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PRODUCTION OF MAGNESIA FROM SAVAGE RIVER MAGNESITE

(Progress Report No. 8: March-December 1981)

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SUMMARY

This report details the results of a number of batch pilot-scale autoclave leach tests. Prior to the generation of this data it was necessary to carry out further modifications to the test rig. These modifications were made necessary because of the physical and chemical properties of the pregnant leach slurry.

In general terms, the effects of leach conditions (slake time, carbon dioxide pressure, temperature, pulp density, aluminium sulphate addition) on the kinetics and degree of magnesium extraction were essentially identical with those encountered in the previously reported laboratory-scale tests. However, the greater degree of turbulence in the pilot-scale autoclave resulted in more rapid iron dissolution rates and, more importantly, an increase in the absolute amount of iron dissolved. This can be attributed to the higher total "dissolved" carbon dioxide concentration in solution which stabilizes the soluble, relatively unstable iron "carbonato" complex. This increased iron dissolution is obviously undesirable.

The results will allow the proposed continuous (>24 h) leach tests to be planned and carried out more logically. The results of these latter tests will be used to design, construct and operate a more realistic pilot plant, capable of processing a minimum of 5 tpd crude magnesite from feed preparation through to final calcination and densification.

INTRODUCTION

During the previous period [1], work on the development of the calcination/carbon dioxide leach process centred around the following areas.

- Preparation and characterization of feed for the pilot-scale autoclave leach test programme. Because of difficulties experienced with the operation of the technical-scale rotary kiln, the sample of bulk calcine was not uniform in composition, and although blending was carried out, the final product showed variable reactivity. Thus grab samples of blended bulk calcine, when leached under standard conditions (0.5 h slake, 3% solids, 100 psig carbon dioxide, 15.5°C, 1200 rpm), gave magnesium extractions in the range of 72.2 to 86.3%. Although these results were less satisfactory than expected, they do indicate the importance of calcination under strictly controlled conditions.
- Completion of the modifications to the pilot-scale autoclave leach test rig.
- Calibration of the indicating temperature controllers, while determination of heating rates and slurry pump flowrates were commenced.
- Development and implementation of operator training programmes.
- Determination of the effect of calcine temperature on iron dissolution. The data obtained clearly illustrates the need to cool the calcine to ambient temperature prior to slurrying in order to minimize iron dissolution. Thus every effort should be made to recover all of the sensible heat from the calcine as it exits from the rotary kiln.
- Investigation of the effect of addition of aluminium sulphate to precipitate iron on the precipitation of hydrated magnesium carbonate or basic magnesium carbonate from clarified pregnant liquors. Tests showed that, although the final precipitate contained a significant amount of sulphate, the amount of magnesium precipitated was not substantially affected by the presence of sulphate in the clarified pregnant liquor. It was concluded that final product purity would not be affected since the final calcination temperature would be significantly greater than the sulphate decomposition temperature. The only problem would be that the carbon dioxide off-gases from the calcination stage would contain sulphur dioxide/sulphur trioxide. These impurities would have to be removed before the carbon dioxide could be recovered and recycled to the leaching circuit.
- Characterization and processing of the Mg-Al-Fe hydroxycarbonate precipitates. Apart from the cost of the aluminium sulphate, a major disadvantage of the aluminium sulphate method of iron removal is that a significant amount of magnesium is precipitated so that the overall

recovery is reduced. Two methods of both recovering at least part of this magnesium and reducing the amount of aluminium sulphate that has to be used to attain the desired [Fe × 100/Mg] concentration ratio were investigated. Both methods, involving controlled calcination and dissolution in sulphuric acid, were at least partly successful.

In the present period, it was anticipated that following final commissioning, much of the batch pilot-scale leaching tests using the autoclave rig would have been completed. It was also anticipated that any necessary modifications to the rig and/or to the operating procedures for continuous operation would have been established. As indicated below, a number of totally unexpected problems became apparent after 3 or 4 batch leach tests had been completed. It was thus necessary to spend a considerable amount of time designing and testing new components before reliable batch and continuous tests could be carried out on a regular basis.

All of the batch leach tests have now been carried out; details of these tests are reported here. The first of several continuous tests (>24 h duration) has already been successfully carried out although several other modifications were considered essential for efficient completion of the research programme. It was decided that the results of the batch tests should be reported now, albeit some months after completion, rather than wait until all the continuous tests were finalized.

EXPERIMENTAL

SLURRY PUMPING RATE

The slurry-metering pump of the rig is of the positive pressure displacement type which means that the autoclaves have to be pressurized before the calcine slurry can be added.

In the first series of pumping tests, nitrogen was used as the pressurizing atmosphere. Tests were carried out with a nitrogen overpressure in the range 20-200 psig. In each test the required pressure was set, the pump setting adjusted as necessary, and pumping commenced. Pumping was continued for periods in the range 2-60 min, depending upon the flowrate, as judged by the change of volume of slurry in the feed tank.

As the volume of slurry pumped into the autoclave increased, the pressure increased; the excess pressure was reduced continuously by venting. At the end of the pump test the slurry was discharged via the bottom drain port (see Fig. 9, Ref. 1) and the volume recorded. Slurries ranging from 1 to 10% solids prepared from crude bulk calcine were used throughout the tests.

It soon became apparent that the measured pumping rates varied over an unacceptably large range for each pump setting, particularly at low operating pressures and high pulp densities. Dismantling of the pump valves showed excessive build-up of solids and some scoring of the Teflon valve seats. It was concluded that the quartz in the solid feed (bulk calcine), which represented the hardest and coarsest of the particles, caused the majority of the problems with the slurry pump. It was decided to dry screen the feed, removing the +0.4 mm fraction. X-ray diffraction analysis showed that this was principally quartz plus a lesser amount of dolomite with virtually no free magnesium oxide. The proposed commercial flowsheet for treating Savage River magnesite by the calcination/carbon dioxide leach process incorporates such a selective gangue removal stage [2-4]. As expected, the X-ray diffraction analysis also showed that the calcine contained an appreciable (10-15%) amount of magnesite, consistent with the need to process an under-calcined feed. As the data in Table 1 show, dry screening and re-blending of the bulk calcine not only removed undesirable gangue minerals but also produced a more uniform feed.

Using the screened feed, reproducible slurry pumping rates were readily achieved. A significant observation was that the pumping rate was not affected by the operating pressure. It was thus clear that the pump seals were operating in a satisfactory manner. Figure 1 gives typical pumping data (average of 5 sets of data for various pressures) using nitrogen as the pressurizing atmosphere and pumping into No. 1 (the top) autoclave only. When the slurry fed into No. 1 autoclave was allowed to overflow into the other autoclaves by opening the valves between the autoclaves, it was not possible to operate at the maximum pumping rate. Apparently the ball valves between each set of autoclaves acted as constrictions to flow so that the No. 1 autoclave tended to overfill. The level indicator was activated and the alarm bells/lights came into operation. The pumping rate at which overflowing took place was affected by the operating pressure; the higher the operating pressure, the higher the workable pumping rate.

For continuous leach tests, the operating pressure is carbon dioxide so that reaction between the slurry and the carbon dioxide commences immediately the slurry is introduced into the autoclave. Under these conditions it is necessary to add additional carbon dioxide on a continuous basis to maintain the desired operating pressure. This compares with the need to have a continuous bleed-off when using nitrogen as the pressurizing atmosphere. Provided the carbon dioxide operating pressure was maintained above 20 psig, the nature of the pressurizing atmosphere had no effect on the pumping rate.

MODIFICATIONS TO THE AUTOCLAVE LEACH RIG

Several preliminary batch leach tests using No. 1 autoclave were carried out in order to ascertain if the proposed operating procedures were realistic or not. The first of these tests proceeded reasonably satisfactorily, samples of slurry being recovered for analysis every 15 min. As with the laboratory scale leach tests [2], the sampling port was freed of slurry by blowing back with high pressure carbon dioxide, and a bleed volume (approximately twice the internal volume of the sampling line) recovered before the analytical sample was recovered. The sampling port was immediately re-purged with carbon dioxide. Towards the end of the leach period (2.5 h) the sampling port ball valve became difficult to operate because of the gradual build-up of solids in the stem of the valve. Although the valve was not completely blocked, a long-term operating problem was recognized at this point.

More serious, however, was the fact that a considerable amount of solids had settled into the stem of the drain valve, completely blocking it. Thus when the leach was completed it was not possible to remove the pregnant leach liquor plus unreacted solids from the autoclave. These could only be removed by releasing the operating pressure and removing the level indicator to allow entry of a 5 mm suction tube. It was necessary to turn off the impeller during this procedure and it was apparent that a significant amount of solids had settled to the bottom of the autoclave.

The above observations suggested that the impeller was not correctly positioned so as to prevent solids settling below the impeller and falling into the stem of the drain valve. That is, there was a null point beneath the impeller. To check this, the autoclave was dismantled, the nature of the problem examined visually and the position of the impeller accurately determined. Two features immediately became apparent. The first was that even if the drain valve had been operable, a small volume (~100 ml) of pregnant leach slurry would remain in the autoclave because the drain valve assembly unit was sitting slightly proud of the dished bottom of the autoclave. The other important observation was that the leach residue in the stem of the drain valve and the slurry remaining in the autoclave had set like concrete and could only be removed by chemical dissolution. Prior to its removal, a small amount of the solid material was recovered for X-ray diffraction analysis. This revealed that it consisted principally of quartz, magnesite and very fine hematite, cemented together by hydrated magnesium carbonate ($MgCO_3.3H_2O$, nesquehonite). The latter had apparently precipitated from the unstable pregnant magnesium bicarbonate solution once the carbon dioxide atmosphere had been removed.

These two observations indicated the necessity to redesign the sampling/draining valve system to allow the impeller to be positioned more closely to the dished bottom of the autoclave to actually sweep the area free of solids. They also indicated the need to drain and clean out the autoclaves while maintaining a carbon dioxide pressure within the autoclaves to prevent precipitation of hydrated magnesium carbonate. This step was regarded as quite critical as previous work had shown that it is not possible to re-dissolve precipitated hydrated magnesium carbonate by re-carbonation.

Examination of the sampling/draining valve system showed that the actual open volume through which the slurry could be discharged was severely restricted by the internal sampling line. In addition, the 90° angle of the drain valve prevented free flow of slurry. The sampling line also meant that the turbine impeller could not be lowered sufficiently close to the bottom of the autoclave (see Fig. 9, Ref. 1). It was decided to separate the sampling/draining system by introducing the sampling line into the top of the autoclave via the blow-down line (Fig. 2 - compare with Figs. 6 and 8, Ref. 1). The sampling tube inside the autoclave was fixed at the same level as in the original configuration. The 90° 6 mm drain valve was replaced by a 180° 13 mm quick-action ball valve. The new valve coupling arrangement was designed to minimize the dead-space volume while maximizing the cross-sectional area of this dead-space. This would help to minimize the possibility of a complete blockage of the valve system, particularly when the turbine impeller was lowered so that the tips of the impeller were approximately 5 mm from the bottom of the autoclave.

The remaining preliminary leach tests using the above modifications were all successful in terms of operating procedures and the "complete" removal of pregnant leach slurry at the conclusion of each test. Removal of the autoclave head after the final test leach revealed only minimal build-up of solids in the bottom of the autoclave.

SAMPLE PREPARATION

Following the dry screening and re-blending of the bulk calcine described above, approximately 1.5 kg batches of feed were re-rolled and then riffled to provide samples for chemical analysis (Table 1) and the appropriate weights of samples for both laboratory and pilot-scale leach tests at the desired pulp density. Thus each pilot-scale test was repeated in the laboratory-scale autoclave using "identical" samples.

OPERATING PROCEDURE

The procedure described below was designed to ensure continuity of results, to reduce the possibility of the development of other, unforeseen problems, and to allow comparison with the laboratory-scale tests carried out on the same samples of feed. The procedures particularly apply to the cases where nitrogen was used as the pressurizing atmosphere. The only significant difference between the operating procedures for the laboratory- and pilot-scale leaches was that the feed was added directly to the autoclave in the former case but was pumped in under nitrogen in the latter case. When using carbon dioxide as the pressurizing atmosphere, a slightly different procedure was adopted (see below) since reaction commenced as soon as the slurry was fed into the autoclave. It was not possible to reproduce these conditions with the laboratory-scale tests because of the method of slurry addition.

The batch leaches using nitrogen as the pressurizing atmosphere were carried out as follows.

- The cooling system (stirrer gland and internal cooling coil) was activated.
- The turbine impeller was turned on.
- The indicating temperature controller was switched on and set at the required temperature.
- The voltage regulators for the heating coils were set at the required values in accordance with the operating temperature. The heater switches were in the off position.
- The autoclave, sampling port and drain line were purged with high-pressure nitrogen to ensure that they were open and free from liquor.
- After shutting the sampling port and drain line valves, the autoclave was pressurized with nitrogen to the required value.
- 35 litres of tap water was added to the slurry mixing vessel and agitation of the latter commenced.
- The slurry pump was set at the required rate. For all of the batch tests in which the operating pressure was maintained with nitrogen, the pumping rate was normally fixed at approximately 0.75 l/m (equivalent to a pump setting of 80) so that the introduction of the required volume of slurry (approximately 20 litres) took less than the minimum slake time (30 min) desired.
- One minute before the slurry pump was switched on, the required weight of feed (PPFS) was added to the slurry mixing vessel. The commencement of the slake period was taken as the time when half of the feed had been added to the slurry mixing vessel.
- The slurry pump was switched on. While the slurry was being pumped into the autoclave the temperature of the slurry within the autoclave was monitored every 5 min. The operating pressure was continuously adjusted as necessary by venting to maintain the desired pressure.
- After 10 min of slurry pumping, the heaters were turned on.
- After the addition of approximately 20 litres of slurry, the pump was turned off, the volume of slurry remaining in the mixing vessel noted, and then the latter discarded. The mixing vessel was thoroughly washed out and filled with fresh water for cleaning out the pump and autoclave once leaching had been completed.

- After 29 min*, or when the leach slurry had reached temperature, the nitrogen overpressure was vented as rapidly as possible, the system purged with carbon dioxide and then pressurized with carbon dioxide to the required value. This was taken as the commencement of the leaching period.
- While leaching took place, the operating pressure was continuously monitored. During the initial stages of reaction the necessary carbon dioxide addition rate was quite high.
- At the appropriate time intervals, the slurry temperature was recorded, and bleed and analytical samples removed via the sampling port. The sampling line was purged before and after sampling with high pressure carbon dioxide. The volumes of the two samples were recorded, the bleed sample discarded, and the analytical sample filtered and processed as described previously [2].
- At the completion of the leach period, the heaters and voltage regulators were turned off and the indicating temperature controller reset at 10°C to cool the pregnant leach pulp down. When this was achieved, the pregnant leach slurry was discharged into a suitable vessel. During this procedure it was necessary to keep the carbon dioxide line open to force the slurry out and to ensure that a carbon dioxide atmosphere was maintained in the autoclave. It was also necessary to switch the turbine impeller off at this stage because of the extreme turbulence over the drain valve. The cooling of the pregnant leach slurry and the carbon dioxide atmosphere ensured that precipitation of hydrated magnesium carbonate during the discharge period was minimized. The volume of pregnant leach slurry was recorded, together with the volumes of the unused feed slurry recovered from the slurry mixing vessel and of the bleed and analytical samples to ensure that a materials balance was obtained. In all cases this was achieved within ±5%.
- Once the pregnant leach slurry had been completely discharged, the drain valve was shut, the turbine impeller turned on, and the autoclave immediately repressurized with carbon dioxide. The slurry pump was turned on and 20 l of clean water pumped into the autoclave. After 5 min agitation, the wash liquor was discharged. The autoclave was washed out with a further 2 × 20 l clean water.

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*For one test, PPL 10, the slake period was 120 min.

- All valves were opened and all lines, plus the autoclave, purged with high-pressure nitrogen.
- The cooling system, temperature controllers and all gas lines were shut down.

As noted previously, reaction commenced immediately the feed slurry was introduced into the autoclave when using carbon dioxide as the pressurizing atmosphere, so that a slightly different operating procedure and treatment of data had to be adopted. The major differences related to the slaking period, the time taken as the commencement of leaching period, the time at which the first analytical sample was taken, and the need to ensure that the carbon dioxide pressure in the autoclave was maintained at the operating pressure during and after completion of slurry feeding to the autoclave.

For convenience, the slaking period was taken as half of the time taken to pump the required volume of slurry into the autoclave plus any additional time the slurry was allowed to slake in the slurry mixing vessel before the slurry pump was turned on. Commencement of the leaching period was taken as the time at which the slurry pump was turned on, while the first analytical sample was recovered simultaneously with the cessation of pumping.

Towards the end of the batch leach test programme, it became apparent that a major difference between the laboratory- and pilot-scale tests was that the reaction kinetics were far superior in the latter case. It was decided to check the possibility of using a flow-through system rather than reaction at an elevated pressure. This test was carried out by pumping the feed slurry into the autoclave under nitrogen, venting the nitrogen (leaving the valve open) and passing carbon dioxide through the autoclave at 7 l/min. This flow was in excess of that required for reaction so that a small flow of unused carbon dioxide was continuously vented. It was also sufficient to allow conventional sampling procedures to be used.

RESULTS AND DISCUSSION

The results of the pilot-scale batch leach tests, together with the results of the comparative laboratory-scale tests carried out on the same sample of feed, are given in Tables 2-19 and Figs. 3-39. The reaction conditions tested were chosen to ascertain the effects of scale-up and were not designed for optimization purposes. The overall development programme for the calcination/carbon dioxide leach process, as applied to Savage River magnesite, involves optimization only at the continuous leach stage. In general terms, the results of the pilot-scale tests are what were expected when compared with the laboratory-scale tests carried out previously [1-3,5-8]. There were, of course, a number of small but significant differences, particularly when considering commercial-scale development and operation.

The experimental data can be summarized as follows.

- Reaction kinetics for both magnesium and iron were enhanced in the pilot-scale tests - see Figs. 32 and 33. This is most likely a result of the much greater turbulence, as measured by the Reynolds Number, in the former (114 000 compared with 19 000 - Ref. 8). This greater turbulence means that there is more rapid transfer of carbon dioxide from the gaseous phase to the surface of the reacting solid particles.
- At the completion of the reaction there was no significant difference in the magnesium concentration of the pregnant liquor for both scales of autoclaves (Fig. 32). However, the iron concentration of the pregnant liquor derived from the pilot-scale autoclave was significantly higher than that from the laboratory-scale autoclave (Fig. 33). This probably can be attributed to the high dissolved carbon dioxide concentration of the leach slurry, since it is known that the solubility of magnesium bicarbonate solutions can be increased by increasing the soluble carbon dioxide concentration. Thus the pregnant liquors from the pilot-scale leaches have higher [Fe × 100/Mg] concentration ratios (Table 2).
- Because the iron concentration of the pregnant liquor increases as the slake time increases, it is essential to keep the slake period to a minimum, particularly when using a high turbulence reactor.
- At 15.5°C, an increase in the operating carbon dioxide pressure increases the rate of magnesium dissolution (Fig. 34). As noted above, the rate of reaction is greater for the pilot-scale tests. At 35°C, an increase in the operating carbon dioxide pressure also leads to an increase in the magnesium dissolution rate (Fig. 36). An important point to note, however, is that at 35°C and 20 psig carbon

dioxide, incomplete reaction takes place when using the laboratory-scale autoclave, whereas this does not occur in the pilot-scale test. This can be attributed to the higher dissolved carbon dioxide concentration in the latter which prevents the precipitation of an intermediate hydroxycarbonate magnesium compound, as occurs with the laboratory-scale autoclave.

- An increase in the operating carbon dioxide pressure increases the iron concentration at both 15.5 and 35°C for both scale of reactors. The difference between the iron concentration of the liquors derived from the two scales of test is greatest at the lowest carbon dioxide pressure used (20 psig) - see Figs. 35 and 37.
- Because of the high turbulence in the pilot-scale autoclave, reaction using a flow-through system is relatively rapid (Table 19, Fig. 31) although less than the case when using a pressurized system. Although a similar test was not carried out using the laboratory-scale autoclave because of problems associated with sampling, a previously reported [5] leach test using an all-glass reaction vessel indicted that with low turbulence, incomplete dissolution of the calcine occurred because of the formation of an insoluble intermediate product.
- At 15.5°C, iron dissolution increases with leaching time, whereas at 35°C the iron concentration passes through a maximum and then decreases. This applies to both sizes of reaction vessel.
- An increase in reaction temperature increases the magnesium dissolution rate but, more importantly, decreases the amount of soluble iron in the pregnant liquor. The decrease is most pronounced with the laboratory-scale autoclave.
- As the pulp density is increased, the iron concentration increases more rapidly than does the magnesium concentration. This is because the increased total soluble carbon dioxide concentration (as dissolved dioxide and the carbonate and bicarbonate anions) stabilizes the soluble iron "carbonato" complex.
- Partial removal of soluble iron by addition of aluminium sulphate to the feed to precipitate an iron-containing hydrotalcite is effective in the pilot-scale autoclave. However, it is to be noted that the final iron concentration for the same addition is somewhat higher than in the case of the laboratory-scale reactor. This is because of the greater degree of dissolution in the pilot-scale autoclave.

- The nature of the pressurizing atmosphere has no effect on the overall magnesium extraction or the time at that this is achieved at 15.5 or 35°C, although the magnesium concentration is initially much lower when using carbon dioxide (Fig. 38). This merely relates to the fact that the first analytical sample was recovered simultaneously with the addition of the last of the feed.
- At 15.5°C, the pressurizing atmosphere affects the iron rate of reaction and ultimate concentration in a manner similar to magnesium. However, at 35°C the nature of the pressurizing atmosphere has a more marked effect on iron dissolution, being greater when pumping in under carbon dioxide (Fig. 39).

PRELIMINARY COST ESTIMATES

Towards the end of 1979, Industrial Mining and Investigations Pty Ltd requested Wright Engineers to carry out preliminary cost estimates for the production of 100 000 tpa of high purity magnesium oxide from Savage River magnesite. The technical information necessary for these estimates was provided by the Division using data collected during the initial stages of the present project. Apart from specifying feed preparation, calcination conditions and treatment of the pregnant leach slurry, the most important data used in the cost estimates were the leaching conditions. These were a minimum slake time, 25 psig carbon dioxide, 3% solids, 25°C and a retention time of 45 min. The capital and operating costs provided by Wright Engineers [9] were encouraging and were not too different to those of alternative processing routes [10].

As the project progressed, it became apparent that the above leach conditions might not be the most appropriate. This was particularly so when the results of the present batch pilot-scale tests were taken into account. It was decided to request Wright Engineers to repeat their calculations using more reliable leaching conditions. Eight sets of operating conditions were chosen, with carbon dioxide pressures, pulp densities and retention times in the ranges 20-50 psig, 35-45°C, 2-3% solids and 15-60 min, respectively. These conditions would yield a range of products of varying purity. Advice provided via Industrial

Mining and Investigations Pty Ltd [11] was that there were no significant changes to the capital and operating costs. This was particularly significant as it was becoming clear that leaching at an elevated temperature and at a reduced pulp density would be advantageous in terms of minimizing iron dissolution. The increased heating costs and reduced throughput per unit volume of reactor capacity were countered by the reduced retention time.

CONCLUSIONS

In general terms, the batch leach tests using the pilot-scale autoclaves proved to be very informative. The most significant finding of the present phase of this project is that the degree of iron dissolution is markedly affected by the degree of turbulence in the reactor. This generates a much higher total dissolved carbon dioxide concentration which stabilizes the soluble but relatively unstable iron "carbonato" complex. This is obviously undesirable. More desirable, however, is the fact that the increased turbulence increases the rate of magnesium dissolution and also, in effect, increases the solubility of magnesium bicarbonate at elevated temperatures.

Previous results [8] have shown that it is possible to virtually eliminate iron dissolution by operating at higher temperatures and lower pulp densities. As noted above, leaching under these conditions has no effect on capital and operating costs. Therefore, there is the need to establish if, on a continuous basis, the increased degree of turbulence still allows an essentially iron-free pregnant magnesium bicarbonate solution to be obtained. The higher degree of turbulence would probably result in an even shorter retention time required to achieve maximum magnesium dissolution. It would probably also be necessary to hold the pregnant liquor for some time under the same operating conditions to ensure that any soluble iron would precipitate as the retention time was increased. Thus the higher turbulence would not decrease the overall retention time but may, in fact, require it to be increased.

Another significant finding of the present work is that the properties of the pregnant leach slurry are somewhat different to those of the majority of hydrometallurgical operations. These properties arise because of the instability of soluble magnesium bicarbonate in a carbon dioxide-free environment. They indicate that all handling of the pregnant leach slurry will have to be carried out under a substantial carbon dioxide pressure. This includes transfer, storage and solid-liquid separation.

During the next period it is planned to carry out several continuous (>24 h) leaches in order to gain a more realistic assessment of "optimum" leaching conditions and of certain aspects of reactor design. It will also provide more experience in materials handling. It is anticipated that the data will assist in the design of a more realistic (>5 tpd) pilot plant covering all unit processes from feed preparation (crushing and grinding) through to precipitation of high purity magnesium carbonate trihydrate and the calcination of the latter to high purity, high bulk density magnesium oxide. The design, construction and operation of this next scale of commercial development would allow more realistic costing and engineering data to be obtained. The Division does not have the expertise or facilities to undertake this work, but will be available to act as "consultants" to the contracting engineers employed for this purpose.

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Table 1. Calcine composition (%).

Unscreened						Screened					
Sample	MgO	CaO	Fe_2O_3	CO_2	Balance	Sample	MgO	CaO	Fe_2O_3	CO_2	Balance
PPF/1	58.9	5.72	3.86	21.0	10.5	PPFS/1	62.0	5.40	3.65	23.4	5.55
PPF/2	56.7	5.73	3.65	21.7	12.2	PPFS/2	61.2	5.26	3.85	22.6	7.09
PPF/3	53.4	5.57	3.75	20.7	16.6	PPFS/3	62.2	5.22	3.72	22.4	6.46
PPF/4	50.2	5.78	3.76	21.8	18.5	PPFS/4	62.2	5.32	3.73	23.1	5.65
PPF/5	52.2	5.29	3.62	21.5	17.4	PPFS/5	65.9	5.36	3.85	18.9	5.99
PPF/6	55.9	5.95	3.65	20.7	13.8	PPFS/6	61.5	5.19	3.89	21.6	7.82
PPF/7	58.2	5.60	3.69	20.6	11.9	PPFS/7	61.2	5.30	3.82	22.0	7.68
PPF/8	56.4	5.90	3.60	21.0	13.1	PPFS/8	61.3	5.37	3.83	22.0	7.50
PPF/9	56.5	5.36	3.70	20.1	14.3	PPFS/9	60.0	5.41	3.82	21.6	9.17
PPF/10	52.4	5.75	3.65	21.4	16.8	PPFS/10	61.3	4.98	3.78	21.7	8.24
PPF/11	57.5	5.36	3.56	22.1	11.5	PPFS/11	60.4	5.29	3.78	20.6	9.93
PPF/12	56.2	5.53	3.95	19.8	14.5	PPFS/12	60.5	5.34	3.82	21.8	8.54
PPF/13	58.3	5.18	4.12	21.9	10.5	PPFS/13	60.7	5.12	3.88	22.0	8.30
						PPFS/14	60.2	5.18	3.80	21.6	9.22
						PPFS/15	60.0	5.19	3.78	21.9	9.13
						PPFS/16	59.5	5.12	3.85	21.3	10.2
						PPFS/17	59.7	5.05	3.69	22.8	8.76
						PPFS/18	60.4	4.94	3.69	23.1	7.87
						PPFS/19	60.8	5.27	3.80	23.0	7.13
						PPFS/20	60.7	4.98	3.75	22.6	7.97
						PPFS/21	60.7	5.22	3.73	20.9	9.45
						PPFS/22	59.7	5.60	3.66	21.1	9.94
						PPFS/23	59.7	5.29	3.70	19.6	11.7
						PPFS/24	59.0	5.09	3.65	20.2	12.1

Table 2. Summary of leach conditions and extraction data.

Test	Pressurizing atmosphere	Operating CO_2 pressure (psig)	Operating temp. (°C)	Pulp density (%)	$Al_2(SO_4)_3.16H_2O$ addition (gpl)	Slake period (min)	Pilot autoclave		Laboratory autoclave	
							Mg extraction (%)	[Fe × 100/Mg]*	Mg extraction (%)	[Fe × 100/Mg]
PPL 1	N_2	100	15.5	2	0	30	84.7	0.75	83.3	0.68
PPL 2	N_2	100	15.5	3	0	30	73.9	1.25	73.3	1.18
PPL 3	N_2	100	15.5	4	0	30	76.5	2.52	76.3	2.31
PPL 4	N_2	100	15.5	5	0	30	83.2	3.47	83.2	3.30
PPL 5	N_2	50	15.5	3	0	30	76.1	1.24	75.1	1.10
PPL 6	N_2	20	15.5	3	0	30	77.0	1.18	76.1	0.93
PPL 7	N_2	100	35.0	3	0	45	76.9	0.23	75.2	0.24
PPL 8	N_2	50	35.0	3	0	40	76.0	0.27	75.4	0.22
PPL 9	N_2	20	35.0	3	0	45	74.1	0.22	55.9	0.11
PPL 10	N_2	100	15.5	3	0	120	76.4	1.49	76.6	1.38
PPL 11	N_2	100	15.5	3	10	30	65.3	0.22	64.9	0.21
PPL 12	N_2	100	35.0	3	10	40	64.7	0.21		
PPL 13	CO_2	100	15.5	3	0	10	75.1	1.42		
PPL 14	CO_2	100	15.5	3	0	130	73.4	1.68		
PPL 15	CO_2	100	35.0	3	0	15	78.0	0.45		
PPL 16	CO_2	100	15.5	2	0	10	82.2	0.74		
PPL 17	N_2	0†	15.5	3	0	30	75.5	1.06		

*Of final liquor.

†CO_2 bubbled through autoclave at 7 lpm.

Table 3. Leach test data.

Test:	PPL 1
Calcine composition (%):	MgO 61.2, CaO 5.26, Fe_2O_3 3.85, CO_2 22.6, balance 7.09
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	2
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave Temp. (°C)	Pilot autoclave Mg (gpl)	Pilot autoclave Fe (gpl)	Laboratory autoclave Mg (gpl)	Laboratory autoclave Fe (gpl)
0		10.8				
5		10.9				
0		10.9				
15		11.0				
20		11.3				
25		11.4				
30	0	15.2				
35	5				4.12	0.019
37.5	7.5	15.4	5.21	0.039		
45	15	15.5	5.63	0.042	5.11	0.026
60	30	15.5	5.95	0.044	5.75	0.034
75	45	15.4	6.17	0.046	6.00	0.038
90	60	15.6	6.23	0.046	6.10	0.040
105	75	15.5	6.26	0.047	6.12	0.041
120	90	15.5	6.26	0.047	6.12	0.042
135	105				6.13	0.042
150	120	15.5	6.25	0.047	6.15	0.042
Mg extraction (%)			84.7		83.3	
[Fe × 100/Mg] of final liquor			0.75		0.68	

See Figs. 3 and 4

Table 4. Leach test data.

Test:	PPL 2
Calcine composition (%):	MgO 62.2, CaO 5.22, Fe_2O_3 3.72, CO_2 22.4, balance 6.46
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		14.3				
5		14.5				
10		14.3				
15		14.7				
20		15.2				
25		15.4				
30	0	15.4				
35	5	15.7	6.88	0.078	6.49	0.055
45	15	15.5	7.31	0.086	6.95	0.067
60	30	16.0	7.84	0.094	7.40	0.080
75	45	15.8	8.13	0.100	7.86	0.088
90	60	15.6	8.26	0.102	8.11	0.093
105	75	15.5	8.30	0.103	8.20	0.095
120	90	15.5	8.30	0.104	8.24	0.096
135	105				8.25	0.098
150	120	15.5	8.31	0.104	8.25	0.097
Mg extraction (%)			73.9		73.3	
[Fe × 100/Mg] of final liquor			1.25		1.18	

See Figs. 5 and 6

Table 5. Leach test data.

Test:	PPL 3
Calcine composition (%):	MgO 62.2, CaO 5.32, Fe_2O_3 3.73, CO_2 23.1, balance 5.65
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	4
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave Temp. (°C)	Pilot autoclave Mg (gpl)	Pilot autoclave Fe (gpl)	Laboratory autoclave Mg (gpl)	Laboratory autoclave Fe (gpl)
0		11.2				
5		11.3				
10		11.3				
15		11.7				
20		12.5				
25		13.8				
30	0	16.4				
35	5	16.2	8.46	0.140	7.00	0.063
45	15	15.5	9.34	0.178	8.41	0.094
60	30	15.4	10.40	0.232	9.74	0.168
75	45	15.5	11.05	0.263	10.55	0.210
90	60	15.5	11.34	0.278	11.06	0.234
105	75	15.5	11.44	0.286	11.30	0.252
120	90	15.4	11.46	0.288	11.41	0.261
135	105	15.5	11.48	0.288	11.45	0.264
150	120	15.5	11.48	0.289	11.45	0.264
Mg extraction (%)			76.5		76.3	
[Fe × 100/Mg] of final liquor			2.52		2.31	

See Figs. 7 and 8

Table 6. Leach test data.

Test:	PPL 4
Calcine composition (%):	MgO 65.9, CaO 5.36, Fe_2O_3 3.85, CO_2 18.9, balance 5.99
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	5
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		13.3				
5		13.5				
10		13.5				
15		13.7				
20		14.0				
25		14.3				
30	0	17.0				
35	5	16.8	11.71	0.120	9.48	0.117
45	15	15.7	13.15	0.225	10.65	0.170
60	30	15.5	14.82	0.366	12.50	0.269
75	45	15.4	15.68	0.456	14.08	0.360
90	60	15.5	16.23	0.508	15.16	0.435
105	75	15.5	16.29	0.539	15.82	0.488
120	90	15.5	16.30	0.559	16.24	0.520
135	105	15.5	16.30	0.565	16.32	0.536
150	120	15.5	16.30	0.566	16.35	0.540
Mg extraction (%)			83.2		83.2	
[Fe × 100/Mg] of final liquor			3.47		3.30	

See Figs. 9 and 10

Table 7. Leach test data.

Test:	PPL 5
Calcine composition(%):	MgO 60.4, CaO 5.29, Fe_2O_3 3.78, CO_2 20.6, balance 9.93
Operating CO_2 pressure (psig):	50
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		12.8				
5		12.8				
10		12.7				
15		13.1				
20		13.6				
25		14.0				
30	0	18.5				
35	5	16.0	6.06	0.066	4.86	0.008
45	15	15.5	6.58	0.074	5.56	0.023
60	30	15.4	7.25	0.085	6.50	0.046
75	45	15.6	7.81	0.095	7.24	0.062
90	60	15.5	8.09	0.098	7.69	0.074
105	75	15.5	8.24	0.102	8.05	0.082
120	90	15.5	8.30	0.102	8.14	0.087
135	105	15.4	8.31	0.102	8.20	0.089
150	120	15.5	8.31	0.103	8.20	0.090
Mg extraction (%)			76.1		75.1	
[Fe × 100/Mg] of final liquor			1.24		1.20	

See Figs. 11 and 12

Table 8. Leach test data.

Test:	PPL 6
Calcine composition (%):	MgO 59.5, CaO 5.12, Fe_2O_3 3.85, CO_2 21.3, balance 10.2
Operating CO_2 pressure (psig):	20
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		13.5				
5		13.5				
10		14.0				
15		14.4				
20		14.5				
25		15.0				
30	0	15.9				
35	5	17.2	3.66	0.015	3.21	0.003
45	15	16.3	4.55	0.028	3.92	0.012
60	30	15.8	5.66	0.046	4.96	0.029
75	45	15.5	6.54	0.064	5.85	0.043
90	60	15.5	7.31	0.079	6.69	0.055
105	75	15.4	7.79	0.088	7.25	0.063
120	90	15.5	8.09	0.095	7.88	0.069
135	105	15.5	8.24	0.097	8.04	0.073
150	120	15.5	8.29	0.098	8.20	0.076
Mg extraction (%)			77.0		76.1	
[Fe × 100/Mg] of final liquor			1.18		0.93	

See Figs. 13 and 14

Table 9. Leach test data.

Test:	PPL 7
Calcine composition (%):	MgO 60.7, CaO 5.12, Fe_2O_3 3.88, CO_2 22.0, balance 8.30
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	35.0
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	45

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		10.5				
5		10.8				
10		11.0				
15		12.4				
20		16.0				
25		19.8				
30		24.0				
35		28.3				
40		33.2				
45	0	36.2				
50	5	35.2	7.51	0.047	7.14	0.040
60	15	34.8	7.79	0.048	7.44	0.041
75	30	35.1	8.04	0.043	7.78	0.037
90	45	35.2	8.26	0.032	8.08	0.030
105	60	34.7	8.35	0.027	8.16	0.024
120	75	34.9	8.42	0.020	8.22	0.022
135	90	35.2	8.45	0.019	8.25	0.021
150	105	35.0	8.44	0.019	8.26	0.020
165	120	35.1	8.44	0.019	8.26	0.020
Mg extraction (%)			76.9		75.2	
[Fe × 100/Mg] of final liquor			0.23		0.24	

See Figs. 15 and 16

Table 10. Leach test data.

Test:	PPL 8
Calcine composition (%):	MgO 59.7, CaO 3.61, Fe_2O_3 3.69, CO_2 22.8, balance 8.76
Operating CO_2 pressure (psig):	50
Operating temperature (°C):	35.0
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	40

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		14.0				
5		14.8				
10		15.3				
15		17.0				
20		20.5				
25		23.2				
30		27.0				
35		31.3				
40	0	35.6				
45	5	35.8	6.80	0.039	6.21	0.027
55	15	35.2	7.11	0.040	6.65	0.038
70	30	35.1	7.50	0.038	7.32	0.036
85	45	34.9	7.84	0.034	7.68	0.029
100	60	35.0	8.09	0.030	7.99	0.023
115	75	35.1	8.23	0.027	8.06	0.021
130	90	34.8	8.20	0.024	8.10	0.019
145	105	35.0	8.22	0.022	8.14	0.019
160	120	35.2	8.21	0.022	8.14	0.018
Mg extraction (%)			76.0		75.4	
[Fe × 100/Mg] of final liquor			0.27		0.22	

See Figs. 17 and 18

Table 11. Leach test data.

Test:	PPL 9
Calcine composition (%):	MgO 60.8, CaO 5.27, Fe_2O_3 3.80, CO_2 23.0, balance 7.13
Operating CO_2 pressure (psig):	20
Operating temperature (°C):	35.0
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	45

Test time (min)	Leach time (min)	Pilot autoclave Temp. (°C)	Pilot autoclave Mg (gpl)	Pilot autoclave Fe (gpl)	Laboratory autoclave Mg (gpl)	Laboratory autoclave Fe (gpl)
0		10.8				
5		11.4				
0		11.8				
15		13.3				
20		18.0				
25		20.5				
30		25.1				
35		28.5				
40		34.1				
45	0	36.2				
50	5	35.6	4.82	0.021	4.86	0.000
60	15	34.9	6.06	0.029	5.12	0.004
75	30	35.2	7.15	0.038	5.59	0.008
90	45	34.8	7.74	0.040	5.90	0.009
105	60	35.0	8.00	0.039	6.01	0.008
120	75	35.2	8.05	0.036	6.14	0.007
135	90	35.1	8.10	0.032	6.18	0.007
150	105	35.0	8.19	0.025	6.15	0.007
165	120	35.0	8.16	0.018	6.16	0.007
Mg extraction (%)			74.1		55.9	
[Fe × 100/Mg] of final liquor			0.22		0.11	

See Figs. 19 and 20

Table 12. Leach test data.

Test:	PPL 10
Calcine composition (%):	MgO 60.0, CaO 5.41, Fe_2O_3 3.82, CO_2 21.6, balance 9.17
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3.16H_2O$ (gpl):	0
Slake period (min):	120

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		10.3				
15		10.8				
30		12.2				
45		13.5				
60		15.1				
75		15.5				
90		15.4				
105		15.5				
120	0	18.5				
125	5	16.2	6.80	0.075	6.51	0.071
135	15	15.4	7.22	0.088	6.99	0.081
150	30	15.5	7.69	0.104	7.63	0.095
165	45	15.6	8.04	0.116	7.96	0.104
180	60	15.5	8.18	0.120	8.18	0.109
195	75	15.4	8.25	0.123	8.30	0.113
210	90	15.5	8.30	0.124	8.32	0.114
225	105	15.5	8.31	0.124	8.31	0.114
240	120	15.5	8.30	0.124	8.32	0.115
Mg extraction (%)			76.4		76.6	
[Fe × 100/Mg] of final liquor			1.49		1.38	

See Figs. 21 and 22

Table 13. Leach test data.

Test:	PPL 11
Calcine composition (%):	MgO 60.5, CaO 5.34, Fe_2O_3 3.82, CO_2 21.8, balance 8.54
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	10
Slake period (min):	30

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		13.0				
5		13.0				
10		13.2				
15		14.0				
20		15.5				
25		15.3				
30	0	16.3				
35	5	16.8	5.59	0.000	5.24	0.000
45	15	16.0	5.81	0.003	5.59	0.003
60	30	15.8	6.19	0.006	6.12	0.007
75	45	15.5	6.60	0.009	6.54	0.010
90	60	15.4	6.81	0.012	6.79	0.012
105	75	15.5	7.03	0.014	7.00	0.014
120	90	15.6	7.15	0.015	7.09	0.015
135	105	15.3	7.12	0.016	7.06	0.015
150	120	15.5	7.15	0.016	7.11	0.015
Mg extraction (%)			65.3		64.9	
[Fe × 100/Mg] of final liquor			0.22		0.21	

See Figs. 23 and 24

Table 14. Leach test data.

Test:	PPL 12
Calcine composition (%):	MgO 60.4, CaO 4.94, Fe_2O_3 3.69, CO_2 23.1, balance 7.87
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	35.0
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	10
Slake period (min):	40

Test time (min)	Leach time (min)	Pilot autoclave			Laboratory autoclave	
		Temp. (°C)	Mg (gpl)	Fe (gpl)	Mg (gpl)	Fe (gpl)
0		12.2				
5		12.8				
10		13.5				
15		15.5				
20		21.3				
25		23.3				
30		28.2				
35		33.3				
40	0	35.9				
45	5	35.6	5.56	0.000	5.12	0.000
55	15	35.4	5.78	0.003	5.41	0.000
78	30	35.2	6.16	0.006	5.95	0.000
85	45	35.5	6.41	0.008	6.34	0.000
100	60	35.5	6.66	0.012	6.64	0.001
115	75	35.5	7.00	0.012	6.89	0.002
130	90	35.6	7.08	0.014	7.12	0.002
145	105	35.5	7.14	0.014	7.10	0.003
160	120	35.5	7.16	0.015	7.12	0.003
Mg extraction (%)			64.7		64.3	
[Fe × of final liquor			0.21		0.04	

See Figs. 25 and 26

Table 15. Leach test data.

Test:	PPL13
Calcine composition (%):	MgO 61.2, CaO 5.30, Fe_2O_3 3.82, CO_2 22.0, balance 7.68
Pressurizing atmosphere:	CO_2
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3.16H_2O$ (gpl):	0
Slake period (min):	10

Leach time (min)	Temperature (°C)	Liquor composition (gpl)	
		Mg	Fe
0	11.0		
5	13.5		
10	15.0		
15	15.6		
20	15.5	5.96	0.051
30	15.4	7.14	0.081
45	15.4	7.90	0.104
60	15.5	8.25	0.113
75	15.6	8.29	0.116
90	15.5	8.30	0.118
105	15.5	8.31	0.119
120	15.6	8.31	0.118

Mg extraction (%)	75.1
[Fe × 100/Mg] of final liquor	1.42

See Fig. 27

Table 16. Leach test data.

Test:	PPL 14
Calcine composition (%):	MgO 61.3, CaO 4.98, Fe_2O_3 3.78, CO_2 21.7, balance 8.24
Pressurizing atmosphere:	CO_2
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	130

Leach time (min)	Temperature (°C)	Liquor composition (gpl)	
		Mg	Fe
0	11.7		
5	15.0		
10	16.0		
15	15.5		
20	15.3	6.27	0.055
30	15.5	7.07	0.081
45	15.4	7.69	0.114
60	15.5	8.03	0.128
75	15.6	8.11	0.134
90	15.3	8.15	0.136
105	15.5	8.15	0.137
120	15.5	8.15	0.137

Mg extraction (%)	73.4
[Fe × 100/Mg] of final liquor	1.68

See Fig. 28

Table 17. Leach test data.

Test:	PPL 15
Calcine composition (%):	MgO 60.2, CaO 5.18, Fe_2O_3 3.80, CO_2 21.6, balance 9.22
Pressurizing atmosphere:	CO_2
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	35.0
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	15

Leach time (min)	Temperature (°C)	Liquor composition (gpl)	
		Mg	Fe
0	12.4		
5	13.8		
10	15.0		
15	19.0		
20	25.0		
25	29.5		
30	33.5		
35	35.0	7.83	0.069
45	34.8	8.05	0.065
60	35.1	8.36	0.058
75	35.0	8.43	0.052
90	35.1	8.46	0.046
105	35.2	8.44	0.041
120	34.9	8.49	0.038
Mg extraction (%)		78.0	
[Fe × 100/Mg] of final liquor		0.45	

See Fig. 29

Table 18. Leach test data.

Test:	PPL 16
Calcine composition (%):	MgO 61.5, CaO 5.19, Fe_2O_3 3.89, CO_2 21.6, balance 7.82
Pressurizing atmosphere:	CO_2
Operating CO_2 pressure (psig):	100
Operating temperature (°C):	15.5
Pulp density (% solids):	2
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	10

Leach time (min)	Temperature (°C)	Liquor composition (gpl)	
		Mg	Fe
0	11.3		
5	13.5		
10	14.2		
15	15.4		
20	15.5	3.84	0.030
30	15.5	4.69	0.036
45	15.4	5.60	0.042
60	15.5	5.95	0.044
75	15.4	6.04	0.044
90	15.6	6.10	0.045
105	15.5	6.11	0.045
120	15.5	6.10	0.045

Mg extraction (%)	82.2
[Fe × 100/Mg] of final liquor	0.74

See Fig. 30

Table 19. Leach test data.

Test:	PPL 17
Calcine composition (%):	MgO 60.0, CaO 5.19, Fe_2O_3 3.78, CO_2 21.9, balance 9.13
Pressurizing atmosphere:	N_2
Operating CO_2 pressure (psig):	0 - CO_2 bubbled through autoclave at 7 lpm
Operating temperature (°C):	15.5
Pulp density (% solids):	3
$Al_2(SO_4)_3 \cdot 16H_2O$ (gpl):	0
Slake period (min):	30

Test time (min)	Leach time (min)	Temperature (°C)	Liquor composition (gpl)	
			Mg	Fe
0		13.6		
5		13.0		
10		13.3		
15		13.5		
20		14.0		
25		14.5		
30	0	15.5		
35	5	15.6	1.68	0.004
45	15	15.3	2.70	0.013
60	30	15.4	4.19	0.030
75	45	15.4	5.35	0.047
90	60	15.5	6.41	0.058
105	75	15.5	7.05	0.070
120	90	15.6	7.58	0.078
135	105	15.5	8.00	0.085
150	120	15.4	8.20	0.087

Mg extraction (%)	75.5
[Fe × 100/Mg] of final liquor	1.06

See Fig. 31

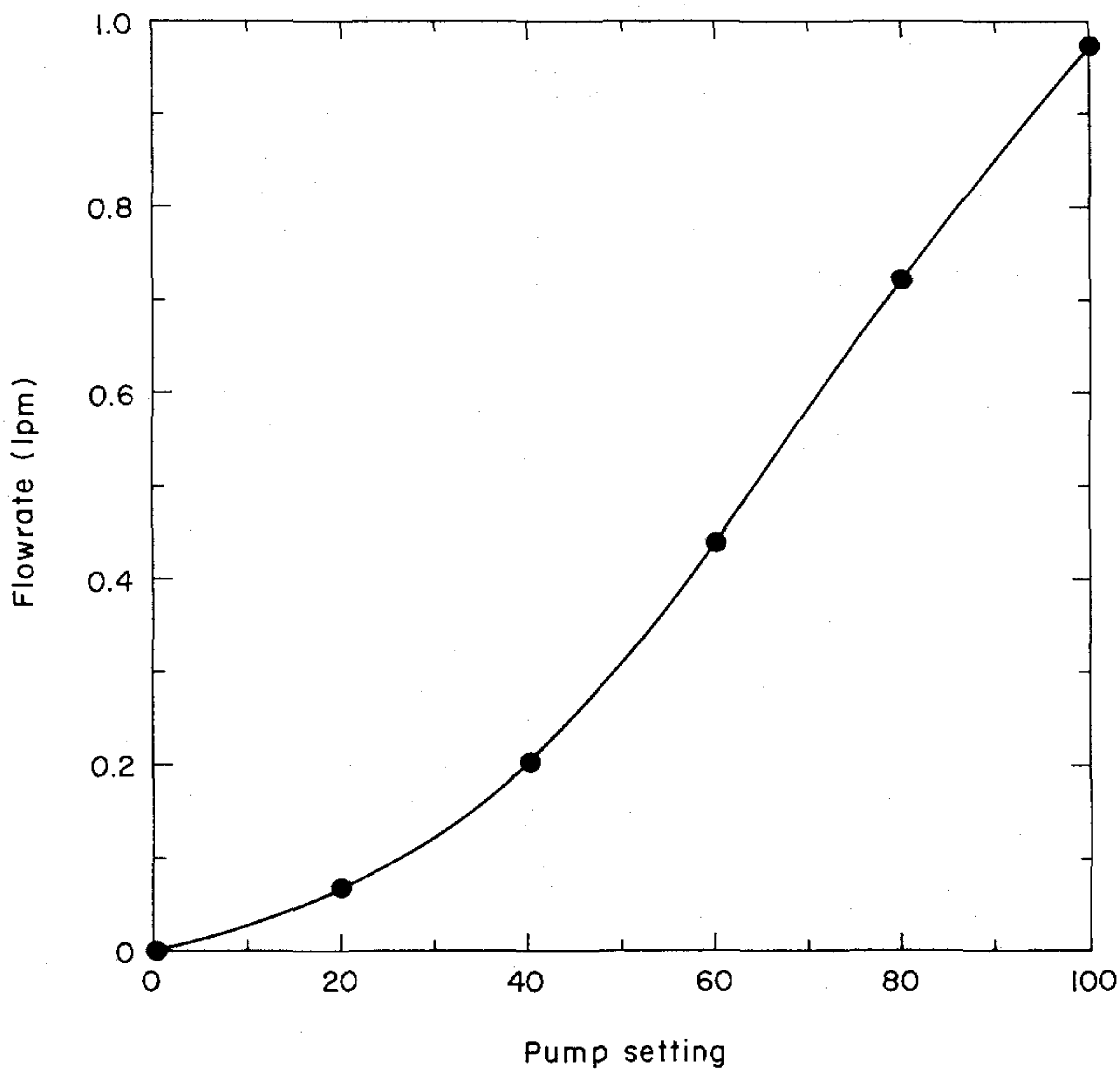

Fig. 1. Slurry pumping rate with 20-200 psig nitrogen overpressure.

Fig. 2. Pilot-scale autoclaves with modified sampling ports and drainage lines.

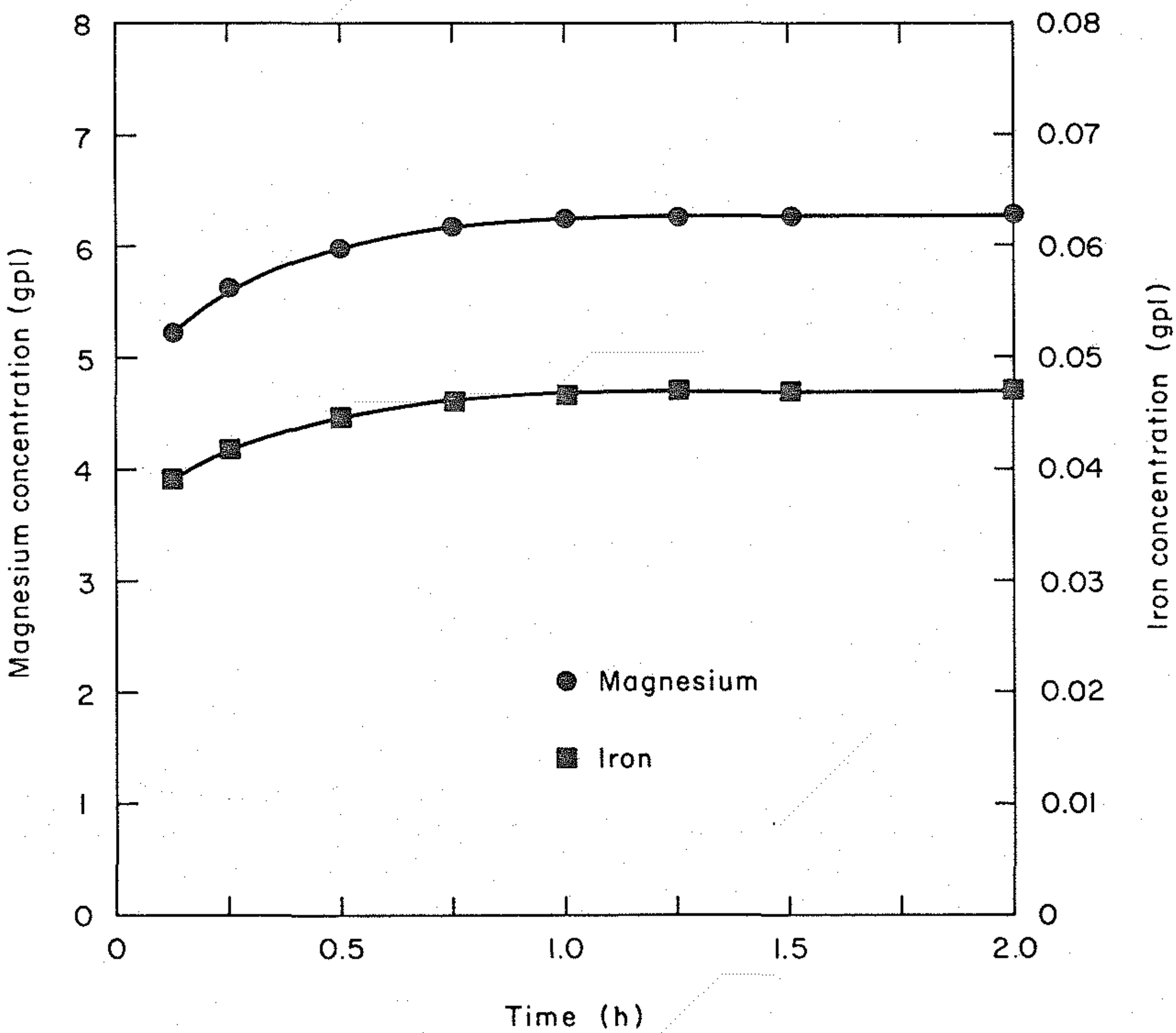

Fig. 3. Pilot-scale autoclave leach test data - PPL 1. See Table 3.

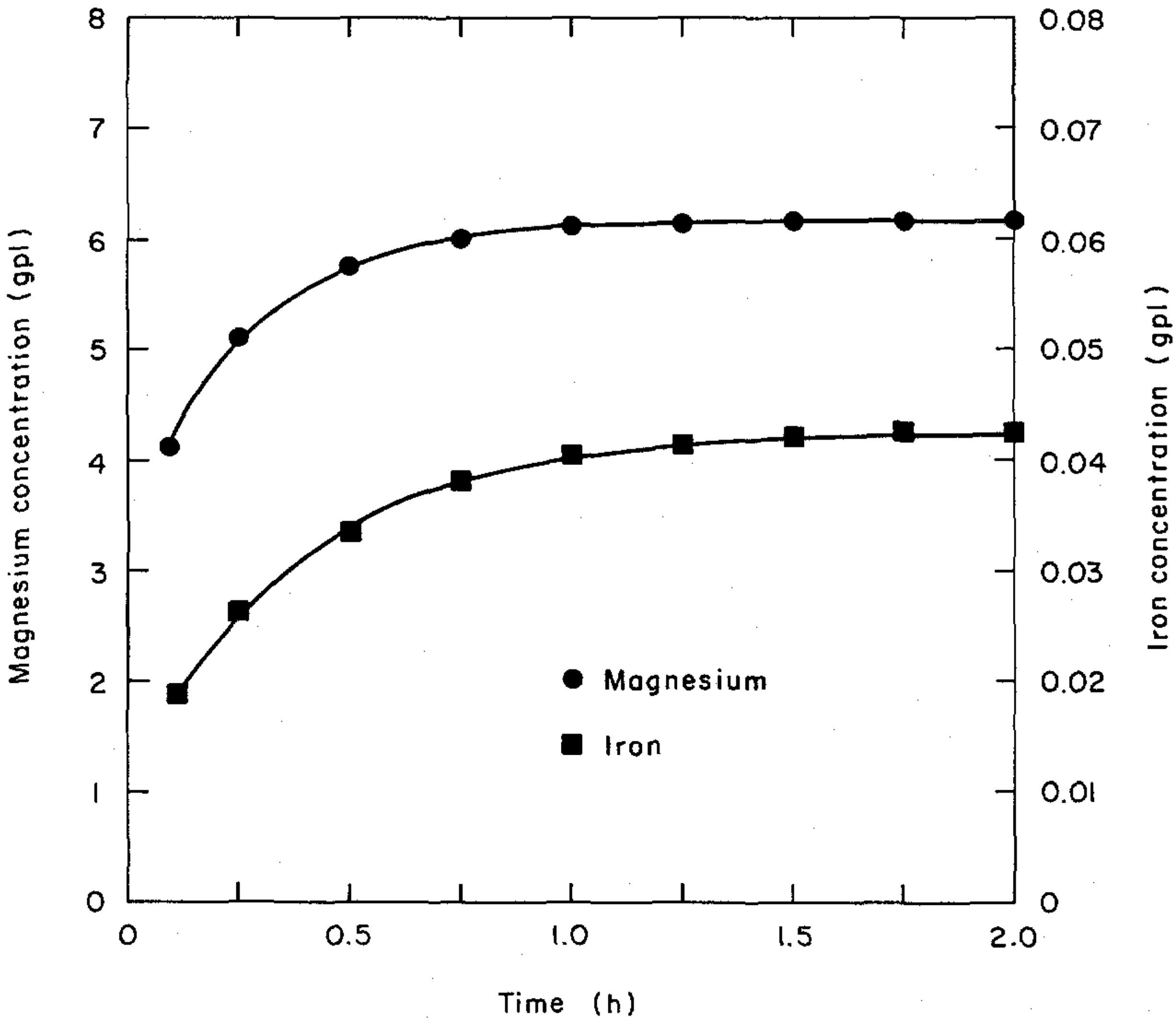

Fig. 4. Laboratory-scale autoclave leach test data - PPL 1. See Table 3.

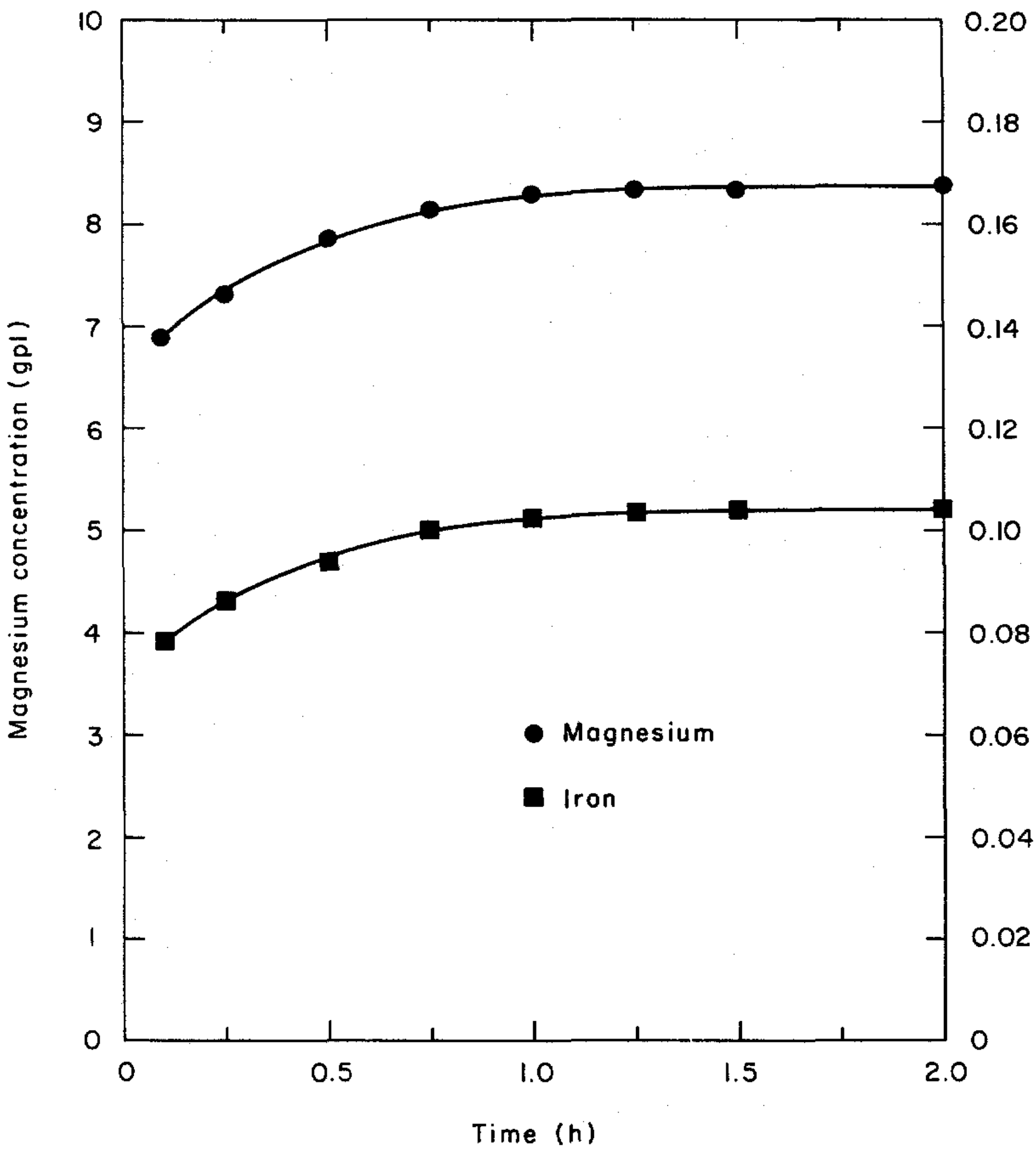

Fig. 5. Pilot-scale autoclave leach test data - PPL 2. See Table 4.

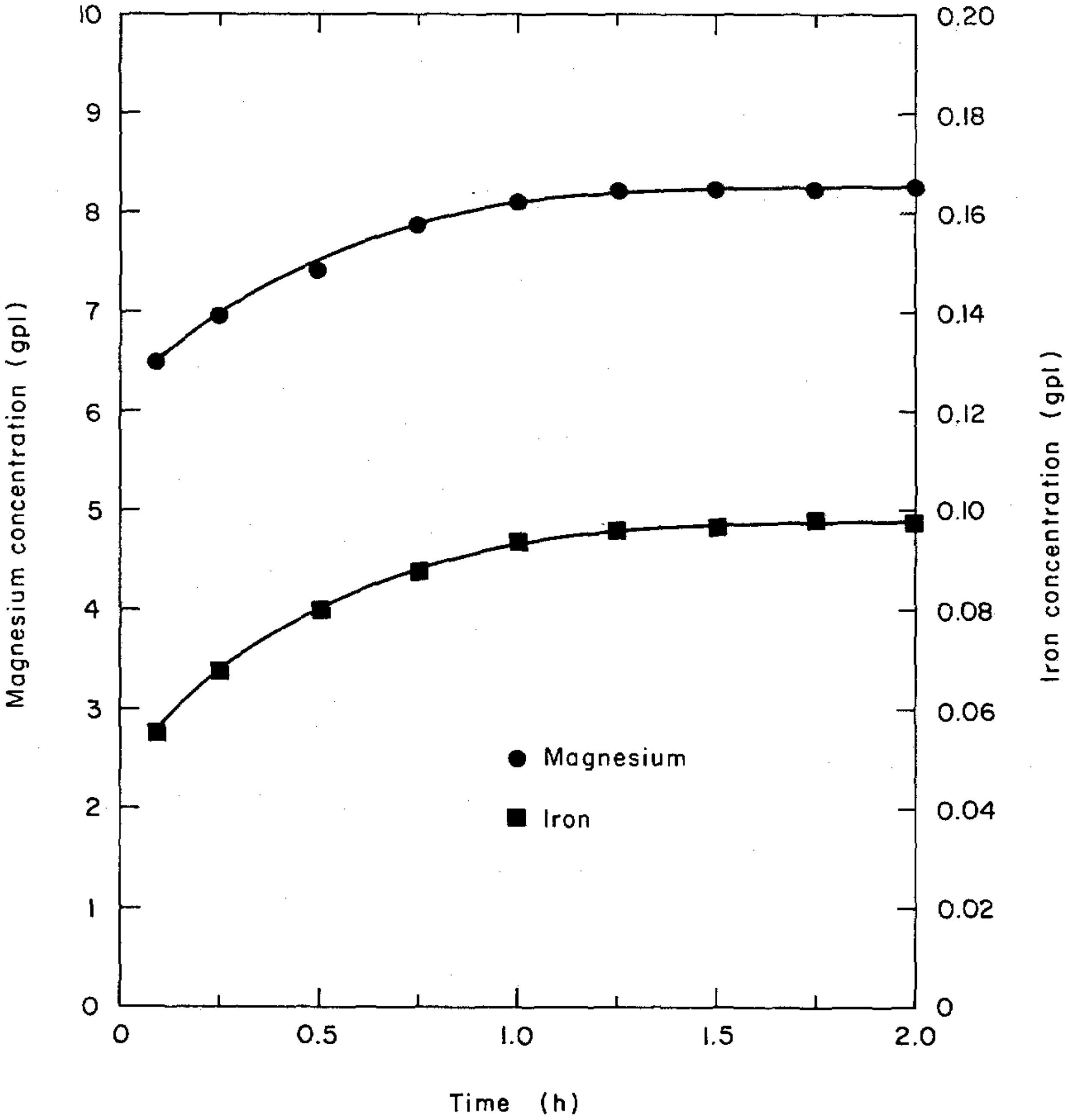

Fig. 6. Laboratory-scale autoclave leach test data - PPL 2. See Table 4.

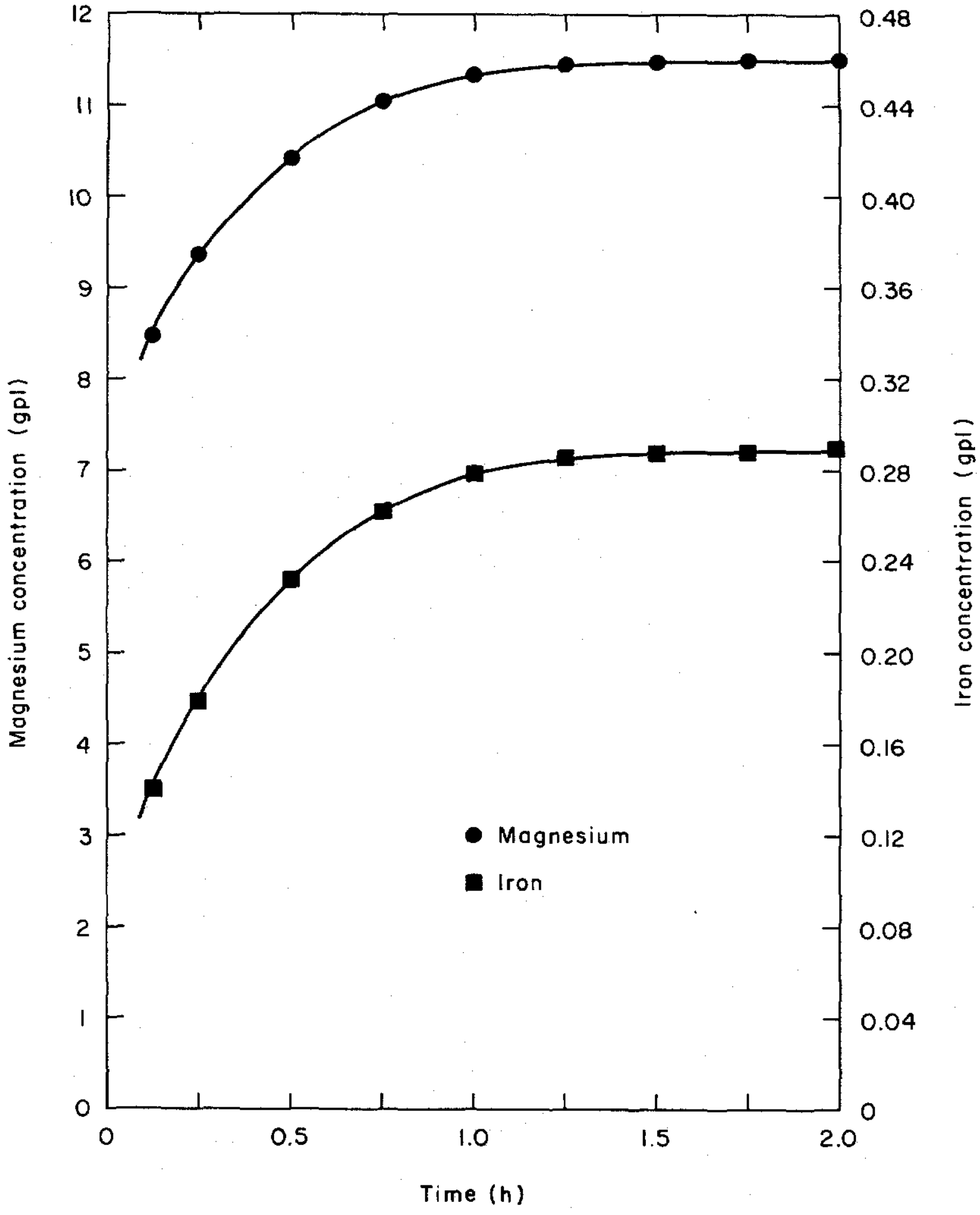

Fig. 7. Pilot-scale autoclave leach test data - PPL 3. See Table 5.

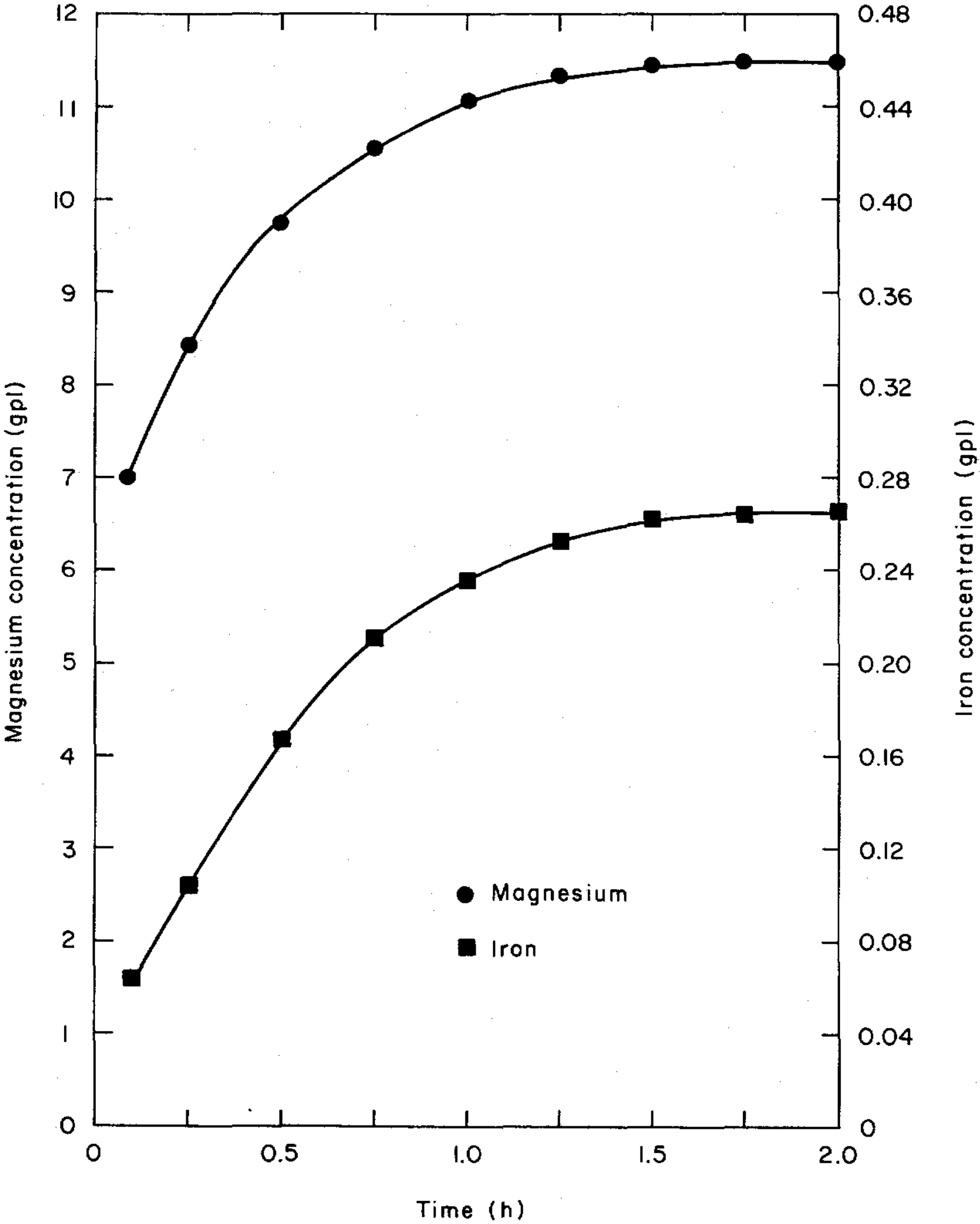

Fig. 8. Laboratory-scale autoclave leach test data - PPL 3. See Table 5.

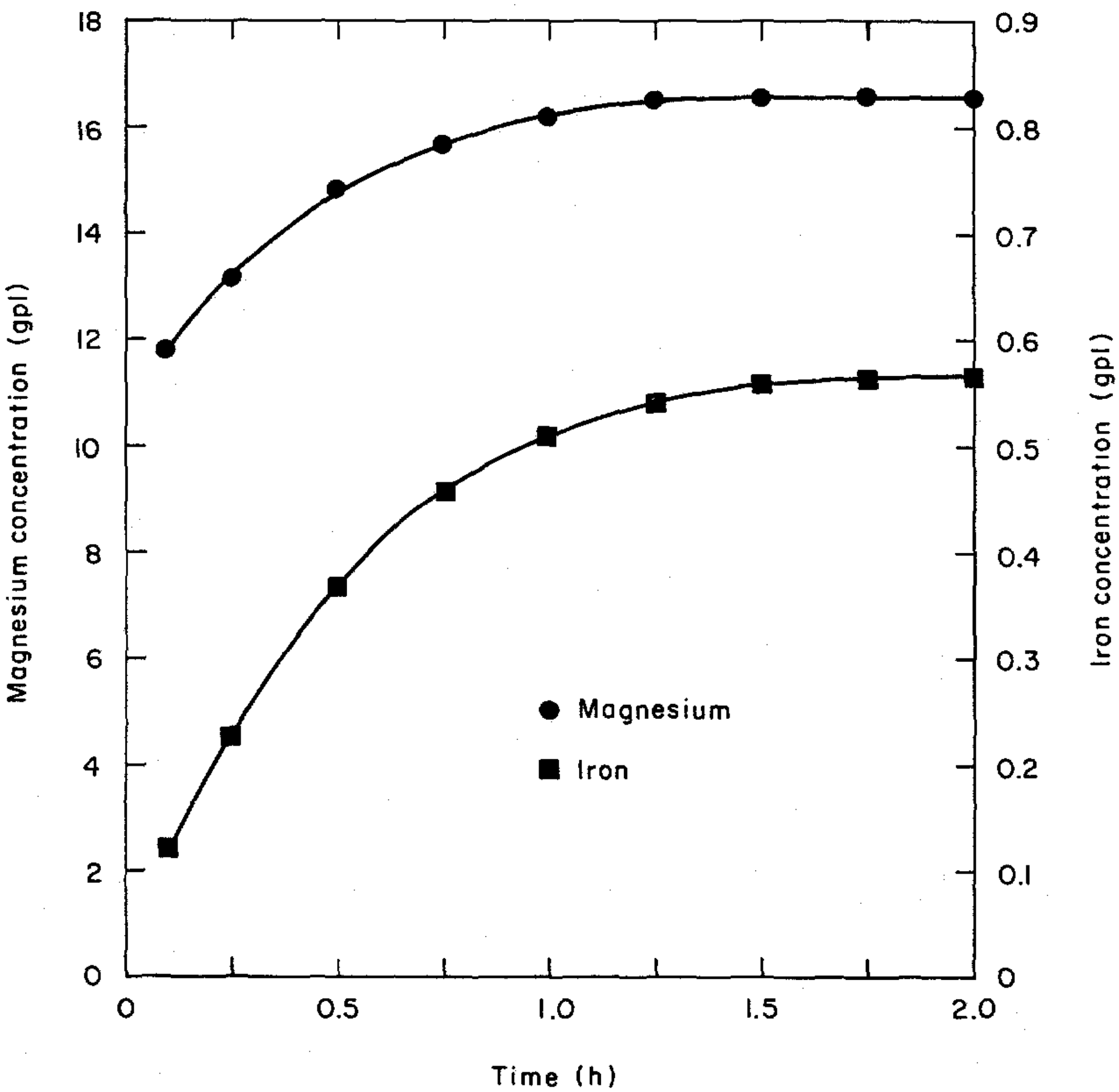

Fig. 9. Pilot-scale autoclave leach test data - PPL 4. See Table 6.

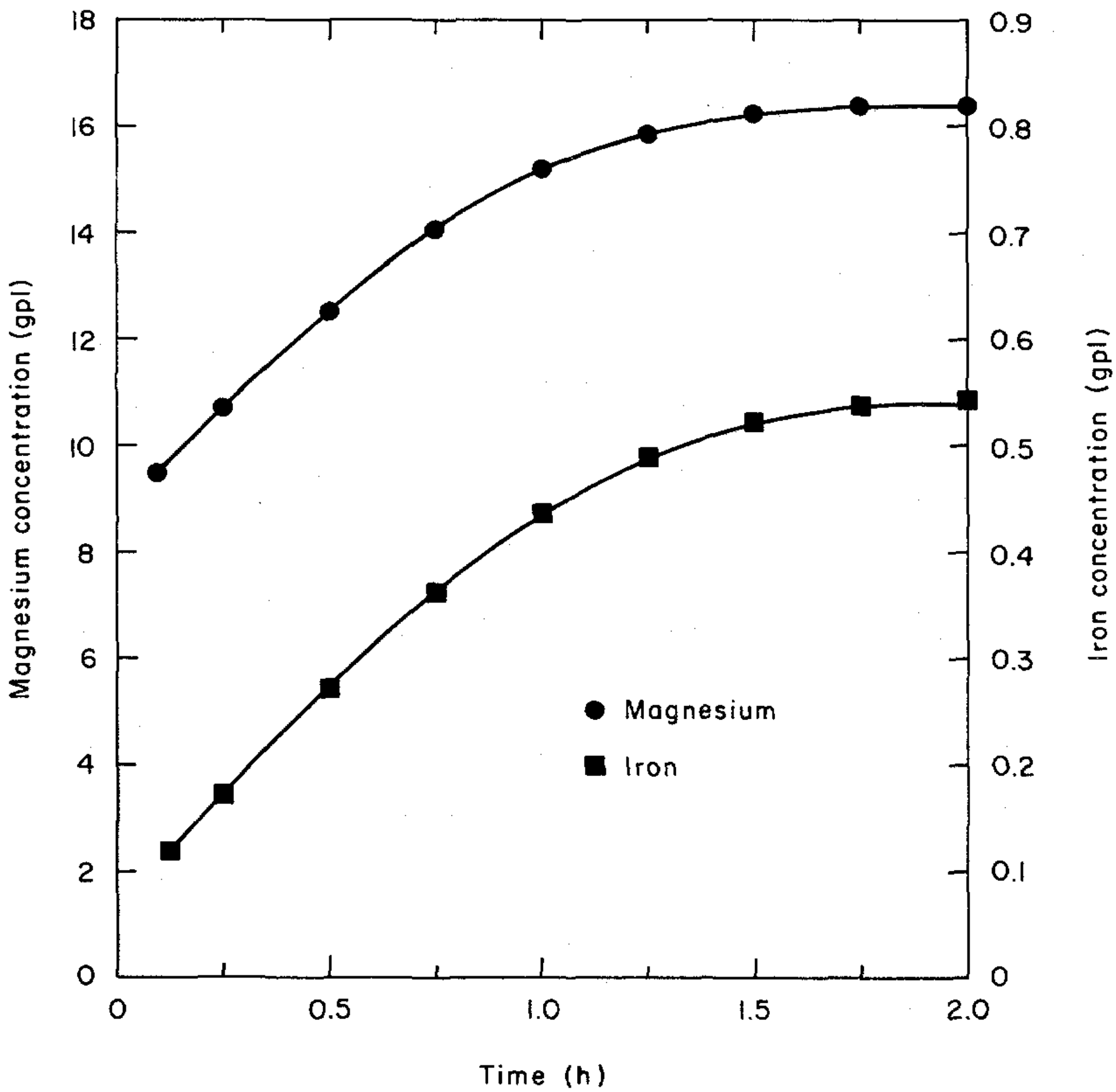

Fig. 10. Laboratory-scale autoclave leach test data - PPL 4. See Table 6.

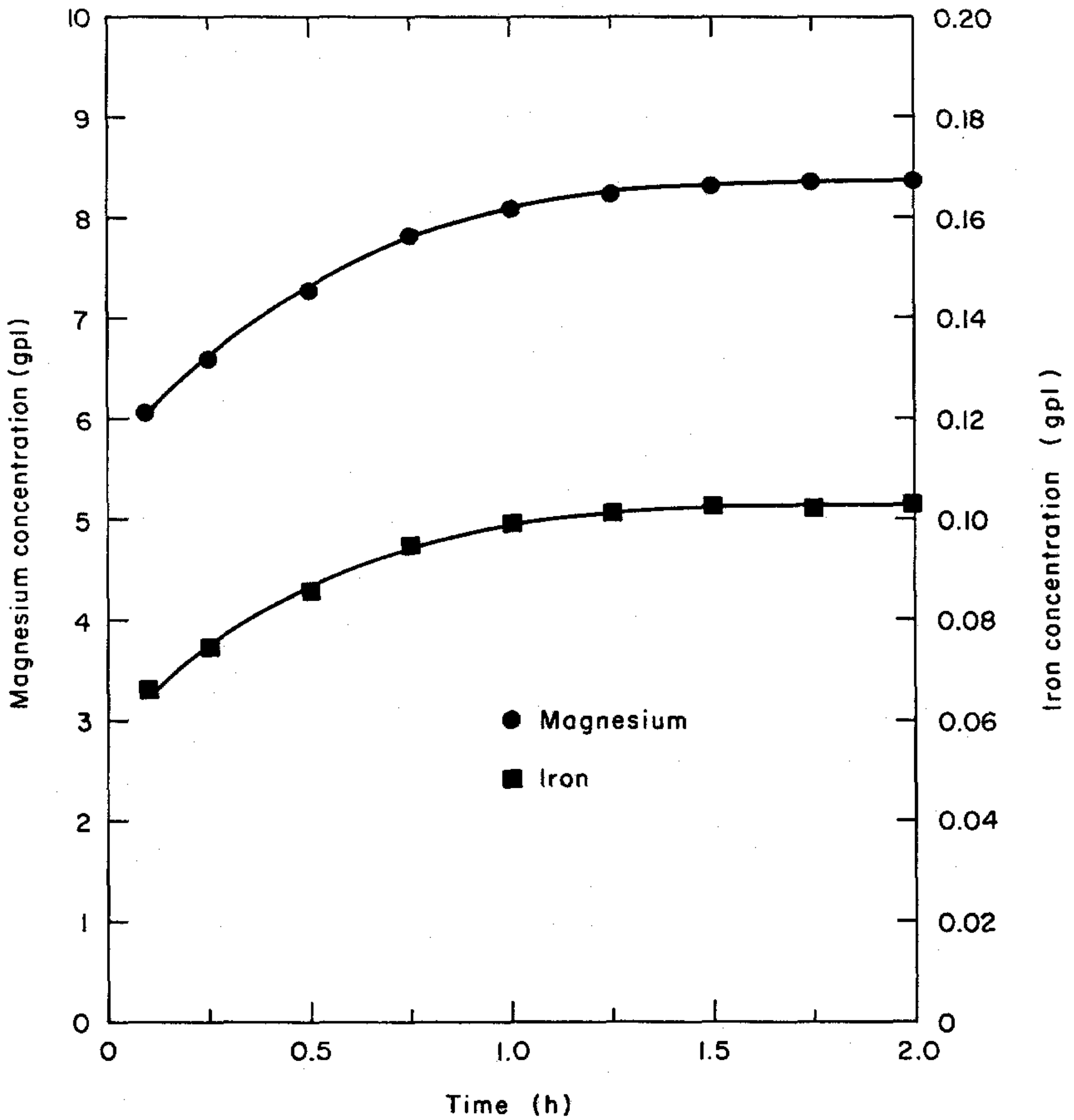

Fig. 11. Pilot-scale autoclave leach test data - PPL 5. See Table 7.

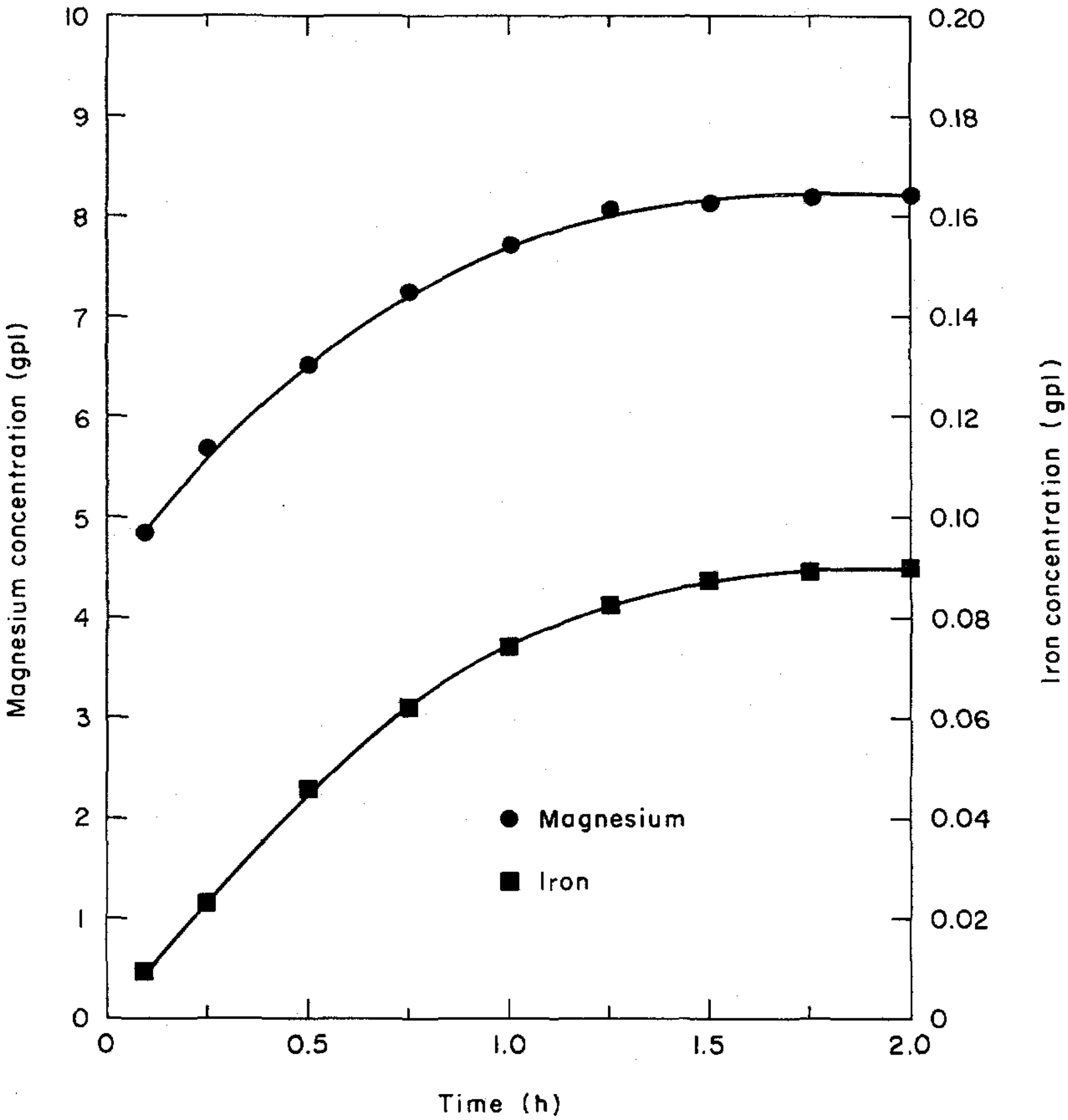

Fig. 12. Laboratory-scale autoclave leach test data - PPL 5. See Table 7.

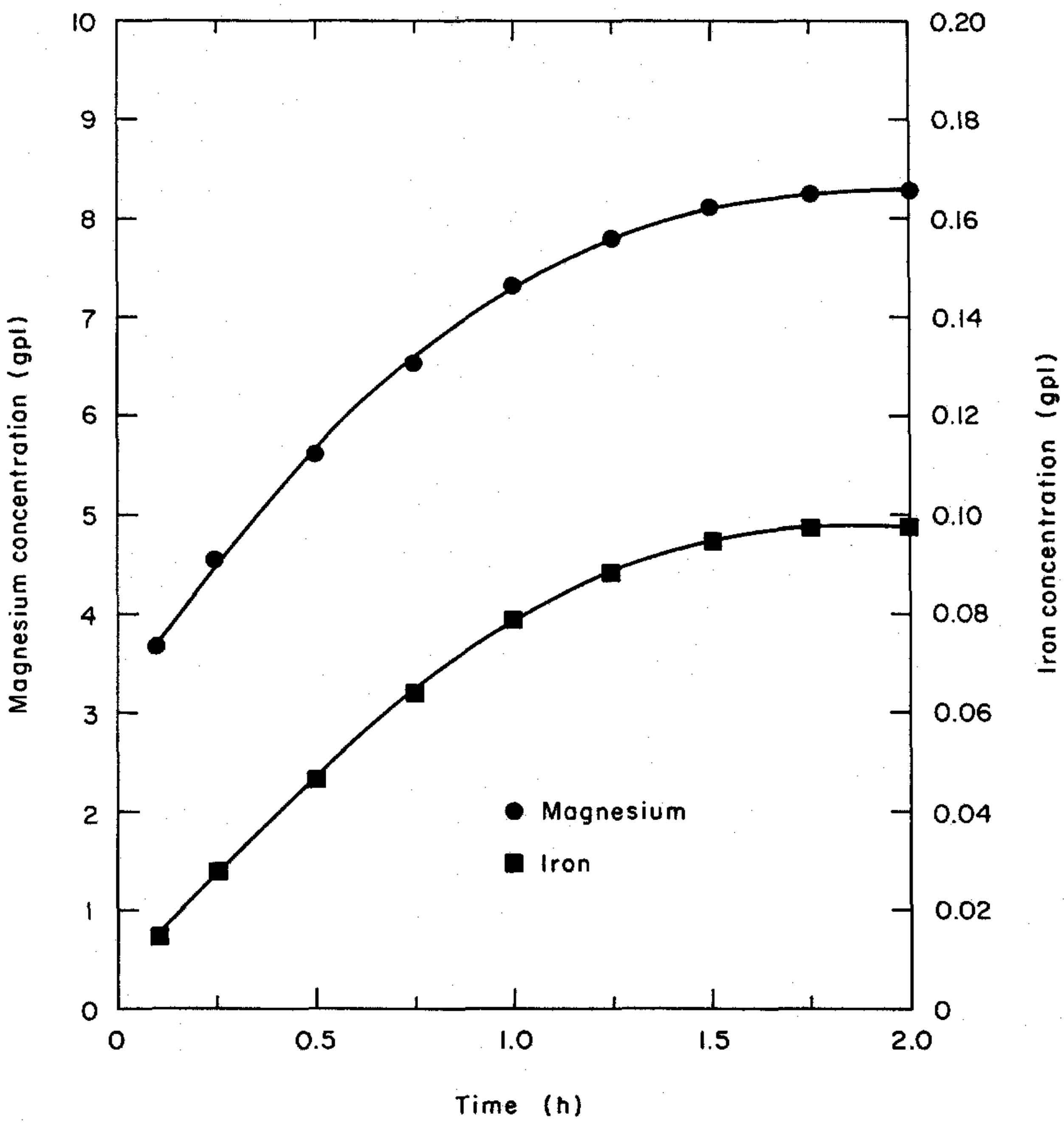

Fig. 13. Pilot-scale autoclave leach test data - PPL 6. See Table 8.

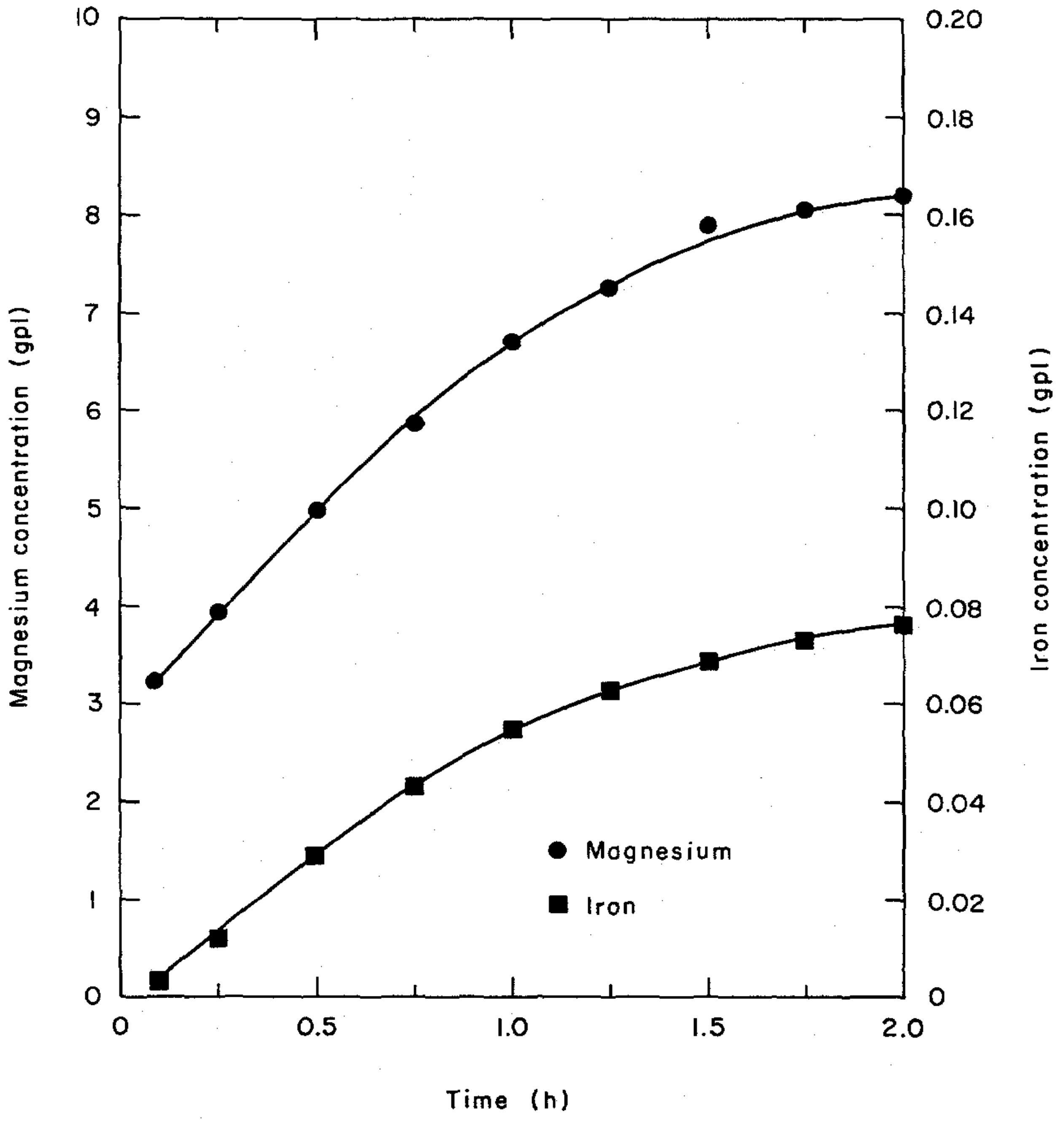

Fig. 14. Laboratory-scale autoclave leach test data - PPL 6. See Table 8.

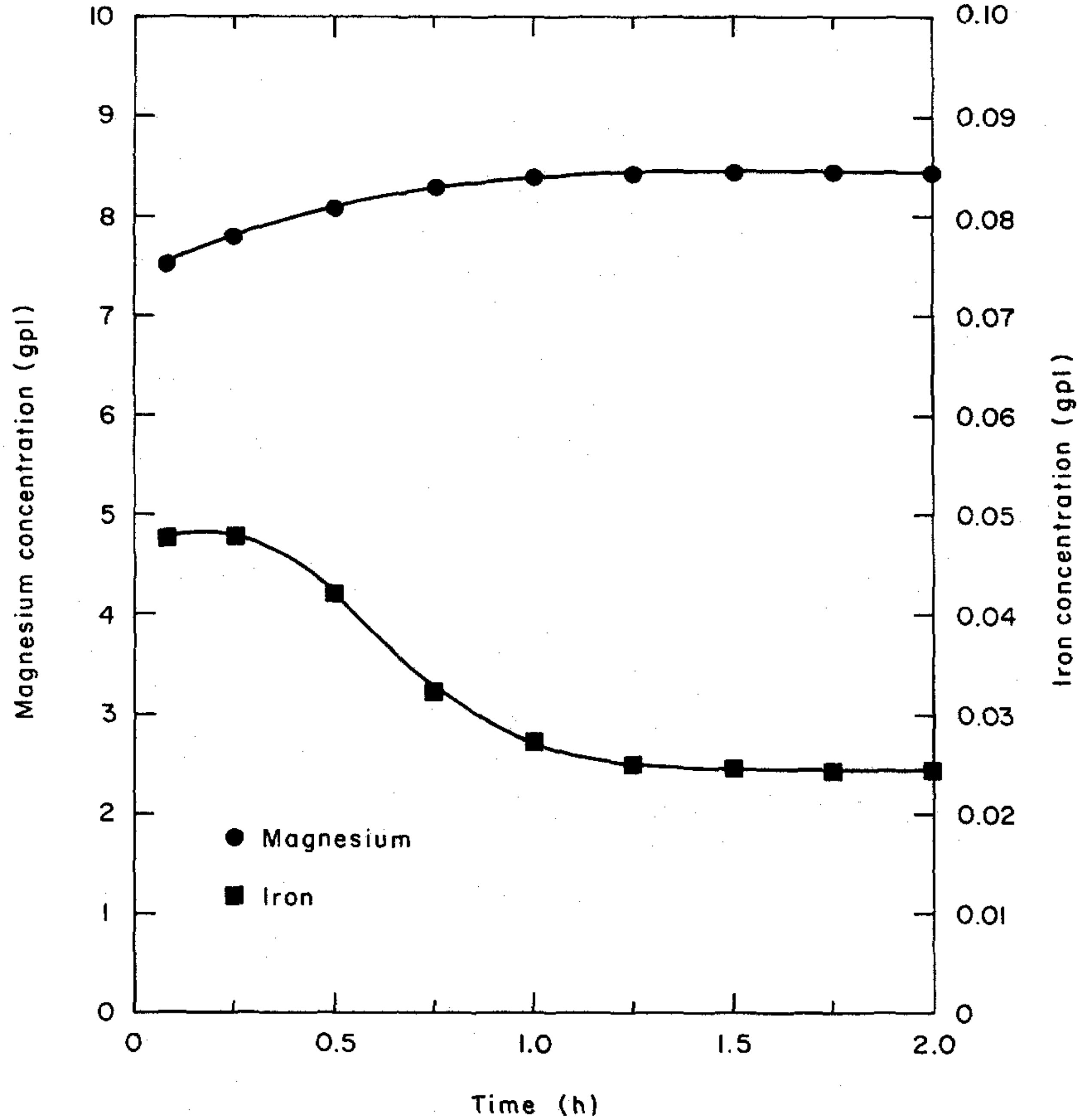

Fig. 15. Pilot-scale autoclave leach test data - PPL 7. See Table 9.

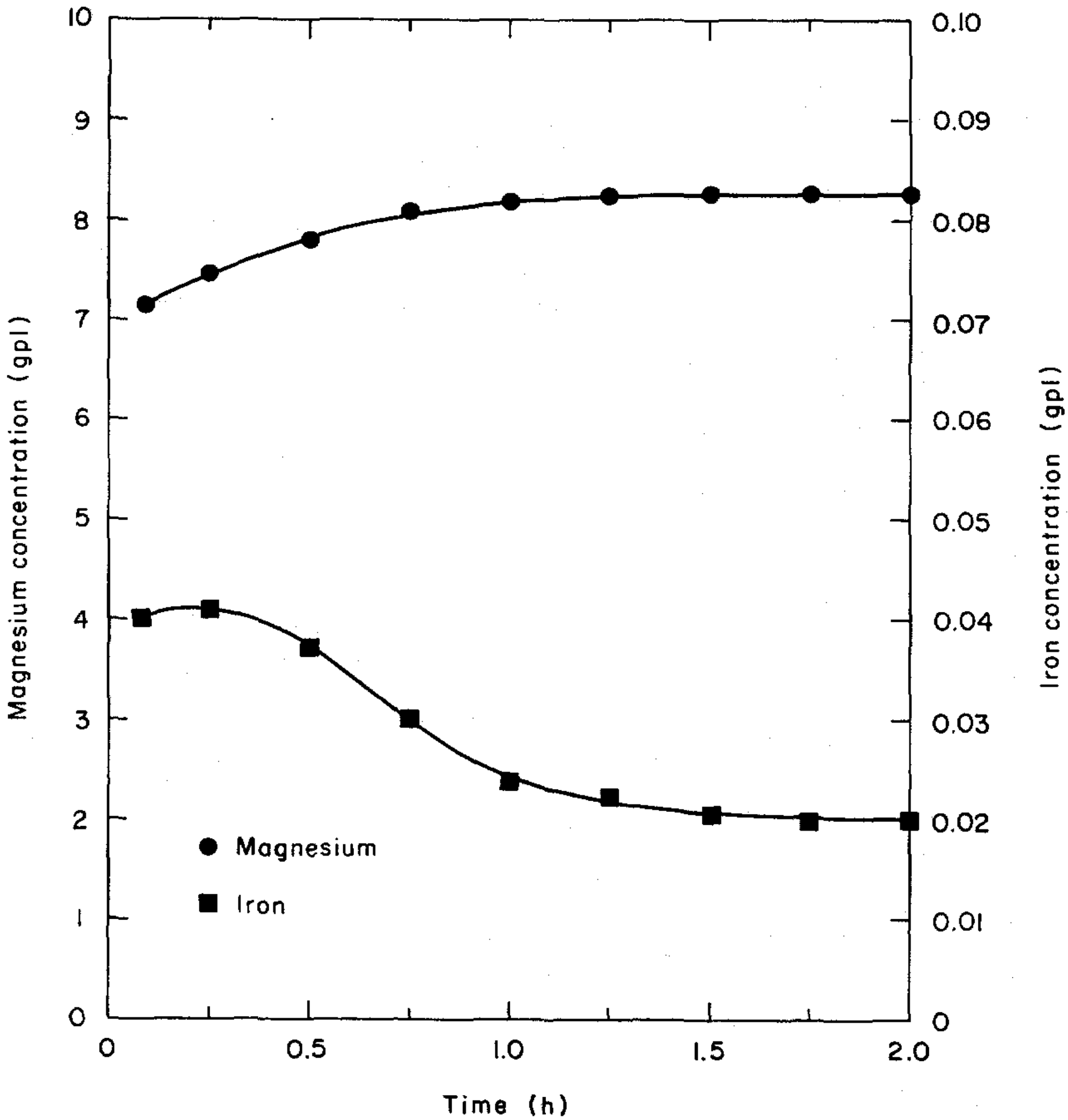

Fig. 16. Laboratory-scale autoclave leach test data - PPL 7. See Table 9.

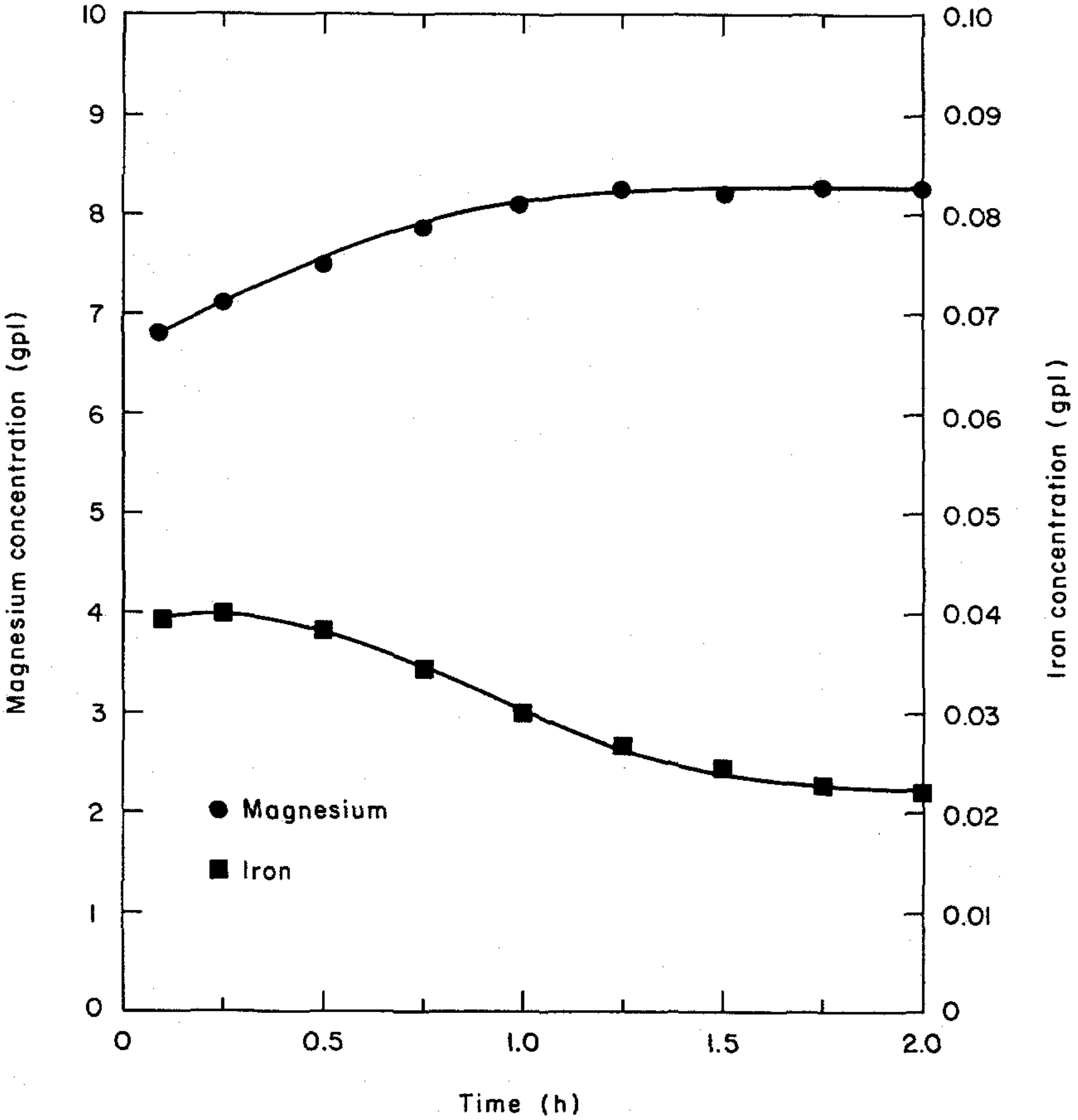

Fig. 17. Pilot-scale autoclave leach test data - PPL 8. See Table 10.

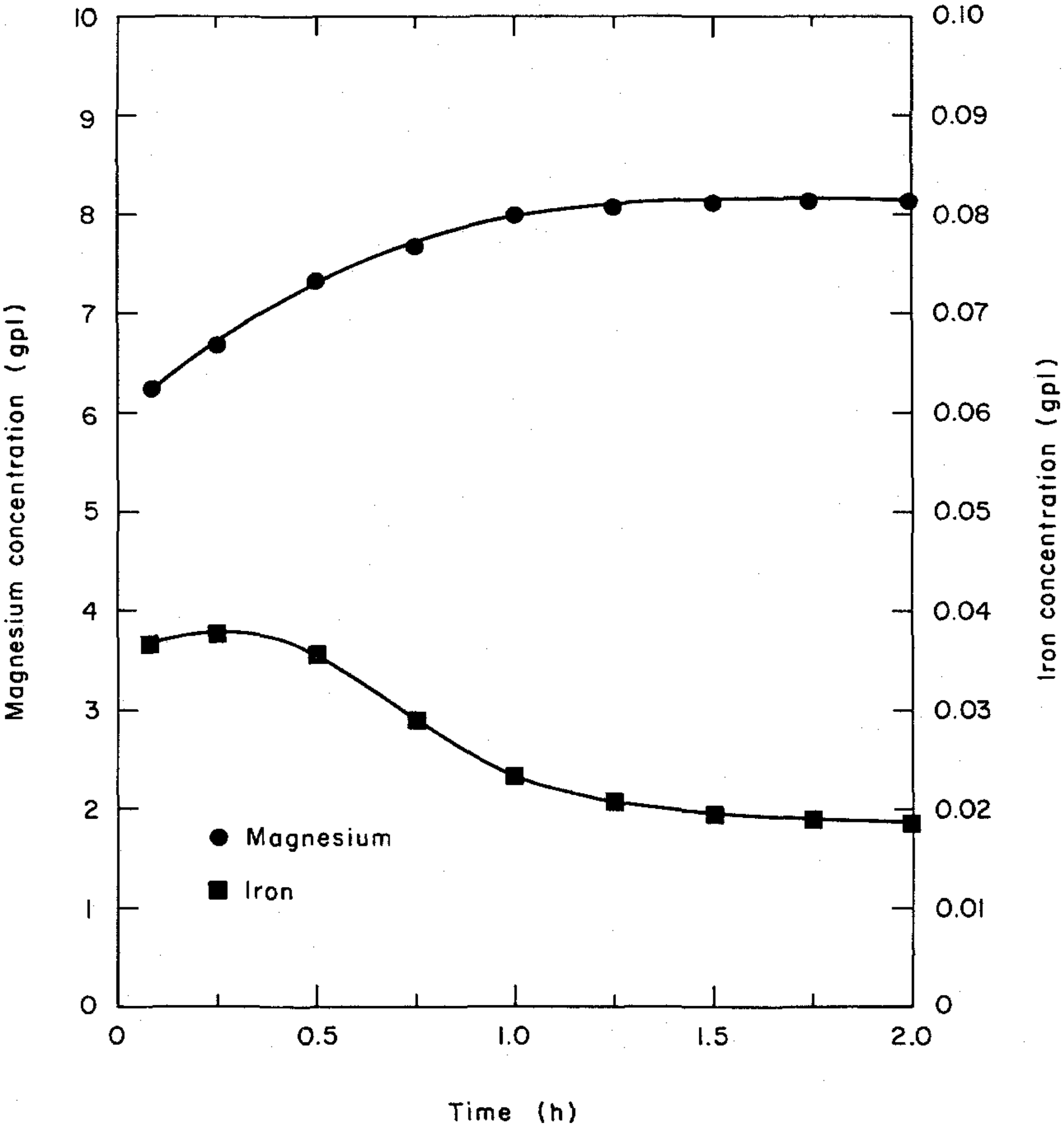

Fig. 18. Laboratory-scale autoclave leach test data - PPL 8. See Table 10.

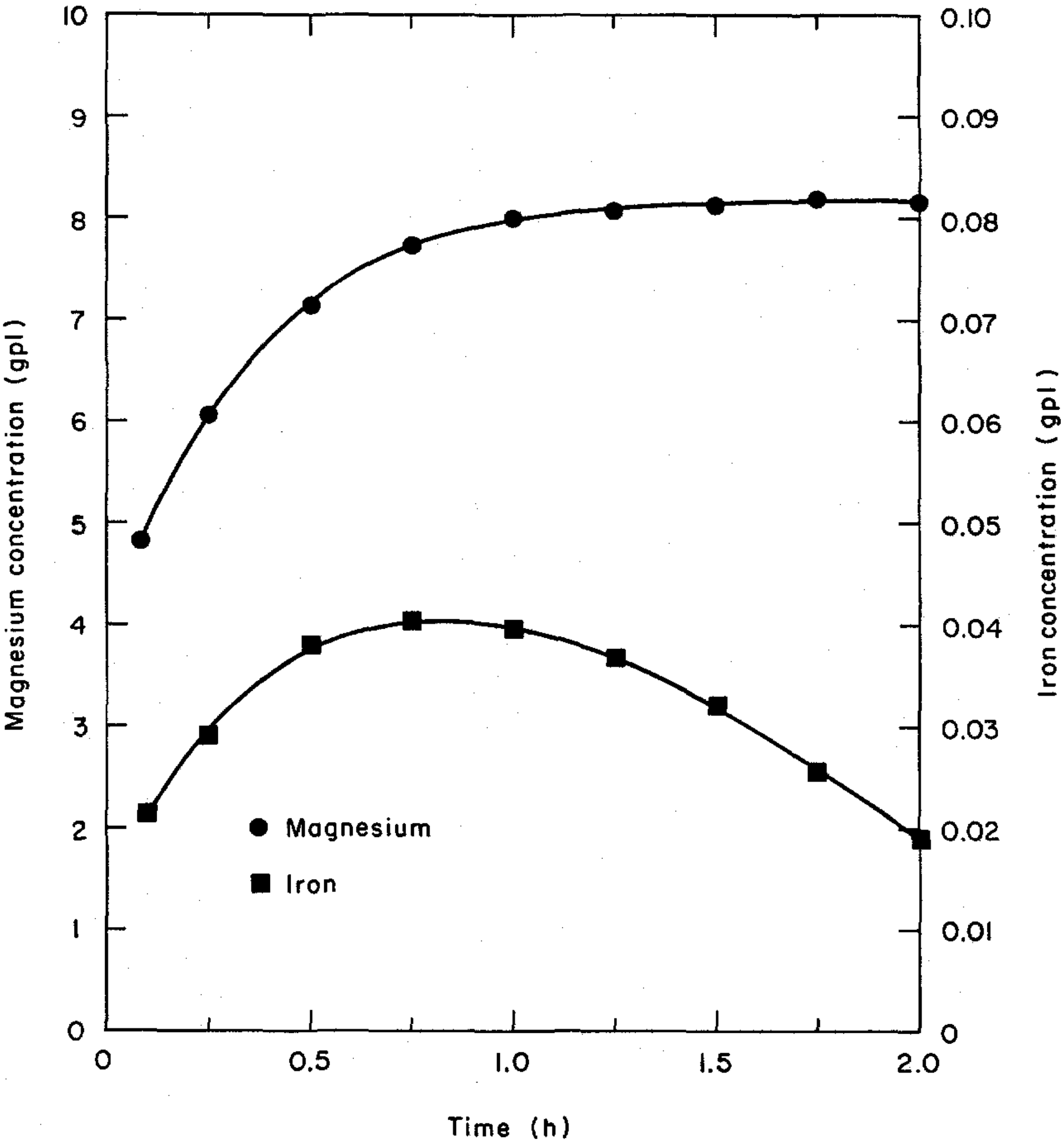

Fig. 19. Pilot-scale autoclave leach test data - PPL 9. See Table 11.

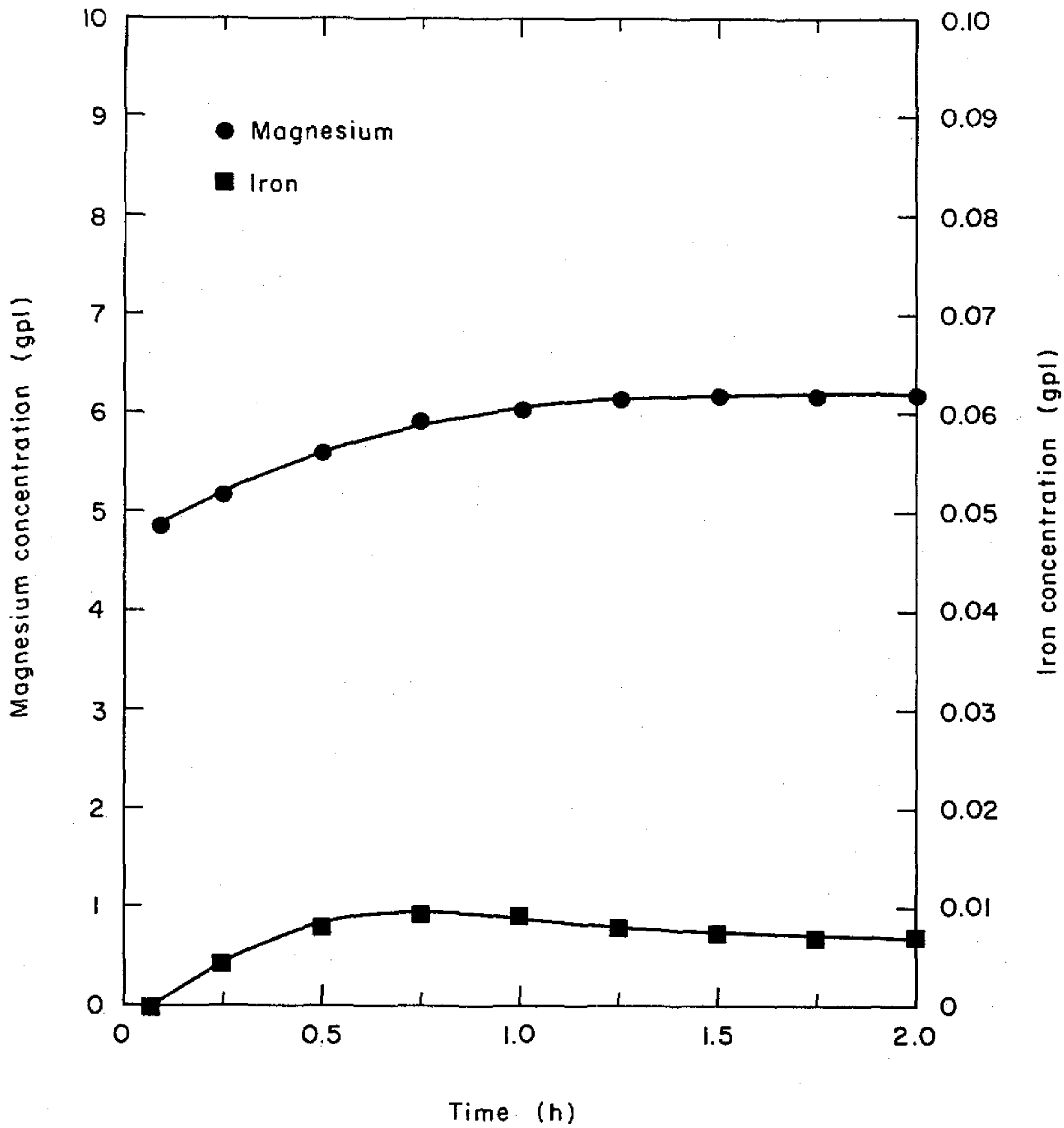

Fig. 20. Laboratory-scale autoclave leach test data - PPL 9. See Table 11.

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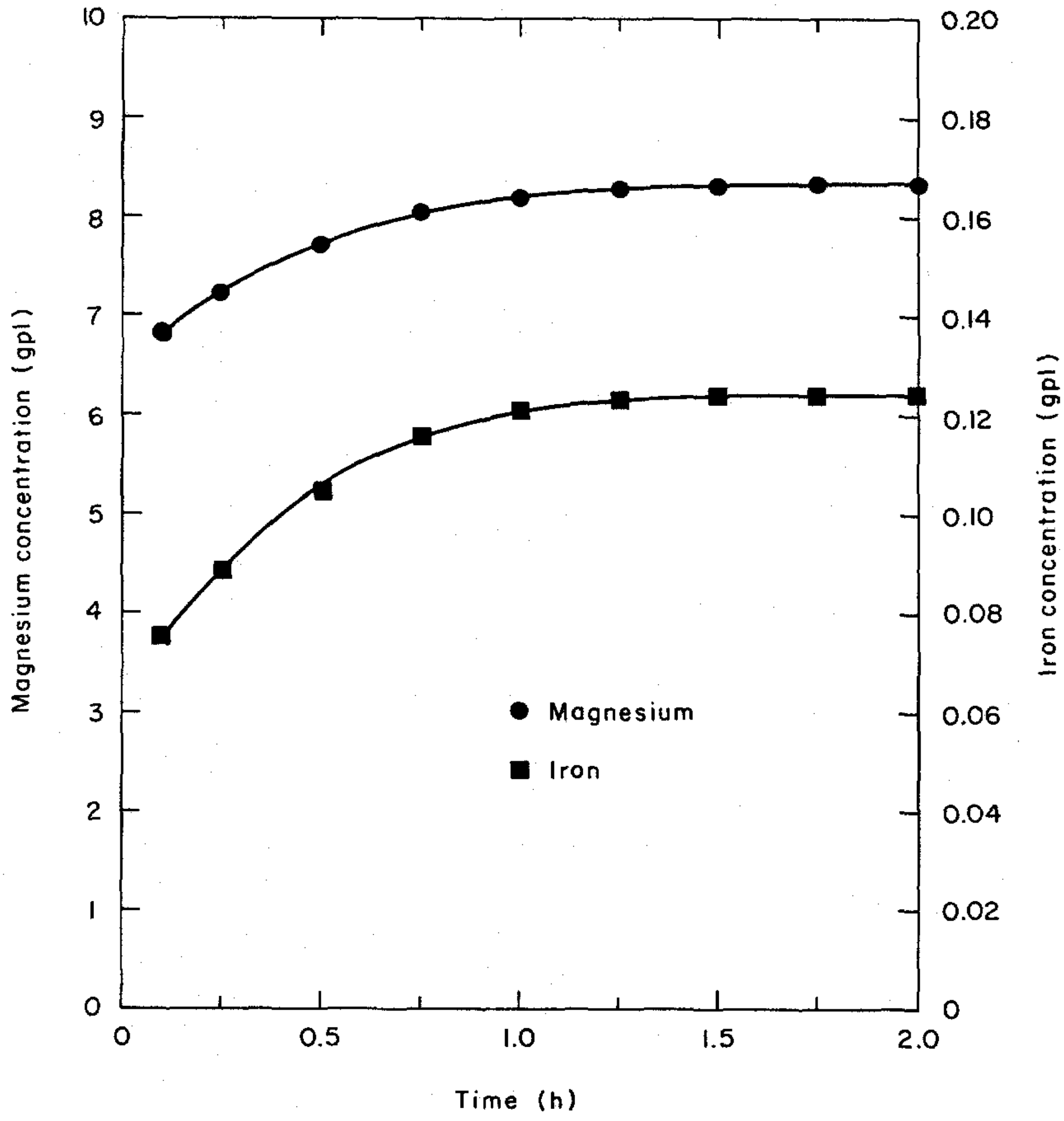

Fig. 21. Pilot-scale autoclave leach test data - PPL 10. See Table 12.

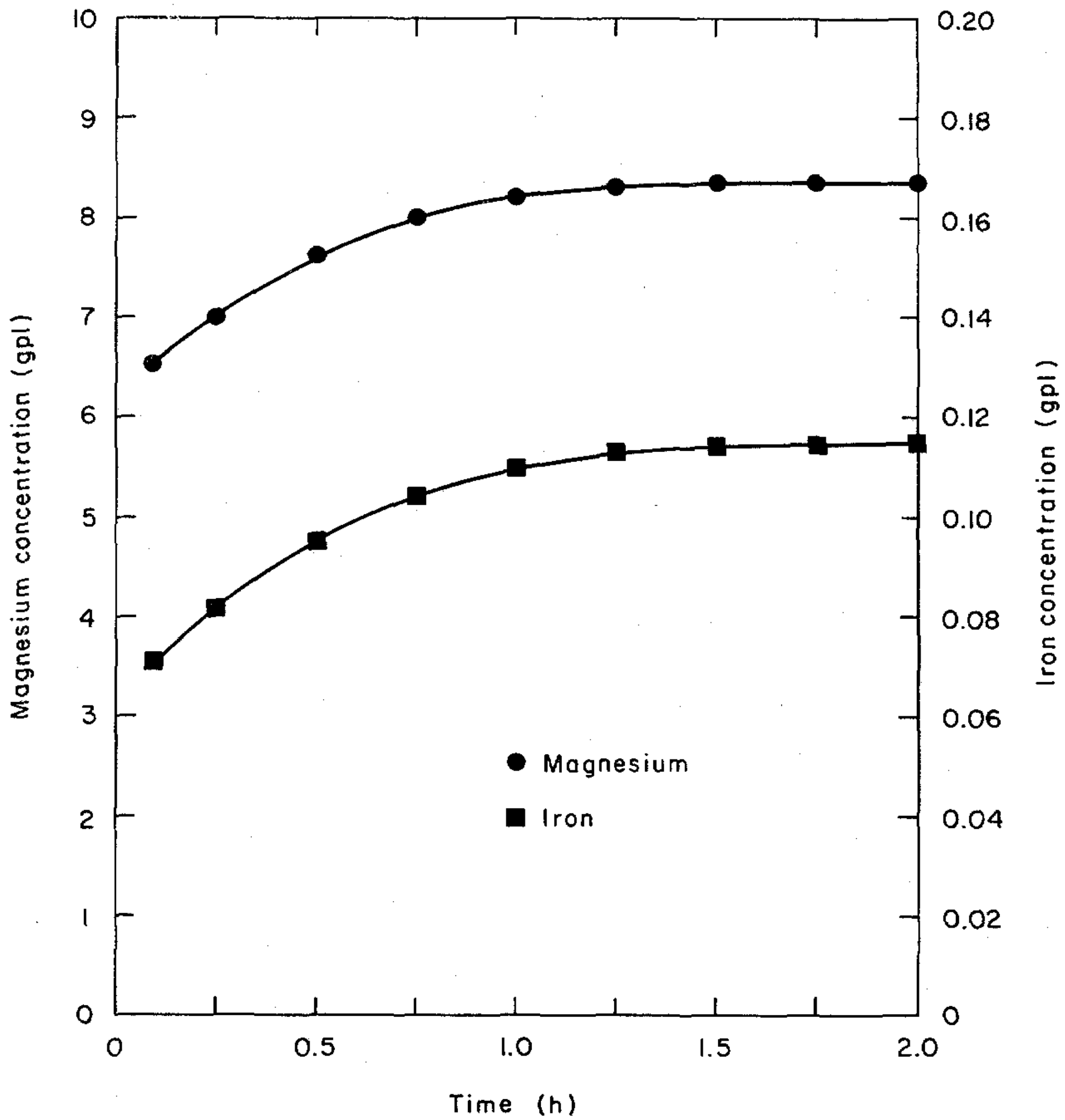

Fig. 22. Laboratory-scale autoclave leach test data - PPL 10. See Table 12.

583059

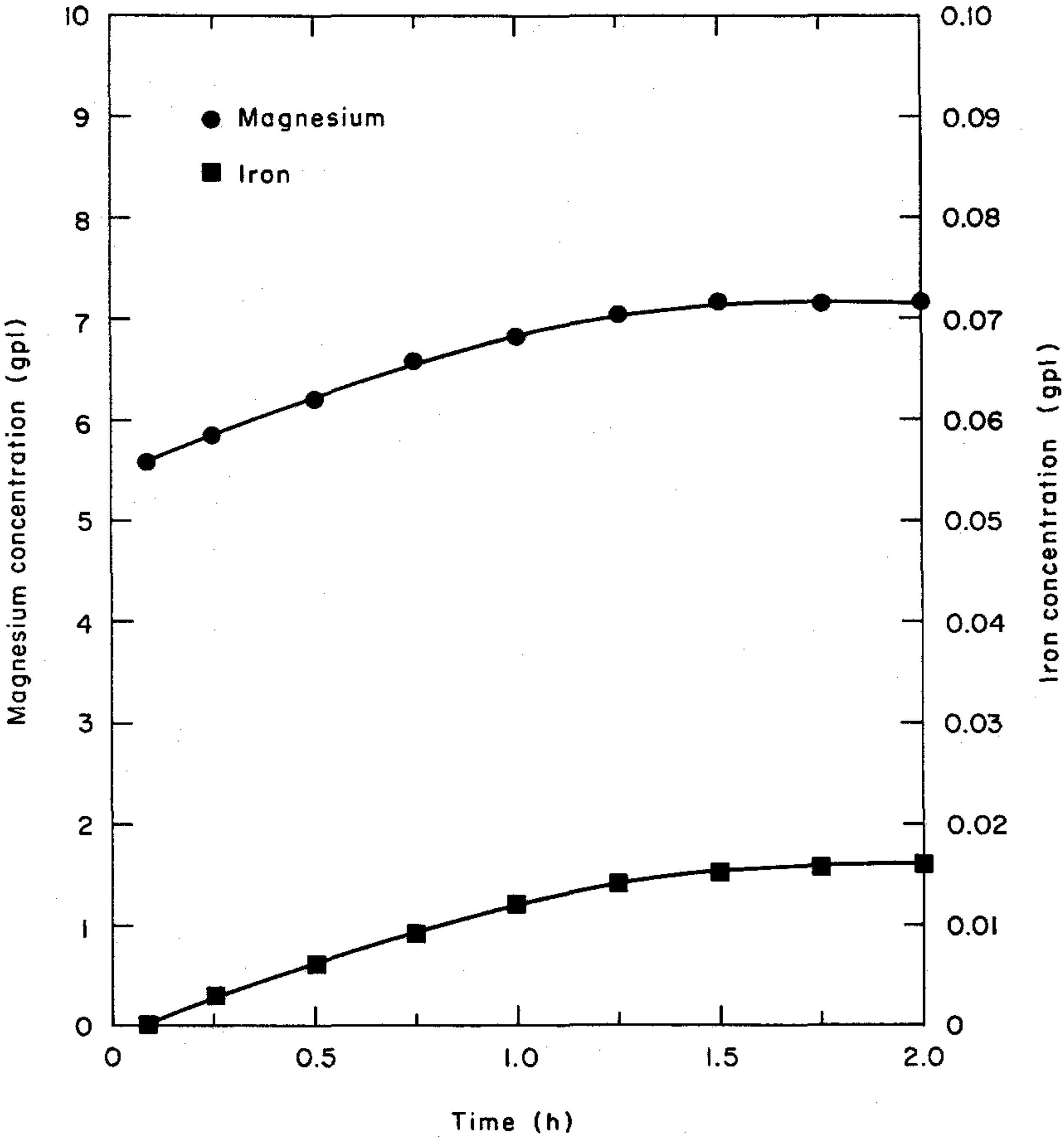

Fig. 23. Pilot-scale autoclave leach test data - PPL 11. See Table 13.

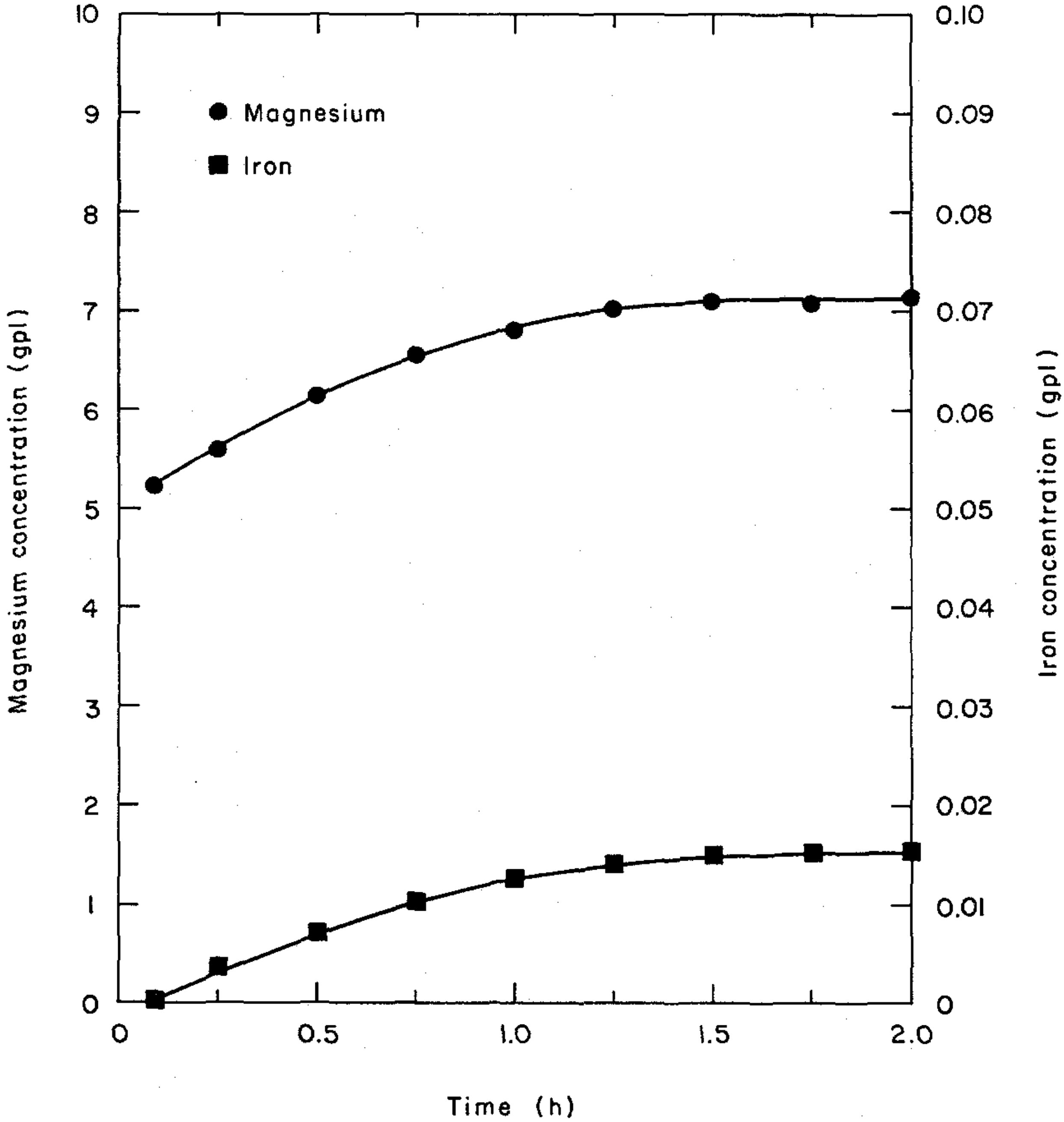

Fig. 24. Laboratory-scale autoclave leach test data - PPL 11. See Table 13.

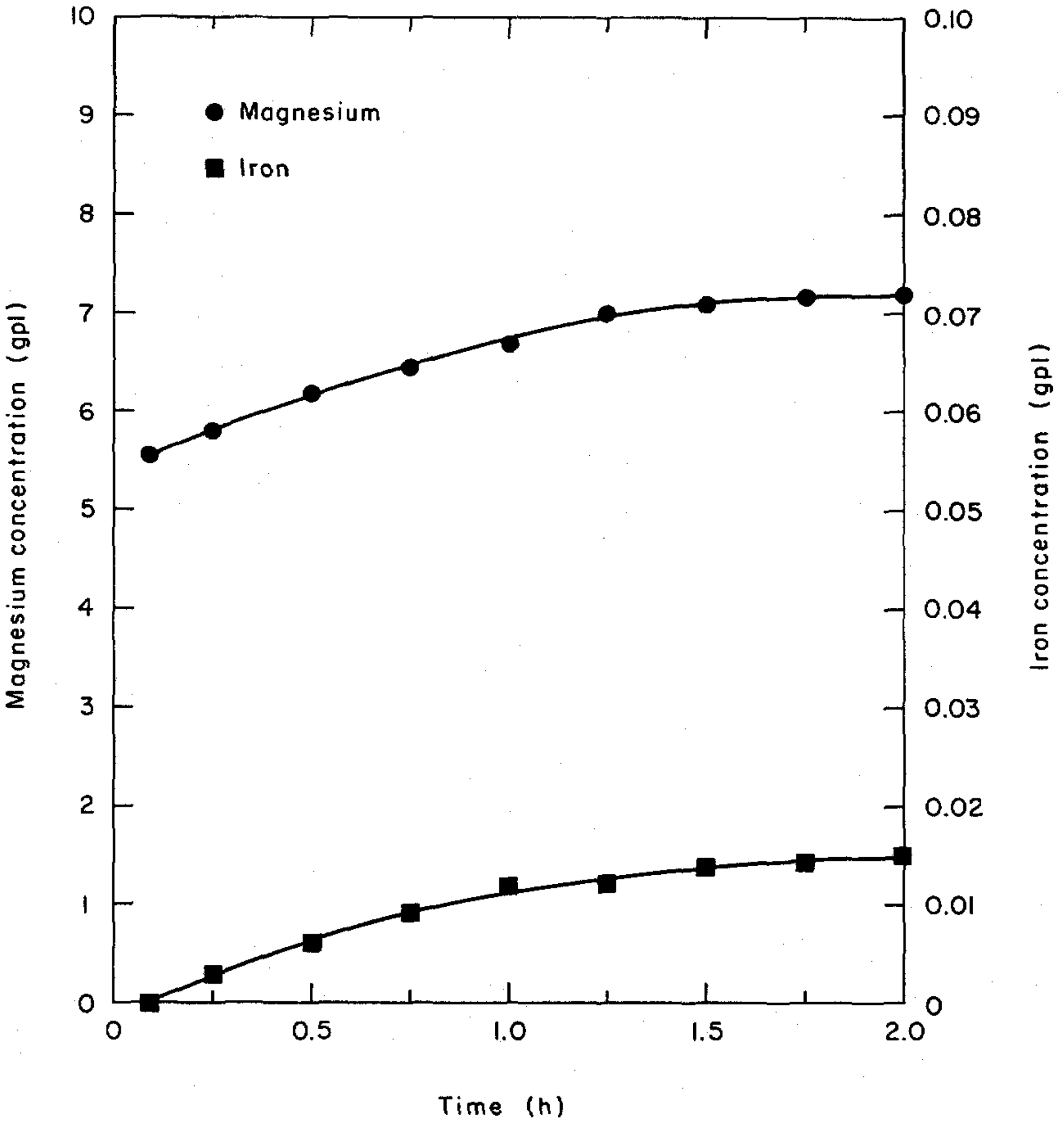

Fig. 25. Pilot-scale autoclave leach test data - PPL 12. See Table 14.

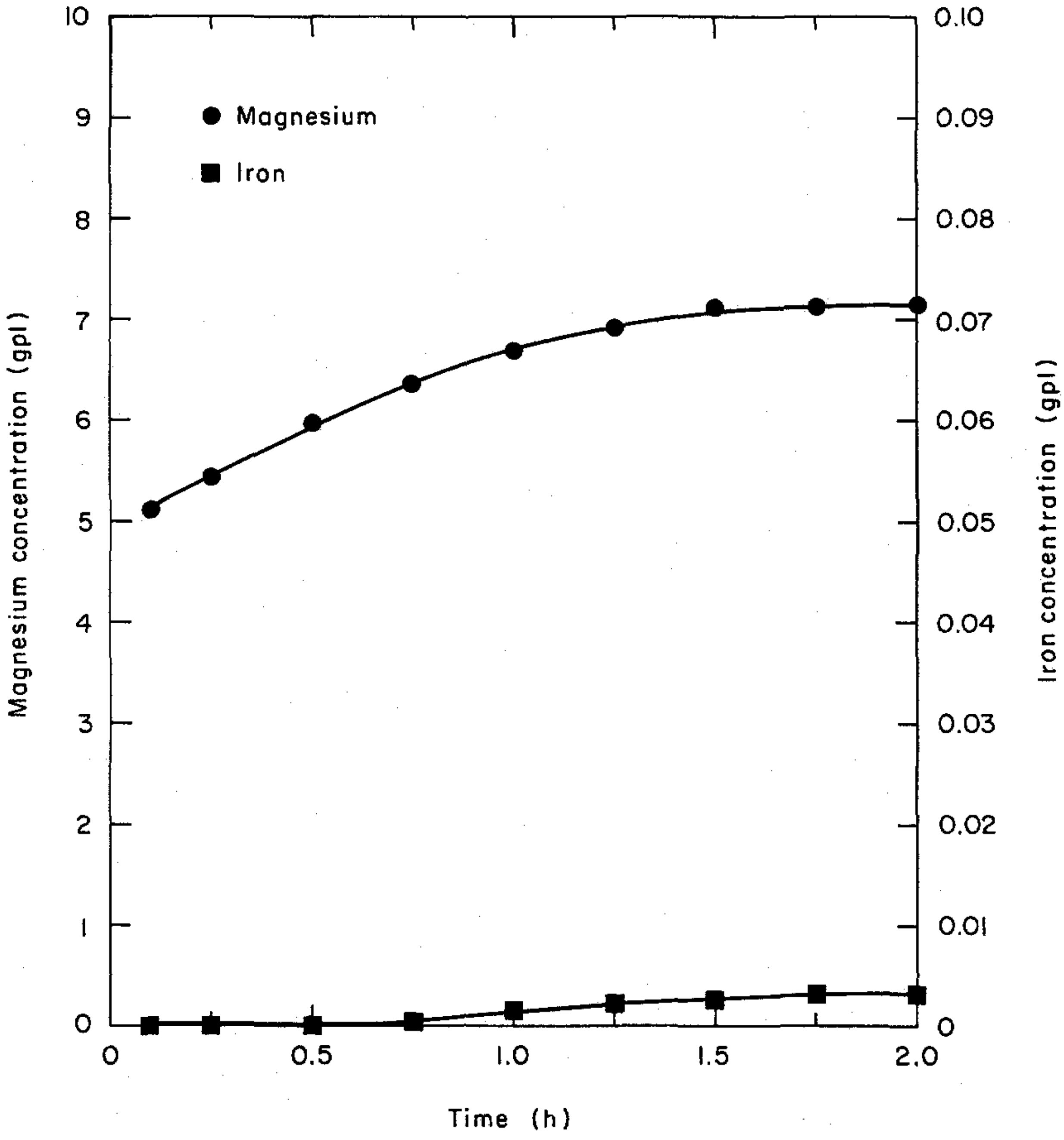

Fig. 26. Laboratory-scale autoclave leach test data - PPL 12. See Table 14.

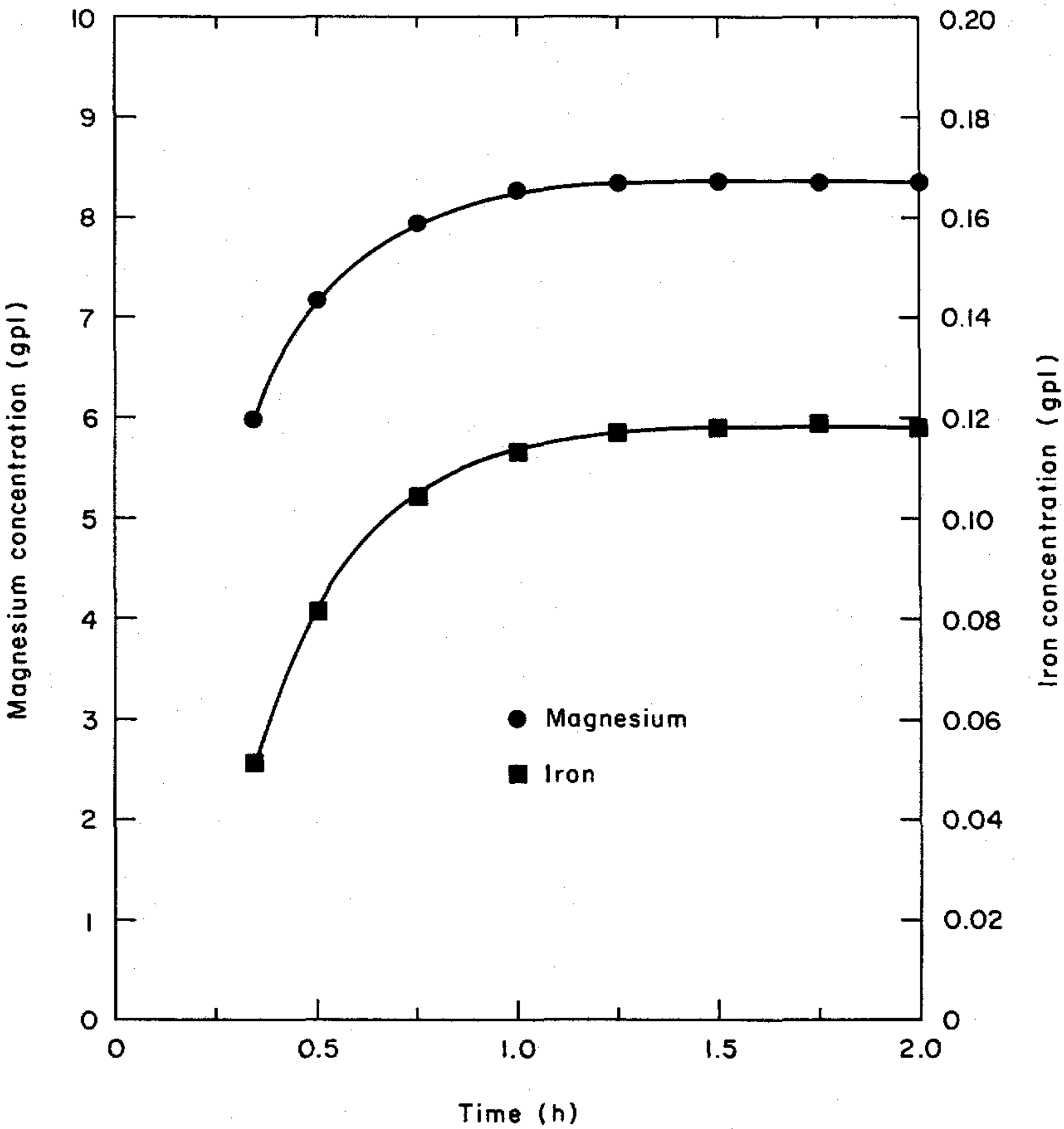

Fig. 27. Pilot-scale autoclave leach test data - PPL 13. See Table 15.

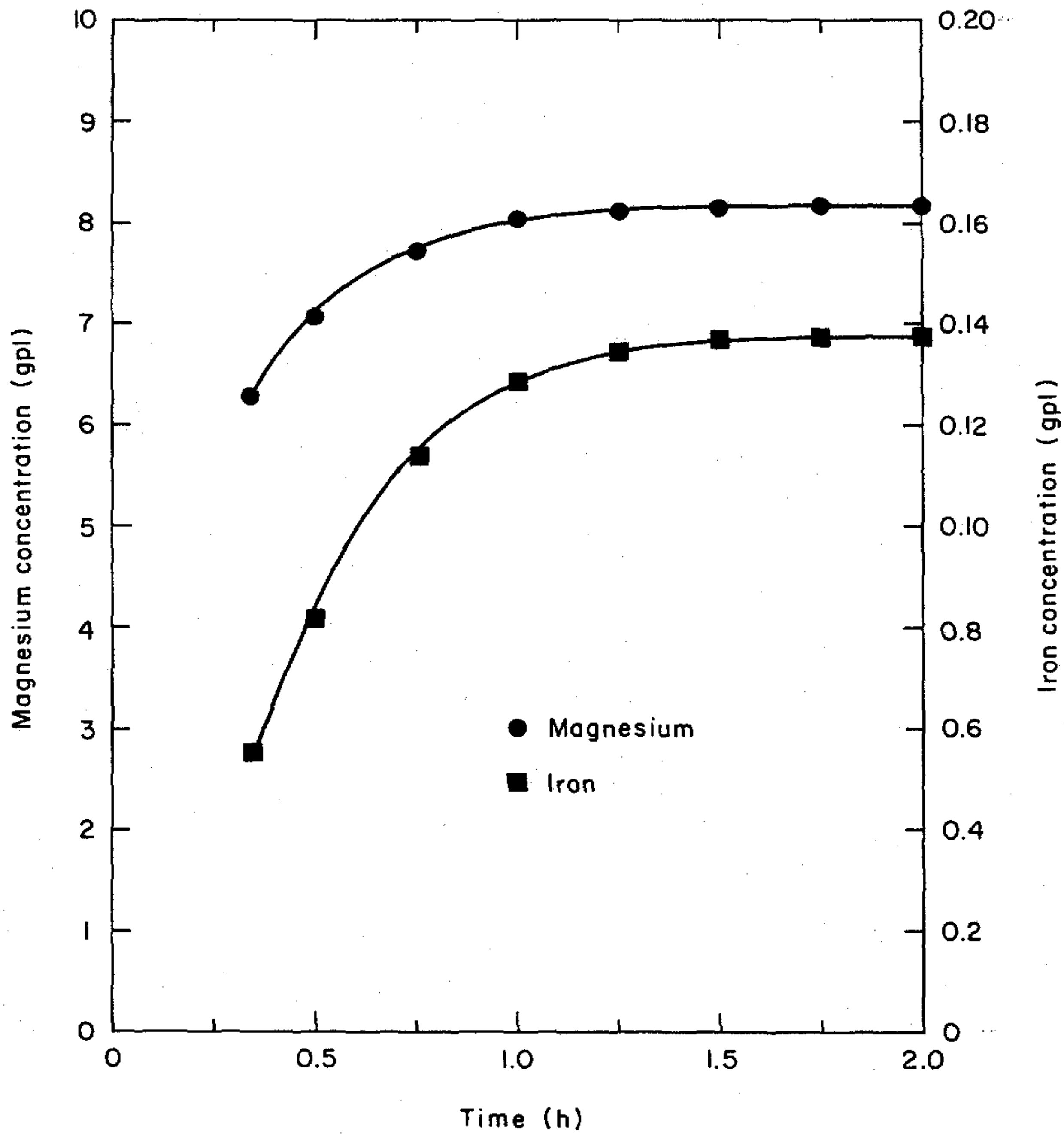

Fig. 28. Pilot-scale autoclave leach test data - PPL 14. See Table 16.

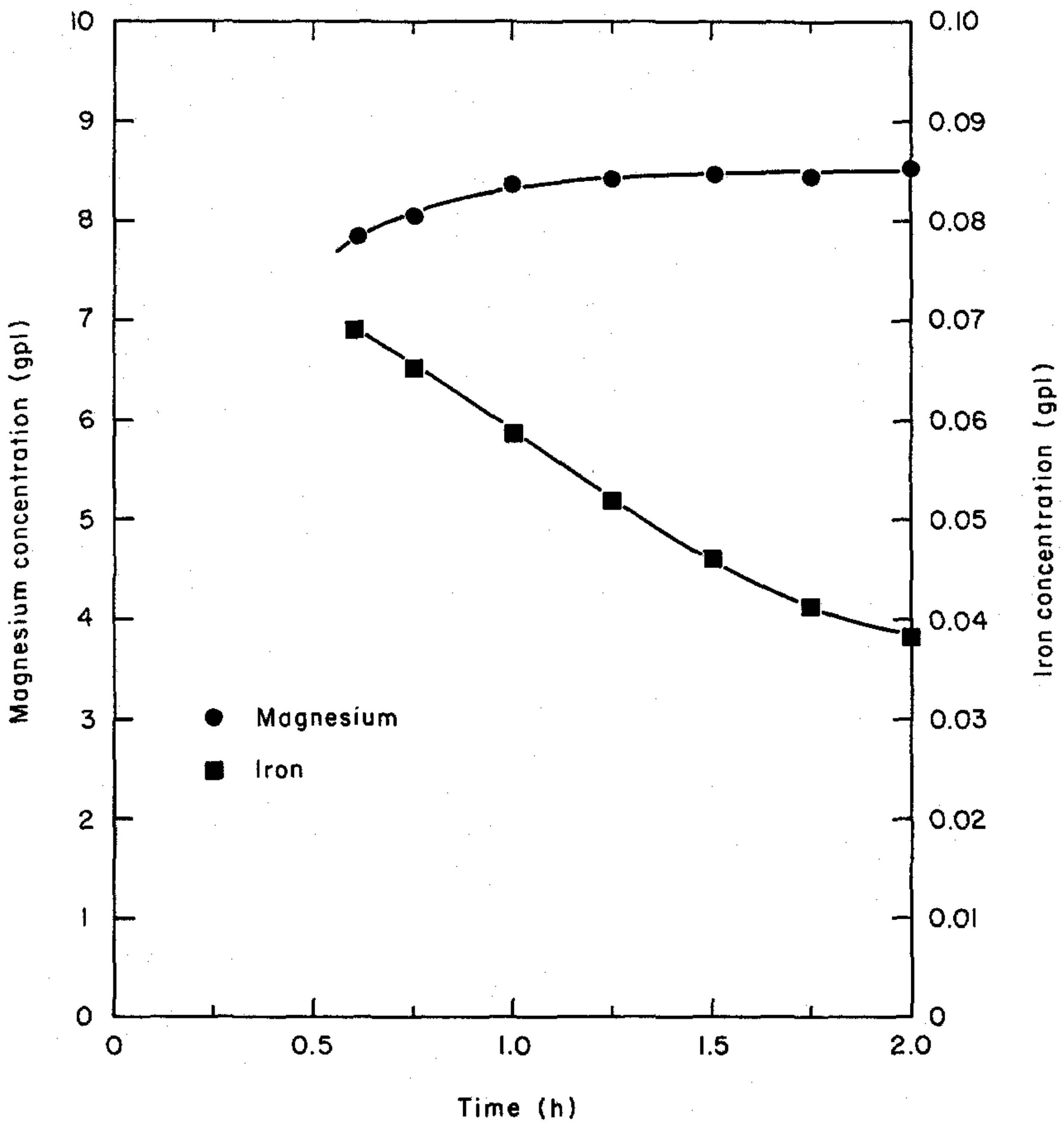

Fig. 29. Pilot-scale autoclave leach test data - PPL 15. See Table 17.

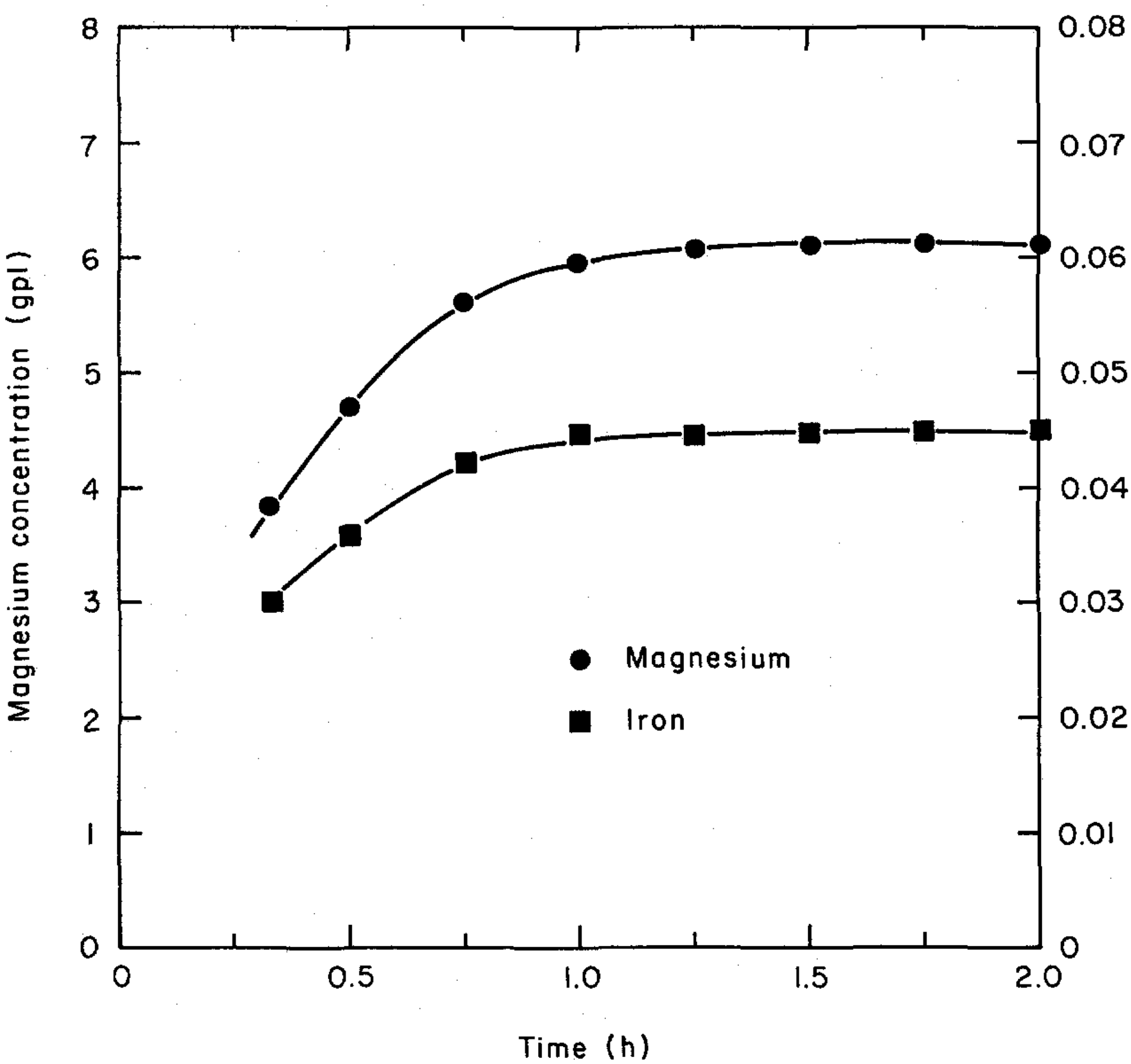

Fig. 30. Pilot-scale autoclave leach test data - PPL 16. See Table 18.

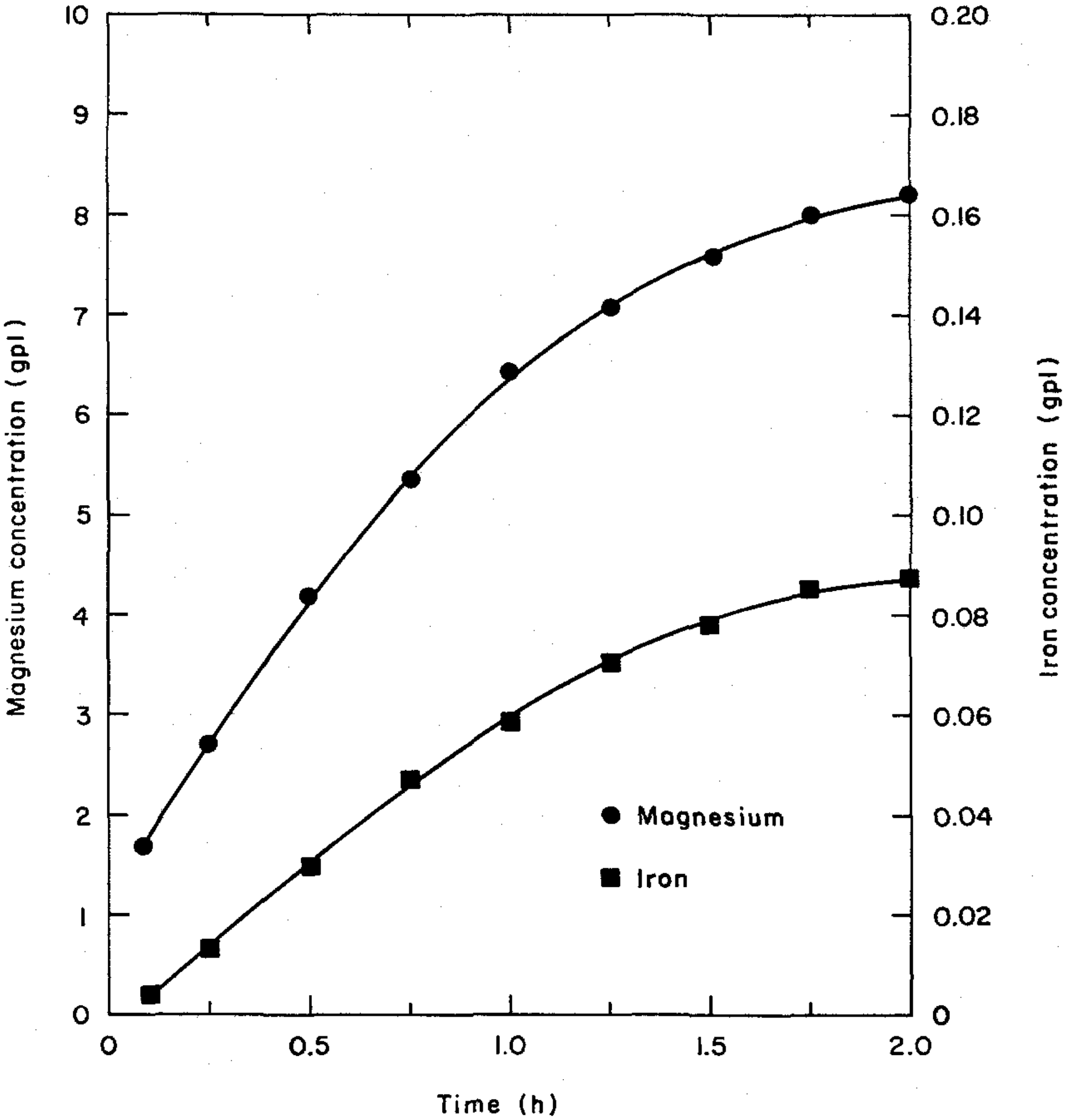

Fig. 31. Pilot-scale autoclave leach test data - PPL 17. See Table 19.

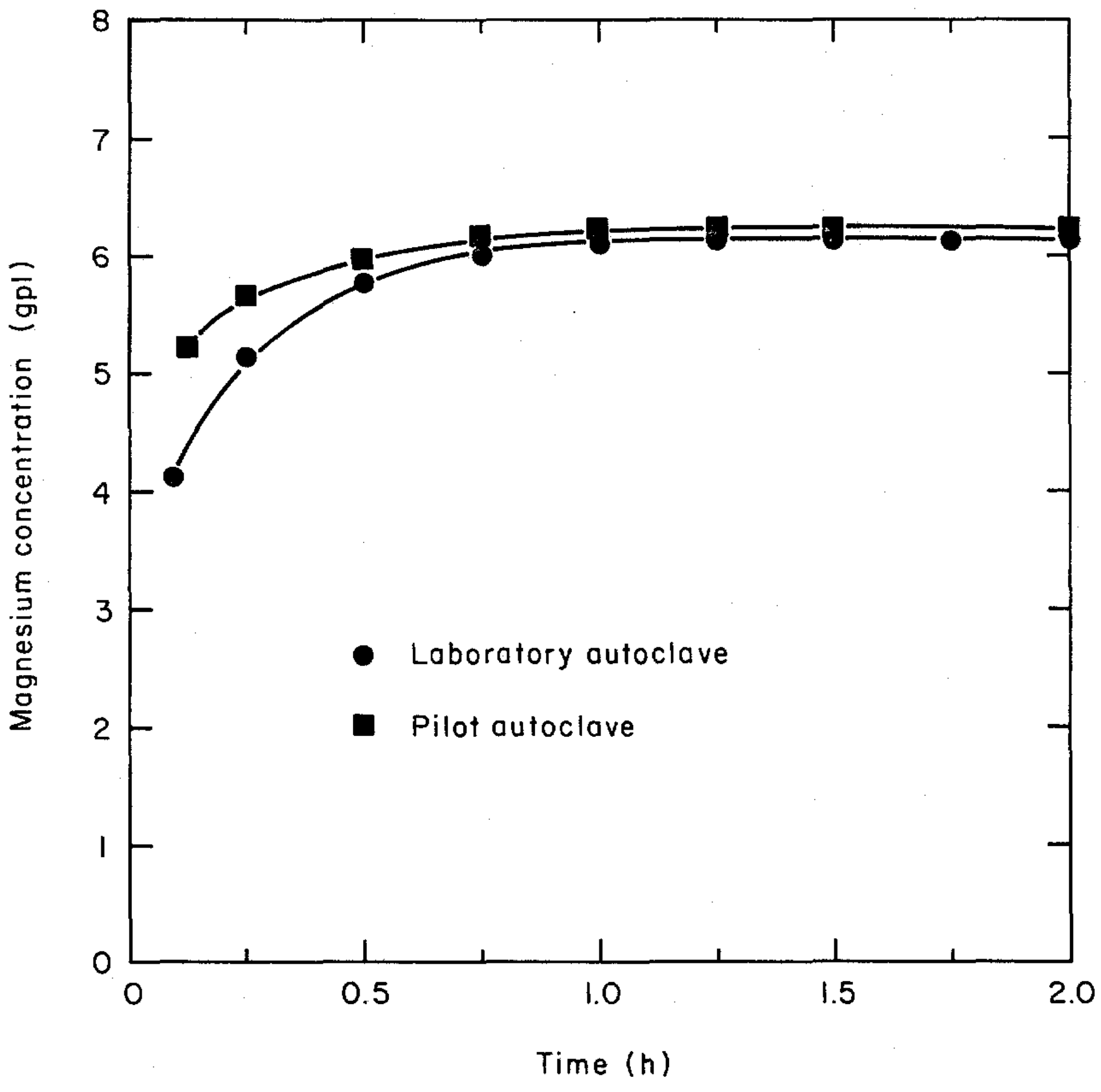

Fig. 32. Magnesium leaching kinetics - effect of autoclave type. Tests PPL 1 and PPL 16: Tables 3 and 18 respectively.

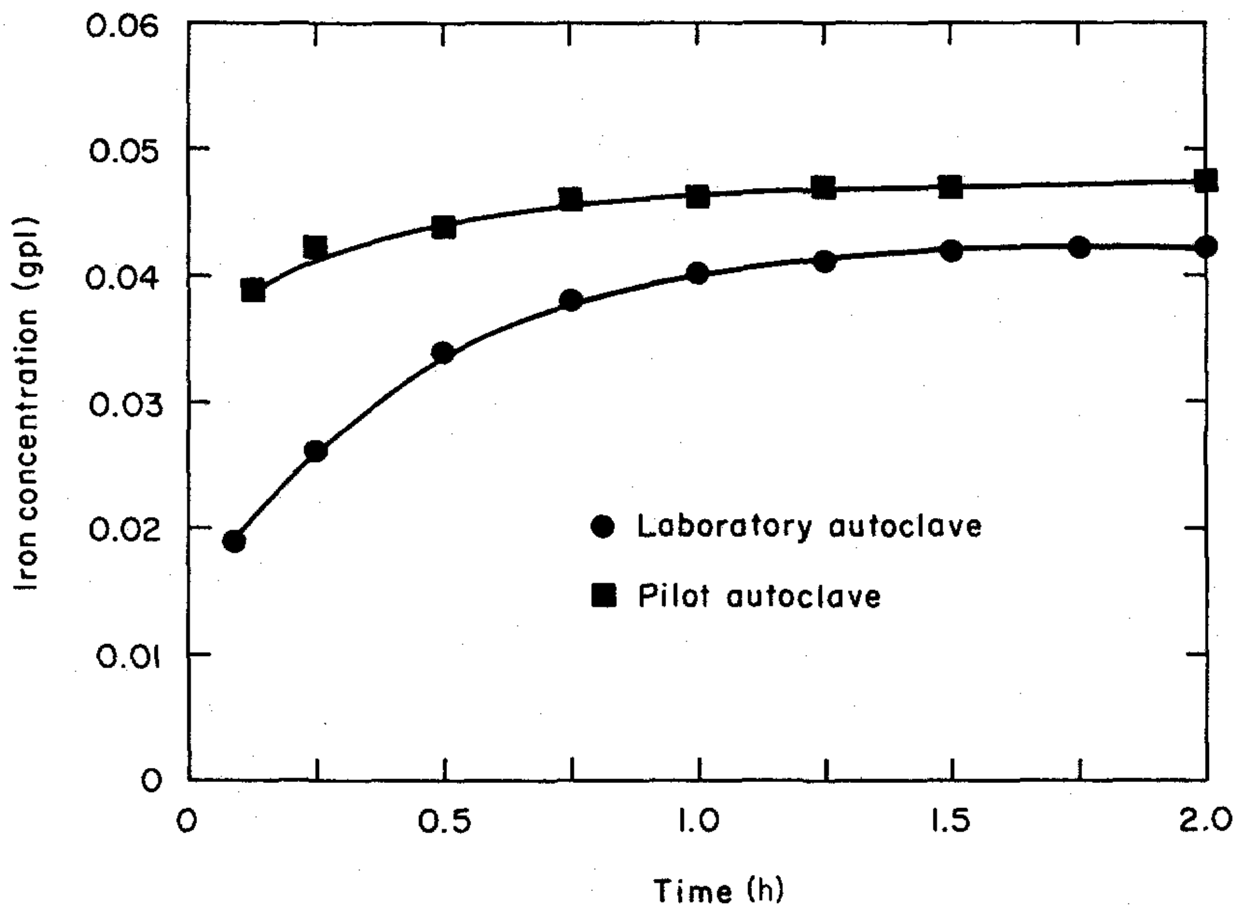

Fig. 33. Iron leaching kinetics - effect of autoclave type. Tests PPL 1 and PPL 16: Tables 3 and 18 respectively.

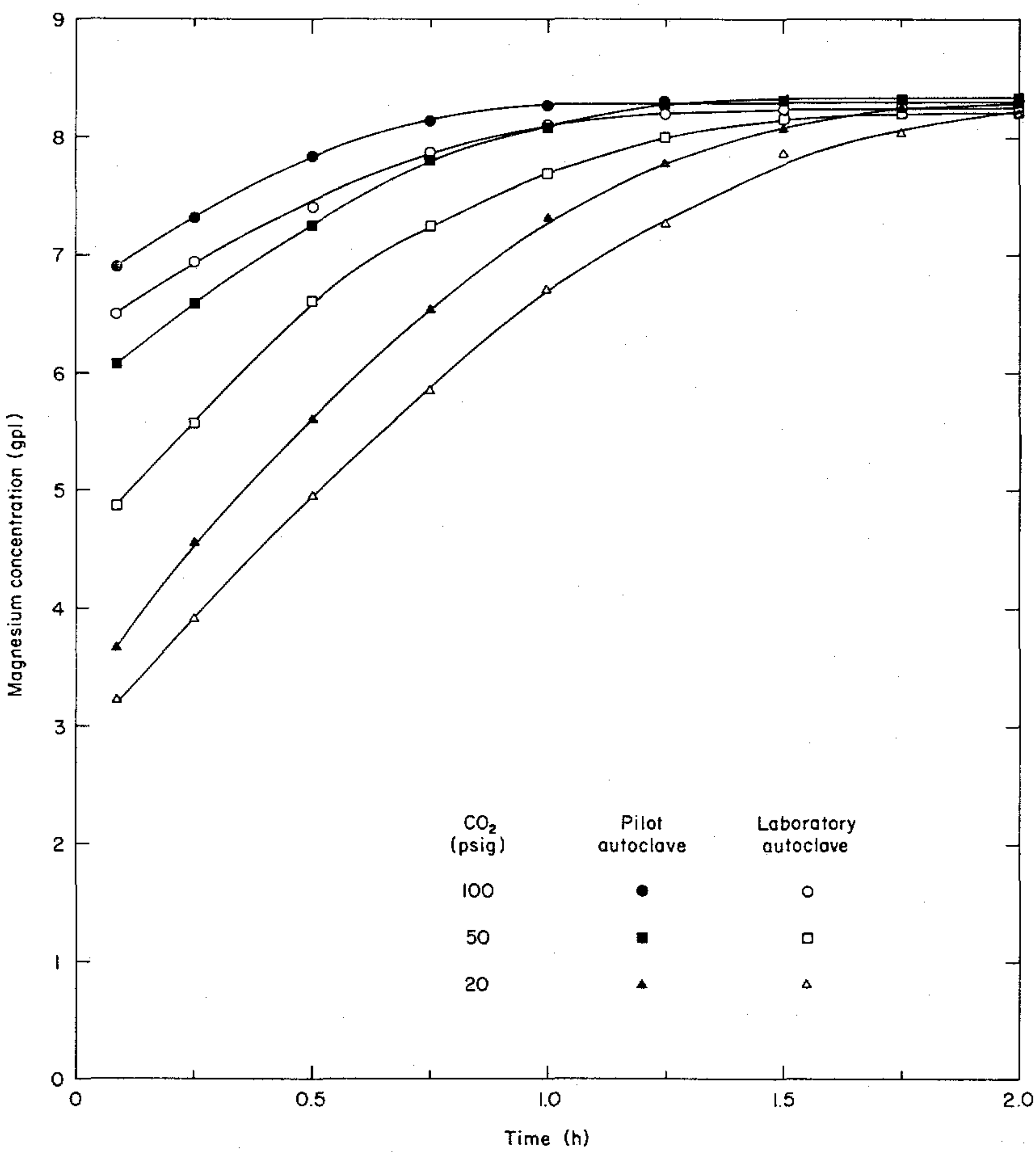

Fig. 34. Magnesium leaching kinetics - effect of operating pressure (all tests at 15.5°C).

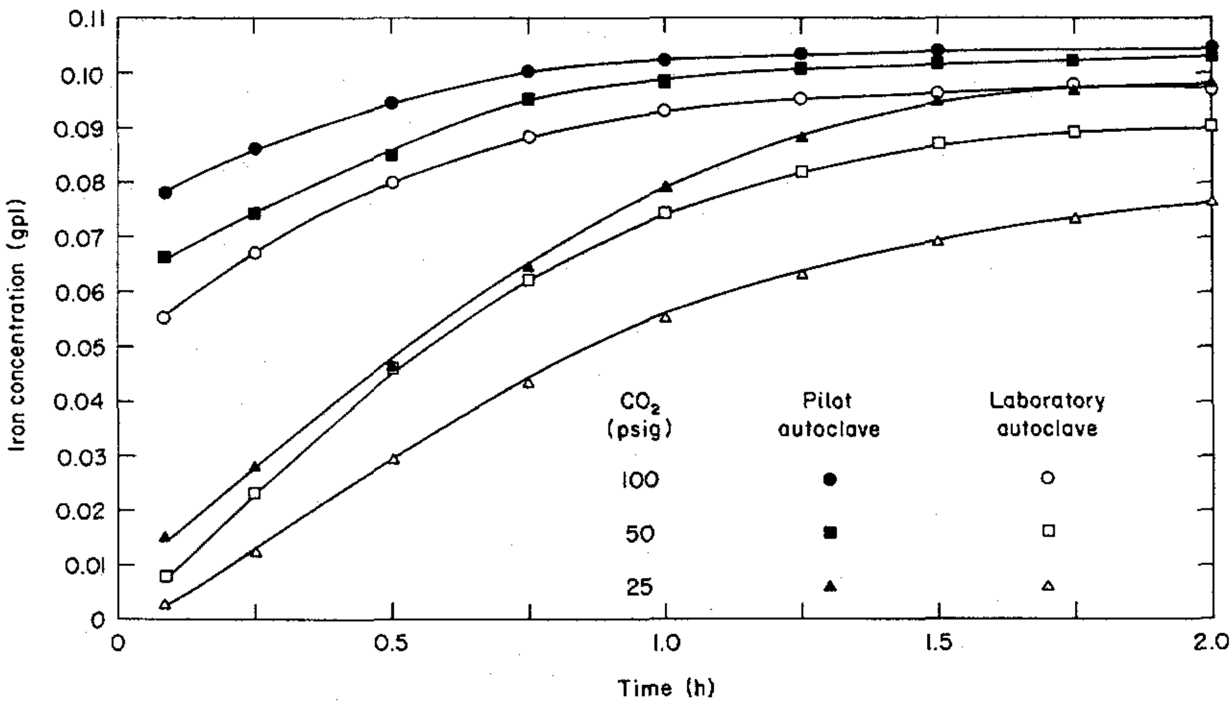

Fig. 35. Iron leaching kinetics - effect of operating pressure (all tests at 15.5°C).

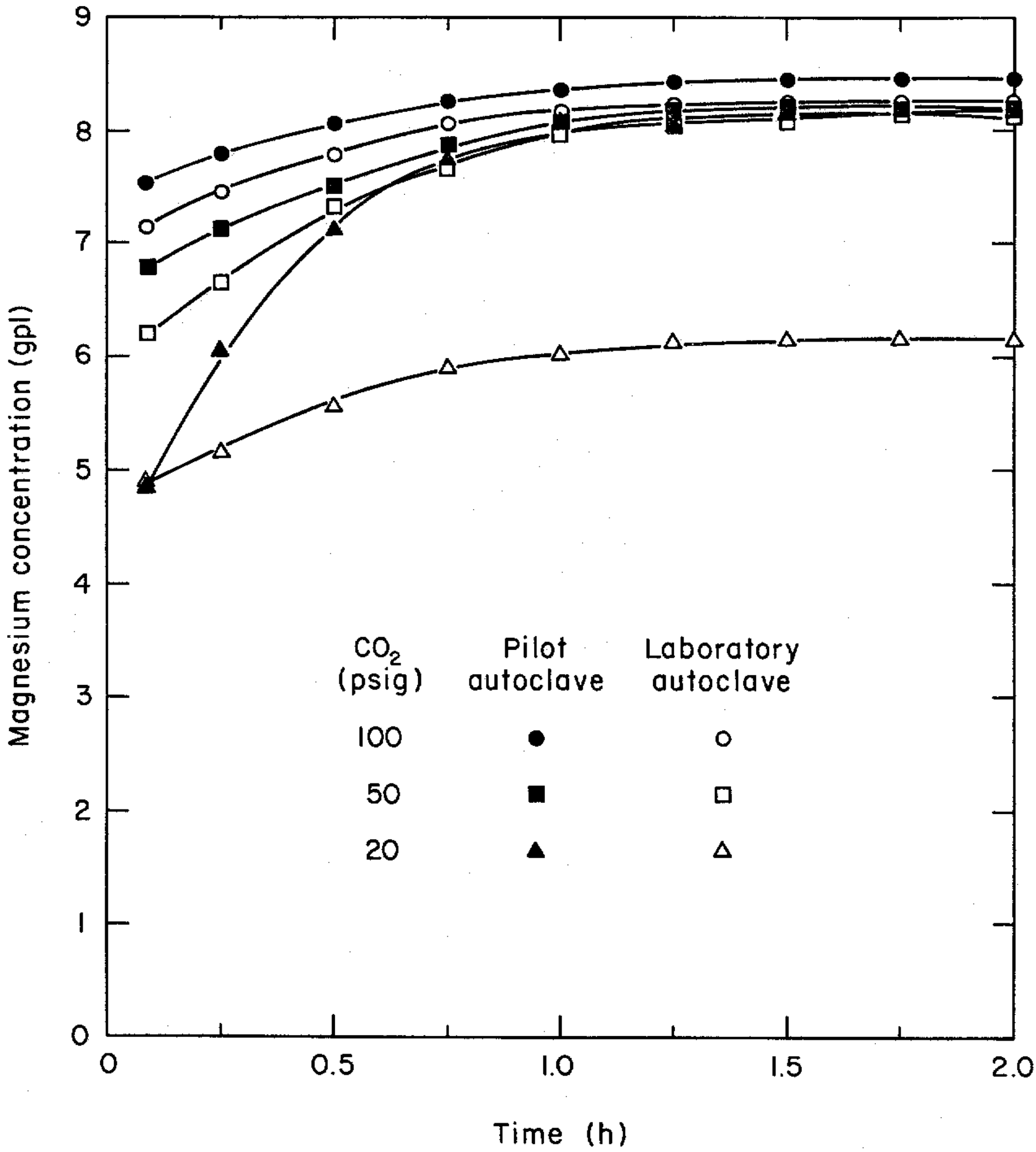

Fig. 36. Magnesium leaching kinetics - effect of operating pressure (all tests at 35°C).

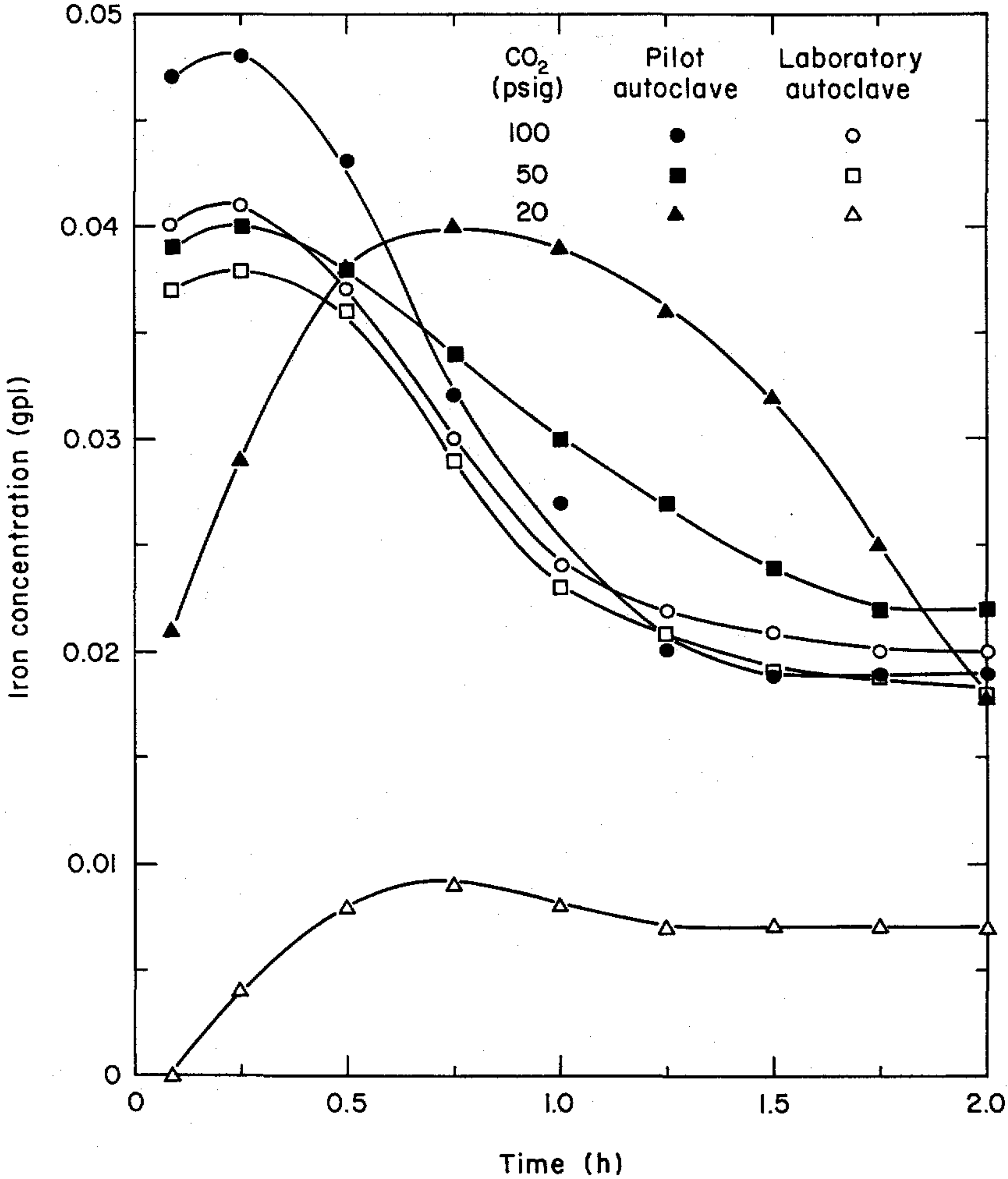

Fig. 37. Iron leaching kinetics - effect of operating pressure (all tests at 35°C).

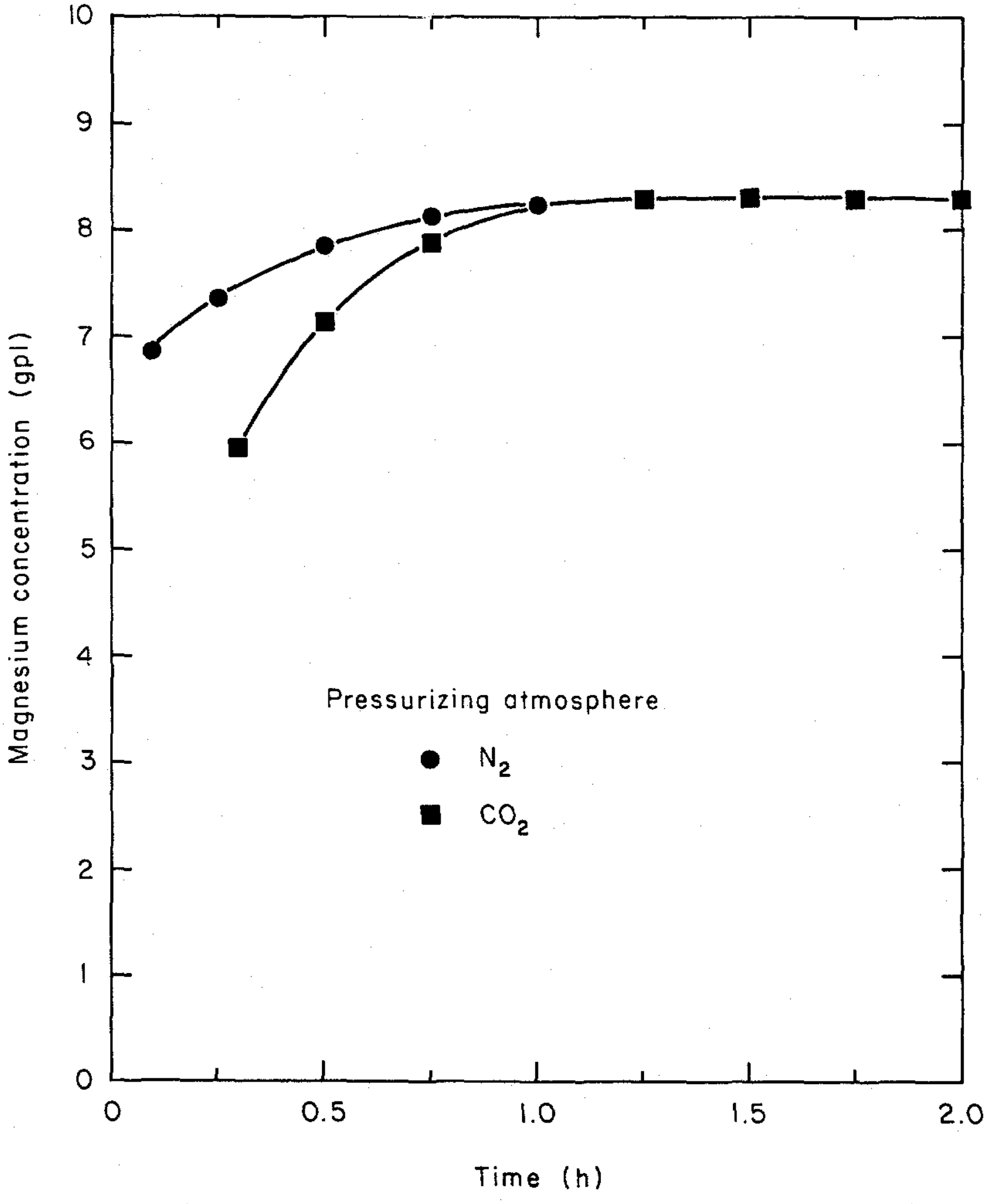

Fig. 38. Magnesium leaching kinetics - effect of pressurizing atmosphere. Tests PPL 2 and PPL 13: Tables 4 and 15 respectively.

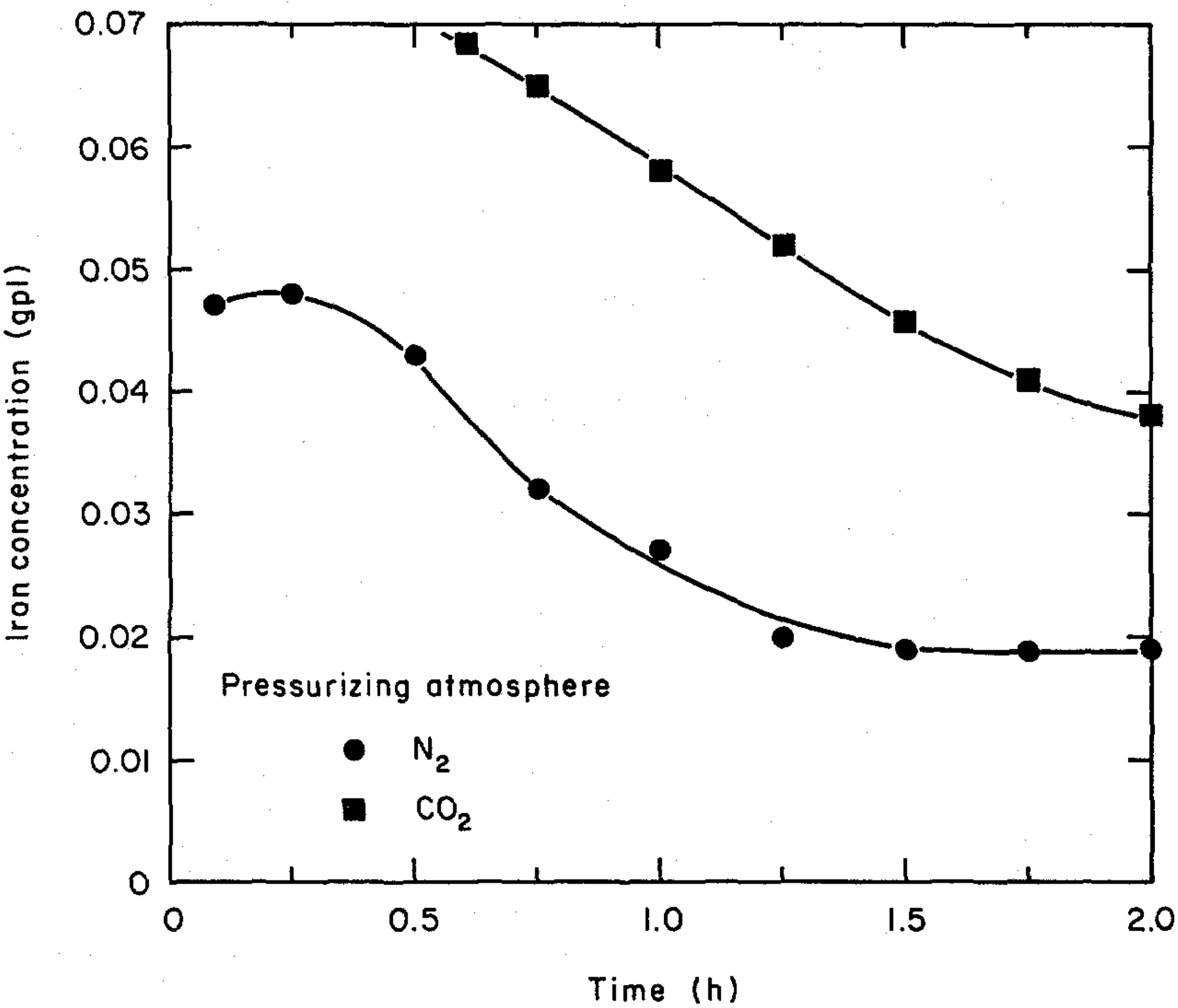

Fig. 39. Iron leaching kinetics - effect of pressurizing atmosphere. Tests PPL 9 and PPL 15: Tables 11 and 17 respectively.