high capacity which they offer. Capacity, expressed for our purposes in terms of milliequivalents per gram or meq./gm.,[a] represents the ion absorption ability possessed in varying degree by ion exchange materials. It is calculated in the following manner:

$$\text{Capacity} = \frac{\text{Milliequivalents (meq.) of ion to be removed}}{\text{Wt. of ion-exchange material required for removal}}$$

$$\text{where: Meq.} = \frac{\text{Wt. of ions to be removed}}{\text{Equivalent wt. of ion}} \times 1000$$

Thus, it may be seen that the higher the capacity, the lower the quantity of ion exchange material required.

The capacity values of the selected Diamond Shamrock and Dow materials are as follows:

MATERIAL	CAPACITY (meq./gm.)
Duolite C20X10	5.1
Duolite A7	9.1
DOWEX 50WX8	4.8
DOWEX 1X8	3.7

(a) Capacity may also be expressed as kilograins per cubic foot, pound-equivalents per pound, milliequivalents per milliliter, etc.

The utilization of these materials in our investigation is described on the following pages. This investigation, it must be pointed out, was admittedly brief in view of the limited time and funds available and the increased emphasis placed on the precipitation method. A summary of these test results and a discussion thereof are presented in Section III.

2. Ion Exchange With Duolite Resins

The Duolite resins, C20X10 and A7, were recommended and supplied for our use by Mr. C. S. Parks, the East Coast representative of Diamond Shamrock Chemical Co. Duolite C20X10 is a strong-acid cation in H^+ form with an affinity for Na^+, Ca^{++}, and Mg^{++} ions; the term "X10" indicates a resin copolymer with 10% cross-linking. Duolite A7 is a weak-base anion in OH^- form with an affinity for $SO_4^{=}$, Cl^-, and HCO_3^- ions. Some typical properties of these resins are given in Table V.

The "starting point" quantity of resins selected for our initial test (#I-1), using one pint of water, was a blend of 3.25 gms. C20X10 and 1.8 gms. A7, determined as shown in Appendix E. The procedure followed for this test, as well as related subsequent tests (#I-2 and I-3), is also described in Appendix E.

In test #I-1 - as in all other tests conducted - the conductivity of the synthetic brackish water fell substantially below the calculated value of 7200 micromhos/cm.; in this case, it was 5700 micromhos/cm - equivalent to a TDS of 5300 ppm as $CaCO_3$ - or about 89% of the predicted level. This indicates, among other things, incomplete ionization of the dissolved solids and should be taken into account in future studies.

TABLE V

PROPERTIES OF DUOLITE RESINS C20X10 AND A7

Property	C20X10 (H^+ Form)	A7 (OH^- Form)
Chemical Classification	Strong acid	Weak base
Functional Groups	Nuclear, sulfonic	Secondary amine
Matrix	Polystyrene	Phenolic
Physical Form	Beads	Granules
Mesh Range	16-50	16-50
Ionic Form Normally Shipped (May Be Regenerated to Other Forms)	Sodium	Salt
Capacity: Meq./ml. (Wet)	2.2	2.4
Meq./gm. (Dry)	5.1	9.1
pH Range	0-14	0-14
Maximum Temp., ^{o}C	150	40
Specific Gravity	1.30	1.12
Moisture Retention Capacity, %	48	55-62

Source: Duolite Data Leaflet No. 2, "Ion Exchange and Adsorbent Resins - List of Products, Their Properties and Uses", Diamond Shamrock Chemical Co., November 1970.

The addition of the initial 5.05 gms. of ion exchange resins reduced the TDS, after 14 minutes, to only 4800 ppm (as $CaCO_3$); our goal was 3200 ppm. Therefore, additional increments of resins were added until the latter value was reached. This required a total of 17.05 gms., added over a period of 60 minutes in 8 increments of 5.05, 1.0, 1.0, 1.0, 2.0, 2.0, 3.0, and 2.0 grams. Unfortunately, the sulfate ion reduction could not be determined due to a test malfunction. However, a reduction of 180 ppm as ion (from 1280 to 1100 ppm), or 14%, was determined for the chloride ions. The pH value dropped from nearly neutral (7.7) to a highly acidic 2.0, indicating a sour taste to the water. Although the pH of potable water is not pathologically significant, some investigation should eventually be made as to the acceptability of drinking acidic water.

In the next test, #I-2, we again used one pint of water but added a larger initial quantity of resin, 12.0 grams, with the intention of adding more if necessary. It was theorized that a relatively large amount of resin added at one time would produce more efficient results than the small increments employed in test #I-1. This proved to be correct in that the TDS reached its goal of 3200 ppm (as $CaCO_3$) in 30 minutes and eventually stabilized at 2800 ppm in 55 minutes. Therefore, no additional quantity of resin was needed. The sulfate ion concentration was reduced 825 ppm as ion (from 1050 to 225 ppm), which was 87% of the calculated amount. This time the chloride reduction was 55% (1260 to 570 ppm as ion), attesting to the increased efficiency of this procedure. Again a very low pH value (2.35) was obtained, which appears to be representative of this reaction.

The final test (#I-3) with the Duolite resins involved a full quart of brackish water, to which was added 24.0 gms. of the C20X10/A7 mixture. The test was stopped after 50 minutes when the TDS dropped below the 3200 ppm (as $CaCO_3$) level but before stabilization occurred.

Inasmuch as the feasibility of this resin system for desalting water has now been demonstrated, it is felt that additional study is warranted in order to - among other things - optimize its weight to achieve stabilization at the desired TDS level.

3. Ion Exchange With DOWEX Resins

A mixture of DOWEX resins, 50WX8 and 1X8, was submitted for our evaluation through Mr. Carney of LWL by Mr. L. J. Lefevre of Dow Chemical Co 's Technical Service and Development Department. DOWEX 50WX8 is a strong-acid cation in H^+ form, while DOWEX 1X8 is a strong-base anion in OH^- form. Table VI presents some typical properties of these resins.

The resin mixture was supplied to us in four 4-ounce bottles, each of which was supposedly sufficient to treat one quart of water containing 5,000 ppm NaCl (calculated as $CaCO_3$) and reduce its salt level to 500 ppm or less. Consequently, no calculations were required on our part in determining the quantities to be used. The mix ratio was reported as 38 gms. 50WX8 to 52.6 gms. 1X8 in the wet state, equivalent to 1.39 parts 1X8 to 1.0 part 50WX8.

In the initial test (#I-4), the procedure consisted of the following simple operations:

TABLE VI

PROPERTIES OF DOWEX RESINS 50WX8 AND 1X8

Property	50WX8 (H^+ Form)	1X8 (OH^- Form)
Type	Strong-acid cation exchanger	Strong-base anion exchanger
Copolymer	Styrene-DVB (DVB = divinylbenzene)	Styrene-DVB
Active Group	$-SO_3^-$	$-N-(CH_3)_3^+$
Standard Crosslinkage, % DVB	8	8
Ionic Form Normally Shipped (May Be Regenerated To Other Forms)	Na^+ or H^+ (20-50 mesh); H^+ (all other mesh)	Cl^-
Physical Form	Spherical beads	Spherical beads
Standard Mesh Size, Wet	20-50	20-50
Effective Size, MM	.54	.51
Shipping Weight, Lbs./Cu.Ft.	50	44
Water Retention Capacity, %	47-50	43-48
Effective pH Range	0-14	0-14
Order of Selectivity for Ions	Monovalent: Ag>Cs>Rb>K>NH_4> Na>H>Li Divalent: Ba>Sr>Ca>Cu> Mg>Be	I>NO_3>Br>Cl> Acetate>OH>F
Capacity: Meq./ml. (Wet)	1.8	1.4
Meq./gm. (Dry)	4.8	3.7
Kgr. as $CaCO_3$/ft.^{3}	39.2	32.7
Thermal Stability, ^oC	150	50

Source: Form No. 176-1080-69, "General Properties of Standard DOWEX Ion Exchange Resins," The Dow Chemical Co., 1969.

a. The wet mixture, having a weight of 81.4 gms., was vacuum dried to a constant weight of 66.6 gms (for an 18% weight loss).

Applying the above ratio of 1.39:1.0, it was assumed that the individual weights of the dried components were 38.8 gms. 1X8 and 27.8 gms. 50WX8.

b. The dried mixture (66.6 gms.) was added to one quart of synthetic brackish water and agitated.

c. Conductivity readings were taken after 2 and 5 minutes of mixing.

d. Sulfate and chloride determinations were made of the treated water after filtering.

Surprisingly, the results reveal that the TDS level decreased drastically from 5400 ppm as $CaCO_3$ (untreated condition) to 410 ppm after only 2 minutes, and less than 100 ppm after 5 minutes. These values were considerably below the maximum goal of 3200 ppm,[a] indicating that the mixture was composed of a large excess of ion exchange materials.

The sulfate and chloride levels were reduced by 95% and 98%, respectively, to 50 ppm and 20 ppm.

In an effort to find the minimum material needed to desalt to an acceptable level of 3200 ppm, in the next test (#I-5), 10-gm. increments of the dried mixture were added to the solution until this TDS value

(a) It is assumed, for convenience, that the ion concentrations theoretically removed by the DOWEX resins are essentially the same as those removed by the Duolite resins and shown in Table XII (Appendix E).

was reached. Three increments were required for a total of 30 gms.; increments #2 and #3 were added at 13 minutes and 25 minutes. The final reading of 3200 ppm was obtained at 28 minutes (although stabilization had not as yet taken place).

In the final test of this series (#I-6), 30 gms. of the dried mixture were added simultaneously to a quart of brackish water. As expected, the conductivity immediately decreased, finally stabilizing at a reading equivalent to 2400 ppm TDS after 17 minutes.

As with the Duolite resins, additional work appears justified with this highly promising DOWEX resin combination to arrive at the minimum weight required for effective desalination.

D. Delivered Items

For delivery to LWL, in fulfillment of the contract, it was mutually agreed between Mr. Carney, the Project Officer, and AAI that the number of desalter packets eventually assembled and furnished would depend upon the status of funds remaining at the time of delivery. As to the contents of these packets, based on the limited development work conducted and reported upon, it was tentatively decided that each would contain ..0 grams of Ionac Chemical Co.'s lot SA-516 desalting chemical (that is, without barium hydrate) in loose form. Each packet was intended to desalt one quart of brackish water to the specified level (except in regard to sulfates) in accordance with a prescribed procedure.

The packaging material, as recommended by LWL, consisted of heat-sealed vapor barrier paper made up of two layers, 1/2 mil mylar on the outside and polyethylene on the inside. Arrangements were subsequently made with a supplier of this material, Continental Can Co. (through its local representative, Mr. A. W. Scholp) to obtain on a no-charge basis sufficient material (identified as C79 blend) to produce at least 400 packets. At this time, for planning purposes, it was estimated that the size of each packet would be approximately the same as that (2-9/16" x 3-3/8") of the water purification tablets already a part of the survival kit. A large roll of the material, 3-1/2 inches wide, silver in color and with green advertising matter preprinted thereon, was received on 9 August 1971 from the Mt. Vernon, Ohio plant of Continental Can.

For identifying the packet and conveying necessary instructions and precautions - as well as to mask out the preprinted advertising matter - a label was devised bearing this pertinent information. This label, initiated by AAI and approved by Mr. Carney, was printed on pressure-sensitive, adhesive-backed paper for affixing to both sides of the packet, as illustrated in Figure 6.

The final configuration of the packet, as stipulated by Mr. Carney, was a two-compartmented closure, folded over and heat-sealed on three sides, with another seal down the center to separate the compartments (Note Figure 6). Each compartment holds 4.5 gms. of chemicals, enough to nominally desalt one pint of brackish water, or one quart if both compartments are utilized. A set of labels is attached to the front and rear of each compartment. A notch is also provided on one edge of each compartment to facilitate the opening of the packet.

The present label, by the way, implies that the contents of each compartment will desalt one quart (rather than one pint). This confusing situation arose because the packet and label were both originally intended for use as one-quart units only. However, when the change was made to the 2-compartment packet - to provide greater flexibility and to make it easier to stow in the survival kit - the labels were already printed and, therefore, left as is rather than reprinted at additional expense.

At the end of the program, there were sufficient funds remaining to cover the preparation of 100 dual packets, enough units to desalt 100 quarts or 200 pints. Consequently, this number of packets was assembled,

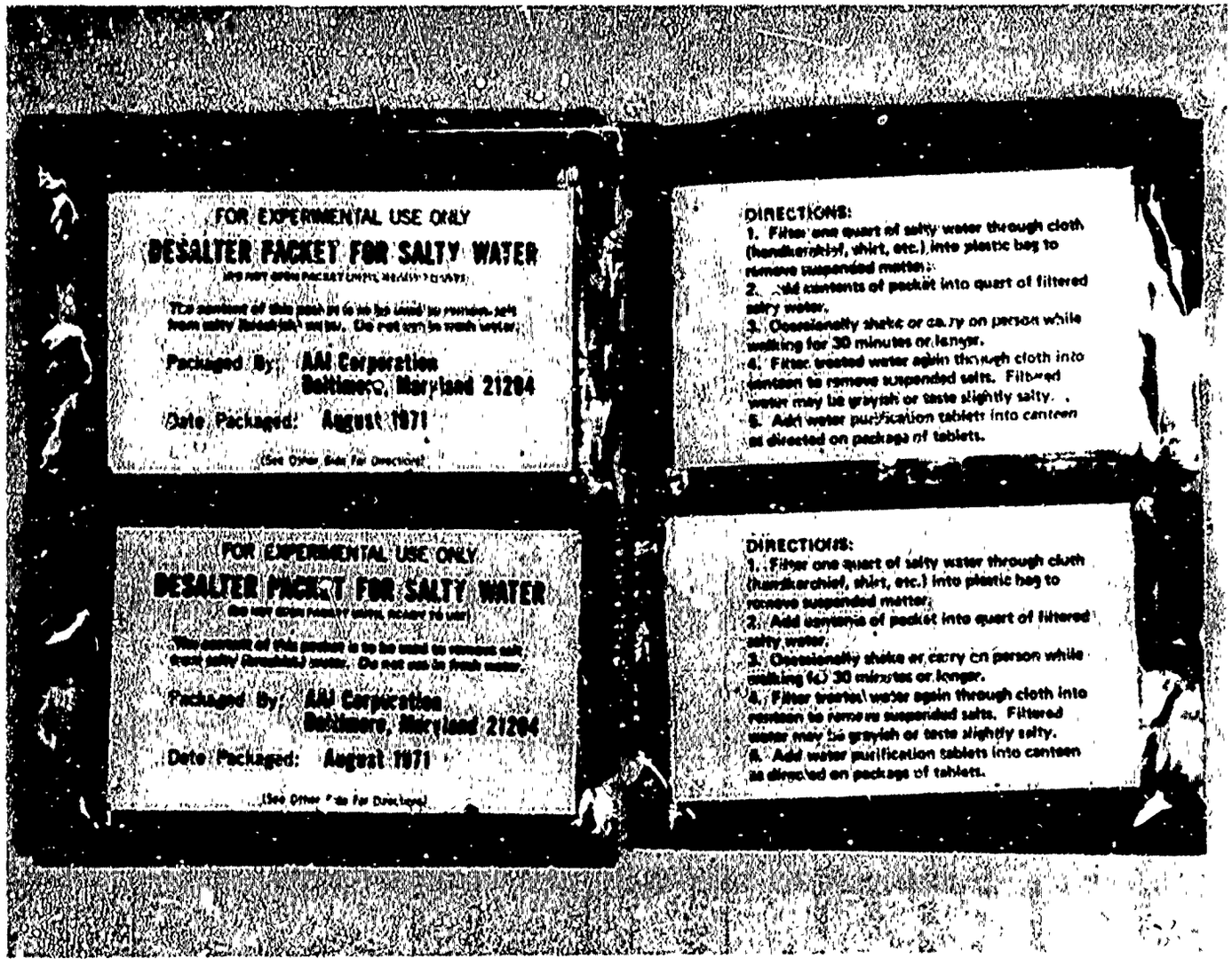


FIGURE 6

Labels Affixed to Both Sides of Desalter Packet

packaged, and delivered to LWL on 8 September 1971. Figure 7 depicts the packets arranged in their shipping carton ready for delivery.

It should be emphasized that the packets delivered at this time were intended to represent a concept only, not an optimized package. For example, a significant reduction in the volume of the packet could be realized by compressing (i.e., briquetting)[a] the desalting chemicals. With this in mind, a comparison follows of the weight and volume of 12 actual packets versus the limits specified in the Scope of Work:

	SPECIFICATION	ACTUAL	DIFFERENCE
Weight of 12 packets, ounces	3.0	5.6	+ 2.6
Volume of 12 packets, $in.^3$	3.0	20.5[b]	+17.5

Thus, it is seen that the specification has not been met and that additional R&D effort is necessary to enable the desalter packets to meet or exceed the prescribed weight and volume limitations.

(a) It is interesting to note that the volume occupied by Ionac's briquette is approximately 42% less than the same weight of material in powder form.

(b) This volume represents a stack of 12 packets measuring 2-9/16 in. wide x 3-3/8 in. long x 2-3/8 in. high.

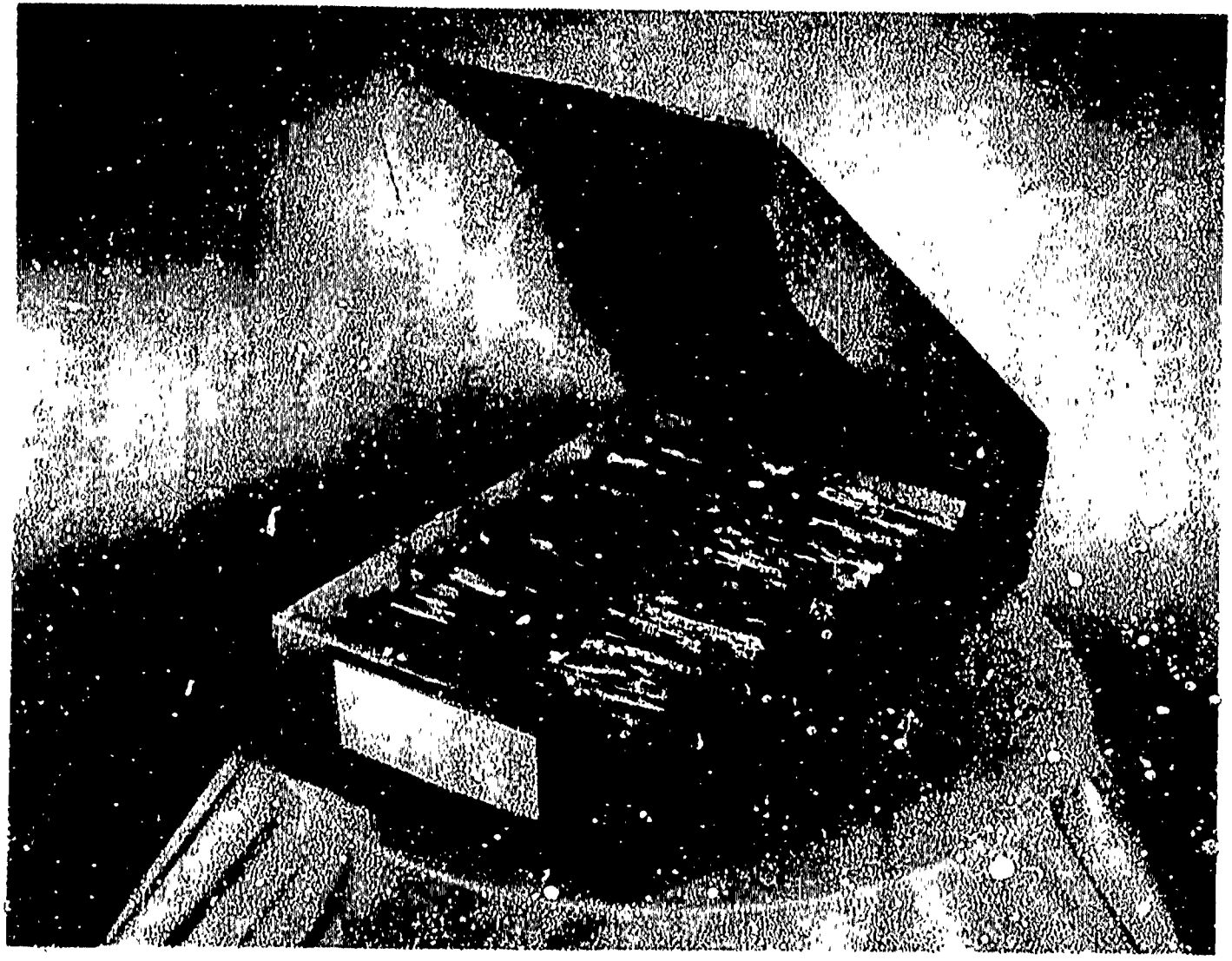


FIGURE 7

100 Dual Type Desalter Packets Arranged in Shipping Carton Ready for Delivery to IWL

III. SUMMARY OF TEST RESULTS

A. Test Data

1. Precipitation

See Table VII.

2. Ion Exchange

See Table VIII.

B. Discussion of Results

1. Precipitation

As a result of compiling and carefully analyzing the test data for this report, it became apparent, due to a shortcoming in our test procedure, that the present quantity of Ionac chemicals (9.0 grams) may be inadequate or, at best, marginal for desalting one quart of brackish water.

As pointed out earlier in the text (page 17), and further indicated by the appropriate data of Table VII, the conductivity <u>before</u> treatment never, in any test, reached the calculated target value of 7200 micromhos/cm.[a] (equivalent to 5700 ppm as $CaCO_3$). In fact, the average conductivity measured for 9 applicable tests was 5700 micromhos/cm. (with a range of 5,400 to 6,000), 21% less than calculated. Nevertheless, for desalting purposes, we considered the job done when the conductivity was reduced to at least 4300 micromhos/cm. (equivalent to 3900 ppm as $CaCO_3$).

---

[a] This figure represents the conductivity of the synthetic brackish water when all ingredients are fully ionized, as shown in Table III.

[illegible]
[illegible]
[illegible]

[illegible]	[illegible]	[illegible]	pH		[illegible]		[illegible]			[illegible]			[illegible]			[illegible]	[illegible]
			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

TABLE III

SUMMARY OF DESALTING TESTS
BY PRECIPITATION METHODS

TEST NO.	DESCRIPTION OF TEST	WT. OF CHEMICALS (GMS.)	pH		CONDUCTIVITY (MICROMHOS/CM.)		TOTAL DISSOLVED SOLIDS (PPM AS CALCIUM CARBONATE)			NITRATE ION CONCENTRATION (PPM AS NO_3)			CHLORI
			BEFORE	AFTER	BEFORE	AFTER	BEFORE	AFTER	DIFFERENCE	BEFORE	AFTER	DIFFERENCE	BEFORE
	TARGET VALUES →	~ 7	7.8	4.0-11.0	7200	4300	6700	3900	-2800	9[illegible]	309	-636	1515
P-1	Precipitation by use of silver oxide and barium hydrate in 1 pint of brackish water.	Silver oxide-2.35 (calc.) Barium Hydrate-0.55 (calc.)	Not recorded	10.7 when test stopped	6000	7750 (when test stopped)	5500	7200 (when test stopped)	+1700	not recorded		---	not
P-2	Ionac chemicals in 1 pint brackish water	4.5	7.7	8.1	5700	3900	5300	3500	-1800	9[illegible]	400	-[illegible]	1280
P-3	Ionac chemicals in 1 quart brackish water; to see if quantity from previous test could be scaled up.	9.0	7.8	8.7	6000	3800	5500	3450	-1850	11[illegible]	500	-600	1330
P-4	Ionac chemicals in 1 quart brackish water; repeat of test #P-3.	9.0	7.7	8.8	5600	3500	5200	3200	-2000	10[illegible]	425	-575	1200
P-5	Ionac chemicals in 1 quart brackish water; agitated for 30 min. in canteen.	9.0	7.8	8.6	5400	3450	5000	3150	-1850	not recorded		---	
P-6	Ionac chemicals packaged in tea-bag and placed in 1 quart brackish water.	9.0	7.8	8.2	6000	5300 @ 30 min. 4200 @ 17½ hours	5500	4900 @ 30 min 3800 @ 17½ hrs.	-600 @ 30 min. -1700 @ 17½ hrs.	9[illegible]	720	-225	1300
P-7	Ionac chemicals added to 1 quart of water sample supplied by LWL; contained mud and sediment; very odoriferous. Not brackish.	9.0	7.3	7.5	220	275	180	230	+ 50	not recorded		---	
P-8	Ionac chemicals in 1 quart brackish water; to check newly arrived lot SA-516 material (without barium hydrate).	9.0	7.9	8.8	5400	3250	5000	2900	-2100	97	825	-150	1390
P-9	Lot SA-516 Ionac chemicals in 1 pint brackish water; to check time required for desalting.	4.5	not recorded		5500	3100	5000	2750	-2250	not recorded		---	T
P-10	Same as test #P-9; supporting data.	4.5	not recorded		5500	2700	5000	2300	-2700	not recorded		---	
P-11	Special test in which extra NaCl was added to 1 quart brackish water to increase initial conductivity to 2700 microhos/cm.	9.0 → + 5.0 → 14.0	7.8	9.6	5600 7200	5200 4250	5200 6700	4800 3900	-2800	[illegible]	750	-200	2100 (before adding NaCl)

SUMMARY [illegible]
BY PREC[illegible]

[illegible]	[illegible] CONCENTRATION (PPM AS [illegible])			CHLORIDE ION CONCENTRATION (PPM AS [illegible])			APPROX. TIME TO FINAL TDS (MIN.)	REMARKS
[illegible]FERENCE	BEF[illegible]	[illegible]FTER	DIFFERENCE	BEFORE	AFTER	DIFFERENCE		
[illegible]0	6[illegible]	[illegible]	-636	1515	0	-1515	[illegible] 30	
[illegible]0	[illegible]	recorded	---	not	recorded	---	5-1/2 Hrs.	[illegible].5 gms. Ag_2O added initially, conductivity went up. add'l. Ag_2O added in .5 gm. increments and conductivity continued going up. Since test did not produce predicted result it was stopped before any barium hydrate was added.
[illegible]00	9[illegible]	[illegible]	-540	1280	790	-490	55	Total wt. of 4.5 gms. found by prorating briquet (normally 67 gms./pint) to go from 4150 to 2000 ppm as ion. briquet normally reduces from 35,000 to 2750.
[illegible]50	11	590	-600	1330	680	-650	35	Chemicals added in bulk at 1 time.
[illegible]00	1[illegible]	425	-575	1200	590	-610	30	
[illegible]50	[illegible]t	recorded	---	not	recorded	---	30	To see if shaking in canteen for 30 min. results in desired TDS level (3900 ppm max.).
[illegible] min. [illegible] hrs.	[illegible]	720	-225	1300	950	-350	17-1/2 Hrs.	Tea-bag configuration does not appear to provide enough intimate contact of chemicals to promote precipitation reaction.
[illegible]50	[illegible]t	recorded	---	not	recorded	---	[illegible]5	Sample contained little or no salt. Difficult to filter using various improvized methods (cloth, bandage); filtrate contained unreacted silver which gave it unappetizing appearance.
[illegible]00	97	825	-150	1390	460	-930	60	New material does not appear to react as fast as former Ionac material, as compared to tests #P-3 and P-4.
[illegible]50	not	recorded	---	not	recorded	---	35	
[illegible]00	[illegible]	recorded	---	not	recorded	---	3[illegible]	
[illegible]00	9[illegible]	750	-200	2100 (1300 before adding NaCl)	410	-1490	60 + [illegible] [illegible]	Necessary to add 1.5 gms. NaCl to increase conductivity to desired level of 7200 micromhos/cm; then added 5.0 gms. desalting chemical to original 9.0 gms. (for total of 14.0 gms.) to reduce conductivity to below 4300 micromhos/cm.

B

TABLE VIII

SUMMARY OF DESALTING TESTS BY ION EXCHANGE METHODS

TEST #	DESCRIPTION OF TEST	ION EXCHANGE RESINS					pH		CONDUCTIVITY (MICROMHOS/CM)		TOTAL DISSOLVED SOLIDS (PPM AS CALCIUM CARBONATE)			SOLIDS
		CATION	FINAL WT. (GMS.)	ANION	FINAL WT. (GMS.)	TOTAL WT. (GMS.)	BEFORE	AFTER	BEFORE	AFTER	BEFORE	AFTER	REMOVAL	REMOVAL
	TARGET VALUES →	--	--	--	--	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
I-1	Dualite resins in 1 pint brackish water, to determine initial resin quantities	C20X10	10.95	A7	6.50	17.95	7.7	7.0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
I-2	Dualite resins in 1 pint brackish water; repeat of test #I-1.	C20X10	7.7	A7	4.3	12.0	7.6	7.4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
I-3	Dualite resins in 1 quart brackish water, scaling-up volume of test #I-2.	C20X10	15.4	A7	9.6	24.0	7.7	7.0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
I-4	DOWEX resins in 1 quart brackish water.	50X8	27.0 (assumed)	1X8	18.0 (assumed)	44.0	7.0	6.6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
I-5	DOWEX resins in 1 quart water; repeat of test #I-4.	50X8	27.5 (assumed)	1X8	17.5 (assumed)	45.0	7.0	6.2	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
I-6	DOWEX resins in 1 quart brackish water	50X8	27.5 (assumed)	1X8	17.5 (assumed)	45.0	7.7	6.0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

[illegible]

[illegible] TESTS BY

[illegible]-25

[illegible]	[illegible] SOLIDS (TDS)			SULFATE ION CONCENTRATION (PPM AS ION)			CHLORIDE ION CONCENTRATION (PPM AS ION)			APPROX. TIME TO FINAL TDS (MIN.)	REMARKS
[illegible]	BEFORE	AFTER	DIFFERENCE	BEFORE	AFTER	DIFFERENCE	BEFORE	AFTER	DIFFERENCE		
[illegible]	[illegible]		-3500	465	0	-465	1515	989	-526	~ 30	
[illegible]			-2300	950	Not Recorded	--	1280	1200	-150	59	Resins (dry) added in increments at following times: Start - 5.05 gms. 23 min.-1.0 gms. 4[illegible] min.-3.0 gms. 14 min.- 1.0 gms. 25 min.-2.0 gms. 57 min.-2.0 gms. 19 min.- 1.0 gms. 42 min.-2.0 gms. TOTAL 17.05
[illegible]	[illegible]	[illegible]	-2400	1050	225	-825	1240	570	-690	55	Resins (dry) added all at one time; put in 12.0 gms., with intention of adding remainder (5.05 gms.) later, but 12.0 gms. produced desired TDS level.
		[illegible]	-2450	1000	175	-825	1200	720	-480	50	Resins (dry) added in bulk all at one time.
	[illegible]	[illegible]	-3510	1500	50	-950	1240	20	-1220	5	Resins supplied by mfr. already blended in container supposed to be used for 1 quart of brackish water; therefore, entire container was used at one time (after drying).
[illegible]		[illegible]	-2150	950	225	-725	1300	410	-890	28	Preblended resins added in increments at following times: Start - 10 gms. 25 min. - 10 gms. 13 min.-10 gms. TOTAL - 30 gms.
[illegible]	[illegible]	[illegible]	-2800	1000	100	-900	1200	590	-610	17	Resins (dry) added in bulk all at one time.

B

[illegible]

Test	Description of Test	[illegible]					pH		Conductivity		[illegible]			[illegible]			[illegible]			Effect, Time to Effect (in days)	Remarks
		Gallon	Final Wt. (kg.)	Added	Final Wt. (kg.)	Total Wt. (kg.)	Before	After	Before	After	Before	After	Difference	Before	After	Difference	Before	After	Difference		
	Target values [illegible]	--	--	--	--	--	7.0	6.0-16	1100	2000	6700	1230	+1100	965	0	+965	1515	955	+735	> 30	
1-1	Mobility testing on 1 part kerosene water to determine initial [illegible] construction	65.0410	10.95	65	9.50	19.65	7.7	7.0	1700	3150	5200	3650	+2100	950	NO Recovered	--	1204	1600	+1000	15	Mobility [illegible] 30 centimeters at following times [illegible]
1-2	Mobility testing on 1 part 50% kero water formed of test oil	22.7810	9.7	67	6.3	12.0	7.6	7.4	5600	3150	3230	2350	+1600	1250	211	+025	1200	170	+195	55	Mobility [illegible]
1-3	Mobility test of 1 part kero water, evaporation [illegible] of test oil	22.7810	13.6	67	8.0	24.0	7.7	7.0	1000	3000	5150	4700	+1650	1000	175	+925	1200	720	+500	56	Mobility [illegible]
1-4	Mobility testing on 1 part kerosene water	50.950	17.0 ([illegible])	100	10.0 ([illegible])	44.0	7.6	9.0	1500	50	5600	--	+1150	1000	50	+950	1160	30	+1130	5	Mobility [illegible]
1-5	Mobility testing on 1 part water, repeat of test [illegible]	50.950	12.5 ([illegible])	110	17.5 ([illegible])	30.0	7.0	7.2	1000	2000	5150	3.00	+2150	950	225	+725	1300	910	+390	30	Mobility testing [illegible]
1-6	Mobility testing on 1 part kerosene water	50.950	12.5 ([illegible])	140	11.5 ([illegible])	30.0	7.7	7.0	1600	1300	5100	2600	+2600	1300	500	+800	1200	595	+615	17	Mobility [illegible]

Thus, comparing this end point with the calculated and actual "before" values, the differences in conductivity after treatment - together with corresponding TDS values - becomes:

	Conductivity (micromhos/cm.)			TDS (ppm as $CaCO_3$)		
	Before	After	Difference	Before	After	Difference
Calculated	7200	4300	2900	6700	3900	2800
Actual	5700	4300	1400	5200	3900	1300

These figures indicate, therefore, that 9.0 grams of Ionac's precipitation chemicals should theoretically reduce the total dissolved solids by only 1300 ppm instead of 2800 ppm. Although this divergence appears serious, the situation is not quite so bad since the "after" conductivities of our earlier (4) tests (on which the selection of 9.0 grams was based) were all less than 4300 micromhos/cm., averaging 3700 micromhos/cm. (3300 ppm); this represents an average difference of 2000 micromhos/cm. (5700-3700) or 1900 ppm (5200-3300).

Then the question arises as to just how much total dissolved solids in terms of ppm as <u>ion</u> (<u>not</u> calcium carbonate) remain in the brackish water after treatment. Taking as an example test #P-4 - which gave the best results in terms of TDS reduction - and analyzing it to determine the TDS remaining, we come up with values of 1993 ppm or 2049 ppm (as ion), depending on which of two assumptions (Method A or Method B) is applied. A tabulation of the data considered in arriving at these figures is given in Table IX. Since the method used is rather complicated, a complete description will not

be given here. Suffice it to say, however, that the changes in $SO_4^{=}$ and Cl^- concentrations are based on actual measurements, while the order of Ca^{++} and Mg^{++} removal[a] is arbitrary. Apparently, little if any HCO_3^- and Na^+ ions are removed. Thus, it is seen that the TDS remaining in the brackish water treated with 9.0 grams of the old Ionac chemical (i.e., containing barium hydrate for sulfate removal) is marginal in regard to meeting the requirement as stated in the scope of work.[b] With the new Ionac chemical, which is not intended to remove $SO_4^{=}$, the TDS remaining, based on test #P-8, was 2040 ppm or 2082 ppm (as ion). The reason the figure was not higher, considering the virtual absence of $SO_4^{=}$ removal, was that this chemical is more effective in Cl^- removal (by about 52%). Table X presents a compilation of the data pertaining to test #P-8.

Now, as to what should have been done during testing; as soon as it was realized that the actual conductivity of a particular solution was not at the calculated level of 7,200 micromhos/cm., additional dissolved solids should have been added to the solution until this level was attained. This could most easily be done by adding sodium chloride.

In reconsidering our approach to the desalting problem and what additional quantity of chemical, if any, might be required, we ran a test (#P-11) involving the above procedure. The conductivity of the brackish

(a) According to the reactions for the Permutit Process (see page 18), the only other ions precipated out - in addition to $SO_4^{=}$ and Cl^- - are Ca^{++} and Mg^{++}.

(b) TDS must be reduced to 2,000 ppm or less.

TABLE IX

ANALYSIS OF PRECIPITATION TEST #P-4 TO DETERMINE TOTAL DISSOLVED SOLIDS REMAINING AFTER DESALTING

Ion	Original Amount (PPM)				Removed (PPM)				Remaining (PPM)			
	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$
SO_4	1000[a]	--	1040	--	575	--	598	--	425[a]	--	442	--
Cl	1200[a]	2200	1692	2732	610	1185	860	1458	590[a]	1015	832	1274
METHOD A[c]												
Ca	180	2380	448	3180	180	1365	448	1906	0	1015	0	1274
Mg	110	2490	451	3631	23	1388	94	2000	87	1102	357	1631
HCO_3	275	2765	226	3857	--	--	--	--	275	1377	226	1857
Na	616	3381	1343	5200[b]	--	--	--	--	616	1993	1343	3200[b]
METHOD B[d]												
Mg	110	2310	451	3183	110	1295	451	1909	0	1015	0	1274
Ca	180	2490	448	3631	37	1332	91	2000	143	1158	357	1631
HCO_3	275	2765	226	3857	--	--	--	--	275	1433	226	1857
Na	616	3381	1343	5200[b]	--	--	--	--	616	2049	1343	3200[b]

NOTES:

(a) These values actually determined by analytical tests.

(b) These values actually determined by conductivity measurements.

(c) Method A assumes that all Ca ions are removed prior to Mg ions.

(d) Method B assumes that all Mg ions are removed prior to Ca ions.

TABLE X

ANALYSIS OF PRECIPITATION TEST #P-8 TO DETERMINE TOTAL DISSOLVED SOLIDS REMAINING AFTER DESALTING

Ion	Original Amount (PPM)				Removed (PPM)				Remaining (PPM)			
	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$	As Ion	Cum. Ion.	As $CaCO_3$	Cum. $CaCO_3$
SO_4	975[a]	--	1014	--	150	--	156	--	825[a]	--	858	--
Cl	1390[a]	2365	1960	2974	930	1080	1312	1468	460[a]	1285	648	1506
METHOD A[c]												
Ca	180	2545	448	3422	180	1260	448	1916	0	1285	0	1506
Mg	110	2655	451	3873	45	1305	184	2100	65	1350	267	1773
HCO_3	275	2930	226	4099	--	--	--	--	275	1625	226	1999
Na	415	3345	901	5000[b]	--	--	--	--	415	2040	901	2900[b]
METHOD B[b]												
Mg	110	2475	451	3425	110	1190	451	1919	0	1285	0	1506
Ca	180	2655	448	3873	73	1263	181	2100	107	1392	267	1773
HCO_3	275	2930	226	4099	--	--	--	--	275	1667	226	1999
Na	415	3345	901	5000[b]	--	--	--	--	415	2082	901	2900[b]

NOTES:

(a) These values actually determined by analytical tests.

(b) These values actually determined by conductivity measurements.

(c) Method A assumes that all Ca ions are removed prior to Mg ions.

(d) Method B assumes that all Mg ions are removed prior to Ca ions.

water sample originally checked out at 5600 micromhos/cm.; consequently, additional NaCl was slowly added until a reading of 7200 micromhos/cm. was obtained. This required 1.5 gms. of sodium chloride, which when added to the 2.50 gms. already in the solution (see Appendix C) gave a total of 4.0 gms. Then, 9.0 grams of new Ionac material were added - at which point the conductivity dropped to 5200 micromhos/cm.: additional material was added until the conductivity stabilized at 4200-4300 micromhos/cm. This required 5.0 gms. of material for a total of 14.0 gms. (9.0+5.0). Total time required was 80 minutes.

This test, of course, leaves several questions unanswered which require additional investigation. For instance, what would be the increase in efficiency - in terms of both quantity and time - in adding the entire amount of material to the solution at one time? This should conceivably reduce the weight of chemical to something less than 14.0 gms. and the time to completion to somewhere between 30 and 80 minutes.

2. Ion Exchange

The same situation existed with the ion exchange study as previously described for the precipitation tests. Here again, the conductivity levels before treatment were considerably below the target value of 7,200 micromhos/cm.; in fact, they averaged 5700 to 5900. No attempt was made, however, to increase the conductivity with NaCl to see how much additional resin, if any, might be required to reduce it to 4300 micromhos/cm. This, perhaps, could be done at some future time. As a matter of interest, an

analysis was made of test #I-3 - using a technique similar to that used for tests #P-4 (Table IX) and P-8 (Table X) - to determine the TDS remaining after treatment. The results are shown in Table XI.

It will be seen here that the final TDS of 1584 ppm (as ion) was significantly below the maximum allowed value of 2000 ppm, which indicates that the ion exchange resins are very effective in removing dissolved solids. One of the factors contributing to this high degree of removal, however, was that the conductivity after treatment stabilized at 3000 micromhos/cm. rather than at the target value of 3600 micromhos/cm.

In actuality, our investigation with ion exchange resins, although highly promising, has been too limited to enable us to render final conclusions. However, the most encouraging factor in using the ion exchange method is that it avoids any problem of toxicity caused by the presence of silver or barium, as is the case with the Ionac precipitation chemicals. Any unreacted resin - such as due to low salt content - is easily removed by filtration together with the spent resins, leaving virtually no unappetizing looking residues.

The weights of the various resins resulting from our tests are by no means optimized. Further reactive studies are indicated in regard to the bulk addition of materials. Furthermore, improved drying should be capable of removing more moisture from the resins than we were able to accomplish. We managed to achieve up to 18% dryness, whereas the manufacturers claim that 25% is reasonable. (Although wet resins retain about 50% moisture, further drying beyond 25% may result in a decrease in ion exchange capacity.)

TABLE XI

ANALYSIS OF ION EXCHANGE TEST ØI-3 TO DETERMINE TOTAL DISSOLVED SOLIDS REMAINING AFTER DESALTING

Ion[a]	Original Amount (PPM)				Removed (PPM)				Remaining (PPM)			
	As Ion	Cum. Ion	As $CaCO_3$	Cum $CaCO_3$	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$	As Ion	Cum. Ion	As $CaCO_3$	Cum. $CaCO_3$
SO_4	1000[b]	--	1040	--	825	--	858	--	175[b]	--	182	--
Cl	1200[b]	2200	1692	2732	480	1305	677	1535	720[b]	895	1015	1197
Ca	180	2380	448	3180	180	1385	448	1983	0	895	0	1197
Mg	110	2490	451	3631	110	1595	451	2434	0	895	0	1197
Na	788	3278	1719	5350[c]	99	1694	216	2650	689	1584	1503	2700[c]

Notes:

(a) Ions arranged in order of affinity for ion exchange resins.

(b) These values actually determined by analytical tests.

(c) These values actually determined by conductivity measurements.

Although not mentioned heretofore, a definite odor is associated with these ion exchange reactions. It may best be described as a fishy smell, which is probably due to the presence of amines in the anion portions. The inclusion of activated carbon or other deodorizers may be necessary for this reason.

3. General

a. Filtration - Although filtration of the effluent liquid was not within the contractual scope of work, we do have a couple of observations to offer in this regard:

(1) Commonly available materials (e.g., shirt, handkerchief, bandage) are not effective as filters since they do not remove ash residue; for this reason, a special purpose filter should be provided in the desalter kit.

(2) The treated solution, after filtering, may retain an unnatural (or dirty) appearance; the user should be warned of this eventuality.

b. Turbidity - The turbidity of a sample is the reduction of transparency due to the presence of suspended particulate matter (such as clay, mud, algae, silica, etc.). The standard method for the determination of turbidity is based on the Jackson candle turbidimeter, which gives readings in terms of Jackson Turbidity Units[a] (JTU's). The U.S. Public Health Service

(a) According to literature issued by Hach Chemical Co., Ames, Iowa, manufacturers of turbidimeters, the unit of turbidity expression is to be changed from the Jackson Turbidity Unit (JTU) to the Formazin Turbidity Unit (FTU); the units are interchangeable.

standard on the turbidity of drinking water is 5 JTU's or less; LWL's scope of work calls for the brackish water to have a turbidity of 65 JTU's.

Actually, no investigation was conducted during this program on turbidity and its effect on the removal of dissolved solids by either the precipitation or ion exchange method. This was attributed to our inability to find a supplier of suitable equipment for this purpose until near the end of the program, at which time a lead time of 30 to 45 days and a price of $345 were quoted. Consequently, in view of the limited time and funds available, it was decided to omit this particular area of investigation at this time.

IV. CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

1. In accordance with the scope of work, an investigation was conducted into the desalting (i.e., removing a prescribed level of dissolved solids) of brackish water by means of precipitants and mixed-bed ion exchange resins.

2. Within the limits of our test procedures, it was found that the necessary level of desalination in one quart of brackish water (the brackish water, in this case, being a synthetic rather than natural solution) could be accomplished within reasonably short periods of time by approximately 9.0 grams of precipitants or 24-30 grams of ion exchange resins. These quantities, although questionable to some extent, nevertheless represent the magnitude of the materials required for this purpose.

3. The precipitants referred to in item 2 are marketed as a proprietary blend by the Ionac Chemical Co. They react in accordance with the patented Permutit Process[a] to remove, theoretically, chloride, sulfate, calcium, and magnesium ions.

4. The two pairs of mixed-bed ion exchange resins investigated were Diamond-Shamrock Chemical Co.'s Duolite C20X10 (H^+ form)/A7 (OH^- form) and Dow Chemical Co.'s DOWEX 50WX8 (H^+ form) / 1X8 (OH^- form). These are high capacity materials capable of removing sodium, calcium, magnesium, sulfate, chloride, and bicarbonate ions, all of which constitute the dissolved solids of the synthetic brackish water.

(a) For description, see page 18.

5. All of the above materials, although possessing certain inherent limitations, would be suitable choices for this application in view of their high reactivity, simplicity of use, and reasonably low bulk and weight.

6. In terms of overall consideration, however, it is felt that the ion exchange resins offer the better choice for the following reasons:

a. They are more highly effective in both sulfate and chloride ion removal.

b. They present no toxicity problem if left unreacted in the solution.

c. They are easily removed by filtering (with cloth) and leave no unsightly residue.

d. They are substantially less expensive.

Although their reactivity (i.e., TDS removed per unit weight) is less than that of the precipitants, it is believed that, through further research, this can be improved upon by better drying and by optimizing the cation/anion ratio of the resins as well as their final combined weight required to reach the desired level of desalination.

7. The tea-bag concept, at least as far as the precipitation method is concerned, is not a satisfactory means for achieving desalination since it does not permit the reactants to make the necessary intimate contact required to complete the reaction process.

8. The Ionac precipitation chemicals, which contain activated charcoal, demonstrated the deodorization of malodorous natural water to a reasonable degree; the ion exchange resins, on the other hand, generate their own distinctive odor, which should be removed for aesthetic reasons.

B. Recommendations

1. The development program conducted by AAI was, admittedly, a very limited one in view of the time and funds available. Considering the important nature of the product involved - drinking water - a much more exhaustive investigation is warranted to develop an operational desalter kit.

2. Based on the effort completed to date, we feel that the ion exchange concept represents the more favorable solution to the subject application; however, we recommend that further work be conducted on both systems - ion exchange and precipitation - before a final decision is rendered as to which method to incorporate in the desalter packet.

3. Specifically, we recommend that further investigation be conducted in - but not necessarily limited to - the following areas:

a. Precipitation Method

(1) Perform tests over complete calculated conductivity range (7200 micromhos/cm. reduced to 4300 micromhos/cm., calculated in terms of calcium carbonate) to optimize the weight of precipitants required; this, in all probability, will be a figure between 9.0 and 14.0 grams per quart. Use additional sodium chloride, if necessary, to increase the initial conductivity to the stated value (i.e., 7200 micromhos/cm.).

(2) Provide means for detecting concentrations of chloride and sulfate ions in water prior to treatment so as to prevent adding overdose of compounds containing potentially harmful silver and barium ions.

b. Ion Exchange Method

(1) Perform tests over complete calculated conductivity range (7200 micromhos/cm. reduced to 3600 micromhos/cm., calculated in terms of calcium carbonate) to optimize the weight of resins required. Use additional sodium chloride, if necessary, to increase the initial conductivity to the stated value (i.e., 7200 micromhos/cm.).

(2) Investigate means of deodorization, particularly in regard to those odors caused by the presence of amine groups in the resins.

(3) Investigate low pH values resulting from use of Duolite resins; this may require supplementary use of a buffering material to increase to a more desirable level.

(4) Optimize the ratio of cation to anion as well as the final weight of both ingredients required for the desalting process; this study should take into consideration such factors as bulk addition of materials, degree of dryness, grinding, etc.

c. General

(1) Devise adequate filtering method for field use to make desalted water appear reasonably attractive for drinking.

(2) Investigate taste of brackish water following desalting, treating with purification tablets, and final filtering; this study, for obvious reasons, should be performed only under close medical supervision.

(3) Investigate effects of turbidity (at level of 65 Jackson Turbidity Units) on the desalting process.

(4) Investigate means of providing optimized packaging of the desalter kit in terms of minimum weight and volume; this should encompass such areas as configuration, drying, compaction, package materials, sealing, etc.

(5) Although the use of synthetic brackish water is entirely acceptable for preliminary investigative purposes, a substantial number of tests should also be performed with natural brackish water as part of the final desalter selection process.

APPENDIX A

Directions for Model 1114

Direct-Reading Portable Conductivity and TDS Meter

General Remarks

The Model 1114 Direct-Reading Conductivity Meter is completely portable, and permits both Conductivity and TDS (Total Dissolved Solids) measurements to be made in the field. It is also ideal for use in the plant or lab, where grounded solutions in tanks or process streams could not otherwise be measured.

The instrument is housed in a small sturdy metal housing which also contains a re-chargeable nickel-cadmium battery. A foam-lined carrying case of chemical-resistant and water-repellant ABS plastic is available. The case can accommodate the instrument and conductivity probe, as well as a thermometer, and is ideal for field work, and to protect the instrument in the plant or lab.

The Model 1114 is rugged, yet sensitive. Give it the same care you would any fine instrument. Protect it from heavy shocks, excessive moisture, strong acids and alkalies, and from direct solar radiation, and it will give long, trouble-free service.

Operation of the Instrument

1. Check the mechanical movement of the meter. The meter needle should coincide with the line of the black number 3 and of the red 10 on the meter scale. If necessary, adjust to this position by turning the slotted screw at the bottom of the meter housing, while instrument is in the "off" position.

2. Before connecting the probe cable jacks to the two terminals marked "Cell", rotate the right-hand control pointer to the "Calibrate" position. (If probe cable is already connected, remove the jacks before this step.)

3. Then rotate the left-hand knob until the meter needle points to the green spot at the extreme right-hand end of the meter scale. The instrument is ready for use.

4. Connect the two jacks of the conductivity probe cable to the two terminals marked "Cell".

5. Immerse the conductivity probe into the solution to be measured, making certain that the solution covers the platinum measuring surfaces completely. The meter needle will rise above the black figure 10 on the scale.

6. Turn the range-control switch (right-hand pointer knob) clockwise until the meter needle comes to rest at a value in the right half of the meter scale. (This is where the full accuracy of instrument is best utilized.)

7. Multiply the meter reading by the range-control switch value to obtain the result in conductivity as micromhos. (The red figures refer to the red scale, and the black figures to the black scale.) Example, if the meter points to the line corresponding to a reading of 7 on the black scale, and the range-control switch points to 10^3, the answer is 7×10^3, or 7000 micromhos. A novel feature of the Model 1114 Conductivity Probe is that the cell constant has been adjusted to be exactly $k=1.00\ cm^{-1}$, so that no mathematical adjustment or correction has to be made to convert the measured conductance to specific conductivity, and readings are direct in terms of umho/cm.

   Note: Read section on "Care and Replatinizing of Probe", and take care never to let probe electrodes become dried out. Also, the platinum-black is very sensitive to certain substances (e.g. concentrated sulphuric acid, supersaturated solutions, suspensions etc.) In measuring such substances check the platinum-black frequently.

8. Check the calibration from time to time, but be sure to remove the cable jacks from the terminals during this step.

9. Turn the range-control switch to "Off" while not using the instrument, to conserve battery charge. NOTE -- The Conductivity Probe should be immersed in distilled water when not in use. During field use, use the rubber cap to keep the tip immersed in distilled water when measurements are not being made. (If distilled water is not available use as clean sample as is available, to fill the rubber cap, since this is better than allowing the platinum electrode to dry out). Naturally the cap must be removed during calibration or measurement.

10. Recharging the Battery -- After approximately 45 hours of continuous use, the rechargeable battery is exhausted. The green calibration check-point on the meter scale can then no longer be reached when calibrating. The battery should be recharged by means of the No. 1114C Charger, as follows:

11. Firmly press the miniature plug of the Charger into the recepticle on the front-edge of the Model 1114.

12. Turn the range-control switch to "Recharge".

13. Connect the Charger to a line voltage outlet (110 volts, 60 cycles AC).

14. Allow the Charger to operate for 14 hours (overnight charging is convenient).

15. Then turn the range-control switch to "Off", and remove the charger plug. The instrument is then again ready for measurement.

Temperature Effects -- If the conductivity measurements are made at a temperature different from 20° C, 68°F for highest accuracy the results should be corrected with a coefficient of between 1.5 to 2.3% per °C, depending on the substance being measured. A convenient average figure is 2% per °C. This is to be deducted for sample temperatures over 20°C. and added for temperatures below 20°C. Many users prefer to bring their sample temperatures to 20°C in a water bath, if possible, to make exact corrections unnecessary.

Care and Re-platinizing of Conductivity Probe electrode surfaces:

The Model 1114 Conductivity Probe has platinum electrode surfaces. These are covered with platinum-black, to avoid disturbing polarisation. It is important to keep the electrode surfaces under distilled water when they are not in use in order to keep the platinum-black active, and to keep the cell constant stable. If the surfaces should ever dry out completely the platinum-black may crumble away and the cell constant may become unstable. The probe should then be replatinized, by coating it with platinum-black as follows:

Coating the electrodes with platinum-black: If necessary the platinum-black layer may be renewed as follows:

1. First strip off any remaining platinum-black by dipping the electrode surfaces in hot nitro-hydrochloric acid (Aqua Regia).

2. Then dissolve 1 gm platinum chloride and 0.01 gm lead acetate in 30 ml of distilled water. Dip the electrode surfaces in this solution and, using an adjustable resistor of 1000 ohms, regulate the current of a 2 to 6 V battery (connect both electrodes) so that an even formation of gas develops (20 mA per square cm of electrode surface). (A special Conductivity Probe Platinizing Instrument is available, as is the platinizing solution -- write for details.) The electrodes are not consumed, because the platinum-black is supplied by the solution. The solution should be renewed from time to time. After having finished the procedure rinse the electrodes in distilled water and then purify them in water, which had been made conductive by adding one drop of diluted sulfuric acid. After 5 minutes reverse the current source and electrolyze the electrodes for an additional 5 minutes. Then rinse the electrodes in distilled water again, and keep the electrode surfaces under distilled water when not in use. If desired, the conductivity probe may be returned to the factory for replatinizing.

Determination of the cell constant k: The cell constant k of a cell can be checked, or determined by conductivity measurements of solutions which have an exactly determined conductivity.

For measuring cells with a k near 1.0, a 1/100 normal KCl-solution is particularly suitable as a calibration liquid. For cells with a considerable higher cell constant a 1/10 normal KCl-solution is also suitable. To prepare a 1/10 Normal KCl-solution, dissolve 7.46 gm dried analytically pure KCl in a 1000 ml volumetric flask and dilute to 1000 ml with distilled water. The 1/10 normal KCl-solution should always be prepared fresh from this basic solution by diluting it 1:10 with distilled water.

The table below shows the conductivity (c) of the solutions at different temperatures:

t°C	1/10 normal KCl-soln.	1/100 normal KCl-soln.
15	0.01048	0.001147
16	0.01072	0.001173
17	0.01095	0.001199
18	0.01119	0.001225
19	0.01143	0.001251
20	0.01167	0.001278
21	0.01191	0.001305
22	0.01215	0.001332
23	0.01239	0.001359

It should be taken into account that the conductivity of ordinary distilled water is approx. $1 \cdot 10^{-5}$ mhos/cm. This conductivity should be added to the values specified in the table in the event of using the 1/100 normal KCl-solution. To determine the cell constant k, dip the probe into the solution in question after having rinsed it thoroughly several times. After reading off the temperature, measure the conductivity of the solution. The cell constant k is calculated based on the table values of the conductivity being in accordance with the measuring temperature as follows: k = c/mho.

SPECIFIC CONDUCTANCE to TDS (TOTAL DISSOLVED SOLIDS)

CONVERSION TABLE

Specific Conductance (micro-mhos/cm)	Total Dissolved Solids mg/l $CaCO_3$	Specific Conductance (micro-mhos/cm)	Total Dissolved Solids mg/l $CaCO_3$
.100	.083	60.0	49.2
.200	.164	80.0	65.6
.300	.246	100.	82.0
.400	.328	120.	98.1
.500	.411	140.	115.
.600	.493	160.	132.
.700	.575	180.	148.
.800	.657	200.	165.
.900	.740	300.	251.
1.00	.821	400.	340.
1.20	.985	600.	515.
1.40	1.15	800.	686.
1.60	1.32	1,000.	865.
1.80	1.48	1,200.	1,035.
2.00	1.64	1,400.	1,210.
3.00	2.46	1,600.	1,388.
4.00	3.28	1,800.	1,565.
6.00	4.93	2,000.	1,740.
8.00	6.56	3,000.	2,670.
10.0	8.20	4,000.	3,640.
12.0	9.80	6,000.	5,540.
14.0	11.4	8,000.	7,440.
16.0	13.1	10,000.	9,480.
18.0	14.8	12,000.	11,580.
20.0	16.4	14,000.	13,580.
30.0	24.6	16,000.	15,700.
40.0	32.8	18,000.	17,800.

Conversion based on a temperature of 25°C (78°F). For absolute conversion at other temperatures, add 2.3% per °C for temperatures below 25°C, and subtract 2.3% per °C for temperatures above 25°C.

APPENDIX B

PROCEDURE MANUAL

DELTA SCIENTIFIC MODEL 50 PORTABLE LABORATORY

IMPORTANT

For the highest possible accuracy, each Model 50 Portable Laboratory is individually calibrated in the Delta Scientific calibration laboratory. The calibration charts in this Handbook are intended for the instrument bearing the same serial number.

PRINCIPLES of OPERATION -- Over the years, chemical tests have been developed which cause a color reaction to occur in water when a sample is treated with the reagent. These reagents are specific for the chemical determination being made. Such tests fall into two distinct categories:

COLORIMETRIC, in which the intensity of the color for a known amount of reagent, is directly related to the quantity of the substances present in a given volume of sample. A photometric device known as "photometer" or "colorimeter" often is used to measure the intensity.

TITRAMETRIC or VOLUMETRIC, in which the color change occurs abruptly as the reagent is slowly added to the sample. Since the reagent is a known concentration, measuring the amount of it necessary to cause the color change in a given volume of sample, gives a direct indication of the amount of the substance present in the sample.

COLORIMETRIC TESTS -- In the Model 50 Portable Laboratory, the tests for Bromine, Chlorine, Copper, Iron, Manganese, Nitrate, Nitrite, Phosphate and Polyphosphate are done colorimetrically, and the color produced by the chemical reactions are measured by a unique, simple and foolproof, battery-operated photoelectric color measuring instrument, called a photometer. No human color judgement is necessary for these tests. The photometer section consists of a long-life battery, a prefocused bulb, a holder for the test cells, and a sensitive and reproducible photocell. Plastic holders containing accurately designed permanent glass color filters are interposed in the light beam. Each photometric test gives instructions for exactly which is to be used. These glass color filters serve to accentuate the photometer's ability to measure minute color differences, which could not possibly be judged by the human eye.

In the Delta Scientific calibration laboratory, very accurate standard solutions are chemically treated by the exact test procedures contained in this manual. The meter readings obtained for each test are carefully plotted on the calibration graphs. Modern advances in electronics assure the continued stability of these calibrations. Since the photometer is thus pre-calibrated, the user can quickly and accurately perform a test merely by treating his samples as directed, and reading the test result directly from the individually prepared calibration chart.

To perform a test, a sample of the water to be analyzed is taken. One of the test cells is filled with a portion of this sample and the cell is inserted into the photometer. The light beam passes through this cell and causes the meter needle to move. The adjustment knob is turned until the meter reads "0".

A second portion of the sample is treated with reagent in accordance with the detailed instructions given for the specific test being made. The second cell filled with this treated sample is substituted for the first. The needle will move to a new setting directly related to the intensity of this color which in turn is directly related to the concentration of the substance being tested.

The meter reading is noted and comparison with the chart instantly and accurately shows the amount of the substance present in parts per million (or pH) without any necessity for computations or conversions.

Treat the Model 50 as you would any fine scientific instrument. With proper care it should give many years of accurate and reliable operation.

TITRAMETRIC or VOLUMETRIC TESTS -- The tests for acidity, alkalinity, chloride, calcium hardness, carbon dioxide, chromate, hardness, hydrogen sulfide, sulfate, and sulfite are volumetric or "drop count" tests. These are performed by adding one reagent to the specimen, and simply counting the number of drops of a second reagent required to cause a color change. The color change is sharp and easily seen. These tests are not performed on the photometer, but directly in a Plastic Sample Tube.

GENERAL REMARKS -- Test cells should be kept clean inside and out and the test procedure should be followed exactly for accurate test results. If the cells become badly soiled, soak in warm water containing soap or detergents, but then rinse very thoroughly with deionized or distilled water, to remove all traces of cleaning chemicals, before using. Wipe the outside of the cells before inserting into the photometer. DO NOT SUBSTITUTE REAGENTS OR CELLS OF ANOTHER MAKE OR TYPE.

Never place the scoops on an unclean surface such as soiled table top, etc. Instead, in order to avoid contaminating the reagent, always replace the scoops in their holders as soon as they have been used.

REAGENT STABILITY -- Cap all reagent bottles snugly after use. Like most chemicals, the reagents should be kept from extreme temperatures, and the case cover should be kept closed to protect reagents from extended exposure to strong sunlight, for maximum reagent life. While reagents properly stored and tightly capped can last for years, it is good practice to replace those over a year old. Prices on request.

PHOTOMETER INSTRUCTIONS -- Check that meter needle points to 50 when button switch is not pressed. If not, adjust to 50 by rotating the screw in the meter cover directly over the meter pivot.

Use only No. 50-T Test Cells. When performing a test, fill the cell at least to graduation mark. Cells must be absolutely clean, dry, and free of finger marks before being inserted.

The Model 50 lamp is used well below its rated voltage, and its replacement will be extremely infrequent. However, should the lamp need to be replaced it is available in its own prefocused holder, with directions, under article No. 50-L. Battery and lamp life will be greatly extended if the push button is depressed only while actually making a reading.

Do not operate photometer instrument with filter holder out of its slot. Replace the "C" battery when it is not possible to set meter to zero when the cell containing untreated sample is inserted in the instrument. When replacing battery observe the polarity markings on the battery and on the battery holder.

When photometer has not been used for considerable time, or unit has been in the cold, the meter needle will move very slowly when the button switch is first pushed down. However, merely continue to hold down until meter swings to desired position. Then, on further use, meter needle will respond more quickly.

ACIDITY

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test is designed to determine Mineral Acid in water occuring in mine drainage, waste effluents, streams, ponds, lakes, etc. It is determined by adding measured amounts of a standard alkali to a measured sample until an arbitrary pH is reached as indicated by a color change of an appropriate indicator.

1. Rinse the No. 8 Plastic Sample Tube with the water being tested and then fill exactly to the mark. (The curved surface or meniscus should be exactly even with the mark).
2. Add three drops of Reagent No. 4 (R-645 Total Alkalinity Indicator). If the sample is green it is alkaline; if red it is acid. If acid add Reagent No. 12 (R-723) Standard Alkali drop by drop (keep count of drops used) from the vertically held dropper bottle, mixing by swirling until the red color changes to green. An intermediate gray color may appear but continue addition of alkali to the first trace of green. Each drop required is equal to 10 parts per million (ppm) of mineral acid in terms of sulfuric acid.

ALKALINITY

Read General Instructions at beginning of this booklet before proceeding for the first time.

Preparation of sample -- Turbid samples should be filtered to remove scale, sludge, rust, etc., before making tests. The only time filtration is inadvisable is when very accurate results are desired in determining caustic alkalinity by titrating in the presence of phenolphthalein. In such cases filtration may cause changes in the caustic content. To make a filter fold a circle of filter paper exactly in half and fold again to quarters. Open the folded paper so that a 60 degree angle cone is formed with three thicknesses of paper on one side and a single thickness on the other. Adjust the paper to fit the funnel, moisten with the sample and press down tightly to the sides of the funnel. Fill with the sample, but not quite to the top. The water going through the filter can be collected in the No. 42 50 ml Erlenmeyer Flask, or in any clean bottle. Discard the first sample that flows through the funnel.

1. Rinse the No. 8 Plastic Sample Tube well with the clear sample and then fill it to the mark.
2. Add 2 drops of Reagent No. 3 (R-638 Phenolphthalein). If the sample has P alkalinity, as is usually the case with boiler waters, it will turn red. If the sample is a raw or natural water, it will usually remain colorless.
3. If the test sample is red, add Reagent No. 5 (R-687 Standard Sulfuric Acid) a drop at a time, mixing well after each drop. Keep track of all drops added and always hold inverted dropping bottle in a vertical position. If held at another angle the size of the drops will vary. Continue until the last trace of red color disappears and the test sample becomes colorless. Stop and record the number of drops of Sulfuric Acid required, as the P reading.
4. Add 3 drops of Reagent No. 4 (R-645 Total Alkalinity Indicator) and mix. The color will be green. Resume the addition of Reagent No. 5 (R-687 Standard Sulfuric Acid) drop by drop with mixing until the color changes to red. An intermediate gray color may appear but continue addition of acid until the first trace of red. Record the total number of drops of acid added to the test sample as the T reading. (Note that the T reading always includes the P reading). If no red color develops on the addition of Phenolphthalein, the Total Alkalinity Indicator is added at once and the T titration carried out as above.

It is customary to calculate alkalinity as calcium carbonate, even though none may be present, so as to have a uniform basis. In this test each drop of Reagent 5 (R-687 Standard Sulfuric Acid) is equivalent to 10 parts per million of calcium carbonate. It is preferable to express the results of the alkalinity determination in terms of P and T alkalinity. Results are sometimes calculated in terms of bicarbonate, carbonate and hydrate on the assumption that titration to the P endpoint is equivalent to the hydrate and one half the carbonate alkalinity, and that the titration to T is equivalent to the total alkalinity. Many factors such as the presence of phosphate, silica, organic and other buffers, however, affect this titration and the calculation of the form of alkalinity present may be in error. Under normal circumstances and in plant control, expression of results as P and T alkalinity is entirely satisfactory and is to be preferred from the standpoint of simplicity. The results of both titrations are frequently recorded as a fraction, P/T.

BROMINE

Read General Instructions at beginning of this booklet before proceeding for the first time.

1. Filter holder should be inserted all the way into slot, so that blue dot or legend "Cl" faces upward.
2. Fill two clean, dry No. 50-T Test Cells to the mark (10 ml) with sample to be tested.
3. Add 12 drops of Reagent No. 38 (R-30 Ortho-tolidine Reagent) to one of the test cells. Note the time.
4. Cap both cells and invert the one to which reagent was added, to mix the reagent and sample thoroughly.
5. Five minutes after step 3, insert the cell not containing reagent 1 to the photometer. Press red button switch, and turn knob to set meter to 0. Then promptly release button switch.
6. Remove cell and replace with cell containing sample to which reagent was added. Press button, and when meter needle comes to rest, read meter value. Obtain test result in parts per million BROMINE directly from graph. Do not touch knob adjustment in this step. To extend range, see Note A. To extend life of battery, release push button promptly, as soon as reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.

BROMINE in TURBID (Cloudy) or COLORED WATER

1. Filter holder should be inserted all the way into the slot, so that blue dot or legend "Cl" faces upward.
2. Fill two clean, dry No. 50-T Test Cells to the mark (10 ml) with sample to be tested. To one cell, which will be called the "blank", add 2 drops Reagent No. 48 (R-69 0.5% Sodium Arsenite), and 12 drops of Reagent No. 38 (R-30 Ortho-tolidine Reagent). Immediately add 12 drops Reagent No. 38 to the second cell, which will be called the "unknown". Note the time, quickly cap both cells and invert to mix contents thoroughly.
3. Fill a third Test Cell with clear and colorless distilled or tap water, and cap the cell. Insert this cell into the photometer. Press the button switch, and turn knob to set meter to 0. Then promptly release the button switch.
4. Five minutes after noting time in Step 2, insert "blank" cell, press button to obtain meter reading, and get corresponding "blank" result from Bromine calibration chart. Immediately replace cell with "unknown" cell, press button to obtain meter reading, and get corresponding "unknown" result from calibration chart. Do not change knob adjustment in this step. To obtain final test result in parts per million residual bromine, subtract "blank" result from "unknown" result. To extend range, see Note A.

   To extend life of battery, release push button promptly, as soon as reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.

Note A — To cover higher values, dilute the sample with zero-chlorine-demand water or with R-30 Ortho-tolidine Reagent, and multiply result by dilution factor. For example, if 10 ml sample is diluted to 50 ml, multiply the result by 50/10, or 5. However, use only 10 ml diluted sample in Step 2.

CALCIUM HARDNESS

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test is designed for quick determination of Calcium Hardness in water under field conditions. Reliable results as high as 200 ppm are attained particularly if the sample to be tested is first filtered to remove scale, sludge, iron rust, etc. Reagents and calibration of the No. 8 Plastic Sample Tube are arranged so that each drop of Hardness Reagent used to produce color change (end point) is equal to 10 ppm hardness in terms of calcium carbonate.

1. Rinse the No. 8 Plastic Sample Tube with the sample to be tested and fill it to the mark (bottom of the curved surface or meniscus should be level with the mark).
2. Add 10 drops of Reagent No. 17 (R-653 Calcium Buffer), mix and add 1 dipper (in cap) of Reagent No. 18 (R-654 Calcium Indicator), mixing gently to dissolve the powder.
3. If Calcium is present the sample will be fluorescent green -- if absent purple. If green add Reagent No. 19 (R-683 Hardness Reagent) drop by drop (keep count of drops used) mixing after each addition. Always hold dropper bottle in a vertical position. If held at another angle, size of drops will vary. When sample turns from green to purple, each drop of reagent required to effect the change represents 10 ppm of hardness (as calcium carbonate).

CARBON DIOXIDE

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test is designed to quickly determine the free carbon dioxide (CO_2) of water being used for drinking, in boiler feeds, cooling systems, swimming pool, and of water occurring in the natural state in streams, lakes, etc. The test is based on the addition of standard sodium carbonate to a sample of water in the presence of phenolphthalein. The carbon dioxide reacts with the sodium carbonate to form sodium bicarbonate which is colorless to phenolphthalein. As soon as an excess of sodium carbonate is present, a red color appears and this is taken as the endpoint.

1. Rinse the No. 8 Plastic Sample Tube with the water being tested and then fill exactly to the mark. (The curved surface or meniscus should be exactly even with the mark). If chlorine or bromine is present add 2 drops of Reagent No. 25 (R-697 Thiosulfate) and mix gently.
2. Add two drops of Reagent No. 3 (R-638 Phenolphthalein). If the sample turns red, no free carbon dioxide is present.
3. If the sample remains colorless, add Reagent No. 24 (R-717 Standard Sodium Carbonate) drop by drop (keeping count of drops), swirling the sample gently, until a definite pink color develops throughout the sample which remains permanent for 30 seconds. This is the endpoint. Each drop of Standard Sodium Carbonate with a 25 ml sample is equal to 5 parts per million of free carbon dioxide (CO_2). Free mineral acids, if present, will be measured by this test. Iron and aluminum salts will interfere. Some loss of carbon dioxide may occur, so the addition of Standard Sodium Carbonate should be completed as soon as possible. Mixing should be as gentle as possible, to avoid loss of carbon dioxide.

CHLORIDES

Read General Instructions at beginning of this booklet before proceeding for the first time.

Preparation of Sample — Turbid samples should be filtered to remove scale, sludge, rust, etc., before making the tests. To make a filter fold a circle of filter paper exactly in half and fold again to quarters. Open the folded paper so that a 60 degree angle cone is formed with three thicknesses of paper on one side and a single thickness on the other. Adjust the paper to fit the funnel, moisten with the sample and press down tightly to the sides of the funnel. Fill with the sample, but not quite to the top. The water going through the filter can be collected in the No. 42 50 ml Erlenmeyer Flask, or in any clean bottle. Discard the first sample that flows through the funnel.

1. Rinse the No. 8 Plastic Sample Tube with the clear filtered sample and fill it to the mark.
2. Add 2 drops of Reagent No. 3 (R-638 Phenolphthalein). If the sample is red, add Reagent No. 5 (R-687 Standard Sulfuric Acid) drop by drop until the test sample is colorless. If the test sample is colorless on adding Phenolphthalein, do not add any acid.
3. Place 5 drops of Reagent No. 1 (R-630 Chromate Indicator) in the measured sample and add Reagent No. 2 (R-706 Standard Silver Nitrate) drop by drop with thorough mixing until a permanent reddish color results. Keep track of all drops added and always hold inverted dropping bottle in a vertical position. If held at another angle the size of the drops will vary. Each drop of Standard Silver Nitrate solution is equal to 10 parts per million of chloride.

CHLORINE

Read General Instructions at beginning of this booklet before proceeding for the first time.

1. Filter holder should be inserted all the way into slot, so that blue dot or legend "Cl" faces upward.
2. Fill two clean, dry No. 50-T Test Cells to the mark (10 ml) with sample to be tested.
3. Add 12 drops of Reagent No. 38 (R-30 Ortho-tolidine Reagent) to one of the test cells. Note the time.
4. Cap both cells and invert the one to which reagent was added, to mix the reagent and sample thoroughly.
5. Five minutes after step 3, insert the cell not containing reagent into the photometer. Press red button switch, and turn knob to set meter to 0. Then promptly release button switch.
6. Remove cell and replace with cell containing sample to which reagent was added. Press button, and when meter needle comes to rest, read meter value. Obtain test result in parts per million CHLORINE directly from graph. Do not touch knob adjustment in this step. To extend range, see Note A. To extend life of battery, release push button promptly, as soon as reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.

CHLORINE in TURBID (Cloudy) or COLORED WATER

1. Filter holder should be inserted all the way into the slot, so that blue dot or legend "Cl" faces upward.
2. Fill two clean, dry No. 50-T Test Cells to the mark (10 ml) with sample to be tested. To one cell, which will be called the "blank", add 2 drops Reagent No. 48 (R-69 0.5% Sodium Arsenite), and 12 drops of Reagent No. 38 (R-30 Ortho-tolidine Reagent). Immediately add 12 drops Reagent No. 38 to the second cell, which will be called the "unknown". Note the time, quickly cap both cells and invert to mix contents thoroughly.
3. Fill a third Test Cell with clear and colorless distilled or tap water, and cap the cell. Insert this cell into the photometer. Press the button switch, and turn knob to set meter to 0. Then promptly release the button switch.
4. Five minutes after noting time in Step 2, insert "blank" cell, press button to obtain meter reading, and get corresponding "blank" result from Chlorine calibration chart. Immediately replace cell with "unknown" cell, press button to obtain meter reading, and get corresponding "unknown" result from calibration chart. Do not change knob adjustment in this step. To obtain final test result in parts per million residual chlorine, subtract "blank" result from "unknown" result. To extend range, see Note A.

   To extend life of battery, release push button promptly, as soon as reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.

Note A -- To cover higher values, dilute the sample with zero-chlorine-water, or with R-30 Ortho-tolidine Reagent, and multiply result by dilution factor. For example, if 10 ml sample is diluted to 50 ml, multiply the result by 50/10, or 5. However, use only 10 ml diluted sample in Step 2.

CHROMATE

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test is designed for the rapid determination of chromates in industrial cooling systems. It provides a guide to regulate the concentration of chromate when added to inhibit corrosion. The test procedure is based on the action of chromates on iodide in acid solution to liberate iodine which is then drop titrated with thiosulfate in the presence of starch indicator.

1. Rinse the No. 8 Plastic Sample Tube with the water being tested and then add the water until level with the mark.
2. Using measuring Spoon No. 16, add 2 level spoonfuls of Reagent No. 15 (R-781 Acid Sulfate) and mix by gentle swirling until crystals are dissolved.
3. Add 10 drops of Reagent No. 14 (R-772 Iodide Solution) and mix.
4. Add Reagent No. 13 (R-780 Standard Thiosulfate) drop by drop, mixing by gentle swirling after each drop until the sample has a pale yellow color. Keep track of all drops added and always hold the inverted dropping bottle in a vertical position. If held at another angle the size of the drop will vary.
5. Add 10 drops of Reagent No. 12 (R-636 Starch Solution) and mix. The sample should have a deep blue color.
6. Continue adding Reagent No. 13 (Standard Thiosulfate) until the blue color just disappears. Each drop of Standard Thiosulfate used is equal to 2 parts per million of chromate as CrO_4.

COPPER

Read General Instructions at beginning of this booklet before proceeding for the first time.

1. Filter holder should be inserted all the way into slot, so that blue dot faces upward.
2. Fill two clean No. 50-T Test Cells to the mark (10 ml) with sample to be tested.
3. To one of the cells, add 10 drops of Reagent No. 44 (R-175 Copper Reagent).
4. Cap both cells and invert the one to which reagent was added to mix the contents. Allow to stand 5 minutes.
5. Insert cell not containing reagent (blank) into the photometer. Press button switch, and turn knob to set meter to 0. Then promptly release switch. See Note A.
6. Remove cell and replace with cell containing treated sample. Press button, and when meter needle comes to rest, read meter value. Obtain test results in ppm COPPER (Cu) directly from graph. DO NOT touch knob adjustment in this step. To extend life of battery, release push button promptly, as soon as needle comes to rest and reading has been taken.
7. Range of test is 0-5 ppm Cu. To measure higher values, dilute the test sample with distilled or deionized water, and multiply result by dilution factor. For example, if 10 ml is diluted to 20 ml (before proceeding with Step 2), multiply the result by 20/10, or 2. However, use only 10 ml in Step 2. Range now becomes 0-10 ppm.

Note A -- If sample blank is so highly colored or turbid that the instrument cannot be set to 0, then use distilled water as a blank in Step 5 and read both the sample blank and the unknown. Convert the meter readings to ppm and subtract the value (in ppm) of the sample blank from the value (in ppm) of the unknown. The difference is the true concentration of unknown in the sample.

HARDNESS

Read General Instructions at beginning of this booklet before proceeding for the first time.

Preparation of Sample -- Turbid samples should be filtered to remove scale, sludge, rust, etc. before making tests. This is absolutely essential in the hardness test since even traces of suspended matter may introduce errors. To make a filter fold a circle of filter paper exactly in half and fold again to quarters. Open the folded paper so that a 60 degree angle cone is formed with three thicknesses of paper on one side and a single thickness on the other. Adjust the paper to fit the funnel, moisten with the sample and press down tightly to the sides of the funnel. Fill with the sample, but not quite to the top. The water going through the filter can be collected in the No. 42 50 ml Erlenmeyer Flask, or in any clean bottle. Discard the first sample that flows through the funnel.

1. Rinse the No. 8 Plastic Sample Tube well with the clear filtered sample and then fill it to the mark. The curved surface (meniscus) should be level with the mark.
2. Add 5 drops of Reagent No. 20 (R-619 Hardness Buffer) and a dipper (in cap) of Reagent No. 21 (R-620 Hardness Indicator Powder). Mix gently to dissolve the indicator.
3. If hardness is present, the sample will be red. If absent, the sample will be blue. If red, add Reagent No. 19 (R-687 Hardness Reagent) a drop at a time, mixing well after each drop. Continue until the sample has no red color but is entirely blue. Keep track of all drops added and always hold inverted dropping bottle in a vertical position. If held at another angle the size of the drops will vary. Each drop is equal to 10 parts per million of hardness, as $CaCO_3$.

HYDROGEN SULFIDE

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test is designed for quick field determinations of Hydrogen Sulfide in water. Waters containing hydrogen sulfide (sulfur waters) are distinguished by their offensive rotten-egg odor and their marked corrosiveness. Concentrations may vary from zero to 70 parts per million or above but usually is under 10. The procedure is based on the reducing action of hydrogen sulfide on a standard iodine solution. An excess of iodine solution is added and the excess determined by drop titration with a standard solution of thiosulfate. The iodine consumed is a measure of the hydrogen sulfide. Thiosulfate, sulfite, some organic materials and other reducing substances will give erroneously high results because they also react with iodine.

1. To determine hydrogen sulfide accurately the sample should be freshly obtained with as little contact with the air as possible. Loss of hydrogen sulfide gas or oxidation may lower the content. If possible a stream of the water being tested should be siphoned or run into the No. 8 Plastic Sample Tube to overflow for a minute or so. The excess sample above the mark should be quickly discarded until the curved surface or meniscus of the water is exactly even with the mark on the tube.
2. Add 3 drops Reagent No. 45 (R-121 Sulfuric Acid, 3.6N), mix gently, and proceed at once to Step 3.
3. Add Reagent No. 9 (Sulfide Reagent C (iodide-iodate)), drop by drop, mixing gently after each drop until the sample has a marked brown color. Always hold the dropper bottle in a vertical position and keep accurate count of the drops added. If held at another angle the size of drop will vary.
4. Add Reagent No. 10 (Sulfide Reagent D (thiosulfate)), gently mixing after each drop until the brown color fades to a faint straw color.
5. Add 5 drops Reagent No. 11 (R-636 Starch Solution) to give a deep blue color.
6. Continue careful addition of Reagent No. 10 (Sulfide Reagent D (thiosulfate)) until the blue color just disappears.
7. The number of drops of Sulfide Reagent C minus the number of drops of Sulfide Reagent D times 2 will equal the Hydrogen Sulfide concentration in parts per million. For example if 10 drops of C was added and it took 8 drops of D to destroy the blue color the Hydrogen Sulfide concentration will be 10-8 = 2X2 = 4 parts per million.

IRON

Read General Instructions at beginning of this booklet before proceeding for the first time.

1. Filter holder should be inserted all the way into slot, so that blue dot faces upward.
2. Fill two clean, dry No. 50-T Test Cells to the mark (10 ml) with sample to be tested.
3. To one of the cells, using Scoop No. 33, add 1 scoop of Reagent No. 32 (R-63 Iron Reagent Powder). Close cap on bottle promptly to prevent powder from picking up moisture. Bottle contains capsule of dessicant to absorb moisture.
4. Cap both cells and invert the one to which reagent was added, to mix the reagent and sample thoroughly.
5. Insert cell not containing reagent into photometer. Press button switch, and turn knob to set meter to 0. Then promptly release button switch.
6. Remove cell and replace with cell containing treated sample. Press button, and when meter needle comes to rest, read meter value. Obtain test results in p.p.m. Iron directly from graph. DO NOT touch knob adjustment in this step. To extend life of battery, release push button promptly, as soon as needle comes to rest and reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.
7. To measure higher values, dilute the test sample with distilled or deionized water, and multiply result by dilution factor. For example, if 10 ml sample is diluted to 50 ml, multiply the result by 50/10, or 5.

IMPORTANT: Always replace the cap promptly and tightly on the bottle containing Reagent No. 32 (R-63 Iron Reagent Powder), to prevent the powder from picking up moisture from the air.

MANGANESE

Read General Instructions at beginning of this booklet before proceeding for the first time.

The rapid dry powder method offers the advantage of a field analysis without the need of heat, or it can be used for a quick laboratory analysis in the absence of interference.

1. Fill a No. 50-T Test Cell to the mark (10 ml) with sample.
2. Using the No. 33 Scoop (0.2 gms.), add 1 scoopful of Reagent No. 34 (R-74 Manganese Buffer Powder), cap the cell, and mix until dissolved.
3. Using the No. 37 Scoop (0.1 gm.), add 1 scoopful of Reagent No. 36 (R-73 Sodium meta Periodate), cap the cell, and mix until dissolved. Let stand one minute for color development. IMPORTANT: Do not delay testing the treated sample longer than 4 minutes after full color development.
4. Filter holder should be inserted all the way into slot of instrument, so that 2 green dots face upward.
5. Fill a clean No. 50-T Test Cell to the mark with untreated water sample for use as a blank. Cap the cell.
6. Insert the cell containing the "blank" into the photometer. Press button switch and turn knob to set meter to 0. Then promptly release switch.
7. Remove cell and replace with cell containing treated sample. Press button, and when meter needle comes to rest, read meter value. Obtain test results in ppm MANGANESE directly from graph. Do NOT touch knob adjustment in this step. To extend life of battery, release push button promptly, as soon as needle comes to rest and reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.
8. To measure higher values, dilute the test sample with distilled or deionized water, and multiply result by dilution factor. For example, if 10 ml sample is first diluted to 20 ml, multiply result by 20/10, or 2. (However, do not forget to use only 10 ml in performing the test).

NITRATE

Read General Instructions at beginning of this booklet before proceeding for the first time.

Procedure A -- Rapid dry powder method for Nitrate determinations in the absence of Nitrite interference. This method offers simplicity. See Note A.

1. Filter holder should be inserted all the way into slot, so that 1 Green dot faces upward.
2. Fill a clean No. 50-T Test Cell to the mark (10 ml) with sample to be tested.
3. Using the No. 33 Scoop (0.2 gms) add 1 level scoopful of Reagent No. 46. Cap the cell securely, note the time, and shake the cell vigorously for 30 seconds. IMPORTANT: Always replace the cap promptly and tightly on the bottle containing Reagent No. 46 to prevent the powder from picking up moisture from the air. Bottle contains capsule of dessicant to absorb moisture. Keep measuring scoop clean and dry. Let stand for 5 minutes.
4. Prepare a blank by adding 10 ml of distilled water to a second No. 50-T Test Cell.
5. Mix the treated sample once again by inverting and let stand for 1 minute.
6. Insert the cell containing the "blank" into the photometer. Press button switch and turn knob to set meter to 0. Then promptly release switch. Immediately remove cell, and replace with cell containing treated sample. Press button and when meter needle comes to rest, read meter value. Obtain test results in ppm Nitrogen directly from the Nitrate Nitrogen graph. Do NOT touch knob adjustment in this step. To extend life of the battery, release push button promptly, as soon as needle comes to rest and reading has been taken. Clean cells and caps thoroughly after use. Range of test is 0-1.4 ppm Nitrogen (N). To measure higher values, dilute the test sample with distilled or deionized water, and multiply results by dilution factor. For example, if 10 ml is diluted to 20 ml and 10 ml of diluted sample is used in the test, multiply result by 20/10, or 2. Range of test is now 0-2.8 ppm N. To express results in terms of Nitrate (NO_3) multiply the results by 4.43.

Procedure B -- For tests in the presence of Nitrite interference.

1. Filter holder should be inserted all the way into the slot so that 1 Green dot faces upward.
2. Fill a No. 50-T Test Cell to the mark (10 ml) with sample to be tested.
3. Add 2 drops of Reagent No. 54 (Chlorine Solution) see Note C, and 3 drops of Reagent No. 5. (Standard Sulfuric Acid). Shake 30 seconds, let stand for 2 minutes.
4. Add 1 drop of Reagent No. 48 (Sodium Arsenite) mix for 30 seconds.
5. Using the No. 33 Scoop (0.2 gms) add 1 level scoopful of Reagent No. 46. Cap the cell securely, shake vigorously for 30 seconds. Note the time and let stand for 5 minutes.
6. Prepare a blank by adding 10 ml of distilled water to a second No. 50-T Test Cell.
7. Mix the treated sample once again by inverting and let stand for 1 minute.
8. Insert the cell containing the "blank" into the photometer. Press button switch and turn knob to set meter to 0. Then promptly release switch.

Immediately remove cell and replace with cell containing treated sample. Press button and when meter needle comes to rest, read meter value. Obtain test results in ppm Nitrogen directly from the Nitrate Nitrogen graph. This value represents combined Nitrate/Nitrite Nitrogen. To determine the Nitrate Nitrogen value, subtract the value of the Nitrite test (in ppm) as determined in Note A from the value of the Nitrate/Nitrite determination in ppm. This represents the Nitrate Nitrogen value. To express results as ppm Nitrate, multiply the result by 4.4.

Note A -- If Nitrites are known to be absent then follow procedure A. If Nitrites are suspected, a test for Nitrite should be performed following the procedure for Nitrite. If the determination indicates Nitrites are present, record the results of the Nitrite test and follow procedure B for the Nitrate determination.

Note B -- If the sample is colored or turbid, fill a No. 50-T Test Cell to the 10 ml mark with sample, and read this untreated sample against the distilled water blank. Convert the reading to equivalent ppm Nitrate Nitrogen and subtract this value from the results of the Nitrate Nitrogen test. This represents the true Nitrate Nitrogen value.

Note C -- Since Reagent No. 54 (Chlorine Solution) has limited stability (3-6 months) we recommend that the following procedure be used in order to prepare it:

Dilute 3 ml of Chlorine Bleach (for instance Clorox) with water to 100 ml. Mix and store in plastic bottle provided.

NITRITE

Read General Instructions at beginning of this booklet before proceeding for the first time.

1. Filter holder should be inserted all the way into slot, so that 1 Green dot faces upward.
2. Fill a clean, dry No. 50-T Test Cell to the mark (10 ml) with sample to be tested.
3. Using the No. 33 Scoop (0.2 gms.) add 1 level scoopful of Reagent No. 47. Cap the cell securely and shake to dissolve. Note the time and let stand for 5 minutes for color development. Always replace the cap promptly and tightly on the bottle containing Reagent No. 47 to prevent the powder from picking up moisture from the air. Bottle contains capsule of dessicant to absorb moisture. Keep measuring scoop clean and dry.
4. Prepare a blank by adding 10 ml of distilled water to a second No. 50-T Test Cell.
5. Insert the cell containing the "blank" into the photometer. Press button switch and turn knob to set meter to 0. Then promptly release switch.
6. Immediately remove cell, and replace with cell containing treated sample. Press button and when meter needle comes to rest, read meter value. Obtain test results in ppm Nitrogen directly from the Nitrite Nitrogen graph. Do NOT touch knob adjustment in this step. To extend life of the battery, release push button promptly, as soon as needle comes to rest and reading has been taken. Clean cells and caps thoroughly after use.
7. Range of test is 0-0.2 ppm Nitrogen (N). To measure higher values, dilute the test sample with distilled or deionized water, and multiply results by dilution factor. For example, if 10 ml is diluted to 20 ml and 10 ml of diluted sample is used in the test, multiply result by 20/10 or 2. Range of test is now 0-0.4 ppm N. To express results in terms of Nitrite (NO_2) multiply the results by 3.29.

Note A -- If the sample is colored or turbid, fill a No. 50-T Test Cell to the 10 ml mark with sample, and read this untreated sample against the distilled water blank. Convert the reading to equivalent ppm Nitrite Nitrogen, and subtract this value from the results of the Nitrite Nitrogen test. This represents the true Nitrite Nitrogen value.

pH

A unique dispenser with Universal pH Indicator Paper is supplied with the Model 50 Portable Laboratory. This covers the range 1 to 12 pH, and has been found most convenient for rapid screening tests in the field.

1. Open the dispenser (No. 31) by turning the upper part in a counter clockwise direction.
2. Using dry fingers, pull out the needed length of strip (usually about 1" will do), close the dispenser by turning clockwise, and tear off the exposed strip.
3. Dip the strip into the test liquid and compare it with the color scale while still wet. When working with liquids containing suspended matter, or colored liquids, place a drop of the liquid on one side of the strip, allow the sample to wet the paper thoroughly, and then turn the strip over and compare the reverse side of the paper to the color chart. This method can also be used to test the pH of sludge, moist soil samples, etc.
4. When measuring weakly buffered or unbuffered solutions, place the indicator strip against the inside wall of a test tube, and fill with test solution. After 60 seconds, compare the color with the color scale through the glass.

PHOSPHATE and POLYPHOSPHATE

Read General Instructions at the beginning of this booklet before proceeding for the first time.

Procedure A -- ORTHO-PHOSPHATE

1. Fill a No. 50-T Test Cell to the mark (10 ml) with filtered sample.
2. Simultaneously prepare a reagent blank by adding 10 ml of phosphate-free or distilled water to a second No. 50-T Test Cell.
3. Add 10 drops of Reagent No. 41 (R-143 Sulfuric Acid 15%)to each and mix. Hold bottle vertically, with tip facing down, when counting drops.
4. Add 6 drops of Reagent No. 39 (R-80 Ammonium Molybdate, 2.5%) to each and mix.
5. Add 1 drop of Reagent No. 40 (R-79 Stannous Chloride) to each and mix.
6. Allow to stand 15 minutes for full color development.
7. Filter holder should be inserted all the way into slot of instrument so that yellow dot faces upward.
8. Insert the cell containing the "blank" into the photometer. Press button switch and turn knob to set meter to 0. Then promptly release switch.
9. Remove cell and replace with cell containing treated sample. Press button and when meter needle comes to rest, read meter value. Obtain test results in ppm PHOSPHATE directly from graph. Do NOT touch knob adjustment in this step. To extend the life of the battery, release push button promptly, as soon as needle comes to rest and reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.
10. Range of test is 0-5 ppm PO_4. To measure higher values, dilute the test sample with distilled or deionized water, and multiply result by dilution factor. For example, if 10 ml is diluted to 20 ml, multiply result by 20/10, or 2. However, do not forget to use only 10 ml in the test.

Procedure B -- TOTAL PHOSPHATE

1. Measure 10 ml of filtered sample, using a No. 50-T Test Cell, into the No. 42, 50 ml Erlenmeyer Flask.
2. Add 10 drops of Reagent No. 41 (R-143 Sulfuric Acid, 15%) and mix.
3. Allow acidified sample to boil gently for 90 minutes adding distilled water to keep the volume between 7 and 10 ml. In the field, an inexpensive "Sterno-Type" Stove can be used. See Note A.
4. Cool sample and transfer to a No. 50-T Test Cell. Dilute with distilled water to the 10 ml mark.
5. Simultaneously prepare a reagent blank by adding 10 ml of phosphate-free or distilled water to the mark of a No. 50-T Test Cell and add 10 drops of Reagent No. 41 (R-143 Sulfuric Acid).
6. Add to each, while mixing, 6 drops of Reagent No. 39 (R-80 Ammonium Molybdate) and 1 drop of Reagent No. 40 (R-79 Stannous Chloride Reagent). Let stand for 15 minutes for full color development.
7. Continue as directed in procedure for Ortho-phosphate, beginning with Step 7.

Procedure C -- POLYPHOSPHATE

To determine Polyphosphate, subtract the result obtained in Procedure A (Ortho-Phosphate), from the result obtained in Procedure B (Total Phosphate). The difference represents Polyphosphate, expressed in terms of Phosphate (PO_4).

PHOSPHATE and POLYPHOSPHATE, Con't.

Note A -- Some samples may require less boiling time. This can easily be determined by running duplicate samples, using a 90 minute and a 30 minute boiling cycle and comparing the results on the colorimeter. If readings are the same, then the 30 minute boiling time may be employed for samples of similar composition.

Note B -- The Stannous Chloride method has been adopted because of its sensitivity in the lower Phosphate ranges. The minimum detectable concentration is approximately 0.02 mg/l of PO_4. To avoid contamination from interfering substances, it is recommended that all glassware used in this test be thoroughly cleaned with No. R-95 Acid Dichromatic Solution and rinsed several times with demineralized distilled water. Do not use soap, detergents or glassware cleaners, as they may leave a film of phosphate on the glass, which would cause errors.

SULFATE

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test is designed for the determination of sulfate in water under field conditions. Sulfate is present in all natural waters but the content will vary considerably in different localities depending on the mineral content of the soil. The United States Public Health Service recommends that drinking water should contain less than 250 ppm. The principal objection to sulfate in industrial water is that it combines with calcium to form calcium sulfate scale.

IMPORTANT: This test uses the special No. 30 Plastic Sample Tube, which is graduated at 10 ml and 20 ml. Do not use the No. 8 Plastic Sample Tube (25 ml) used for all other tests.

The procedure on waters which do not contain phosphate is as follows:

1. Rinse the special No. 30 Plastic Sample Tube with the water being tested and fill to the lower mark (10 ml).
2. Add a drop of Reagent No. 3 (R-638 Phenolphthalein). If the sample turns red add Reagent No. 26 (R-760 Hydrochloric Acid solution) drop by drop until the red color disappears. If the sample is colorless when Phenolphthalein is added do not add acid.
3. Add a small dipper (in cap) of Reagent No. 28 (R-761 Sulfate Indicator Powder) to the above sample. When the indicator has dissolved, add Reagent No. 29 (R-762 Isopropyl Alcohol) to the second (top) mark on the No. 30 Plastic Sample Tube and mix by gentle swirling.
4. Add Reagent No. 49 (R-759 Standard Barium Chloride Solution) drop by drop with mixing until the color changes from yellow to a dull red or orange. Keep count of drops added. Always hold the dropper bottle in a vertical position. If held at another angle the size of drops may vary. Each drop required equal 25 parts per million of Sulfate as SO_4.

When phosphates are present as in some boiler waters, the sample should be acidified to bromcresol green, by adding three drops of Reagent No. 27 (R-1003E Bromcresol Green Indicator Solution) to the original 10 ml sample, and adding drops of Reagent No. 26 (R-760 Hydrochloric Acid) until the color changes from blue to yellow. Then add Reagent No. 49 (R-759 Standard Barium Chloride Solution) drop by drop as above, and calculate the sulfate (SO_4) content in the same way.

Samples with high sulfate content can be diluted with sulfate-free (distilled or deionized) water and the results multiplied by the dilution factor. For example, if 10 ml is diluted to 20 ml, multiply by 20/10, or 2. Always thoroughly clean the Plastic Sample Tube after each test.

SULFITE

Read General Instructions at beginning of this booklet before proceeding for the first time.

This test kit is designed for quick determination of sodium sulfite in boiler water. To combat corrosion, dissolved oxygen must be at the lowest possible concentration. One practice is to remove as much as possible from the feed water with a deaerating heater and maintain residual sodium sulfite in the boiler water to remove any oxygen which passes the heater. Under proper conditions sulfite combines with oxygen to form sulfate. The test uses a dual purpose dry starch indicator powder which contains both acid and starch in dry form. The procedure avoids the use of strong mineral acids, deteriorating starch solutions and eliminates a step in the procedure. The acid-starch indicator powder is extremely stable.

The procedure is based on the fact that when iodide-iodate is added to an acid solution, iodine is liberated [illegible] immediately reacts with any sulfite that is present. As soon as all [illegible] sulfite has reacted with this freed iodine, any excess iodine forms a blue color with the starch indicator. Each drop of Reagent No. 22 (R-617 Iodide-Iodate) equals 2 ppm sodium sulfite. To determine sulfite accurately the sample should be freshly obtained with as little contact with the air as possible. The sample should never be filtered since exposure to the air will use up sulfite. It should always be cooled to room temperature before making the test.

1. Carefully rinse the No. 8 Plastic Sample Tube with the water to be tested and then add the water until the bottom of the curved surface or meniscus of the liquid is exactly even with the mark on the tube.
2. Add 1 drop of Reagent No. 3 (R-638 Phenolphthalein) and mix gently. Boiler water should turn red.
3. Add 1, 2, or more dipperfuls of Reagent No. 23 (R-725 Acid-Starch Indicator Powder) to the sample with gentle mixing until the red color disappears.
4. Add Reagent No. 22 (R-617 Regular Iodide-Iodate Solution) drop by drop (keep count of drops used) with gentle mixing until a faint but permanent blue color appears. Always hold the dropper bottle in a vertical position. If held at another angle, the size of drops will vary. Each drop of Regular Iodide-Iodate Reagent required to give the permanent blue color is equal to 2 ppm sodium sulfite (Na_2SO_3).

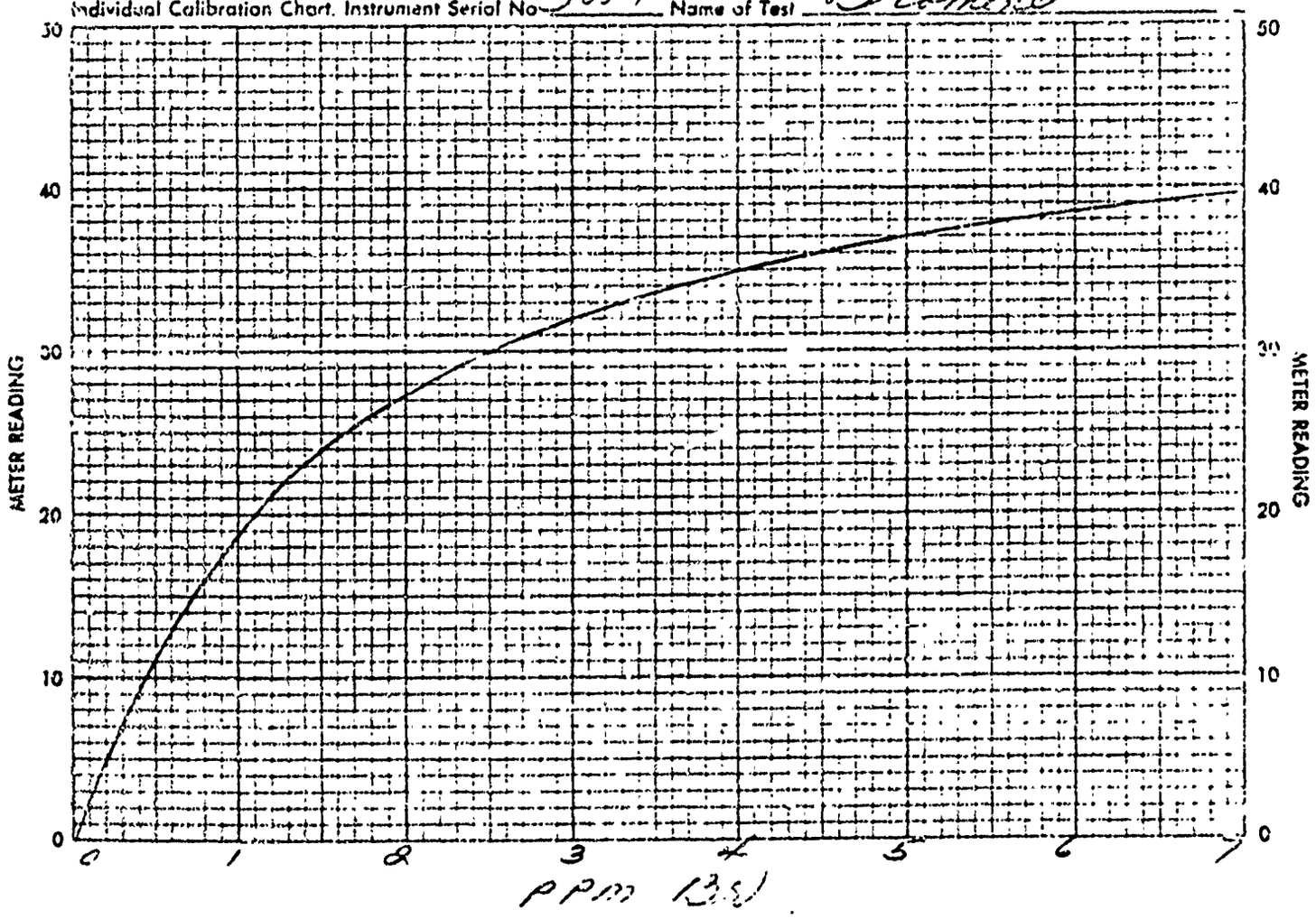
TURBIDITY

Read General Instructions at beginning of this booklet before proceeding for the first time.

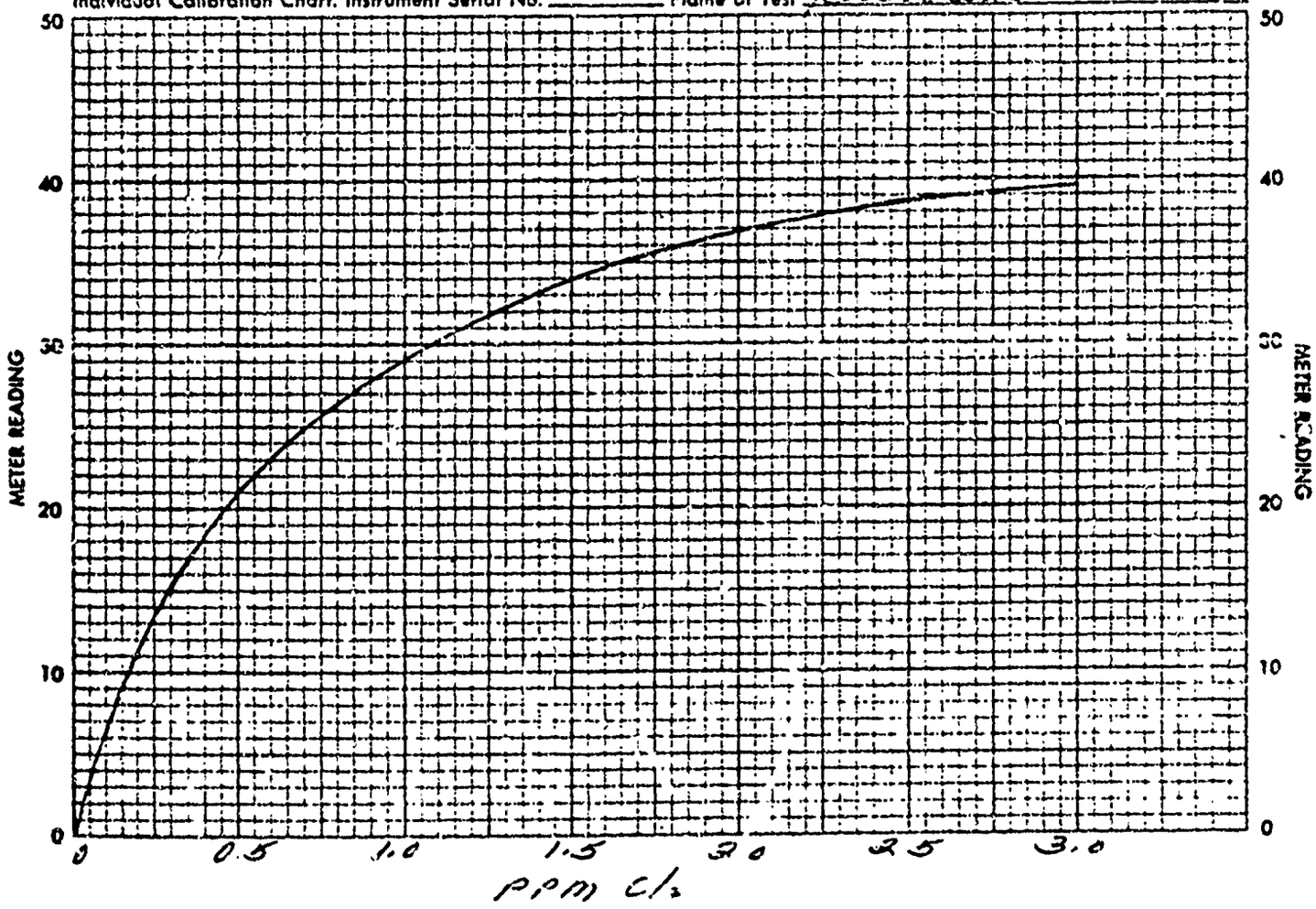
1. Filter holder should be inserted all the way into slot, so that blue dot faces upward.
2. Fill a clean, dry No. 50-T Test Cell to the mark with sample to be tested, and cap the cell.
3. Fill a second clean, dry test cell to the mark with clear, turbidity-free distilled or deionized water, for use as a blank. Cap the cell.
4. Insert the cell with the clear water into the photometer. Press button switch, and turn knob to set meter to 0. Then promptly release button switch.
5. Remove cell and replace with cell containing turbid sample. Press button, and when meter comes to rest, read meter value. Obtain Turbidity test result in parts per million, as silica, direct from Turbidity graph. Do not touch knob adjustment in this step. To extend life of battery, release push button promptly, as soon as needle comes to rest and reading has been taken. Clean cells and caps thoroughly after use, and allow to dry.

Note - The No. 50-T Test Cells must be completely clean and free from film, lint, fingerprints, etc.

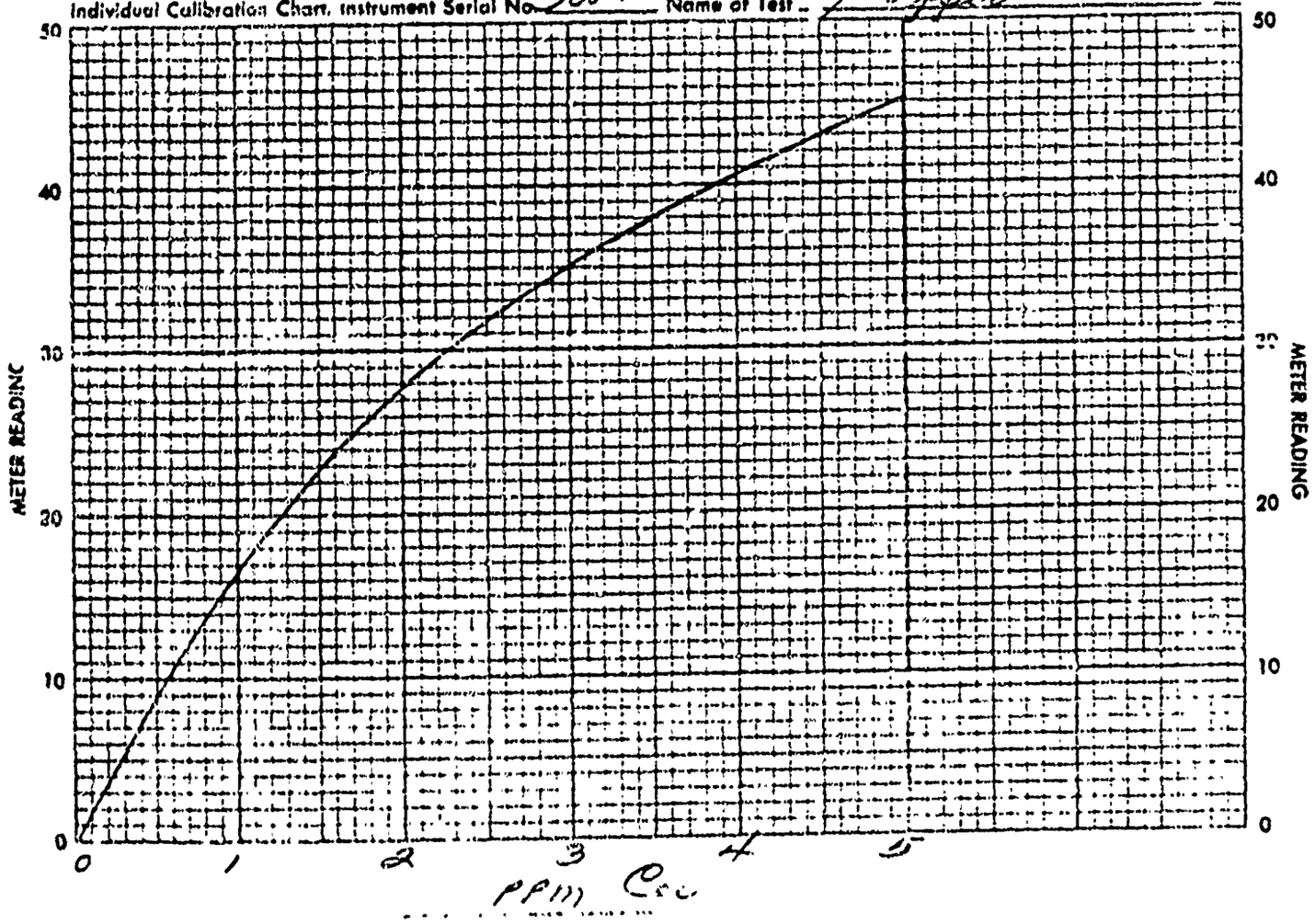
Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Bromine



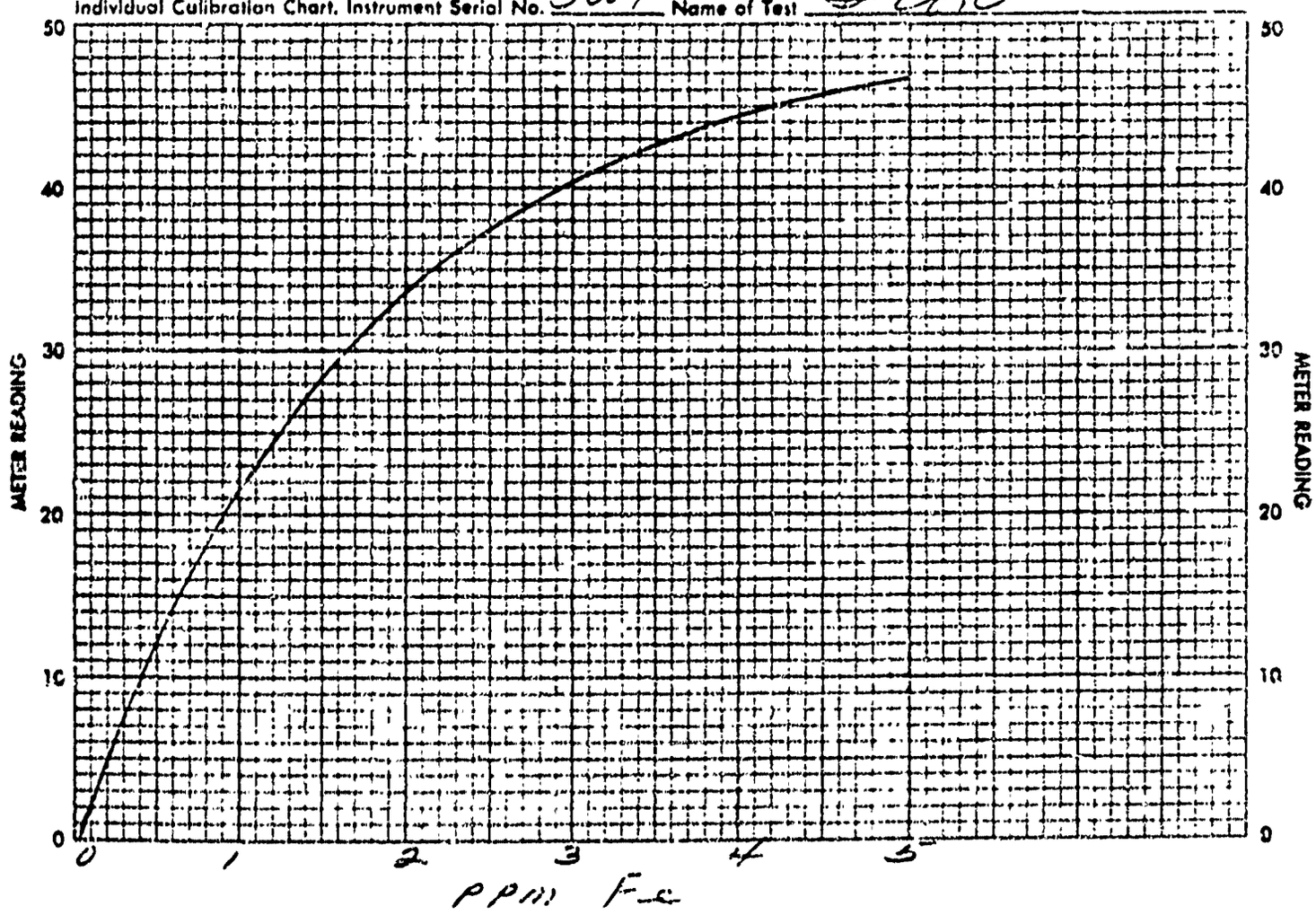
Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Chlorine



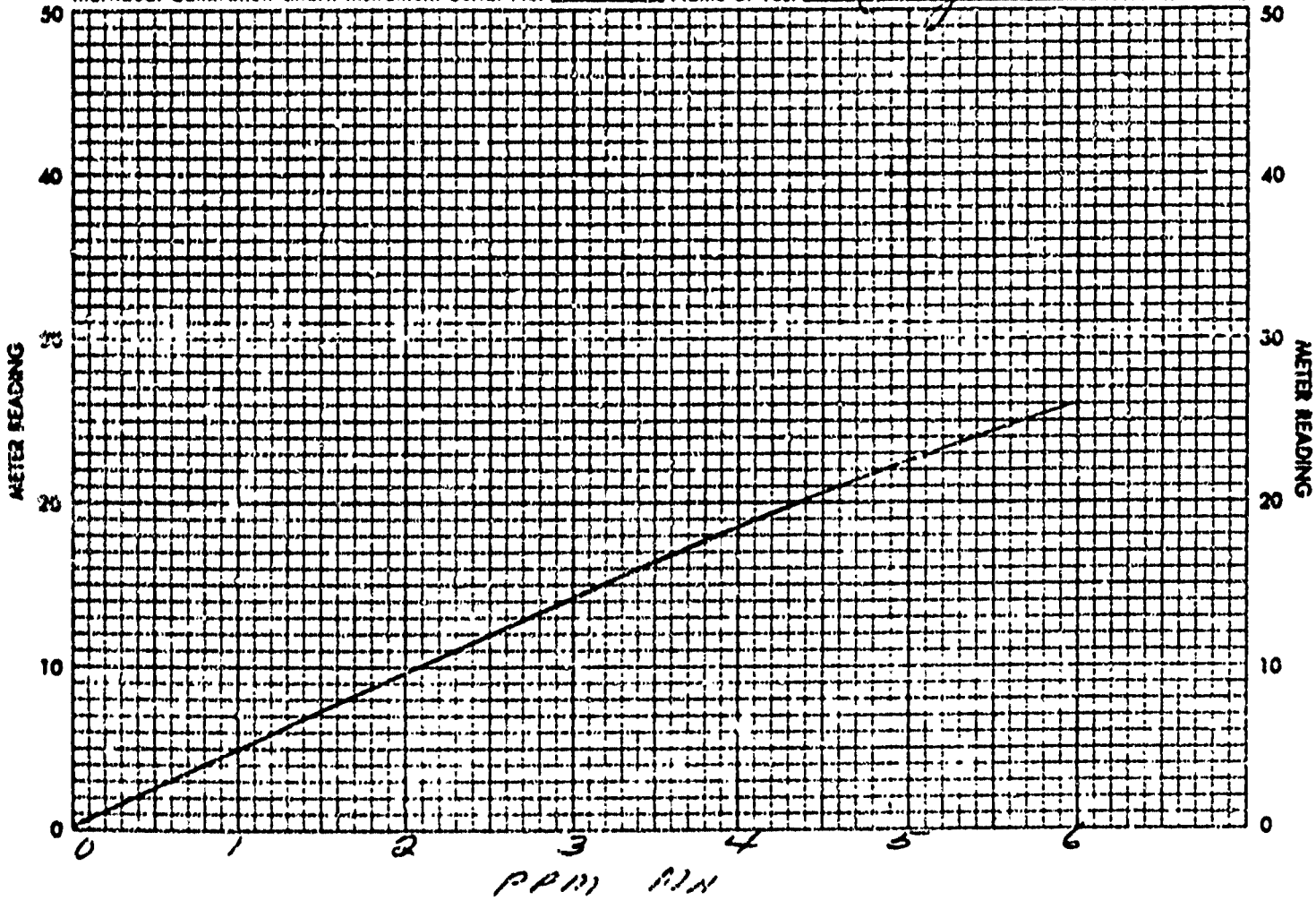
Individual Calibration Chart, Instrument Serial No. 3084 Name of Test Copper



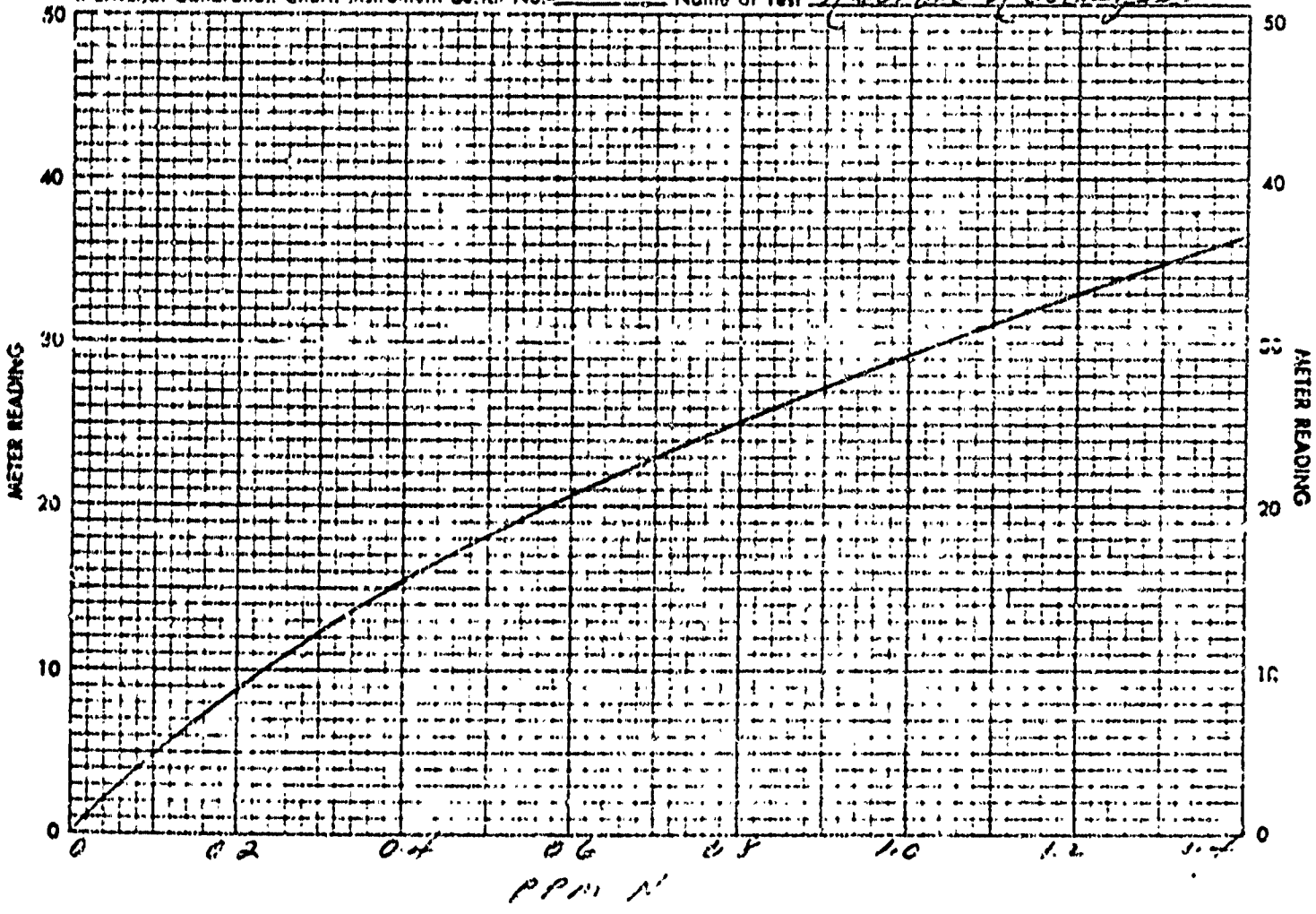
Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Iron



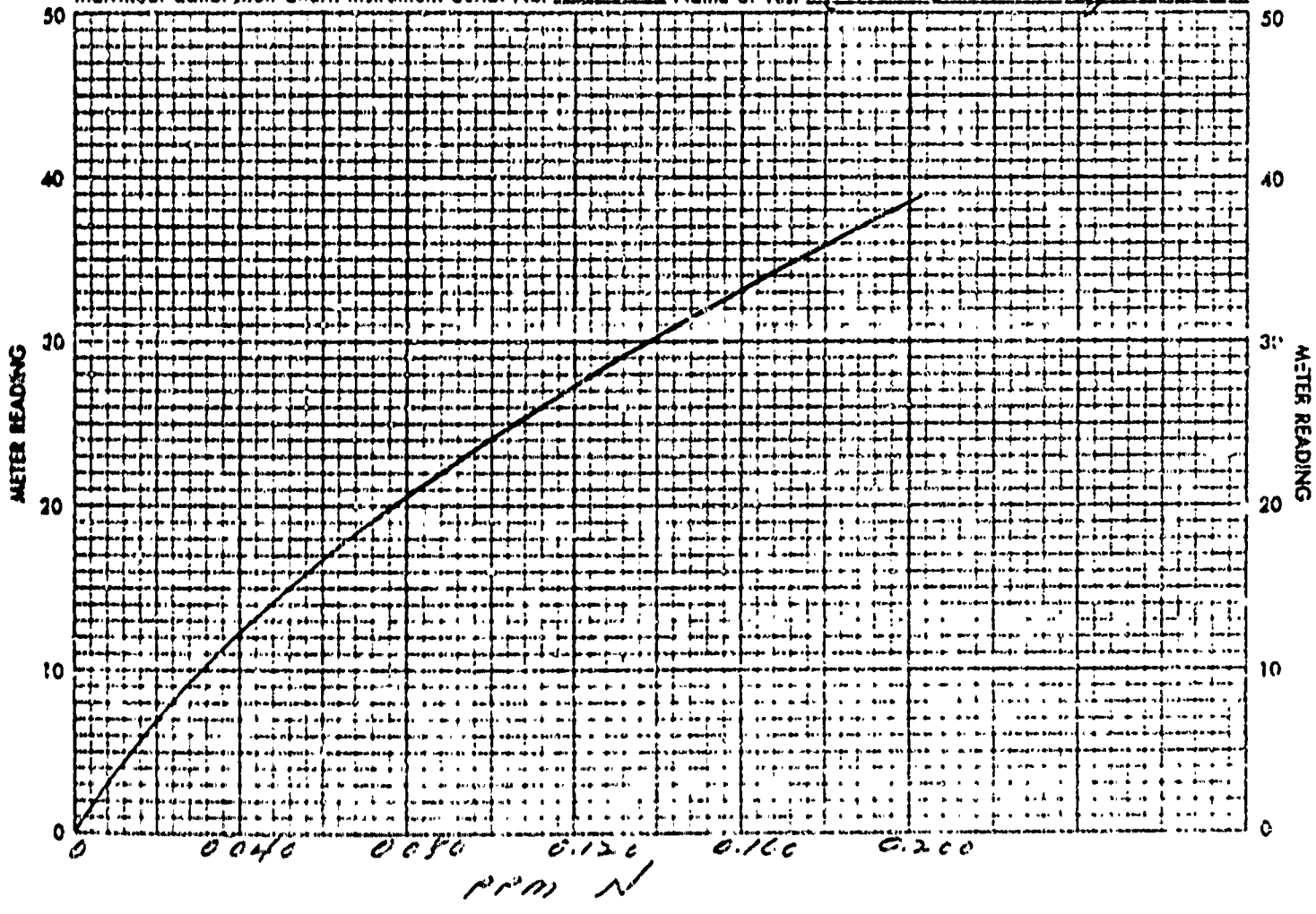
Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Manganese



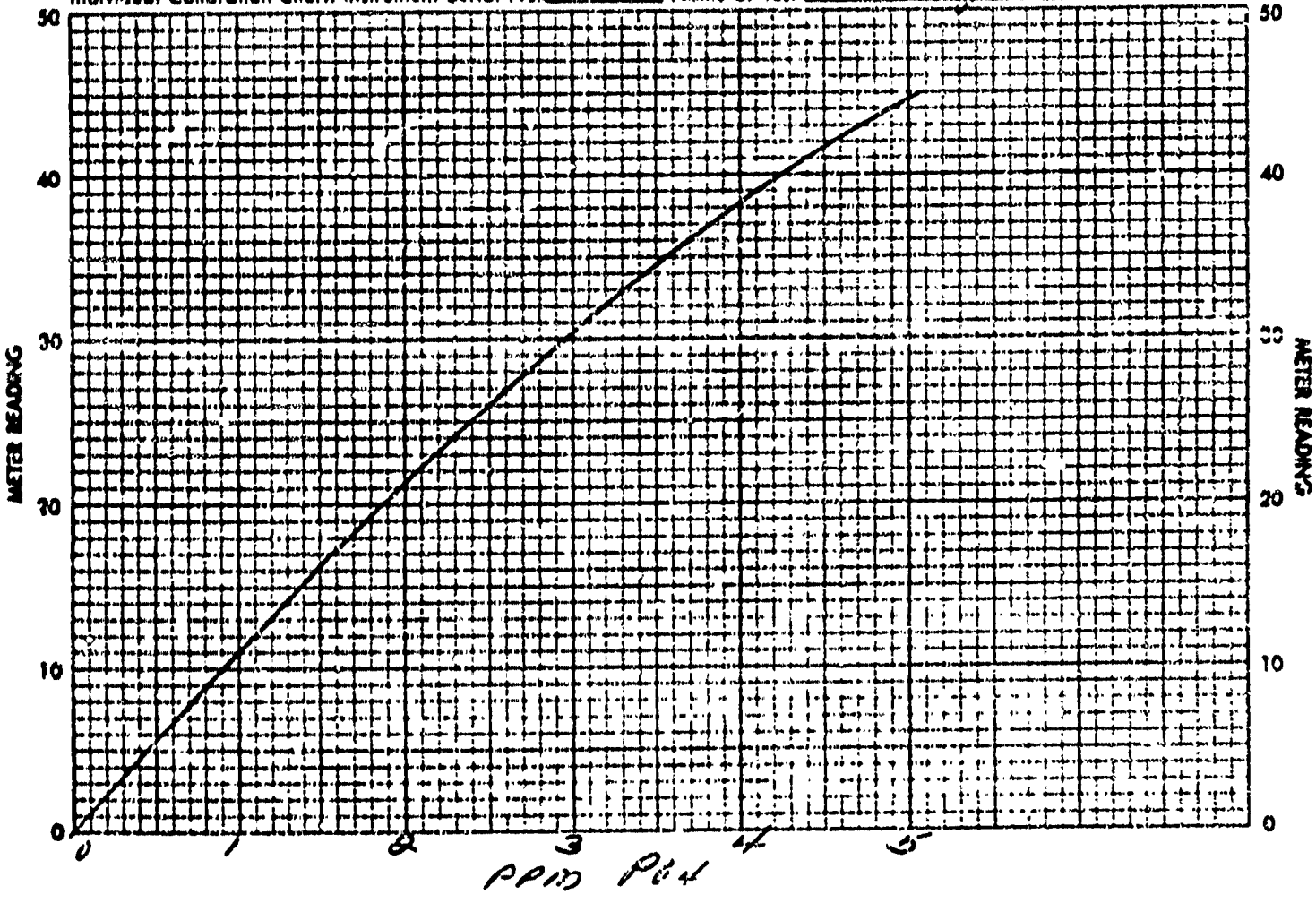
Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Nitrate Nitrogen



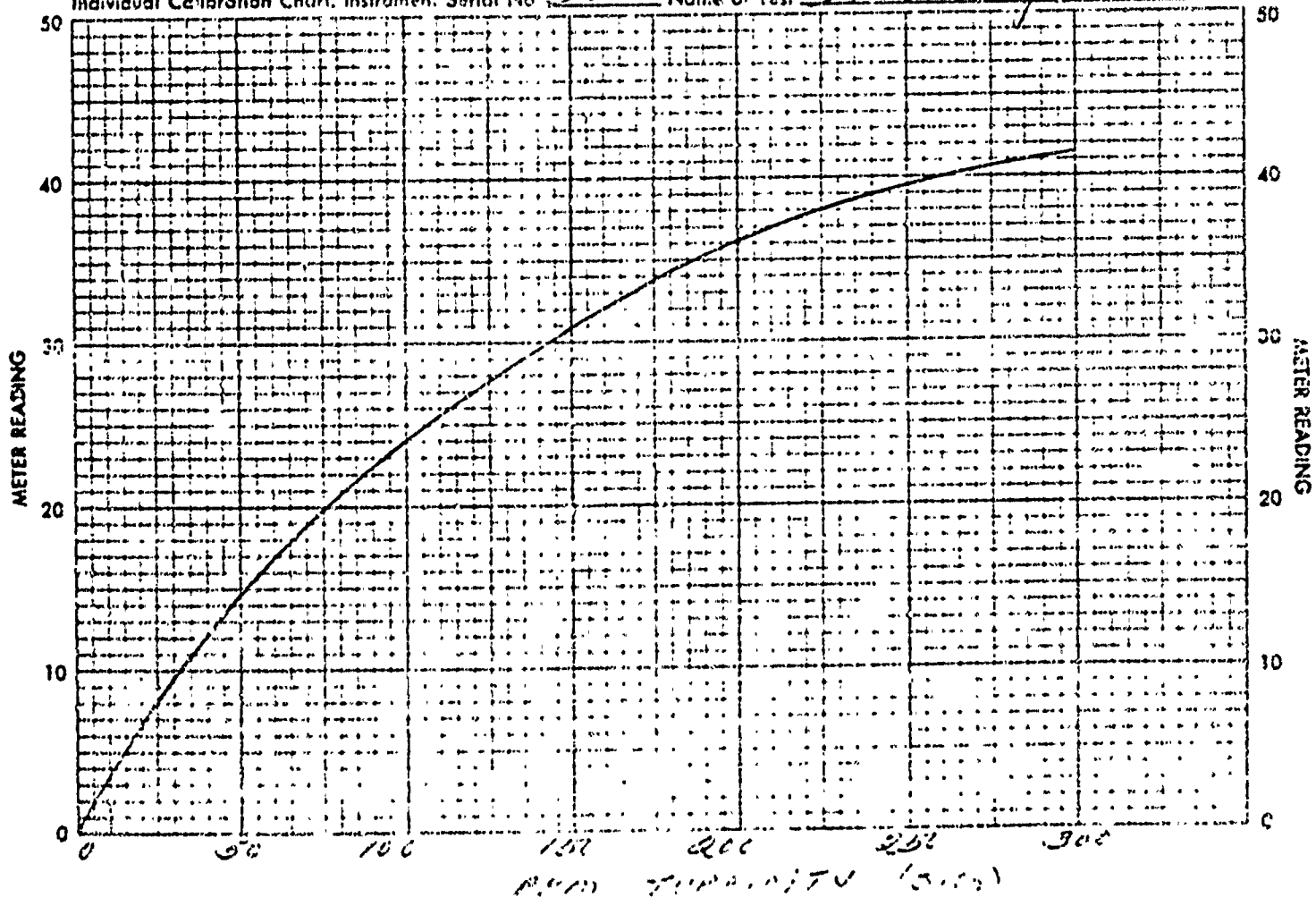
Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Nitrate Nitrogen



Individual Calibration Chart. Instrument Serial No. 3684 Name of Test Phosphate



Individual Calibration Chart. Instrument Serial No. 3084 Name of Test Turbidity



APPENDIX C

PROCEDURE FOR PREPARING SYNTHETIC BRACKISH WATER

The synthetic brackish water utilized throughout this program was prepared in the following manner:

1. The chemical ingredients were weighed out on an analytical balance in the following quantities:

Chemical	Supplier	Purity (%)	Weight (milligrams)
Sodium bicarbonate	J.T.Baker, Stock No. 3506	99.7	379
Sodium chloride	J.T.Baker, Stock No. 3624	99.5	2500
Magnesium sulfate	J.T.Baker, Stock No. 2506	98.0	545
Calcium sulfate	J.T.Baker, Stock No. 1453	98.0	632
Sodium sulfate	J.T.Baker, Stock No. 3898	99.0	135

2. One liter (1000 milliliters) of commercial-grade distilled water[a] (purchased in one-gallon containers from a local drug store) was measured out in a graduated cylinder.

3. The calcium sulfate was first added to the distilled water (at room temperature) and agitated.

(a) The conductivity of this water was found to be less than 1 micromho/cm.

4. After the solution reattained clarity (15-20 minutes), the other ingredients were added one at a time (in no particular order) with agitation until dissolved.

5. The solution was then stirred for another 30 minutes to ensure complete solubility.

6. Prior to use, the conductivity of the solution was measured and recorded.

APPENDIX D

CALCULATIONS AND PROCEDURE FOR INITIAL DESALTING OF BRACKISH WATER BY PRECIPITATION WITH SILVER OXIDE

1. Calculations (Based on Desalting One Quart of Water)

Since these calculations are based on 1 qt. of water, it is necessary to convert mg/l - as given in the specification - to mg/qt. This is done by dividing mg/l by 1.057 qt./l. This changes the weights of the ingredients as follows:

Ingredient	Mol. Wt.	Milligrams/liter	Milligrams/quart
$NaHCO_3$	84.0	379	358
NaCl	58.4	2500	2365
$MgSO_4$	120.4	545	516
$CaSO_4$	136.1	612	579
Na_2SO_4	142.0	115	109
TOTAL		4151	3927

a. Silver Oxide - Amount required to react with sodium chloride to remove all chloride ions:

Mol. wt. → 231.8 116.8

$$Ag_2O + 2NaCl + H_2O \rightarrow 2\ AgCl\downarrow + 2\ NaOH$$

Wt. in gms. → X 2.365

$$\text{Wt } Ag_2O = \frac{2.365}{116.8} \times 231.8 = 4.70 \text{ gm./qt.}$$

b. Barium Hydrate - Amount required to remove sulfates:

First, determine amount needed to remove <u>all</u> sulfates.

<u>$MgSO_4$</u>

Mol. Wt. ⟶ 315.5 120.4

$$Ba(OH)_2 \cdot 8H_2O + MgSO_4 \rightarrow BaSO_4\downarrow + Mg(OH)_2\downarrow + 8H_2O$$

Wt. in gms. ⟶ X .516

$$\text{Wt. BaH} = \frac{.516}{120.4} \times 315.5 = 1.35 \text{ gm}$$

<u>$CaSO_4$</u>

Mol. Wt. ⟶ 315.5 136.1

$$Ba(OH)_2 \cdot 8H_2O + CaSO_4 \rightarrow BaSO_4\downarrow + Ca(OH)_2\downarrow + 8H_2O$$

Wt. in gms. ⟶ X .579

$$\text{Wt. BaH} = \frac{.579}{136.1} \times 315.5 = 1.34 \text{ gm.}$$

<u>Na_2SO_4</u>

Mol. Wt. ⟶ 315.5 142.0

$$Ba(OH)_2 \cdot 8H_2O + Na_2SO_4 \rightarrow BaSO_4\downarrow + 2\ NaOH + 8H_2O$$

Wt. in gms. ⟶ X .109

$$\text{Wt. BaH} = \frac{.109}{142.0} \times 315.5 = 0.24 \text{ gm.}$$

Total BaH required to remove <u>all</u> sulfates from 1 qt. = 1.35 + 1.34 + 0.24
= 2.93 gms.

Since the silver oxide theoretically removed all chloride ions, amounting to 1515 ppm, and thereby leaving 2636 ppm TDS (4151-1515), it is still necessary to remove 636 ppm (or 602 mg/qt.) in the form of sulfate ions in order to reach the specified TDS level of 2000 ppm. At the same time, this would reduce the sulfate ion concentration from its original level of 945 ppm (or 895 mg/qt.) to 309 ppm (or 293 mg/qt.), which is below the specified maximum of 500 ppm.

Therefore, the quantity of BaH actually needed becomes:

$$\frac{602}{895} \times 2.93$$

$$= 1.97 \text{ gm/qt.}$$

c. Citric Acid - Amount required will be experimentally determined (see Procedure, step h).

2. Procedure

a. Using one pint (473.2 ml)[a] of synthetic brackish water, take following readings (as well as at end of test) and record.

Reading	Method	Approximate Value	
		Before	After
pH	pH Meter	7.8	10.5 - 11.0
TDS	Delta Scientific Co. Model 1114 Conductivity Meter	7200 micromhos/cm. (equiv. to 6,700 ppm as $CaCO_3$ or 4150 ppm as ion)	4,300 micromhos/cm. (equiv. to 3,900 ppm as $CaCO_3$ or 2,000 ppm as ion)
Sulfate ion concentration	Delta Scientific Co. Model 50 Water Test Kit, per para. 4.6.4.8, MIL-D-5531.	945 ppm as ion	309 ppm as ion
Chloride ion concentration	Delta Scientific Co. Model 50 Water Test Kit, per para. 4.6.4.9, MIL-D-5531.	1,515 ppm as ion	0 ppm as ion

(a) A pint, rather than quart, was initially used to conserve chemicals during early stages of the investigation.

b. Add 2.35 gms.[a] of silver oxide to the brackish water sample and stir.

c. After 5 minutes, take conductivity readings at 5 minute intervals until 2 consecutive equal readings are obtained.

d. If meter reading is not at 5000 micromhos/cm.,[b] continue adding known weights of silver oxide in small portions, taking readings at 5 minute intervals, until this figure is reached. Record final weight of silver oxide required and pH.

e. Add 0.98 gm.[c] of barium hydrate to the water sample and stir.

f. After 5 minutes, take conductivity readings at 5 minute intervals until 2 consecutive equal readings are obtained.

g. If meter reading is not at 4300 micromhos/cm., continue adding known weights of barium hydrate in small portions, taking readings at 5 minute intervals, until this figure is reached. Record final weight of barium hydrate required and pH.

h. If final pH value is greater than 11.0, indicating that water is highly alkaline due to formation of NaOH, add citric acid until a pH range of 10.5-11.0 is reached. Record weight of citric acid required.

(a) Calculated weight, based on 1 quart, was 4.70 gms.; therefore, amount for 1 pint is one-half, or 2.35 gms.

(b) Assuming silver oxide removes <u>all</u> chloride ions, which amount to 2,136 ppm as $CaCO_3$, this leaves 4,562 ppm TDS in sample (6698-2136); latter is equivalent to conductivity of 5000 micromhos/cm.

(c) Calculated weight, based on 1 quart, was 1.97 gms.; therefore, amount for 1 pint is one-half, or 0.98 gm.

i. Filter liquid to remove precipitated material.

j. Conduct tests on filtrate to determine sulfate and chloride ion concentrations.

APPENDIX E

CALCULATIONS AND PROCEDURE FOR INITIAL DESALTING OF BRACKISH WATER BY ION EXCHANGE METHOD USING DIOLITE C20X10 AND A7 RESINS

1. Calculations (Based on Desalting One Quart of Water)

To determine the quantities of resins needed for initial evaluation (referred to as "starting point" quantities), it is first necessary to calculate the gram-milliequivalents of ions to be removed. This is accomplished as shown in the accompanying Table XII.

Thus, the approximate weight of cation resin (C20X10) required

$$= \frac{\text{meq.'s of cations to be removed/qt.}}{\text{capacity of resin}}$$

$$= \frac{33.1 \text{ meq./qt.}}{5.1 \text{ meq./gm.}}$$

$$= 6.5 \text{ gms./qt.}$$

Approximate weight of anion resin (A7) required

$$= \frac{32.8 \text{ meq./qt.}}{9.1 \text{ meq./gm.}}$$

$$= 3.6 \text{ gms./qt.}$$

Total weight of resins required = 10.1 gms./qt.

Ratio of resins = 1.8 parts C20X10/1.0 part A7.

2. Procedure

a. Using one pint (473.2 ml.)[a] of synthetic brackish water, take following readings (as well as at end of test) and record:

(a) A pint, rather than quart, was initially used to conserve resins during the early stages of the investigation.

TABLE XII

MILLIEQUIVALENTS OF IONS TO BE REMOVED PER QUART

Ion (a)	Equivalent Weight (b)	Theoretical Concentration in Solution (c) (ppm as ion)	Assumed To Be Removed (ppm as ion)	Milliequivalents of Ion to be Removed Per Quart of Solution (e)	Ions Remaining in Solution	
					PPM as Ion	PPM as $CaCO_3$ (f)
Ca^{++}	20.04	180	180	8.5	0	0
Mg^{++}	12.15	110	110	8.6	0	0
Na^{+}	23.0	1,126	390 (d)	16.0	736	1604
$SO_4^{=}$	48.0	945	945	18.7	0	0
Cl^{-}	35.5	1,515	526 (d)	14.1	989	1394
HCO_3^{-}	61.0	275	0	0	275	226
Total	--	4,151	2,151	Cations - 33.1 Anions - 32.8	2,000	3,224

(a) Listed in order of relative affinity for respective ion exchange resin; i.e., Ca > Mg > Na and SO_4 > Cl > HCO_3.

(b) Equivalent wt. = $\frac{\text{Atomic wt.}}{\text{Valence}}$

(c) See "Totals" shown earlier in Table III.

(d) Proportion of Na^+ and Cl^- ions to be removed is same as originally in solution, i.e., 1.35 parts Cl^- to 1.0 part Na^+.

(e) Meq./qt. = $\frac{\text{ppm to be removed}}{1.057 \times \text{equivalent wt.}}$

(f) Using conversion factors shown in Table II.

Reading	Method	Approximate Value	
		Before	After
pH	pH Meter	7.8	4.0-11.0
TDS	Delta Scientific Co. Model 1114 Conductivity Meter	7,200 micromhos/cm (equiv. to 6,700 ppm as $CaCO_3$ or 4150 ppm as ion)	3,600 micromhos/cm. (equiv. to 3,200 ppm as $CaCO_3$ or 2000 ppm as ion)
Sulfate ion concentration	Delta Scientific Co. Model 50 Water Test Kit, per para. 4.6.4.8, MIL-D-5531	945 ppm as ion	0 ppm as ion
Chloride ion concentration	Delta Scientific Co. Model 50 Water Test Kit, per para. 4.6.4.9, MIL-D-5531	1,515 ppm as ion	989 ppm as ion

b. Prepare Duolite ion-exchange resins (C20X10 and A7) in following manner, if wet[a] (otherwise, proceed to step (5):

(1) Weigh out approx. 50 gms. of each resin.

(2) Put each resin in separate container and dry in vacuum at 40°C (104°F) max. for 2 hours.

(3) Cool down resins in desiccator for approx. 10 minutes and weigh.

(4) Repeat step (2) for 1 hr. and weigh. Continue this procedure (i.e., dry 1 hr. and weigh) until 2 consecutive uniform weighings are obtained.

(a) These resins, as received, are already regenerated.

(5) Blend resins together at ratio of 1.8 parts C20X10 to 1.0 part A7 (by weight).

(6) Use mortar and pestle to grind up blended material as uniformly as possible.

c. Add 5.05 gms.[a] of dried, ground, blended material (i.e., consisting of 3.25 gms. C20X10 and 1.80 gms. A7) to 1 pint of synthetic brackish water and mix.

d. Check conductivity at 5 minute intervals until stabilized, i.e., until 2 consecutive readings are obtained.

e. Record final conductivity and pH.

f. Filter liquid to remove resins and any precipitated material which may form.

g. Conduct tests on filtrate to determine sulfate and chloride ion concentrations.

(a) Calculated total weight, based on 1 quart, was 10.1 gms.; therefore, quantity for 1 pint is one-half, or 5.05 gms.

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