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RESEARCH AND DEVELOPMENT REPORT

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PREPARATION OF FEED FOR THE TRIBUTYL PHOSPHATE WASTE METAL RECOVERY PROCESS

SPECIAL RE-REVIEW
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BY JP DEROUIN DATE 3-2-81
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TECHNICAL DIVISIONS

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**PREPARATION OF FEED FOR THE
TRIBUTYL PHOSPHATE WASTE METAL RECOVERY PROCESS**

by

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Chemical Development Section
Separations Technology Division

July 13, 1950

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-2-

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PREPARATION OF FEED FOR THE
TRIBUTYL PHOSPHATE WASTE METAL RECOVERY PROCESS

I. INTRODUCTION

The uranium in the present 200 Area separations process neutralized wastes is to be recovered by the tributyl phosphate solvent extraction process. During its period of underground storage, this neutralized waste has separated into liquid and solid phases, the latter being a complex mixture of sodium uranyl carbonate and phosphates. These phases will be mechanically mixed to form a slurry or suspension representing as closely as possible homogenization of each three tank cascade. This slurry, estimated to be eight parts by volume of supernate to one part of solid, will be treated with nitric acid to dissolve the solids and to produce a homogeneous solution. The acid molarity of this solution was originally stipulated by TBP-HW-3 Flowsheet to be 2.45. Serious corrosion problems encountered in the recovery of nitric acid from TBP-HW-3 Flowsheet wastes have led to the abandonment of nitric acid recovery, and subsequent Flowsheets TBP-HW-4 and TBP-HW-5 which were devised to minimize nitric acid consumption result in a reduction of acidity of the solution from the slurry dissolution step to 2.01 molar.

It has been demonstrated by experimental studies reported in Document HW-18407, that this reduction in acid molarity does not invalidate the present study with respect to slurry dissolution, since the dissolution of the solids proceeds to completion under the final acid concentration specified by any of the Flowsheets TBP-HW-1 through 5.

II. OBJECTIVES

The objectives of this investigation were:

1. Determination of the chemical and physical properties of the system during dissolution of the slurry with nitric acid.
2. Determination of the rate of reaction between nitric acid and slurry with variations in the system temperature and composition.
3. Determination of the characteristics of the evolution of carbon dioxide as a function of the type of reaction system, i. e. alkaline going to acid or entirely acidic.

4. Determination of the rate of solution of sodium uranyl carbonates (hard sludge) in water.

This information was anticipated as being necessary to supply adequate information for defining the preferred conditions for the preparation of feed, and to form the basis for engineering design.

III. SUMMARY AND CONCLUSIONS

The dissolving of metal waste slurries to prepare feed solutions for the TBP process has been demonstrated in the laboratory, using synthetic incubated metal waste as the starting material.

The preparation of this feed solution by adding 60 per cent nitric acid to the slurry which was composed of an 8 to 1, liquid to solid, volume ratio (the "direct strike") was investigated as the first step. The adiabatic temperature rise was found to be 16°C., and the peak evolution of CO₂ was found to be in excess of 600 cubic feet per minute, on the basis of a 100 gallon per minute acid addition rate as prescribed for plant operation.

At a pH of approximately 9, a precipitate of sodium uranate began to form which reached a maximum viscosity of 850 centipoises at a pH of 1, as determined at 20 RPM with the number 2 spindle of a Brookfield RVF viscometer. The precipitate was not completely dissolved until excess acid was added to bring the final concentration to approximately 2 M in free HNO₃.

The "reverse strike," in which the slurry was added to 60 per cent nitric acid, resulted in completely uniform evolution of CO₂, corresponding to a rate of approximately 200 cubic feet per minute on the basis of plant dissolver operation. The slurry dissolved as rapidly as added, and the maximum viscosity for the solution (before concentration) was found to be 1.5 centipoises as measured at 25°C. with a modified Ostwald viscometer. Foaming was eliminated as a serious problem by this reverse strike, as it was noted that a rate of slurry addition equivalent to twice that proposed for plant operation resulted in a layer of foam less than one inch in thickness above the surface of the solution.

Additional studies at acid temperatures of 60-65°C., indicated that elevated temperatures would have but slight effect (less than 10 per cent) on foaming and rate of dissolving.

It was concluded that the "reverse strike" represented the logical method, and suggested optimum conditions for Flowsheet TBP-HW-4 feed preparation are as follows: The slurry in a ratio of 8 parts of supernate to one part of

solid should be pumped to the 15,000 gallon dissolver tank containing 49,700 pounds of 60 per cent nitric acid (under continuous agitation,) at a rate of 100 gallons per minute, until the total volume reaches 13,500 gallons. The CO₂ evolution rate will be approximately 200 cubic feet per minute for the duration of this dissolving step. Two to two and one-half hours will be required for the completion of a charge, representing 2.5 tons of uranium, or one-fourth of one days' feed requirements.

Hard sludge components, primarily sodium uranyl carbonate, may have to be removed from the U. G. S. tanks by dissolution with water since their physical strength is such that uncertainty exists regarding the effectiveness of the high velocity supernate streams to reduce the agglomerates to small particles permitting removal to the accumulator tank by pumping. The rate of dissolution in water under moderate agitation at 25 ± 5°C. of a specimen of synthetic hard sludge expressed in decrease in cake thickness vs. time is 0.02 cm./hour.

Physical data obtained on both slurry and prepared feed were previously presented in a progress report, HW-18149, and are included in this final report.

IV. EXPERIMENTAL

A. Waste Preparation and Properties

1. Synthetic waste composition

The material used in this work was incubated metal waste (IMW) prepared as indicated in Table I. Solutions were blended to correspond as closely as possible to the U. G. S. waste.

TABLE I
MAKE-UP OF SYNTHETIC METAL WASTE

<u>Component Added</u>	<u>Molarity</u>	<u>grams/liter</u>
UNH	0.29	146 ± 7
H ₃ PO ₄	0.40	66 ± 3
Na ₂ SO ₄	0.25	35.5 ± 2
Na ₂ CO ₃	0.97	103 ± 5
NaNO ₃	0.16	13.6 ± 0.7
NaHCO ₃	0.34	28.6 ± 1.5

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-6-

HW-18367

The batches of approximately 21 gallons each, were stored in 30 gallon black iron drums which were placed in a steam heated incubation vat held at a temperature of $80 \pm 5^{\circ}\text{C}$. to simulate the conditions existing in the underground tanks. The particular batch used for these laboratory studies had been incubated for one month and contained only finely divided solids, although different batches of the same material have been noted to contain large, crystalline masses. Obtaining representative samples of such a slurry was difficult, as can be seen from the data in Table II, where comparisons are made between two supposedly identical aliquots of slurry, and also between the solids from two additional samples. Supernate "as received" is also compared with acidified supernate. These data, showing the variation in chemical composition between samples, also illustrate what may be expected in plant operation where solid stratification may result in phases of varying carbonate, phosphate, sulfate and uranium ratios. Any such variation will have a direct bearing on the ability of the feed preparation step to produce column feeds as required by Flow-shee specifications.

The volume ratios referred to throughout this report were determined by centrifuging the solids from the supernate in a graduated vessel at 1000 g's for 15 minutes. The phase volumes were then read directly.

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TABLE II
SOLUTIONS PREPARED BY DISSOLVING INCUBATED
SYNTHETIC METAL WASTE

Sample	Solution Preparation			Solution Composition							
	Initial Volume (ml.)	H ₂ O Added (ml.)	70% HNO ₃ Added (ml.)	Final Volume (ml.)	UNH (g./l.)	PO ₄ ⁼ (g./l.)	SO ₄ ⁼ (g./l.)	CO ₃ ⁼ (g./l.)	Na+ (g./l.)	H+ (g./l.)	Density (g./ml.)
Slurry I	100.0	----	27.4	116.0	110.0	31.20	21.40	----	60.0	2.04	1.289
Slurry II	100.0	----	30.0	126.0	100.6	31.20	20.28	----	70.4	2.18	1.277
Super-nate I	100.0	----	----	100.0	27.3	25.30	25.87	25.17	57.6	----	1.170
Super-nate II	83.5	----	11.5	95.0	21.76	26.21	23.30	----	63.6	1.0	1.178
Sludge I *	14.0	86.0	15.9	107.5	80.5	12.40	2.86	----	17.6	1.47	1.149
Sludge II *	16.5	83.5	23.0	111.0	106.1	13.90	4.22	----	25.6	2.18	1.193

* These sludges were obtained by centrifuging two supposedly identical aliquots similar to samples 1 and 2.

2. Synthetic Waste Viscosity

Viscosities were determined, using the Brookfield Model RVF viscometer, as a function of shear rate for various supernate to sludge volume ratios. These data are given in Tables III and IV, and are plotted in Figures 1 and 2. The volumes of solids were determined by centrifuging at 1000G's for 15 minutes, and on this same basis, the apparent solid density was also determined.

The more dilute slurries exhibit almost no change in viscosity with increase in shear, while the heavier slurries (8:1 and 5:1, liquid: solid) which decrease in viscosity with increasing shear, appear to be thixotropic. Should this prove to be true of the actual stored metal waste also, it would indicate pumping and blending power requirements to be perhaps somewhat less critical than originally anticipated. Further information on the viscosity as determined in a pipeline type viscometer for slurries ranging from 0.44 to 5.85 volume of supernate to sludge is presented in HW-17775 "Apparent Viscosities of Simulated Underground Metal Waste Slurries".

As may be seen, the effect of temperature on viscosity is almost negligible when the liquid to solid ratio is 10 or over. Included on each graph is the comparison curve at 20°C. for the 8 to 1 volume ratio. This ratio is considered as the standard since it represents what are believed to be the actual relationships of the underground stored wastes.

TABLE III
VISCOSITY IN CENTIPOISES OF SYNTHETIC SLUDGE AT 40°C

Liquid Solid	No. 2 Spindle Speed (rpm)			
	10	20	50	100
5	172.0	108.0	61.6	58.8
10	18.0	15.0	18.4	21.2
15	12.0	12.0	12.8	17.2

TABLE IV

VISCOSITY IN CENTIPOISES OF SYNTHETIC SLUDGE AT 60°C.

Liquid Solid	No. 2 Spindle Speed (rpm)			
	10	20	50	100
5	160.0	102.0	53.6	44.4
10	----	16.0	16.8	18.4
15	----	15.0	15.0	18.0

3. Density relationships synthetic slurry mixtures

The density of sludge and supernate combinations has been measured over the range 1:1 to 20:1, liquid: solid, as a function of temperature. The data are given in Table V and Figure 3.

The density of the supernate alone was also determined as a function of temperature, and is shown in Figure 4. The apparent density of the centrifuged sludge was found to be 1.675 gms./ml. at 25°C., and was not appreciably affected by temperature between 20° and 70°C.

TABLE V

APPARENT DENSITY OF SYNTHETIC SLURRIES AS A FUNCTION OF TEMPERATURE

25°C.		40°C.		65°C.	
Liquid Solid	Density (g./ml.)	Liquid Solid	Density (g./ml.)	Liquid Solid	Density (g./ml.)
1	1.436	1	1.399	1	1.390
4	1.240	5	1.215	5	1.200
9	1.179	10	1.173	10	1.157
14	1.170	15	1.157	15	1.141
19	1.161	20	1.149	20	1.132

B. Direct Strike Dissolving**1. Viscosity**

As a qualitative observation, it was noted that during the dissolving step, the viscosity of the sludge-supernate mixture increased tremendously as acid was added. This is due to the formation of the very viscous di- or poly-uranates as the pH is lowered from 9 to 0. This was also true of sludge alone and supernate alone, as well.

To determine this effect quantitatively, an experiment was performed in which a sample of the slurry was acidified step-wise with 70 per cent nitric acid, with viscosity measurements being taken at each step.

The readings were made with a Brookfield Model RVF viscometer, using three different spindles, and four different speeds for each spindle. The data are presented in Table VI and Figure 5 (spindle number 2 only). This plot shows that for part of such an acidification cycle (pH 1.5 to 0), high speed, high shear agitation would be required, while for the balance of the time, slow moving, low shear agitation would suffice.

2. pH Changes

Figure 6 illustrates the pH profile of this direct strike acidification.

3. CO₂ Evolution

It was noted that when the calculated amount of 60 per cent nitric acid was added rapidly to the slurry the resulting mixture increased to 7-10 times its volume due to CO₂ foam formation. The reaction was complete, and the solution volume was again normal in 10 seconds. This was done on a 50 ml. scale in a 500 ml. graduated vessel.

TABLE VI
VISCOSITY AS A FUNCTION OF SLURRY ACIDIFICATION

Viscosity Measurements: Brookfield viscometer. Results given in centipoises.

Slurry: 350 milliliters of synthetic waste prepared by incubating simulated waste metal solutions.

Nitric Acid: 15.69 Normal

Temperature: Readings were all made at 21°C.

Acid (ml.) pH	Spindle No. 1 RPM			Spindle No. 2 RPM			Spindle No. 3 RPM					
	10	20	50	100	10	20	50	100	10	20	50	100
0	80.0	57.5	43.0	47.5	88.0	66.0	44.0	46.0	170.0	123.0	54.0	40.0
10	8.7	19.0	21.0	24.5	---	20.0	22.4	32.8	220.0	125.0	40.0	37.0
20	7.2	168.0	107.5	47.5	180.0	112.0	56.0	52.0	230.0	135.0	60.0	40.0
25	6.9	193.0	105.0	48.8	220.0	126.0	60.8	51.2	450.0	250.0	110.0	65.0
30	6.8	127.0	78.0	45.0	176.0	100.0	48.0	48.0	122.0	65.0	240.0	115.0
35	6.7	160.0	88.0	41.5	160.0	100.0	48.0	45.2	1350.0	800.0	390.0	235.0
40	6.0	175.0	102.5	46.2	200.0	112.0	60.0	46.0	1100.0	665.0	312.0	185.0
45	1.5	335.0	187.5	51.8	380.0	210.0	101.6	70.0	610.0	350.0	160.0	115.0
50	---	750.0	390.0	96.5	812.0	460.0	212.0	122.0	1350.0	800.0	390.0	235.0
60	---	*	*	*	1520.0	860.0	405.6	228.0	1350.0	800.0	390.0	235.0
70	0.2	*	*	*	1120.0	640.0	292.0	171.2	1100.0	665.0	312.0	185.0
80	0.0	550.0	290.0	85.0	540.0	320.0	144.0	88.0	610.0	350.0	160.0	115.0
90	"	**	**	**	104.0	76.0	60.0	46.0	150.0	75.0	36.0	38.0
100	"	**	**	**	20.0	20.0	24.0	28.0				
110	"	15.0	10.0	17.0	12.0	12.0	20.0	18.0				

* Off scale readings.

** Non reproducible results due to spindle configuration.

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-12-

HW-18367

4. Heat of Neutralization

The heat evolved in acidifying the slurry to prepare an RAF solution was determined using a Dewar flask for the calorimeter, and assuming a specific heat of 0.8 for the final solution. The isothermal heat release at 25°C. per gram of slurry (7:1: liquid: solid volume ratio) was approximately 13 calories. This corresponds to a temperature increase of 16°C. under adiabatic conditions.

C. Reverse Strike Dissolving

1. Viscosity

The viscosity (of the material corresponding to TBP HW-3 Flow sheet) after acidification and before concentration is too low to be measured with the Brookfield, and so was determined with the modified Ostwald viscometer at 25°C. This value was 1.42 centipoises, which was increased to 2.01 centipoises upon concentration to RAF as specified in the TBP HW-3 Flowsheet. The approximate viscosity-temperature relationships are shown in Figure 7, compared with water.

2. Rate of Slurry Addition

Slurry was added to well agitated 60 per cent nitric acid, and the amount of foam produced was determined as the measurable layer depth above the surface of the liquid. The rates are given in Table VII, and are plotted in Figure 8. A very slight vacuum (2-3 inches of water) was applied to the vessel during one run, but no appreciable effect on the foam height was apparent. Two additional experiments were run at 60-65°C., in which an attempt was made to determine the effect of temperature in foam height. The results were inconclusive, due to the low rate of addition employed, but indications are that the effect is very slight if noticeable at all. This is shown on Figure 8. The rate of dissolution, already very rapid at 25°C. was not materially increased at the higher temperature. It was further noted during the course of these experiments that the height of foam was independent of the vessel diameter when this diameter was greater than a minimum of approximately 1.5 inches, where "bridging" could not occur and capillary action was no longer a factor.

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TABLE VII
FOAM THICKNESS VS. SLURRY ADDITION RATE 25°C.

<u>Addition Rate</u> <u>(Gal. / (Hr.) (Sq. Ft.)</u>	<u>Foam Height</u> <u>(Mm.)</u>
2.5	0
8.0	0.4
10.0	0.5
15.5	1.5
20.5	2.0
22.0	3.0
24.5	3.2
30.5	4.0
38.0	6.0
49.3	7.0
88.0	12.3
124.0	17.3

Experiments were run in which the slurry, added to the acid, was introduced beneath the surface of the liquid very near the bottom of the vessel. The results were decisively unfavorable, since the CO₂ evolved at this point was forced to pass upward through the bulk of the solution, and in doing so, expanded the total volume 300 per cent at a slurry addition rate of 40 gal. / (hr.) (sq. ft.). This method is thus definitely to be discouraged.

D. Water Dissolving of Carbonate Sludge

For this experiment, a sample of hard, crystalline sludge was obtained from a vat of synthetic metal waste which had been incubated for six months. Two pieces of known surface area were prepared, and were then covered with water (insufficient to dissolve all the sludge) which was circulated slowly. The solutions were sampled at intervals and analyzed for UNH as a function of time. The resulting rate curves are shown in Figure 9. The data are in Table VII.

TABLE VIII
RATE OF URANYL CARBONATE DISSOLVING IN WATER

Time (Hrs.)	Sample B UNH in Sol'n (g./l.)	Sample A UNH in Sol'n (g./l.)
1	35.9	55.4
2	57.5	82.5
3	66.3	83.4
4	66.4	84.3
22	115.0	116.6

Note: Sample A was harder than B, although the densities were approximately the same at 2.5.

A second experiment was run in which samples of the hard sludge were imbedded in paraffin with only one surface exposed to the circulating water. The density of this material was approximately 3.5. From considerations of time, known surface area, and uranium found in solution, the solubilization of the sludge was determined to be 0.02 cm./hour expressed as "penetration rate". This was determined at approximately 1.5 per cent of saturation; therefore the amount of carbonate in solution had no appreciable effect on the dissolving, thus keeping the penetration rate roughly linear over the total elapsed time. The work reported in HW-18140 (Table VIII, this report) was done with samples which were free to disintegrate as they dissolved, in a system in which saturation was approached much more rapidly. The penetration rate was here found to be about twice that noted in the previous experiment.

V Discussion of Results

A Physical Properties of Synthetic Waste Slurries

The material which was used for the bulk of these tests, cannot be considered representative of the U. G. S. slurries, due to the fact that it was incubated for only one month, and contained no sizable masses of the "hard" sludge, $UO_2 \cdot Na_4(CO_3)_3$. It does, however, resemble in many ways what may be expected of the "hot" wastes following the jetting, stirring and pumping, which are proposed for the removal from U. G. S.

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-15-

HW-18367

and subsequent feed preparation operations. In spite of difficulties involved in attempting to make physical measurements of viscosity, density, etc., on such materials, the data are believed to be reliable enough to allow extrapolation to proposed plant operations.

B. Direct Strike Acid Dissolving

The data which have been presented in the preceding section have shown conclusively that this method of feed preparation can no longer be considered. Slow acidification becomes extremely awkward in view of the great peak evolution of CO_2 and the formation of the uranate precipitates which are very difficult to handle.

The "one shot" dissolving step (adding excess acid in one charge) is no more desirable from the standpoint of foaming, although the agitation power requirements would be lessened somewhat.

C. Reverse Strike Acid Dissolving

This method, in which the blended slurry is added to an excess of acid appears to be the logical choice for this operation. Viscosity is no longer a factor, never rising much above 2 centipoises, even upon concentration to the desired RAF composition. The CO_2 evolution presents no cause for concern, being completely uniform and dependent upon the rate of slurry addition. At the rates presently proposed and even at twice these rates of addition, CO_2 foaming in the dissolving vessel will give no difficulties other than the usual off-gas disposal when the dissolver vessel is provided with adequate agitation facilities. The rate of dissolving at 25°C . is extremely rapid throughout the range of solution compositions and does not appear to be significantly increased by an increase of temperature to 65°C .

D. Water Dissolving of Carbonate Sludge

Should the planned sluicing operation for U. G. sludge removal fail to break up the tri-carbonate satisfactorily, it will be possible to remove it by dissolving with water. Rate studies have been carried out, and penetration rates have been calculated from which extrapolation to plant requirements may be made.

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VI. Future Work

No additional work is in progress at the present time on the acidification step, nor is any planned in the foreseeable future.

One experiment is underway at present, however, aimed at obtaining supplemental data on the settling rates and viscosities of slurries as a function of shear (high speed stirring). Parallel experiments are being run on synthetic incubated wastes and "hot" material from the 101-U tank. The details and results of this work will be issued as a separate report when they become available.

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FIGURE 1

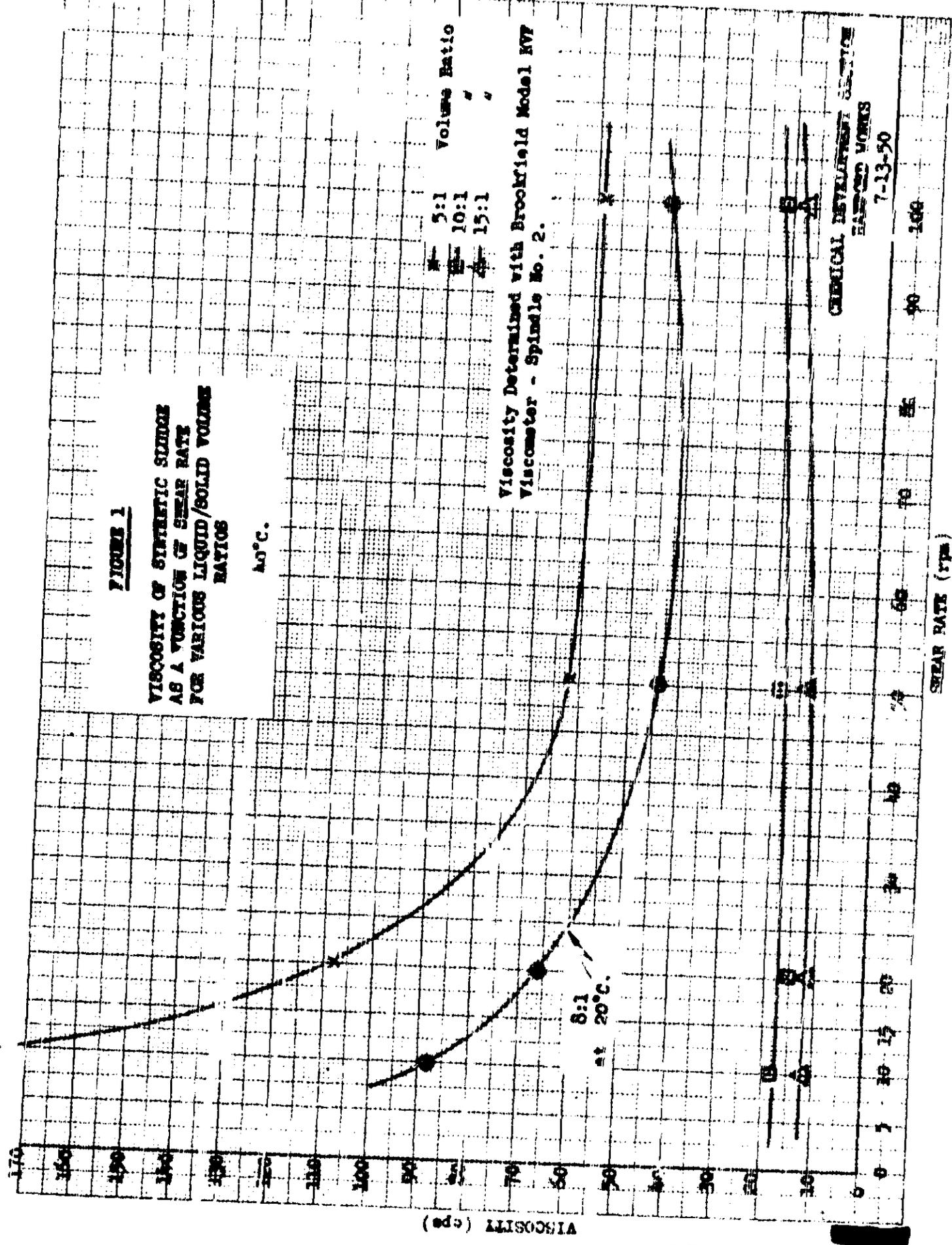
VISCOSITY OF SYNTHETIC SLUDGE AS A FUNCTION OF SHEAR RATE FOR VARIOUS LIQUID/SOLID VOLUME RATIOS

40°C.

Volume Ratio
5:1
10:1
15:1

Viscosity Determined with Brookfield Model KVF
Viscometer - Spindle No. 2.

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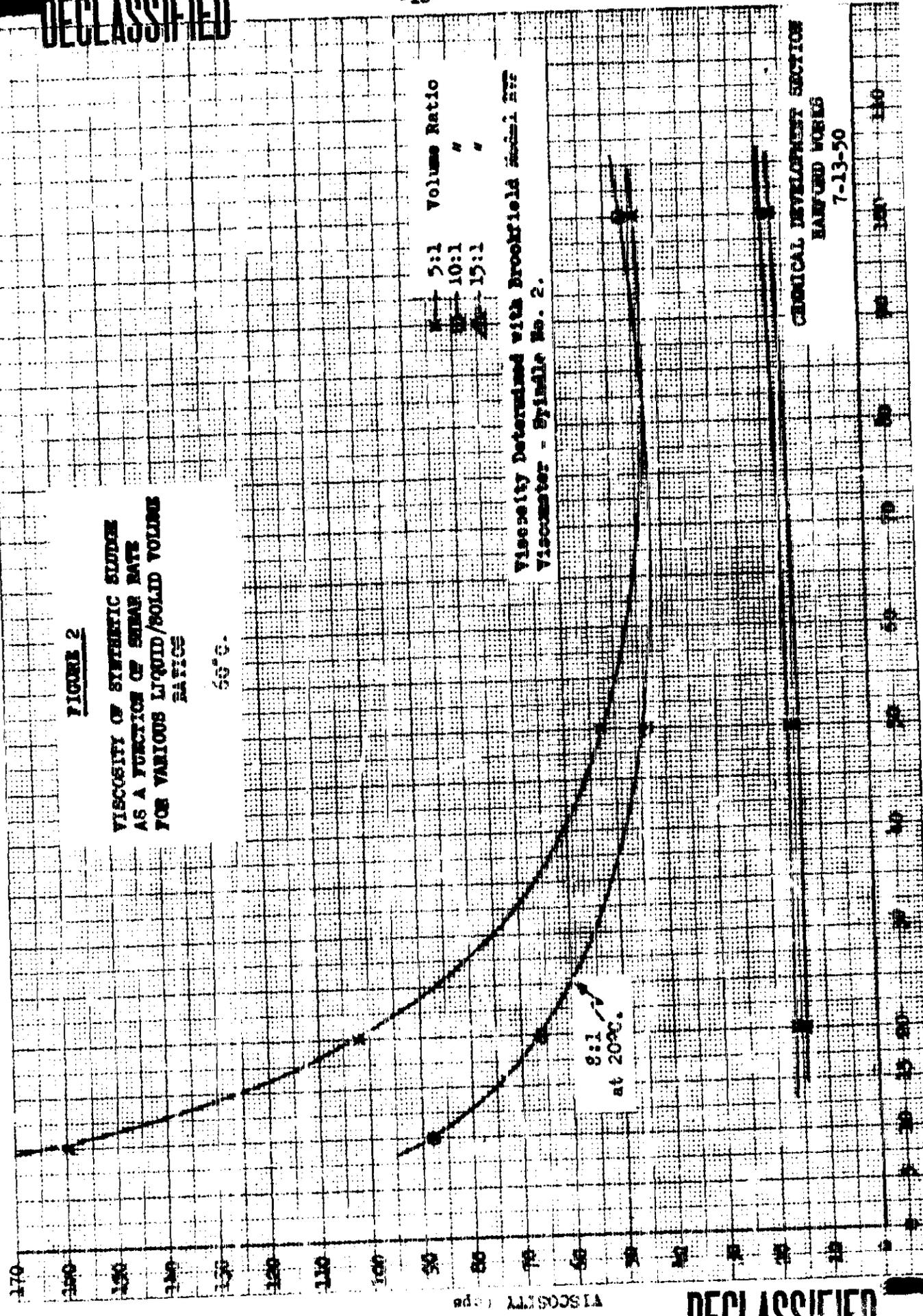
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KELUFFEL & ECKER CO

No. 39914 Millimeters, 5 mm lines centered, cm lines heavy
GRADE 4000

FIGURE 2
VISCOSITY OF SYNTHETIC SLUDGE
AS A FUNCTION OF SHEAR RATE
FOR VARIOUS LIQUID/SOLID VOLUME
RATIOS
65°C.



5:1 Volume Ratio
 10:1 " "
 15:1 " "

Viscosity Determined with Brookfield Mini. Etc
 Viscometer - Sydnale No. 2.

8:1
 at 20°C.

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No. 359 14. Millimeters, 5 mm lines, selected 2 mm lines heavy

FIGURE 3

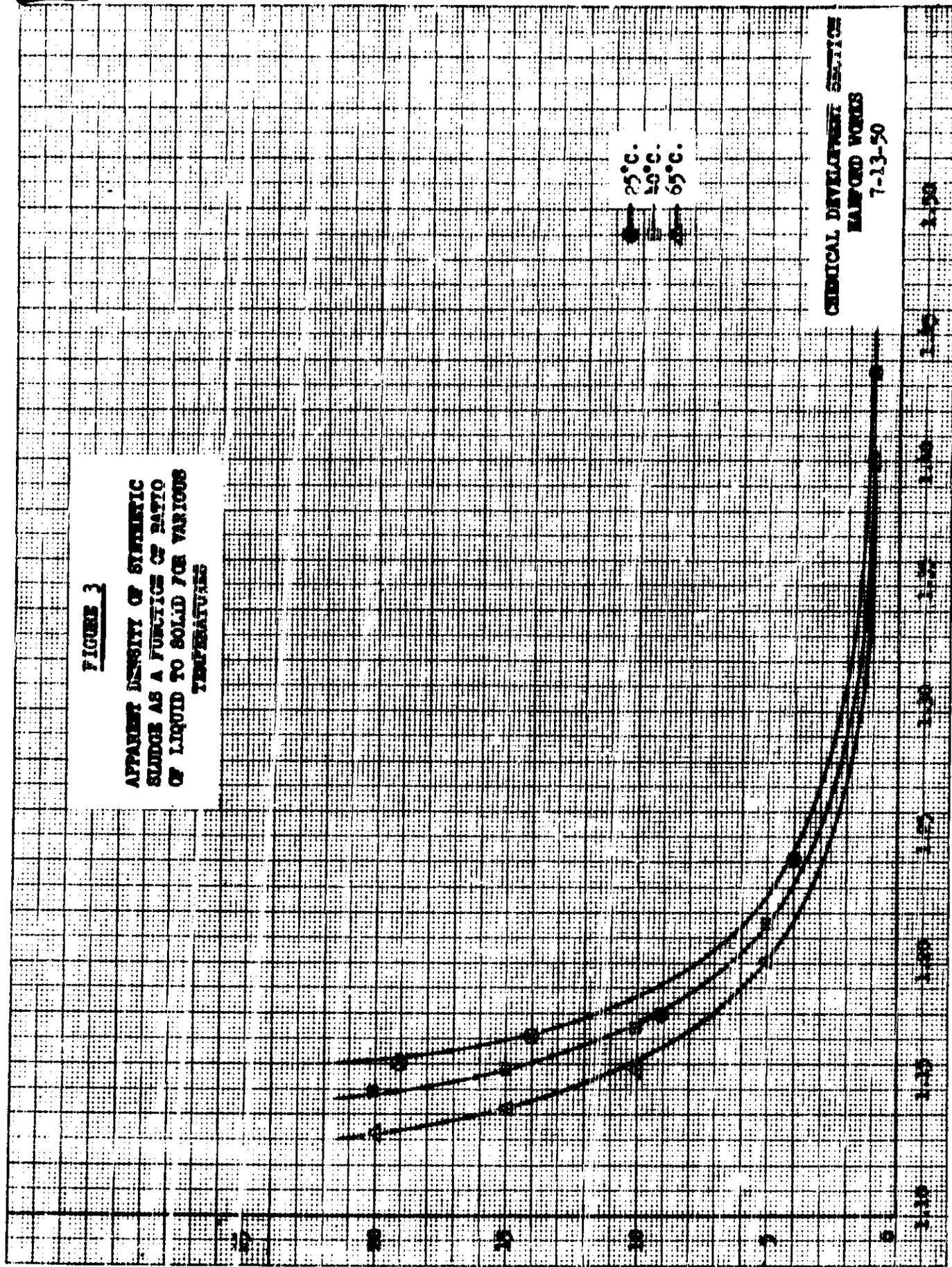
APPARENT DENSITY OF SYNTHETIC SLUDGE AS A FUNCTION OF RATIO OF LIQUID TO SOLID FOR VARIOUS TEMPERATURES

LIQUID-SOLID VOLUME RATIO

25°C.
40°C.
65°C.

CHEMICAL DEVELOPMENT SECTION
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TEMPERATURE (°C.)

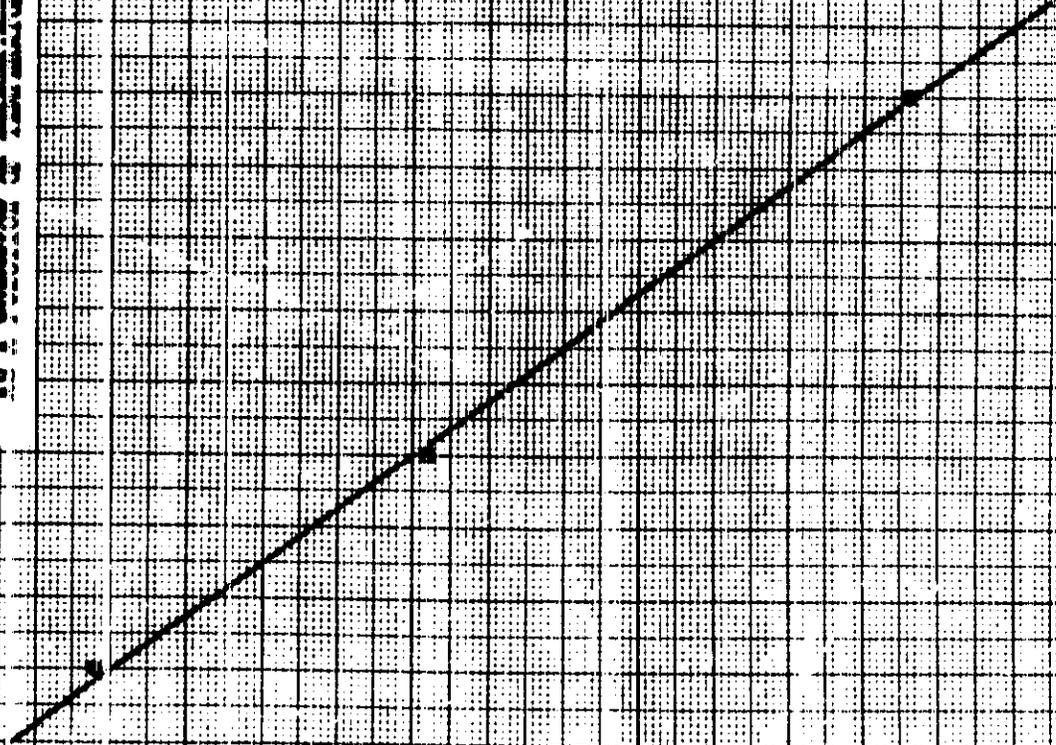


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FIGURE 4

**DENSITY OF SUPERNATE
AS A FUNCTION OF TEMPERATURE**

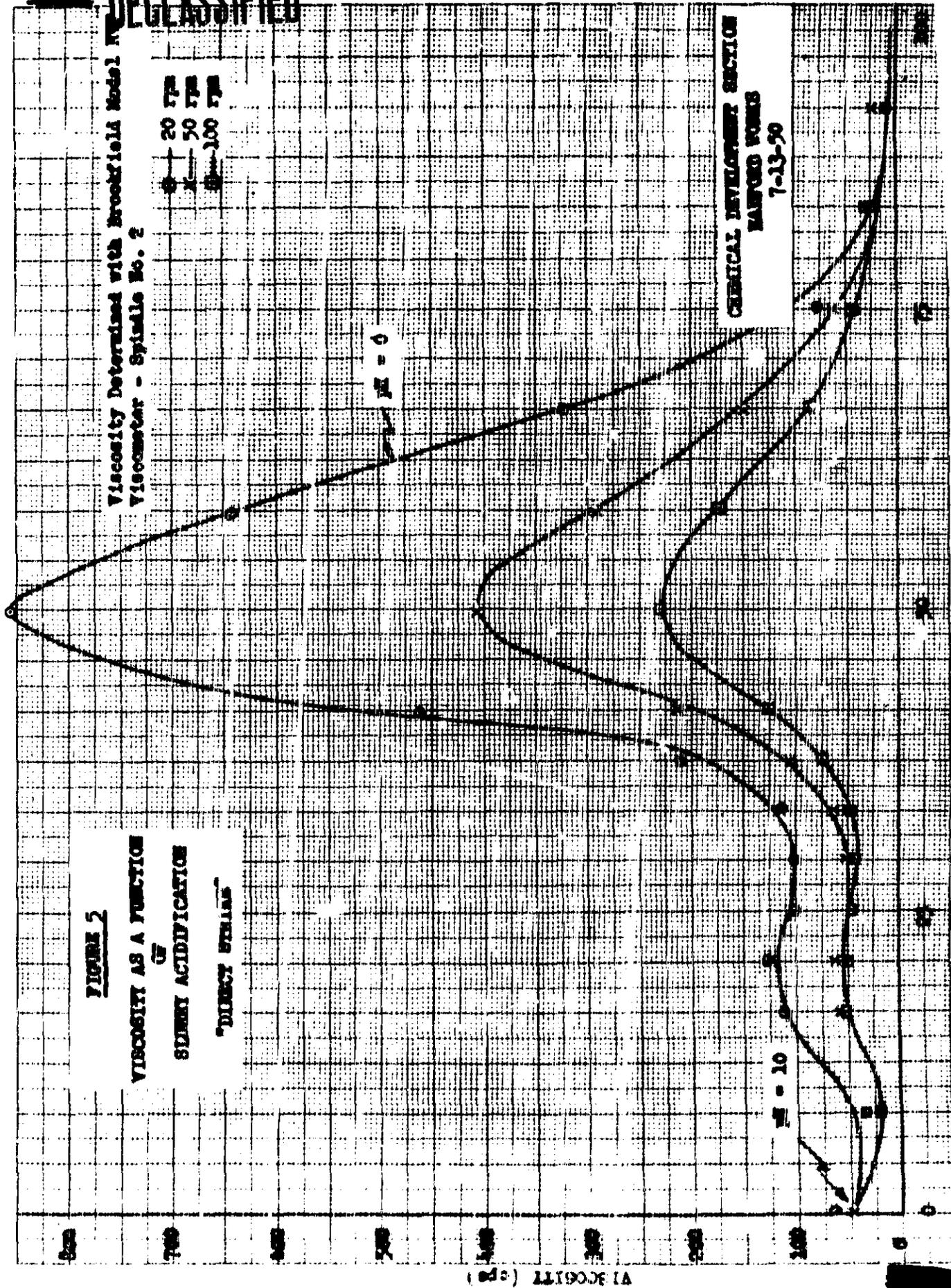


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DENSITY (Gms./ML.)

TEMPERATURE (°C.)

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FIGURE 6

PH AS A FUNCTION OF SLURRY ACIDIFICATION

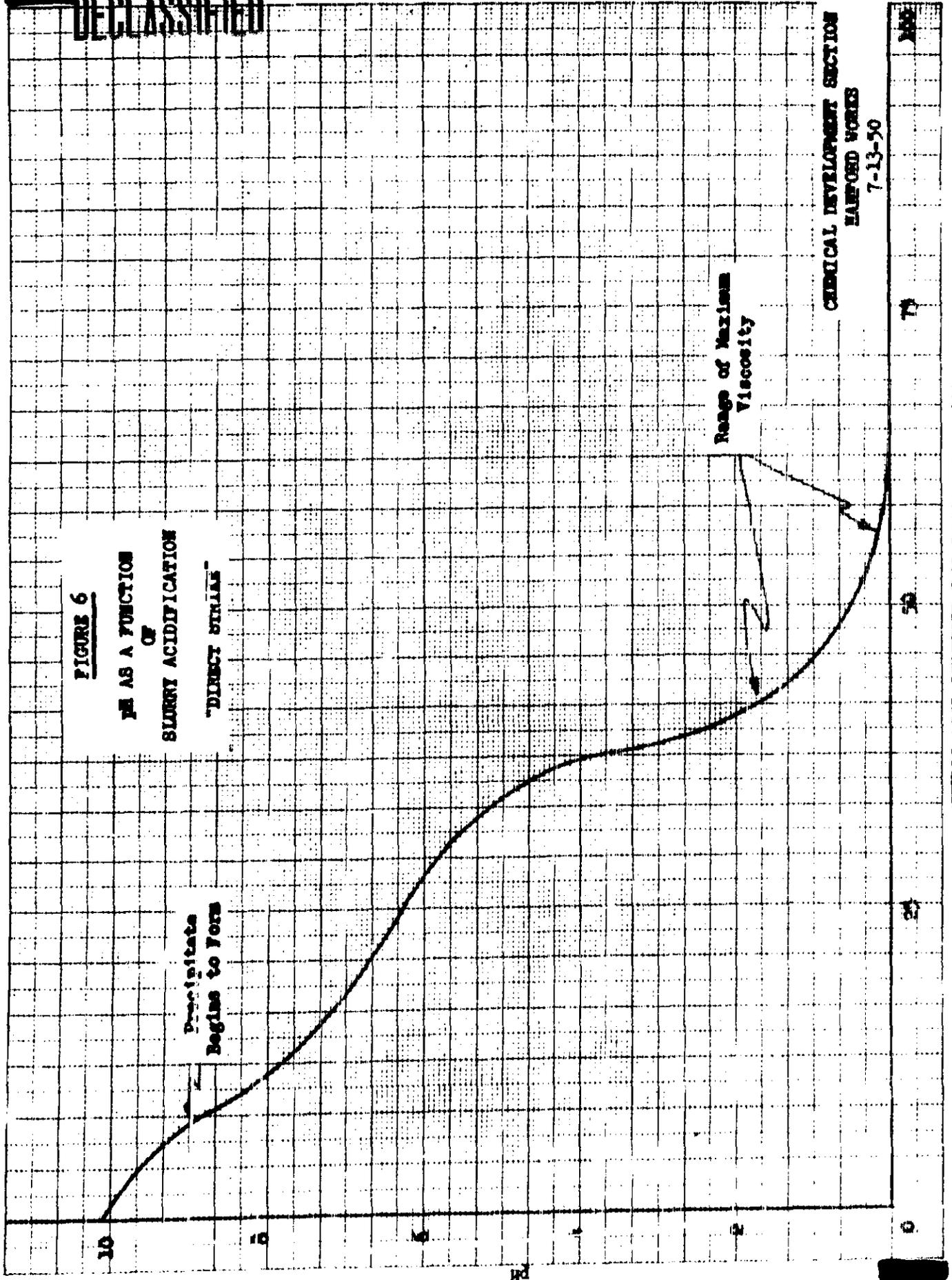
"DIRECT STRAIN"

Precipitate Begins to Form

Range of Maximum Viscosity

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NETRIC ACID REQUIRED FOR DISSOLUTION (%)



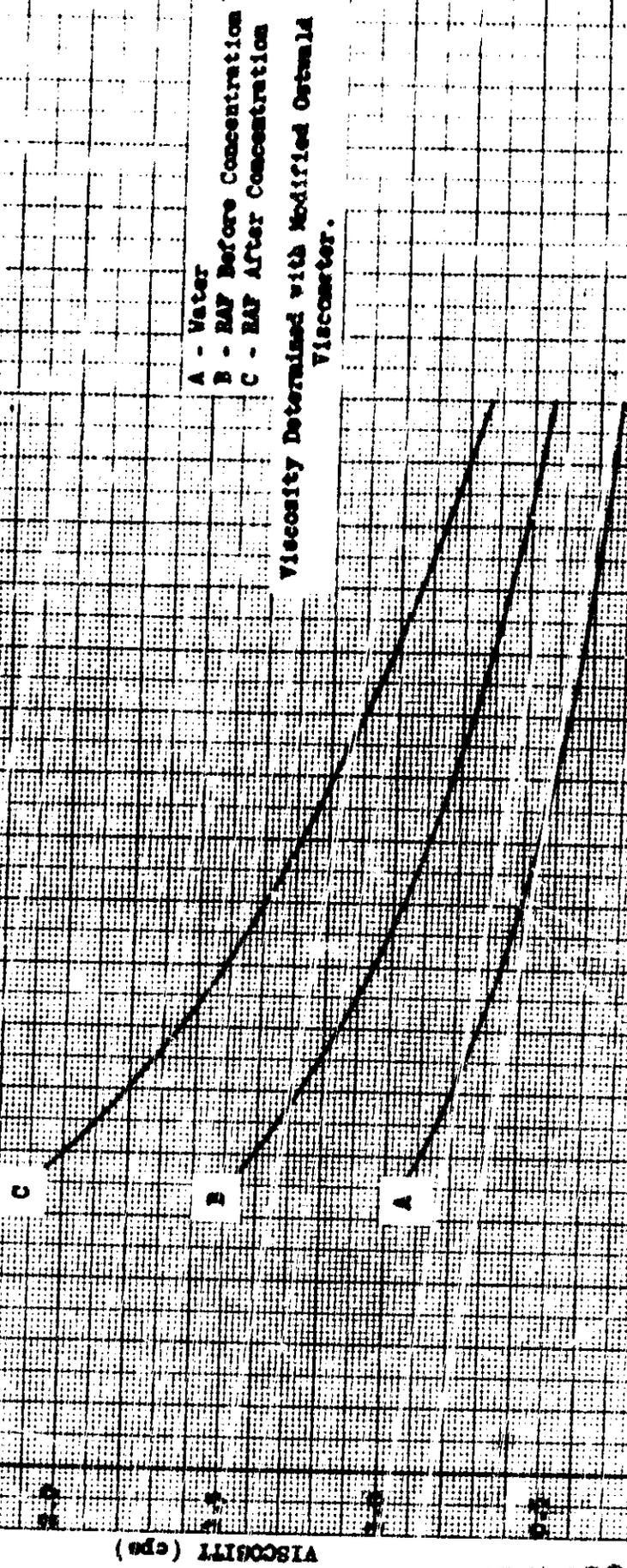
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McUFFEL & ESSEY CO

No. 359 14 Millimeters, 5 mm lines accents, 10 lines heavy

FIGURE 7

**VISCOSITY OF RAY
AS A FUNCTION OF TEMPERATURE**



A - Water
 B - RAY Before Concentration
 C - RAY After Concentration

Viscosity Determined with Modified Ostwald
 Viscometer.

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1108

VISCOSITY (cps)

FIGURE 8

FOAM HEIGHT AS A FUNCTION OF SLURRY ADDITION RATE

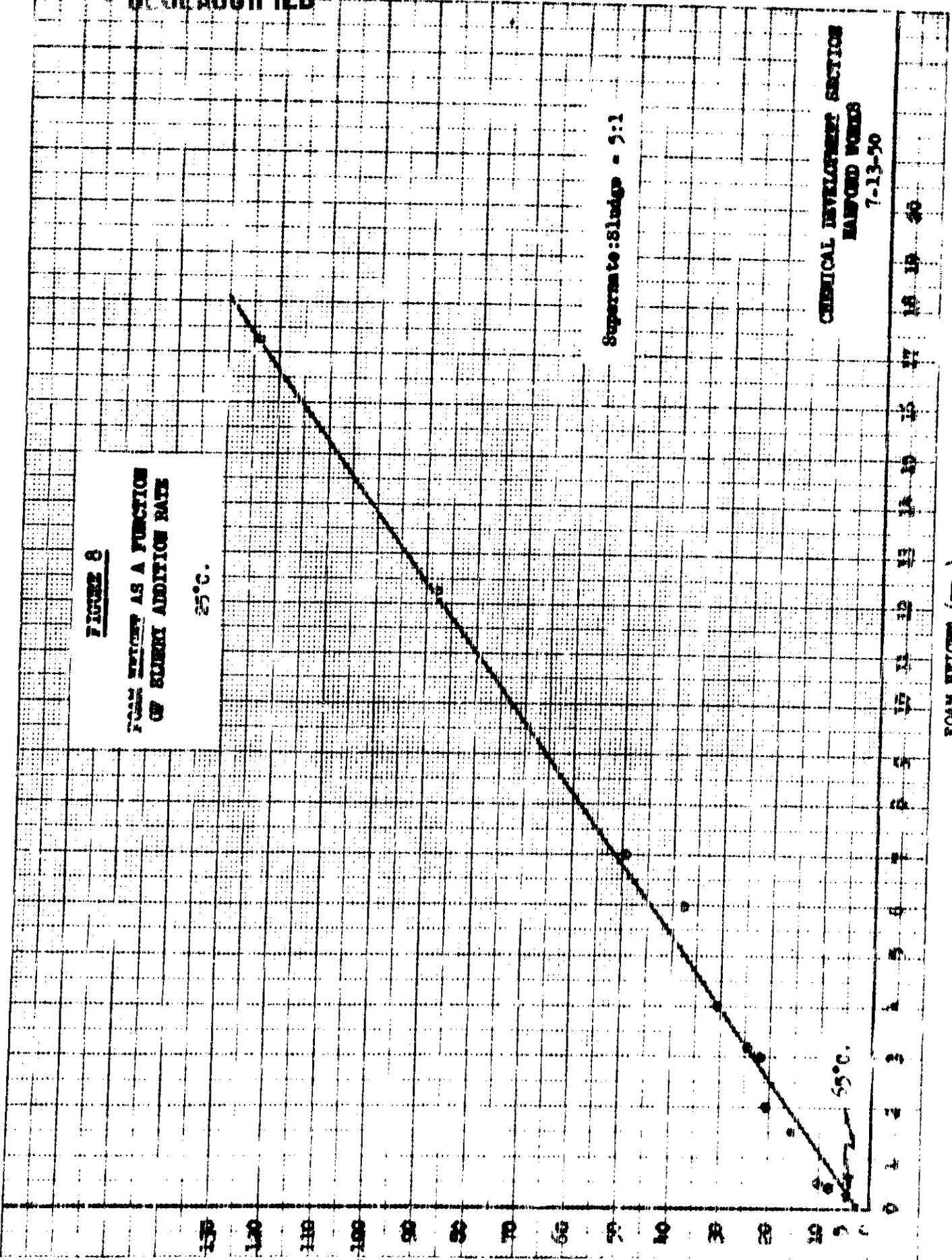
25°C.

SLURRY ADDITION RATE (g./hr./ft.²)

Supernatant: Sludge = 5:1

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FOAM HEIGHT (mm.)

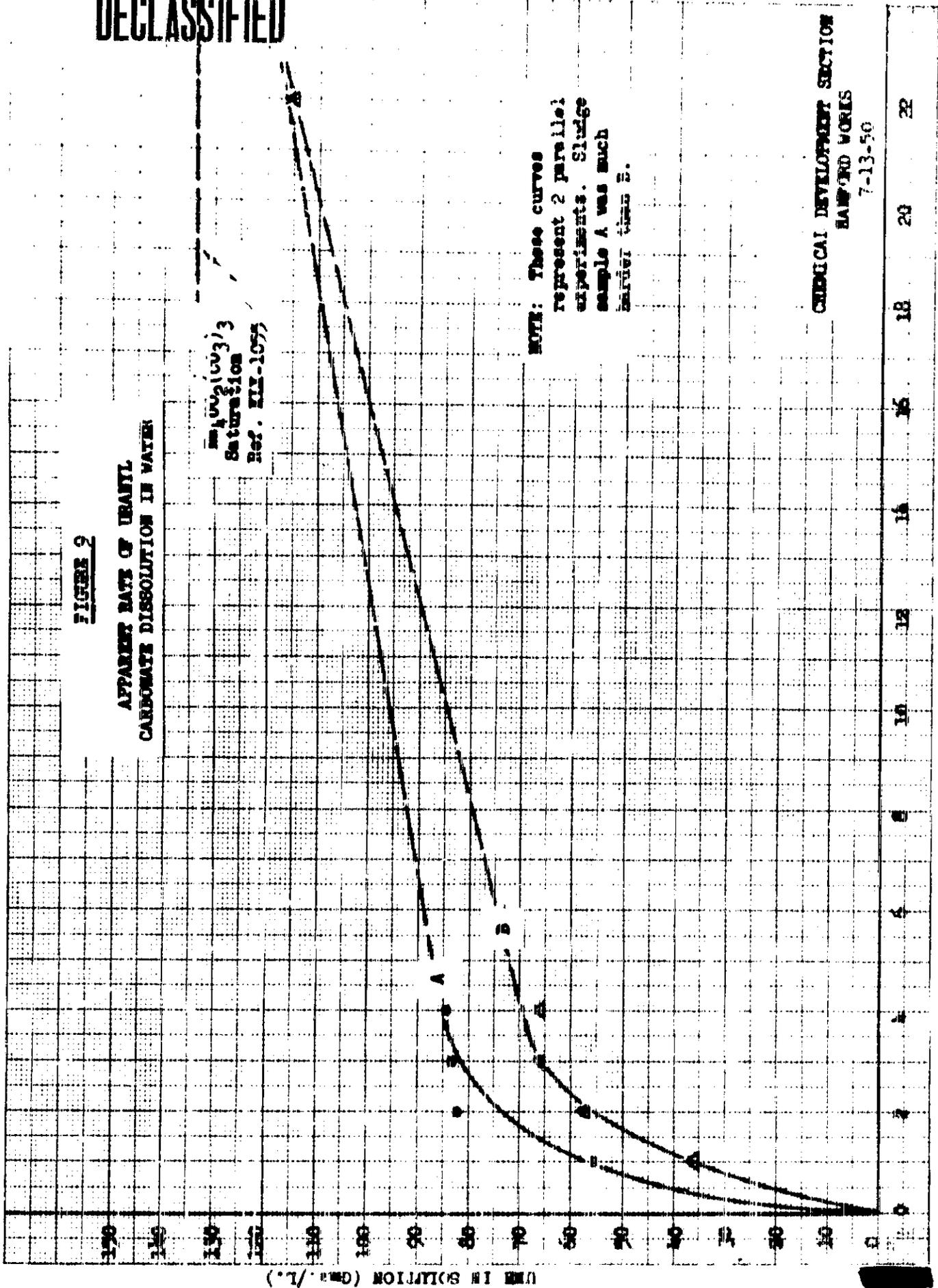


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FIGURE 2

APPARENT RATE OF URANYL CARBONATE DISSOLUTION IN WATER

Uranium Saturation
Ref. ILL-1077



NOTE: These curves represent 2 parallel experiments. Sludge sample A was much larger than B.

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TIME (Hrs.)

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