ECONOMIC EVALUATION OF A "KRYSTAL" CRYSTALLIZER

by

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Fulfillment of the Requirements

for the

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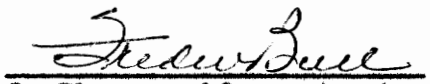
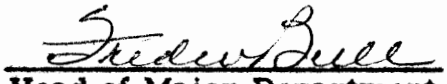
Approved:



In Charge of Investigation



Head of Major Department

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Blacksburg, Virginia

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I. INTRODUCTION

The final question applied to any proposed process in the highly competitive chemical industry is: "Will it make money?" Many times the economic feasibility of a process is inherent in such basic factors as markets and raw materials or the process itself. However, quite often the decision to go ahead on a process or to stop at the pilot plant level may be influenced by the choice of operating conditions.

Sometimes pressure, temperature and other variables may change with no effect on the quality of the product but with appreciable effects upon operating costs. Such factors as heat loss by radiation and the attendant cost of insulation, greater equipment and maintenance costs when high pressures are used, and fluctuating power costs at different flow rates all affect the total cost of an operation.

In order to determine the optimum operating conditions for any process a cost analysis or economic evaluation must be performed.

Continuous crystallization with a "Krystal" crystallizer involves primarily two variables, operating temperature and recirculation rate. High operating temperatures will increase the product yield per unit of time because of increased rate of evaporation and decreasing heat transfer coefficients also accompany high temperatures. Operating costs will vary with recirculation rate, increasing as pumping costs increase.

The purpose of this investigation was to determine the effect of operating temperature and recirculation rate upon the cost of production of magnesium sulfate in a "Krystal" crystallizer, using a feed stock of saturated magnesium sulfate solution at 150 °F, at operating temperatures from 150 °F to 180 °F and at recirculation rates from 20 to 50 gallons per minute.

II. LITERATURE REVIEW

This section includes information obtained from the literature regarding: sources, uses, physical properties, and thermal properties of magnesium sulfate; the theory of crystallization; and the principle of the "Krystal" crystallizer.

Sources and Uses of Magnesium Sulfate

Magnesium sulfate heptahydrate, hereafter referred to simply as magnesium sulfate, occurs naturally as white, granular, fibrous or earthy masses or in crusts. It is found in Stassfurt, Germany, the Western United States, and Canada[18]. It is manufactured synthetically as a by-product of the manufacture of CO_2 for carbonating drinks, from treatment of dolomitic limestone with sulfuric acid, and by the "Dow"[8] method in which sulfur dioxide is absorbed in magnesium hydrate to form the sulfite and this in turn is blown with air at 50 to 60 ^{o}C in the presence of metal catalysts to oxidize the sulfite to the sulfate. It is impossible to crystallize magnesium sulfate in any form but that of granular crystals in a continuous operation.

Magnesium sulfate is used in pharmacy, dyeing, fireproofing, and as a fertilizer. The pharmaceutical trade[2] always requires that the

product be in the form of needle-shaped crystals, necessitating batch operation to supply this market.

Theory of Crystallization

The Miers theory of a supersaturation curve suggested that there existed a region between the solubility curve of a substance and its supersaturation curve in which crystals would grow but nuclei would not form spontaneously. This region he called the metastable region. Beyond the supersaturation curve nuclei form spontaneously and this area of the temperature-concentration curve he called the labile region[9], see Figure 1, page 5.

In 1934 Ting and McCabe[26] suggested that there existed three regions: the metastable region immediately adjacent to the solubility curve, where crystals would grow but would not nucleate spontaneously; the intermediate range in which crystals would grow and nuclei would form at an increasing rate; and the labile region where nucleation was spontaneous and crystal growth was rapid and copious, see Figure 2, page 6.

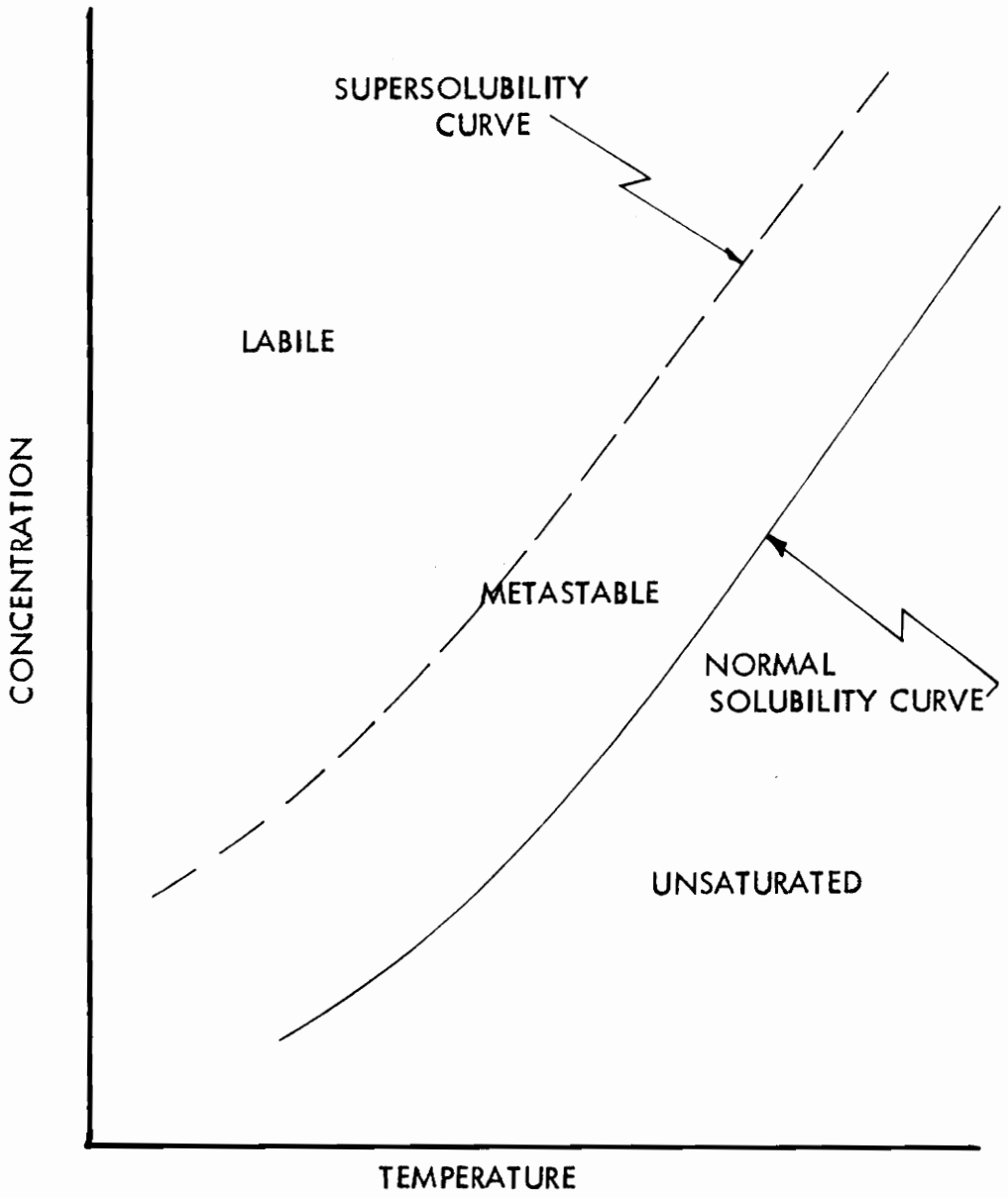


FIGURE 1. DIAGRAMMATIC REPRESENTATION OF MIER'S THEORY

Gloss, G. H.: Magnesium Compounds, "Encyclopedia of Chemical Technology," (Kirk, R. E. and D. F. Othmer, Editors), Vol. 4, pp. 619-636, Interscience Encyclopedia, Inc., New York, N.Y., 1952.

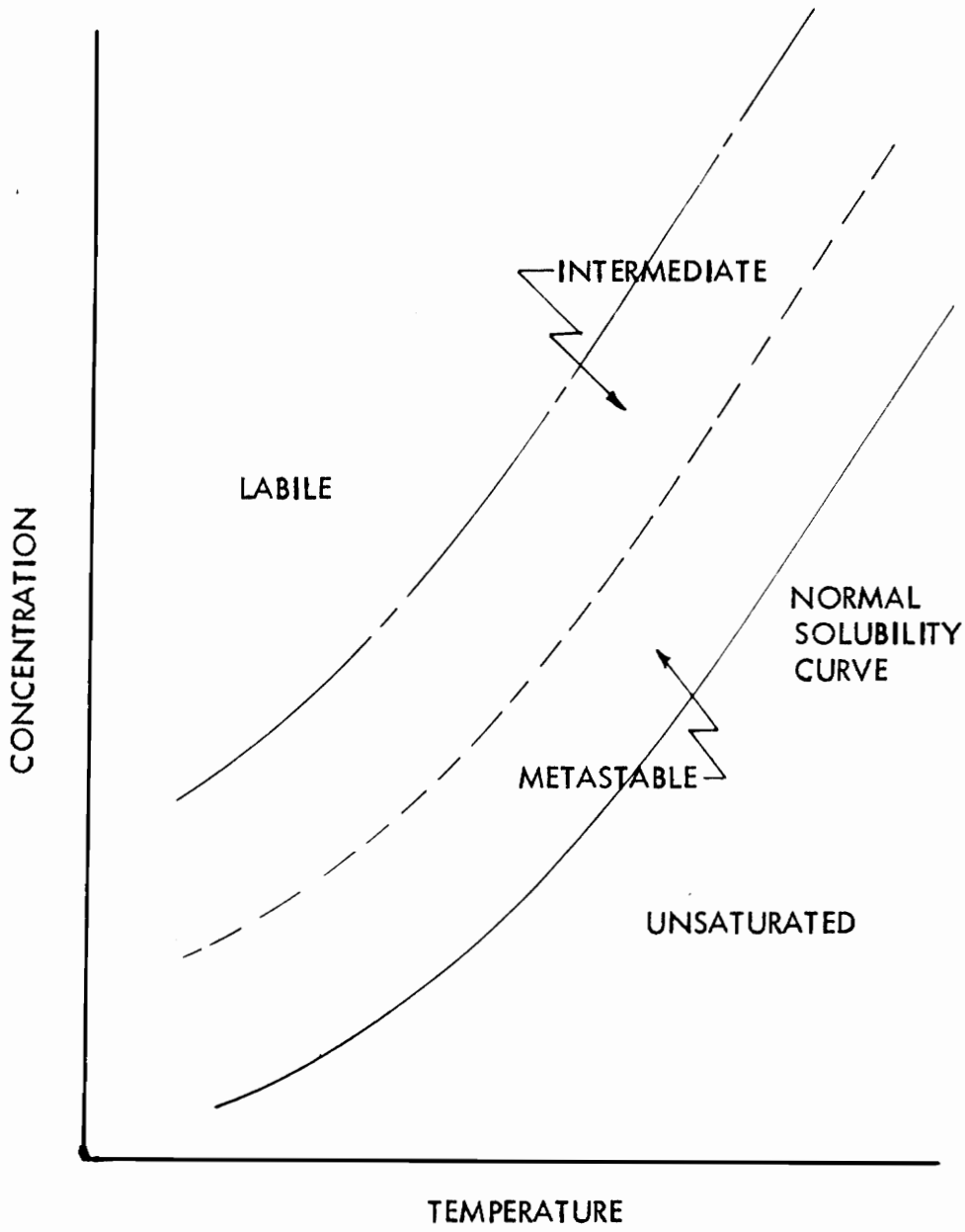


FIGURE 2. DIAGRAMMATIC REPRESENTATION OF TING AND McCABE'S THEORY

Ting, H. H. and W. L. McCabe, Supersaturation and Crystal Formation in Seeded Solutions, Ind. Eng. Chem., 26, 1201-1210 (1934).

The following formula is presented by McCabe[20] for the calculation of the yield of a crystallization process. This equation is valid for either hydrated or anhydrous crystals.

$$C = (R)(100W_o - S(H_o - D))/(100 - S(R-1))$$

where, for any consistent set of units:

- C = Weight of crystals in final magma
- R = Molecular weight of hydrated solute / Molecular weight of anhydrous solute
- S = Solubility (parts by weight anhydrous solute per parts by weight of total solvent) of material at final temperature
- W_o = Weight of anhydrous solute in original batch
- H_o = Total weight of solvent in batch at the beginning of the process
- E = Evaporation during the process.

The heat requirement per unit of product may be calculated from the difference in the amount of heat required to heat the solution to the desired temperature and to vaporize the solvent at the required pressure and the heat released to the system upon crystallization of the product[21].

Physical Properties

Magnesium sulfate[15] is a colorless, crystalline solid with an index of refraction of 1.45, molecular weight of 246.50 and a specific gravity equal to 1.636. Its melting point is difficult to ascertain since, at

elevated temperatures, the water of hydration is evolved and at still higher temperatures the anhydrous form decomposes into its oxides. However, several values for the melting point of the anhydrous magnesium sulfate have been reported[10] ranging from 1127 to 1170 °C.

Magnesium sulfate exists in two forms, the stable rhombic (I) and the unstable monoclinic (II). The monoclinic form exists only under special conditions below 21 °C. The solubility relations of the $MgSO_4$-H_2O system are complex because of the many metastable phases and the slow equilibrium adjustments inherent in it. The various hydrates form supercooled and metastable saturated solutions. For the purpose of this review the data will be omitted. However, a graph[27] illustrating the variation of the solubility and the hydrates formed is presented in Figure 3, page 9.

Ting and McCabe[28] derived an expression which closely approximates the solubility curve of magnesium sulfate in the temperature range 30 to 45 °C. This expression is as follows:

$$S = 0.00360t^3 - 0.3140t^2 + 12.140t - 43.80$$

where:

S = $MgSO_4 \cdot 7H_2O$ solubility, parts per 100 parts
t = Temperature, °C.

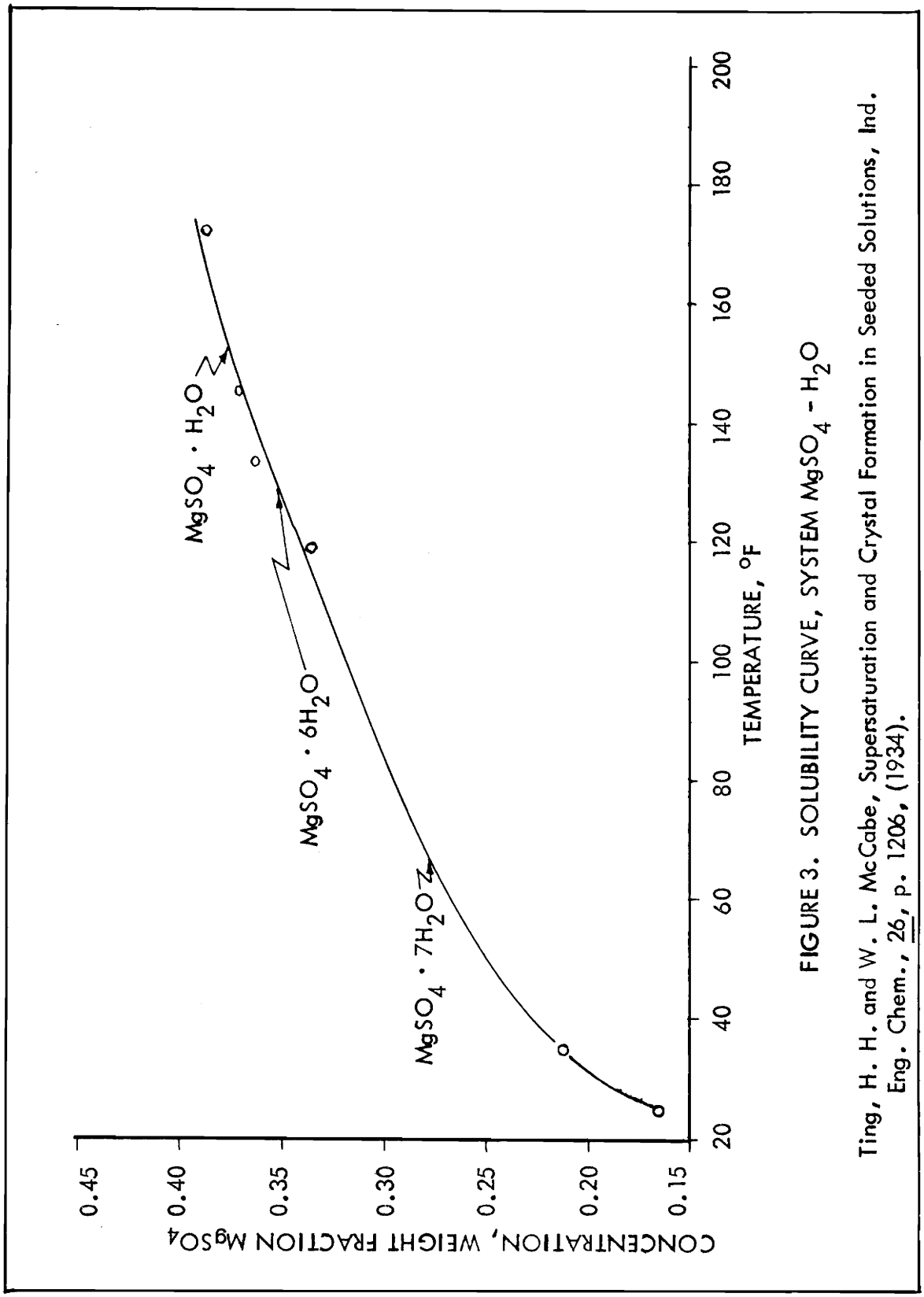


FIGURE 3. SOLUBILITY CURVE, SYSTEM $MgSO_4$ - H_2O

Ting, H. H. and W. L. McCabe, Supersaturation and Crystal Formation in Seeded Solutions, Ind. Eng. Chem., 26, p. 1206, (1934).

Substances exhibiting solubility curves with high positive slopes lend themselves well to crystallization by cooling and those with nearly horizontal curves are best removed from solution by evaporation. Magnesium sulfate, having an intermediate slope, is produced best by a combination of the two effects.

Thermodynamic Properties

The heat of formation of anhydrous magnesium sulfate from its elements is -302,310 cal/gram. The formation[17] of the heptahydrate from water and the anhydrous compound is accompanied by a heat of -24,680 cal/gram.

The heat effect observed during the crystallization of magnesium sulfate is the differential heat of solution (with sign reversed) plus the heat of dilution. Since the heat of solution is ordinarily much higher than the heat of dilution and since data on the heat of dilution for most substances are unavailable, the heat of solution is assumed to be the heat of crystallization. The heat of solution[22] of magnesium sulfate is -3180 cal/gram mole.

Effect of Variables Upon Crystallization

The effects of variables upon the rate and degree of crystallization are included in this section.

Temperature. The effect of temperature upon the size of crystals of magnesium sulfate was found to be negligible by an investigating group[5] at Purdue University. It has also been determined by other investigators[13] that temperature has no effect on the shape of the crystals.

Agitation. Variation of the degree of agitation[16] was found to have no effect on crystal size under specified conditions. Other observers[29] found that up to a certain point, agitation affected the location of the supersolubility curve; beyond this point, it had no effect. Both low frequency[25] and supersonic[24] vibrations were found to markedly affect the rate of crystallization.

Concentration. Concentration was found to exert great influence in determining the size of the crystals. As supersaturation decreased the size of crystals forming increased rapidly[7]. The shape of the crystals was also found to be affected by supersaturation. Grzymek[14] and Vadilo[32] agreed that as supersaturation increased the needle-shaped crystal was favored. Concentration, however, had little effect on the location of the supersolubility curve.

Quantity of Seed Crystals. The weight of seed crystals was found to affect the number of collisions between particles and the amount of surface area available for deposition. This change brought the supersolubility curve closer to the solubility curve[30].

Size of Seed Crystals. Variation of the size of the seed crystals affected the location of the supersolubility curve in a peculiar manner. With 28-mesh crystals the supersolubility curve is located a maximal distance from the solubility curve, but at larger or smaller sizes the curves are closer together[31].

Cooling Rate of Solution. The cooling rate was found to affect the location of the supersolubility curve by moving it away from the solubility curve. This is probably due to the inability of the rate of crystallization to keep up with the drop in temperature.

Hydration and Dehydration

Anhydrous magnesium sulfate is obtained only through dehydration of the hydrated forms[11]. It will hydrate, at 20 °C in moist air, to form the hexahydrate and finally the heptahydrate. The monohydrate, however, transforms directly from the monohydrate to the heptahydrate. In dehydration the heptahydrate always changes to the hexahydrate and

from thence through the subhydrates to the anhydrous form[16]. During hydration the formation of the higher hydrates is catalyzed by the subhydrates[33].

Crystallization Equipment

The "Krystal" evaporator crystallizer is a continuous crystallization apparatus in which the crystals are classified by size. The mother liquor is heated above the saturation temperature in the vaporizer. At the vaporizer the solvent flashes and the mother liquor is both cooled and concentrated to supersaturation[12]. The supersaturated solution falls through a drop pipe to a crystallizing vessel where it contacts crystals and loses its supersaturation. The mother liquor is then recycled through the heater and back to the vaporizer. The crystals are classified by size as they settle to the bottom of the crystallizer where they are withdrawn[23]. To prevent salting-up and to maintain a more constant crystal size, flow in the salt-leg and the salt trap of an evaporative crystallizer is kept at a high rate by recycling slurry either back to the crystallizer itself or to various points in the salt-leg circuit. A slip-stream is taken to a centrifuge for solids removal and mother liquor separation[1].

III. EXPERIMENTAL

This section includes information relating to laboratory material, apparatus, procedure, data obtained, and results calculated.

Purpose of Investigation

The purpose of this investigation was to determine the effect of operating temperature and recirculation rate upon the cost of production of magnesium sulfate in a "Krystal" crystallizer, using a feed stock of saturated magnesium sulfate solution at 150 °F, at operating temperatures from 150 °F to 180 °F and at recirculation rates from 20 to 50 gallons per minute.

Plan of Investigation

The following plan was used in conducting the investigation.

Literature Review. A study was made of the current literature and scientific texts and handbooks on crystallization and magnesium sulfate.

Collection of Data. A study of the literature and equipment was made to determine what range of rates and temperatures could be varied without affecting product quality. The pump was able to absorb a choking from 50

gallons per minute to 20 gallons per minute and still function properly. As indicated in Figure 3, page 9, the only range of temperatures in which evaporative crystallization would be feasible and the composition of product would be constant was from 150 to 180 °F. A thirty-degree range was necessary in order to maintain a temperature differential with the equipment available.

Materials

The specifications, manufacturer and use of the materials used in this investigation are in the following section.

Magnesium Sulfate Heptahydrate. U.S.P. grade, item No 12650. Manufactured by the Drackett Co., Cincinnati, Ohio. Purchased from the U. S. War Department, 1943. Used as solute in preparation of feed stock solution.

Water. Tap; obtained from the water mains of Virginia Polytechnic Institute, Blacksburg, Virginia. Used as solvent in preparation of feed stock solution.

Apparatus

A description of the apparatus used in this investigation is included in the following section.

Beaker. Five-hundred ml capacity, pyrex. Purchased from Fisher Scientific Co., Silver Spring, Maryland. Used for calibration of thermometers.

Centrifuge. Size 17 inch, type Master, 1800 rpm, Number 5001. Manufactured by Fletcher Works, Philadelphia, Pennsylvania. Used in separating product from mother liquor.

Crystallizer. "Krystal." Donated to the Chemical Engineering Department of V.P.I. by E.I. du Pont, Belmont, Virginia. The crystallizer consists of a resonance tank (serial No 73570), heat exchanger (serial No 73571), condenser (serial No 73572), two glass lined tanks (65 gallon capacity), and a steel salt catch tank 20 inches high by 14 inches OD. The resonance tank, heat exchanger and condenser were manufactured by Struthers-Wells, Warren, Pennsylvania, and the tanks by The Pfaudler Company. Used to produce magnesium sulfate monohydrate crystals.

Cylinder. Glass, 2 inch ID x 15 inches long. Purchased from Fisher Scientific Co., Silver Spring, Maryland. Used for specific gravity measurements.

Hot Plate. "Autemp," 115 volts ac, 450 watts. Manufactured by Fisher Scientific Co., Pittsburgh, Pennsylvania. Used for calibration of thermometers.

Hydrometer. Catalog Number 13-1758. Range: 1.20 to 1.42. Manufactured by Fisher Scientific Co., Pittsburgh, Pennsylvania. Used for specific gravity measurements.

Ice. Obtained from the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Virginia. Used for calibration of thermometers.

Mixer. "Lightnin" 250 rpm. Manufactured by the Mixing Equipment Co., Inc., Rochester, New York. Used to mix feed stock solution.

Motor. Induction type APZ, 5 hp, 3 phase, 220/440 volts, 60 cycle, 1740 rpm, serial number 9A8-51-652-804-620. Manufactured by Allis-Chalmers, Norwood, Ohio. Used to power recirculation pump.

Motor. Induction type NPL 324, 15 hp, 3 phase, 220/440 volts, 60 cycle, 38/19 amp, 3525 rpm, serial number 540949. Manufactured by the Continental Electric Co., Newark, New Jersey. Used to power Nash Hytor.

Nash Hytor. Compressor, size AL-574, test number P3919, 3525 rpm. Manufactured by the Nash Engineering Co., South Norwalk, Connecticut. Used to create vacuum in flash chamber.

Pump. Single suction centrifugal, 100 gpm capacity, 1740 rpm, 45 feet head, number 13666. Manufactured by Lawrence Machine and Pump Corporation, 371 Market Street, Lawrence, Massachusetts. Used to recirculate feed stock solution.

Scales. Toledo "One Spot," capacity 800 lb, 0.25 lb increments. Manufactured by the Toledo Scale Co., Toledo, Ohio. Used to weigh solute and product.

Scales. Model 2312, serial number 2315, factory number 2312-0-021 FD, capacity 1125 lb, 0.25 lb increments. Manufactured by the Toledo Scale Co., Toledo, Ohio. Used to measure steam condensate.

Thermometer. Mercury 30 to 300 °F, 2 °F increments. Manufactured by the Fisher Scientific Co., Silver Spring, Maryland. Used to measure feed stock solution temperature.

Thermometers. Gas filled, bayonet type metal, -10 - 110 °C, 1 C° increments, 0 - 220 °F, 2 F° increments, 25 - 125 °F, 1 F° increments. Manufactured by Fisher Scientific Co., Pittsburgh, Pennsylvania. Used to measure temperature of heater condensate, feed stock after heater, flash chamber, cooling water in, cooling water out, vapor condensate, and steam to heater.

Method of Procedure

The following paragraphs describe the method of procedure followed in this investigation.

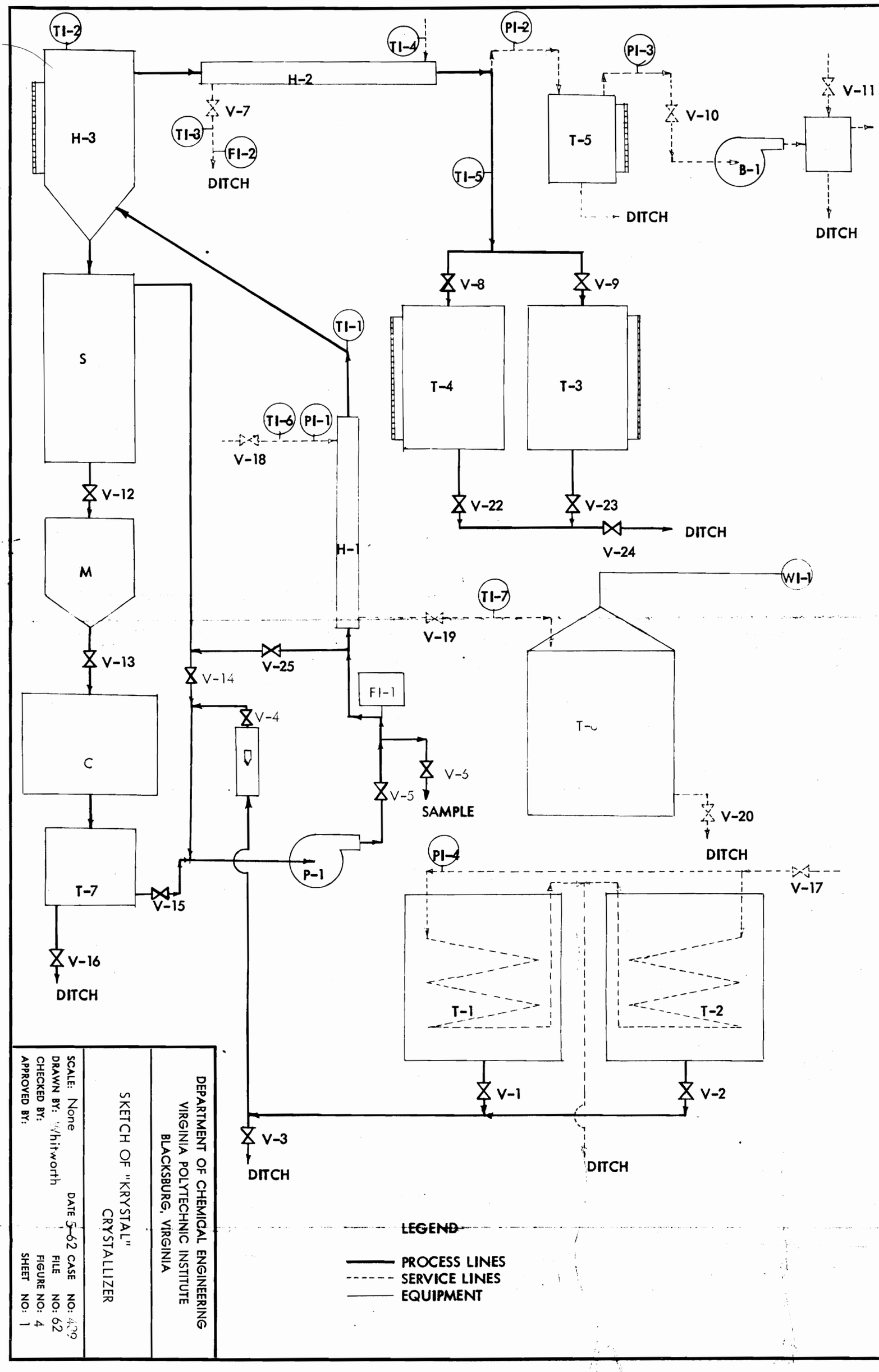
Calibration of Thermometers. The bayonet thermometers used in recording the temperatures at all points indicated in Figure 4, page 20, except the feed stock and preheater condensate, were calibrated by immersion in an ice bath, a beaker of boiling water, and comparison with a mercury thermometer at an ambient temperature of 76 °F. The mercury thermometer was found to be accurate within 0.25 F° (estimated) at the ice and steam points. The data for this calibration may be found on page 25.

Cleaning of Apparatus. The crystallizer was filled with water at 180 °F and recirculated for one hour with continuous feed and draw-off to remove impurities.

Calibration of Flow Indicator. The flow indicator mounted on the equipment was calibrated for a liquid of specific gravity equal to 1.0. It was necessary to recalibrate the instrument for a feed stock of approximately 1.39 specific gravity. This procedure refers to Figure 4, page 20.

The system was filled with water and recirculated with valve V-14 fully opened. The flow at this setting was noted from flow indicator F1-1.

FIGURE 4: SKETCH OF "KRYSTAL" CRYSTALLIZER


TI-2
H-3
TI-4
H-2
PI-2
PI-3
V-11
V-7
TI-3
FI-2
DITCH
T-5
V-10
B-1
TI-5
DITCH
DITCH
V-8
V-9
S
TI-1
T-4
T-3
TI-6
PI-1
V-18
V-12
V-22
V-23
DITCH
V-24
M
H-1
WI-1
TI-7
V-19
V-13
V-25
V-14
FI-1
V-4
T-6
C
V-6
V-5
SAMPLE
V-20
DITCH
PI-4
P-1
V-17
T-7
V-15
T-1
T-2
V-16
DITCH
V-1
V-2
V-3
DITCH
DITCH
LEGEND
PROCESS LINES
SERVICE LINES
EQUIPMENT
DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA
SKETCH OF "KRYSTAL"
CRYSTALLIZER
SCALE: None
DRAWN BY: Whitworth
CHECKED BY:
APPROVED BY:
DATE 5-62
CASE NO: 409
FILE NO: 62
FIGURE NO: 4
SHEET NO: 1

Valve V-14 was then closed until the reading at F1-1 was 80 per cent of maximum. While valve V-14 was at this setting a center punch was used to mark the stem of the valve at a point immediately adjacent to the valve wheel. Upon resetting the valve stem to this position the flow rate could be duplicated. This procedure was followed for 60 and 40 per cent of maximum flow also. The system was then filled with feed stock and the flow readings at F1-1 recorded for the predetermined settings of the stem of valve V-14. The data and curve for this calibration may be found on pages 26 and 27.

Start-up, Steady-state, and Shut-down Procedure. This description refers to Figure 4, page 20. All valves were closed.

Water was added to tanks T-1 and T-2, valve V-17 opened to admit steam to the coils, and enough magnesium sulfate heptahydrate added to make a saturated solution at 150 °F. This solution will hereafter be referred to simply as feed stock. Valves V-1, V-4, V-5, V-9, V-23 and V-24 were then opened and pump P-1 was started. Valves V-11 and V-10 were opened and the Nash Hytor B-1 was turned on. Pumping continued until the flash chamber sight glass indicated a level of approximately 24 inches. Valves V-1 and V-4 were then closed and V-14 opened. Valves V-18 and V-19 were opened to activat e the feed stock heater H-1. Valve V-21 was then opened, V-23 closed, and the Nash

Hytor B-1 was turned on. Valve V-21 was then closed and V-14 adjusted until the desired flow rate was indicated at F1-1. When tank T-3 had filled, V-8 was opened, V-9 closed and V-23 opened. By manipulation of valves V-8, V-9, V-22, V-23 and V-24 and recording the sight glass readings of tanks T-3 and T-4, the amount of vapor removed was observed. When the level in H-3 dropped below the sight glass, V-14 was closed and V-1 or V-2 opened to bring the level back up. Periodically, samples were withdrawn at V-6 and the feed stock tested for specific gravity. When the specific gravity had risen to 1.39, steady-state was assumed. Valve V-12 was then opened and the salt catch tank M was filled. Valve V-12 was closed, the centrifuge C was turned on, and V-13 was opened. The crystals were removed from C and the mother liquor collected in T-7. The mother liquor was put back into the system by opening valve V-15 and closing V-14. When T-7 was empty, V-15 was closed and V-14 reopened. Crystals were collected over a period of time varying from 45 minutes to one hour to establish production per unit of time. When all phases of the experimental work had been performed, the pump P-1 was cut off, B-1 was cut off, valves V-12, V-13, V-16, V-3, and V-20 were opened, and the system drained. All valves were closed.

Collection of Data. The equipment was put into operation in the manner described in the previous paragraph. Valve V-14 was fully opened and V-18 regulated so as to maintain a temperature of 150 °F. When the system appeared to be nearing steady-state, as indicated by specific gravity tests, data taking was commenced. At fifteen-minute intervals the temperature and pressure of the steam to the heater were recorded as well as the temperature and weight of the condensate. The temperatures of the inlet and outlet cooling water and the accumulated flow, the temperature in the flash chamber, of the vapor condensate, the pressure in the condensate line, and the accumulated vapor condensate were recorded. In the flow system itself the temperature of the feed stock after passing through the heat exchanger was monitored, and the total pounds of crystals obtained at each set of conditions was noted.

The procedure for varying conditions was as follows. The recirculation rate was set at 50 gallons per minute and held constant while steady-state was attained at 150, 160, 170, and 180 °F. The flow rate was then decreased to 40 gallons per minute by partially closing valve V-14. This procedure was repeated at flow rates of 30 and 20 gallons per minute.

Static heating data were obtained before operations began. The feed stock was heated from 150 to 180 °F with constant steam pressure and the pounds of condensate noted at the 160, 170, and 180 °F intervals.

Data and Results

The data and results of this investigation are described in the following paragraphs.

Calibration Data. The thermometer calibration data are presented in Table I, page 25. Seven bayonet thermometers were calibrated and found to be in error not more than 3 °F. The flow indicator measuring recirculation rate was calibrated for a feed stock of specific gravity equal to 1.39 (H_2O = 1.0). These data and the calibration curve are included in Table II, page 26 and Figure 5, page 27, respectively.

Production Tests. The experimental data obtained from the production of magnesium sulfate monohydrate are presented in Table III, page 28. This table includes operating temperature and flow rates; operation time and amount of product obtained; the pressure in the vacuum and steam lines; the weights of steam condensate, cooling water and vapor condensate; and the temperatures of the heating steam, steam condensate, flash chamber, vapor condensate and cooling water. The steam necessary to heat 1450 pounds of feed stock from 150 to 180 °F may be found in Table IV, page 29.

Production Costs. The results of this investigation indicating the costs per pound of crystals produced are included in Table V, page 30. Figures 6 and 7, pages 31 and 32, indicate the variation of cost per pound of crystals produced with flow rate and temperature.

TABLE I

Calibration Data for Thermometers Used in Measuring Temperatures at the Condenser, Heater and Flash Chamber of the "Krystal" Crystallizer

Location on Crystallizer	Thermometer	Thermometer Reading at 32 °F/0 °C	Thermometer Reading at 76 °F/24.4 °C	Thermometer Reading at 209 °F/99.5 °C
Steam to Heater, °F	1	30	74	209
Condensate from Heater, °C	2	1	25	97
Cooling Water Out, °F	3	32	74	--[a]
Feed Stock (after heater), °F	4	35	78	207
Flash Chamber, °C	5	0	24	97
Vapor Condensate, °F	6	34	77	208
Cooling Water In, °F	7	32	75	207

[a]Maximum temperature of thermometer number 3 was 125 °F.

TABLE II

Calibration Data for Flow Indicator Measuring Recirculation Rate

Test No.	Flow Indicator Reading, gal/min	Flow Rate gal/min
1	70	50
2	56	40
3	42	30
4	28	20

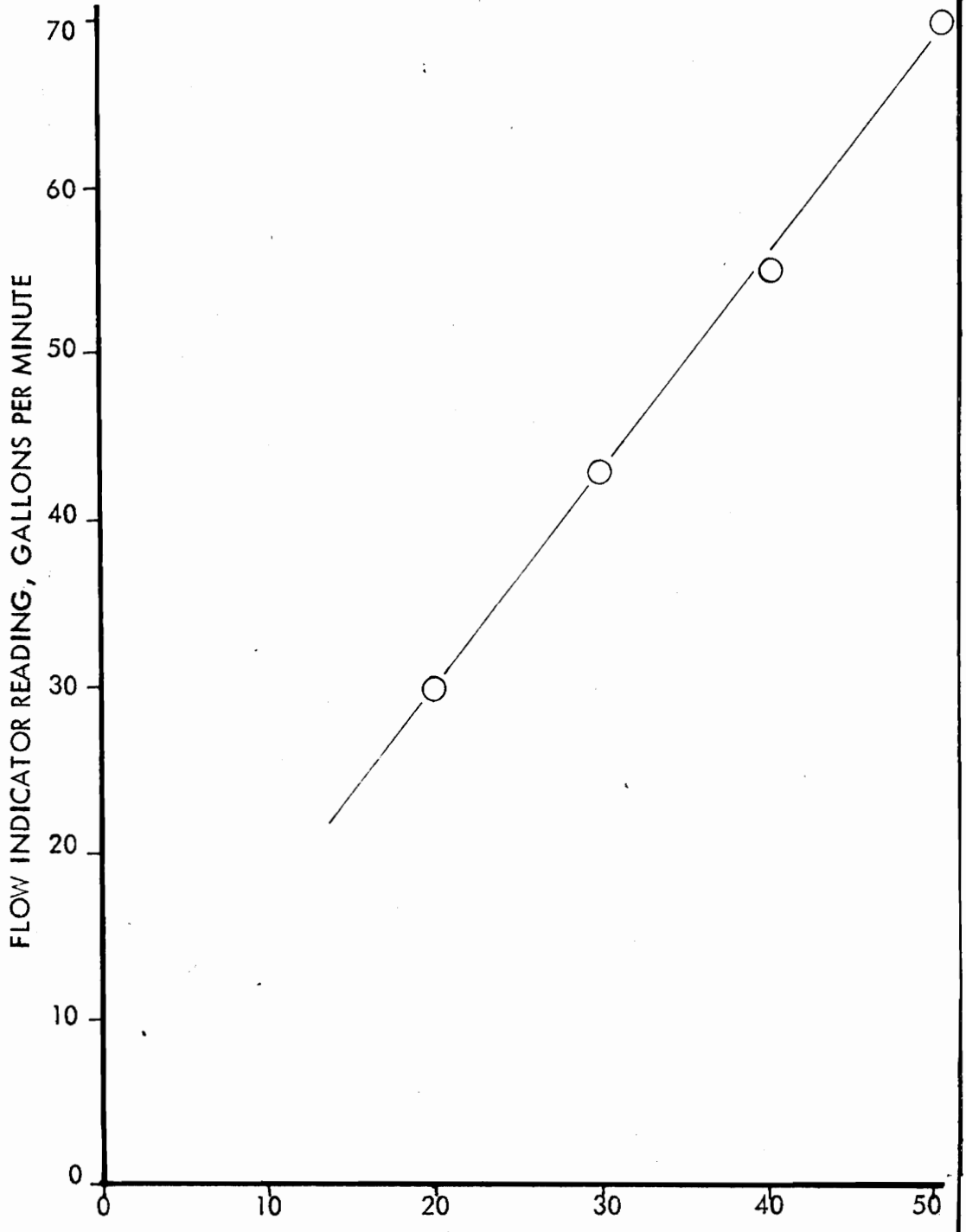


FIGURE 5. CALIBRATION CURVE FOR FLOW INDICATOR MEASURING RECIRCULATION RATE

TABLE III

Data Obtained During Operation of a "Krystal" Crystallizer

Using the System Magnesium Sulfate - Water

TABLE III

Data Obtained During Operation of a "Krystal" Crystalliser Using the System Magnesium Sulfate - Water

Operating Temperature	Time	Flow Rate	Temperatures: Steam to Heater	Condensate from Heater	Feed Stock (after Heater)	Flash Chamber	Vapor Condensate	Cooling Water Out	Cooling Water In	Pressures: Vacuum Line	Steam to Heater	Accumulative Flows: Steam Condensate	Vapor Condensate	Cooling Water	Specific Gravity	Product
°F	min	gpm	°F	°C	°F	°C	°F	°F	°F	in. Hg	psi	lbs	lbs	lbs	H_2O = 1.0	lbs
						Test I										
150	30	50	210	50	150	58	105	65	50	25	--	14	114	333	1.39	20.5
160	30	50	230	78	160	60	110	72	50	24	4	41	139	350	1.39	25
170	45	50	240	84	171	65	114	75	50	22	9	91	224	558	1.39	43
180	45	50	248	90	181	70	118	77	50	21	15	105	327	500	1.39	52
						Test II										
150	30	40	209	51	148	58	99	60	50	25	--	10	82	350	1.39	18
160	45	40	229	78	160	62	107	72	50	24	4	52	176	466	1.39	32
170	30	40	242	85	172	65	113	76	49	23	8	86	143	350	1.39	38
180	45	40	247	91	181	71	118	79	49	22	14	122	302	541	1.39	58
						Test III										
150	30	30	210	50	150	58	99	63	49	25	--	8	82	358	1.39	15
160	45	30	230	71	159	61	106	72	49	23	3	41	147	525	1.39	27
170	45	30	241	82	168	65	112	75	49	22	7	70	204	533	1.39	41
180	45	30	248	91	180	71	118	79	50	21	13	85	257	508	1.39	65
						Test IV										
150	30	20	211	49	152	60	100	65	49	25	--	11	82	300	1.39	17
160	30	20	230	70	160	63	108	72	49	23	3	30	98	342	1.39	24
170	45	20	241	84	170	65	111	75	49	22	7	67	208	550	1.39	42
180	45	20	249	91	179	70	118	79	49	21	13	74	245	491	1.39	63

TABLE IV

Pounds of Steam Required to Preheat 1450 Pounds of Feed Stock

Temp of Stock, °F	Steam Pressure, lb/sq in	Accumulative Condensate, lbs	Temp of Condensate, °F
150	--	--	--
160	5	15.25	190
170	5	31.5	190
180	5	48.5	190

TABLE V

Cost Per Pound of Magnesium Sulfate Monohydrate Produced

Flow Rate gpm	Operating Temperature °F	Power Cost mills/lb	Steam Cost mills/lb	Total Cost mills/lb
50	150	1.058	0.296	1.354
50	160	0.867	0.718	1.585
50	170	0.757	0.932	1.689
50	180	0.625	0.896	1.521
40	150	0.965	0.241	1.206
40	160	0.814	0.705	1.519
40	170	0.457	0.996	1.453
40	180	0.449	0.934	1.383
30	150	0.866	0.231	1.097
30	160	0.722	0.666	1.388
30	170	0.475	0.754	1.229
30	180	0.300	0.588	0.888
20	150	0.510	0.280	0.790
20	160	0.362	0.550	0.912
20	170	0.310	0.706	1.016
20	180	0.207	0.530	0.737

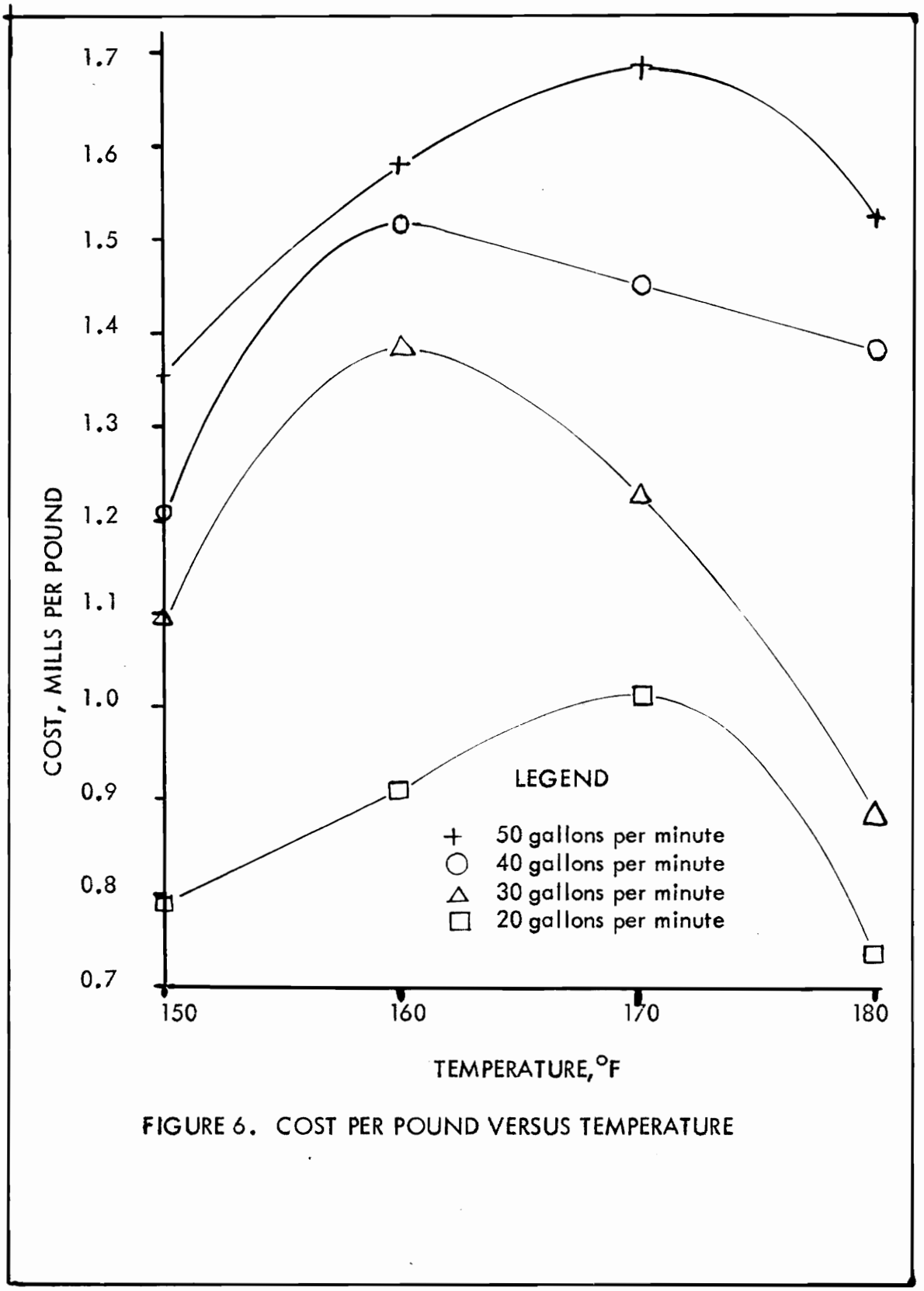


FIGURE 6. COST PER POUND VERSUS TEMPERATURE

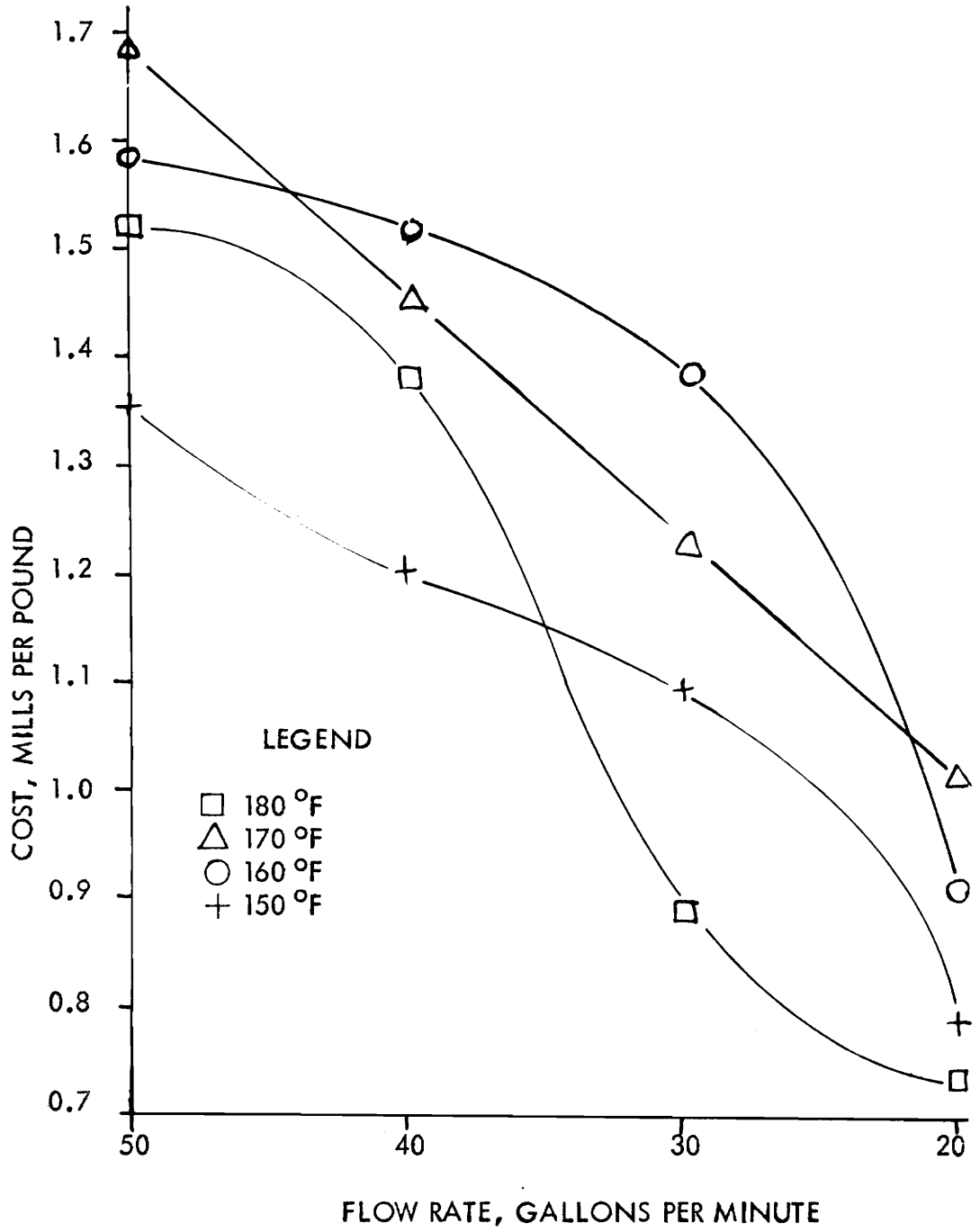


FIGURE 7. COST PER POUND VERSUS FLOW RATE

Sample Calculations

The following calculations are samples of the calculations used in this investigation. Static heating costs were calculated from data in Table IV, Part B, page 29. All other sample calculations are from data appearing in Table III, Test I, Part B, page 28.

Determination of Power Costs[19]. The power cost required for pumping a volume of liquid necessary to obtain one pound of product was calculated from the following equation:

$$A = \frac{(UC)\left(\quad R \quad\right)\left(\right).00315)(s)(H)}{(16.66)\ (PE)\ (ME)\ (P)}$$

where:

A = Cost of power, mills/lb of product

UC = Unit cost of power, mills/Kwhr

R = Recirculation rate, gal/min

s = Specific gravity, (H_2O = 1)

H = Total head, ft

PE = Pump efficiency

ME = Motor efficiency

P = Weight of product, lbs/hr

The efficiency of the pump was assumed to be 0.45 and that of the motor 0.80. From the literature[3] UC = 11.9 mills/Kwhr. Total head for the equipment was 10 ft.

$$A = (11.9)\frac{(50)\ (0.00315)(1.39)(10)}{(16.66)(0.450)(0.800)(25)(2)}$$

$$= 0.867 \text{ mills/lb of product}$$

Determination of Static Heating Steam Costs. The cost of steam necessary to heat an amount of feed stock containing one pound of product was calculated from the following equation:

$$B = \frac{(SC)(L)(M)}{W}$$

where:

B = Cost of heating, mills/lb product

SC = Unit cost of steam, mills/lb steam

L = Weight of condensate, lbs

M = Weight of feed stock containing one pound of product, lb/lb

W = Total weight of feed stock, lb

The unit cost of steam was 0.434 mills/lb[4].

$$B = \frac{(0.434)(15.250)(1.520)}{1450}$$

$$= 0.007 \text{ mills/lb product}$$

Determination of Dynamic Heating Cost. The cost of steam necessary to maintain the feed stock at the required temperature was calculated from the following equation:

$$D = \frac{(L)(SC)}{P}$$

where:

D = Cost of heating, mills/lb product

L = Weight of condensate, lb

SC = Unit cost of steam, 0.434 mills/lb steam

P = Weight of product, lb

$$D = \frac{(41)(0.434)}{25}$$

$$= 0.711 \text{ mills/lb product}$$

Determination of Total Cost. The total operating cost per pound of product was calculated from the following equation:

$$C = A + B + D$$

where:

C = Total cost, mills/lb product

A = Power cost, mills/lb product

B = Static heating cost, mills/lb product

D = Dynamic heating cost, mills/lb product

$$C = 0.867 + 0.007 + 0.711$$

$$= 1.585 \text{ mills/lb product}$$

IV. DISCUSSION

A discussion of the results obtained in this investigation, recommendations for future study, and the limitations of the investigation are included in this section.

Discussion of Results

The purpose of the study of the operating conditions of a "Krystal" crystallizer was to optimize the equipment with respect to recirculation rate and operating temperature. Murphy [34] had suggested that the optimum operating temperature would be the highest tested. There was some doubt, however, about the magnitude of the effect of rate and temperature upon heat transfer to the medium, radiation to the atmosphere, and heat of vaporization. The effects of these factors were rather surprising. The prediction was made that efficiency would increase with rising temperature and decrease with rising flow rate.

With regard to flow rate, the cost decreased with decreasing rate. This was expected since at lower flow rates the pumping costs decrease. However, as indicated by the data in Table V, page 30, the steam cost decreases as the rate decreases (i.e., from 0.718 mills per pound at

50 gpm to 0.550 mills per pound at 20 gpm at 160 °F). Therefore, the heat transfer must improve with decreasing rates and the improvement of cost versus product is not solely a function of decreasing power requirements.

Variation with temperature indicated a maximum for intermediate temperatures, at all flow rates. Only at recirculation rates of 40 and 50 gpm is the cost at 180 °F greater than at 150 °F. However, at flow rates of 20 and 30 gpm the cost per pound of product at 180 °F appears to be approaching a minimum, whereas the cost per pound at 150 °F is decreasing rapidly. At flow rates of 15 and 10 gpm it is believed that the lower temperature would be optimum. To go further, it may be said that maximum efficiency would be reached when the flow rate is equal to the rate of evaporation.

It may be seen from Figure 6, page 31, that at all flow rates the cost versus temperature curve indicates a maximum at temperatures of 160 and 170 °F. This suggests that at a given flow rate the heat transfer from the steam to the feed stock is greater at 150 °F because of the high temperature drop across the heat exchanger wall; and at 180 °F the heat of vaporization is less, thereby lowering the heat requirement to maintain operating temperature.

Within the scope of this investigation the most economical operating conditions are a recirculation rate of 20 gallons per minute and an

operating temperature of 180 °F. At these conditions magnesium sulfate monohydrate may be produced at a cost of 0.737 mills per pound. Between the extremes of the data represented in Table V, page 30, a savings of 0.952 mills per pound of product may be realized by choice of the correct operating temperature and recirculation rate.

Magnesium sulfate monohydrate is not stable at room temperatures, going rapidly to the heptahydrate if its temperature drops below 150 °F and moisture is present. In order to verify the purity of the product, a small sample of the product was placed in an oven before it had time to cool. The mother liquor water evaporated quickly and the weight of the sample remained constant up to the temperature of the decomposition of the monohydrate.

The data presented in Table V, page 30, and Figures 6 and 7, pages 31 and 32, were connected by the best curve rather than straight lines. This was done because of the complexity and therefore non-linearity of the variables associated with the investigation.

The solubility curve, Figure 3, page 9, suggested that the only temperature range suitable for vaporization crystallization in which the product composition would be constant was from 150 to 180 °F.

Recommendations

The recommendations made as a result of this investigation are included in the following paragraphs and should be considered in additional experimentation.

Selection of Solute. A solute should be selected whose temperature-solubility curve has either a negative slope or a very slightly positive slope (i.e., Na_2SO_4). The feed lines and valves will freeze up and present a considerable problem if a temperature sensitive substance such as magnesium sulfate is used.

Lower Flow Rates. Without variation efficiency increased with decreasing flow rates. Lower rates should be tried to see if this tendency persists.

New Pump. A new pump should be installed or the present one overhauled before the crystallizer is used again.

Personnel. No one man should attempt to operate the equipment. To efficiently control and operate the apparatus, at least two and preferably three men should be used.

Modification of Equipment. A pitot tube, manometer or other suitable flow measuring device should be placed downstream from the pump in order to take advantage of the by-pass valve presently installed.

Heating Coil Repair. The heating coil in the left-hand feed tank should be repaired.

Basis of Analysis. The data should be analyzed on the basis of cost per hour as well as cost per pound.

Limitations

The investigation was carried out under the following limitations.

Apparatus. The tests were made with a "Krystal" crystallizer, using a 5 hp pump as recirculator. A Nash Hytor compressor was used to evacuate the flash chamber to a maximum vacuum of 26 inches of Hg and a 17-inch industrial centrifuge was used for collection of product.

Temperature and Flow Rate. Operating temperature was restricted to the range 150 to 180 ^{o}F and recirculation rate from 20 to 50 gallons per minute. Sixteen tests were conducted, one at each combination of rate and temperature in steps of ten.

Materials. The only form of magnesium sulfate available was the heptahydrate. All solubility data was based on the anhydrous form. A saturated solution of magnesium sulfate at 150 ^{o}F was used as feed stock.

Scope of Study. All costs which would be essentially the same at any set of operating conditions such as labor, depreciation, and cooling, were not taken into account.

V. CONCLUSIONS

A study of the effect of recirculation rate and operating temperature upon the cost per pound of magnesium sulfate monohydrate was made under the following conditions:

The operating temperature varied from 150 to 180 °F. The recirculation rate varied from 20 to 50 gallons per minute. The sixteen tests were made for duration of 45 minutes to one hour each. The ambient temperature varied from 74 to 78 °F. The crystallizer used was of the "Krystal" type using a 5 hp pump as recirculator and a Nash Hytor compressor to create a vacuum of 26 inches of mercury. The feed stock was a saturated solution of magnesium sulfate at 150 °F. Only costs which varied with operating conditions were considered.

The tests made under the above conditions led to the following conclusions:

1. The cost of production of magnesium sulfate monohydrate crystals decreased as the recirculation rate decreased.
2. No trend was indicated upon variation of operating temperature.

VI. SUMMARY

A study was made of the cost per pound of magnesium sulfate monohydrate produced by a "Krystal" crystallizer at recirculation rates from 20 to 40 gallons per minute and operating temperatures from 150 to 180 °F. From these tests the optimum operating conditions within the range of these variables were derived.

The problem was studied by operating the equipment at all sixteen combinations of these variables in unit steps of ten. The crystallizer was operated at each set of conditions, the product collected, and the cost per pound determined.

The operating conditions were found to affect power and steam costs greatly, varying from 1.689 mills/lb at 50 gallons per minute and 170 °F to 0.737 mills/lb at 20 gallons per minute and 180 °F, representing a decrease of 0.952 mills per pound upon correct choice of operating temperature and recirculation rate.

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Addendum

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To his wife, Katherine, who typed this thesis for the ridiculously low price of one ocelot coat, the author extends his most loving thanks.

IX. VITA

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