NASA CR 65735

HOUSTON RESEARCH INSTITUTE, INC.

DESIGN AND ENGINEERING DIVISION OF HOUSTON RESEARCH INSTITUTE, INC.,

6001 GULF FREEWAY, HOUSTON, TEXAS 77023, 713 WA 8-5001

CAUSES AND EXPLANATION OF "BREAKTHROUGH PHENOMENON" WHEN LEM COOLING SYSTEM SUBLIMATOR IS FED WITH CHLORINATED FEEDWATER.

LIBRARY COPY

SEP 29 1967

MANNED SPACECRAFT CENTER
HOUSTON, TEXAS

Design and Engineering

Research and Development

Economics, Marketing and Management Services

N67-37555 (ACCESSION NUMBER) (THRU)

(PAGES) (CODE)

CR-65735 (NASA CR OR TMX OR AD NUMBER) 13 (CATEGORY)

FACILITY FORM 602

Contract No. NAS 9-6698
Task Order No. 5
August 30, 1967

Prepared for:
NASA MANNED SPACECRAFT CENTER
CREW SYSTEMS DIVISION
HOUSTON, TEXAS

HOUSTON RESEARCH INSTITUTE, INC.
DESIGN AND ENGINEERING — RESEARCH AND DEVELOPMENT — ECONOMICS AND MARKETING
6001 GULF FREEWAY · HOUSTON, TEXAS 77023 · 713 WALNUT 8 5001

FINAL TASK ORDER REPORT
ON
CAUSES AND EXPLANATION OF "BREAKTHROUGH PHENOMENON" WHEN LEM COOLING SYSTEM SUBLIMATOR IS FED WITH CHLORINATED FEEDWATER.

Contract No. NAS 9-6698
Task Order No. 5
August 30, 1967

BY
Dr. Frank L. Worley
Dr. W. I. Honeywell
Allen H. Blair
Of
Houston Research Institute Inc.
Houston Texas

Prepared For
NASA MANNED SPACECRAFT CENTER
CREW SYSTEMS DIVISION
HOUSTON, TEXAS

Copy No. 1

Project No. 41,805

Allen H. Blair, Task Manager

1. INTRODUCTION

On June 23, 1967 NASA-MSC directed Houston Research Institute Inc. to perform Task Order No. 5 under contract NAS 9-6698. After several meetings with technical personnel to obtain background and other information, the below-described information, conclusions, and recommendations resulted from this effort.

2. BACKGROUND

A sublimator is to be used to remove excess heat from the LM. This device vaporizes water into steam, which is exhausted into the vacuum of space, ice being an intermediate material in the process. A subcontractor test of a test section of the module resulted in unexplained phenomena when chlorinated water was used as feed water. A green color was formed, ice or snow appeared on the downstream side of the sublimator sintered plate, and this seemed to occur in a cyclic manner. Details appear in contractor report No. PMP-LEM-140 of May 1, 1967.

3. OBJECTIVE

As stated summarily in Task Order No. 5 and subsequently elaborated verbally by Mr. W.W. Guy and staff, HRI Inc. was retained to analyze and explain the unusual phenomena which occur when chlorine is used as a bactericide, and also to comment about the probability of like or similar happenings which might occur when Iodine is used as a bactericide.

4. ANALYSIS OF BREAKTHROUGH PHENOMENA (Dr. WIH)

The breakthrough phenomena occurring with the use of chlorinated feed water may be explained (1) by examining the chemistry of the chlorine-water-nickel system and (2) by considering the consequent effects on the physical and hydrodynamic behavior of the system.

4.1 Solubility of Chlorine

When chlorine gas is bubbled through water, several reactions take place consecutively. Some of the chlorine gas is dissolved

but at low concentration almost all of it reacts with water according to the reaction:

$$Cl_2 + H_2O \longrightarrow HCl + HOCl \qquad (I)$$

In fact, at a total chlorine concentration of 6 ppm, the amount of Cl_2 unreacted, i.e. present as dissolved gas, is in the order of 10^{-10}% (See Figure 1).

The covalent HCl almost completely dissociates in dilute aqueous solution.

$$HCl + H_2O \rightarrow H_3O^+ + Cl^- \qquad (II)$$

HOCl is a weak acid and only partly dissociates according to

$$HOCl \rightleftharpoons HCl + 1/2\ O_2 \qquad (III)$$

4.2 Reaction with Nickel

A dilute solution of HCl will react with nickel to give the salt nickelous chloride.[13]

$$Ni + HCl\ (dilute) \longrightarrow H_2 + NiCl_2 \qquad (IV)$$

An additional effect of the presence of HCl on this reaction is to reduce the solubility of the $NiCl_2$.[14] Removal of $NiCl_2$ from solution by this mechanism will tend to displace reaction (IV) toward completion. This reaction further has the effect that it removes HCl from its equilibrium feed water concentration causing reaction (I) to proceed further toward completion.

One would expect that the concentration of HOCl would build up as reaction (I) shifts toward the right. This will not occur with Ni present for two reasons. Even though the HOCl dissociates only slightly by reaction (III), the removal of HCl by reaction (IV) will consequently shift (III) to the right and its equilibrium concentration will decrease. A second reason involves the rate

of HOCl dissociation. Normally, the reaction proceeds very slowly. However, in the presence of a rough surface and NiO, the reaction proceeds readily.[1]

Both of these conditions are present in the sintered nickel plate of the sublimator.

The validity of the above mechanism is substantiated by the experimental results of the sublimator tests. First of all, $NiCl_2$ was found to be the principal compound on the surface of the sublimator at the end of the tests. Secondly, the presence of a green film on the sublimator during the test suggests that nickel chloride was present, since it forms several hydrates, all of which are green.[2]

4.3 Chlorine Hydrate Formation

The possibility of chlorine hydrate formation has been suggested. It is true that chlorine can form hydrates at low temperatures. However, all of these are difficult to form under ordinary concentrations (they are all unstable above 9°C). At the very low concentrations of Cl_2 present in the feed water (refer to Figure I), the formation of chlorine hydrates is virtually impossible. Further, they are yellow in color, whereas the ice formation on the sublimator was indicated to be green.

4.4 Concentration Calculations

4.41 Solubility of Chlorine

The concentration of chlorine present as dissolved gas or as the hydrolytic products HCl or HOCl may be calculated from the equilibrium constant for reaction (I).

$$K_1 \equiv \frac{[HCl]\ [HOCl]}{[Cl_2]\ [H_2O]}$$

The brackets indicate the activities of each of the components. In dilute solutions the activity coefficients are unity [3]--the ideal solution approximation--and the activities may be replaced with mol fractions or other suitable concentration units.

Since the HCl ionizes readily according to reaction (II), the equilibrium constant is more frequently reported as

$$K \equiv \frac{[H^+]\ [Cl^-]\ [HOCl]}{[Cl_2]}$$

where the mol fraction of water in dilute solution is assumed to be unity. The hydrated ion H_3O^+ is written for simplicity as H^+.

From the stoichiometry of the reaction the concentration of HOCl will be the same as that for H^+ and Cl^- in the event that it dissociates only slightly. Since the equilibrium constant for reaction (III), dissociation of HOCl, is about 10^{-8}, this will indeed be the case.[4] Therefore, we can solve for the concentration of chloride ion:

$$[Cl^-] = \left\{K[Cl_2]\right\}^{1/3}$$

The total chlorine present in solution may thus be expressed as:

Total Chlorine present	=	Chlorine as dissolved gas	+	Chlorine present as Hydrolytic products
	=	$[Cl_2]$	+	$\left\{K[Cl_2]\right\}^{1/3}$

Experimental values for K have been reported by Whitney and Vivian.[5]

Temperature °C	$K(\text{lb-moles/cu.ft})^2$
10	7.10
15	8.55
20	10.7
25	12.8

Examining the percent of chlorine present as dissolved gas, we have at 25°C.:

$$\text{Percent Dissolved Chlorine Gas} = \frac{[Cl_2]}{[Cl_2] + \left\{K[Cl_2]\right\}^{1/3}}$$

$$= \frac{1}{1+2.34\,[Cl_2]^{-2/3}}$$

The results with chlorine expressed as lb. Cl_2/ft^3 are plotted in Figure 1.

4.42 $NiCl_2$ Salt Accumulation

For the reasons discussed above, the total amount of $NiCl_2$ salt formed will be equivalent to the total amount of Cl_2 put into the sublimator feed water. The Cl_2 concentration of 6 ppm gives an equivalent amount of 6.9×10^{-4} lb. $NiCl_2/ft^3$ solution. At a feed water rate of 0.5 lb. solution per hour, this amounts to the accumulation of

$$6.9 \times 10^{-4} \quad (62.4)^{-1}\ 0.5 = 5.5 \times 10^{-6} \text{ lb. } NiCl_2/\text{hr.}$$

In the 10 hour period between observed cycles, about 5.5×10^{-5} lb. of $NiCl_2$ could have accumulated. As shown below (Section 7.3) this accumulation is large enough to have caused the observed breakthrough phenomena.

5. Alternate Possible Mechanism for $NiCl_2$ Formation (AHB)

An alternate possible mechanism of $NiCl_2$ formation can be expressed as follows:

$$Cl_2 + H_2O \longrightarrow HOCl + HCl \qquad \text{(I)}$$

$$HOCl \longrightarrow HCl + [O] \qquad \text{(VI)}$$

$$[O] + Ni \longrightarrow NiO \qquad \text{(VII)}$$

$$NiO + 2HCl \longrightarrow NiCl_2 + H_2O \qquad \text{(VIII)}$$

Since the porous nickel plate presents an enormous surface area per unit volume to the HOCl, HCl, and nascent oxygen reactants, the likelihood of such reactions occurring at a rapid rate is very great.

6. Performance of Iodine Solution in the Sublimator (Dr. W.I.H.)

The reactivity of iodine is much less than that of chlorine. It is much less electronegative and accordingly a much poorer oxidizing agent [6]. In aqueous solution the tendency is for the I_2 to remain in solution rather than to form hydroiodic acid analogous to the chlorine reaction (I). The reaction

$$I_2 + H_2O \rightleftharpoons HI + HOI \qquad \text{(V)}$$

has an equilibrium constant of 3×10^{-13} at 25°C [7], and is therefore of no practical importance. This reaction has no chance to be shifted to the right by decomposition of HOI since the dissociation constant[8] is also 10^{-13}. The reaction of water with iodine takes place in a complicated way (9) giving the end products hypoiodite, IO^-, and polyiodide ions such as I_3^-.

Another possible reaction

$$3I_2 + 3H_2O \rightleftharpoons IO_3^- + 5I^- + 6H^+ \qquad \text{(VI)}$$

lies so far to the left that it is used to estimate the presence of free hydrogen ion [10].

The presence of iodine in aqueous solution is therefore mostly confined to the form I_2. Thus, the solubility of this species should be examined. At 25°C. it is soluble to the extent of 0.34 gm/ liter and decreases solubility rapidly to 0.16 gm/ liter at 0°C.[11]. The effect of these solubilities on the operation of the sublimator is examined below.

The possibility of formation of the salt NiI_2 is small since it requires a definitely alkaline solution of the hydroxide

in hydroiodic acid [12]. The possibility of any significant form of iodine besides I_2 in the sublimator is therefore discounted.

7. Analysis of Chlorine Case (Dr. F. L. W.)

It has been experimentally observed that breakthrough of liquid water does occur under certain conditions. This effect appears to offer a source of potential operating problems when the feedwater has been treated with chlorine.

In previous sections of this report it has been concluded that the use of chlorine as a bactericide will lead to the formation of nickel chloride by the action of the chlorine acids on nickel present in the system. The presence of this salt in the water will cause several physical properties of the water phase to change. The changes arise because water is selectively removed from this phase by vaporization in the porous plate of the evaporator.

A material balance around this porous element requires that the concentration of nickel chloride increase with time in direct proportion to the amount of chlorine entering the system. This concentration increase causes the density of the water phase to increase, the vapor pressure (Figure 2) and the freezing point of the solution (Figure 3) to decrease. The latter two are the changes of interest as they have a direct effect on the breakthrough.

The observations reported in the "Preliminary Test Report", Test Results on Sublimer Performance with Chlorine Feedwater, dated May 1, 1967, indicate several events which are reported to be unique with the chlorinated feedwater supply. These are: (a) green film formation and subsequent disappearance, (b) wetting of the surface and (c) formation of low density ice on the surface. These items are to be discussed and probable mechanisms presented in the following sections.

7.1 Green film: The appearance of the green film is due to the formation of nickel chloride and the subsequent formation of a very thin film of nickel chloride solution on or at the surface of the porous metal. The appearance of the green color and its intensification is directly related to the vaporization of the water from the pores and the increase in nickel chloride concentration due to the selection process.

At the higher heat loads vapor is formed at a higher rate and the interface between the vapor phase and the water-ice phase recedesinto the porous plate. This effect causes the nickel chloride solution to be drawn back into the pore structure by surface tension. Although the exterior surface may still be wetted by the solution, the effective thickness on the peaks will be extremely thin. If the solution has not concentrated to a high enough level, local freezing will also occur.

Such a very thin layer will not be visible* to the observer even though the nickel chloride is still present at the surface. To the observer the surface will appear dry. Any local breakthrough which will provide fluid to increase the fluid volume (as ice or solution) will cause the green color to "re-appear". If breakthrough does not occur the plate will be dry and have the appearance and color of the porous nickel plate.

7.2 Wetting of the surface: The appearance of the slush and wet areas on the surface is due to minor local breakthrough which provides a temporary surplus of liquid which forms a solution with the nickel chloride previously deposited in and on the porous plate. This breakthrough was observed to be closely connected with external variables such as change in heat load, feedwater, pressure variation, etc. In general, this is a self-correcting effect in that the water expelled has a high vapor pressure and will

* See Appendix: Experimental Work/Green Film

vaporize at a very high rate causing rapid cooling and freezing in the region. This ice then sublimes and the "wet" region vanishes as soon as the ice layer retreats into the pores.

7.3 Ice formation: The formation of ice masses on the surface of the plate is due to local breakthrough at a rate such that: 1) a vapor cloud forms, producing ice crystals, 2) small jets of water form streamers of ice. Although this results in large masses of ice on the surface, the effect is self-correcting as previously noted.

In all of these cases the effects are related to the phenomena called "breakthrough" by the observer in the report covering the preliminary test. The following section presents a mechanism by which this can occur. It should be noted that the presentation is related to local or isolated effects and that the effects are time dependent. One area will be at one "age" and another at another "age". This "age" is not just a certain number of clock hours, but is also related to the local heat flux which provides energy to vaporize water. The nickel chloride associated with the solution is deposited in the pore structure causing a lowering of the local vapor pressure and freezing point. The gross effects observed are due to an average for any particular area on the plate. Thus, it is possible for breakthrough to occur in any isolated region if the average age reaches the critical value.

Breakthrough requires that water flow to the surface at a relatively high rate. This is easily accomplished if the pore structure does not contain ice, as the pressure drop for feed rates expected (0.30 to 0.70 lbs/hr for a plate having about 25 in^2) is much less than the 4 to 6 psi pressure differential available. This is true even if the flow is through less than 1% of the superficial area.

An estimate of the location of the ice front at the time of vaporization may be obtained by calculating the thickness of porous plate required to yield the gas phase pressure drop known to exist. The concentration of nickel chloride at the interface was

assumed to be between 10 and 20 % by weight. This concentration corresponds to the amount of salt as calculated in Section 4.42* The equilibrium vapor pressure in this solution is about 1.5 psia. The thickness of plate required for a gas phase pressure drop of this magnitude at a feed rate of 0.5 lb/hr for the test module is 0.023 inches. The thickness of the actual plate was 0.024 ± inches. While the accuracy of the calculation is not the best, it is felt that the result obtained is reasonable.

This implies that just before breakthrough the ice interface has moved to the feed water side of the porous plate. At this location the ice layer becomes very thin due to freezing point depression by the nickel chloride and as a result not able to withstand the 4 to 6 psi gradient. An idealized view of this model is presented as Figure 4. Any sharp change in this pressure due to external conditions will result in the rupturing of ice although breakthrough would eventually occur in either case. Breakthrough is followed by a rapid influx of water into the pore structure. This water carries with it the accumulated nickel chloride associated with the pore. The flow can be visualized as a plug of high concentration solution followed by relative pure feed water. As soon as the feed water reaches the low pressure side, rapid vaporization occurs and generates a local dense water vapor cloud which condenses on the adjacent surfaces as low density ice or snow. If the breakthrough is large enough, streams of liquid will jet into the free space, forming ice. The $NiCl_2$ salt appears as a contaminate and causes coloration of this mass.

This local effect can spread (due to heating) due to the influx of feed water, which can cause adjacent pores to breakthrough "ahead of schedule". The self-propogating feature of the system is damped by the vaporization of feed water and within a short period of time the region again returns to the initial condition (ice and low temperature feed water) in the pore structure.

However, unless the ice mass can be physically lifted and removed from the surface (for example, it might separate

* See appendix: Experimental Work/Green Film

at the surface due to local temperature and fall from the surface if a gravity field is present), the nickel chloride will again be deposited on the porous metal surface. This accumulation must occur because the water present in the ice will sublime, leaving the non-volative nickel chloride. Thus, the breakthrough will not remove the cause of the upset when the ice is removed by sublimation. However, when the surface ice sublimes, the net energy removed from the coolant system will not be changed by the breakthrough if the heat exchanger does not obtain heat from external sources. This does not imply that satisfactory operation can be obtained under breakthrough conditions, as secondary effects such as loss of liquid through the steam exhaust system are very undesirable.

The magnitude of this breakthrough will be dependent upon several factors, namely, magnitude and duration of the pressure pulse, temperature of the local feed water, total $NiCl_2$ deposited in the pore structure, or inability of the steam-side chamber to remove the vapor formed by the breakthrough. These factors are closely tied to the actual design material and construction of module and physical test system.

8. Iodine Case (Dr. F.L.W.)

Investigation of the iodine-water system properties in the presence of nickel metal has been presented in Section 6. The following deals with the potential problems associated with the use of iodine (dissolved in the feed water) as a bactericide in the concentration range 1 to 10 parts per million (ppm). The feed water rate is taken to be between 0.30 and 0.70 lbs/hr. The results are reported in terms of the amount of iodine delivered to the test module per test module porous surface.

A search of the literature reveals that elemental iodine is very insoluble in water (0.16 gms iodine/liter solution at 32°F).

The desired requirement is that all of the iodine entering the module with the feed be removed by some mechanism.

If the iodine does not accumulate or react to form acids and subsequently salts, the module will perform the same as the pure water feed case.

In the previous sections it was shown that iodine does not form acids or react and therefore remains in solution as elemental iodine. It is therefore necessary to determine if the partial pressure of iodine will be such that it will vaporize at the rate it enters with the feed water.

An important factor in this situation is the state of the iodine-water system. If the iodine exists in a homogeneous solution it exerts one partial pressure; however, if it exists in a heterogeneous solution it will exert a substantially different partial pressure. Both cases will be examined.

Because iodine is very insoluble it will be necessary to determine if the concentration in the liquid (or solid) phase required to provide this partial pressure is greater than the known solubility for iodine.

This calculation was made by assuming ideal solutions (both phases), local conditions of $32^{o}F$ in the porous plate and a local total pressure equal to the vapor pressure of water. This implies that the vapor is formed in the interior of the porous plate at a depth such that the pressure drop (due to gas flow) is equal to the difference between the vapor pressure and the steam jacket pressure. The result of this calculation yields a required concentration of iodine equal to 1.54×10^{-3} gm iodine/gm solution. The literature value of iodine solubility at this temperature is 1.4×10^{-4} gm/gm solution. Thus, it is certain that iodine will accumulate in the porous metal and will crystallize out of solution.

The question which must now be answered is whether or not the amount of solid iodine formed will be detrimental to the operation of the module. The following operating conditions were employed: 1) a basis of 100 hours of operation, 2) 10% vaporization factor for iodine (90% to crystal), 3) feed rates of 0.30 to 0.70 lbs/hr, 4) feed water containing 1.0 to 10 ppm iodine, 5) a porous plate unit 0.024 inches thick

having 25 square inches of superficial surface area. A graph has been constructed to indicate the percent of the pore volume occupied by the iodine crystal. This is presented in Figure 5. As these calculations are based upon an inlet feed water concentration of 10 ppm iodine as a maximum; any reduction below this level will result in an equal reduction in residual solid iodine. This reduction will continue until a feed water iodine concentration of 0.91 ppm is reached. At this level all incoming iodine will be vaporized and no residual solid iodine will be present in the porous metal. However, this approach (homogenous case) has ignored the fact that any iodine solid surface in contact with the gas phase will be generating iodine (vapor) at its vapor pressure (Figure 6) and thus aid in reducing the amount of residual solid iodine.

Examination of the heterogeneous case leads to the result that the equilibrium partial pressure of iodine in the vapor phase at 32°F is equal to its vapor pressure (.0299 mmHg). This situation results from the vapor pressure property of immiscrible mixtures - each exerts a partial pressure equal to its vapor pressure and the total pressure is the sum of the vapor pressures. At 32°F, this yields a possible gas phase iodine concentration of 8900 ppm iodine. This is much greater than the required 10 ppm and as a result there will not be any difficulty in removing iodine entering in the feed water at any concentration up to the level dictated by saturation data.

Although the permissible level is sixteen times the 10 ppm specified, this favorable situation will exist only if the iodine crystallizes in the porous plate and the resulting crystal surface is exposed to the gas phase. This is certain to occur during normal operation of the module although the effect will be cyclic in nature. A buildup of iodine crystals will be followed by rapid vaporization until all free iodine is exhausted. Any wetting of the surface by water will change the mechanism to that of a homogeneous solution. As soom as the iodine crystals are exposed the system will again behave according to

the heterogeneous mechanism.

9. Conclusions

In summary, the following conclusions have been reached as a result of this study:

(A) The phenomenon called "breakthrough" is the result of vapor pressure and freezing point depression caused by an accumulation of the salt $NiCl_2$. This salt is produced when chlorinated water is exposed to the highly divided nickel surface in the sublimator plate, particularly in the presence of nickel oxide (which is present intiially and also may be formed by oxidation due to the nascent oxygen formed by decomposition of HOCl).

(B) If iodine is used as a bactericide there will not be any reaction between the iodine and the nickel. Therefore there cannot be an accumulation of soluble salts to cause the kind of undesirable effects observed with chlorinated feed water.

(C) The exact mechanism for removal of the iodine from the porous plate cannot be determined. It is probable that the iodine removal rate will vary with time, resulting in a variable concentration of iodine both in the porous plate and in the effluent vapor (this effect is not to be confused with the cyclic breakthrough phenomena associated with the chlorinated feed water).

Whether the iodine removal is cyclic or uniform however, is irrelevant. In either case the rate of iodine removal will be adequate to prevent any long-range accumulation, and short-term cyclic accumulations probably will not have a period or amplitude great enough to be detrimental to the operation of the sublimator.

(D) Based on B and C above, it is concluded that the system should operate on iodated feed water in the same manner as it does with pure feed water, and that the presence of up to 10 ppm of iodine in the feed water should cause no appreciable degradation of performance.

10. Recommendations

Since the mechanism controlling the rate of removal of iodine is very dependent upon the physical configuration and operating conditions, it is recommended for safety consideration that empirical physical tests of the iodine system be conducted. Adequate reliable data should be collected to make an accurate quantitative analysis of the system to determine whether the Iodine will accumulate under actual flight conditions during the time period anticipated for the flight duration.

It is possible to do this with a small test module in a manner similar to the tests previously conducted, although the data collected should include more exact pressure control, feed water analysis, etc., and must also include means to monitor continously (or at frequent intervals) the Iodine content of the effluent water vapor.

References

(1) E. Chirnoaga, J. C. S., 1693, (1926).

(2) N. V. Sidgwick, The Chemical Elements and Their Compounds, p. 1433, Oxford, The Clarendon Press, (1950).

(3) J. M. Smith and H. C. Van Ness, Introduction to Chemical Engineering Thermodynamics, 2nd ed. McGraw-Hill Book Company, Inc., p. 434, (1959).

(4) Sidgwick, ibid, p. 1213.

(5) Ind. Eng. Chem. 33, 741, (1941).

(6) L. Pauling, General Chemistry, 2nd ed., W. H. Freeman & Co.

(7) W. C. Bray and E. L. Connally, J.A.C.S., 33, 1485, (1911).

(8) A. Skrabal, Ber., 75, 1570, (1942).

(9) C.H.L. and C. F. White, J.A.C.S., 65, 335, (1943).

(10) Sidgwick, ibid, p. 1229.

(11) Sidgwick, ibid, p. 1146.

(12) Sidgwick, ibid, p. 1433.

(13) McKaes and Worthington, Corrosion Resistance of Metals and Alloys, ACS Monograph Series, #71, page 359.

(14) Friend, Trans. AIChE, 39, 731 (1943).

APPENDIX

EXPERIMENTAL WORK/GREEN FILM

In order to verify the observations (contractor) concerning the green film and ice formed in the preliminary test, a simple experiment was carried out. This experimental work consisted of preparing aqueous solutions of nickel chloride $NiCl_2$ in the concentration range0.76% by weight to 21.1%. In order to approximate the conditions believed to exist on the module surface, thin films of these solutions were prepared. These films were placed against a surface similar to the porous nickel reportedly used in the module.

From these tests, it was concluded that a $NiCl_2$ concentration between 10 and 20 percent for films .002" to .005% thick must exist if the green color is to be readily seen. Subsequently the films were frozen and the observation repeated with the same conclusions.

It should be pointed out, that the ability to "see" the green color associated with the nickel chloride is very dependent upon the background material (1% is readily observed against a diffuse white surface), and the angle and intensity of incident light.

KEUFFEL & ESSER CO. MADE IN U.S.A.
3 X 5 CYCLES

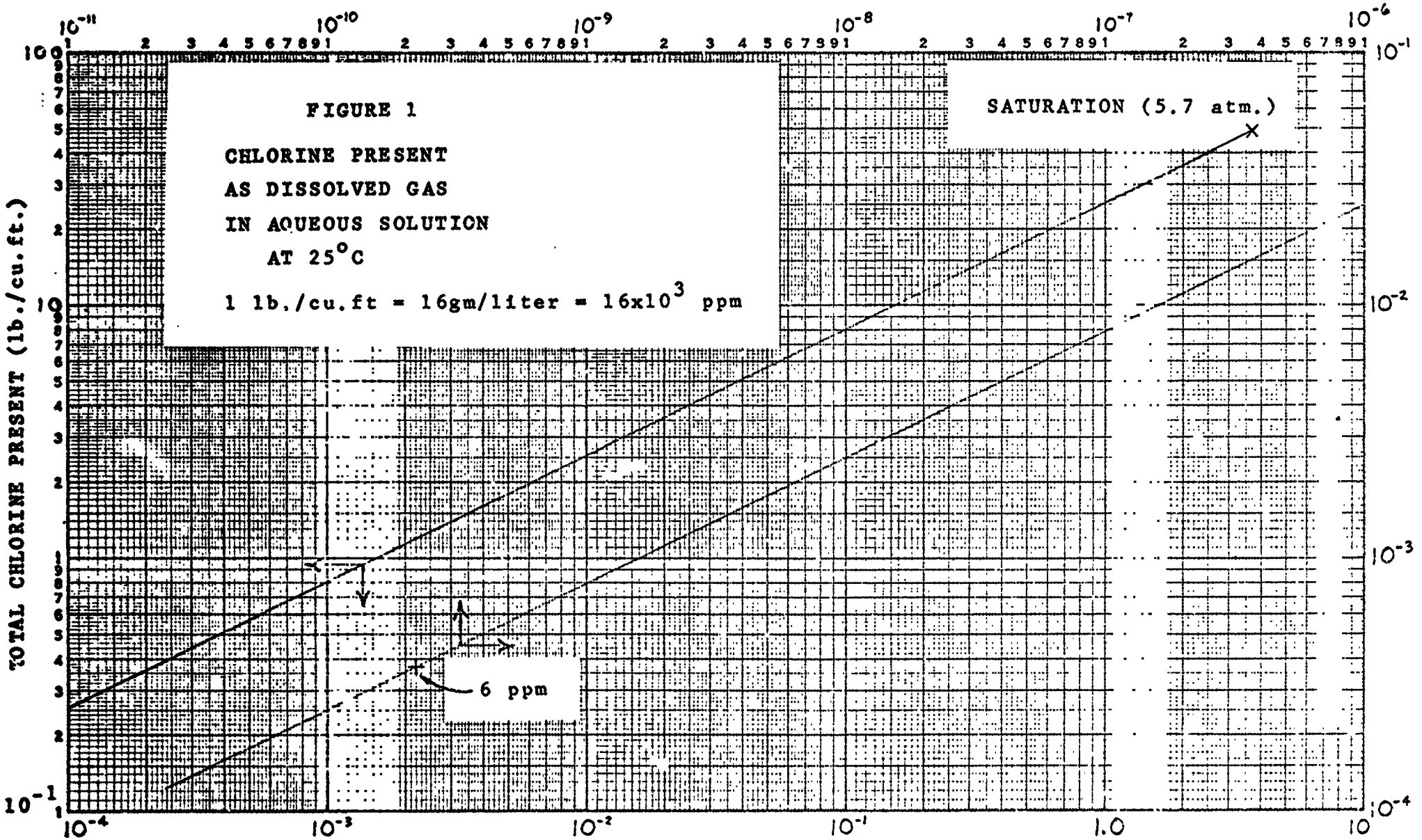


FIGURE 1

FIGURE 2

$NiCl_2$ - H_2O System

VAPOR PRESSURE

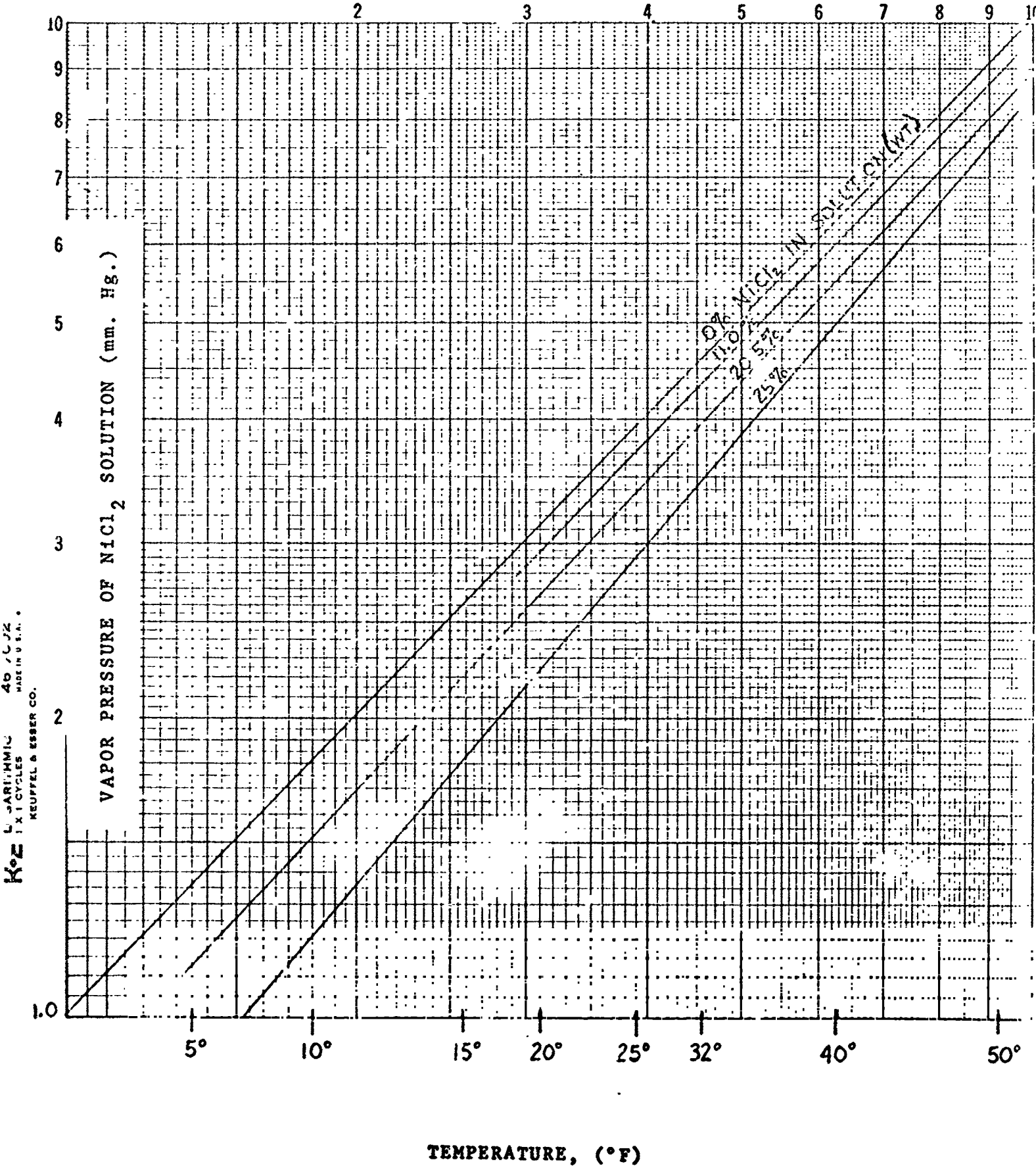
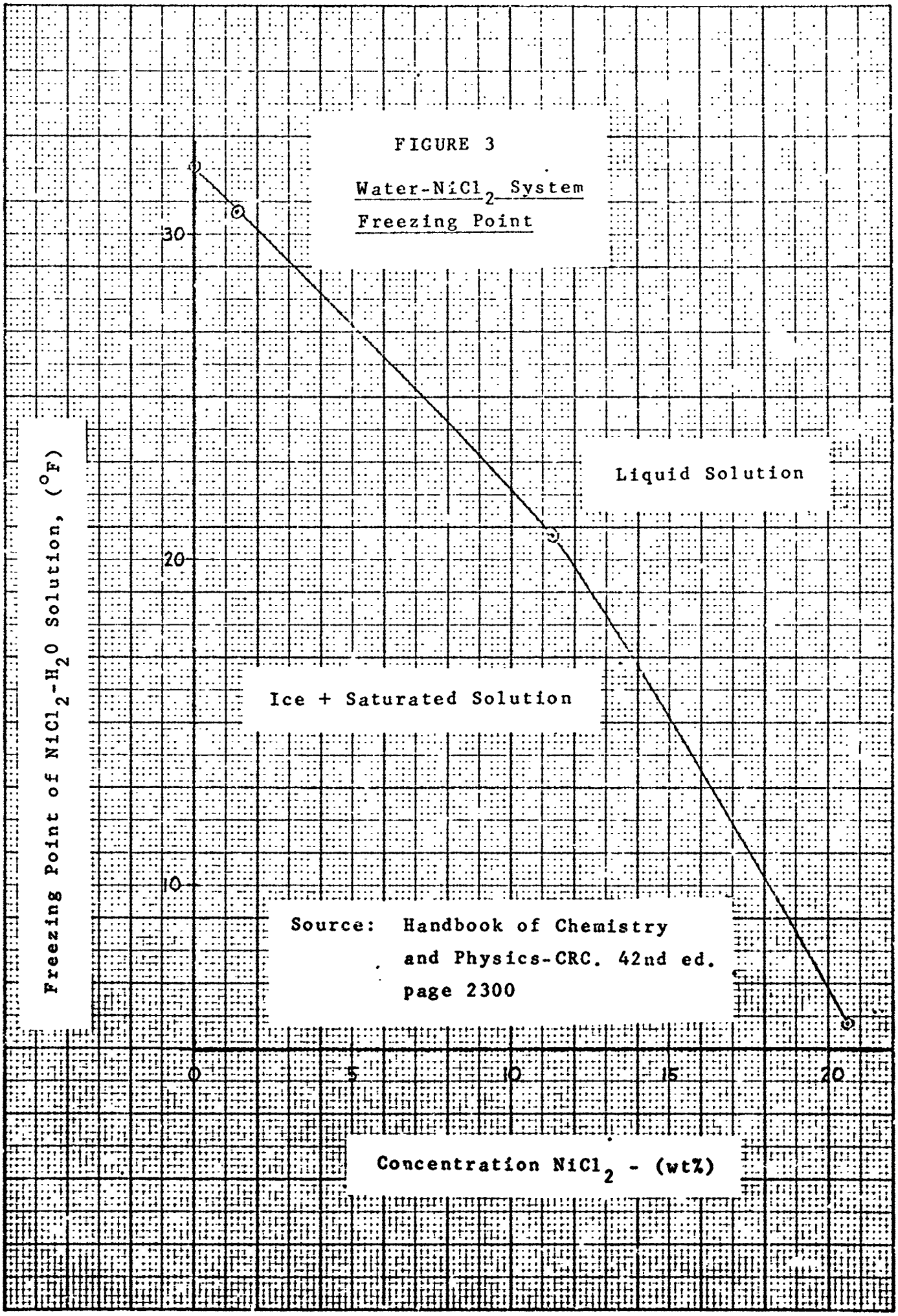


FIGURE 3

Water-$NiCl_2$ System

Freezing Point



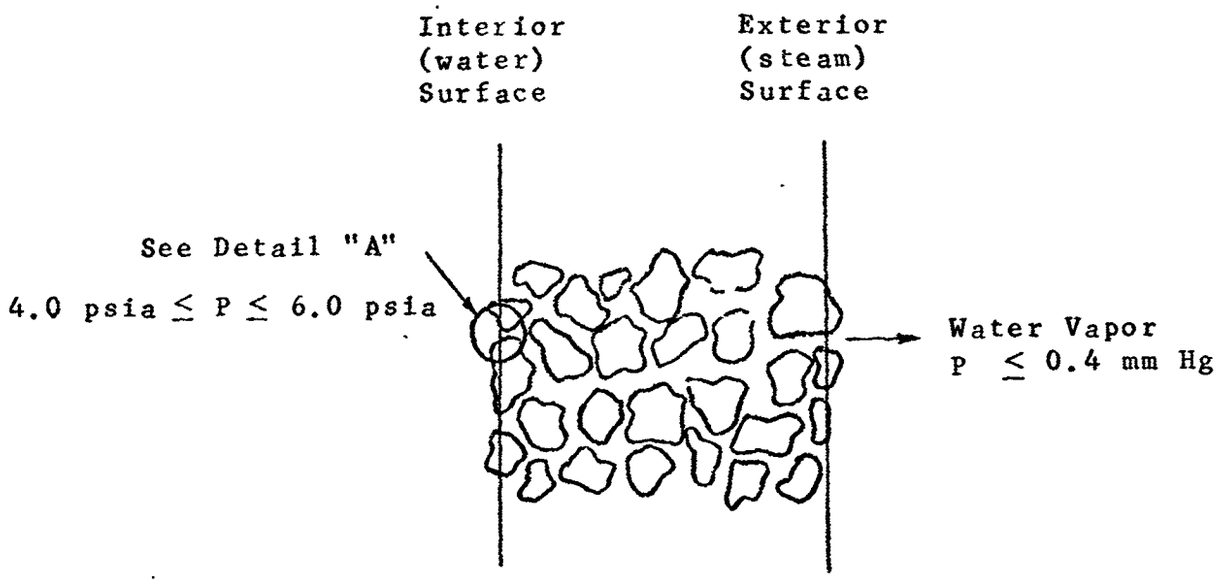
Source: Handbook of Chemistry and Physics-CRC. 42nd ed. page 2300

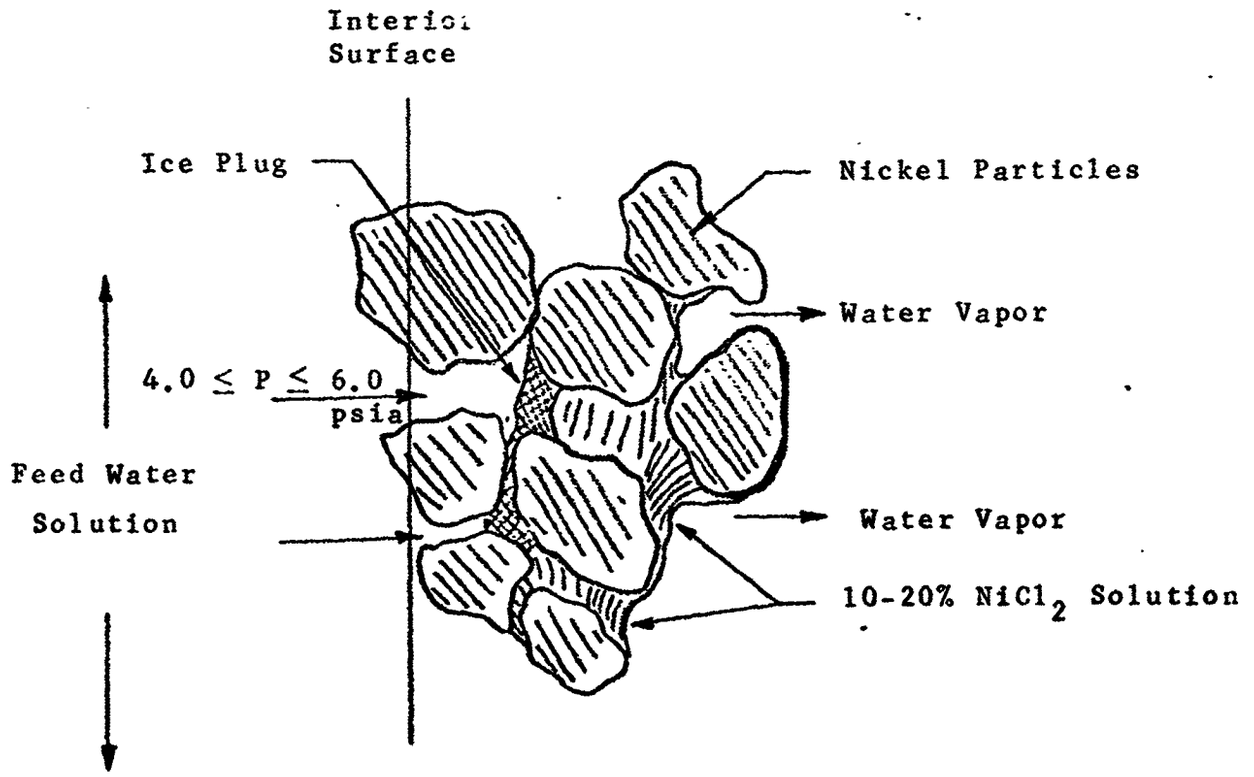
K+E 20 X 20 TO THE INCH 46 1242
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.

FIGURE 4

Idealized Cross-Section

Porous Plate





DETAIL "A"

FIGURE 5

Solid Iodine Deposit

25 in^2 x 0.024" porous plate

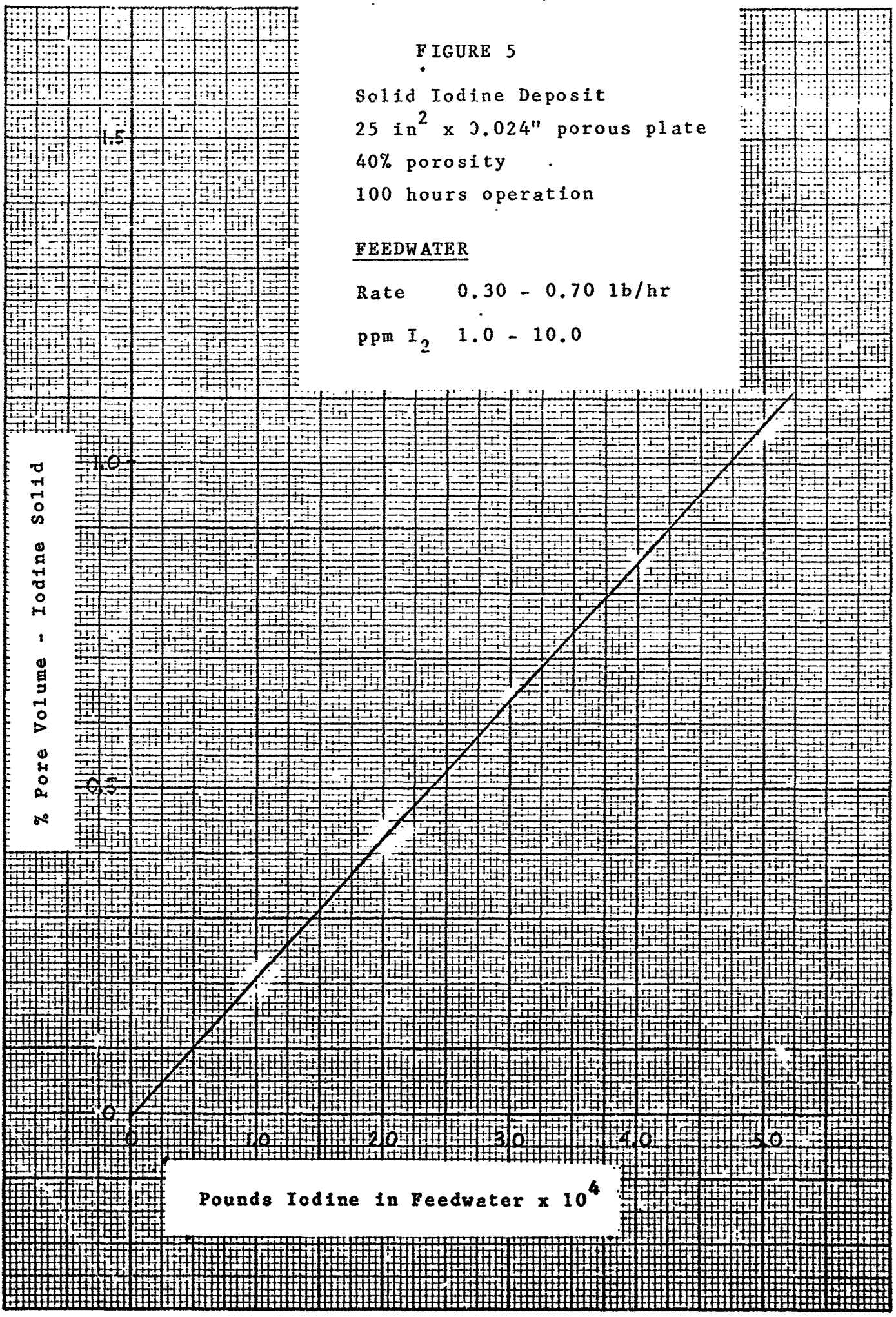
40% porosity

100 hours operation

FEEDWATER

Rate 0.30 - 0.70 lb/hr

ppm I_2 1.0 - 10.0

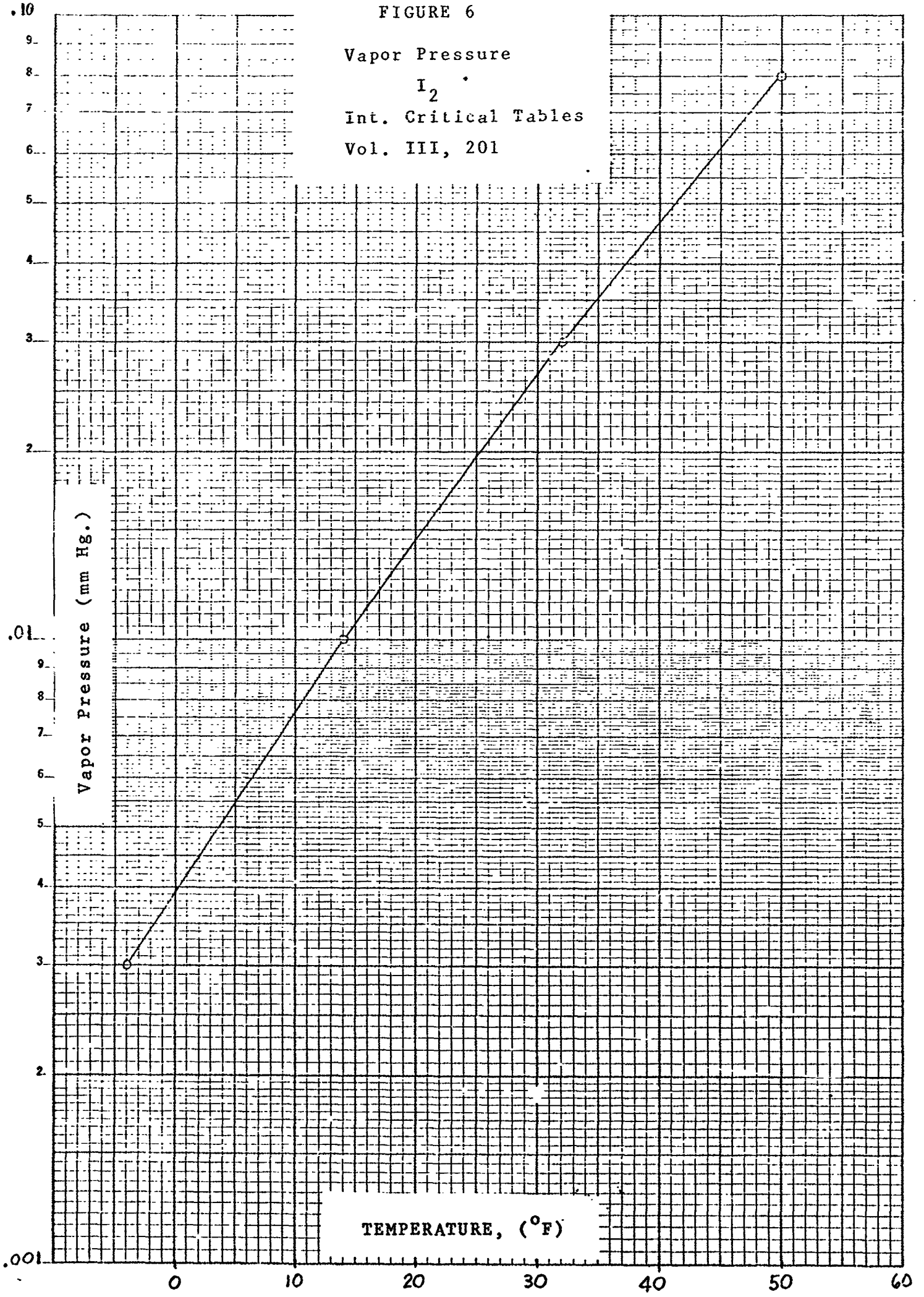


K E 2 7 X 10 INCHES .01 F .H MADE IN U.S.A. KEUFFEL & ESSER CO.

FIGURE 6

Vapor Pressure

I_2

Int. Critical Tables

Vol. III, 201