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DIVISION OF MINERAL CHEMISTRY

PRODUCTION OF MAGNESIA FROM SAVAGE RIVER MAGNESITE

by

J.H. CANTERFORD and P.T. EVERSON

Report No. 3 ========== September-November, 1979

Carried out on behalf of

Industrial and Mining Investigations Pty Ltd

December 1979

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SUMMARY

Leaching tests indicate that optimum calcination conditions depend upon the magnesite/dolomite ratio of the feed. Magnesite is more readily calcined than dolomite and optimum calcination conditions should be based on the magnesite component of the feed.

Treatment of MAG 3 bulk calcine clearly indicates that the excessive iron dissolution is related both to the physical nature of the iron in the calcine and the formation of a relatively stable ferric bicarbonate complex. Optimum leaching conditions are those which give the best rate and percentage magnesium dissolution with minimum iron dissolution. To meet specification requirements, the [Fe × 100/Mg] concentration ratio of the clarified pregnant liquor should be less than 0.15. Various techniques have been investigated on how this might be achieved and results to date suggest that addition of aluminium sulphate whilst leaching offers the most promise. This technique of iron removal has yet to be optimized.

Throughout the report, the proposed process is discussed in terms of calcination, leaching and recovery of intermediate product.

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INTRODUCTION

In the previous quarter (1) experimental work was mainly concerned with the influence of calcination and leaching conditions on the recovery of magnesium from sample MAG 3 (high magnesite). The formation and recovery of the intermediate product was also investigated. On the basis of the experimental data obtained and consideration of engineering principles, a flowsheet for the calcination/carbon dioxide pressure leach process was described.

The following conclusions were drawn from the experimental results. As noted previously (1), the conclusions reached as to the influence of leaching variables and calcination conditions refer specifically to sample MAG 3.

- Chemical analyses of MAG 1 and MAG 3 calcines are consistent with their X-ray diffraction patterns and the corresponding weight loss on ignition and can be used to ascertain the temperature/time profile to which they have been subjected.

- Iron is dissolved to a significantly greater extent than the literature indicates. Because the soluble iron reports with the intermediate and final products, the level of iron dissolution that can be tolerated is determined by market requirements.

- For a given calcine, magnesium and iron dissolution depend upon slaking time and temperature, pulp density, leaching temperature, carbon dioxide partial pressure and agitation.

- Apart from leaching temperature, the above leaching variables have virtually no effect on the percentage magnesium extracted although the kinetics of magnesium dissolution may be altered.

- At elevated leaching temperatures and/or low carbon dioxide partial pressures, there may be precipitation of a basic magnesium carbonate during the early stages of leaching. This precipiate dissolves quite rapidly as the leaching time is extended provided the solubility limit of magnesium bicarbonate is not exceeded and the carbon dioxide partial pressure is greater than atmospheric pressure.

- The amount of iron dissolved increases as slake time and temperature, pulp density, carbon dioxide partial pressure and the agitation rate are increased and the leaching temperature is decreased.

- Using a fixed set of leaching conditions, which do not necessarily represent optimum leaching conditions, it is readily shown that calcination temperature, and to a lesser extent calcination time, have a pronounced effect on the percentage and rate of magnesium dissolution.

- For calcines where magnesium dissolution has gone to completion at the end of the specified leaching period, there is a direct correlation between the amounts of magnesium and iron dissolved; the greater the magnesium dissolution the greater the iron dissolution. For the same series of calcines there is an optimum magnesium content with respect to the amount of magnesium involved. This approximates to calcination at 700°C for one hour. At a lower calcination temperature there is incomplete conversion of magnesite (magnesium carbonate) to magnesium oxide, while at higher calcination temperatures there is a reduction in reactivity due to a decrease in surface area.

- Optinum calcination conditions depend upon the magnesite/dolomite content of the feed.

- Two major products can be obtained from clarified pregnant leach liquors. These have different physical and chemical properties although both yield the desired product, magnesium oxide (magnesia), at approximately the same calcination temperature. Low temperature precipitation yields nesquehonite, $MgCO_3 \cdot 3H_2O$. This has a higher bulk density and purity than the high temperature precipitate, hydromagnesite, $Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$, whereas the latter has a lower heat of decomposition per mole of magnesium oxide product and is easier to recover from the precipitation vessel.

One of the major findings of the previous quarter was that the agitation rate has a marked influence on the shapes of the kinetic curves for both magnesium and iron dissolution. At the lower agitation

rate (900 rpm) dissolution of magnesium occurs via at least two stages; an intermediate hydrated magnesium carbonate or basic magnesium carbonate is formed and this is subsequently converted into soluble magnesium bicarbonate. At the higher agitation rate (1200 rpm) the kinetic curves indicate that the intermediate precipitate either reacts to yield soluble magnesium bicarbonate as it is formed, or in fact is not formed at all. More important, however, is the fact that the time required for complete magnesium dissolution is signigicantly reduced at the higher agitation rate. Rather surprisingly, the rate *and* amount of iron dissolution increases significantly at the higher agitation rate. The increased rate of iron dissolution is expected because of the more efficient dispersion of the carbon dioxide throughout the slurry. As noted above, there is no formation of an insoluble intermediate at the higher agitation rate. It is possible that at the lower agitation rate (900 rpm) some of the soluble iron is coprecipitated with the intermediate product in a form which does not react and go into solution when the intermediate precipitate redissolves.

The increased rate of magnesium and iron dissolution and the increased amount of iron dissolution at the higher agitation rate have a significant bearing on process design. The increased power consumption required for the higher agitation rate would be more than offset by the reduction in the optimum retention time. The increased degree of iron dissolution means that development of an iron removal step or the development of leaching conditions which limit iron dissolution is of prime importance.

For the above reasons, optimization of leaching conditions using the MAG 3 bulk calcine were re-investigated at the higher agitation rate. Similarly the effects of calcination temperature and calcination time for both MAG 1 (high dolomite) and MAG 3 (high magnesite) were determined. Associated with the above test programme, rates of precipitation, the reasons why there is excessive iron dissolution, methods of limiting iron dissolution and techniques for removing soluble iron were investigated.

EXPERIMENTAL

All leaching tests were carried out as previously described (1).

A sample of MAG 3 bulk calcine was examined under a scanning electron microscope in order to ascertain the distribution of iron oxide in the sample. The instrument (JEOL - U3M) was operated at 1×10^{-9} A and 25 kV using a 50 second scan period. The back-scattered electrons were analysed by the EDAX (Energy Dispersive X-ray Analysis) technique.

RESULTS AND DISCUSSION

CHARACTERIZATION OF MAG 3 BULK CALCINE

It was previously reported (1) that although the MAG 3 bulk calcine had a significant iron content (2.91%), iron oxide (hematite) could not be identified either by X-ray diffraction analysis or optical microscopy. Under the scanning electron microscope, each grain was of the same or similar intensity; in addition, there was no significant variation in intensity within each grain. This indicates that there are no separate grains of iron oxide or localized high iron contents within the magnesium oxide grains. Figure 1 shows the back-scattered electron image of a typical magnesium oxide grain, while Figure 2 shows an EDAX trace for point b of the same grain. The integrated peak heights of the three point analyses (Figure 1, points a,b,c) are:

Point	Mg (Kα)	Fe (Kα)	Mg/Fe
a	14149	11197	1.264
b	14253	10739	1.327
c	14814	11491	1.289

It is apparent that the iron oxide is dispersed throughout the magnesium oxide, varying in concentration from point to point, and is probably present on a molecular basis. This is consistent with the earlier finding that the siderite is in solid solution with the magnesite in the original sample.

INFLUENCE OF CALCINATION CONDITIONS ON LEACHING BEHAVIOUR

Samples of MAG 1 and MAG 3 calcined at different temperatures and times were leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig carbon dioxide and 1200 rpm. Apart from the increased agitation rate, these conditions are identical to those used for MAG 3 reported previously (1). As with the 900 rpm data, these conditions do not necessarily represent optimum leaching conditions but do allow the effects of calcination conditions to be determined and comparisons made between the behaviour of MAG 1 and MAG 3. A different set of leaching conditions would probably yield different kinetic data but would not change, to any significant extent, the general trends with respect to calcination conditions noted below.

Behaviour of MAG 1 Calcines

Tables 1-4 and Figures 3-6 quite clearly show that the amount and percentage of magnesium dissolved increases as the calcination temperature is increased from 600°C to 970° (1 h or 3 h calcination period) and when the calcination time is increased from 0.25 h to 3 h (calcination at 700°C or 850°C). It is to be noted that in many cases reaction has not gone to completion after the standard 2.5 h leach period.

At the highest calcination temperature used (970°C) there is a small decrease in the amount and percentage of magnesium dissolved when the calcination time is increased from 1 h to 3 h. This decrease is probably associated with a decrease in surface area and hence reactivity. Evidence for the reduced reactivity is indicated by a comparison of the leaching data for the 970°C/3 h calcine with that of the 970°C/1 h calcine; the former is still reacting after the standard 2.5 h leach period whereas for the latter magnesium dissolution is complete. The data indicate that a high calcination temperature, of the order of 950-1000°C for a relatively short period, of the order of one hour, is required to yield maximum magnesium extraction for samples with a high dolomite content.

The amount of calcium dissolved is relatively low, falling in the range 1-7.5% of the total calcium content and varying according

to the calcination conditions. Only at the very lowest calcination temperature (600°C) is the amount of calcium dissolved in excess of 10% of the amount of magnesium dissolved. The amount of calcium dissolved does not follow the same trends noted for magnesium dissolution. For calcination times of 1 h and 3 h, the amount of calcium dissolution passes through a minimum at a calcination temperature of 700°C, the amount of calcium dissolved from the 700°C/3 h calcine being lower than that from the 700°C/1 h calcine. With a calcination temperature of 850°C, calcium dissolution increases as the calcination period is increased from 0.25 h to 0.5 h, decreases at 1 h and then increases for 2 h and 3 h periods. It is not apparent, at this stage, why calcium dissolution passes through these minima.

With the 590°C/3 h, 600°C/1 h and 700°C/1 h calcines, the amount of soluble calcium progressively increases as leaching proceeds. With all other calcines there is an initial high soluble calcium concentration; the soluble calcium concentration falls to a minimum after about 0.25-0.5 h and then begins to increase as leaching proceeds. Figure 7 shows the magnesium and calcium dissolution curves for calcines formed at 590°C/3 h and 970°C/3 h (runs 61 and 64 respectively). As the temperature and time of calcination increase, so does the conversion of dolomite to calcium and magnesium oxide (1,2). Calcium oxide readily undergoes a slaking reaction.

$$CaO + H_2O \longrightarrow Ca(OH)_2$$

Magnesium oxide undergoes a similar reaction, although to a lesser extent. The slaking reaction takes place prior to carbonation. Once carbon dioxide is introduced some of the calcium is precipitated, resulting in a decrease in the soluble calcium concentration. As leaching proceeds the bicarbonate content of the slurry increases (as the magnesium goes into solution) and calcium begins to go into solution, the magnesium and calcium contents running in parallel.

Although the MAG 1 calcines contained a small amount of iron (1.16-1.88%), in most cases there was no detectable iron in the pregnant leach liquors. The reasons for the non-dissolution of the iron were not established; however, two possible reasons are that the iron in MAG 1 has a different physical form to that in MAG 3 and that the dissolution of calcium from the MAG 1 calcines suppresses iron dissolution.

Behaviour of MAG 3 Calcines

Tables 5 and 6 and Figures 7 and 8 show the effects of calcination time and temperature on the dissolution of magnesium and iron from MAG 3 calcines. These data, obtained at an agitation rate of 1200 rpm, are completely consistent with the previously reported (1) 900 rpm data. Thus:

- There is an optimum calcine magnesium content which in turn reflects the degree of calcination. Under calcination, giving a calcine with less than about 41% Mg, and over calcination, yielding calcines with greater than about 41% Mg, both result in a reduction in magnesium extraction.

- Where reaction has gone to completion the amount of iron dissolved follows a similar trend to that of magnesium. Moreover, there is a pronounced correlation between magnesium extraction and the [Fe × 100/Mg] concentration ratio of the pregnant liquor.

- Optimum calcination conditions are of the order of 700°C for one hour.

Comparison of the Behaviour of MAG 1 and MAG 3 Calcines

Figures 10-13 clearly indicate the differing effects of calcination time and temperature on the dissolution of magnesium from MAG 1 (high dolomite) and MAG 3 (high magnesite) calcines. In particular, maximum magnesium extraction from MAG 3 is achieved at relatively low calcination temperatures for relatively short periods. Only at temperatures in excess of about 800°C is the magnesium extraction from MAG 1 greater than that from MAG 3.

Because the magnesium content of the MAG 3 calcines are significantly greater than those of the MAG 1 calcines, it is quite clear that it is advisable to process a feed with as high a magnesite/dolomite ratio as possible and that calcination conditions be determined by the behaviour of the magnesite component of the feed.

INFLUENCE OF LEACHING CONDITIONS ON THE BEHAVIOUR OF MAG 3 BULK CALCINE

Tables 7-9 and Figures 14-22 show the effects of slake time, slake temperature, pulp density, leach temperature, carbon dioxide partial pressure, agitation and initial leachant composition on the dissolution of magnesium and iron from the MAG 3 bulk calcine. The results can be summarized as follows:

- *Slake time* has no effect on the rate or degree of magnesium extraction nor on the rate of iron dissolution, but affects the amount of iron dissolved; as the slake time is increased, so the amount of iron dissolution increases. Thus the slake time should be kept to a minimum, suggesting dry grinding of calcined feed.

- *Slake temperature* has little effect on the rate or amount of magnesium dissolved but an increase in slake temperature increases the amount of iron dissolved although the rate of iron dissolution is reduced during the first 0.75 h of leaching. Crushed and ground calcine should therefore be cooled to room temperature (or the leaching temperature if this is greater than room temperature) prior to slurrying and introduction into the leaching autoclaves.

- At carbon dioxide partial pressures of 25 and 100 psig, an increase in *pulp density* results in a significant increase in the [Fe × 100/Mg] concentration of the pregnant liquor and also in a small increase in the percentage magnesium dissolved. At 25 psig carbon dioxide, an increase in the pulp density results in an increase in the time required to reach maximum magnesium dissolution, whereas at 100 psig carbon dioxide, the retention time is little affected by pulp density. From a process point of view, it is advantageous to operate at as high a pulp density as possible since this increases the throughput per unit of plant capacity. The maximum pulp density applicable is thus determined by the solubility limits of magnesium bicarbonate and the amount of iron dissolution than can be tolerated.

- Provided the solubility limit of magnesium bicarbonate is not exceeded, then as expected, an increase in *leaching temperature* results in an increase in the rate of magnesium dissolution, thus

reducing the time required to achieve maximum magnesium dissolution. In addition, the amount of iron dissolved decreases rapidly as the leaching temperature is increased. Thus leaching should be carried out at as high a temperature as it practical. Although it is possible to form supersaturated magnesium bicarbonate solutions, it is concluded that, from a process point of view, the operating pulp density should be such that the solubility limit of magnesium bicarbonate for the given leaching conditions (particularly temperature and *carbon dioxide partial pressure*) is not exceeded.

- As would be expected, an increase in the carbon dioxide partial pressure increases the rate of magnesium dissolution but has little or no effect on the total amount of magnesium dissolved. At higher pulp densities, it would be expected that, on the basis of measured and calculated solubilities (2), an increase in the carbon dioxide partial pressure would result in an increase in the soluble magnesium concentration. In terms of process design and operation, the carbon dioxide partial pressure should be kept as low as possible to ensure minimum iron dissolution but should be such that leaching times are not excessive. The lower operating pressure will also simplify the leaching equipment required.

- Since the dissolution of magnesium and iron involves the interaction of carbon dioxide dissolved in the slurry with particles of calcine, an increase in the *rate of agitation* would result in a significant increase in the rate of magnesium and iron dissolution. The experimental data clearly show the importance of the dispersion of carbon dioxide throughout the slurry. At an agitation rate of 900 rpm, precipitation of an intermediate insoluble basic carbonate is apparent in the early stages of leaching; this subsequently dissolves to form soluble magnesium bicarbonate. At 640 rpm there is insufficient carbon dioxide dispersed throughout the slurry for the intermediate precipitate to react to any significant degree; at the end of the standard 2.5 h leach period the kinetic curve suggests that the precipitate is slowly reacting to form soluble magnesium bicarbonate. At 1200 rpm the carbon dioxide content of the slurry and the transfer of carbon dioxide to the slurry are such that the intermediate precipitate either

reacts with the carbon dioxide as it is formed or is in fact not formed at all. If the sub-aerator is blocked, then there is still sufficient agitation at 1200 rpm for reasonably rapid magnesium dissolution without the formation of any significant quantity of intermediate precipitate. Provided there is sufficient agitation to ensure that the intermediate precipitate reacts to form soluble magnesium bicarbonate, then the rate of agitation has no effect on the amount of magnesium extracted. However, a change in agitation rate does affect the amount of iron dissolved. It is possible that where there is formation of the intermediate product some or all of the soluble iron is coprecipitated in such a form that it is not solubilized when the intermediate product dissolves to form magnesium bicarbonate. From a process point of view it is advantageous to use as high an agitation rate as practical since this reduces the time required to achieve maximum magnesium dissolution. The cost of the increased power consumption required to achieve the increased agitation rate must be less than the economic advantage (capital and operating costs) achieved by use of the shorter retention time. The increased iron dissolution at the high agitation rate means that the development of an iron removal step is of importance.

- In most leaching operations it is desirable to recycle as much process water as possible. In the calcination/carbon dioxide pressure leach process, the filtrate from the intermediate product solid/liquid separation stage would be used to slurry fresh calcine being fed to the leaching circuit (see Figure 1, reference 2). Normally it is not practical to recover all the desired metal(s) from the clarified pregnant leach liquor. The recycled liquor thus has a low concentration of the desired metal(s). Unless there is a specific impurity removal step, the impurities present in the clarified pregnant leach liquor will also report in the recycled leach liquor. Thus for the calcination/carbon dioxide pressure leach process it is essential to establish the effects of *the magnesium and iron concentrations of the recycled liquor* (which thus becomes the leachant) on the subsequent dissolution of magnesium and iron from a fresh sample of calcine. The experimental data (Table 9 and Figure 22) clearly show that the magnesium

and iron contents of the leachant have no effect on the rate and degree of magnesium extraction. However, the magnesium content of the leachant has a dramatic effect on the amount of iron dissolved. This indicates that it is essential that there is as complete as possible precipitation of magnesium from the clarified pregnant liquor to yield a recycle leachant which has a low as possible magnesium content.

Taking into account the effects noted above which can be directly related to the increased agitation rate, the influence of the leaching parameters on the rate and percentage of magnesium and iron dissolution are essentially the same as those reported previously (1) using a 900 rpm agitation rate. Thus optimum leaching conditions are largely determined by the amount of iron dissolution that can be tolerated. If it is possible to develop a process for either treating the calcine prior to leaching so as to prevent iron dissolution, or alternatively, to develop a process for removing soluble iron, the leaching should be carried out under the following conditions:

- the shortest slake time,
- slaking to be carried out at, or preferably, below the leaching temperature,
- at as high a pulp density consistent with the solubility of magnesium bicarbonate at the operating temperature and pressure,
- at as high a temperature consistent with the solubility of magnesium bicarbonate at the operating pulp density and pressure,
- under the lowest practical carbon dioxide partial pressure which ensures a reasonably rapid rate of reaction, and
- at as high an agitation rate to ensure rapid dissolution kinetics consistent with capital and operating costs.

PRECIPITATION OF INTERMEDIATE PRODUCT

Following leaching of the calcine, the gangue is removed from the pregnant leach slurry by solid/liquid separation (preferably

pressure filtration to prevent precipitation of intermediate product) and the intermediate product precipitated by removing excess carbon dioxide from the clarified pregnant liquor. As noted previously (1), two intermediate products can be obtained:

Nesquehonite	$MgCO_3 \cdot 3H_2O$	$T < 52°C$
Hydromagnesite	$Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$	$T > 52°C$

Because the carbon dioxide recovered from the precipitation stage has to be purified and compressed prior to recycling to the leaching circuit, because the physical and chemical properties of the intermediate product have a bearing on the properties of the final product, and because it is essential to recover as much magnesium as possible to ensure the recycle leach liquor is depleted in magnesium, precipitation data are of importance.

Table 10 and Figure 23 show four sets of precipitation data. From these data, experimental observations and previously reported data (1), the following points can be made.

- As the temperature of precipitation is increased, so the rate of precipitation increases.

- The magnesium and iron contents of the mother liquor decrease simultaneously as precipitation takes place.

- At room temperature ($20 \pm 2°C$) without aeration, the precipitate forms a crust on the liquor surface and on the walls of the precipitation vessel; the latter is difficult to recover from the precipitation vessel.

- At all temperatures with aeration, the major portion of the precipitate forms in the bulk of the solution; this precipitate settles quite rapidly once agitation is stopped and is readily recovered from the precipitation vessel.

- The bulk density and average particle size decrease rapidly as the temperature of precipitation is increased.

- The heat of decomposition per mole of magnesium oxide product is lower for the high temperature precipitate (hydromagnesite) than it is for nesquehonite, the low temperature precipitate.

The magnesium concentration of the clarified pregnant liquor is relatively low compared with many other hydrometallurgical process solutions, and heating this liquor, even over 10°C, would consume considerable energy per unit weight of magnesium. This suggests that precipitation should be carried out at a temperature not significantly greater than the leaching temperature. Low pressure steam could be used to heat and agitate the clarified pregnant liquor to a temperature of about 35°C; by this means the carbon dioxide evolved would not be significantly diluted and would be readily treated for recycling to the leaching circuit. It may be necessary to pass the slurry to a second precipitation vessel where high pressure air could be injected to ensure complete precipitation of the magnesium. The precipitate formed under the above conditions would be nesquehonite and would have a reasonable bulk density and particle size such that it could be readily recovered by conventional solid/liquid separation techniques.

THE DISSOLUTION OF IRON

The amount of iron dissolved in the various leaching tests described here and elsewhere (1) is considerably greater than the literature and the pH of the clarified pregnant liquors (7.5-8.0) would suggest. Chemical tests confirmed that the iron in solution was in the ferric state. One would normally not expect to detect ferric iron in solutions so alkaline. Ferric iron normally precipitates at pH's more alkaline than 2.5-3.0; that is, ferric iron normally precipitates even under mildly acidic conditions. A solution of ammonium ferric sulphate was make alkaline to pH 8 by addition of a sodium hydroxide solution and the precipitated ferric hydroxide removed by vacuum filtration; the filtrate contained no detectable ferric ion (<0.05 ppm).

Because of excessive iron dissolution during carbon dioxide leaching and the need to develop an efficient iron removal step, it was considered essential that the reasons for the excessive iron dissolution be determined. There are basically two reasons for the ferric iron going into solution; the calcine contains the iron oxide in a particularly active form and/or the leaching system is such that the ferric iron in solution is chemically stabilized.

To ascertain if it is the physical form of the iron in the MAG 3 bulk calcine that determines the high iron dissolution, samples of natural hematite, synthetic hematite and freshly precipitated ferric hydroxide were leached with and without magnesium oxide or sodium carbonate under conditions similar to those used for the MAG 3 bulk calcine. The results of these tests are given in Table 11; the following conclusions can be drawn.

- There is no dissolution of hematite (synthetic or natural) or ferric hydroxide when they are slurried and pressurized with carbon dioxide even though the particle size of the source of ferric iron was quite small (<1 μm in the case of synthetic hematite) and thus had a high surface area.

- When magnesium oxide or sodium carbonate is added with the resultant formation of a considerable bicarbonate concentration, each of the sources of ferric iron dissolves to a limited extent, with up to 6 ppm ferric iron going into solution.

- For natural hematite, the higher the bicarbonate concentration, the greater the iron dissolution.

- The dissolution of ferric iron from MAG 3 bulk calcine is considerably greater than that from the other potential sources of iron even when an excess of bicarbonate forming additive is added to the latter.

Thus the dissolution of ferric iron from MAG 3 bulk calcine is directly related to the physical nature of the iron in the calcine. It has already been shown that the iron in the bulk calcine is in the form of hematite dispersed throughout the magnesium oxide grains, possibly on a molecular basis. In such a form the hematite will have an extremely high surface area and surface properties that allow it to react to a degree greater than would be expected for hematite with "normal" surface area and surface properties.

Data given in Tables 7-11 and Figures 14-23, as well as those reported previously (1), indicate that, in general, the amount of iron dissolved increases as the magnesium (and hence bicarbonate) content of the pregnant leach slurry increases. This suggests that

the iron goes into solution via the formation of a ferric bicarbonate complex, possibly of the type $Fe(HCO_3)_{3+n}^{n-}$ (n = 0-3). The only case where the amount of iron in solution does not increase as the bicarbonate concentration increases is when the leaching temperature is increased. This indicates that the stability of the ferric bicarbonate complex is very temperature dependent; as the leaching temperature is increased the ferric bicarbonate complex progressively decomposes to ferric oxide. Further evidence for the low stability of the ferric bicarbonate complex comes from the fact that it readily decomposes when the bicarbonate content of the pregnant leach liquor is reduced by air sparging to induce evolution of carbon dioxide and precipitation of magnesium carbonate.

Further evidence for the formation of a soluble ferric bicarbonate complex comes from the fact that addition of sodium carbonate to the MAG 3 bulk calcine prior to leaching results in a substantial increase in the concentration of soluble iron in the pregnant liquor. As Table 11 shows, there is a five-fold increase in iron concentration on addition of 50 g sodium carbonate to the standard leach system. The solution containing the 460 ppm ferric iron was bright red and the iron remained in solution for several months, indicating that the ferric bicarbonate complex is relatively stable in the presence of a large bicarbonate concentration.

The above observations are consistent with the findings of Zaitsev (3) who showed that relatively concentrated ammonium or alkali metal carbonate solutions dissolve considerable quantities of ferric salts (nitrate, chloride, sulphate) forming clear yellow to dark red solutions, the colour depending upon the iron concentration. Zaitsev (3) believed the iron was present in solution as complexes of the type $[Fe(CO_3)_nL_m]^{x-}$ where L = H_2O or OH. Several dark coloured precipitates were recovered from these solutions, but their low purity precluded their characterization.

In summary, the high ferric iron contents of the liquors derived from MAG 3 bulk calcine arises both because of the physical properties of the hematite in the calcine and because of the formation of a stable ferric bicarbonate complex.

ELIMINATION OF IRON DISSOLUTION AND REMOVAL OF SOLUBLE IRON

The specification of magnesium oxide required for furnace linings used in BOF steelmaking furnaces is such that the Fe_2O_3 content should be less than 0.25% and preferably less than 0.15%. To achieve this using the calcination/carbon dioxide pressure leach process, the clarified pregnant leach liquor containing, say, 10 gpl magnesium must have an iron content of less than 0.015 gpl (15 ppm), that is the [Fe × 100/Mg] concentration ratio should be less than 0.15. Hence the successful application of the calcination/carbon dioxide pressure leach process depends on preventing any iron going into solution, or if this cannot be prevented, the removal of the soluble iron prior to precipitation of the intermediate product.

There are three basic approaches to the production of iron-free pregnant liquors.

- Treatment of the calcine prior to leaching in such a way that the iron oxide is made non-reactive.
- Development of leaching conditions in which iron dissolution is inhibited.
- Precipitation of iron from the pregnant leach pulp, preferably during leaching, or from clarified pregnant liquor.

The ideal process is one that requires no extra unit process such as a separate roast or an additional solid/liquid separation, which requires no additional reagents, or at least utilizes inexpensive reagents, is compatible with the rest of the process, and which does not result in significant magnesium losses.

In their extensive study of the atmospheric pressure carbonation of calcined magnesite and dolomite, Doerner and coworkers (4) noted a small but significant degree of iron contamination of their pregnant leach liquors. The maximum iron dissolution reported by these workers is significantly lower than that reported here and is probably related to the nature of the iron contents of the respective calcines and the carbon dioxide pressures used. Even so, Doerner *et al.* (4) were concerned by the iron dissolution and suggested that

sulphidization of the iron content of the calcine prior to leaching would be one way of reducing iron dissolution. They suggested that calcination of the magnesite/dolomite be carried out in a multiple hearth furnace and that a suitable mixture of sulphur and hydrogen be introduced into the lower hearths of the furnace to form iron sulphide. They pointed out that it would be necessary to cool the sulphidized calcine in an inert atmosphere to prevent reoxidation of the iron sulphide.

This approach seems to have several disadvantages. In particular, there is the problem of reoxidation of the iron sulphide during calcine cooling and leaching, while a multiple hearth furnace is must less efficient than the proposed rotary kiln. With the calcines derived from the Savage River magnesite, the iron oxide is in such a form that it is doubtful that complete sulphidization of the iron oxide could be readily and efficiently achieved using the above technique. Moreover, it is likely that the cost of the admittedly small amounts of hydrogen would tend to make this technique uneconomic.

It is possible that the iron oxide in the calcine could be selectively reduced to magnetite and the latter magnetically removed from the calcine. Once again the highly dispersed nature of the iron oxide within the magnesium oxide grains would probably make this removal technique relatively inefficient. As with the sulphidization technique, it is likely that the cost of the reductant would make the technique uneconomic.

Although a wide variety of leaching conditions were tested (Tables 7-8 and Figures 14-21), no case was found where there was an acceptable rate and percentage magnesium dissolution at a "practical" pulp density which yielded a [Fe × 100/Mg] concentration ratio of less than 0.15. Thus it was apparent that the development of a technique for removing soluble iron from the pregnant leach slurry or clarified liquor offered the most potential for the production of specification magnesium oxide using the calcination/carbon dioxide pressure leach process.

Because solid/liquid separation is a costly hydrometallurgical unit process, it is preferable that the soluble iron be precipitated from the pregnant leach slurry during leaching. This approach has the additional advantage in that precipitation of the iron from clarified pregnant liquors is likely to be complicated by simultaneous precipitation of intermediate product (nesquehonite or hydromagnesite, depending upon temperature) resulting in magnesium losses. However, in order to be confident of the potential of alternative iron removal techniques, it was considered that it would be appropriate to carry out initial testwork on clarified pregnant liquors.

As Table 12 shows, the following techniques were tested for iron removal: physical adsorption, formation of stable complex anions, precipitation of ferric salts, ion exchange and solvent extraction. These techniques were chosen since they form the base of many analytical methods and hydrometallurgical unit processes. In each case the magnesium and iron contents of the aqueous phases were determined 30 minutes after mixing and again after 6 days had elapsed. In a number of tests nesquehonite precipitated before the second series of analyses. A reference solution of clarified pregnant liquor without additive was used to determine if there had been any selective iron precipitation by the various additives.

The expected and observed reactions are listed in Table 12; only in a small number of cases were the two reactions identical or the observed reaction indicative of some potential as an iron removal step.

- Precipitation of iron sulphide can be achieved by injection of hydrogen sulphide or addition of potassium polysulphide. The iron sulphide tends to be extremely fine, much of it passing through a 1 µm filter. On standing or heating, the iron sulphide precipitate agglomerated and could be removed on a 10 µm filter. However, a considerable amount of nesquehonite precipitated at the same time and this would represent an unacceptable loss of product.

- With both aluminium sulphate and to a lesser extent sodium aluminate, a white precipitate was formed and there was a significant

reduction in the [Fe × 100/Mg] concentration ratio of the mother liquor 30 minutes after mixing. With both additives there was also a small drop in the magnesium contents of the mother liquors.

The effectiveness of aluminium sulphate in removing iron from clarified pregnant liquors was carefully checked in a second series of tests—see Table 13 and Figure 24. It can be seen that the amount of aluminium sulphate added has a marked effect on the [Fe × 100/Mg] concentration ratio of the filtrate although it is to be noted that a high addition of aluminium sulphate causes a significant reduction in the magnesium concentration of the filtrate. However, in no case did the [Fe × 100/Mg] concentration ratio reach the desired level (<0.15). The white precipitate was extremely fine and quite difficult to remove by filtration.

On the basis of the observations made with several of the additives included in Table 12, it was decided to carry out a series of autoclave leach tests with these additives mixed with the MAG 3 bulk calcine. By incorporating the additive during leaching, it was anticipated that the expected reaction would be able to proceed more efficiently. This technique, as noted before, would not result in an additional solid/liquid separation stage. Although the addition of hydrogen sulphide or potassium polysulphide resulted in the precipitation of iron sulphide from clarified pregnant liquors, the use of these reagents during leaching was not attempted. Both act as reducing agents and would react with and remove the oxide film which protects the stainless steel autoclave from corrosion.

The results of the removal of iron during leaching tests are summarized in Table 14. Only with sodium citrate, aluminium sulphate and sodium aluminate is there any significant change in the [Fe × 100/Mg] concentration ratio at the end of the 2.5 h leach period. The addition of sodium citrate has a deleterious effect on the [Fe × 100/Mg] concentration ratio since 80% of the ferric iron is solubilized, compared with about 10% dissolution in the standard (no sodium citrate addition) leach. The increased amount of iron dissolution is most likely due to the high stability of the ferric citrate complex. Sodium aluminate causes a small reduction in the [Fe × 100/Mg]

concentration ratio for this particular addition of aluminium sulphate is below the maximum acceptable level.

Although addition of 10 g of aluminium sulphate per 30 g of MAG 3 bulk calcine causes a satisfactory reduction in the [Fe × 100/Mg] concentration ratio, there is an excessive loss of magnesium and the cost of the aluminium sulphate would probably be prohibitive. Thus there is the need to carefully establish the optimum addition of aluminium sulphate under a variety of leaching conditions. It is also considered that the form in which the iron and magnesium are precipitated should also be determined.

The first set of tests was carried out using the standard leaching conditions—the results of these leaches are given in Table 15 and Figures 25 and 26. The following conclusions can be drawn from the data.

- There is no change in the rate of magnesium dissolution as the aluminium sulphate addition is increased.
- The magnesium and iron contents of the pregnant liquor at the end of the leaching period decrease in a parallel manner as the aluminium sulphate addition is increased.
- The lowering of the iron and magnesium concentrations in solution is because of the formation of a precipitate containing both metals and probably also aluminium. As the formation of this precipitate seems to be related to the dissolution of magnesium and the formation of magnesium bicarbonate, it seems likely that the precipitate is a Mg-Al-Fe carbonate (basic or normal).
- Semi-quantitative analysis indicates that the pregnant liquor is free of aluminium but contains all of the added sulphate.
- The influence of the sulphate content of the pregnant liquor on the precipitation of the intermediate product (nesquehonite or hydromagnesite) and on leaching fresh calcine with recycled liquor needs to be established.
- Under the standard leaching conditions, precipitation of iron during leaching by addition of aluminium sulphate is probably

uneconomic. However, on the basis of the leaching tests carried out (Tables 7-9 and Figures 14-22) it is likely that leaching at an elevated temperature (of the order of 30°C) will result in the formation of a pregnant liquor with an acceptable [Fe × 100/Mg] concentration ratio, minimum magnesium losses and a minimum addition of aluminium sulphate.

Because the feed for a commercial calcination/carbon dioxide pressure leach operation will normally contain a certain amount of dolomite, it was considered essential that the effects of aluminium sulphate addition on the leaching of magnesium and calcium from a dolomite-rich sample be examined. Table 16 and Figure 27 show the kinetic data for leaching MAG 1 calcined at 850°C/3 h and 970°C/3 h. It has previously been noted that there is no detectable iron dissolution from these calcines.

- Addition of aluminium sulphate alters the shape of the dissolution curves of both magnesium and calcium.

- Addition of aluminium sulphate *increases* the amount of calcium dissolved to such an extent that although there is a reduction in the magnesium content of the pregnant liquor, the Ca/Mg concentration ratio increases as the aluminium sulphate addition increases.

- Where there is even greater calcium dissolution, say from a pure dolomite sample (MAG 1 contains about 80% dolomite), it is possible that upon addition of aluminium sulphate, the solubility product of gypsum, $CaSO_4 \cdot 2H_2O$, would be exceeded so that the latter would precipitate.

In summary, of all the techniques used to date to eliminate iron dissolution, only the addition of aluminium sulphate is effective in reducing the [Fe × 100/Mg] concentration ratio.

SUMMARY OF RESULTS/GENERAL COMMENTS

- The iron content (2.91%) of MAG 3 bulk calcine is dispersed throughout the magnesium oxide grains, and is probably present on a molecular basis.

- There is no iron dissolution from MAG 1 calcines using standard leach conditions.

- In terms of maximum magnesium dissolution the optimum calcination conditions for MAG 1 are probably 970°C/1 h.

- As the temperature and time of calcination of MAG 1 are increased, so, in general, does the calcium dissolution increase.

- With most MAG 1 calcines there is an initial high soluble calcium concentration, which decreases to a minimum after 0.25-0.5 h and then begins to increase again as leaching proceeds.

- For MAG 3, there is an optimum calcine magnesium content which in turn reflects the degree of calcination. Under calcination, giving a calcine with less than 41% Mg, and over calcination, yielding calcines with greater than 41% Mg, both result in a reduction in magnesium extraction.

- Where reaction has gone to completion, the amount of iron dissolved from MAG 3 calcines follows a similar trend to that of magnesium. Moreover, there is a pronounced correlation between magnesium extraction and the [Fe × 100/Mg] concentration ratio of the pregnant liquor.

- Optimum calcination conditions for MAG 3 are of the order of 700°C/1 h.

- Because the magnesium contents of MAG 3 calcines are significantly greater than those of the MAG 1 calcines, it is advisable to process a feed with as high a magnesite/dolomite ratio as possible and that calcination conditions be determined by the behaviour of the magnesite component of the feed.

- An increase in slake time, slake temperature, pulp density, carbon dioxide partial pressure, rate of agitation and magnesium content

of the leach liquor result in an increase in the iron dissolution from MAG 3 bulk calcine. An increase in the leaching temperature results in a decrease in the dissolution of iron.

- The percentage magnesium dissolution is determined by the leaching temperature and carbon dioxide partial pressure.

- Both the leaching temperature and agitation rate have a marked effect on the rate of magnesium dissolution.

- As the temperature of precipitation of the intermediate product is increased, so the rate of precipitation increases while the bulk density and particle size of the precipitate decrease.

- On an energy consumption basis, precipitation is probably best carried out at or just above the leaching temperature using low pressure steam to heat the pregnant liquor and induce agitation for efficient carbon dioxide evolution.

- A series of leaching tests with a range of ferric iron-containing solids clearly indicates that the iron dissolution from MAG 3 bulk calcine is directly related to the physical nature of the iron in the calcine.

- Iron dissolution from MAG 3 bulk calcine is related to the magnesium dissolution, that is, bicarbonate formation. It is apparent that the iron goes into solution as a ferric bicarbonate complex.

- To meet specification requirements, the calcination/carbon dioxide pressure leach process must yield a product containing less than 0.25% Fe_2O_3; this is equivalent to a pregnant liquor with a maximum [Fe × 100/Mg] concentration ratio of 0.15.

- The ideal process for producing a pregnant liquor with an acceptable [Fe × 100/Mg] concentration ratio is one that requires no extra unit process, which requires no additional reagents or at least utilizes inexpensive reagents, which is compatible with the rest of the process and which does not result in significant magnesium losses.

- Of the following techniques tested on clarified pregnant liquors or during leaching—physical adsorption, formation of stable complex anions, precipitation of ferric salts, iron exchange, solvent extraction—only the addition of aluminium sulphate to form a white Mg-Al-Fe precipitate results in the generation of pregnant liquors with [Fe × 100/Mg] concentration ratios of the order required.

- Addition of aluminium sulphate does not affect the rate of magesium dissolution.

- The [Fe × 100/Mg] concentration ratio and magnesium losses are directly related to the aluminium sulphate addition; optimum conditions have yet to be determined for a range of leaching conditions.

PROPOSED FUTURE WORK PROGRAMME

During the next three months period, experimental work will be chiefly directed towards developing the iron removal step using aluminium sulphate. If this development is successful, then a range of intermediate and final products will be prepared and carefully characterized in terms of their physical and chemical properties.

The capital and operating cost estimates currently being carried out by consultants to Industrial and Mining Investigations Pty Ltd should become available and it is hoped that these will highlight any process areas requiring refinement.

On the basis of the results obtained, further consideration will be given to the commencement of technical scale studies.

The current literature of the production of high grade magnesium oxide using various alternative routes will continue to be assessed for suitability for treatment of Savage River magnesite.

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TABLE 1

Autoclave Leach Test Data - Effect of Calcination Conditions

Sample: MAG 1
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	57		58		59		60	
Calcination Temperature (°C)	600		700		850		970	
Calcination Time (h)	1		1		1		1	
Calcine Mg Content (%)	13.6		15.1		17.6		21.9	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)							
	Mg	Ca	Mg	Ca	Mg	Ca	Mg	Ca
0.08	0.320	0.126	0.44	0.118	0.30	0.264	0.12	0.374
0.25	0.410	0.174	1.11	0.130	1.04	0.235	0.96	0.190
0.50	0.540	0.186	1.72	0.140	1.62	0.204	3.84	0.305
0.75	0.640	0.188	1.92	0.146	2.16	0.204	3.84	0.305
1.00	0.705	0.190	2.09	0.150	2.40	0.205	4.30	0.350
1.25	0.762	0.196	2.19	0.158	2.66	0.210	4.60	0.391
1.50	0.805	0.196	2.22	0.161	2.86	0.210	4.80	0.410
1.75	0.840	0.194	2.29	0.161	2.99	0.210	4.92	0.422
2.00	0.840	0.196	2.34	0.161	3.10	0.210	5.02	0.434
2.25	0.840	0.196	2.36	0.161	3.24	0.210	5.02	0.434
2.50	0.840	0.198	2.36	0.161	3.32	0.210	5.02	0.434
Ca/Mg of Final Liquor	0.24		0.07		0.06		0.09	
Mg Extraction (%)	21		52		63		77	

See Figure 3

TABLE 2

Autoclave Leach Test Data - Effect of Calcination Conditions

Sample: MAG 1
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	61		62		63		64	
Calcination Temperature (°C)	590		700		850		970	
Calcination Time (h)	3		3		3		3	
Calcine Mg Content (%)	13.8		16.6		21.2		22.6	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)							
	Mg	Ca	Mg	Ca	Mg	Ca	Mg	Ca
0.08	0.124	0.094	0.20	0.060	0.08	0.386	0.12	0.442
0.25	0.373	0.106	0.60	0.036	1.22	0.208	0.70	0.302
0.50	0.477	0.120	1.22	0.024	2.94	0.268	1.98	0.279
0.75	0.490	0.131	1.66	0.027	3.80	0.340	3.02	0.372
1.00	0.499	0.141	2.08	0.034	4.18	0.390	3.72	0.488
1.25	0.509	0.150	2.40	0.040	4.36	0.420	4.20	0.566
1.50	0.518	0.156	2.60	0.048	4.50	0.436	4.42	0.610
1.75	0.518	0.162	2.74	0.052	4.56	0.446	4.63	0.633
2.00	0.520	0.164	2.86	0.060	4.64	0.456	4.70	0.651
2.25	0.520	0.169	3.00	0.068	4.68	0.460	4.84	0.660
2.50	0.520	0.174	3.10	0.071	4.73	0.466	4.95	0.674
Ca/Mg of Final Liquor	0.33		0.02		0.10		0.14	
Mg Extraction (%)	13		62		74		73	

See Figure 4

TABLE 3

Autoclave Leach Test Data - Effect of Calcination Conditions

Sample: MAG 1
Particle Size: $-\frac{1}{4}$"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	58		62		65	
Calcination Temperature (°C)	700		700		700	
Calcination Time (h)	1		3		5	
Calcine Mg Content (%)	15.1		16.6		16.5	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)					
	Mg	Ca	Mg	Ca	Mg	Ca
0.08	0.44	0.118	0.20	0.060	0.70	0.250
0.25	1.11	0.130	0.60	0.036	1.40	0.224
0.50	1.72	0.140	1.22	0.024	2.10	0.216
0.75	1.92	0.146	1.66	0.027	2.50	0.220
1.00	2.09	0.150	2.08	0.034	2.86	0.230
1.25	2.19	0.158	2.40	0.040	3.10	0.240
1.50	2.22	0.161	2.60	0.048	3.20	0.244
1.75	2.29	0.161	2.74	0.052	3.30	0.250
2.00	2.34	0.161	2.86	0.060	3.30	2.540
2.25	2.36	0.161	3.00	0.068	3.30	0.250
2.50	2.36	0.161	3.10	0.071	3.30	0.250
Ca/Mg of Final Liquor	0.07		0.02		0.08	
Mg Extraction (%)	52		62		67	

See Figure 5

TABLE 4

Autoclave Leach Test Data - Effect of Calcination Conditions

Sample: MAG 1
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	66		67		59		68		63	
Calcination Temperature (°C)	850		850		850		850		850	
Calcination Time (h)	0.25		0.5		1		2		3	
Calcine Mg Content (%)	16.1		17.4		17.6		20.6		21.2	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Ca	Mg	Ca	Mg	Ca	Mg	Ca	Mg	Ca
0.08	0.48	0.130	0.16	0.290	0.30	0.264	0.10	0.288	0.08	0.386
0.25	1.22	0.124	0.64	0.281	1.04	0.235	0.98	0.128	1.22	0.208
0.50	1.72	0.126	0.30	0.255	1.62	0.201	2.34	0.134	2.94	0.268
0.75	2.04	0.132	1.78	0.255	2.16	0.204	3.18	0.252	3.80	0.340
1.00	2.20	0.140	2.10	0.255	2.40	0.205	3.62	0.278	4.18	0.390
1.25	2.26	0.142	2.38	0.260	2.66	0.210	3.82	0.300	4.36	0.420
1.50	2.34	0.146	2.56	0.264	2.86	0.210	3.96	0.314	4.50	0.436
1.75	2.35	0.146	2.68	0.268	2.99	0.210	4.10	0.324	4.56	0.446
2.00	2.33	0.146	2.80	0.271	3.10	0.210	4.24	0.330	4.64	0.456
2.25	2.35	0.148	2.86	0.275	3.24	0.210	4.32	0.332	4.68	0.460
2.50	2.35	0.146	3.00	0.280	3.32	0.210	4.44	0.334	4.73	0.466
Ca/Mg of Final Liquor	0.06		0.09		0.06		0.08		0.10	
Mg Extraction (%)	49		57		63		72		74	

See Figure 6

TABLE 5

Autoclave Leach Test Data - Effect of Calcination Conditions

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	69		70		71		72		73	
Calcination Temperature (°C)	600		700		800		850		970	
Calcination Time (h)	1		1		1		1		1	
Calcine Mg Content (%)	29.4		40.8		44.6		46.5		46.3	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	1.93	0.000	1.44	0.013	0.24	0.000	0.20	0.000	0.02	0.000
0.25	3.18	0.001	6.06	0.027	1.30	0.000	0.34	0.000	0.21	0.000
0.50	3.29	0.001	9.44	0.041	3.10	0.001	0.87	0.000	0.45	0.000
0.75	3.31	0.001	10.40	0.099	4.14	0.003	1.49	0.000	0.66	0.000
1.00	3.25	0.001	10.76	0.102	5.20	0.006	2.01	0.000	0.94	0.000
1.25	3.29	0.001	10.82	0.102	5.86	0.009	2.60	0.000	1.25	0.000
1.50	3.29	0.001	10.96	0.102	6.60	0.010	3.09	0.000	1.38	0.000
1.75	3.28	0.001	11.05	0.102	7.10	0.012	3.60	0.000	1.66	0.000
2.00	3.29	0.001	11.10	0.102	7.50	0.013	4.20	0.001	2.02	0.000
2.25	3.29	0.001	11.10	0.102	8.00	0.012	4.58	0.002	2.30	0.000
2.50	3.29	0.001	11.10	0.102	8.40	0.012	5.03	0.003	2.55	0.000
[Fe × 100/Mg] of Final Liquor	0.03		0.93		0.14		0.06		0.00	
Mg Extraction (%)	57		91		63		36		18	

See Figure 8

TABLE 6

Autoclave Leach Test Data - Effect of Calcination Conditions

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	74		75		70		76		77		78	
Calcination Temperature (°C)	700		700		700		700		700		700	
Calcination Time (h)	0.25		0.5		1		2		3		5	
Calcine Mg Content (%)	26.2		33.5		40.8		43.5		43.0		43.3	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)											
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	1.00	0.000	3.00	0.013	1.44	0.013	1.46	0.000	1.54	0.000	1.90	0.000
0.25	1.18	0.000	4.46	0.022	6.06	0.027	5.78	0.028	6.10	0.017	5.90	0.018
0.50	1.30	0.000	5.00	0.026	9.44	0.041	8.50	0.050	8.50	0.044	7.88	0.041
0.75	1.51	0.000	5.20	0.027	10.40	0.099	9.40	0.065	9.40	0.055	9.10	0.050
1.00	1.52	0.000	5.40	0.029	10.76	0.102	9.80	0.072	9.96	0.059	9.48	0.058
1.25	1.52	0.000	5.44	0.028	10.82	0.102	10.26	0.077	10.32	0.063	9.70	0.064
1.50	1.52	0.000	5.42	0.029	10.96	0.102	10.60	0.079	10.60	0.066	10.00	0.066
1.75	1.51	0.000	5.44	0.029	11.05	0.102	10.81	0.080	10.60	0.069	10.15	0.069
2.00	1.51	0.000	5.44	0.029	11.10	0.102	10.94	0.080	10.60	0.071	10.31	0.071
2.25	1.52	0.000	5.44	0.029	11.10	0.102	10.94	0.080	10.60	0.073	10.46	0.073
2.50	1.52	0.000	5.44	0.029	11.10	0.102	10.94	0.080	10.60	0.074	10.60	0.074
[Fe × 100/Mg] of Final Liquor	0.00		0.53		0.93		0.73		0.70		0.70	
Mg Extraction (%)	19		54		91		84		82		82	

See Figure 9

TABLE 7

Autoclave Leach Test Data - Effect of Leaching Conditions

Sample: MAG 3
Particle Size: $-\frac{1}{4}$"+7 mesh ground to 100% -100 mesh
Calcination Conditions: 700°C for 3 h
Leach Conditions: 1 litre water (Mg and Fe free), 1200 rpm

Run Number	42		44[b]		45		46		47	
Calcine Weight (g)	30		30		30		30		30	
Slake Time (h)	0.5		0.5		0.2		15.5		0.5[c]	
Slake Temperature (°C)	15.5		15.5		15.5		15.5		85	
Leach Temperature (°C)	15.5		15.5		15.5		15.5		15.5	
CO_2 Pressure (psig)	100		100		100		100		100	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	3.66	0.010	0.90	0.000	3.26	0.011	4.30	0.026	2.00	0.066
0.25	7.66	0.058	3.24	0.012	7.52	0.058	7.08	0.108	6.32	0.125
0.50	8.88	0.082	6.88	0.042	9.10	0.080	8.60	0.152	8.72	0.238
0.75	9.24	0.089	8.56	0.060	9.30	0.090	9.19	0.172	9.40	0.312
1.00	9.60	0.095	9.10	0.072	9.30	0.093	9.20	0.174	9.41	0.366
1.25	9.60	0.095	9.30	0.074	9.30	0.093	9.30	0.172	9.41	0.366
1.50	9.50	0.093	9.30	0.072	9.30	0.093	9.20	0.172	9.41	0.366
1.75	9.60	0.093	9.30	0.072	9.30	0.093	9.20	0.172	9.41	0.366
2.00	9.50	0.093	9.30	0.072	9.30	0.093	9.20	0.172	9.41	0.366
2.25			9.30	0.072	9.30	0.093	9.20	0.172	9.41	0.366
2.50			9.30	0.072	9.30	0.093	9.20	0.172	9.41	0.366
[Fe × 100/Mg] of Final Liquor	0.98		0.77		1.00		1.87		3.89	
Mg Extraction (%)[a]	81		79		79		78		80	

[a] Based on calcine composition of 39.1% Mg.
[b] Gas inlet of sub-aerator sealed.
[c] Plus additional 0.25 h for cool down.

See Figures 14-21

TABLE 7 (continued)

Autoclave Leach Test Data - Effect of Leaching Conditions

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Calcination Conditions: 700°C for 3 h
Leach Conditions: 1 litre water (Mg and Fe free), 1200 rpm

Run Number	48		49		50		51		52	
Calcine Weight (g)	20		40		50		30		50	
Slake Time (h)	0.5		0.5		0.5		0.5		0.5	
Slake Temperature (°C)	15.5		15.5		15.5		15.5		15.5	
Leach Temperature (°C)	15.5		15.5		15.5		15.5		15.5	
CO_2 Pressure (psig)	100		100		100		25		25	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	3.00	0.004	4.50	0.024	3.50	0.018	1.20	0.018	1.70	0.010
0.25	5.10	0.014	9.60	0.136	11.91	0.190	4.80	0.052	5.90	0.078
0.50	5.66	0.013	11.92	0.201	15.00	0.300	7.70	0.068	10.80	0.180
0.75	6.08	0.014	12.44	0.232	16.60	0.368	8.90	0.073	13.90	0.253
1.00	6.18	0.013	12.60	0.247	16.60	0.390	9.20	0.077	14.75	0.324
1.25	6.18	0.014	12.60	0.252	16.60	0.390	9.20	0.076	15.55	0.365
1.50	6.18	0.013	12.60	0.257	16.60	0.395	9.20	0.076	16.00	0.398
1.75	6.21	0.013	12.60	0.263	16.60	0.400	9.20	0.076	16.30	0.420
2.00	6.18	0.013	12.60	0.263	16.60	0.400	9.20	0.076	16.30	0.430
2.25	6.18	0.014	12.60	0.263	16.60	0.400	9.20	0.076	16.30	0.430
2.50	6.18	0.013	12.60	0.263	16.60	0.400	9.20	0.076	16.30	0.430
[Fe × 100/Mg] of Final Liquor	0.21		2.09		2.41		0.83		2.64	
Mg Extraction (%) [a]	79		81		85		78		83	

[a] Based on calcine composition of 39.1% Mg.

See Figures 14-21

TABLE 7 (continued)

Autoclave Leach Test Data - Effect of Leaching Conditions

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Calcination Conditions: 700°C for 3 h
Leach Conditions: 1 litre water (Mg and Fe free), 1200 rpm

Run Number	53		54		55		56		79[b]	
Calcine Weight (g)	50		30		30		30		30	
Slake Time (h)	0.5		0.5		0.5		0.5		0.5	
Slake Temperature (°C)	30.0		6.0		30.0		15.5		15.5	
Leach Temperature (°C)	30.0		6.0		30.0		15.5		15.5	
CO_2 Pressure (psig)	25		100		100		50		100	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	1.50	0.000	2.41	0.006	5.50	0.018	2.76	0.000	0.38	0.000
0.25	3.10	0.001	5.30	0.040	9.10	0.021	6.90	0.040	0.76	0.000
0.50	4.75	0.003	6.60	0.076	9.30	0.025	8.99	0.066	1.07	0.000
0.75	5.66	0.004	7.30	0.098	9.35	0.027	9.28	0.072	1.45	0.000
1.00	5.80	0.004	8.10	0.118	9.30	0.028	9.30	0.074	1.70	0.000
1.25	5.85	0.003	8.50	0.128	9.30	0.029	9.31	0.074	1.21	0.000
1.50	5.85	0.003	8.80	0.140	9.30	0.027	9.28	0.074	0.85	0.000
1.75	5.83	0.004	9.05	0.146	9.30	0.030	9.30	0.074	0.73	0.000
2.00	5.85	0.003	9.20	0.150	9.32	0.028	9.30	0.074	1.02	0.000
2.25	5.83	0.003	9.25	0.153	9.30	0.029	9.30	0.074	1.45	0.000
2.50	5.83	0.003	9.25	0.154	9.30	0.028	9.30	0.074	2.23	0.000
[Fe × 100/Mg] of Final Liquor	0.05		1.66		0.30		0.80		0.00	
Mg Extraction[a]	50		79		79		79		19	

[a]Based on calcine composition of 39.1% Mg.

[b]At 640 rpm.

See Figures 14-21

TABLE 8

Magnesium Extraction (%) and [Fe × 100/Mg] Concentration Ratio as a Function of Leaching Conditions

Run Number	Slake Time (h)	Slake Temperature (°C)	Calcine Weight (gpℓ)	Leach Temperature (°C)	CO_2 Pressure (psig)	Agitation (rpm)	Mg[a] Extraction (%)	Concentration Ratio [Fe × 100/Mg]	Figure
45	0.2	15.5	30	15.5	100	1200	79	1.00	
42	0.5	15.5	30	15.5	100	1200	81	0.98	14
46	15.5	15.5	30	15.5	100	1200	78	1.87	
42	0.5	15.5	30	15.5	100	1200	81	0.98	15
47	0.5	85	30	15.5	100	1200	80	3.89	
51	0.5	15.5	30	15.5	25	1200	78	0.83	16
52	0.5	15.5	50	15.5	25	1200	83	2.64	
48	0.5	15.5	20	15.5	100	1200	79	0.21	
42	0.5	15.5	30	15.5	100	1200	81	0.98	17
49	0.5	15.5	40	15.5	100	1200	81	2.09	
50	0.5	15.5	50	15.5	100	1200	85	2.41	
54	0.5	6.0	30	6.0	100	1200	79	1.66	
42	0.5	15.5	30	15.5	100	1200	81	0.98	18
55	0.5	30.0	30	30.0	100	1200	79	0.30	
52	0.5	15.5	50	15.5	25	1200	83	2.64	19
53	0.5	30.0	50	30.0	25	1200	50	0.05	
51	0.5	15.5	30	15.5	25	1200	78	0.83	
56	0.5	15.5	30	15.5	50	1200	79	0.80	20
42	0.5	15.5	30	15.5	100	1200	81	0.98	
79	0.5	15.5	30	15.5	100	640	19	0.00	
16	0.5	15.5	30	15.5	100	900	81	0.58	21
42	0.5	15.5	30	15.5	100	1200	81	0.98	
44[b]	0.5	15.5	30	15.5	100	1200[b]	79	0.77	

[a]Based on calcine composition of 39.1% Mg.
[b]Gas inlet of sub-aerator sealed.

TABLE 9

Autoclave Leach Test Data - Effect of Leachant Composition

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Calcination Conditions: 700°C for 3 h
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	42				80				81			
Initial Mg Content of Leachant (gpℓ)	0.000				1.52				4.26			
Initial Fe Content of Leachant (gpℓ)	0.000				0.000				0.020			
Leach Time (h)	Pregnant Liquor Composition (gpℓ)											
	Measured		Corrected[b]		Measured		Corrected		Measured		Corrected	
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	3.66	0.010	3.66	0.010	5.20	0.021	3.68	0.021	6.82	0.032	2.56	0.012
0.25	7.66	0.058	7.66	0.058	9.40	0.075	7.88	0.075	11.82	0.142	7.56	0.122
0.50	8.88	0.082	8.88	0.082	10.36	0.108	8.84	0.108	13.44	0.180	9.18	0.160
0.75	9.24	0.089	9.24	0.089	10.90	0.115	9.38	0.115	13.80	0.200	9.54	0.180
1.00	9.60	0.095	9.60	0.095	11.00	0.122	9.48	0.122	13.80	0.210	9.54	0.190
1.25	9.60	0.095	9.60	0.095	11.00	0.124	9.48	0.124	13.80	0.210	9.54	0.190
1.50	9.50	0.093	9.50	0.093	11.00	0.122	9.48	0.122	13.85	0.210	9.59	0.190
1.75	9.60	0.093	9.60	0.093	11.00	0.124	9.48	0.124	13.80	0.210	9.54	0.190
2.00	9.50	0.093	9.50	0.093	11.00	0.124	9.48	0.124	13.80	0.210	9.54	0.190
2.25					11.00	0.124	9.48	0.124	13.80	0.210	9.54	0.190
2.50					11.00	0.124	9.48	0.124	13.80	0.210	9.54	0.190
[Fe × 100/Mg] of Final Liquor			0.98				1.31				1.99	
Mg Extraction[a]			81				81				81	

[a]Based on calcine composition of 39.1% Mg.
[b]Measured concentration - Initial concentration.

See Figure 22

TABLE 10

Rate of Precipitation Data

Run Number	82		83		84		85	
Temperature (°C)	20 ± 2		20 ± 2		44 ± 1		65 ± 2	
Air Flow (ℓpm)	0		5		5		5	
Precipitation Time (h)	Pregnant Liquor Composition (gpℓ)							
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0	8.71	0.092	9.40	0.095	9.30	0.097	8.93	0.092
0.25			7.80	0.087	2.54	0.002	1.25	0.000
0.50			6.26	0.073	1.10	0.000	0.55	0.000
0.75			4.74	0.055				
1.00			3.70	0.038	0.52	0.000	0.38	0.000
1.25			3.00	0.025				
1.50			2.41	0.014	0.40	0.000		
1.75			1.99	0.008				
2.00	8.50	0.090	1.70	0.004	0.30	0.000		
2.25			1.39	0.003				
2.50			1.20	0.002				
2.75			1.16	0.002				
3.00			1.11	0.001				
3.75			1.05	0.001				
4.00	8.31	0.088						
24	7.99	0.068						
30	7.70	0.066						
46	7.10	0.061						
70	6.42	0.053						
148	5.21	0.031						

See Figure 23

TABLE 11

Dissolution of Iron from Calcined Magnesite, Natural and Synthetic Ferric Oxide and Freshly Precipitated Ferric Hydroxide in the Absence and Presence of Additives

Leaching Conditions: 15.5°C, 100 psig CO_2, 1200 rpm, 1.5 h, 1 litre water (Mg and Fe free)

Run Number	Iron-containing Reactant (g)				Additive (g)		Iron Concentration ppm
	MAG 3[a]	N-Fe_2O_3[b]	S-Fe_2O_3[c]	$Fe(OH)_3$[d]	MgO[e]	Na_2CO_3	
42	30	0	0	0	0	50	93
86	30	0	0	0	0	50	460
87	0	2	0	0	0	0	0
88	0	2	0	0	0	50	3.1
89	0	2	0	0	20	0	0.92
90	0	2	0	0	50	0	4.2
91	0	0	2	0	0	0	0
92	0	0	2	0	0	50	2.9
93	0	0	2	0	20	0	0.86
94	0	0	0	≈5	0	0	0
95	0	0	0	≈5	0	50	5.7
96	0	0	0	≈5	20	0	2.6

[a] Bulk calcine.

[b] Type III Tom Price natural hematite.

[c] Synthetic hematite.

[d] Freshly precipitated ferric hydroxide added as wet paste.

[e] AR $MgCO_3$ calcined at 700°C for 3 hours.

TABLE 12

Removal of Iron from Clarified Pregnant Liquors[a]

Test No.	Additive	Expected Reaction[b]	Observed Reaction
1	Chromatographic silica gel	Adsorption of iron oxide	No measurable adsorption
2	Molecular sieve (Type 3A)	Adsorption of iron oxide	No measurable adsorption
3	Activated charcoal	Adsorption of iron oxide	No measurable adsorption
4	Chromatographic alumina	Adsorption of iron oxide	No measurable adsorption
5	Kaolin	Adsorption of iron oxide	No measurable adsorption
6	Sodium chloride	Stabilization of iron as chloroferrate (III) complex anion	No apparent reaction
7	Sodium sulphate	Stabilization of iron as sulphatoferrate (III) complex anion	No apparent reaction
8	Sodium citrate	Stabilization of iron as ferric citrate	Solution changed from red-brown to pale yellow indicating formation of complex anion
9	*d*-glucose	Stabilization of iron as ferric *d*-glucose complex	Slight change in solution colour
10	Sodium dihydrogen orthophosphate	Precipitation of ferric phosphate	White precipitate (magnesium phosphate)
11	Disodium hydrogen arsenate	Precipitation of ferric arsenate	White precipitate (magnesium arsenate)
12	Sodium fluoride	Stabilization of iron as fluoroferrate (III) complex anion	White precipitate (magnesium fluoride)
13	Potassium ethyl xanthate	Precipitation of ferric ethyl xanthate	No apparent reaction
14	Hydroxylamine sulphate	Reduction of ferric iron to ferrous iron	Solution turned dark red indicating reduction; nesquehonite badly stained

15	Potassium polysulphide	Precipitation of iron sulphide	Precipitation of very fine black sulphide; very difficult to filter
16	Ammonium thiocyanate	Stabilization of iron as thiocyanato-ferrate (III) complex anion	No apparent reaction
17	Aluminium sulphate	Precipitation of iron-aluminium basic salt	Effervescence and formation of white precipitate
18	Sodium aluminate	Precipitation of iron-aluminium basic salt	Formation of white precipitate
19	Hydrogen sulphide[c]	Precipitate of iron sulphide	Precipitation of very fine black sulphide, very difficult to filter
20	Tributyl phosphate[d]	Extraction of ferric iron into organic phase	No measurable extraction
21	Alamine 336[e]	Extraction of ferric iron into organic phase	No measurable extraction
22	Aliquot 336[e]	Extraction of ferric iron into organic phase	No measurable extraction
23	Cross-linked casein	Ion exchange of iron	Casein turned red-brown; pregnant liquor lighter in colour after treatment

[a] Mg and Fe contents in the ranges 9.20–950 gpℓ and 0.090–0.097 gpℓ respectively, derived from MAG 3 bulk calcine.

[b] 10 g additive per litre pregnant liquor.

[c] Bubbled through liquor at rate of 200 ml/min.

[d] Aqueous/organic ratio = 5/1, no diluent, shaken for 30 min.

[e] 5% (V/V) in Shell EC561 kerosene, aqueous/organic ratio = 5/1, shaken for 30 min.

TABLE 13

Removal of Iron from Clarified Pregnant Liquors as a Function of Aluminium Sulphate Addition

Test Number	$Al_2(SO_4)_3 \cdot 16H_2O$ (gpℓ)	Filtrate Composition (gpℓ)[a]		[Fe × 100/Mg]	Mg Extraction (%)[b]
		Mg	Fe		
23	0	8.62	0.092	1.07	100
24	1	8.62	0.079	0.92	100
25	4	8.60	0.045	0.52	99.8
26	7	8.60	0.027	0.31	99.8
27	10	8.00	0.019	0.24	92.8

[a] Filtered after standing at room temperature for 30 min.

[b] Based upon Mg content of pregnant liquor without additive

See Figure 13

TABLE 14

Removal of Iron during Leaching

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Calcination Conditions: 700°C for 3 hours
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm, 2.5 h leach

Run No.	Additive[a]	Pregnant Liquor Composition (gpℓ) Mg	Fe	[Fe × 100/Mg]	Mg Extraction (%)[b]
42	Nil	9.50	0.093	0.98	81
97	Chromatographic silica gel	9.50	0.090	0.95	81
98	Molecular sieves (Type 3A)	9.49	0.094	0.99	81
99	Activated charcoal	9.50	0.093	0.98	81
100	Chromatographic alumina	9.20	0.089	0.97	78
101	Kaolin	9.50	0.094	0.99	81
102	Sodium chloride	9.30	0.094	1.01	79
103	Sodium sulphate	9.50	0.088	0.93	81
104	Sodium citrate	9.41	0.700	7.44	80
105	*d*-glucose	9.35	0.094	1.01	80
106	Aluminium sulphate	7.78	0.009	0.12	66[c]
107	Sodium aluminate	8.80	0.069	0.78	75
107A	Cross-linked casein	9.42	0.091	0.97	80

[a] 10 g additive.

[b] Based on calcine composition of 39.1% Mg.

[c] This is equivalent to 82% extraction of available Mg (assumed to be equivalent to 9.50 gpℓ)

TABLE 15

Removal of Iron during Leaching as a Function of Aluminium Sulphate Addition

Sample: MAG 3
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Calcination Conditions: 700°C for 3 hours
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm

Run Number	42		108		109		110		106	
$Al_2(SO_4)_3 \cdot 15H_2O$ (g)	0		1		4		7		10	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	3.66	0.010	3.69	0.008	2.90	0.001	2.55	0.000	1.96	0.000
0.25	7.66	0.058	7.64	0.045	6.40	0.013	5.92	0.003	5.74	0.001
0.50	8.88	0.082	8.50	0.061	7.22	0.016	7.43	0.006	7.10	0.005
0.75	9.24	0.089	8.82	0.066	8.10	0.019	7.80	0.008	7.50	0.007
1.00	9.60	0.095	8.90	0.066	8.30	0.020	8.00	0.009	7.78	0.009
1.25	9.60	0.095	8.90	0.066	8.29	0.021	8.10	0.010	7.78	0.009
1.50	9.50	0.093	8.90	0.066	8.29	0.022	8.10	0.012	7.78	0.009
1.75	9.60	0.093	8.90	0.066	8.29	0.022	8.10	0.013	7.78	0.009
2.00	9.50	0.093	8.90	0.066	8.29	0.022	8.10	0.013	7.78	0.009
2.25			8.90	0.066	8.29	0.022	8.10	0.013	7.78	0.009
2.50			8.90	0.066	8.29	0.022	8.10	0.013	7.78	0.009
[Fe × 100/Mg] of Final Liquor	0.98		0.74		0.27		0.16		0.12	
Mg Extraction (%)[a]	81		76		71		69		66	
Mg Extraction (%)[b]	100		94		87		85		82	

[a] Based on calcine content of 39.1% Mg.

[b] Based on available Mg (assumed to be equivalent to 9.50 gpℓ).

See Figures 25 and 26

TABLE 16

Addition of Aluminium Sulphate during Leaching of Calcines derived from Dolomitic Sample

Sample: MAG 1
Particle Size: -¼"+7 mesh ground to 100% -100 mesh
Leach Conditions: 30 g calcine/litre, 0.5 h slake, 15.5°C, 100 psig CO_2 1200 rpm

Run Number	63		111		112		64		113	
Calcination Temperature (°C)	850		850		850		970		970	
Calcination Time (h)	3		3		3		3		3	
Calcine Mg Content (%)	21.2		21.2		21.2		22.6		22.6	
$Al_2(SO_4)_3 \cdot 16H_2O$ (g)	0		4		10		0		4	
Leach Time (h)	Pregnant Liquor Composition (gpℓ)									
	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe	Mg	Fe
0.08	0.08	0.386	0.26	0.636	0.24	1.12	0.12	0.442	0.24	1.17
0.25	1.22	0.208	1.03	0.418	0.84	0.860	0.70	0.302	0.64	0.976
0.50	2.94	0.268	2.11	0.328	1.66	0.745	1.98	0.279	1.81	0.700
0.75	3.80	0.340	3.03	0.328	2.02	0.722	3.02	0.372	2.75	0.618
1.00	4.18	0.390	3.68	0.436	2.31	0.710	3.72	0.488	3.36	0.605
1.25	4.36	0.420	3.95	0.490	2.50	0.815	4.20	0.586	3.86	0.605
1.50	4.50	0.436	4.08	0.491	2.53	0.839	4.42	0.610	4.27	0.652
1.75	4.56	0.446	4.21	0.491	2.53	0.860	4.63	0.633	4.53	0.665
2.00	4.64	0.456	4.24	0.491	2.54	0.860	4.70	0.651	4.66	0.676
2.25	4.68	0.460	4.29	0.491	2.54	0.860	4.84	0.660	4.62	0.676
2.50	4.73	0.460	4.32	0.491	2.53	0.860	4.95	0.674	4.66	0.676
Ca/Mg of Final Liquor	0.10		0.11		0.34		0.14		0.15	
Mg Extraction (%)	74		68		40		73		69	

See Figure 27

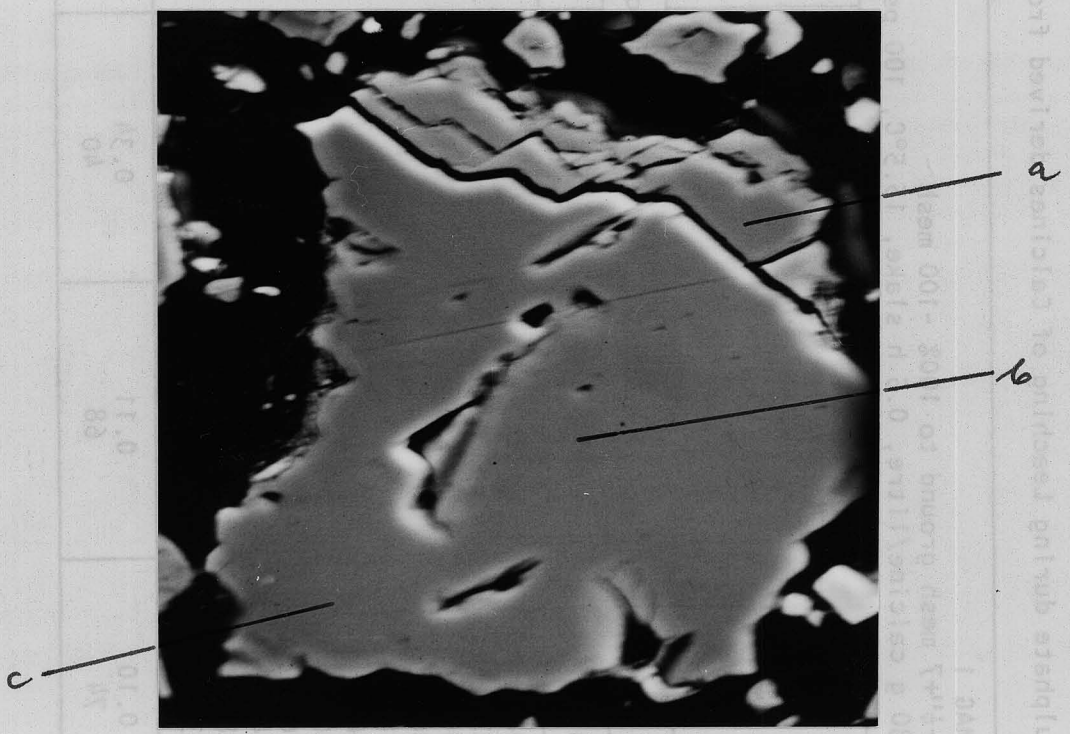

FIGURE 1. Back-scattered electron image of magnesium oxide grain of MAG 3 bulk calcine showing EDAX analysis points. ×800.

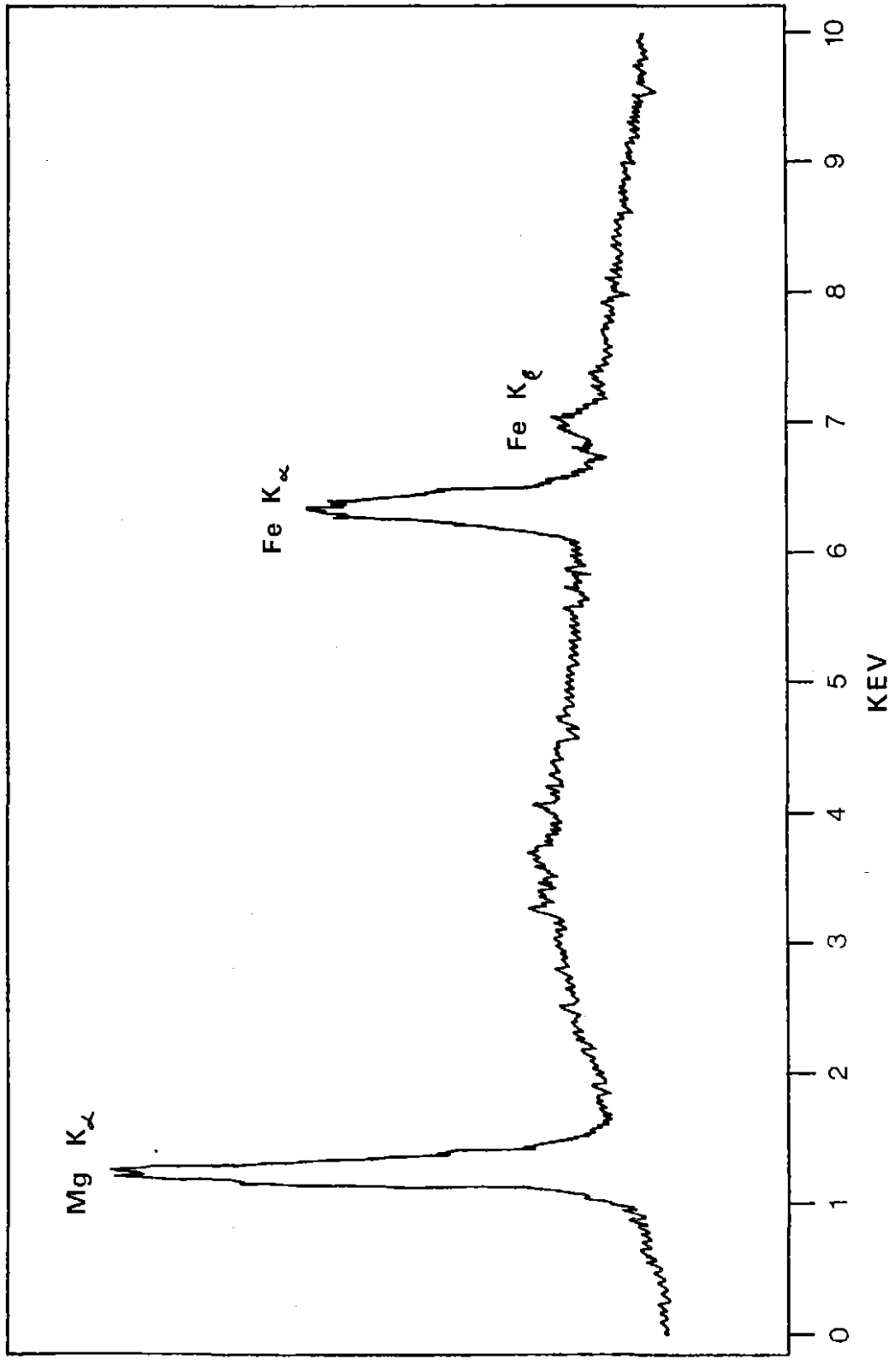

FIGURE 2. EDAX trace of magnesium oxide grain (point *C*).

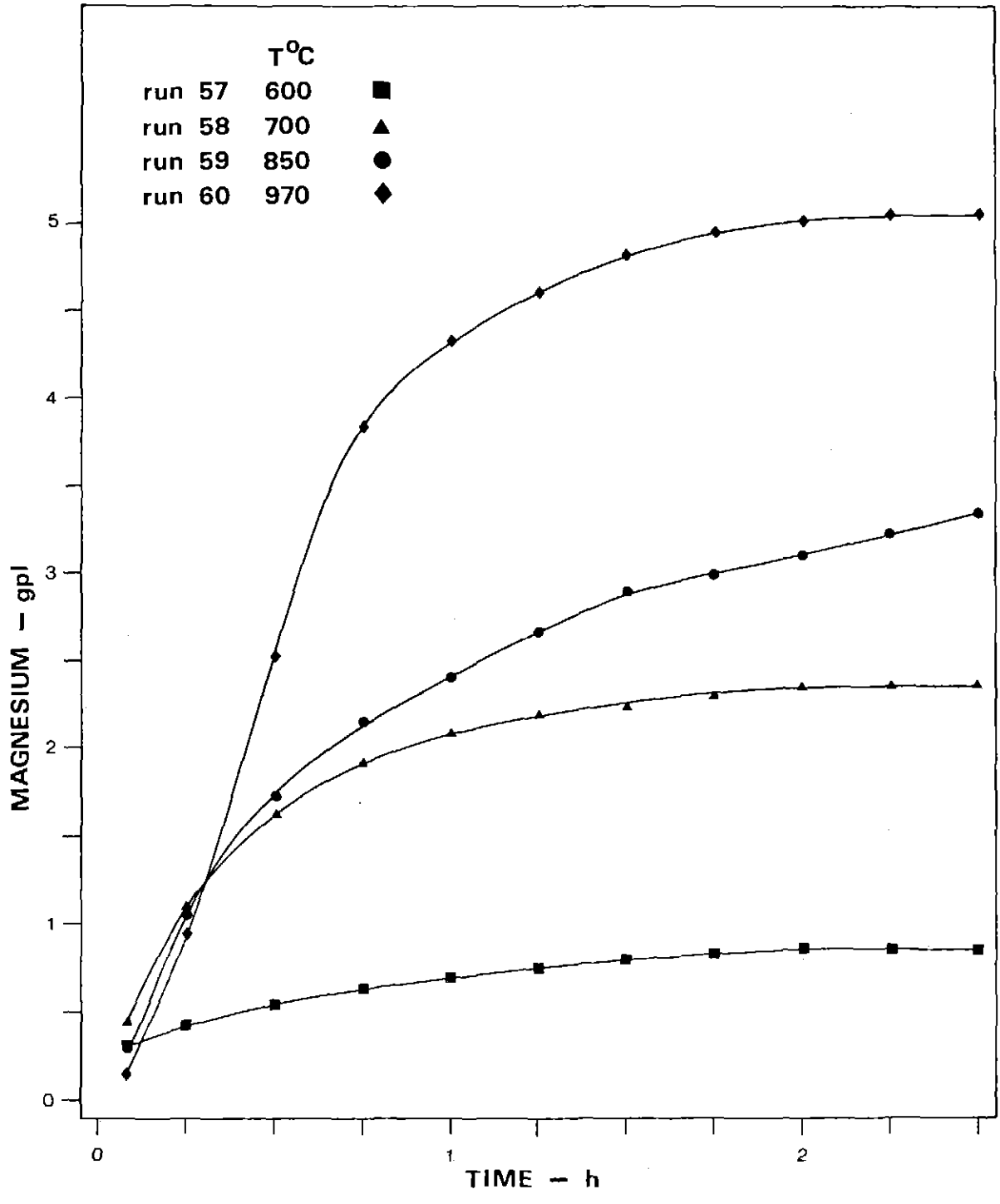

FIGURE 3. Autoclave leaching kinetic data; the effect of calcination time on magnesium concentration. MAG 1 calcined for 1 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 1).

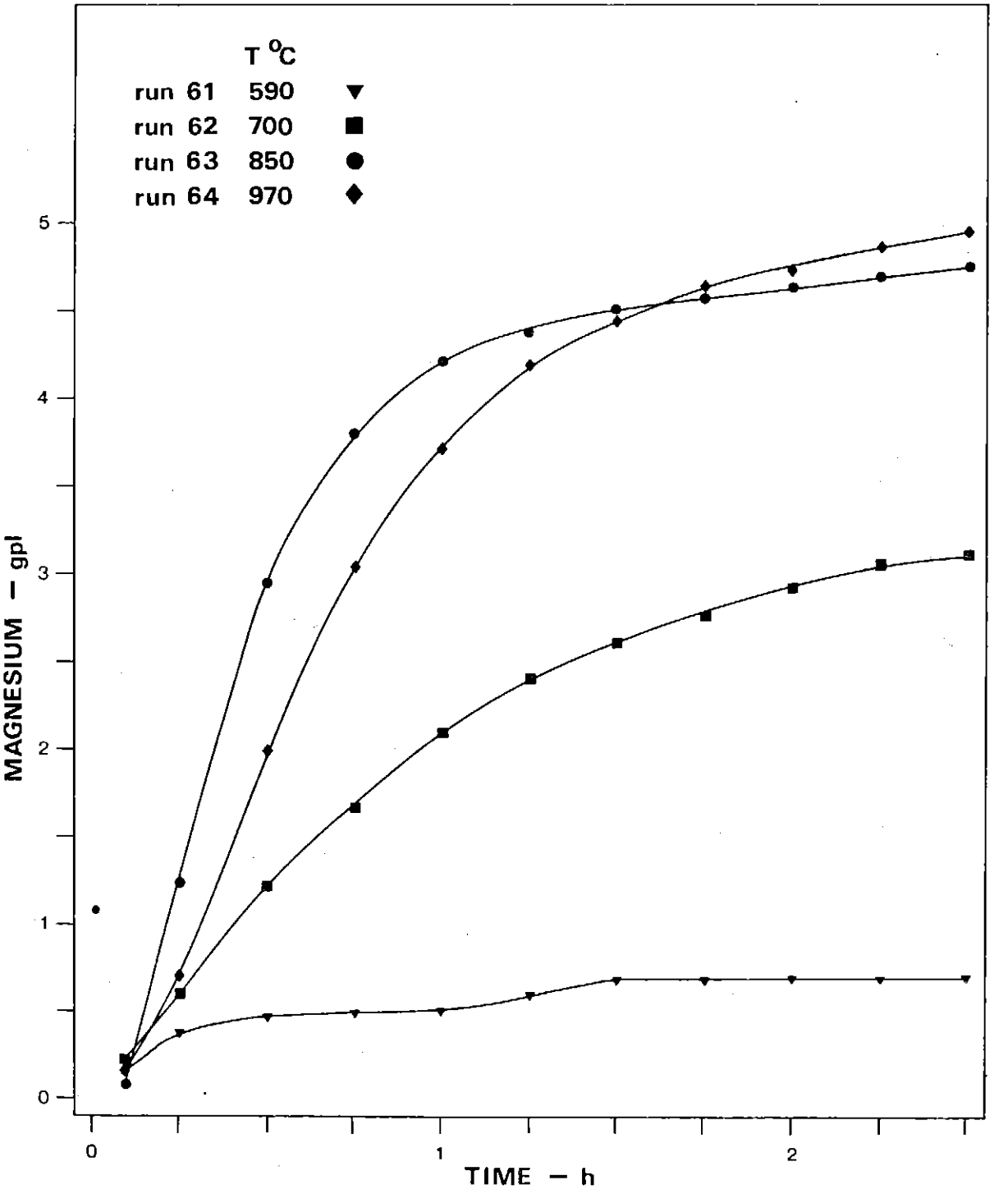

FIGURE 4. Autoclave leaching kinetic data; the effect of calcination temperature on magnesium concentration. MAG 1 calcined for 3 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 2).

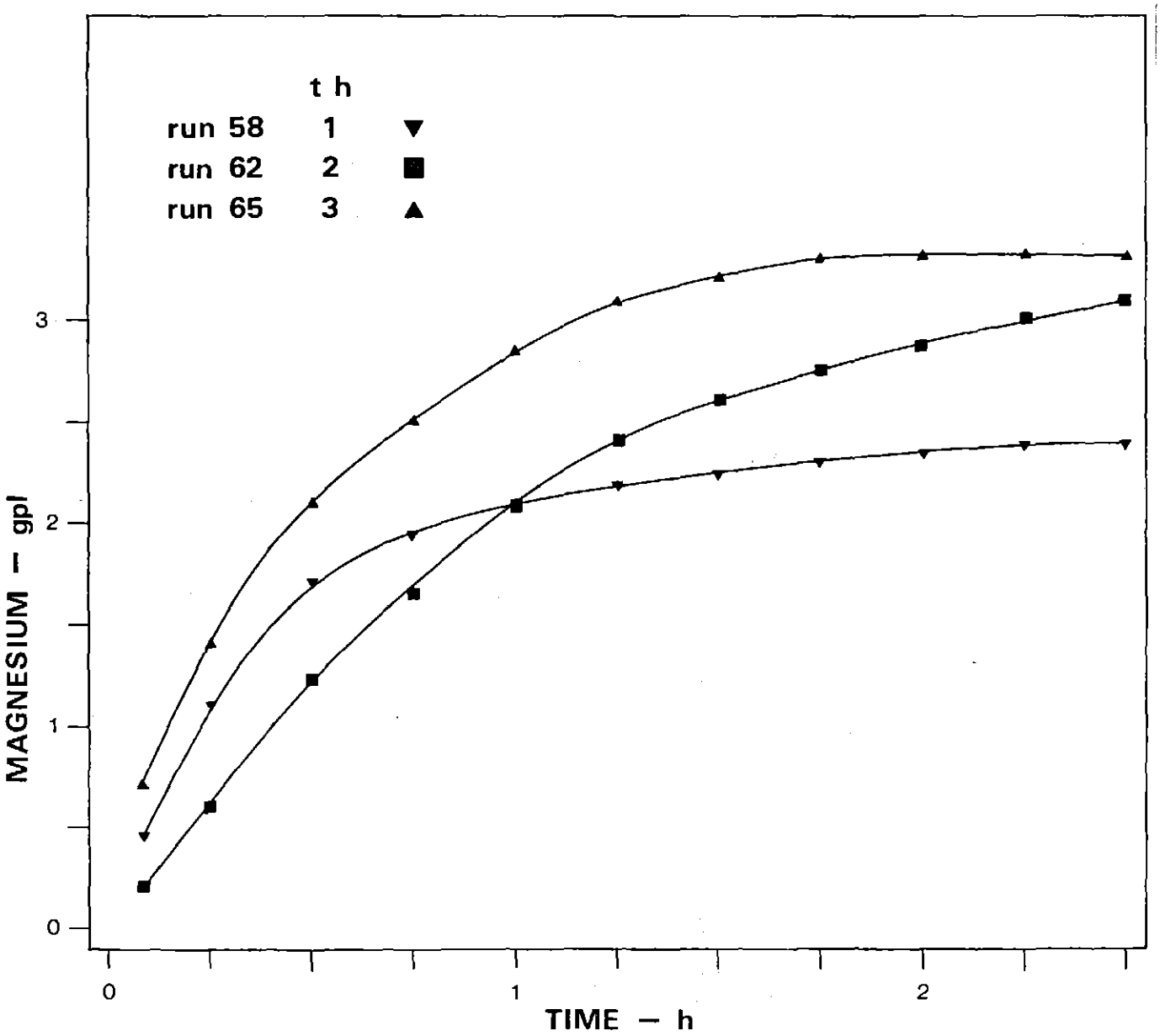

FIGURE 5. Autoclave leaching kinetic data; the effect of calcination time on magnesium concentration. MAG 1 calcined at 700°C, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 3).

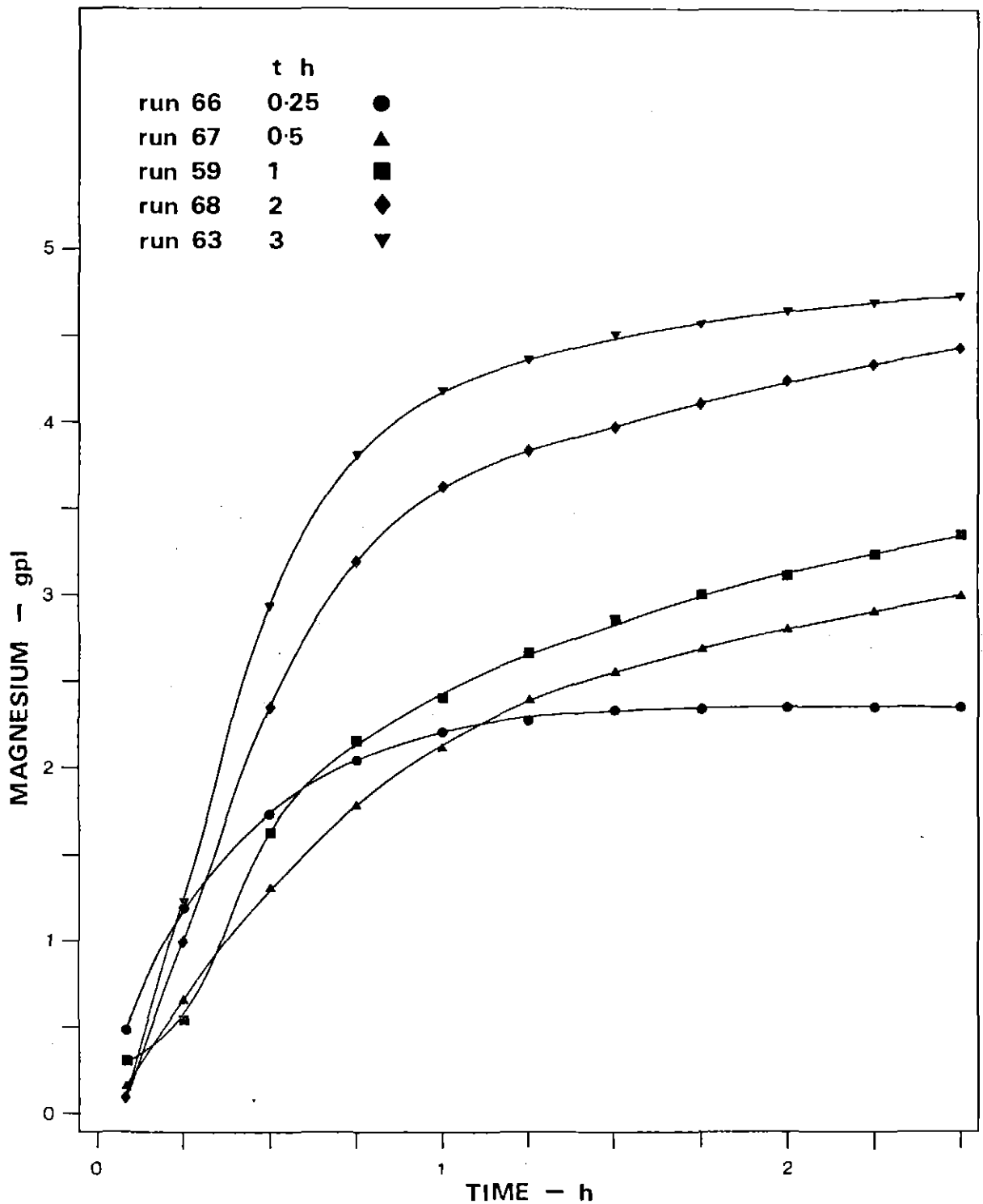

FIGURE 6. Autoclave leaching kinetic data; the effect of calcination time on magnesium concentration. MAG 1 calcined at 850°C, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 4).

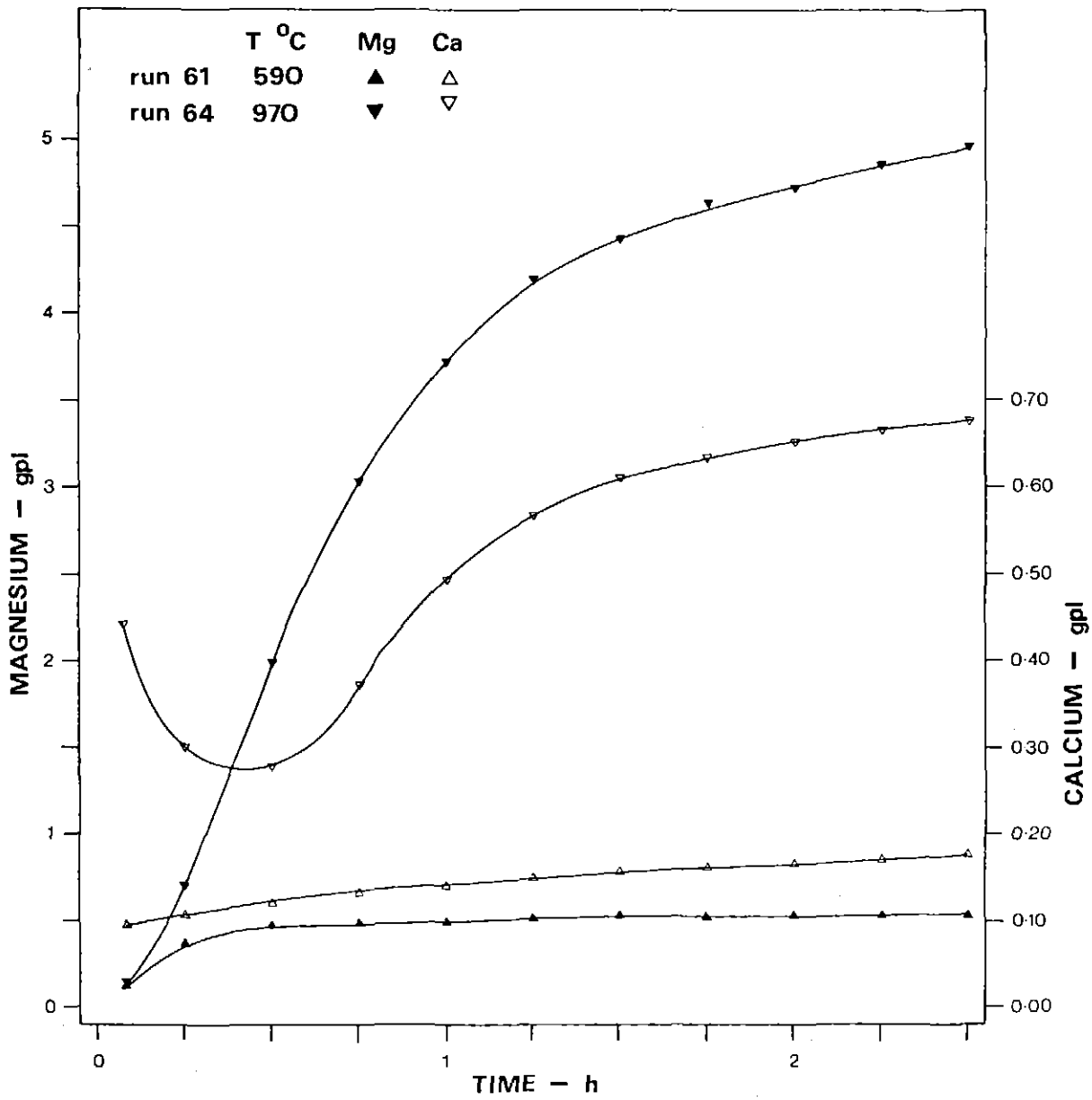

FIGURE 7. Autoclave leaching kinetic data; the effect of calcination temperature on calcium concentration. MAG 1 calcined for 1 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 2).

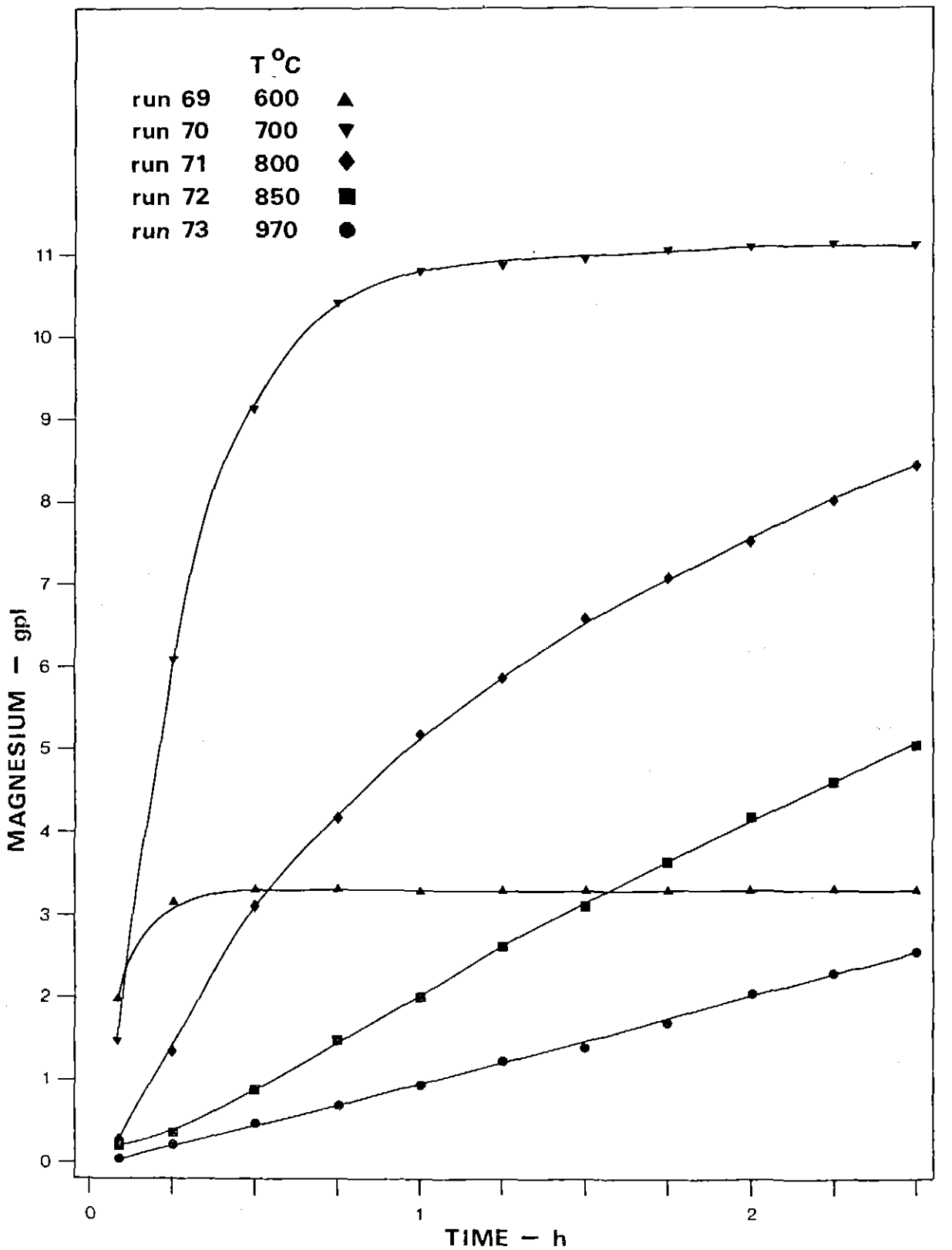

FIGURE 8. Autoclave leaching kinetic data; the effect of calcination temperature on magnesium concentration. MAG 3 calcined for 1 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 5).

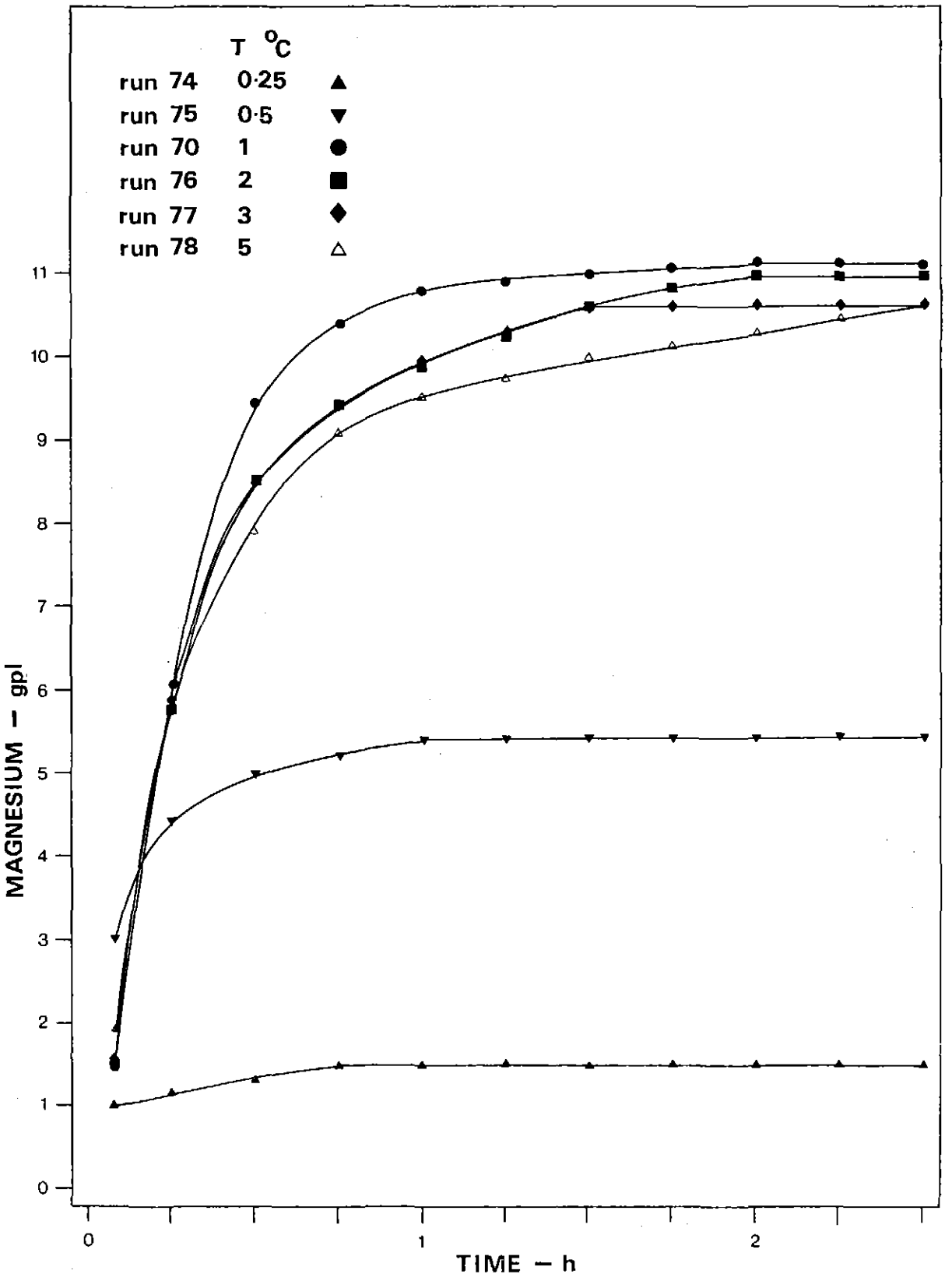

FIGURE 9. Autoclave leaching kinetic data; the effect of calcination time on magnesium concentration. MAG 3 calcined at 700°C, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 6).

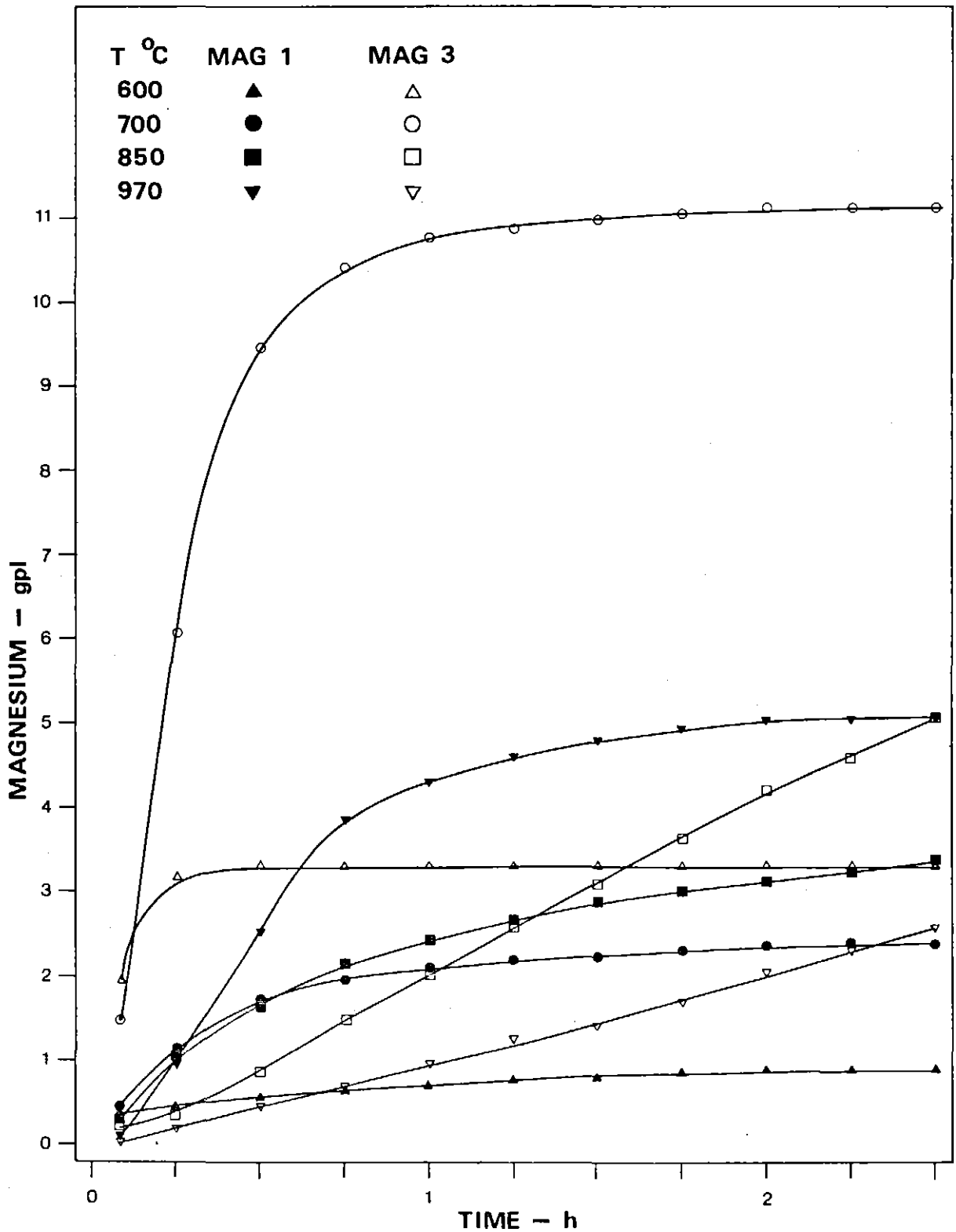

FIGURE 10. Autoclave leaching kinetic data; comparison of the effect of calcination temperature on magnesium concentration for MAG 1 (high dolomite) and MAG 3 (high magnesite). Calcined for 1 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Tables 1 and 5).

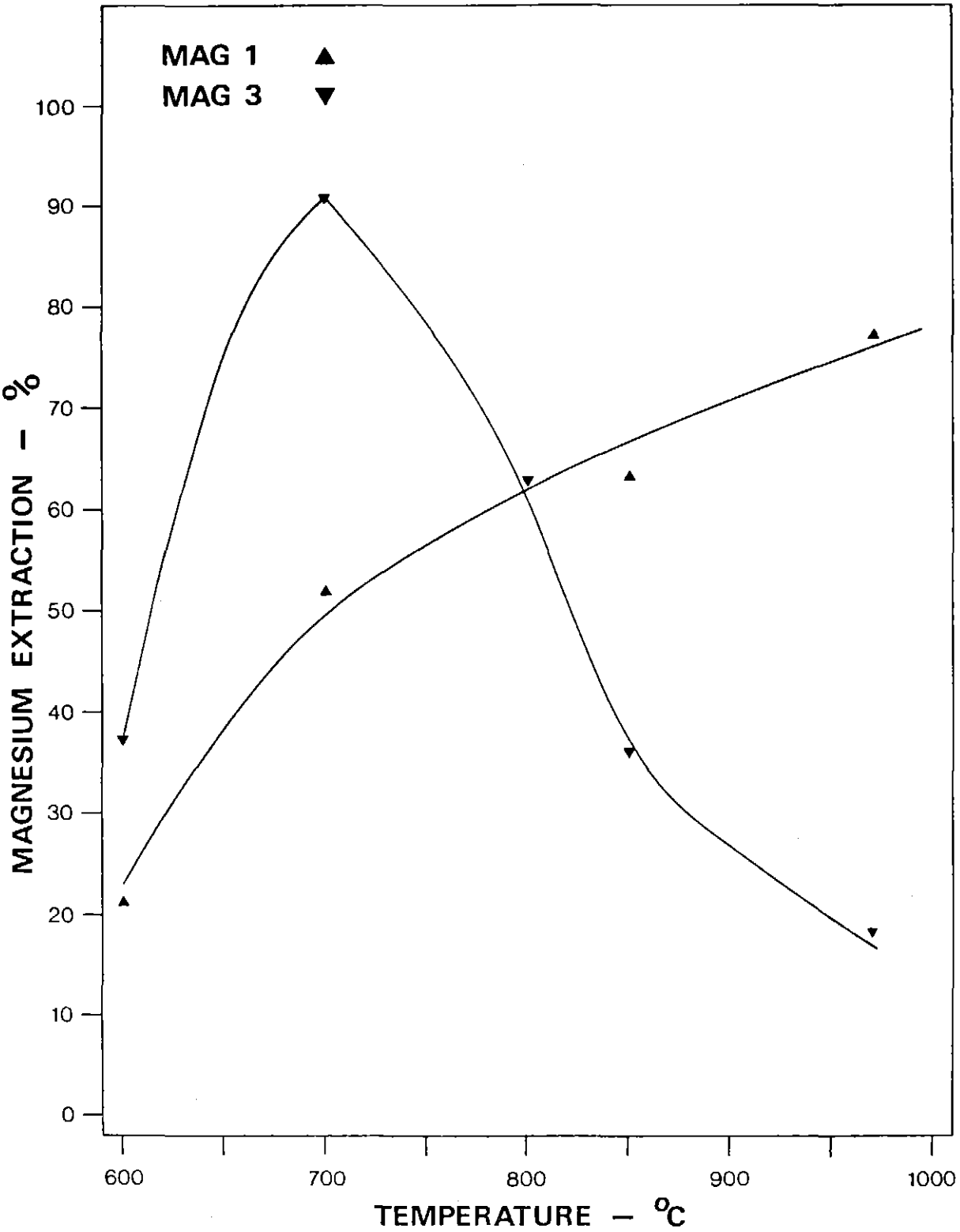

FIGURE 11. Autoclave leaching kinetic data; comparison of the effect of calcination temperature on magnesium extraction for MAG 1 (high dolomite) and MAG 3 (high magnesite). Calcined for 1 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Tables 1 and 5).

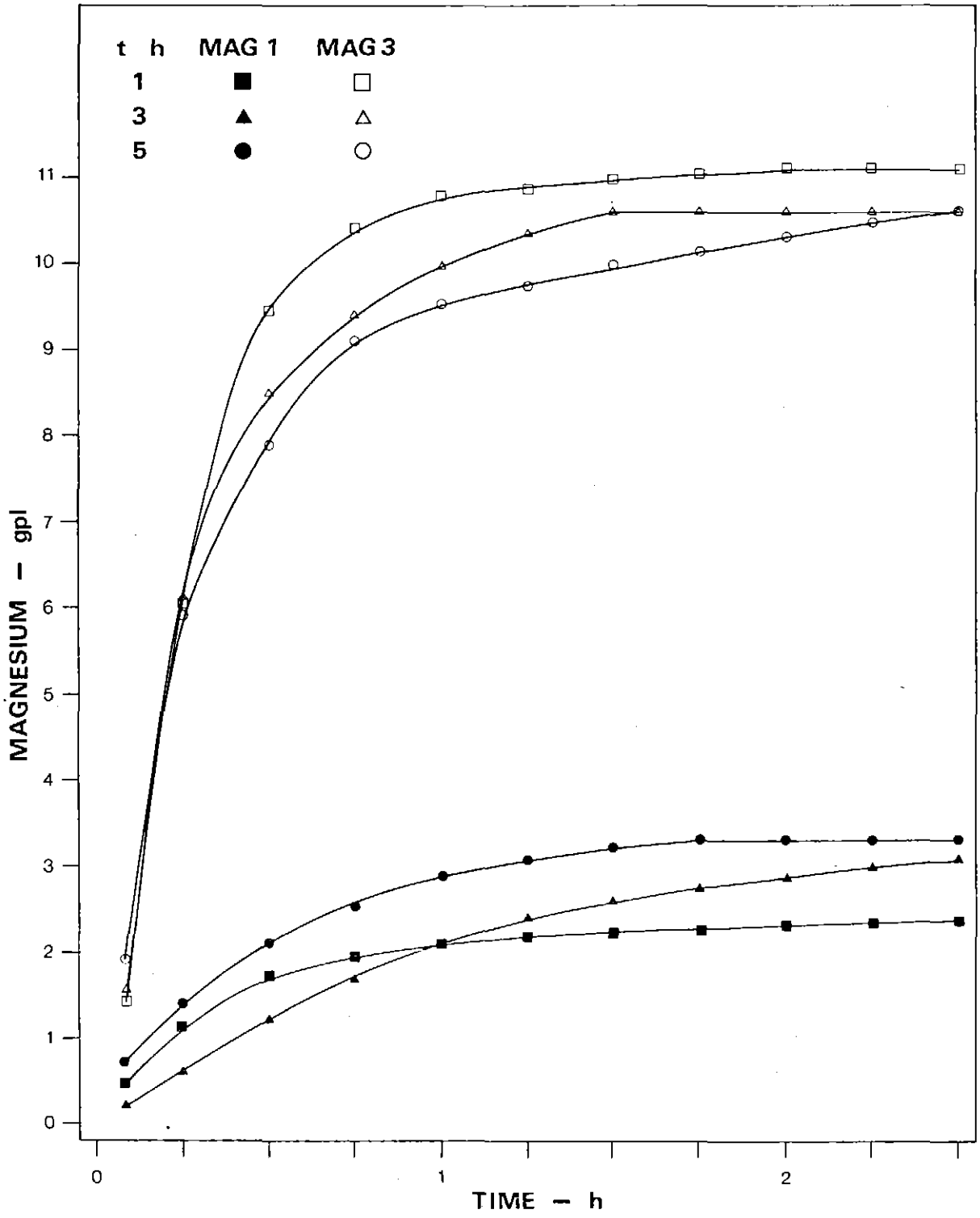

FIGURE 12. Autoclave leaching kinetic data; comparison of the effect of calcination time on magnesium concentration for MAG 1 (High dolomite) and MAG 3 (high magnesite). Calcined at 700°C, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Tables 3 and 6).

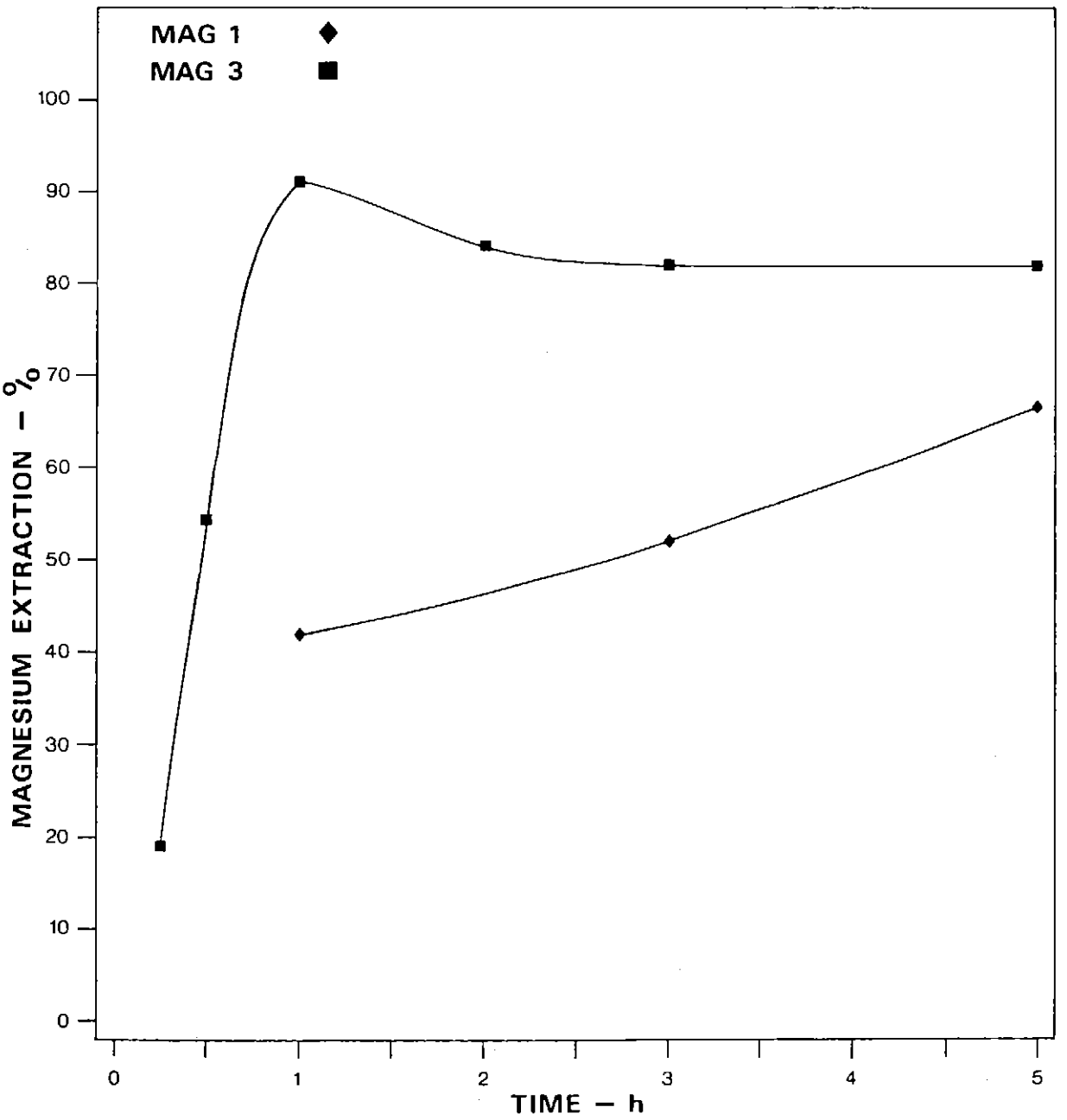

FIGURE 13. Autoclave leaching kinetic data; comparison of the effect of calcination time on magnesium extraction for MAG 1 (high dolomite) and MAG 3 (high magnesite). Calcined at 700°C, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Tables 3 and 6).

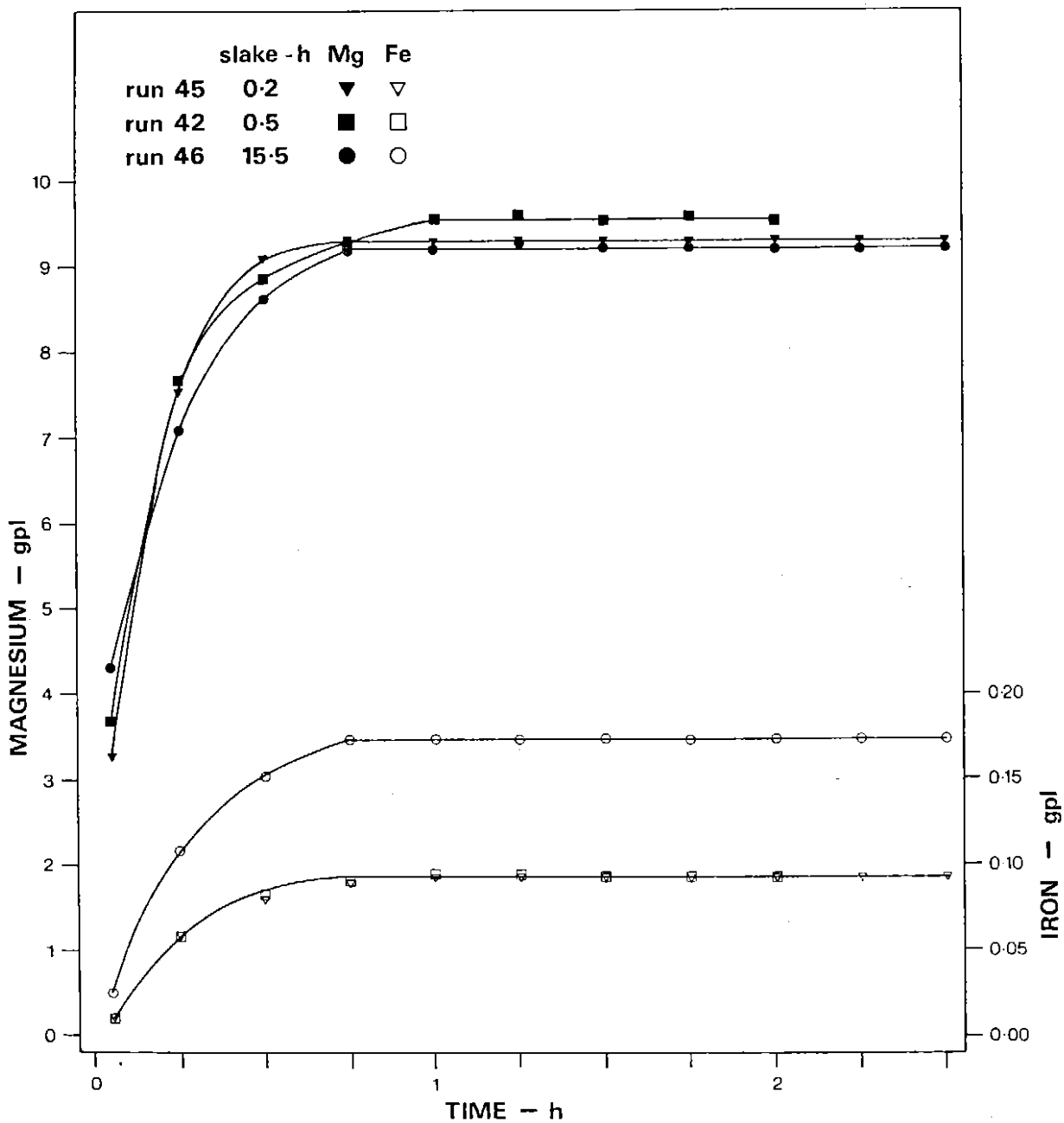

FIGURE 14. Autoclave leaching kinetic data; the effect of slake time on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Tables 7 and 8).

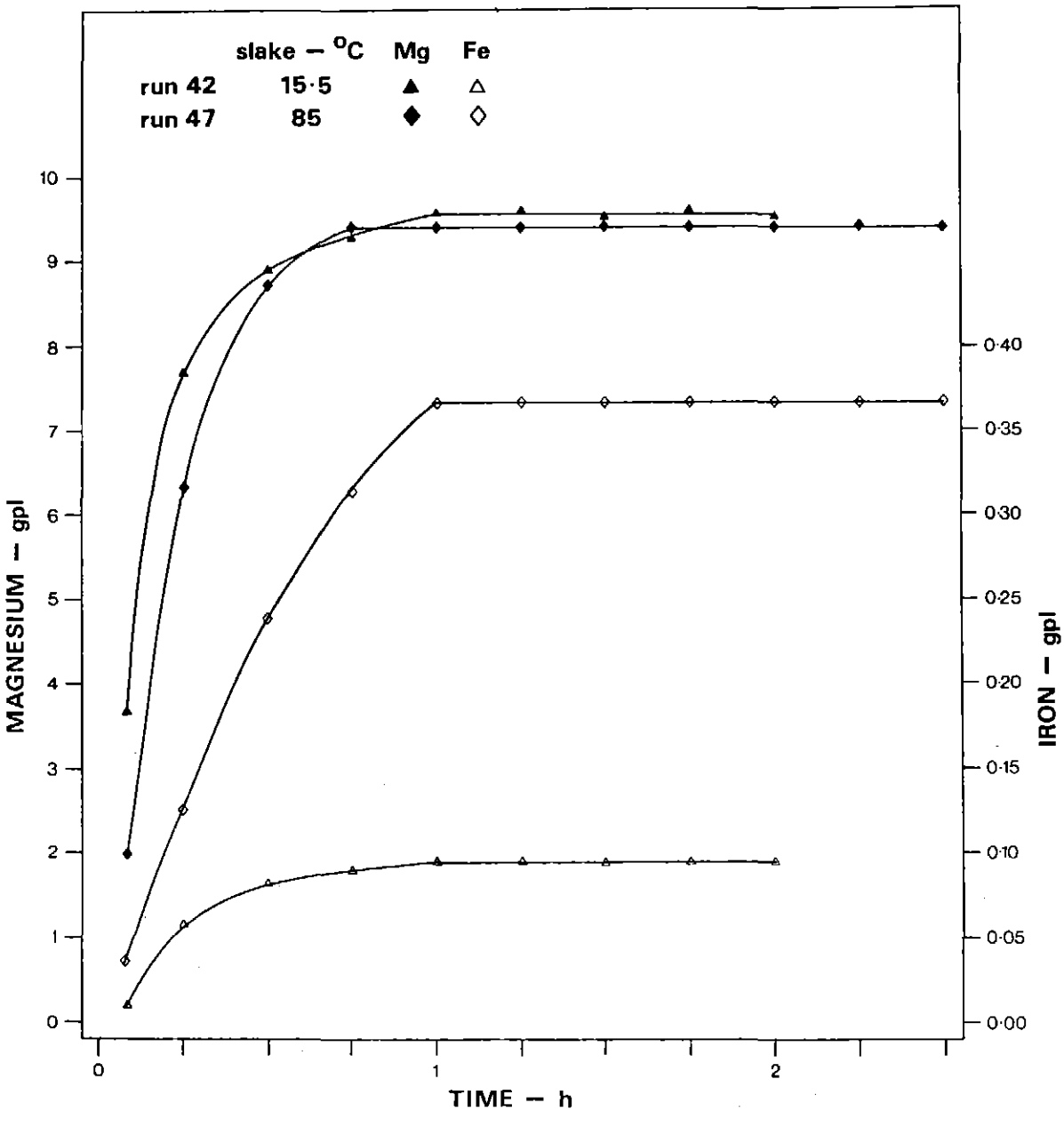

FIGURE 15. Autoclave leaching kinetic data; the effect of slake temperature on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 39 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm. (See Tables 7 and 8).

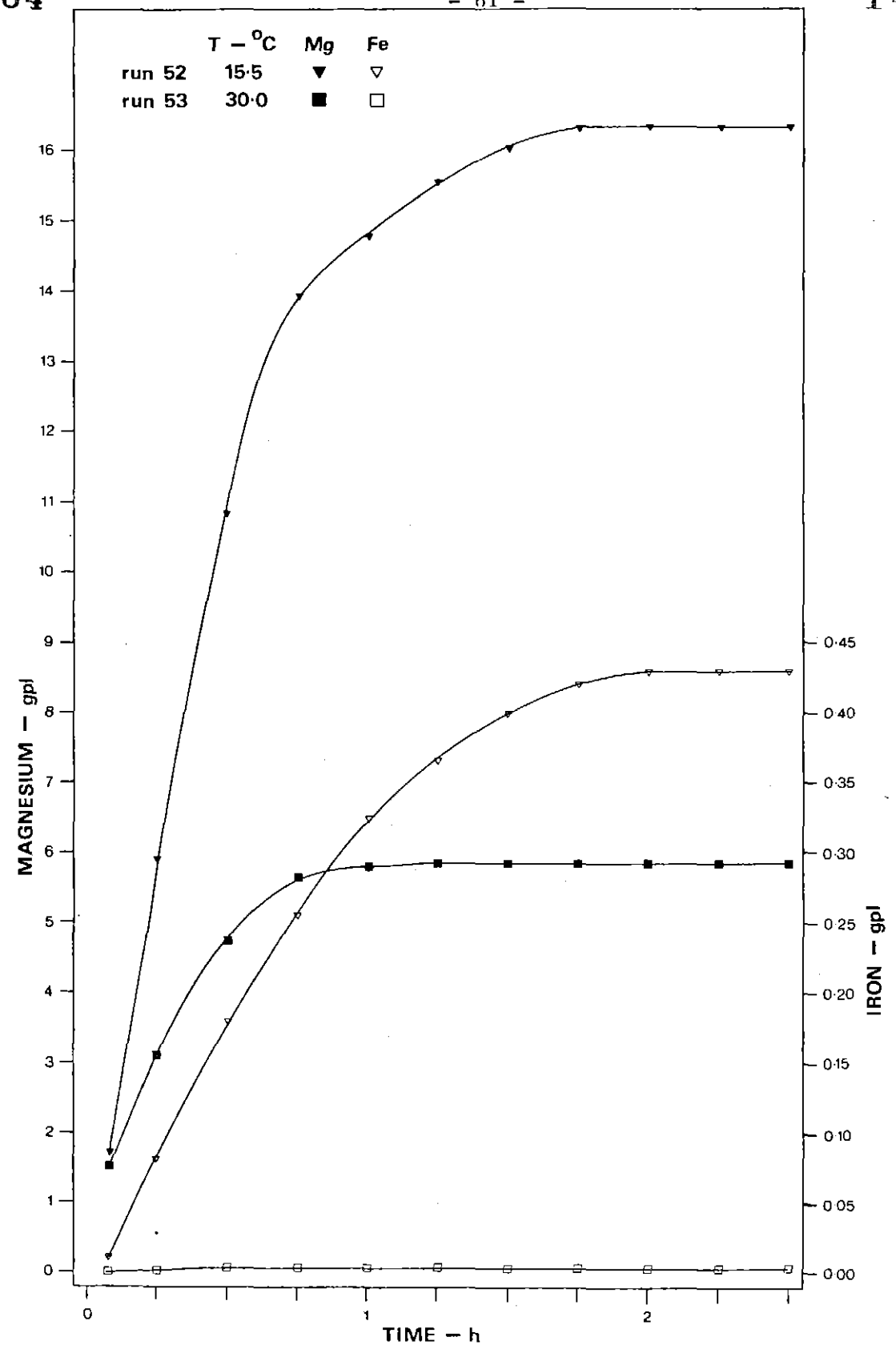

FIGURE 16. Autoclave leaching kinetic data; the effect of pulp density on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 0.5 h slake, 15.5°C, 25 psig CO_2, 1200 rpm.
(See Tables 7 and 8).

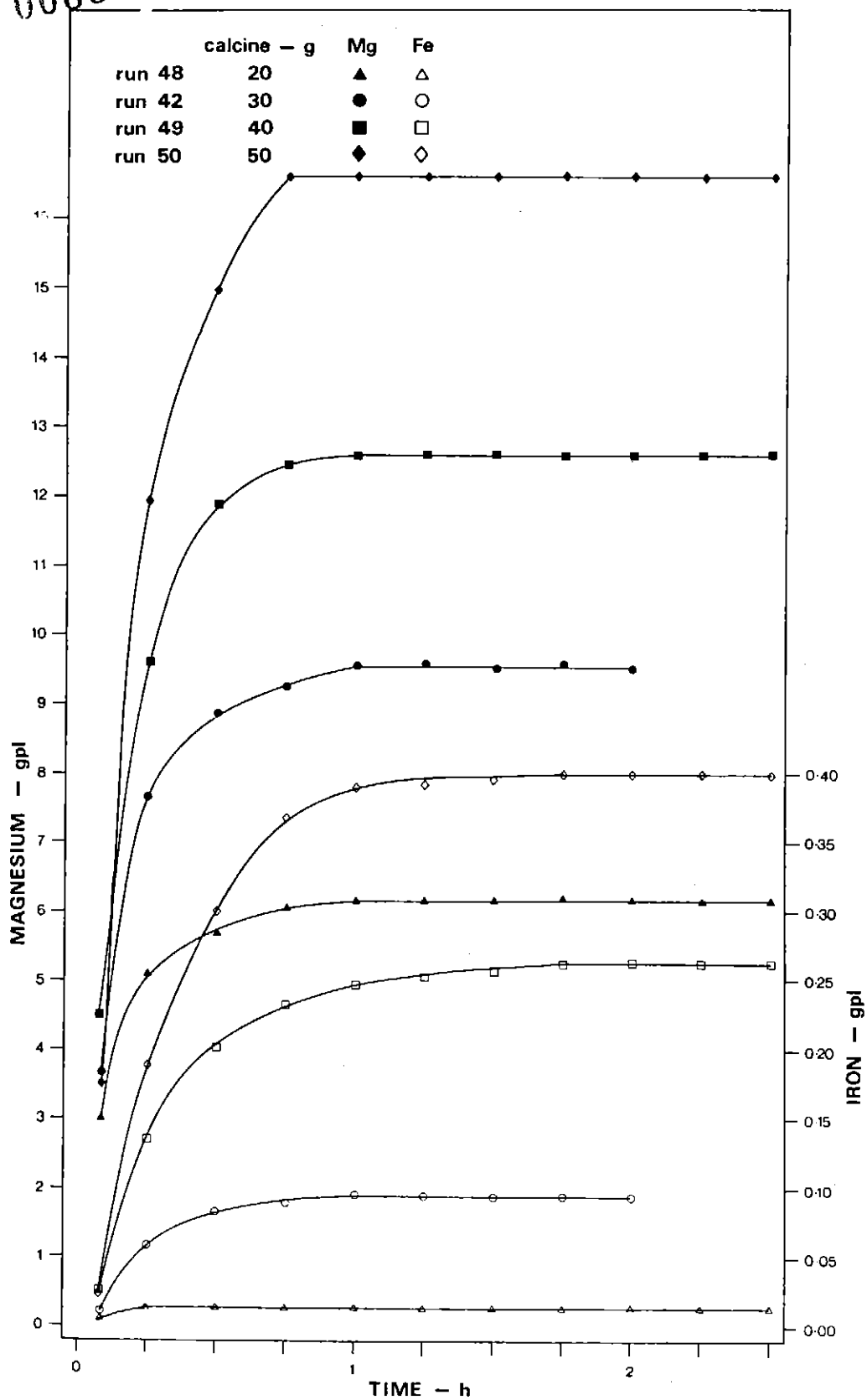

FIGURE 17. Autoclave leaching kinetic data; the effect of pulp density on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Tables 7 and 8).

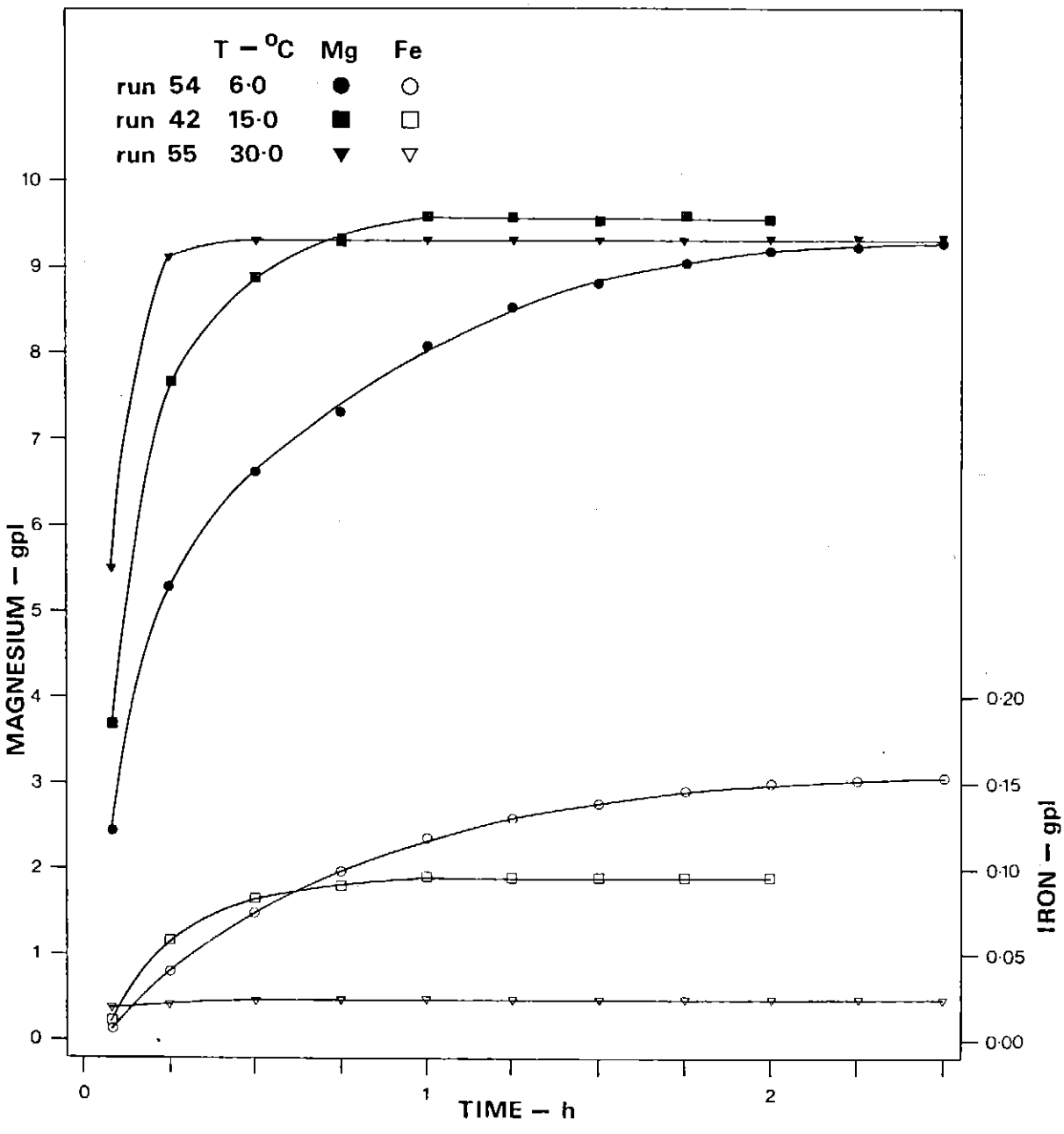

FIGURE 18. Autoclave leaching kinetic data; the effect of leaching temperature on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/ litre, 0.5 h slake, 100 psig CO_2, 1200 rpm. (See Tables 7 and 8).

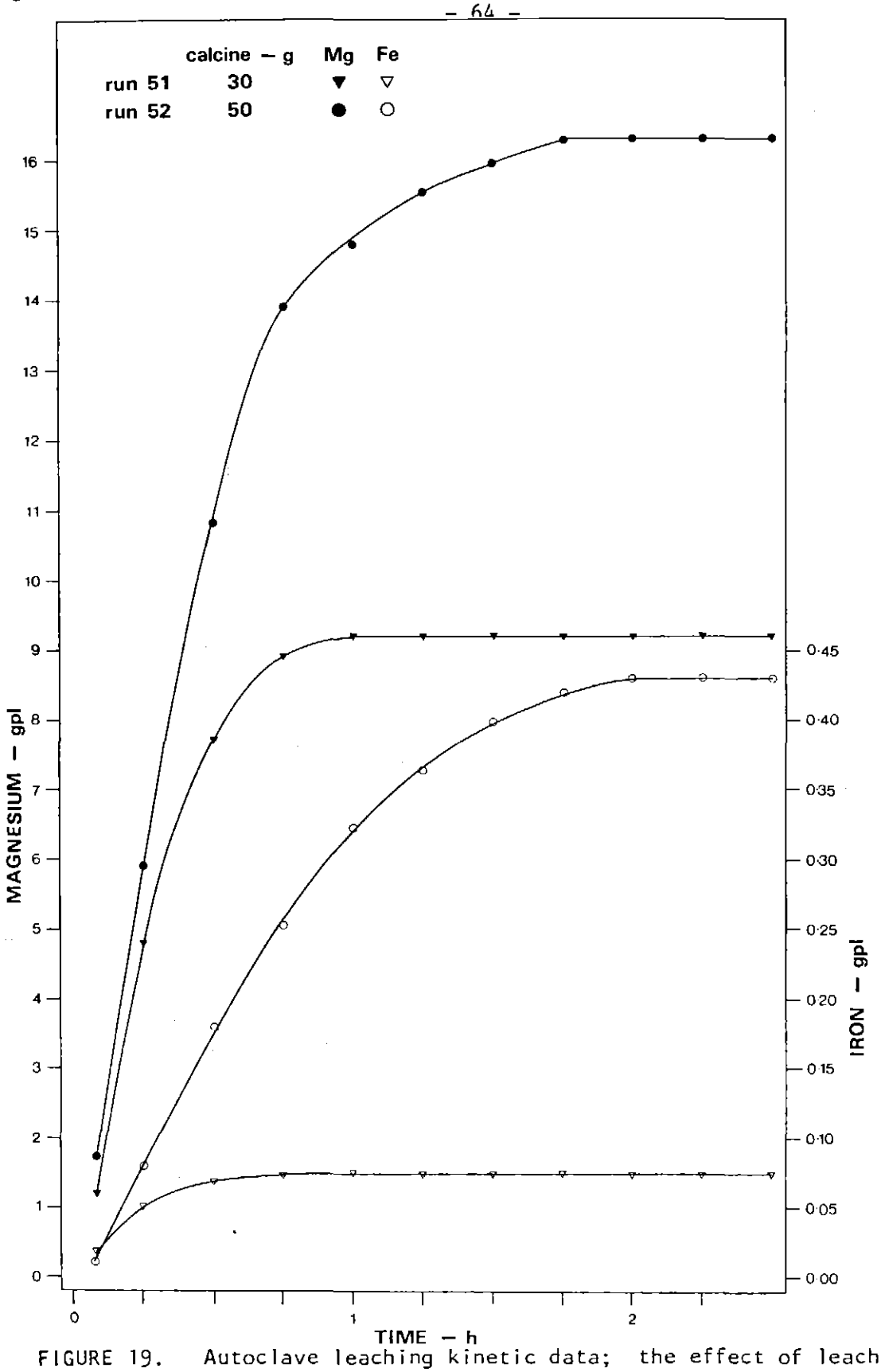

FIGURE 19. Autoclave leaching kinetic data; the effect of leaching temperature on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 25 psig CO_2, 1200 rpm.
(See Tables 7 and 8).

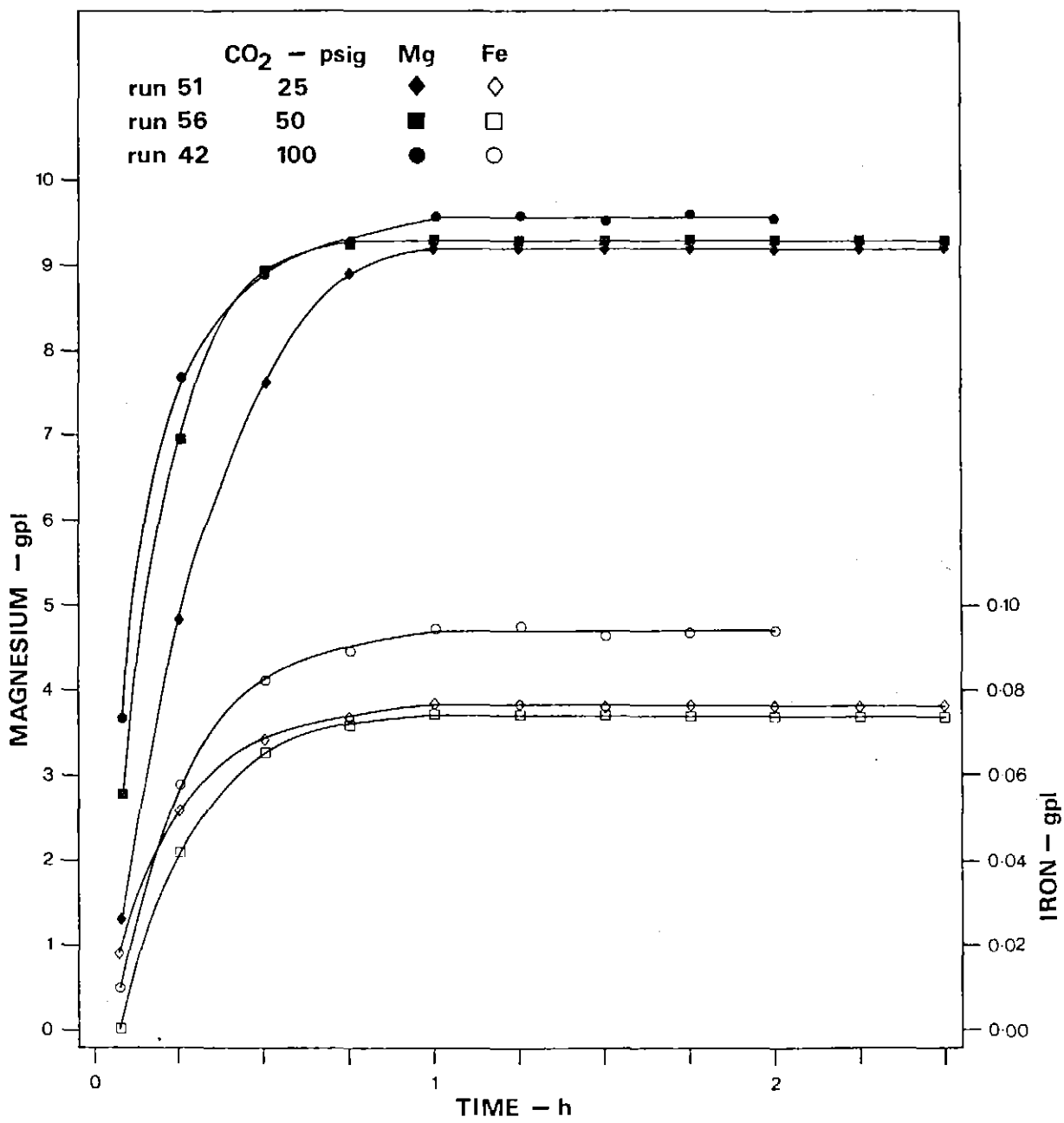

FIGURE 20. Autoclave leaching kinetic data; the effect of carbon dioxide partial pressure on magnesium and iron concentrations. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 1200 rpm.
(See Tables 7 and 8).

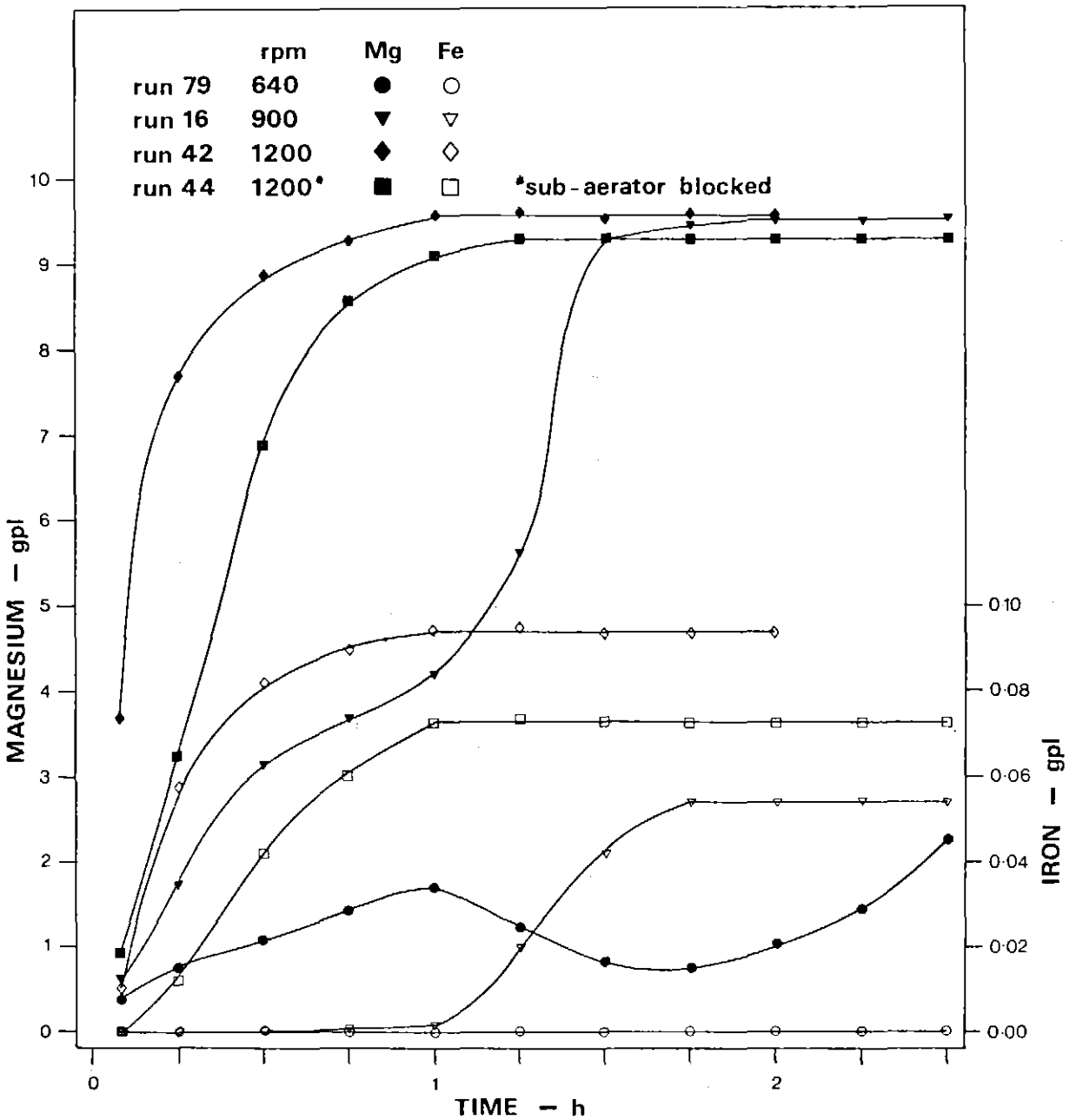

FIGURE 21. Autoclave leaching kinetic data; the effect of agitation rate on magnesium and iron concentration. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2.
(See Tables 7 and 8).

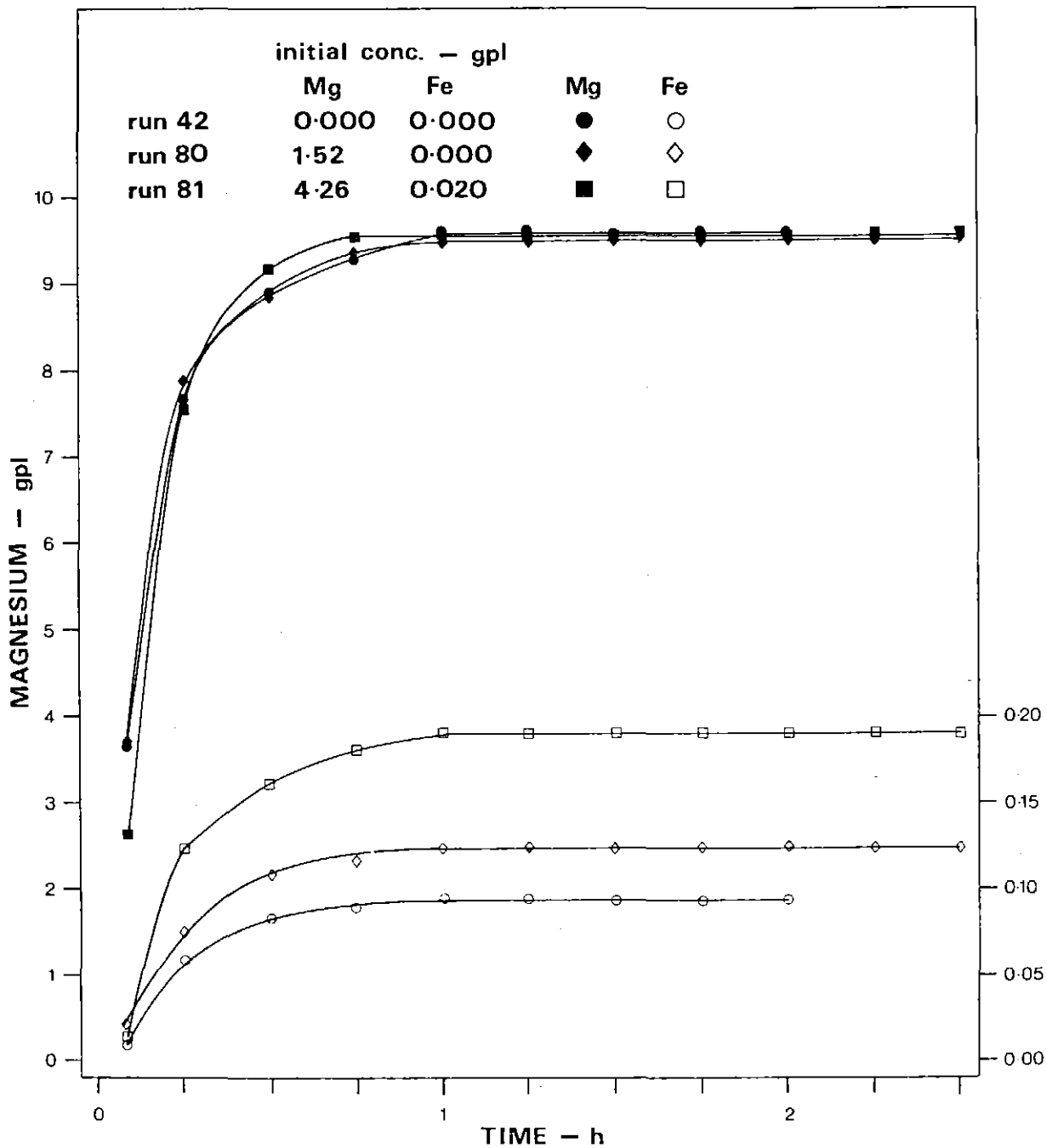

FIGURE 22. Autoclave leaching kinetic data; the effect of the initial leachant composition of magnesium and iron concentration corrected for the initial leachant concentration. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Table 9).

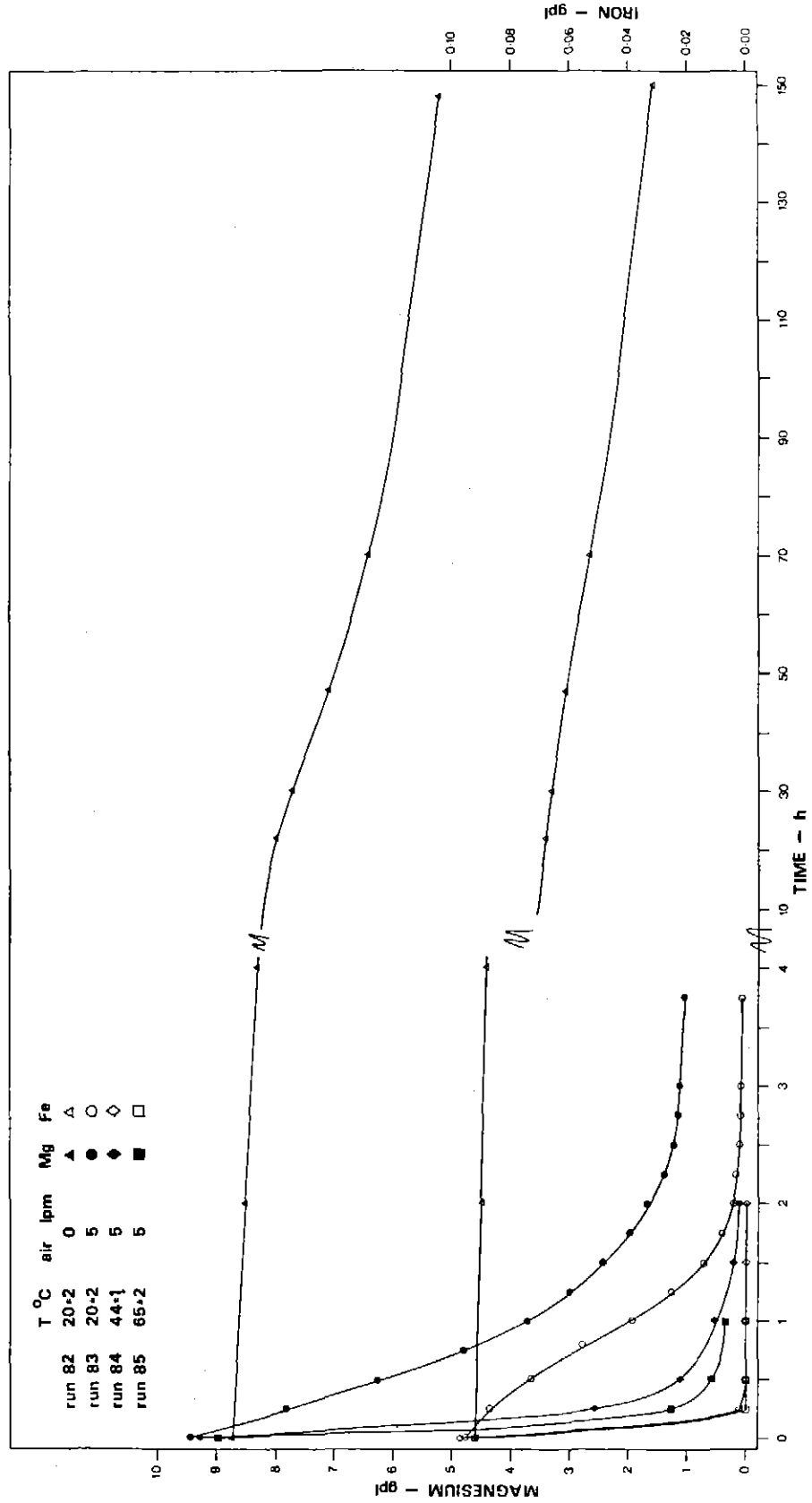

FIGURE 23. Precipitation rate data; the effect of temperature and air sparging on magnesium and iron concentration in solution.
(See Table 10).

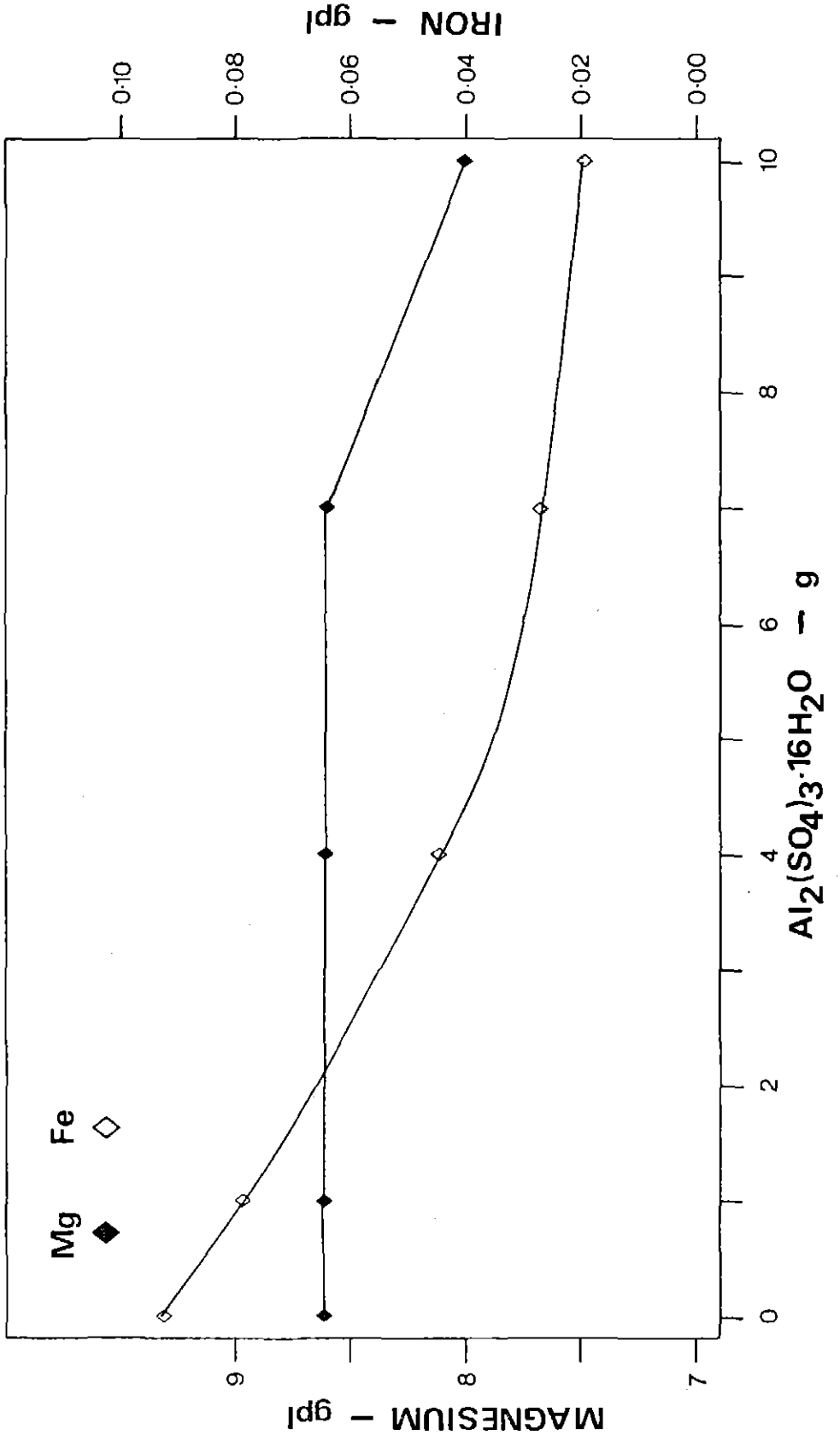

FIGURE 24. Magnesium and iron concentration in solution as a function of aluminium sulphate addition to clarified pregnant leach liquor.
(See Table 13).

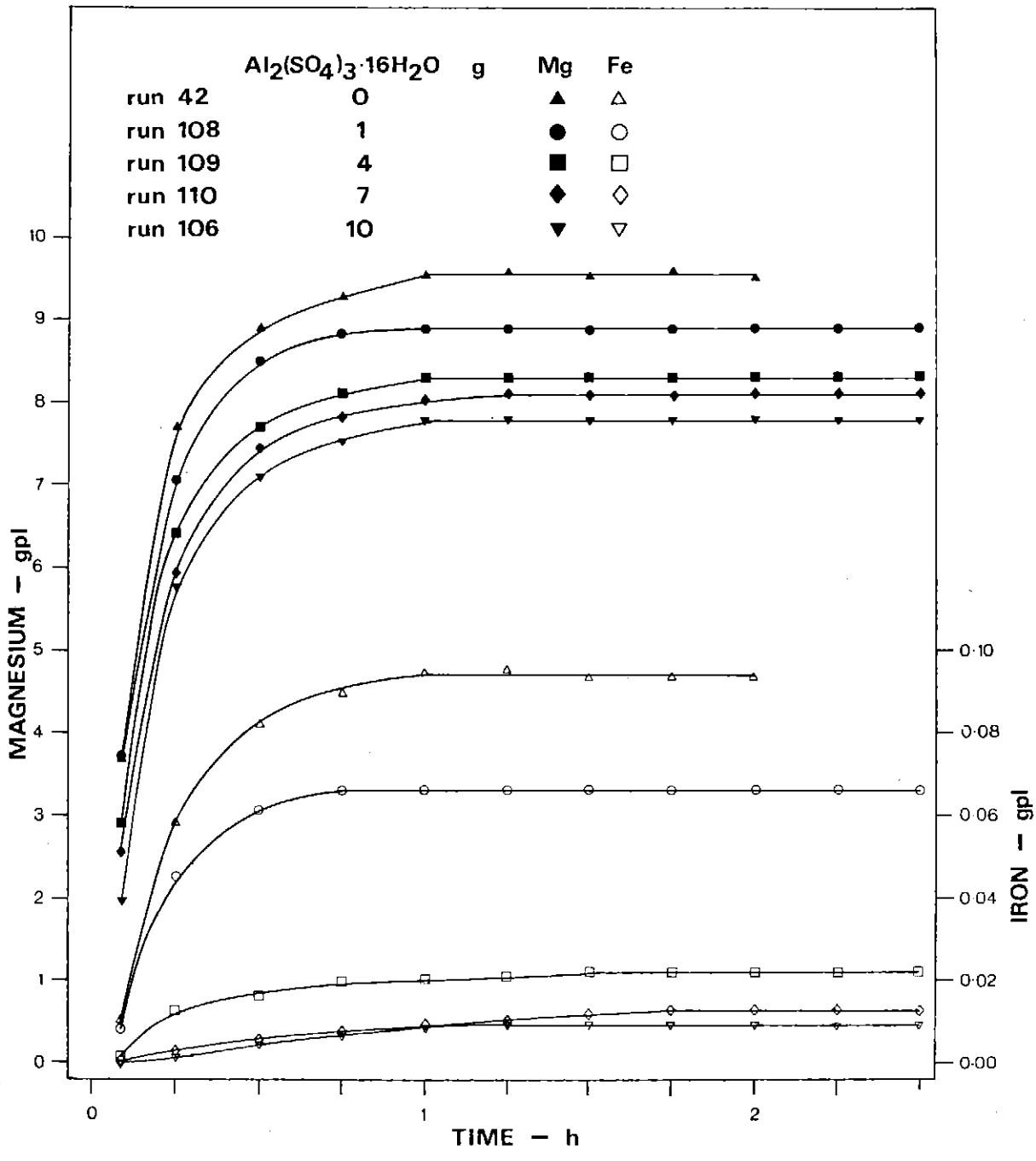

FIGURE 25. Autoclave leaching kinetic data; the effect of addition of aluminium sulphate on magnesium and iron concentration during leaching. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Table 15).

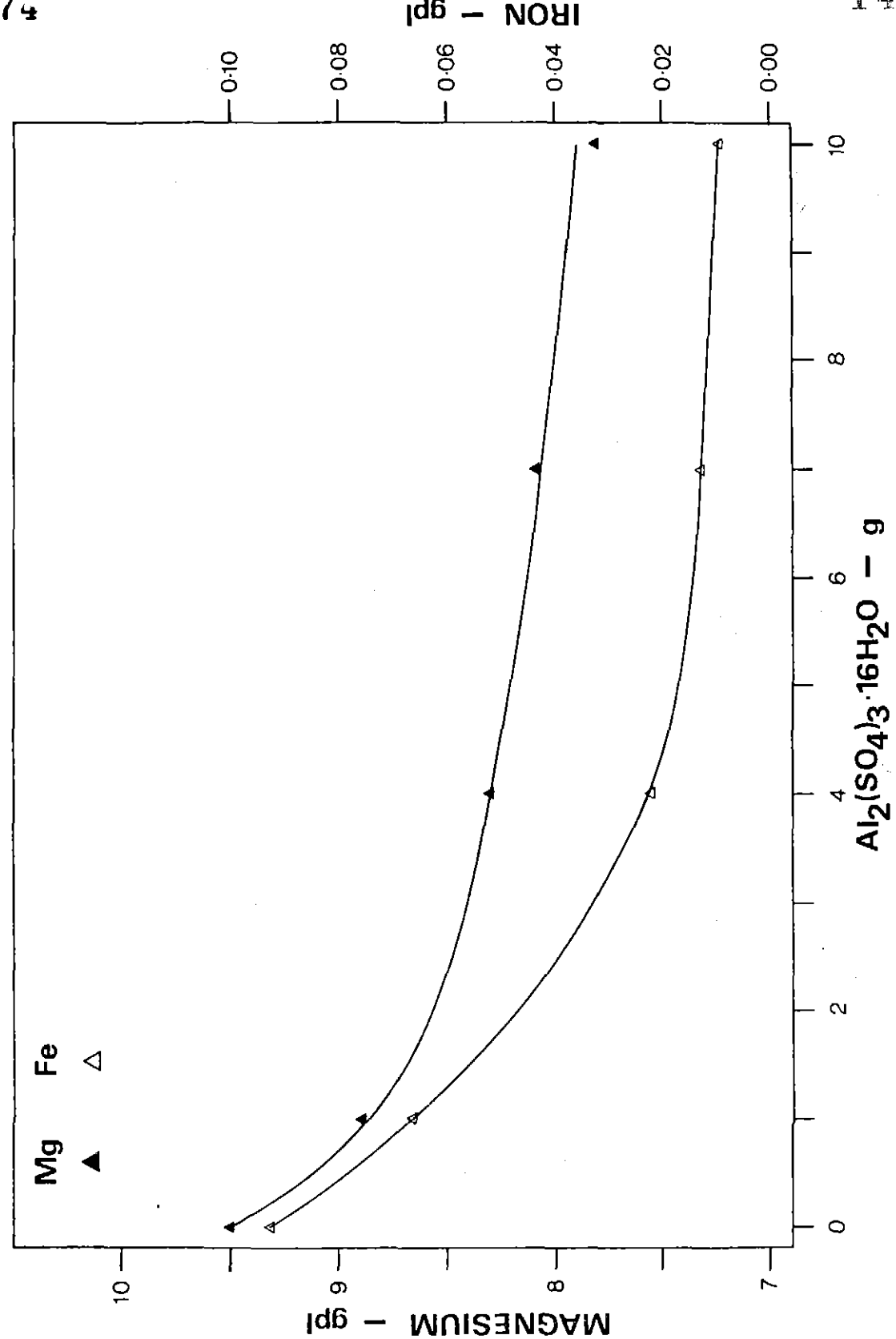

FIGURE 26. Magnesium and iron concentration in solution as a function of aluminium sulphate addition during leaching. MAG 3 calcined at 700°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 2.5 h leach, 15.5°C, 100 psig CO_2, 1200 rpm. (See Table 15).

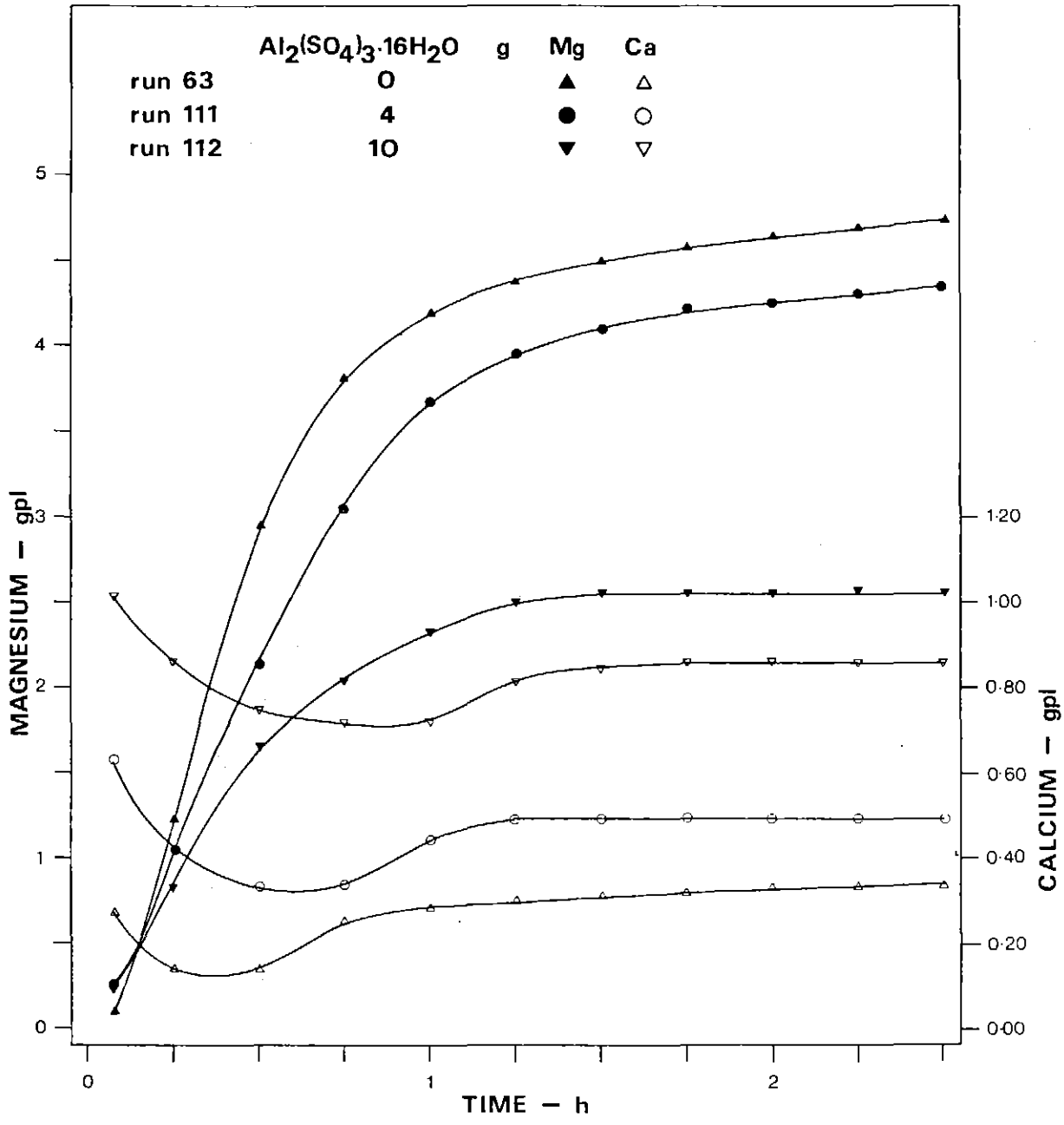

FIGURE 27. Autoclave leaching kinetic data; the effect of addition of aluminium sulphate on magnesium and calcium concentration during leaching. MAG 1 calcined at 850°C for 3 h, leached at 30 g solid/litre, 0.5 h slake, 15.5°C, 100 psig CO_2, 1200 rpm.
(See Table 16).