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A THEORETICAL TREATMENT OF MIXED SMOKES AS ICE NUCLEI

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ABSTRACT

A proposed nucleation mechanism is described for mixed smokes composed of potassium iodide and silver iodide.

The dynamic dissolution of such smoke particles is considered to be theoretically of significance in the nucleation mechanism of supercooled fogs of water droplets.

TABLE OF CONTENTS

page

I. INTRODUCTION 1

II. BACKGROUND 2

III. POSSIBLE NUCLEATION MECHANISMS 4
A. Condensation-freezing. 4
B. Direct Contact Freezing. 5
C. Sublimation. 7
D. Summary. 7

IV. THE DYNAMIC DISSOLUTION PROCESS. 8
A. General Description. 8
B. Heat and Mass Flow 9
C. Temperature-Concentration-Time Interactions. 12
D. Silver Iodide Precipitation. 15

V. THE SURFACE PARAMETER, m 15

VI. CONCLUSIONS. 17

VII. ACKNOWLEDGMENTS. 19

TABLE I. 20
FIGURE 1 21
FIGURE 2 22
FIGURE 3 23
FIGURE 4 24
FIGURE 5 25
FIGURE 6 26
FIGURE 7 27
FIGURE 8 28

BIBLIOGRAPHY 29

I. Introduction

Early in May 1967, the Research and Development Department, Naval Ammunition Depot, Crane, Indiana, was introduced to an application of pyrotechnics which differed grossly from normal uses.

The Naval Weapons Center (NWC), China Lake, California, requested the assistance of this Department in the development of a pyrotechnic nuclei generator to be utilized in atmospheric research projects such as Project Stormfury.

After a brief educational period, predominant processes, involved in the conversion of supercooled water droplets to ice crystals, were studied and there appeared to be an area of investigation which demanded further study.

One of the pyrotechnic formulations developed during this program yielded abnormally high nucleation efficiencies at supercoolings of approximately six degrees Centigrade when the pyrotechnically generated smoke was evaluated in a cloud chamber.

It was felt that the nucleating mechanism of this smoke was probably different from that of previous pyrotechnic smokes utilized and warranted study.

II. Background

During the educational process, the nucleation characteristics of a previously developed NWC pyrotechnic formulation was investigated. This formulation, designated as TB-2, yielded nucleation results as illustrated by Figure 1.

Estimations of the flame products for this formulation were made assuming equilibrium was reached in the flame at a temperature of 2000°C and one atmosphere pressure. Table 1 lists the results of these calculations. The reaction which was considered is illustrated below.

$$AgI_{(g)} + K_{(g)} \rightleftharpoons Ag_{(g)} + KI_{(g)}$$

If it is assumed that equilibrium is reached in the flame of the pyrotechnic, then it becomes obvious that the nucleating efficiencies of the silver iodide present is approximately twelve times greater than the values listed in Figure 1 where all of the silver present was assumed to be in the form of silver iodide. When the equilibrium values of silver iodide are used with the nucleation efficiency points of Figure 1 and a second efficiency curve is plotted, the results indicate that all test points lie above Fletcher's theoretical sublimation curve[1] for silver iodide. (See Figure 2.)

This information, assuming the cloud chamber tests to be valid, indicated that a nucleating process was occurring which was significantly more efficient than that expected for silver iodide acting as a sublimation nuclei. The nucleation efficiency at -5°C is definitely indicative of processes other than sublimation, and the other two points may likewise be assumed to be acting by other processes also.

Another pyrotechnic formulation, TB-7, was developed at the request of Dr. F. K. Odencrantz of NWC. During some investigations which were conducted in a cloud chamber, he determined that a mixed smoke composed of a 3:1 mole ratio of potassium iodide to silver iodide produced a very efficient nucleant. The TB-7 formulation was formulated to theoretically produce the desired 3:1 mole ratio product. The smoke from this formulation produced an efficiency which is in between the efficiency values for the TB-2 formulation (see Figure 3).

The final smoke products from either formulation produces particles which should be hygroscopic due to the presence of potassium iodide and other potassium by-products, such as, potassium oxide. The basic literature revealed very little information regarding nucleation mechanisms involving mixed smokes. The effect

of soluble salts admixed or complexed with insoluble nucleants is admittedly not known.[2,3,4] In fact, one authority in this area has chosen to assume that mixed smokes of silver iodide behave simply as silver iodide sublimation nuclei.[5]

It is the purpose of this report to present (1) a proposed mechanism which could explain the nucleation efficiencies observed and (2) a mechanism by which the action of mixed smokes may be better understood.

III. Possible Nucleation Mechanisms

A. Condensation-freezing

Assuming that the mixed smoke produced by these pyrotechnic formulations contains a complex salt of potassium iodide and silver iodide, e.g. AgI · 3KI, AgI · 2KI or AgI · KI, it can be assumed that the smoke particles produced would be hygroscopic. From work of Tompkins, Muus and Pearson[6], it is obvious that appreciable quantities of water would condense on these complex salts at normal saturation ratios existent in clouds.

Solubility data drawn from the International Critical Tables for the KI-AgI-H_2O system indicate, as illustrated by Figure 4, that silver iodide would be precipitated if the double salt (AgI · 3KI) concentration was greater than about 73 percent by weight or

less than about 45 percent by weight. As illustrated, the double salt would be completely soluble between these limits at the prescribed temperature.

The smoke products from pyrotechnics will normally have particle sizes around 0.01 to 0.1 micron, the exact size being a function of properties of the specific products formed and the properties of the flame.

For particle sizes of this range, condensation can occur depending on the specific properties of the particle. As pointed out, the double salt would be subject to condensation due to the hygroscopic nature of the salt. Once the particle was coated with a water layer, droplet growth would be expected. Realizing that dissolution of the double salt would result in precipitation of silver iodide within the droplet, it would be expected that freezing of the droplet could occur under certain conditions. Such a mechanism of condensation-freezing has been proposed by Vonnegut.[7]

B. Direct Contact Freezing

Direct contact freezing is defined as a freezing process initiated by the intimate contact of a nucleant and a supercooled water droplet. In theory, this process occurs by the Brownian motion of small particles.

If, for some reason, the water droplets are electrically charged and the nucleant particles are oppositely charged, a very high collision rate or contact rate would be expected. However, without both particles being charged in this manner, the direct contact process of nucleation would be expected to be insignificant in droplet fogs and seeding densities normally considered.

Some experimental basis for the direct contact freezing process exists. Maybank and Barthakur[8] used fog droplets labeled with uranyl nitrate and seeded the radioactive fog with an organic nucleant, leucine. The ice crystals formed were collected and analyzed. At temperatures warmer than -20°C, almost all ice crystals were radioactive. If a transfer of water molecules had occurred via sublimation, the ice crystals would have ideally no radioactivity. This experiment strongly suggests the predomination of a direct contact process. It should be realized that the leucine molecule is readily polarized and space charges on a leucine crystal may be induced by presence of other charged particles, e.g. charged water droplets. The ability to become polarized and slightly charged may account for the results of this experiment.

The smoke products of pyrotechnics and other combustion processes can be produced with an electrical charge. For example, the magnesium oxide aerosol produced by the combustion of

magnesium ribbon is reported to form 0.8-1.5 micron particles with 44 percent of the particles charged positively, 42 percent negatively and 14 percent neutrally.[9]

Thus, it would appear that the mixed smoke particles produced by the two pyrotechnic formulations could be charged and the direct contact process would be significantly encouraged.

C. Sublimation

The ability of a water soluble material to produce ice formation via sublimation has been demonstrated. Hosler and Spalding[10] demonstrated that a very soluble salt, potassium iodide, was able to promote ice formation in supercooled fogs at temperatures of -22.9°C to -26.5°C depending on the particle size of the potassium iodide. Ammonium iodide was reported to have induced ice formation at temperatures as warm as -15°C.

The temperature at which a double salt particle of potassium iodide and silver iodide might induce sublimation is presently not known, however, it would be doubtful that the double salt would act as sublimation nuclei at warmer temperatures of approximately -6°C.

D. Summary

Of the three nucleation mechanisms discussed, it would appear that the condensation-freezing and the direct contact freezing

processes probably predominate in the production of ice crystals when a double salt nucleant is utilized.

For the purposes of this report, it is not immediately important to determine which of these two processes predominate.

Therefore, it will be assumed that either the direct contact process or the condensation-freezing process is predominating during further discussions in this report.

IV. The Dynamic Dissolution Process

A. General Description

To better understand the mechanism which might be occurring in nucleation processes involving mixed smokes, i.e. a double salt in this instance, it is necessary to investigate the dynamic dissolution process and the energy transfer which occurs.

If a planar solid-solution interface is considered, as illustrated in Figure 5, the ions from the dissolving solid would be seen to be most concentrated next to the interface and less concentrated away from the interface. At the immediate surface of the solid, the solution would have a concentration equal to the concentration of a saturated solution of the solid soluble material. Due to diffusion processes and concentration gradients within the solution, the ions would diffuse towards less concentrated areas while solid material was dissolved at the solid-solution interface.

Depending on the heat of solution of the dissolving solid material, a change in temperatures within this system would be expected.

As the double salt begins to dissolve, precipitation of silver iodide at some critical level of concentration would be initiated and would continue along with the diffusion process.

B. Heat and Mass Flow

To understand the potential significance of the double salt-dissolution process, it is necessary to establish the energy and mass transfer processes occurring in this type of system.

The mass flow rate, R, in units of mass per time, is given by[11]

$$R = -DA \frac{dc}{dx} \qquad \text{(EQ. 1)}$$

where D is the mass diffusion coefficient, A is the area of mass transfer, C is the concentration in units of mass per volume and X is distance for a diffusion controlled dissolution process. If a planar solid-solution interface is considered, as illustrated in Figure 5, the concentration of the solution at the interface is C_s, the concentration of a solution saturated with the given soluble material.

Since this diffusion equation has the same form as the heat conduction equation

$$q = -KA \frac{dT}{dx} \qquad \text{(EQ. 2)}$$

and the diffusion equation, Fick's "Second Low of Diffusion",

$$\frac{\partial c}{\partial t} = D\left(\frac{\partial^2 c}{\partial x^2}\right) \qquad \text{(EQ. 3)}$$

has the same form as the basic heat flow equation,

$$\frac{\partial T}{\partial t} = K\left(\frac{\partial^2 T}{\partial x^2}\right) \qquad \text{(EQ. 4)}$$

the mathematical solutions of these mass diffusion equations are all identical to the solutions which have been developed for the heat equations.[12]

In the above equations, t is time, q is rate of heat flow, K is thermal conductivity and T is temperature.

From Equation 4, it can be shown that[13,14]

$$\frac{dT}{dx} = \frac{(T_a - T)}{\sqrt{\pi \alpha t}} \cdot e^{-\frac{x^2}{4\alpha t}} \qquad \text{(EQ. 5)}$$

is a solution which has the limitations that t>0 and 0<X>L where L is the length of the conducting medium, X is the thermal diffusivity, T_a is the initial temperature of the medium and T is the temperature at the distance, X, and time, t for the system to be described.

Combining Equations 2 and 5 yields Equation 6.

$$q = \frac{KA(T - T_a)}{\sqrt{\pi \alpha t}} \cdot e^{-\frac{x^2}{4\alpha t}} \qquad \text{(EQ. 6)}$$

Therefore, treating Equations 1 and 3 in a similar manner, Equation 7 results.

$$R = A(C_s - C_a)\left(\frac{D}{\pi t}\right)^{0.5} \cdot e^{-\frac{x^2}{4\alpha t}} \qquad \text{(EQ. 7)}$$

It is assumed that the temperature, T, is different than the ambient or initial temperature, T_a, of the solution due to the

heat of solution, ΔH_s, of the soluble solid material. As the dissolution process occurs, a cooling or heating effect should occur, dependent on the value of the heat of solution, ΔH_s.

Then by definition, the rate of heat flow, q, is equal to the product of the rate of mass flow, R, and the heat of solution, ΔH_s.

$$q = R\Delta H_s \qquad \text{(EQ. 8)}$$

It is assumed that the heat flow into the solid crystalline material is neglible under the conditions to be considered.

Substituting for q and R from Equations 6 and 7 and solving for the temperature difference, $(T-T_a)$, yields the following equation,

$$(T-T_a) = \left[\frac{(C_s - C_a)(\alpha D)^{0.5}\Delta H_s}{K}\right] \cdot e^{-\frac{x^2}{4\phi t}} \qquad \text{(EQ. 9)}$$

where

$$\phi = \frac{D}{\alpha - D} \qquad \text{(EQ. 10)}$$

This equation may be utilized to calculate the temperature difference, (T_s-T_a), by letting X and t approach zero. This temperature difference is that which would be expected to be present in a dynamic situation at the instant of soluble particle-water droplet contact.

It should be noted that this temperature difference is not nearly as large as would be calculated for the solution temperature change observed under completely adiabatic dissolution of a soluble

material to form a saturated solution,

$$\Delta T = \frac{C_s \Delta H_s}{C_{ps}} \qquad \text{(EQ. 11)}$$

where C_s is the concentration of solute in the solution (mass of solute per mass of solution), ΔH_s is the heat of solution and C_{ps} is the specific heat of the solution.

If these equations for mass flow and heat flow are examined, it is seen that extremely high rates of heat and mass flows occur at the instant of water contact. The high rate of heat flow is responsible for significant temperature changes within the system described. For a material such as silver iodide which is highly insoluble, it is obvious that a very small temperature difference, rate of heat flow and rate of mass flow would exist.

C. Temperature-Concentration-Time Interactions

The integration of Equations 3 and 4 yield the two following equations, respectively.

$$\frac{T - T_s}{T_o - T_s} = \mathrm{erf} = f\left(\frac{x}{2\sqrt{\alpha t}}\right) \qquad \text{(EQ. 12)}$$

$$\frac{C - C_s}{C_o - C_s} = \mathrm{erf} = f\left(\frac{x}{2\sqrt{D t}}\right) \qquad \text{(EQ. 13)}$$

In these equations, $f\left(\frac{x}{2\sqrt{\alpha t}}\right)$ and $f\left(\frac{x}{2\sqrt{D t}}\right)$ are functions which are called Gauss's error integral. Tables are available[15] for evaluation of these functions for given values of x, t and the diffusivity terms, x and D.

By substituting for values of the above variables, the temperature, T, at a given distance, X, and time, t, may be calculated, as likewise may be the concentration, C.

In order to establish if the double salt concentration would be relatively high when significant temperature changes occurred, calculations utilizing Equations 12 and 13 were performed.

A search of the literature failed to reveal values for α and D. However, calculations were possible utilizing values for potassium iodide solutions. It is realized that neither α or D are constant when the temperature or concentration is changed, but it is felt that the values utilized are adequately correct to demonstrate the magnitude of changes in the temperature-concentration-time-distance system.

Using values of $\alpha = 1.35 \times 10^{-3}$ cm^2/second, $D = 1.40 \times 10^{-5}$ cm^2/second, $C_s = 0.56$, $Ca = 0.00$, $k = 1.35 \times 10^{-3}$ calories/cm. deg. second and $\Delta H_s = 28.9$ calories/gram, various calculations were performed utilizing Equations 9, 12 and 13.

By letting x approach zero in Equation 9, the value for the temperature, T_s, at the solid-solution interface was estimated to be 2.8 degrees centigrade lower than the ambient temperature (or initial water temperature).

Assuming an ambient temperature, T_a, of -6.0°C, the interface temperature, T_s, would be -8.8°C.

Using these values for T_s and T_a, calculations were performed utilizing Equation 12 to determine at what points in time and distance a temperature, T, in the solution would exist where T = -6.5°C. These values are plotted in Figure 6.

Inspecting this graphical information leads to the conclusion that a 0.5 degree undercooling by a dissolution process would occur quite rapidly - less than a millisecond - at distances of ten microns away from the solid-solution interface.

Calculations were similarly made for the concentration, C, and it was seen that the concentration was effectively zero with the distances and times considered in Figure 6.

To better evaluate the variables of temperature and concentration versus distance, values were calculated for a fixed time of $t = 2.05 \times 10^{-6}$ seconds using Equations 12 and 13. Figure 7 demonstrates quite vividly that undercoolings of over two degrees could be realized by a dissolution process.

It is likewise observed that the freezing point of the solution at undercoolings of approximately two degrees or less would hardly be affected by the salt concentrations present.

D. Silver Iodide Precipitation

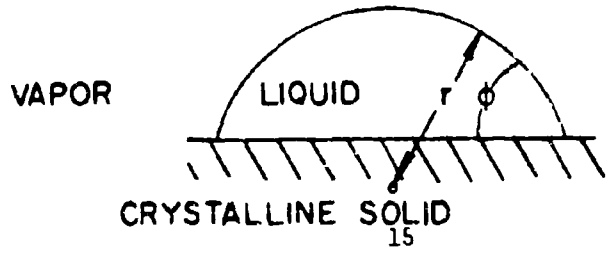
For the double salt, AgI · 3KI, it would be predicted that silver iodide would be precipitated continually near the leading edge of the diffusing ionic species. Later in time, as the concentration of the potassium and iodide ions increased, the precipitated silver iodide would be redissolved and transported by the diffusion process towards lower concentration areas where precipitation would once again occur.

It is postulated that the freshly precipitated silver iodide would have a very high concentration of crystalline imperfections. The imperfections could serve as potential nucleating points in this system.

V. The Surface Parameter, m

In heterogeneous nucleation theories involving condensation or sublimation, the surface parameter, m, is considered to be of utmost importance[16]. The surface parameter, m, is defined by Equation 14 and illustrated below,

$$m = \text{COSINE } \phi = \frac{\sigma_{cv} - \sigma_{cl}}{\sigma_{lv}} \qquad \text{(Eq. 14)}$$



where the subscripts C, V and L connote crystalline solid, vapor and liquid phases of a system and σ is the free surface energy between phases.

Figure 8 illustrates the temperature, T, at which a spherical particle of radius r and surface parameter m will nucleate an ice crystal in one second by sublimation from a water-saturated environment and by freezing from water (direct contact)[17]. This figure illustrates the importance of the surface parameter, m, in both processes. It is obvious that a particle must have a very high value for m, as well as a rather large particle size, before the sublimation process can be of any importance in the warmer temperature zones.

However, it is seen that the contact freezing process is much less subject to these restrictions. Much lower values of m and particle sizes may be tolerated in this process.

In the potassium iodide-silver iodide direct contact nucleation process, it is proposed that the normal m value assumed for silver iodide may not be applicable. The basis for this statement becomes evident if the interfacial free energy terms of Equation 14 are examined.

In the silver iodide precipitation areas, the potassium iodide solution would surely cause a decrease in the crystalline solid-

liquid interfacial free energy term, σ_{CL}, due to the continuous diffusion of ions to and from the silver iodide precipitate. Likewise, a slight increase in the liquid-vapor interfacial free energy term, σ_{LV} would be expected. Without a precise knowledge of these values, it is not known whether the true value of m would be increased or decreased in this situation.

VI. Conclusions

While this report has not proven conclusively the exact nature by which a mixed smoke of potassium iodide and silver iodide affects the formation of ice crystals, enough data is presented to draw some useful conclusions regarding experimental results and probable mechanisms.

The apparently high nucleation efficiencies obtained by the TB-2 and TB-7 pyrotechnic compositions can very probably be explained by a condensation-freezing mechanism or a direct contact mechanism. If the double salt is formed, it is obvious that sublimation could not occur due to the very low m value expected from a soluble salt.

Chemical analysis of the smoke product from the TB-2 composition indicates that approximately 8.35 percent of the silver in the smoke was present as silver iodide. Comparing this data to the flame equilibrium data listed in Table 1, it appears that the

equilibrium values predicted are achieved. From Table 1, it is seen that 8.24 percent of the silver present should be in the form of silver iodide.

Thus, it would appear that if the double salt complex is formed, the nucleation efficiency of the TB-2 composition would be best demonstrated by the values in Figure 2.

It is concluded that the nucleation efficiencies are better than normally observed for silver iodide simply because the proposed mechanism allows smaller smoke particle sizes to be utilized via the direct contact process. (See Figure 8.) A second benefit may be also realized through the undercooling induced by the dissolution process. This undercooling would serve to increase the rate of nucleation as well as lower the Δm value required for a given particle size to cause freezing at a given ambient temperature.

It is likewise concluded that other nucleants can be developed utilizing the proposed dissolution-precipitation technique and these nucleants would not necessarily have to be based upon silver iodide to be useful.

VII. Acknowledgments

Stimulating discussions held during the initial learning period were the basic "nuclei" which caused this report to "precipitate".

The author wishes to express his appreciation for the thought-provoking discussions between himself and Mr. Donald Hazelton, Mr. William Ripley, Mr. Charles Lipscomb and Mr. Harold Benham, all of the Research and Development Department, Naval Ammunition Depot, Crane, Indiana.

Likewise, without the patient encouragement and discussions with Dr. F. Kirk Odencrantz and Dr. Pierre St. Amand of Naval Weapons Center, China Lake, California, the author would have been significantly slower in consolidating the thoughts which went into the report.

TABLE I

COMBUSTION PRODUCTS @ 2000°C

(in moles per 100 grams of composition)

Combustion Products	t=0	t=teq.
Silver iodide	0.085	0.007
Silver	--	0.078
Potassium iodide	--	0.078
Potassium	0.433	0.355

Note: Binder products and other diluent species are not listed.

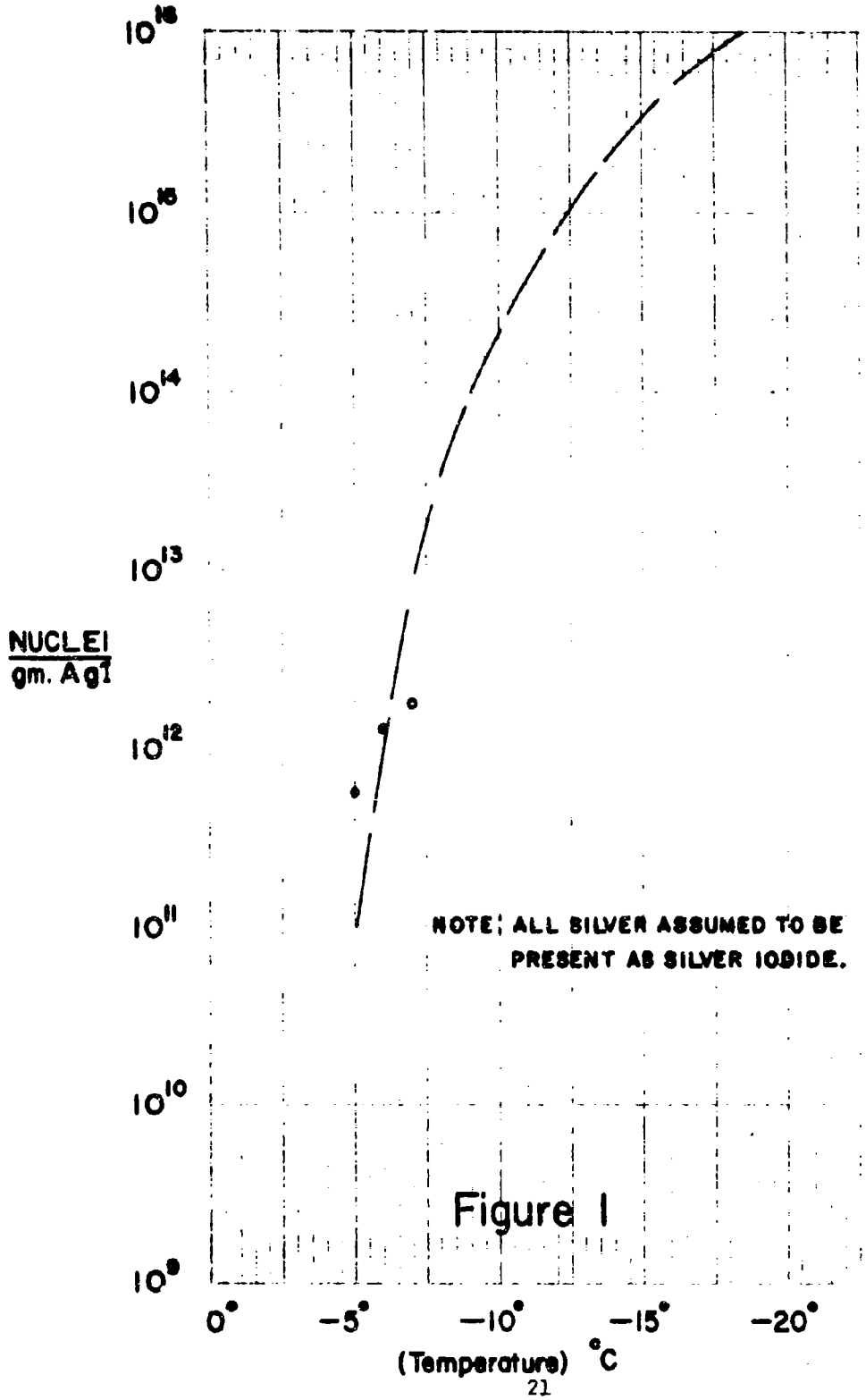

10^{16}
10^{15}
10^{14}
10^{13}
10^{12}
10^{11}
10^{10}
10^{9}
NUCLEI / gm. AgI
NOTE; ALL SILVER ASSUMED TO BE PRESENT AS SILVER IODIDE.
0°
−5°
−10°
−15°
−20°
(Temperature) °C

Figure 1

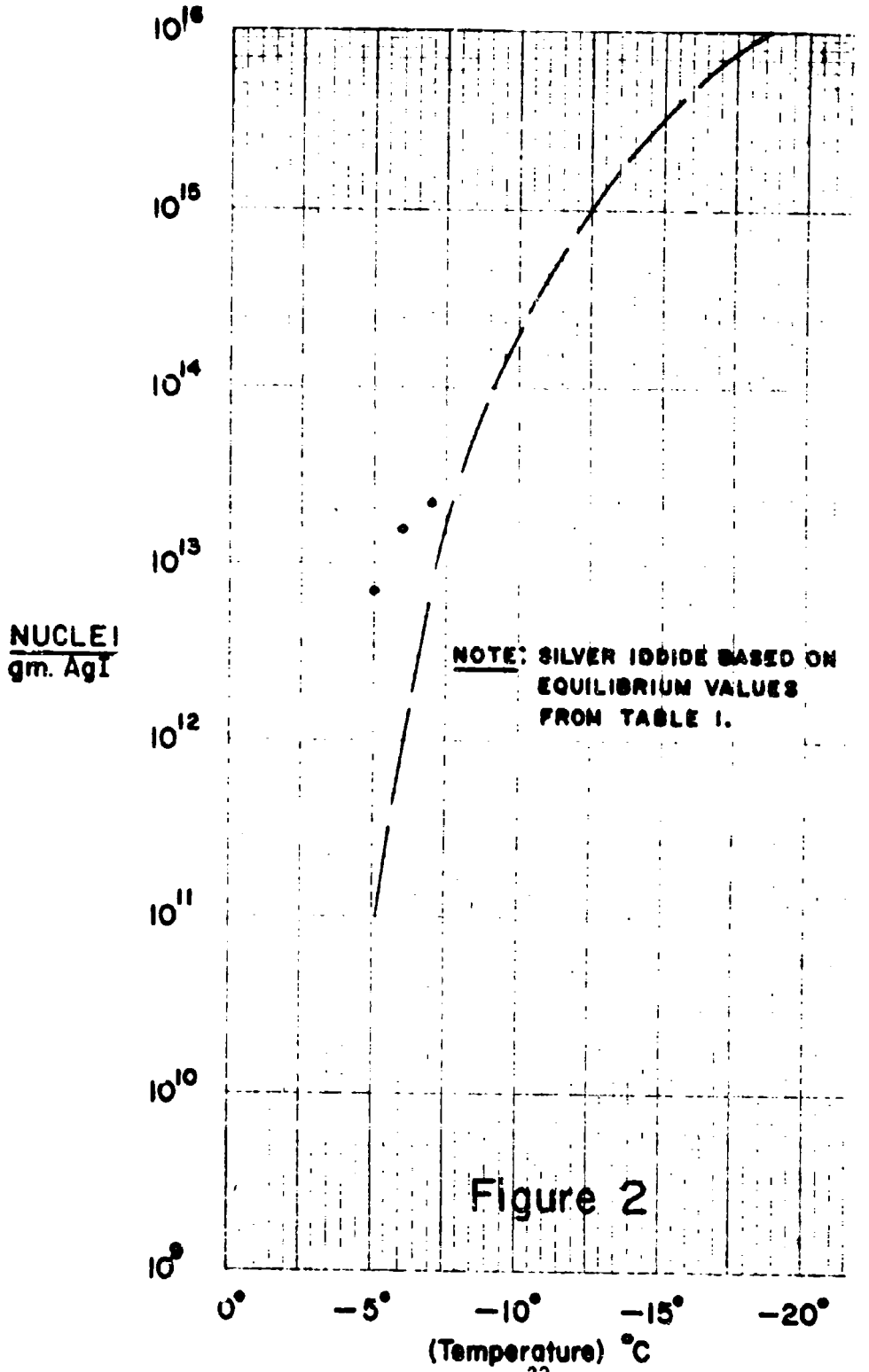

10^{16}
10^{15}
10^{14}
10^{13}
10^{12}
10^{11}
10^{10}
10^{9}
NUCLEI / gm. AgI
NOTE: SILVER IODIDE BASED ON EQUILIBRIUM VALUES FROM TABLE I.
0°
−5°
−10°
−15°
−20°
(Temperature) °C

Figure 2

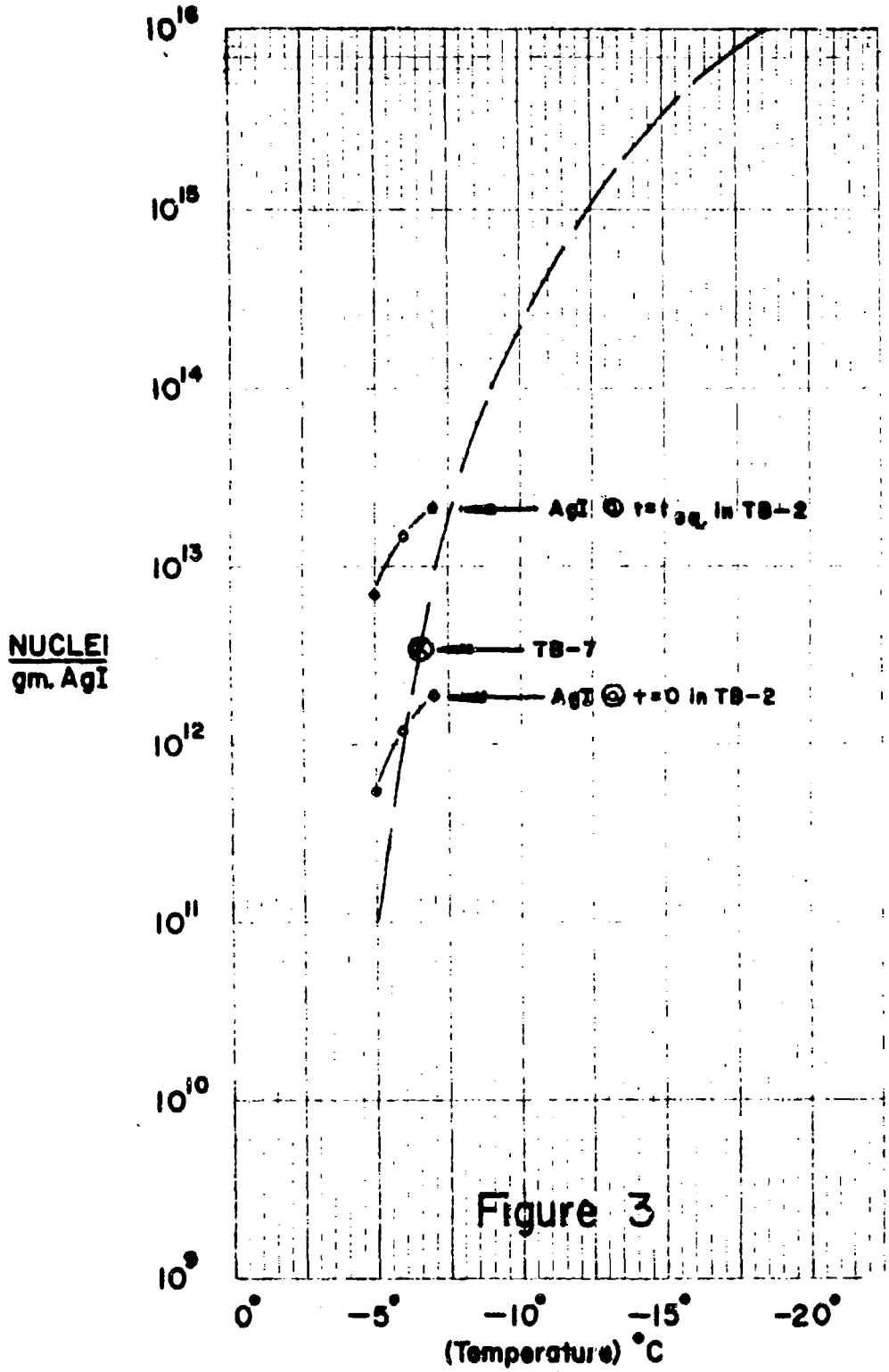

10^{16}
10^{15}
10^{14}
10^{13}
10^{12}
10^{11}
10^{10}
10^{9}
NUCLEI / gm. AgI
AgI @ t=t eq. in TB-2
TB-7
AgI @ t=0 in TB-2
0°
-5°
-10°
-15°
-20°
(Temperature) °C

Figure 3

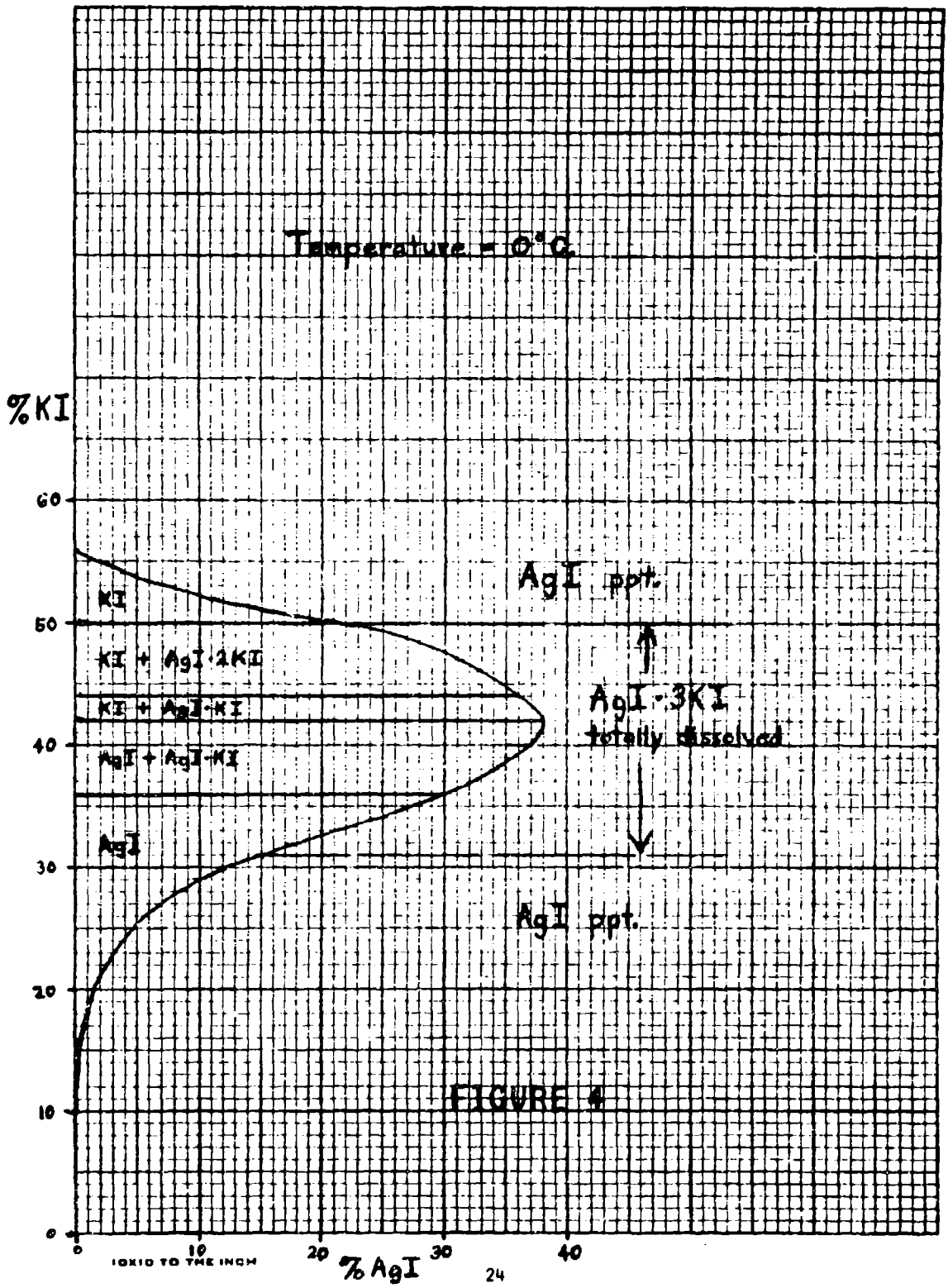

Temperature = 0° C.
% KI
60
50
40
30
20
10
0
KI
KI + AgI·2KI
KI + AgI·KI
AgI + AgI·KI
AgI
AgI ppt.
AgI·3KI
totally dissolved
AgI ppt.
0
10
20
30
40
% AgI
10 X10 TO THE INCH

FIGURE 4

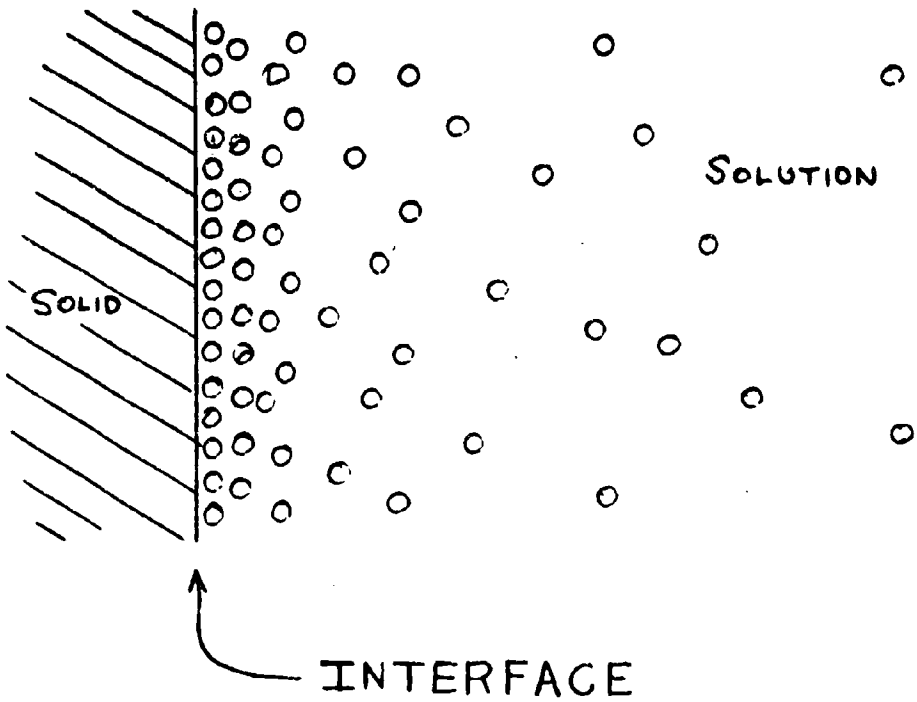


FIGURE 5

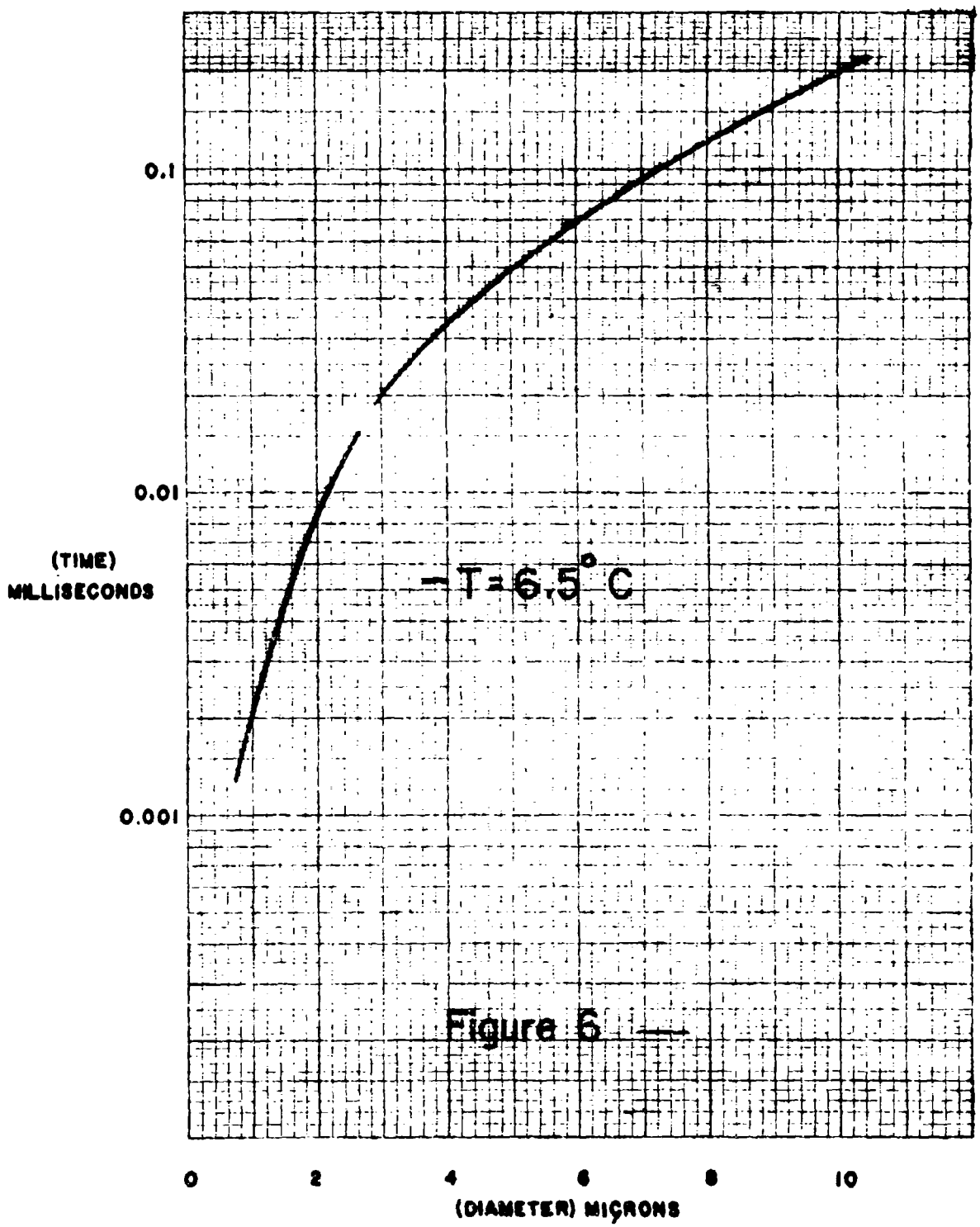

0.1
0.01
0.001
(TIME)
MILLISECONDS
T=6.5° C
0
2
4
6
8
10
(DIAMETER) MICRONS

Figure 6

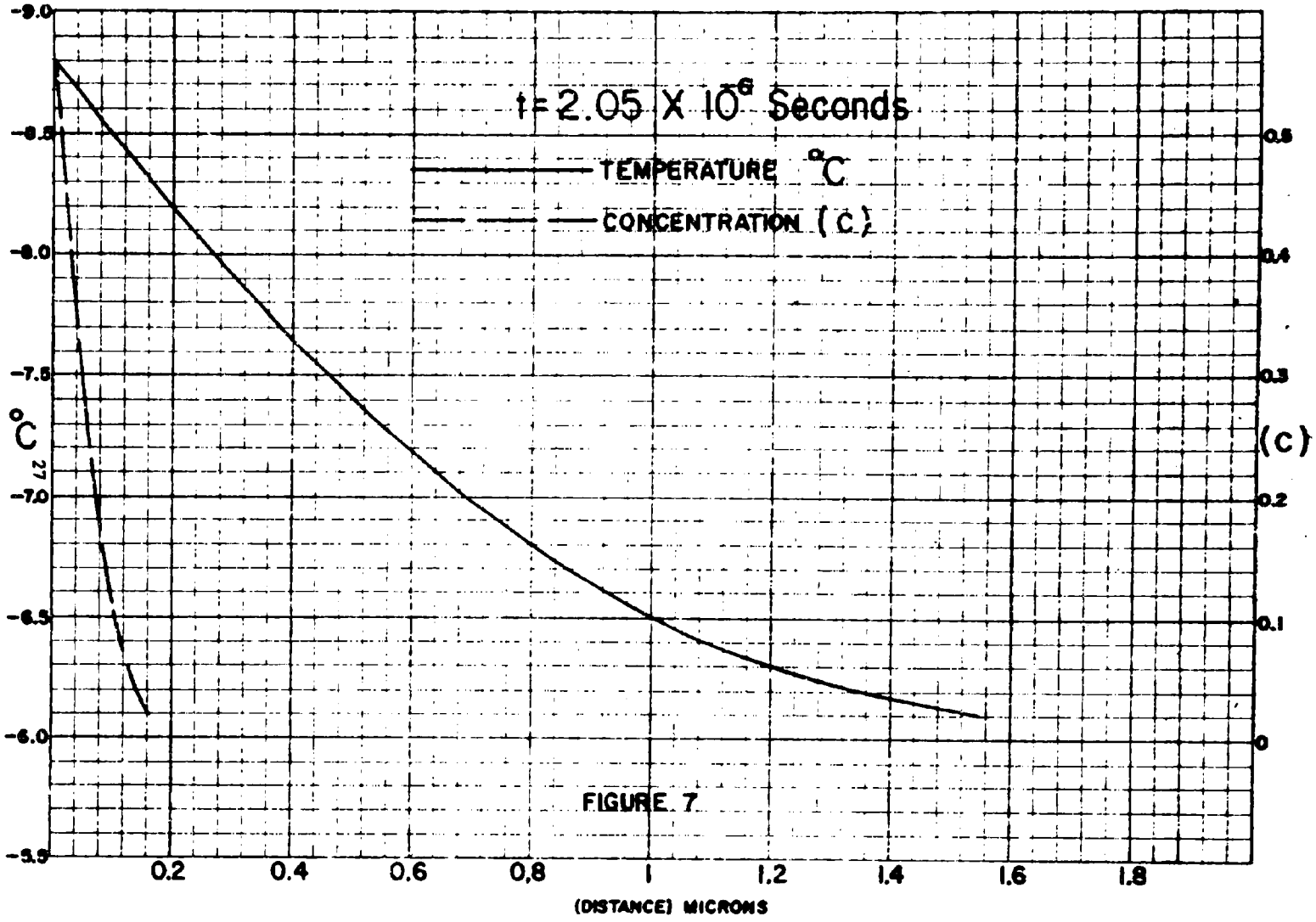

t = 2.05 X 10^-6 Seconds
TEMPERATURE °C
CONCENTRATION (C)
°C
(C)
-9.0
-8.5
-8.0
-7.5
-7.0
-6.5
-6.0
-5.5
0.5
0.4
0.3
0.2
0.1
0
0.2
0.4
0.6
0.8
1
1.2
1.4
1.6
1.8
(DISTANCE) MICRONS

FIGURE 7

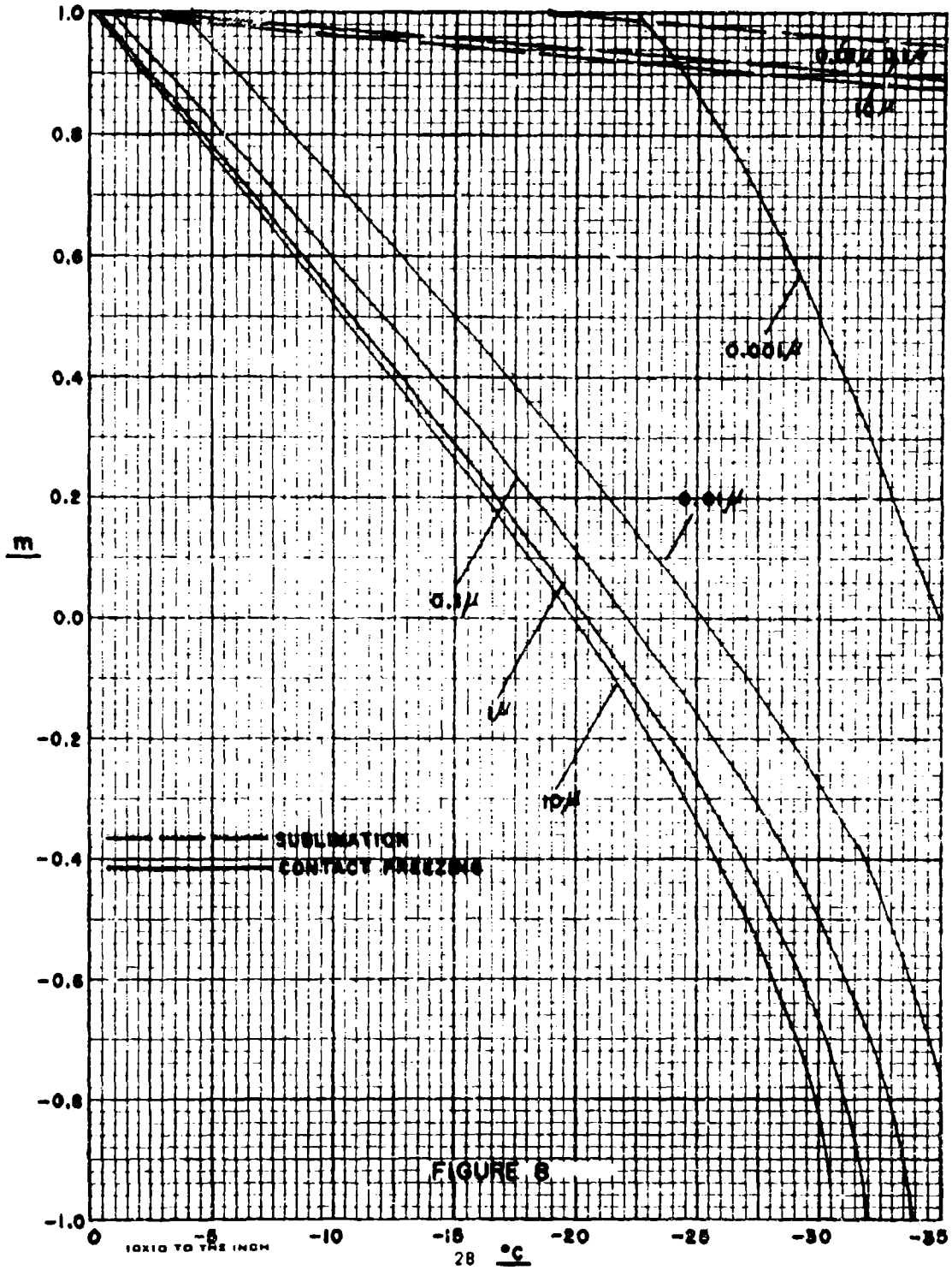

1.0
0.8
0.6
0.4
0.2
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
m
0.001μ
0.1μ
1μ
10μ
SUBLIMATION
CONTACT FREEZING
0
-5
-10
-15
-20
-25
-30
-35
°C
10X10 TO THE INCH

FIGURE 8

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A proposed nucleation mechanism is described for mixed smokes composed of potassium iodide and silver iodide.

The dynamic dissolution of such smoke particles is considered to be theoretically of significance in the nucleation mechanism of supercooled fogs of water droplets.

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