

# The Metsep Process for the separation and recovery of zinc, iron, and hydrochloric acid from spent pickle liquors

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## SYNOPSIS

A spray roaster has been installed in the Johannesburg area for the treatment of spent liquors from a variety of small pickling operations. A new process was developed for the processing of the liquor that is contaminated with zinc chloride, and a commercial plant was built incorporating this process.

An ion-exchange plant separates the zinc from the iron chloride, and a liquid-liquid-extraction plant converts the zinc chloride to zinc sulphate and recovers the hydrochloric acid.

## SAMEVATTING

Daar is 'n spuitrooster in die Johannesburgse gebied geïnstalleer vir die behandeling van gebruikte vloeistowwe afkomstig van verskeie klein bytondernemings. Daar is 'n nuwe proses ontwikkel vir die verwerking van die vloeistof wat met sinkchloried gekontamineer is en daar is 'n kommersiële aanleg gebou wat hierdie proses insluit.

'n loonuitruilaanleg skei die sink van die ysterchloried en 'n vloeistof-vloeistofekstraksieaanleg sit die sinkchloried om in sinksulfaat en herwin die soutsuur.

## INTRODUCTION

The regeneration of hydrochloric acid from spent pickle liquors is now well established, and processes such as those using the Woodall-Duckham Spray Roaster are well known. Unfortunately, such processes can tolerate only minor contamination with zinc chloride, which, at the reactor temperatures used in commercial spray roasters, generally volatilizes without hydrolysing.

In South Africa, the spent pickle liquor from galvanizing operations usually contains appreciable concentrations of zinc chloride — up to 100 g/l. The Metsep Process was therefore developed to handle such contaminated liquors, which represent approximately half of the spent liquor available for treatment.

The process has been described previously<sup>1</sup>, but at that time was still in the development stage. This paper reports on the process after it had been in full-scale operation for more than a year.

A novel feature of the operation is that, for the first time in South Africa, a central plant has been established for the treatment of such waste liquors. This was made possible as the result of a remarkable degree of co-operation: a contract is

drawn up with each pickling organization by which the spent pickle liquor is collected, and recovered hydrochloric acid is supplied in return. The regeneration plant operates as a central antipollution measure in that the recovery of acid in this way is completely economic, whereas the erection of individual plants at each galvanizing or pickling works would have been far too costly for individual operators.

## DEVELOPMENT OF THE PROCESS

It was realized in the initial stages that the project would not be viable if it was not able to treat zinc-contaminated liquors. Because there was no process for the treatment of such solutions, scientists from the National Institute for Metallurgy and engineers from Woodall-Duckham worked together to develop a process that would separate the zinc impurity from the basically ferrous chloride solutions that are commonly known as spent pickle liquor. The ferrous chloride solution could then be treated in a conventional spray-roaster plant.

This research resulted in the Metsep Process, which consists of three stages. The first stage involves the separation of the zinc chloride in a continuous countercurrent ion-exchange plant, the zinc-free liquor

being hydrolysed in a spray-roaster plant. The dilute zinc chloride solution from the ion-exchange plant is then converted to concentrated zinc sulphate solution in a liquid-liquid-extraction plant, and the dilute hydrochloric acid from this stage is used for absorption of the hydrogen chloride produced in the spray-roaster plant. The process produces no effluent and results in the maximum recovery of all the chloride present in both the contaminated and the zinc-free liquor.

## THE PROCESS

The essential steps in the Metsep Process are given in Fig. 1, and typical assays are given in Tables I to III.

The spent pickle liquor is received in tanker loads of up to 25 tonnes and is transferred into the relevant feed tanks, the zinc-contaminated liquor as feedstock for the ion-exchange plant, and the zinc-free liquor as feedstock for the spray roaster.

The object of the ion-exchange plant is to remove all the zinc present in the contaminated liquor because zinc chloride can be tolerated only in very small quantities in the spray roaster. The two streams leaving the ion-exchange plant are as follows:

- (1) liquor free of zinc, which joins the other zinc-free liquor as

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TABLE I  
SUMMARY OF TYPICAL ASSAYS IN THE ION-EXCHANGE PLANT

	HCl g/l	Fe <sup>2+</sup> g/l	Zn <sup>2+</sup> g/l
Zinc-contaminated feedstock	26	95	40
Zinc-free liquor	22	80	<1
Eluate	1,3	1,5	15
Resin from the loading column			65 to 75
Resin from the washing column			55 to 65
Resin from the eluting column			0 to 3

TABLE II  
SUMMARY OF TYPICAL ASSAYS IN THE LIQUID-LIQUID-EXTRACTION PLANT

	HCl g/l	Fe <sup>2+</sup> g/l	Zn <sup>2+</sup> g/l	H <sub>2</sub> SO <sub>4</sub> g/l	Cl mg/l
Eluate from ion-exchange plant	1,3	1,5	15	—	—
Raffinate from liquid-liquid-extraction plant	15,5	1,5	2,3	—	—
Zinc sulphate product	—	—	95,0	300	4,5
First-stage washing liquor	—	—	0,01	5	—
Second-stage washing liquor	—	—	Traces	Traces	—
Loaded solvent	—	—	0,8	—	—
Stripped solvent	—	—	Traces	Traces	—

TABLE III  
SUMMARY OF TYPICAL ASSAYS IN THE SPRAY-ROASTER PLANT

	HCl g/l	Fe <sup>2+</sup> g/l	Zn <sup>2+</sup> g/l	Cl g/l
Feed to reactor	24	85	<1	—
Recovered acid	16 to 18%	10	<3	—

feedstock for the spray roaster, and

(2) the eluate, which is a solution of zinc chloride containing traces of iron and hydrochloric acid.

The object of the liquid-liquid-extraction unit is to remove all the zinc present in the eluate from the ion-exchange plant and to convert it to zinc sulphate in a concentrated solution. The following major streams leave this plant:

- (a) zinc sulphate product, which is a fairly concentrated solution of zinc sulphate in sulphuric acid (approximately 90 g of zinc per litre of solution), and
- (b) raffinate, which consists of hydrochloric acid (15 g per litre of solution) with traces of iron, zinc, and sulphuric acid.

The zinc sulphate is sold to an electrolytic zinc refinery, and the raffinate is returned to the hydrochloric acid absorber of the spray-roaster unit. Thus, all the chlorides are recovered.

The following are the two main products leaving the spray roaster:

- (i) hydrochloric acid (16 to 18 per cent), which is used as pickling acid, and
- (ii) iron oxide in the form of

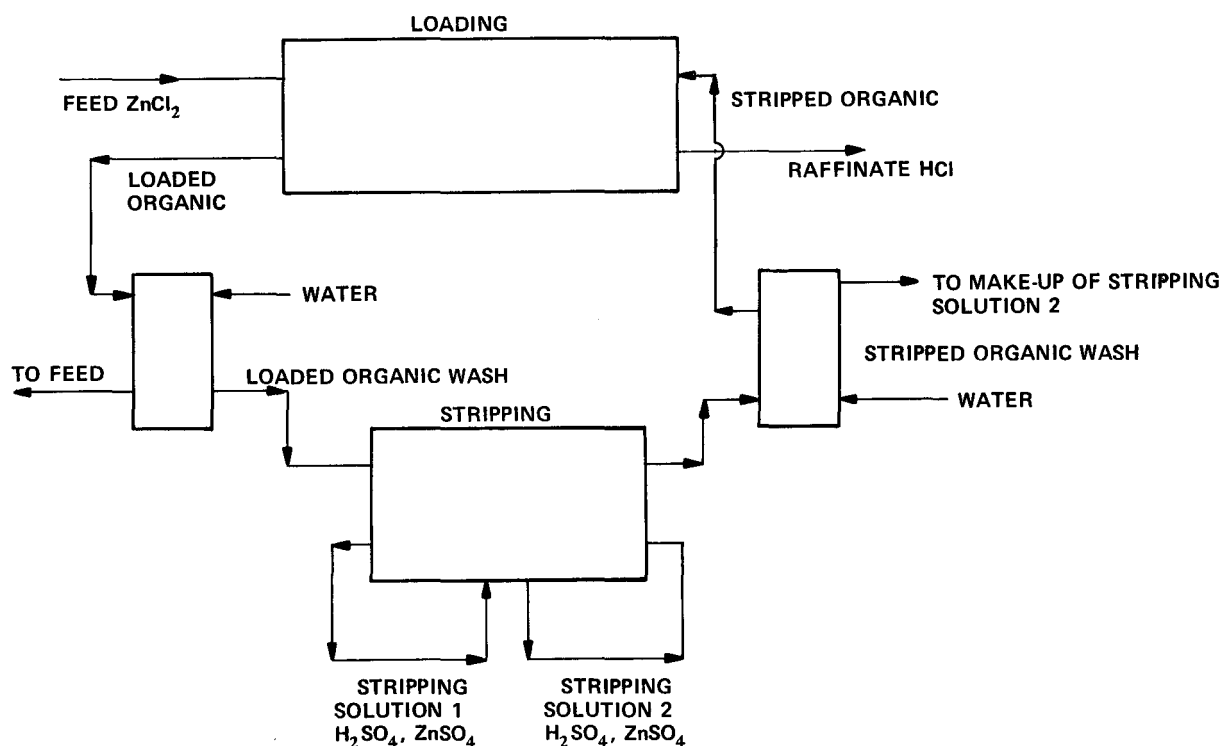


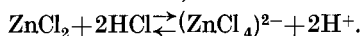
Fig. 1—Block flowsheet of the Metsep Process

Fe<sub>2</sub>O<sub>3</sub>, which is sold as low-grade pigment, or as a raw material for the manufacture of high-grade pigments, or for use in the ferrite industry.

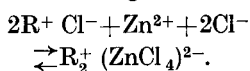
## THE ION-EXCHANGE PLANT

### Chemistry of the Process

The zinc-contaminated spent pickle liquor is contacted with a weak-base ion-exchange resin (Relite MG) in a continuous countercurrent ion-exchange plant working on the Cloete-Streat principle. The theory behind the ion exchange of zinc is that ZnCl<sub>2</sub>, in the presence of free hydrochloric acid, forms (ZnCl<sub>4</sub>)<sup>2-</sup>:



At this concentration of hydrochloric acid, neither Fe<sup>2+</sup> nor Fe<sup>3+</sup> ion forms complex anions. The complex zinc chloride ion is adsorbed on the anion-exchange resin, the reaction being



The zinc is then removed from the resin by elution with demineralized water, and the resin reverts to the chloride form.

### Outline of the Process

The use of continuous columns is an essential feature of this ion-exchange process. The liquor is so concentrated that the resin in a fixed-bed system would be fully

loaded in only two bed volumes, and washing of the resin for the removal of iron chloride would seriously dilute the liquor and would result in some elution. Thus, the feed liquor is fed to the bottom of the loading column while fresh resin is fed to the top of the column in countercurrent manner. The loaded resin is transferred to the top of the washing column, where the resin is washed with a portion of the eluate. The washed resin is then sent to the top of the elution column, where demineralized water is used to elute the zinc chloride from the resin.

The dimensions of the ion-exchange plant are given in Table IV, and the plant is illustrated in Fig. 2.

TABLE IV  
DIMENSIONS OF THE ION-EXCHANGE PLANT

	Loading column	Washing column	Eluting column
Height, m	7,0	7,0	7,0
Diameter, m	0,84	0,341	0,56
Number of stages	7	4	7

In each column, the resin is fluidized by the upward flow of liquor, and true countercurrent operation is obtained. It will be noted (Fig. 2) that the resin is filtered before being introduced into the loading column. This is done to

prevent undue dilution of the iron chloride solution. The amount of washing liquor is kept to a minimum for the same reason. The iron chloride content should be kept to more than 10 per cent, which is essential for the economy of the spray roaster to which this liquor is sent.

The ion-exchange columns are fitted with perforated trays, and a special tray at the bottom of the column prevents the resin from draining out of the column when it is shut down. A chamber is provided at the base of each column to collect the resin moving downwards from the column, the resin being hydraulically elevated to the next column in series.

The operation of the columns is cyclic, and the cycles are the same for each column. During the upflow period, the feed liquor to each column is pumped at a steady flowrate, which has the effect of expanding the resin in the column to a greater or lesser degree. The flow of liquor is then stopped, and the resin is allowed to settle in the trays. During the downflow cycle, resin is transferred from tray to tray, and some leaves the bottom of the column. The resin from the bottom tray is collected in resin receivers and is then transferred to the next column by hydraulic pressure. There is a delay between the

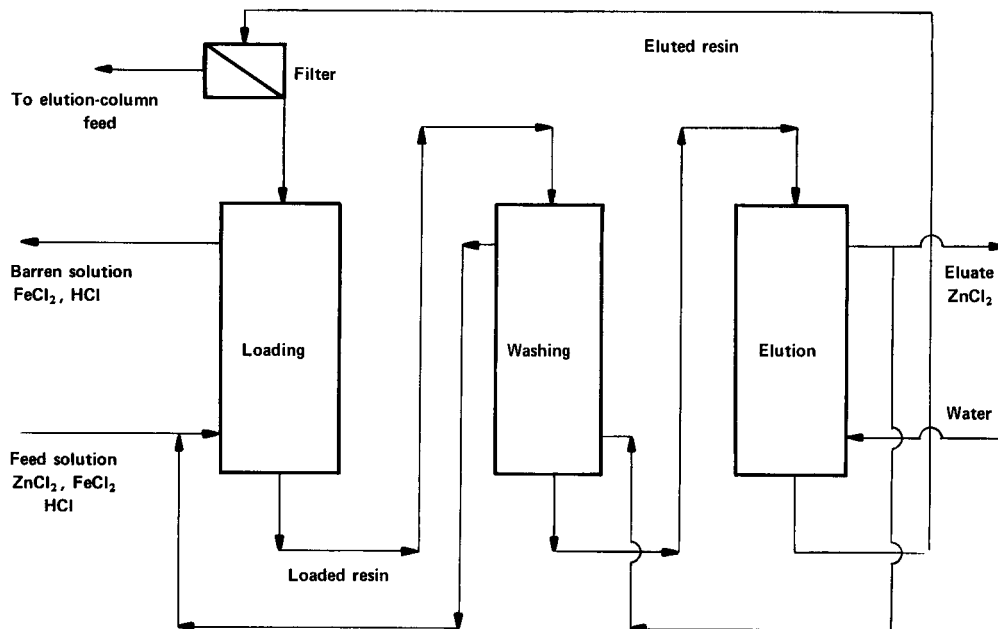


Fig. 2—Flowsheet of the ion-exchange plant

TABLE V

TIMING CYCLE IN THE ION-EXCHANGE PLANT

Period	Minutes	Seconds
Settling of resin	1	
Draining of resin	2	
Delay	1	
Transfer of resin	1	40
Upward flow of solution	12	

downflow period and this transfer period to ensure that all the resin that collects in the conical section of the column during the downflow period is transferred through to the receiver.

The sequence of operations, which is given in Table V, is controlled by a series of timers. It will be noticed that, because the concentration of the solutions is high, the period of liquor flow is relatively short.

The loading of zinc on the resin is kept as constant as possible, and, to prevent loss of zinc, the flowrate of pickle liquor is varied according to the concentration of the zinc in the liquor. The average flowrates are given in Table VI.

TABLE VI

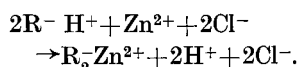
AVERAGE FLOWRATE OF SOLUTION IN THE ION-EXCHANGE PLANT

Zinc-contaminated liquor, m <sup>3</sup> /h	1,3
Eluate to washing column, m <sup>3</sup> /h	0,4
Water to eluating column, m <sup>3</sup> /h	1,5
Resin flow per cycle, m <sup>3</sup>	0,130

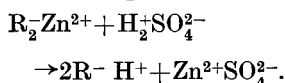
### THE LIQUID-LIQUID-EXTRACTION PLANT

#### Chemistry of the Process

The chemistry of the process is that the zinc ion (Zn<sup>2+</sup>) forms a complex with the solvent DEHPA (di-2-ethyl-hexyl-phosphoric acid). The reaction is



The hydrochloric acid formed during the above reaction is removed in the raffinate, and the loaded solvent is contacted with strong sulphuric acid. The following reaction takes place:



The zinc is removed from the solvent and forms zinc sulphate, the solvent being converted into its original form.

#### Outline of the Process

The liquid-liquid-extraction plant

consists of ten mixer-settlers of pump-mix design. Its dimensions are listed in Table VII, and typical flowrates are given in Table VIII. The flowsheet is given in Fig. 3. Because the organic flow takes longer than the aqueous flow, the mixer-settlers can recycle a large proportion of the aqueous phase in each mixer-settler.

The feed to the liquid-liquid-extraction circuit has a zinc concentration of 15 g/l, the elution flow to ion exchange being controlled so that this concentration is not exceeded. As this solution should not contain any free acid or iron, the flowrate of washing liquor in the ion-exchange plant is controlled to ensure that this is so.

TABLE VII

DIMENSIONS OF THE LIQUID-LIQUID-EXTRACTION PLANT

Height of weir, mm	715
Length of settler, mm	5600
Length of overflow compartment, mm	250
Volume of mixer, litre	1400

TABLE VIII

AVERAGE FLOWRATE OF SOLUTION IN THE LIQUID-LIQUID-EXTRACTION PLANT

Flow of eluate to plant, m <sup>3</sup> /h	1,23
Flow of solvent, m <sup>3</sup> /h	11,10

The stripping operation proceeds in five stages:

(a) washing of the loaded solvent

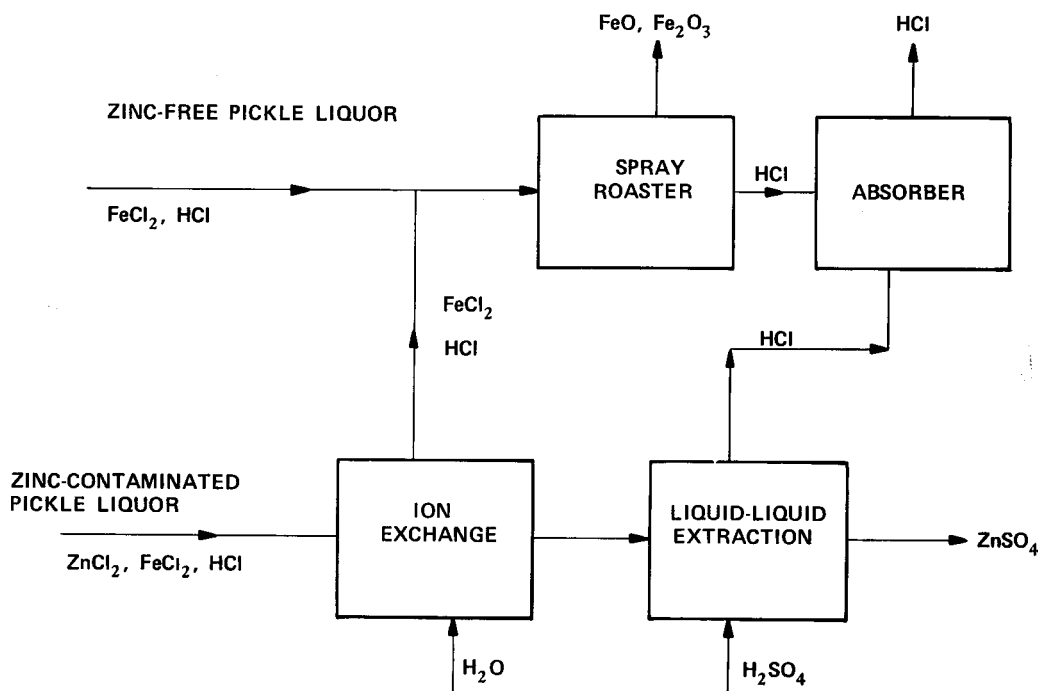


Fig. 3—Flowsheet of the liquid-liquid-extraction plant

with demineralized water for the removal of entrained zinc chloride, the liquor being used until the chloride rises to a level at which working becomes inefficient and then being used as eluate makeup,

- (b) stripping of the solvent with a 30 per cent solution of sulphuric acid to produce a concentrated solution of zinc sulphate, the large batch of acid being recirculated until the zinc concentration reaches 90 g/l,
- (c) stripping with a 20 per cent solution of sulphuric acid for the removal of any remaining traces of zinc, and
- (d) two washing stages with demineralized water for the removal of entrained sulphuric acid.

It has been found that efficiency is best when the washing stages are operated with a continuous organic phase.

### THE SPRAY-ROASTER PLANT

The recovery operation is centred around the reactor (Table IX), which is designed to provide intimate contact between the spent pickle liquor and a stream of hot combustion gases obtained by the burning of fuel gas. The pickle liquor is introduced into the top of the reactor through a number of spray nozzles, which divide the liquor into many small drops. These drops

TABLE IX  
DIMENSIONS OF THE SPRAY-ROASTER PLANT

Diameter of reactor, m	5,2
Diameter of absorber, m	1,0

provide a large surface of liquor in contact with the hot gases. In the top part of the reactor, the water and hydrochloric acid in the drops are given off, and the drop is reduced to a hollow sphere of ferrous chloride. In contact with the hot gases at the middle and bottom of the reactor, the ferrous chloride is converted into solid iron oxide and gaseous HCl. The bulk of the iron oxide drops into the bottom of the reactor and is elevated by a pneumatic conveying system into an oxide bunker. About 10 per cent of the oxide formed in the reactor is carried out with the gases leaving the top of the reactor, and a cyclone separator removes the bulk of this oxide. The gases then enter an absorber that is designed to remove the HCl in the raffinate. The concentration of the acid leaving the absorber is between 16 and 18 per cent. The average flowrates achieved are given in Table X.

TABLE X  
AVERAGE FLOWRATE OF SOLUTION IN THE  
SPRAY-ROASTER PLANT

Feed (spent pickle liquor), m <sup>3</sup> /h	1,8
Recovered acid, m <sup>3</sup> /h	1,44
Iron oxide, t/h	0,2

### MATERIALS OF CONSTRUCTION

As can be expected on all plants handling hydrochloric acid and chloride solutions, the materials of construction play a vital role.

In the ion-exchange plant, vessels, tanks, and columns are constructed of mild steel lined with rubber,

and the trays of rigid PVC. The piping is of high-density polyethylene, and the pumps are of Keebush or polypropylene.

The materials in the spray-roaster plant are identical to those in other Woodall-Duckham Spray Roasters, in which wide use is made of titanium, Keebush, acid brick, and rubber lining. The piping in cold service is of high-density polyethylene, whereas the hot-service piping is polypropylene.

### PERFORMANCE

Both the ion-exchange and spray-roaster plants came into full operation towards the end of 1973 and have operated very satisfactorily. The resin is loaded to its full capacity of 70 to 80 g of zinc per litre of wet-settled resin, and no degradation has been observed. The operation of the spray-roaster plant has shown that, with reasonable maintenance and good housekeeping, the plant can be kept clean at all times of iron oxide dust.

The liquid-liquid-extraction plant was commissioned in July 1974.

### ACKNOWLEDGEMENT

This paper is published by permission of the Director General, National Institute for Metallurgy, Johannesburg, and of Messrs Woodall-Duckham Metsep (Pty) Ltd, Germiston.

### REFERENCE

1. HAINES, A. K., *et al.* The recovery of zinc from pickle liquors by ion exchange. *J. S. Afr. Inst. Min. Metall.*, vol. 74, no. 4, Nov. 1973. pp. 149-157.

## ISEC '77

Papers are invited on the following aspects of solvent extraction for the International Solvent Extraction Conference, which is to be held in Toronto from 10th to 16th September, 1977:

- Organic Reagents
- Equipment
- Nuclear Processes

- Dispersion and Coalescence
- Organic Processes
- Base Metal Processes
- Analytical
- Mass Transfer
- Modelling.

Prospective authors should submit titles and abstracts (minimum 500 words) to Dr M. H. I. Baird,

Secretary ISEC '77, Dept of Chemical Engineering, McMaster University, Hamilton, Ontario, L8S 4L7, Canada. All abstracts must be in English and must be received by 1st September, 1976.

Further details of the Conference can be obtained from Dr Baird at the above address.

## NIM reports

The following reports are available free of charge from the National Institute for Metallurgy, Private Bag 7, Auckland Park 2006.

### Report no. 848

*The distribution of nickel in serpentinite from Lusinzi, north-eastern Transvaal.* (23rd Oct., 1969. Re-issued Jan. 1976).

Nine samples of serpentinite and related rocks from the Lusinzi nickel prospect of the Phosphate Development Corporation Limited were investigated. The serpentinites are derived from harzburgite and appear, through residual weathering, to have become enriched in nickel.

The nickel content of the serpentinites varies between 0,5 and 1,7 per cent, with an average of 1,0 per cent. Detailed studies indicate that 60 per cent of the total nickel content of these rocks is to be found in the serpentinite minerals. The remainder of the nickel occurs mainly in the limonite and magnetite.

This deposit, if not economically exploitable, is obviously a borderline case, and some recommendations are made to aid in the evaluation of the ore-body.

### Report no. 1614

*The extraction of rutile from smelter slags.* (22nd Feb., 1974. Re-issued Nov. 1975.)

Factors affecting the precipitation of rutile from smelter slags were investigated, with particular reference to the effect of additions of phosphorus pentoxide to the slag. Identification of the phases and determination of the phase boundaries were made for slags having a phosphorus pentoxide content of 0 to 33 per cent. The liquidus temperatures of these slags were determined, and a pseudo-binary phase diagram was constructed.

Rutile crystallized as the primary phase from melts having a phosphorus pentoxide content of more than 15,5 per cent. Prismatic rutile crystals whose  $\text{TiO}_2$  content was more than 97 per cent, and whose dimensions were 100 microns in cross-section and 500 to 1000

microns in length, were grown from melts that had a phosphorus pentoxide content of 23 per cent.

Attempts were made to extract these crystals by gravity separation at three particle-size ranges, and use was made of a phosphoric acid leach for the removal of the glassy matrix. Low recoveries and low grades resulted. The very small size of the crystals would make any ore-dressing process difficult.

A brief investigation was made of the morphology of rutile crystals precipitated from melts having rutile contents of 40, 60, and 80 per cent, and it was shown that higher concentrations of  $\text{TiO}_2$  in the slag are associated with crystals of larger size that still possess the acicular habit.

### Report no. 1778

*An apparatus for the automatic measurement of current efficiencies during the electrodeposition of nickel in chloride media.*

An apparatus that automatically measures the current efficiencies during the electrodeposition of metal is described. The apparatus can take separate deposition and stripping solutions, and make measurements at four different current densities (preset) and three electrolyte flow rates in one sequential operation.

The apparatus is designed to operate from 5 to 70°C under conditions of high purity, in that only Teflon and glass come into contact with the electrolytes. The system can be purged and can operate under inert atmospheres.

It operates without attention after the 'start' switch is activated, going through alternate cathodic and anodic modes, with intermediate washing and draining periods. Currents of up to 100 mA and electrolysis times of up to 15 minutes can be preset.

With slight modifications, the apparatus can be used to provide data, as well as Tafel slopes, for the diagnosis of kinetic mechanisms.

### Report no. 1781

*The determination, by ion-selective*

*electrode, of low concentrations of fluoride in complex materials.*

The direct determination of fluoride in sulphides and aluminosilicates by use of an ion-selective electrode and the method of standard addition was examined. A single decomposition technique, involving fusion in a vitreous-carbon crucible, was adopted for both types of sample. The manner in which the Nernstian slope changes at low concentrations of fluoride for different buffer systems was established, and a suitable TISAB buffer was developed for the formation of complexes with the interfering ions contributed by the types of sample considered.

The addition to the sample solution of a standard solution prepared from the sample solution achieved virtual matching of all the interfering ions, particularly aluminium. Aging of the standard solution is necessary if equilibrium conditions are to be achieved. Fluoride from 80 p.p.m. upwards can be determined in sulphide samples having a copper, lead, or zinc content of up to 80 per cent, and from 40 p.p.m. upwards can be determined in aluminosilicates having an  $\text{Al}_2\text{O}_3$  content of up to 40 per cent. The coefficient of variation of the method is about 4 per cent, and the accuracy is within 5 per cent.

### Report no. 1782

*The application of air-sparging techniques to the industrial electrodeposition of zinc from acidic zinc sulphate solutions.*

Air sparging was tested on a pilot-plant basis in an attempt to improve the current efficiency during the electrodeposition of zinc from acidic zinc sulphate solutions.

The only advantage noticed from the use of air sparging was an increase in the purity of the zinc deposit, which had a lower lead content. As smoother deposits were obtained, it is supposed that this small decrease in the lead content results from a lower adsorption of solid lead sulphate particles at the metal surface.

A design that incorporates permanent sparging tubes at the bottom of each anode is proposed.

### Report no. 1783

*The spectrographic determination of minor and trace elements in copper, lead, and zinc concentrates.*

This report deals with the development of a method for the determination, by an emission-spectrographic technique, of magnesium, manganese, aluminium, silver, calcium, chromium, cobalt, titanium, antimony, cadmium, molybdenum, zirconium, nickel, boron, vanadium, arsenic, beryllium, tin, germanium, and bismuth in copper, lead, and zinc sulphide concentrates.

The method involves the preparation of complex standards in which the volatile elements arsenic, antimony, cadmium, tin, and bismuth are incorporated as sulphide compounds at temperatures of 800°C in evacuated silica tubes together with a synthetic sulphide matrix. These standards are then mixed with the other minor and trace elements to form composite standards. The conditions for excitation with a direct-current arc, and the analytical lines for the elements and internal standards, are given. The procedure is rapid and convenient, and involves the minimum of sample preparation. The accuracy is about 10 per cent, and the method has a coefficient of variation for the various elements of between 2 and 13 per cent.

### Report no. 1787

*The determination, by atomic-absorption spectrophotometry, of trace elements in sulphide concentrates.*

The separation, concentration, and determination of trace elements in base-metal and sulphide concentrates are described. After the sample has been dissolved, the trace elements that form insoluble hydroxides are precipitated with lanthanum as the coprecipitant and are separated from those elements that form soluble amines with ammonia. The precipitate is dissolved, and the trace elements selenium, tellurium, arsenic, antimony, bismuth, tin, vanadium, chromium, manganese, and aluminium are determined by atomic-absorption spectrophotometry.

Coefficients of variation between

1 and 10 per cent, depending on the amount of the element, were obtained, with limits of determination ranging from 0,1 to 50 p.p.m.

### Report no. 1788

*A computer programme for emission-spectrographic calculations.*

A computer programme written in BASIC, which calculates Seidel functions for emission-calibration curves and linear or non-linear concentration-calibration curves with the use of a least-squares regression method, is described.

The computer programme allows for easy rejection of points for both types of calibration curves (i.e., emulsion and concentration), and requires a 16 K mini-computer for operation.

### Report no. 1795

*The determination of minor and trace elements in the leach liquors from anode sludges.*

The solutions arising from the processing of anode sludges contain up to fifteen elements in minor and trace concentrations for which analysis is required. The atomic-absorption determination of copper, lead, nickel, and silver following dilution of the solution, and of platinum, palladium, and gold after destruction of the organic content of the processing solution with aqua regia, is briefly examined. The remaining elements — arsenic, antimony, bismuth, selenium, tellurium, iron, tin, and aluminium — which cannot be determined by either of these procedures, are concentrated by co-precipitation with titanium hydroxide and, after dissolution of the hydroxide precipitate, are determined by atomic-absorption spectrophotometry.

Factors affecting the co-precipitation of the elements of interest were examined, and the precision and accuracy were determined by the use of synthetic solutions. Recoveries of more than 93 per cent, except for arsenic, were obtained, and the precipitation ranged from 1 to 8 per cent. The limits of determination for the dilution technique vary between 2,5 to 5 p.p.m. in the solution for analysis, and, for the co-precipitation technique, from 0,1 to 1 p.p.m. in the sample solution.

### Report no. 1797

*A rapid atomic-absorption method for the determination of silver in sulphide ores and concentrates.*

A rapid method for the determination of silver in sulphide ores is described. At concentrations of less than 100 p.p.m. of silver, the sample is dissolved in brominated hydrochloric acid and nitric acid, and the silver determined by atomic-absorption spectrophotometry. When the silver is present at concentrations of greater than 100 p.p.m., the sample is fused with sodium peroxide and, after leaching, diluted to a suitable volume for measurement by atomic absorption.

### Report no. 1798

*The determination, by atomic-absorption spectrophotometry, of copper, lead, and zinc in sulphide concentrates.*

The direct procedure described uses a nitrous oxide-acetylene flame. Tests leading to its development are briefly described. The precision is approximately 1 per cent of the content over the ranges 4 to 63 per cent for lead, 5 to 21 per cent for copper, and 5 to 54 per cent for zinc. With the exception of zinc, for which there was a small negative bias, the values obtained agreed to within 5 per cent with those obtained by the method of standard additions, which, in the absence of suitable reference materials, was used for comparison.

### Report no. 1814

*An updated summary of the World's fluorspar industry, 1975.*

The consumption of all grades of fluorspar in the Western World decreased by 4,7 per cent, from 2,54 million tonnes in 1974 to approximately 2,42 million tonnes in 1975, but is expected to increase from the last quarter of 1976 onwards. In 1975, steelmaking accounted for 53 per cent of the consumption, the balance being consumed, in order of importance, by the following industries: fluorocarbons (25,7 per cent), aluminium (16,0 per cent), stainless-steel pickling (3,2 per cent), petroleum alkylation (1,8 per cent), and uranium enrichment (1,1 per cent).

South Africa has the largest

proven ore reserves containing approximately 29 million tonnes of CaF<sub>2</sub> and, with a production capacity of 420 000 tonnes in 1975, ranks third amongst the fluorspar-

producing countries. South African exports of some 133 000 tonnes in 1974 represented 2,6 per cent of World production.

It can be expected that, from the

mid-1980s onward, South Africa will become a major supplier of fluorspar to the World, since exploitable ore-deposits all over the World face accelerated depletion.

## Progress report, AS&TS Trust

Donations to the AS&TS Trust this year were as follows:

<b>Companies</b>	
Messrs CNA Investments Limited, Barlow Rand Limited, South African Cement Producers Association, Philips Telecommunications (Pty) Limited, Sappi Limited, Van Leer South Africa (Pty) Limited, Zakrzewski Associates Incorporated .....	
	R2 630,00
<b>Individual Members via Their Institutes</b>	
Geological Society of South Africa .....	48,00
Institute of Land Surveyors of the Transvaal .....	103,00
Institution of Certificated Mechanical and Electrical Engineers, S.A. ....	969,00
Royal Aeronautical Society (Southern Africa Branch) ..	73,30
S.A. Chemical Institute ....	5,00
S.A. Institute of Assayers and Analysts .....	33,00
S.A. Institute of Electrical Engineers .....	2 423,00
S.A. Institute of Mining and Metallurgy .....	1 469,00
S.A. Institution of Civil Engineers .....	4 022,00
S.A. Institution of Mechanical Engineers .....	601,00
	R9 746,00
<b>Individuals</b>	
J. D. Aungiers, G. E. Burgess, D. Dryborough, R. Bailey-McEwan, R. E. Purdham ..	
	94,00
Total donations for the year 1974/75.....	R12 470,00
Plus interest on funds invested	1 721,00
Add balance as at 30th September 1974 .....	11 522,00
Total accumulated funds at 30th September, 1975 ...	R25 713,00

An increasing number of individual members are contributing to the Trust through their institutions and societies, and I appeal to all societies to advance the cause of the Trust by giving publicly to the aims of the Trust and by assisting members to make donations when remitting their annual dues. The Foundation and Admitted Societies of the AS&TS have over 19 000 members, and the Affiliated and Associate Societies another 6000 members, a total of 25 000 in all, and the Trustees are anxious to raise at least R4 per member to provide an initial fund of R100 000, which would be large enough to provide significant assistance to further the interests of the AS&TS.

The AS&TS in itself is a service organization supplying managerial and secretarial facilities to its member societies. It cannot build up worth-while capital reserves from the subscriptions received, and thus finds itself severely restricted when contemplating the expansion necessary to fulfil its function as a national organization. The Trust appeal was launched three years ago to provide some financial backing, and the steady growth in donations is sufficiently encouraging to suggest that some funds could now be used as a catalyst to start specific projects.

The primary aim should be the building up of branch organizations to foster the enthusiasm for and the

spread of science and technology throughout the country.

Other valuable suggestions, which must be regarded as longer-term aims, include backing for scientific associations for teachers, support for technician societies, and help for non-white scientific and technical organizations. It is thus imperative that the Trust should continue to grow. Let us go for the R100 000 immediately!

May I ask individual members of societies again to be generous with their donations; and, secondly, with a view to the longer-term effort, would members please not forget the merits of the Trust when making bequests in their wills.

The Trustees during the year were Drs T. F. Muller, H. J. Nel, J. D. Roberts, A. E. Rupert, and Messrs R. S. Lawrence (President of the Chamber of Mines), Stanley Craib, G. A. P. Low, Prof. D. C. Midgley, with myself as Chairman. Once again I am greatly indebted to them for their help, and my thanks must also go to those members and companies who have contributed to the Trust, and to Mr Boden and his Staff at the AS&TS, who have administered the Trust free of charge.

R. C. J. Goode

22nd December, 1975 Chairman

## Verslag oor vordering, GW&TV-trust

Skenkings aan die GW&TV-trust was vanjaar soos volg:

<b>Maatskappye</b>	
Mnre CNA Beleggings Beperk, Barlow Rand Beperk, Suid-Afrikaanse Vereniging van Sementvervaardigers, Philips Telecommunications (Edms) Beperk, Sappi Beperk, Van Leer Suid-Afrika (Edms) Beperk, Zakrzewski Associates Incorporated .....	
	R2 630,00

<b>Individuele Lede deur Hulle Verenigings</b>	
Geologiese Vereniging van Suid-Afrika .....	48,00
Instituut van Landmeters van die Transvaal .....	103,00
Instituut van Gediplomeerde Werktuigkundige en Elektrotegniese Ingenieurs, S.A. ..	969,00
Royal Aeronautical Society (Afdeling Suidelike Afrika)	73,30
S.A. Chemiese Instituut ....	5,00

S.A. Instituut van Essaieurs en Analitici .....	33,00
S.A. Instituut van Elektriese Ingenieurs .....	2 423,00
S.A. Instituut vir Mynbou en Metallurgie .....	1 469,00
S.A. Instituut van Siviele Ingenieurs .....	4 022,00
S.A. Instituut van Meganiese Ingenieurs .....	601,00
	R9 746,00



### Individue

J. D. Aungiers, G. E. Burgess,  
D. Dryborough, R. Bailey-  
McEwan, R. E. Purdham . . . .

94,00

Totaal vir die jaar 1974/75 . . R12 470,00

Plus rente op gelde belê . . . . 1 721,00

Voeg balans soos op 30 Sep-  
tember 1974 by . . . . . 11 522,00

Totaal van opgehoopde fonds  
soos op 30 September 1975 R25 713,00

'n Toenemende getal individuele lede dra deur middel van hulle Institute by tot die Trust, en ek doen 'n beroep op alle verenigings om die Trust se saak te bevorder deur die doelstellings van die Trust bekend te maak en dit vir lede maklik te maak om donasies by die betaling van hulle jaargelde in te sluit. Die Sigter- en Toegelate-verenigings van GW&TV het meer

as 19 000 lede, en die Affiliaat- en Assosiaatverenigings 'n verdere 6000 lede, alles tesame 25 000. Die Trustees is gretig om minstens R4 per lid op te neem ten einde 'n aanvanklike fonds van R100 000 te verskaf wat groot genoeg sal wees om vir betekenisvolle hulp vir die bevordering van GW&TV se belange te sorg.

Die GW&TV is op sigself 'n diensorganisasie wat bestuurs- en sekretariële fasiliteite aan sy lede-verenigings verskaf. Hy kan nie kapitaalreserwes wat die moeite werd is, uit die ledegeld wat ontvang word, opbou nie, en word dus ernstig gestrem wanneer die nodige uitbreiding om sy funksies as 'n nasionale organisasie uit te voer, oorweeg word. Die Trust se oproep het drie jaar gelede begin om gelde-lyke steun hiervoor te verskaf, en die

bestendige groei van donasies is voldoende aan moediging om voor te stel dat geld nou as 'n katalisator gebruik kan word om spesifieke projekte mee te begin.

Die hoofdoel behoort te wees om takorganisasies tot stand te bring om die geesdrif vir en die uitbreiding van natuurwetenskap en tegnologie dwarsdeur die land aan te moedig.

Ander waardevolle voorstelle sluit steun in vir wetenskaplike verenigings vir onderwysers, vir tegnikus-verenigings en hulp vir nie-blanke wetenskaplike en tegniese verenigings. Hierdie moet egter as lang termynmikpunte gesien word. Dit is dus noodsaaklik dat die Trust steeds moet groei. Kom laat ons dadelik na die R100 000 mik.

R. C. J. Goode

22 Desember 1975

Voorsitter

## Hydrometallurgy

The South African Institute of Mining and Metallurgy has great pleasure in announcing its intention of holding a Vacation School on the topic of **Hydrometallurgy — Theory and Practice**. The motivation for this vacation school is to provide the opportunity for engineers in industry and research and development to participate in short, intensive technical programmes, to encourage technical interaction between engineers engaged in similar spheres, to supply a refresher course with particular emphasis on trends and new developments, and to encourage continued familiarity with the relevant current technical literature. The school will be of interest to metallurgical engineers, chemical engineers, industrial chemists, and chemists in research and development organizations.

It is the intention to conduct this vacation school on an educational level and to provide notes on each of the topics covered by the programme, and so encourage an enhanced understanding of the topic.

The Vacation School Committee

has been particularly fortunate in attracting lecturers of a very high calibre. The school leaders will be Professor E. Peters and Professor I. H. Warren, Department of Metallurgy, University of British Columbia. These two eminent lecturers are well known for their contribution in the field of hydrometallurgy and in 1972 presented the First Tutorial Symposium on Hydrometallurgy sponsored by The Extractive Metallurgy Institute of the Colorado School of Mines, the University of Denver, and the American Institute of Mining.

In addition, the following seven local lecturers will participate in the course:

**Dr A. W. Bryson, Senior Lecturer, Department of Chemical Engineering, University of the Witwatersrand**

**Mr F. L. D. Cloete, Senior Chief Research Officer, Chemical Engineering Research Group, CSIR**

**Dr A. K. Haines, Director: Process Development Division, National Institute for Metal-**

**Dr R. D. Hancock, Chief Scientist, Mineral and Process Chemistry Division, National Institute for Metallurgy**

**Dr P. Laxen, Chief Scientist, Process Development Division, National Institute for Metallurgy**

**Dr D. I. Legge, Consulting Chemical Engineer, Anglo-Transvaal Consolidated Investment Co. Ltd**

**Dr M. J. Nicol, Chief Scientist, Mineral and Process Chemistry Division, National Institute for Metallurgy.**

The school will be held at the University of the Witwatersrand, Johannesburg, from 9th to 13th August, 1976.

The registration fee is R200,00 per person, which covers all aspects including lunches, cocktail party, dinner, and lecture notes. Persons wishing to register should contact the Secretary, SAIMM, P.O. Box 61019, Marshalltown, 2107 South Africa. Participants who wish to be accommodated in the Men's Hall of Residence should register as soon as possible.

# Rock mechanics

The South African Institute of Mining and Metallurgy has great pleasure in announcing its intention of holding two vacation schools on the topic of **The Use of Rock Mechanics Principles in Practical Underground Mine Design**. These schools are intended for practical mining engineers whose work requires the use of rock mechanics results, but who do not need to know the detailed background theory of the subject.

The programme is designed to provide a working knowledge of basic theory and of rock mechanics applications. Each school will be held for 5 days at the University of the Witwatersrand, Johannesburg. Demonstrations will be given of laboratory tests on rock samples and of the use of the Chamber of Mines electrical resistance analogue. Examples of output from the main rock mechanics computer programs — Finite Element Analysis and Minsim 1 — will be provided and discussed.

The following broad topics will be covered by various lectures during the course:

**Fundamental principles of solid mechanics**

**Mechanical properties of rocks and rock masses**

**Response of the rock mass to underground excavations**

**Stresses and displacements in-**

**duced by mining tabular deposits**

**Selection and design of tunnel supports**

**Principles of the support of production excavations**

**Protection of service excavations and shafts**

**Design of the layout of tubular excavations**

**Pillar load and stability**

**Control of rockburst hazards.**

The Institute has been able to draw on the wealth of rock mechanics experience available in this country. The following is a provisional list of some of the lecturers who have agreed to contribute to the proceedings:

BUDAVARI, Professor S.,  
Professor of Rock Mechanics in the Mining Department of the University of the Witwatersrand.

MORE O'FERRALL, R. C.,  
Group Rock Mechanics Engineer, General Mining and Finance Corporation;

ORTLEPP, W. D.,  
Group Rock Mechanics Engineer, Rand Mines Limited;

PATCHET, Dr S. J.,  
Head of Rock Mechanics Department, Anglo American Corporation of S.A. Limited;

SALAMON, Dr M. D. G.,  
Research Adviser, Chamber of Mines of South Africa;

WAGNER, Dr H.,

Assistant Director of Mining Operation Laboratory (Gold), Chamber of Mines Research Laboratories.

It is intended to provide reference material covering the subjects of the course. These documents will be available beforehand and will be a useful reference volume after the vacation school has been completed. Please note that the registration fee for attending this school will be R200,00 per person and that this fee covers all lunches and social activities. The Men's Residence at the University of the Witwatersrand has kindly agreed to arrange board and lodging at R10,00 per day for dinner, bed, and breakfast for any participant who requires accommodation.

The dates of the two schools are as follows:

**SCHOOL A:**

**26th July to 30th July, 1976**

**SCHOOL B:**

**2nd August to 6th August, 1976.**

Numbers at each school will be restricted and registration will be accepted in the sequence in which applications are received. Attendance will not be limited to members of the Institute. Overseas visitors are particularly welcome.

Further details are available from the Secretary, S.A.I.M.M., P.O. Box 61019, Marshalltown, 2107 South Africa.