

PRELIMINARY DESIGN AND FEASIBILITY STUDY  
FOR A CALCIUM-MAGNESIUM ACETATE UNIT

FINAL REPORT

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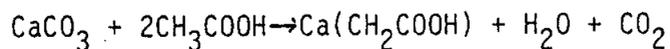
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## FOREWORD

Fundamental regard for winter time driving safety has resulted in an extensive and continuing application of NaCl and CaCl<sub>2</sub> de-icing salts to the road surface. The de-icing benefit of these materials is however, negatively offset by secondary costs due to metal corrosion and pollution effects. Literature sources place the total cost of salt usage at approximately 10-15 times that of the salt product itself. The public's objection to secondary costs such as rusty automobile fenders often provides a pungent criticism of roadway salting practices. In addition, corrosion of bridge reinforcing steel is a problem of great magnitude to the highway engineer.

National research into alternative de-icing compounds has led to recognition of Calcium Magnesium Acetate (CMA) as the most viable substitute for NaCl and CaCl<sub>2</sub>. CMA provides an ice melting capability similar to chloride salts while raising little or no corrosion potential for most steel or aluminum vehicle components. Furthermore, CMA has been shown to be a safe, non-toxic substance with respect to animal and plant life.

CMA is fairly easily produced by the action of acetic acid on calcium carbonate (limestone) as indicated below:



Details of critical reaction parameters and a chemical process design are presented within this report.

The extremely high cost of CMA has prevented its use in all but limited testing situations. CMA is commercially available only in laboratory reagent grade as either the pure calcium or pure magnesium acetate. These products are intended for laboratory usage and are priced at many dollars per pound. This study was initiated to investigate production methods and economics associated with making a low grade of CMA from raw materials readily available to a potential Alaskan producer.

This report evaluates the small scale production (14 - 72 tons per day dry

weight equivalent) of a "highway grade" CMA solution. Findings indicate that CMA becomes economical at production levels of more than 17 tons per day. It was calculated that the cost of CMA would range between \$290 and \$590 per ton depending on rate of manufacture and the base price of acetic acid. These figures compare very favorably with the current cost of  $\text{CaCl}_2$  used by the Alaska DOTPF of almost \$650 per ton (F.O.B. Fairbanks), especially in view of secondary damage attributable to the chloride salt.

An interesting point is emphasized by the authors regarding potential economic ramifications of Alaskan CMA. It is reasonable to expect that petroleum derived, low grade, low cost acetic acid can be made available within Alaska as a result of oil related industry. Under this circumstance, with acetic acid costs possibly below \$1.00 per gallon, larger scale production of CMA could become a significant Alaskan enterprise. It is further conceivable that Alaska could eventually become a major producer and exporter of low grade CMA to a national or international market.

As project manager I would recommend a course of action leading to manufacture and use of CMA as a de-icing agent in Alaska. In view of the promising nature of this report's findings, work is continuing with construction of a small "bench scale" pilot plant, a refining of the process design and field trials of experimentally produced CMA. An important first-trial of CMA will involve treatment of a roadway sand stock pile to prevent freezing.

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## ABSTRACT

The adverse environmental effects and corrosion problems associated with the use of chloride salts as de-icing agents have prompted a search for alternative de-icing compounds. Calcium and Magnesium Acetates (CMA) exhibit excellent de-icing characteristics yet are not corrosive or harmful to the environment.

A viable process design for the production of CMA has been developed based on the results of a series of kinetic reaction experiments conducted at the University of Alaska. Acetic acid and native Alaskan limestones were used as the raw materials. An economic evaluation of the process indicates a selling price of less than \$600/ton of solid CMA, based on the production of a saturated, aqueous CMA solution in small scale facilities (10,000 - 50,000 gallons/day). At the upper range of production rates studied (50,000 GPD) and for an acetic acid cost of \$1.25/gallon, the calculated CMA price was \$290/ton of solid. This represents a minimum price and is attractive when compared with the cost of other de-icing compounds.

The results of this cooperative project between the Petroleum Engineering Department at the University of Alaska, Fairbanks and the State D.O.T. are highly encouraging. With the use of native Alaskan limestone and acetic acid, the process economics point toward a high quality, competitively priced product. The environmental advantages of CMA over chloride salts and reduced secondary costs due to lower corrosion rates for vehicles and bridges indicate that CMA may become the premier de-icing agent.

## CONCLUSIONS

The project has resulted in a number of original findings. The de-icing and reaction characteristics using Alaskan limestone have been well established.

A process design and economic evaluation has been presented. The prices of CMA vary from \$590/ton for a 10,000 gallon per day plant at \$2.50 per gallon of acetic acid to \$290/ton for a 50,000 gallon per day plant at \$1.25 per gallon of acetic acid. These prices are within the ranges of present de-icing compounds.

The reaction scheme and vessels that we have used thus far were simple, primarily intended to demonstrate the viability of the process and the effectiveness of the ensuing product. The process design is presently in the block diagram stage while the process equipment was sized for a feasibility analysis. The economic evaluation has resulted in a plausible price for the product.

While the scope of this work had in mind Alaskan conditions and potential Alaskan (localized) uses, its findings may prove useful to a private venture. The remoteness of Alaskan locations and the small size of the demand suggested the size of the plants.

However, the abundance of Alaskan limestone and natural gas that can easily be converted to acetic acid (methane-methanol-acetic acid) may provide the impetus for a major industry, especially if CMA becomes a dominant factor in de-icing. This subject is, though, beyond the scope of our work.

Future research needs will involve the construction of a bench scale pilot plant using a continuous trickle-bed reactor and a wide surface test application of the material, in Fairbanks, as soon as the weather permits. This work is dependent on additional funding.

## INTRODUCTION

Road and airport runway de-icing are serious problems in the northern latitudes. Sodium and calcium chlorides have been used successfully for many years as de-icing agents. However, adverse environmental effects and corrosion problems associated with chloride salts have prompted a search for alternative, cost-effective, de-icing agents. (1,2,3,4)

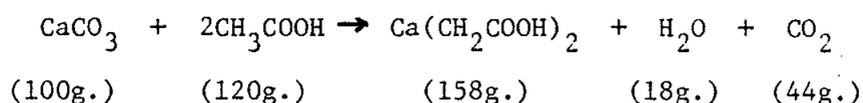
A report entitled "Alternate Highway De-icing Chemicals" from the Bjorksten Research Laboratories, Inc. presented a review of the de-icing characteristics of various compounds. Two candidates were selected which proved economical, and more importantly, less corrosive than NaCl.

The first, methanol, was found to be "less persistent" due to its low flash point. The second, a mixture of calcium and magnesium acetate (CMA) was found to exhibit superior de-icing characteristics at a competitive price. In contrast to NaCl, CMA is a corrosion inhibitor, is beneficial to most soils, and has no potential for harming drinking supplies. Hence, CMA does not exhibit many of the "extended costs" associated with the application of NaCl.

CMA can be produced by the simple dissolution of naturally occurring carbonates in acetic acid. These carbonates are readily available in the form of native limestones and dolomite. We have discovered that, for economic and technical reasons, the production of calcium acetate (CA) alone is preferred. This subject is dealt with in a later section of this report.

## REACTION EXPERIMENTS

Calcium acetate is formed by the dissolution of calcium carbonate in an acetic acid solution. Water and carbon dioxide are byproducts. The stoichiometric equation is given below:



The reaction is slightly exothermic ( $\Delta H_r^0 = -4.6\text{kcal/gmol.}$ ). The solubility limit of CaAc (calcium acetate) is 28% by weight. Hence, if a saturated solution of CaAc is desired, a simple material balance indicates a water requirement of 388 g.  $\text{H}_2\text{O}/100\text{g CaCO}_3$ .

A series of experiments was undertaken to define the process kinetics and ultimate degree of completion functions of the reaction temperature, reactant concentrations, and extent of particle size reduction for the calcium carbonate. Alaskan limestone delivered from a quarry near Livengood, Alaska, and laboratory acetic acid were used as reactants.

The reaction behavior of limestone and acetic acid is reported in Figure 1. The reactions were conducted in a simple stirred tank reactor. At a given residence time, there was no discernible difference between the various water to limestone ratios, as long as enough water was present to provide an unsaturated solution of calcium acetate at the conclusion of the reaction. This minimum ratio of 4 to 1 on a mass basis proved inhibiting at high acetic acid excess. This result is to be expected since the reaction is based on the dissociation of acetic acid. An insufficient amount of water results in inadequate dissociation of acetic acid.

Since various water to limestone ratios above 5:1 (for brevity not all results are plotted on Figure 1.) did not exhibit any discernible difference in the reaction completion rate, the entire subsequent set of experiments was done at a 5:1 weight ratio of water to limestone.

A significant conclusion can be drawn from the above. The quality of the acetic acid does not have to be highly refined. Industrial grade acetic acid (95% purity) which is significantly less expensive than reagent grade, can be

(5)

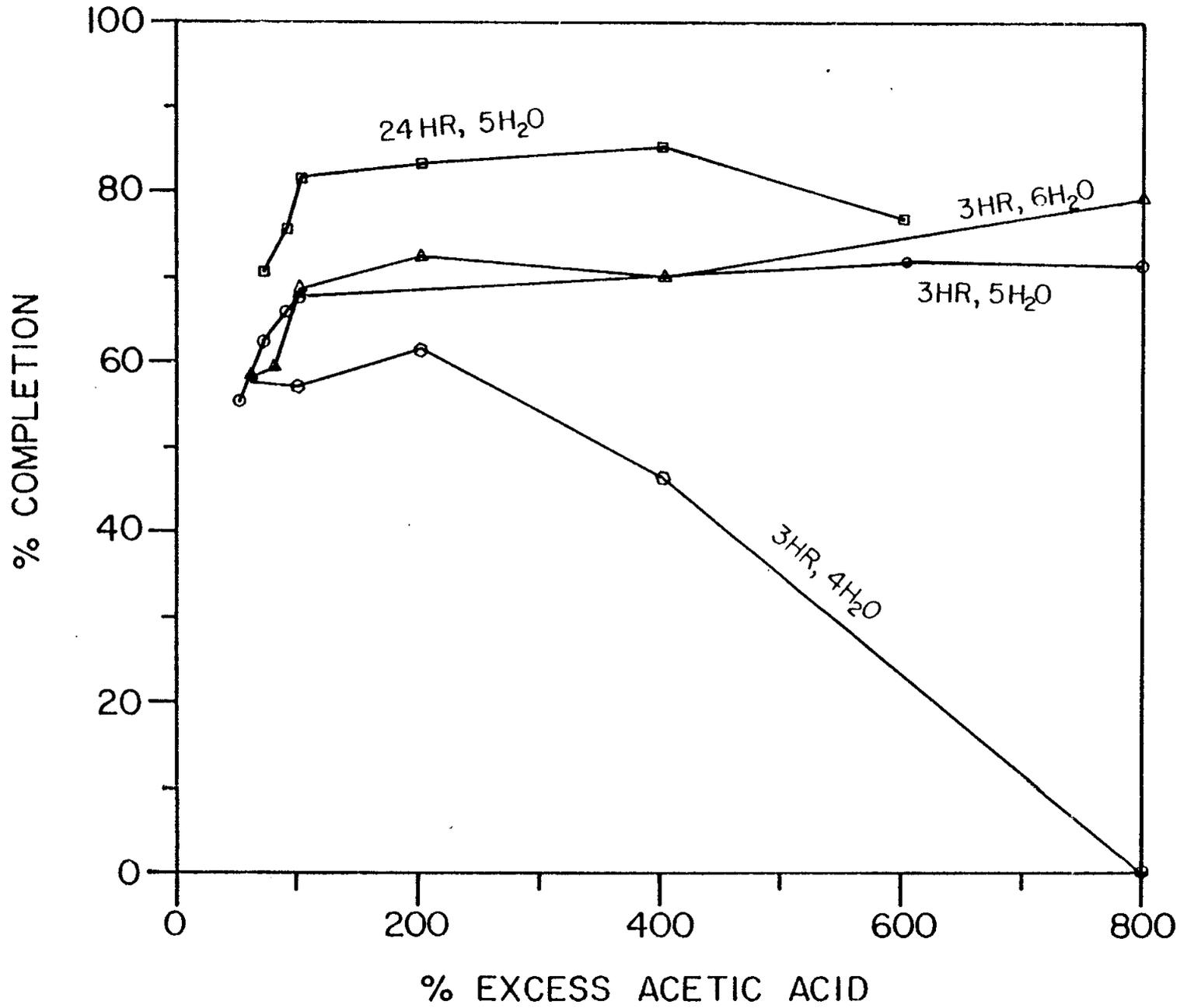


Figure 1. Reaction Behavior of Limestone and Acetic Acid: Percent Completion vs. Excess of Acid at Various Residence Times and Water Content.

used since it will have to be diluted to 50% in any event.

There is another major benefit out of the findings. Liquid phase oxidation (LPO) of waste cellulose (or methanol carbonylation) produce acid at about the optimal 50% concentration. Both methods of production could be implemented in Alaska. Especially attractive is the methanol carbonylation since several industrial concerns are contemplating methanol production in the state. The supply of carbonate for the acetate process seems assured. The state houses large deposits of limestone and dolomite.

Figures 2 through 5 present a comprehensive picture of the reaction results. The series of experiments were intended to gauge the effects of excess acid, reaction temperature and limestone particle sizes on the completion of the reaction. The results are for batch reactions with a three hour residence time.

The amount of excess acetic acid did not appear to affect the reaction completion significantly. Hence, acid consumption can be held at a manageable level.

Neutralization of acetic acid by NaOH can be considered. However, a further economic evaluation may point toward an associated acid recovery unit. An extraction step using an appropriate solvent, followed by a distillation step may be indicated.

The effect of the temperature is significant. An increase in the reaction temperature from room level of 20°C to 60°C results in an increase in the rate of completion of over 20%. A similar effect was observed in using more finely ground limestone. The "first" and "second" crushing that appear in Figures 2 through 5 refer to two consecutive outputs of a commercially available rock crusher. Both of the last two findings will have an effect on the economic optimization of the plant. The beneficial effect on the completion rate must be balanced against the operating and energy costs. Finally, the residence time in the reactor appears significant. A 20% difference in the reaction completion was observed if a reaction time of 24 hours was used instead of 3 hours.

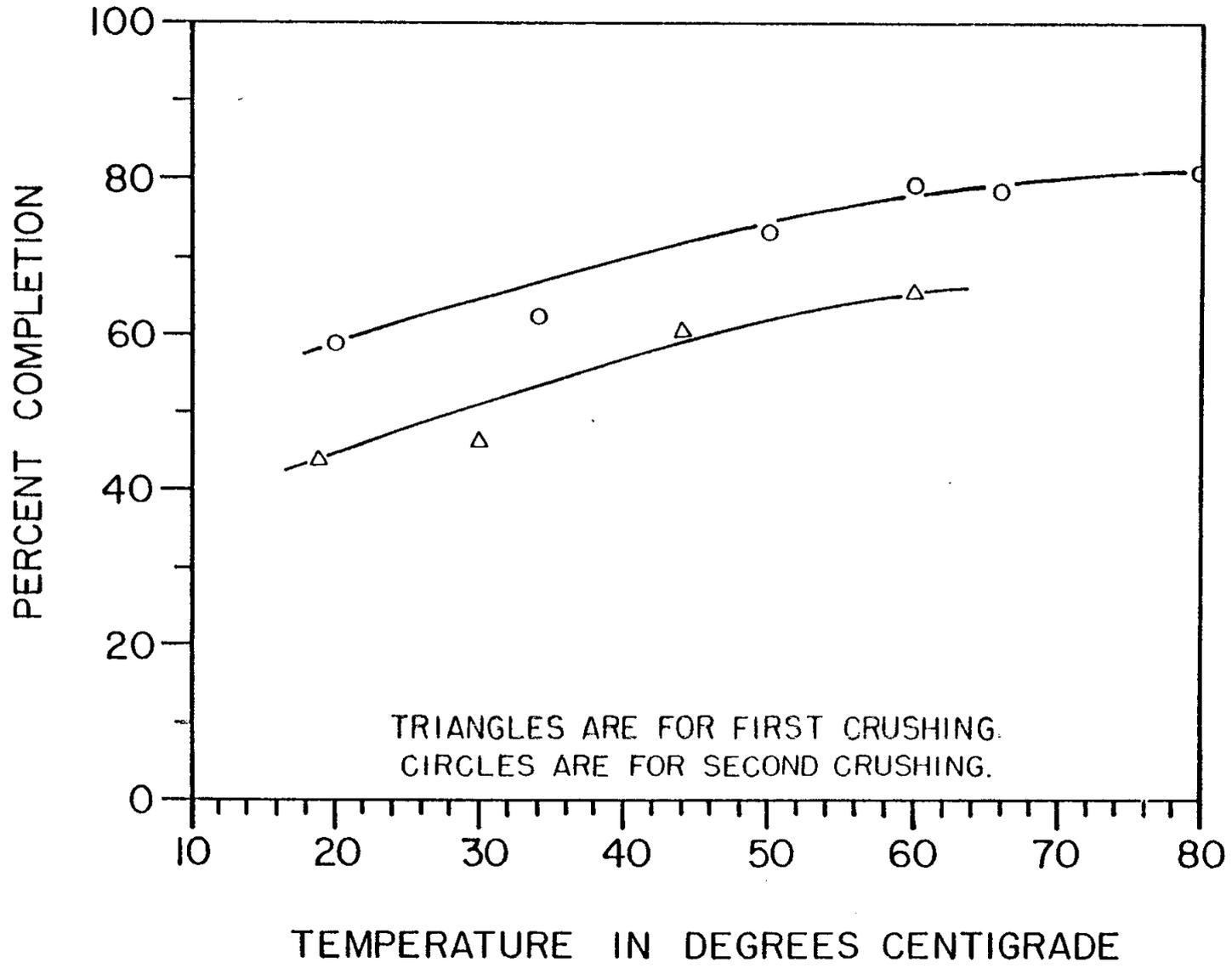


Figure 2. Reaction Performance for Limestone and 0% Excess Acetic Acid

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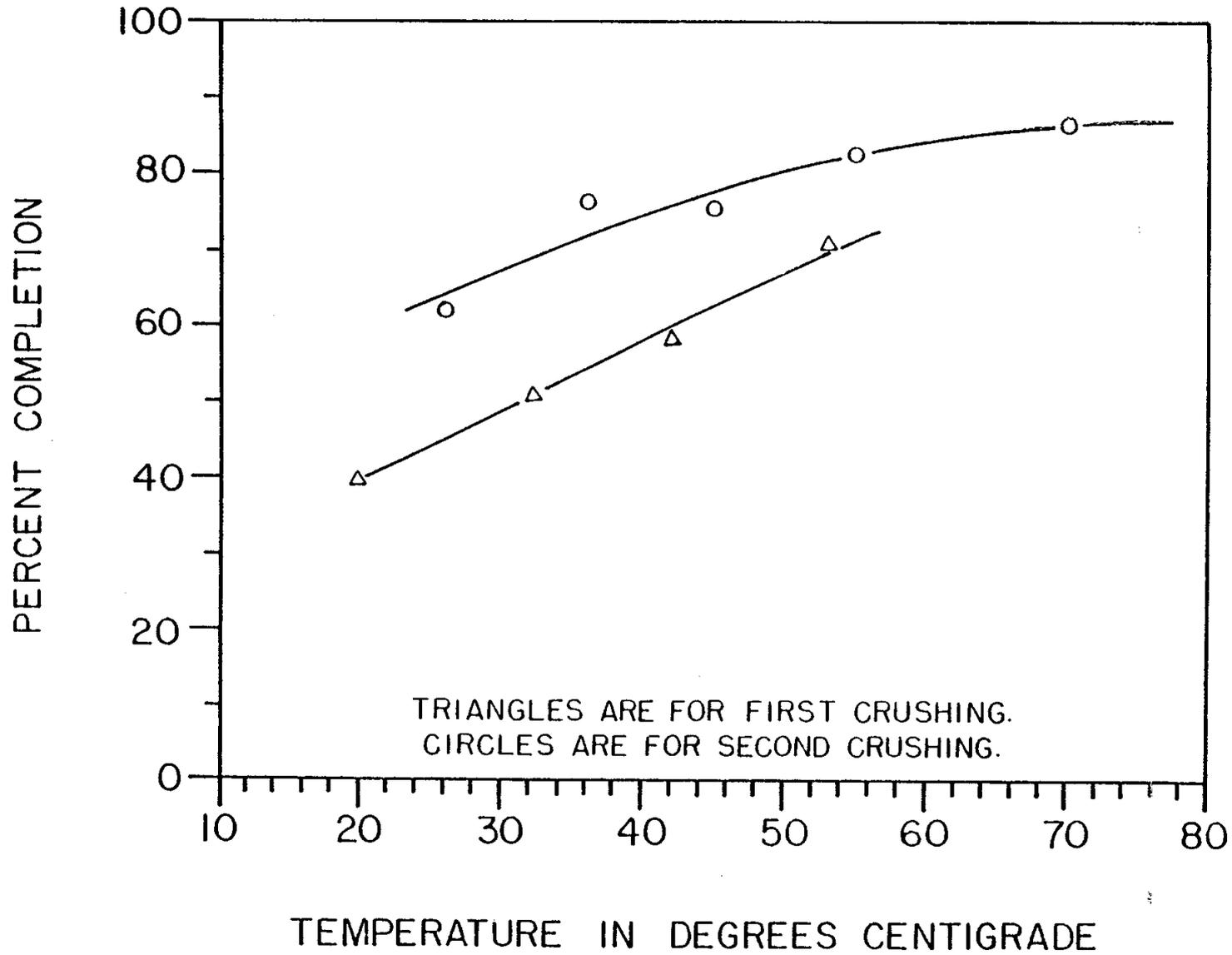


Figure 3. Reaction Performance for Limestone and 20% Excess Acetic Acid.

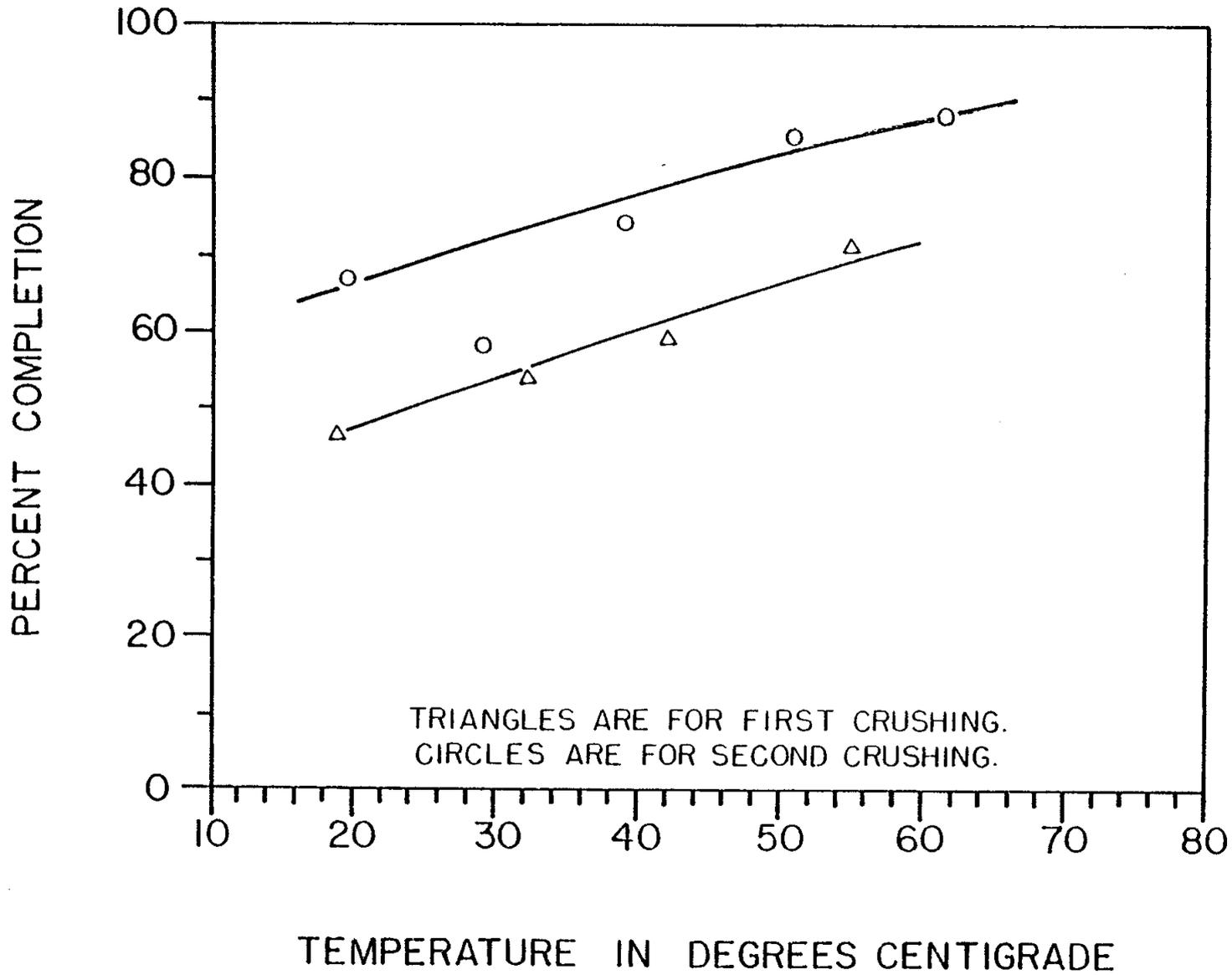


Figure 4. Reaction Performance for Limestone and 50% Excess Acetic Acid.

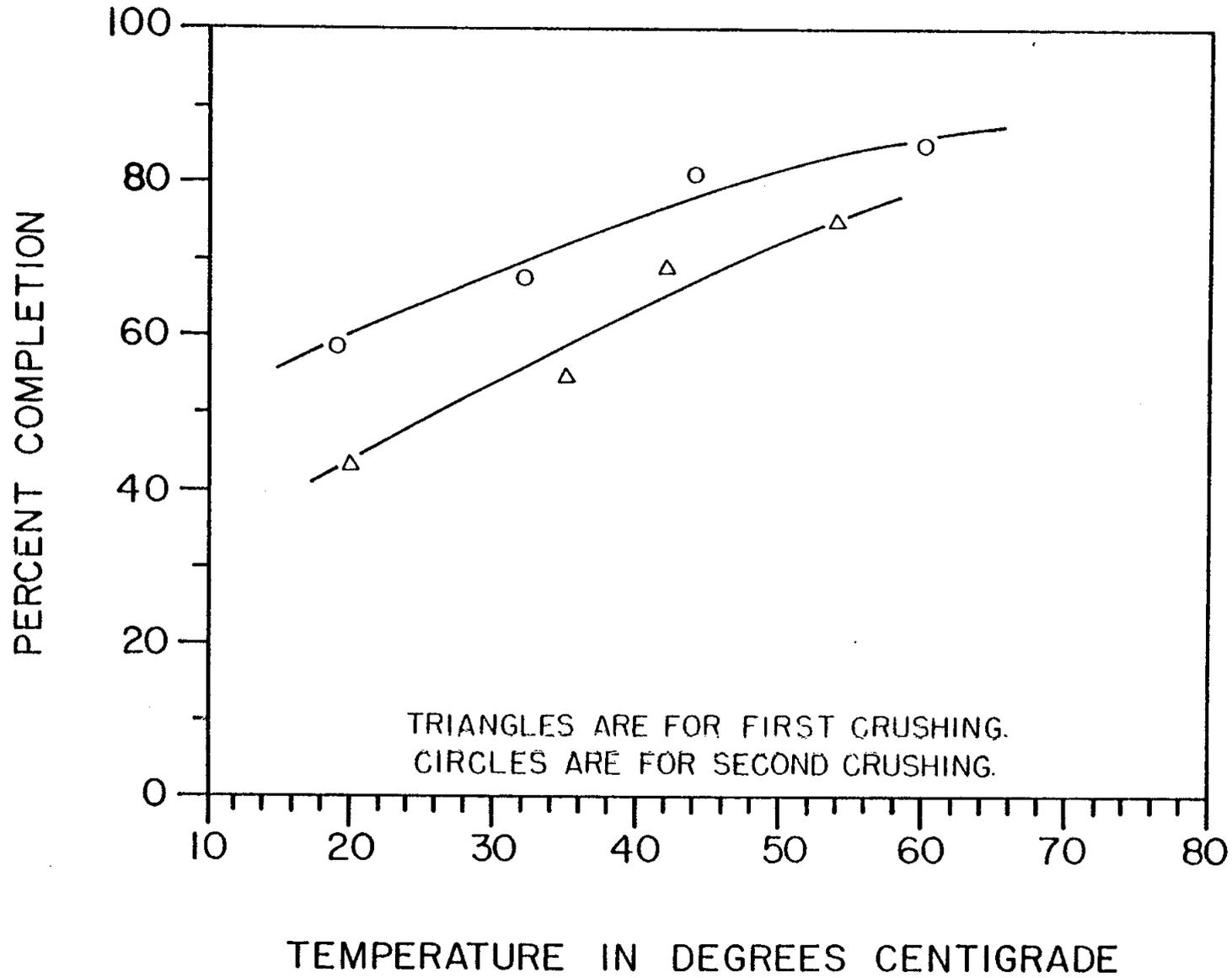


Figure 5. Reaction Performance for Limestone and 100% Excess Acetic Acid.

## PROCESS DESIGN AND ECONOMICS

A rigorous economic analysis for a proposed process requires a detailed process design. However, an approximate analysis can be completed on the basis of a preliminary design. This is common practice in the industry.

For a simple reaction process such as that of production of CMA, capital costs do not contribute a very large proportion of the required selling price of the product. Rather, it is the operating cost and more specifically the raw materials costs which are controlling. At moderate capacities any errors or omissions in the plant capital cost estimation do not alter the selling price significantly. Thus, it is possible to obtain a reasonably reliable estimate of selling price with only a process design.

Figure 6 shows a block diagram of the preliminary process design envisioned for the CMA production. Raw limestone is first crushed in two stages to 1/8" to 1" diameter aggregate particles. The crushed limestone is then mixed with acetic acid and water in a continuous stirred tank reactor. The reactor is sized for a three hour mean residence time and is heated to 70°C by an external steam heating jacket. The reaction product, consisting of the mixture of unspent acetic acid and unreacted limestone and extraneous inert material is then passed through a filtration unit. In the filtration unit unreacted solids are removed and sent to a neutralizing settling pond where they are neutralized by addition of sodium hydroxide. The filtrate containing the product acetates is then sent to a neutralizer reactor where excess acetic acid is neutralized by the addition of sodium hydroxide. The resulting acetate solution is then checked for appropriate pH value and sent to liquid product storage tank. In an optional process addition, it would be possible to evaporate the liquid product to produce a solid calcium or magnesium acetate product.

The major process components are described below:

1. Crusher: Particle size was found to be a significant variable in the reaction efficiency. A two-stage crusher will be utilized to reduce the limestone to 1/8" to 1" diameter aggregate particles.

Crusher Price --- \$14,000.00

(12)

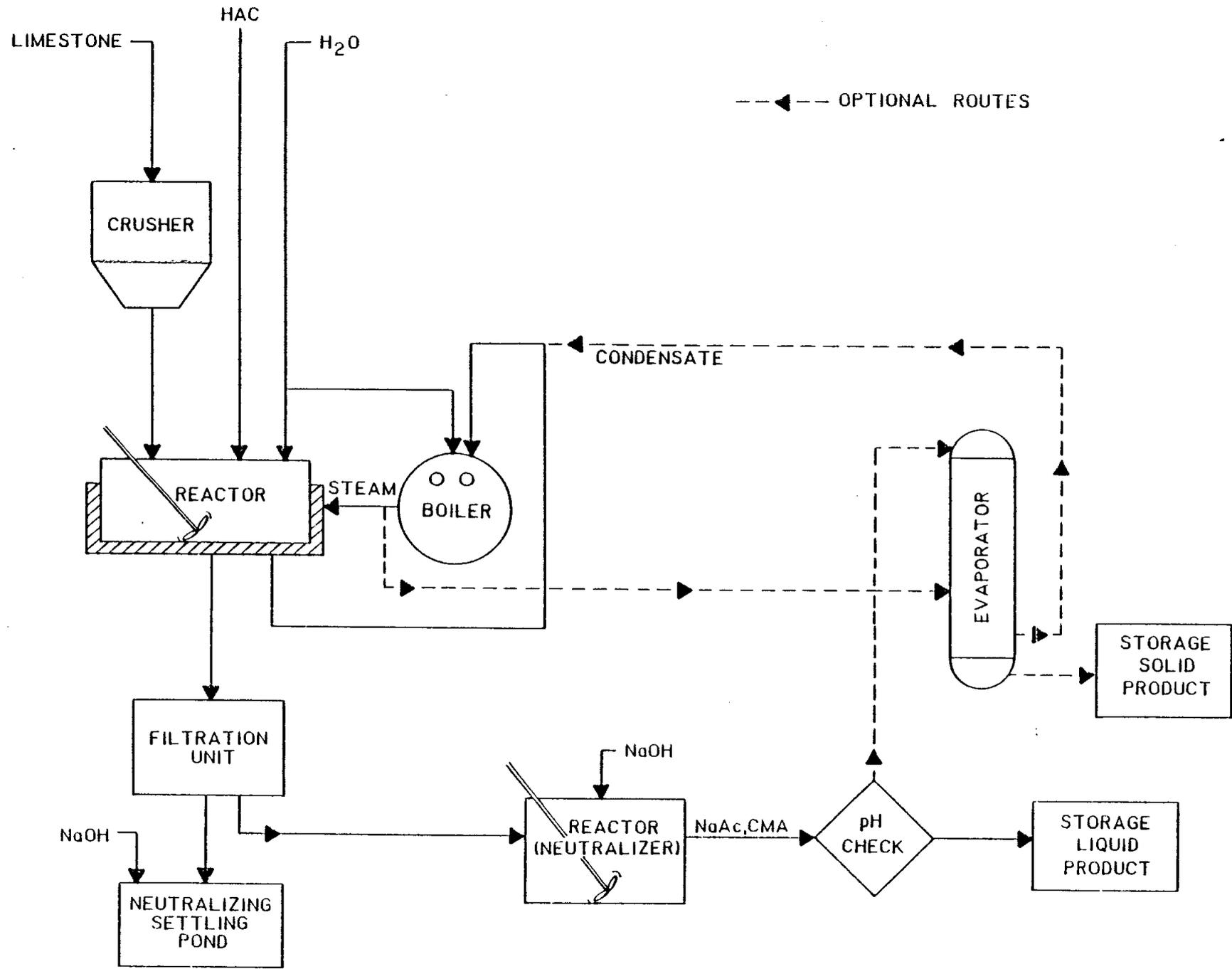


Figure 6. Block Diagram of Conceptual Process Design.

2. Reactors: Two 8,000 gallon reactors are required. The first reactor must be equipped with an external steam heating jacket. Since acetic acid is corrosive to mild carbon steel, the reactors must be constructed of 316 stainless steel.

Reactor Price --- \$110,000.00

3. Boiler: A turnkey boiler package capable of producing 4,000 pounds per hour of 250 psig steam will be required for the reactor jacket heating and space heating. The packaged unit will include a boiler, feed water de-aerator, boiler feed pumps, chemical injection pumps, stack and shop assembly.

Boiler Cost --- \$24,000.00

4. Vessel: A carbon steel storage tank capable of handling three days production (150,000 gallons) is included.

Vessel Cost --- \$70,000.00

5. Filter Press: A 304 stainless steel horizontal-plate filter press is also included.

Filter Press Cost --- \$28,000.00

6. Front End Loader: A front end loader with an 18 cu.ft. capacity is required.

Front End Loader Cost --- \$27,000.00

A summary of the major capital equipment costs for the plant is listed in Table 1. In addition, a 10% contingency has been added on giving a total major capital cost of \$300,000.00 for the CMA plant. It is common practice in capital cost estimating to use multipliers to estimate instrumentation, installation, piping, electrical, and indirect costs for the construction of chemical process plants. (5) Table 2 presents a summary of these additional costs for the CMA plant. This gives a total of \$185,000.00 for the additional costs associated with

TABLE 1

MAJOR CAPITAL EQUIPMENT COSTS

CRUSHER -----	\$ 14,000
REACTORS (2) -----	110,000
PACKAGED BOILER -----	24,000
STORAGE TANKS -----	70,000
FILTER PRESS -----	28,000
FRONT END LOADER -----	27,000
	-----
Subtotal -----	\$273,000
10% Contingency -----	27,000
	-----
TOTAL MAJOR CAPITAL EQUIPMENT -----	\$300,000

TABLE 2

ADDITIONAL PLANT COSTS

INSTRUMENTATION ( 6% of Major Capital Equipment (MCE)) -----	\$ 18,000
INSTALLATION (30% of MCE) -----	90,000
PIPING ( 7% of MCE) -----	21,000
ELECTRICAL ( 5% of MCE) -----	15,000
INDIRECT COSTS (15% of MCE) -----	45,000
	-----
Subtotal -----	\$189,000
MCE -----	300,000
	-----
TOTAL CAPITAL INVESTMENT -----	\$489,000
WORKING CAPITAL (10% of Capital Investment) -----	49,000
	-----
TOTAL INVESTMENT -----	\$538,000

the CMA plant. When combined with the capital equipment estimate, this yields a capital cost estimate of \$489,000.00. Working capital can be estimated roughly as 10% of the total investment. Inclusion of working capital results in a total plant cost of \$538,000.00. This figure represents the total investment which must be recovered through the selling of the CMA product.

On the basis of the preceding design, we have completed an economic analysis of the proposed CMA plant. It should be emphasized that this analysis represents a best case scenario, allowing for continuous operation, and minimal storage facilities. The required selling price of the CMA product can be calculated by the following equation:

$$P = \frac{CF - TD + A(1-T)}{G(1-T)}$$

where;

P = price of solution (\$/gallon)

G = annual production rate (gallons)

D = annual depreciation (straight line method)

A = annual operating cost

T = tax rate (50%)

CF = cash flow using uniform series present worth value with ROR = 20% (rate of return)

This equation has been used to calculate the required selling price (dollars per gallon of solution) required as a function of the price of acetic acid and the production capacity of the plant. A detailed example of the derivation and use of this equation is presented in Appendix 1. Figure 7 shows the required selling price as a function of acetic acid cost for a 10,000 gallon per day plant and a 50,000 gallon per day plant. It is apparent that there is some economy of scale. However, the final plant size will most likely be dictated by market considerations. Finally, Figure 8 presents the selling price for CMA as a function of production rate alone given a fixed cost for acetic acid (\$1.25/gallon). It is apparent that a minimum plant capacity of about 12,000 gallons per day is dictated by the economics. Little additional economy of scale is indicated above the production rate of 30,000 gallons per day. In Figures 7 and 8 the price of CMA has been reported in dollars per ton of solid in solution to allow comparison with current prices of sodium chloride and calcium chloride.

From an analysis of Figures 7 and 8 it is apparent that the cost of acetic

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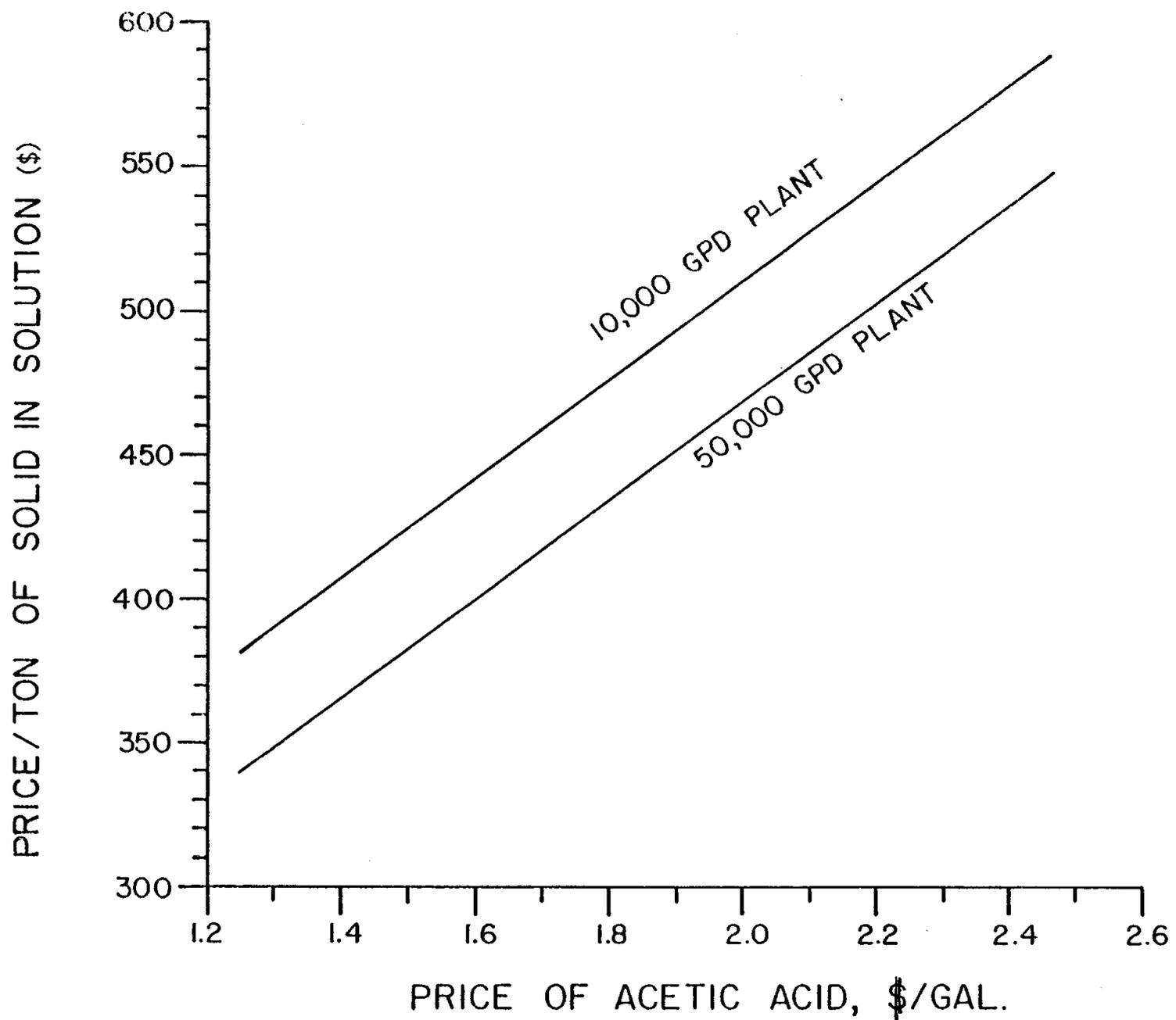


Figure 7. Required Selling Price of CMA for Various Acetic Acid Prices.

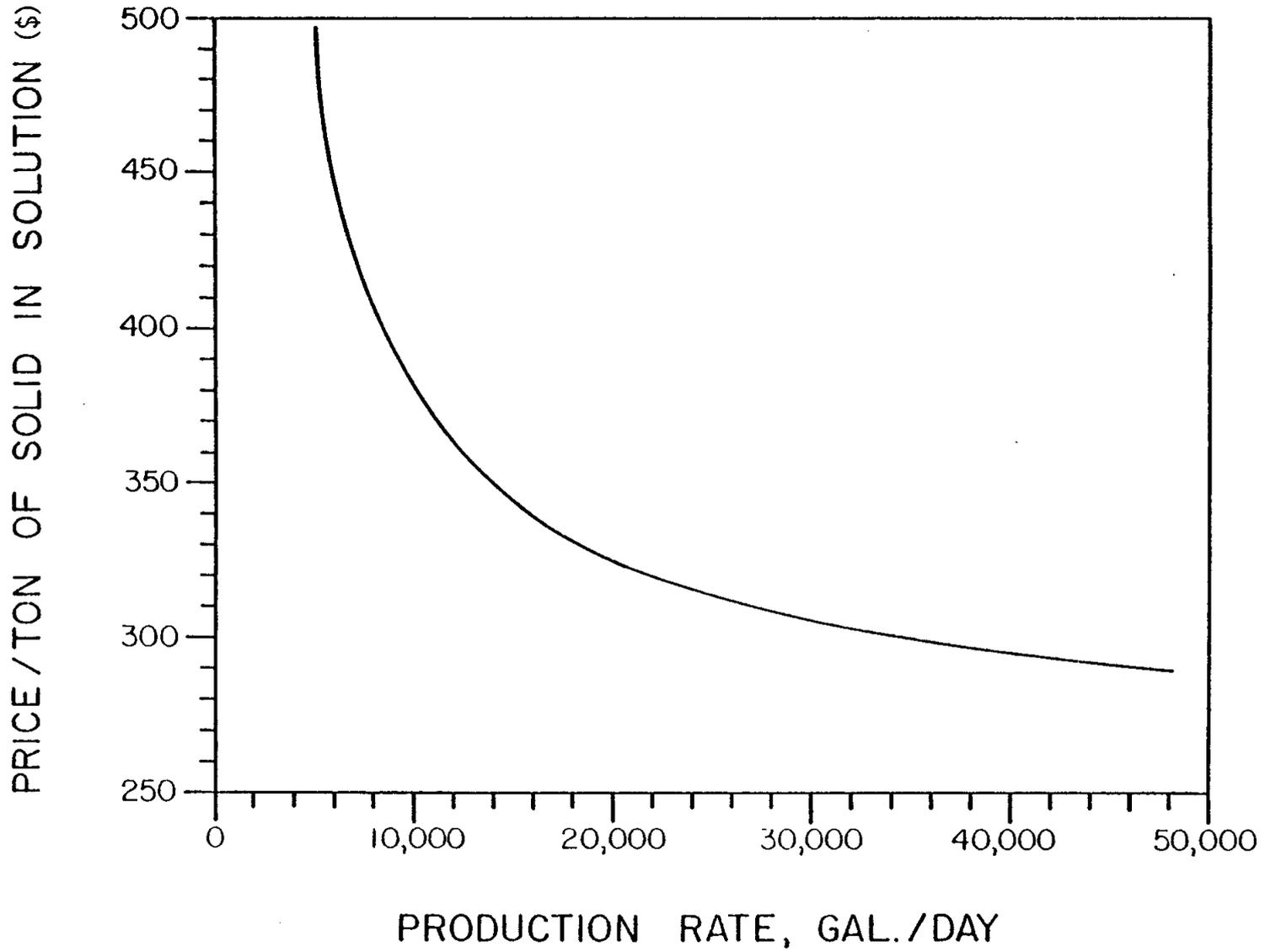


Figure 8. The Effect of Plant Size on the CMA Price. (\$ 1.25/ gallon acid)

acid is the single most important factor in determining the selling price of the CMA product. If acetic acid must be shipped to Alaska from Texas, for example, at a cost of over \$2.5 per gallon, the price of CMA will be in excess of \$500 per ton of solid in solution. However, acetic acid is normally shipped and delivered in purities in excess of 90%. Since in our process acetic acid is diluted with water to roughly 50%, it does not make sense to pay an additional premium for high purity acetic acid. In fact, one of the major costs of producing acetic acid is in the purification stage taking the acid from 50% to purities in excess of 90%. Hence, if a local production facility for acetic acid were located in or near Alaska, it should be possible to purchase dilute acetic acid at a considerable savings, perhaps as low as \$1 per gallon.

Even utilizing imported acetic acid, the price of \$500 per ton of solid CMA may not be excessive. CMA is not corrosive as is calcium chloride and sodium chloride. Hence, there is an inherent cost advantage in using CMA as opposed to chloride salts. Therefore, while CMA may cost more in the initial stage of application, significant cost savings may be realized in the area of maintenance. In addition, since airports cannot use chloride salts, but rely on more expensive methanol or urea, CMA may have a particular advantage.

## FREEZING AND EUTECTIC EXPERIMENTS

There were two objectives in this series of experiments: (1) Determination of eutectic diagrams for various solutions of salts; and (2) evaluation of the de-icing performance of the salts under varying atmospheric conditions.

The eutectic diagrams are the result of measurements gathered in the laboratory. A cold testing chamber was used to evaluate freezing properties over a wide spectrum of ambient temperatures (0°C to -75°C). A comprehensive laboratory study involved the use of different de-icing salts (CaAc, MgAc, NaCl and CMA) at different percentage solutions. The results seen in the accompanying figures represent the average readings gathered over three test runs. The ideal de-icing salt is obviously a concentrated solution (27% by wt.) of MgAc. At the lowest temperature tested (-75°C), the solution merely gelled.

Figure 9 contains the eutectic behavior of sodium chloride solutions. Our data agrees with published data. In the case of the acetates, a 28% solution of CaAc was the upper limit (saturation point) while a slightly lower concentration was the saturation limit for MgAc. Figures 10 and 11 contain the eutectic characteristics of CaAc and MgAc respectively. While CaAc did not prove more effective than NaCl, MgAc was decidedly more effective with a eutectic point depression of 75°C. Hence, a mixture of Calcium-Magnesium Acetate (CMA) with a high concentration of MgAc should be the most effective de-icer. A 25% solution of CMA with 70-80% MgAc would be sufficient for the most severe climatic conditions, as it is shown in Figure 12.

The findings of our freezing experiments, in addition to corroborating the de-icing performance of the acetate compounds, have also pointed out a major conclusion. Since the best dolomite deposits contain less than 50% magnesium carbonate, the limit of the CaAc/MgAc ratio is de facto determined.

A re-examination of the findings shown in Figure 12 lead to the observation that a 50% MgAc contribution in a CMA solution is not any more effective than a "pure" calcium acetate solution. For example, a 20% CMA solution containing 50% MgAc has a freezing point of -29°C (Figure 12) while a similar solution of CaAc has a freezing point of -26°C (Figure 10).

Dolomite with magnesium carbonate concentration of greater than 50% is not found in appreciable quantity in Alaska. Since the use of dolomite containing lower concentrations of magnesium carbonate results in a solution which is no more effective than calcium acetate derived from abundant limestones, and since

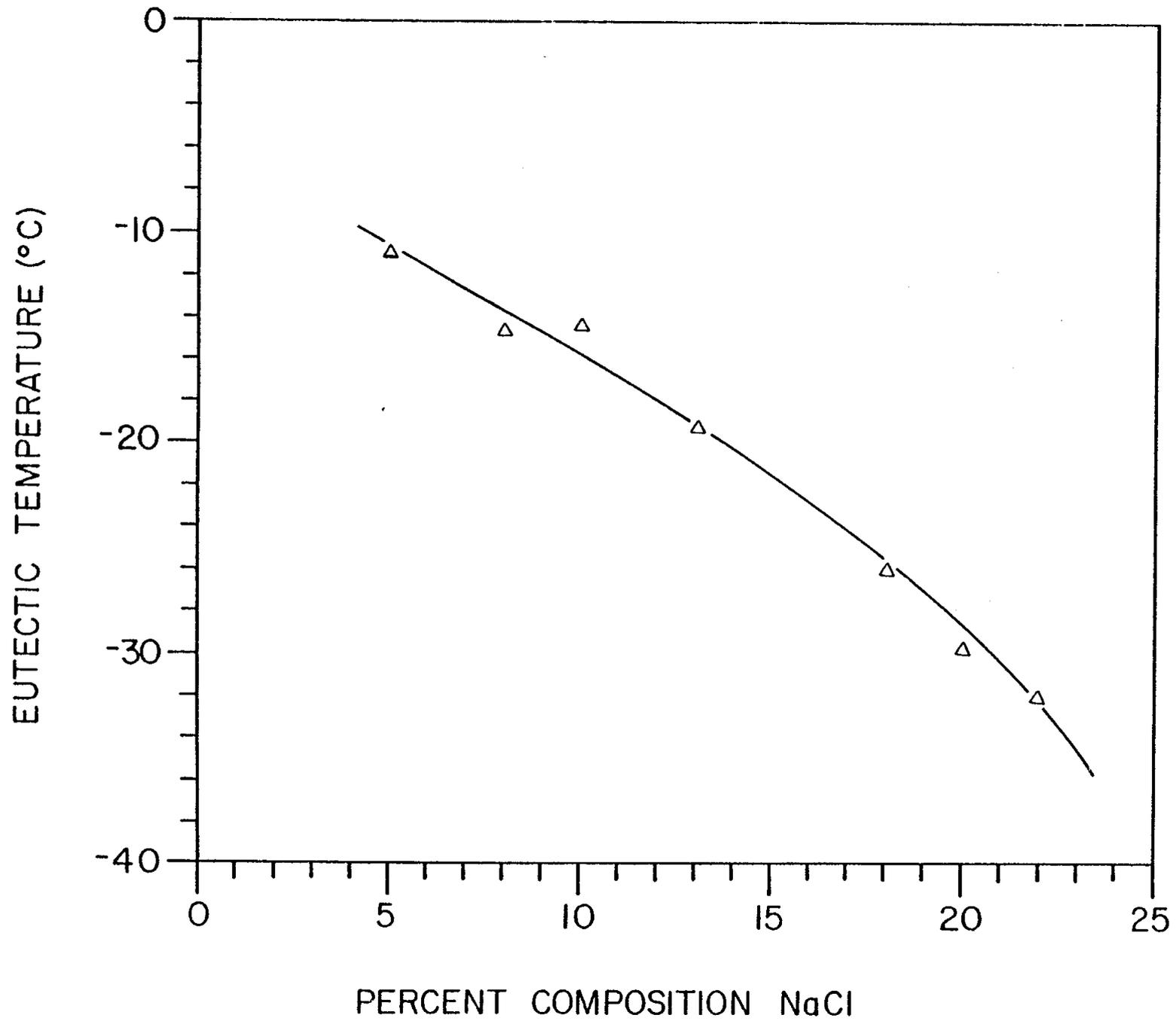


Figure 9. Experimental Determination of the Freezing Temperature of NaCl Solutions.

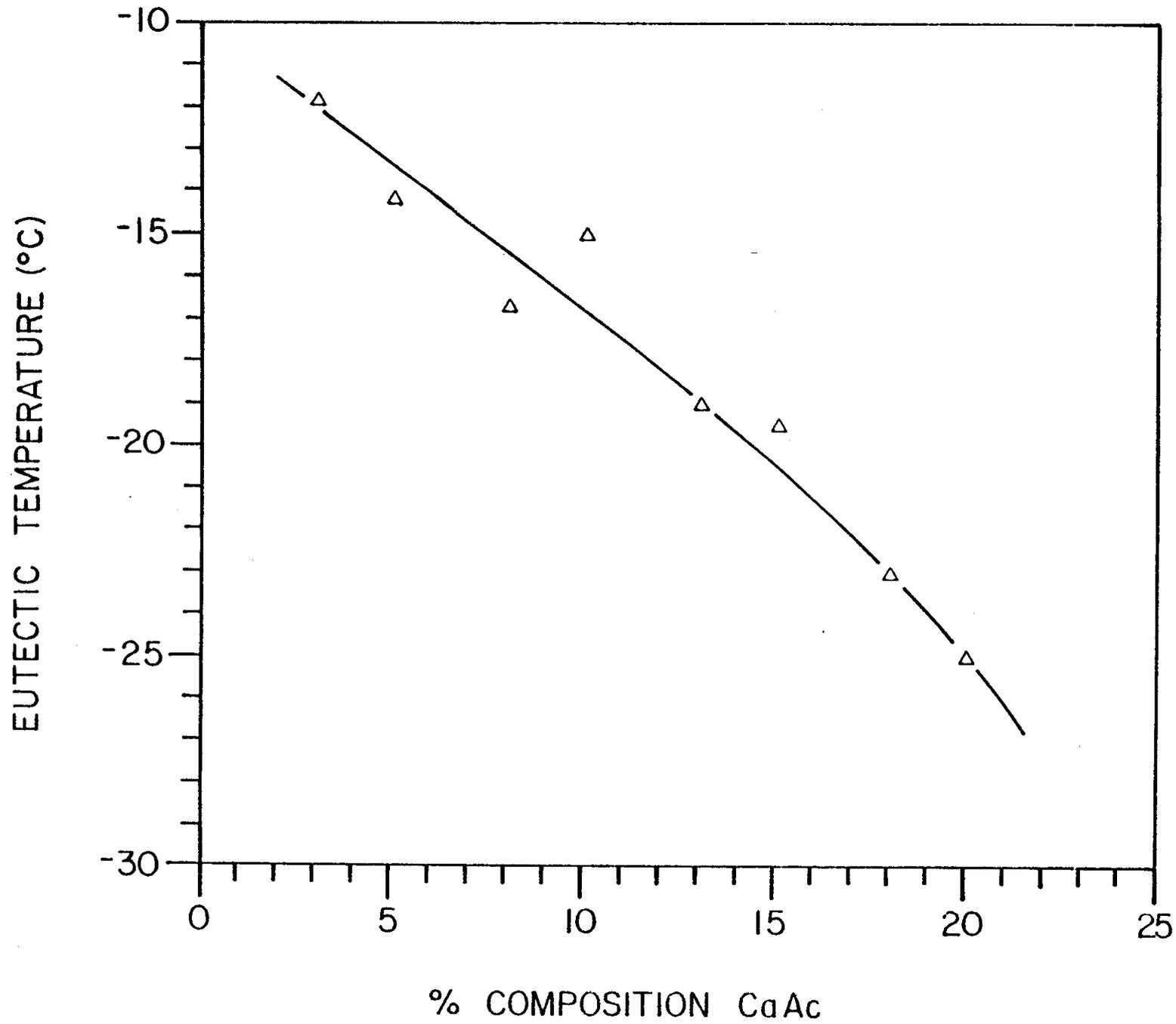


Figure 10. Eutectic Diagram of CaAc Solutions.

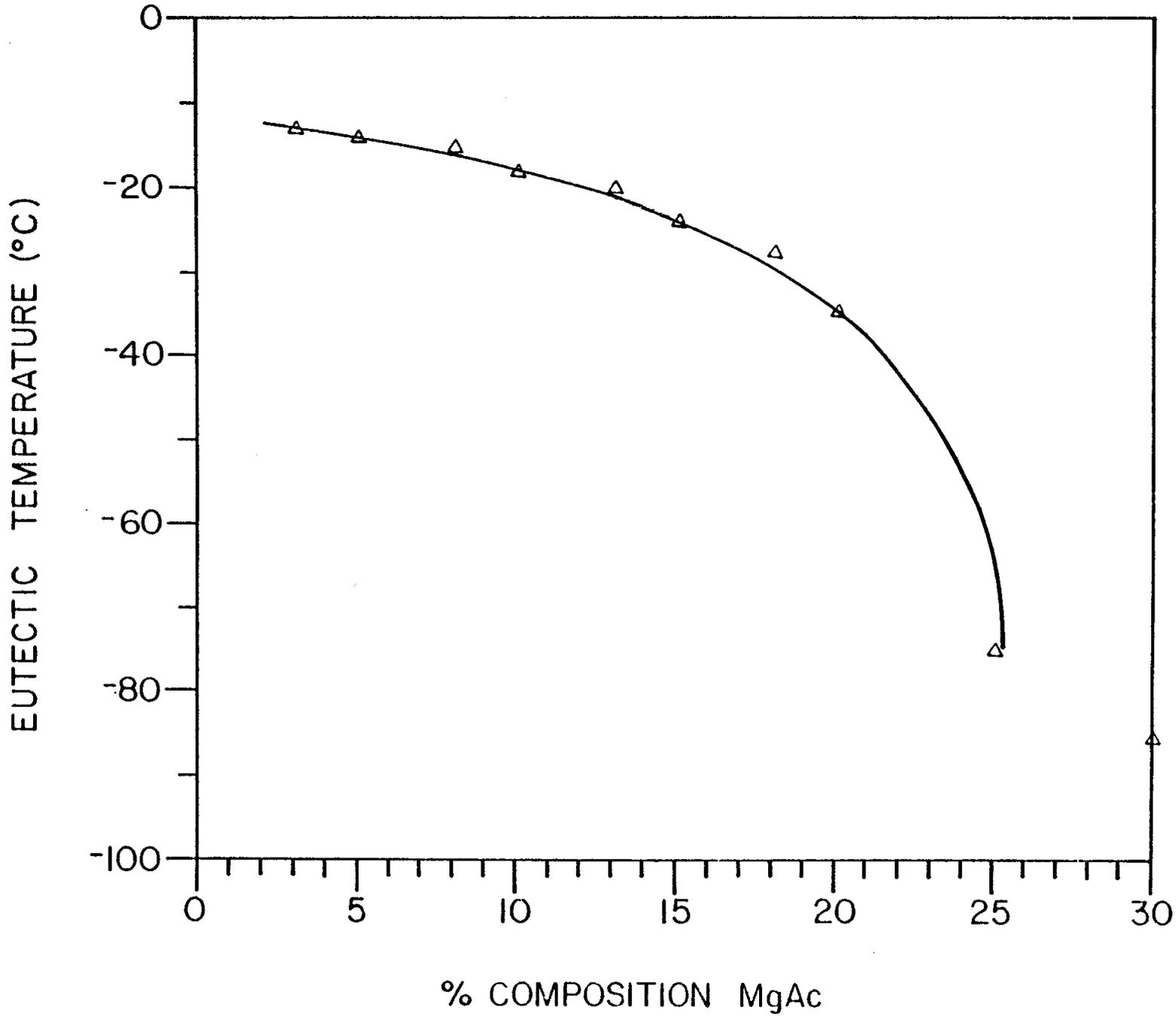


Figure 11. Eutectic Diagram of MgAc Solutions

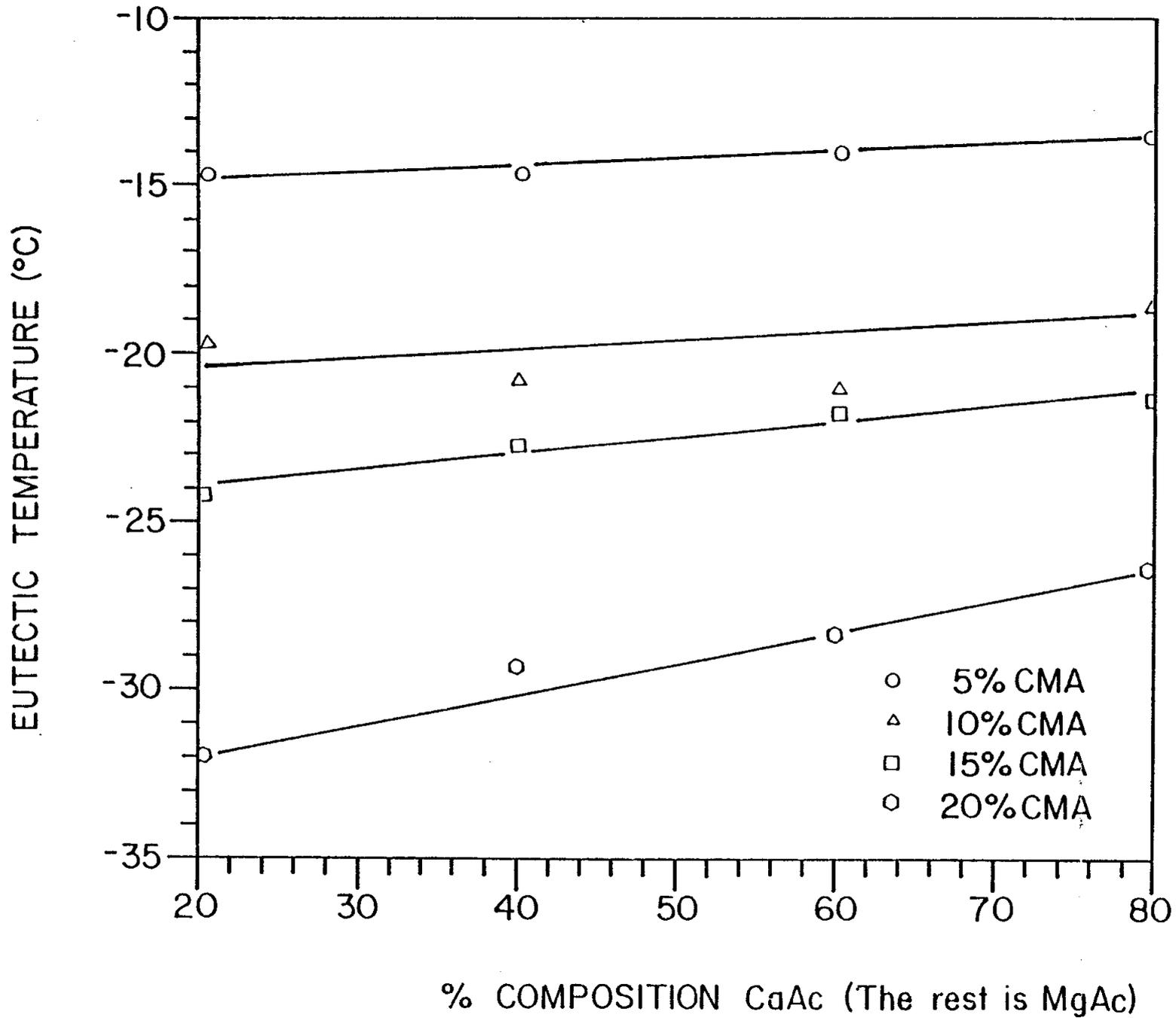


Figure 12. Eutectic Characteristics of Various Compositions of CMA Solutions.

dolomite is more expensive than limestone, it can be concluded that calcium acetate would be the most economic route for the process. To avoid confusion, we continue to use the nomenclature CMA throughout this report. However, it should be realized that we refer to a calcium acetate solution containing only small amounts of magnesium acetate.

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APPENDIX 1

SAMPLE ECONOMIC CALCULATION

The working equation is:

$$P = \frac{CF - TD + A(1-T)}{G(1-T)} \quad (1)$$

where:

P = price of solution (\$/gallon)

G = annual production rate (gallons)

D = annual depreciation (straight line method)

A = annual operating cost

T = tax rate (50%)

CF = cash flow using uniform series present worth value with ROR = 20% (rate of return)

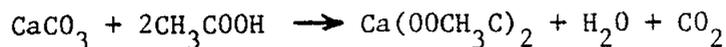
The variables in equation (1) can be evaluated:

G: 365 days x 50,000 GPD = 18.25 x 10<sup>6</sup> gallons

D: The depreciation is 1/10 of the capital investment (i.e., \$53,800).

A: The operating costs can be divided into the costs of raw materials and the fixed costs such as labor, instrumentation and maintenance.

To calculate the costs of raw materials a stoichiometric balance of the general reaction is indicated:



In order to produce 50,000 gallons of solution per day there is a need for:

48 tons of CaCO<sub>3</sub> (at \$60/ton)

12,700 gallons of Acetic Acid (at e.g. \$2.00/gal.)

53,000 gallons of water (at \$2.25/1000 gal)\*

Then A = \$10,800,000 which includes \$10,400,000 for materials and \$400,000 for fixed operating costs, instrumentation, labor and maintenance.

T: The tax rate taken at 50%

---

\* Calculated using density of solution of 87.4 lbm/ft<sup>3</sup>, 80% reaction completion, 97.14% pure Alaskan limestone, and a saturated acetate solution (25% calcium acetate, 75% water).

CF: Is the amount of money required to recover a capital investment using a certain rate of return. In this case a 20% ROR would require a uniform series present worth factor of .235.

$$\text{Hence, } CF = \$538,000 \times .235 = \$129,000$$

Using equation (1) the price of solution is \$0.60/gal which can be converted to \$420/ton of solid in a saturated CMA solution (25%).

Derivation of Equation (1)

Equation (1) is essentially a simple rearrangement of the cash flow equation:

$$CF = \text{Net Income} - \text{Tax} \quad (2)$$

Where:

$$\text{Net Income} = PG - A \quad (3)$$

and

$$\text{Tax} = (PG - A - D)T \quad (4)$$

Combining Terms:

$$CF = (PG - A) - (PG - A - D)T \quad (5)$$

Rearranging

$$P = \frac{CF - TD + A(1-T)}{G(1-T)} \quad (6)$$