



# Electrokinetic and flotation studies of hematite using di(2-ethyl hexyl) phosphoric acid

by B. Das and P.K. Naik\*

## Synopsis

The flotation studies on hematite were carried out using di (2-ethyl hexyl) Phosphoric acid (D2EHPA) as the collector. The possible mineral reagent interaction was studied through electrokinetic and dissolution studies. The shift in zeta potential (PZC) and decrease in dissolution of iron in the presence of D2EHPA indicated that D2EHPA was specifically adsorbed on hematite surface. The adsorption imparted necessary hydrophobicity for flotation of hematite. Hematite could be floated selectively from hematite and quartz synthetic mixture.

Key words: Hematite, D2EHPA, flotation, electrokinetics, dissolution.

## Introduction

The selective and specific adsorption of collector on the mineral surface is of vital importance in economic flotation separation. Therefore, there is incessant search for new efficient flotation reagents. Flotation has been applied to low-grade iron ores for removing siliceous gangue or preparing super grade concentrates for special purposes. Flotation of iron ore is carried out in both acid and alkaline pH using different reagents<sup>1</sup>. A wide variety of collectors such as oleates, sulphonates, sulphates, amines and hydroxamates have been used for the flotation of iron ores containing different iron and gangue minerals<sup>2</sup>. The reverse flotation of iron ore is being carried out by using standard long chain amine collectors<sup>3,4</sup>. More recently, a column flotation technique has been applied to Indian iron ore slimes using dodecylamine as the collector. It was possible to obtain 64.22% Fe 1.63% Al<sub>2</sub>O<sub>3</sub> and 2.0% SiO<sub>2</sub> with 72% recovery from a feed containing ~ 52% Fe<sup>5</sup>.

Solvent extractants such as LIX-65NS have been used as collector in the flotation of copper ore containing sulphide and mixed sulphide oxide ores<sup>6,7</sup>. The di (2-ethyl hexyl) phosphoric acid (D2EHPA) is a well known for its specificity towards metal ions and widely used as a solvent extractant<sup>8</sup>. It could be used as a potential flotation reagent for metallic

mineral flotation. In the present work, the amenability of D2EHPA as a reagent to float hematite and separate from siliceous gangue is tested.

## Experimental methods

Lumpy hard variety of hematite obtained from Orissa Mining Corporation (OMC) was stage crushed and ground dry in a ball mill to obtain size fraction of -150 +45  $\mu\text{m}$  and -75  $\mu\text{m}$  for flotation studies. The dissolution studies were carried out on the -75  $\mu\text{m}$  particles while they were further ground in an agate mortar for electro-kinetic studies. All the samples were kept in airtight containers. The complete chemical analysis of the hematite is given in Table I. Crystalline variety quartz was jaw crushed and then ground in a ball mill. The -75 micron pure quartz sample obtained by repeatedly washing it in dilute HCl followed by distilled water was used for preparation of a synthetic mixture of hematite-quartz for flotation studies.

The Di (2-ethyl hexyl) phosphoric acid (D2EHPA) used in this study was obtained from heavy water plant, Talcher, India. Its physical properties are given in Table II. D2EHPA is soluble in organic solvents like acetone, benzene, carbon tetrachloride and non-ionizing solvents like kerosene. The D2EHPA solution was prepared freshly before experiments by adding a known quantity of D2EHPA in acetone.

NaOH and HNO<sub>3</sub> were used as a pH regulator and KNO<sub>3</sub> of 10<sup>-3</sup> molar concentration was used to maintain the ionic strength of the solution in the electrokinetic studies. All the chemicals used are of analytical reagent grade. The distilled water, having a neutral pH, was used for all tests except flotation studies for which tap water was used.

\* Council of Scientific and Industrial Research, Regional Research Laboratory, Bhubaneswar, India.

© The South African Institute of Mining and Metallurgy, 2003. SA ISSN 0038-223X/3.00 + 0.00. Paper received Mar. 2003; revised paper received Jun. 2003.

# Electrokinetic and flotation studies of hematite using di(2-ethyl hexyl) phosphoric acid

Table I

## Chemical analysis of hematite

Constituents	Per cent
Fe <sub>2</sub> O <sub>3</sub>	95.88
SiO <sub>2</sub>	0.73
Al <sub>2</sub> O <sub>3</sub>	1.43
LOI	1.38

Table II

## Physical properties of Di (2-ethyl hexyl) phosphoric acid

1	Formula	(C <sub>8</sub> H <sub>17</sub> O) <sub>2</sub> P(O)OH
2	Chemical structure	$\begin{array}{c} \text{C}_8\text{H}_{17}\text{O} \quad \text{O} \\   \quad \quad   \\ \text{P} \\   \quad \quad   \\ \text{C}_8\text{H}_{17}\text{O} \quad \text{OH} \end{array}$
3	Molecular weight	322.431
4	Appearance	Pale yellow to colourless liquid
5	Purity	>95 wt% Diester, <2 wt% Monoester
6	Phosphorous content	9.8 wt%
7	Acid number	174 mg KOH/gm
8	Density, gm/cc at 20°C	0.974
	Density, gm/cc at 30°C	0.957
	Density, gm/cc at 40°C	0.952
	Density, gm/cc at 60°C	0.937
9	Refractive Index	1.441
10	Flash point °C (open cup method)	171
11	Viscosity at 200°C, cps Brookfield	56
12	Solubility of D2EHPA in water, % at 20°C	<0.01
13	Solubility of water in D2EHPA, % at 20°C	2.4
14	Vapour pressure at 25°C	<1

## Methods

The electrophoretic mobility measurements of hematite with and without D2EHPA at different pH were determined using Rank Brothers Mark II electrophoresis apparatus in the flat cell configuration and were converted to Zeta-potential using the Smoluchowski equation. All the measurement was conducted in a dilute suspension of 0.05% (w/v) in 10<sup>-3</sup> KNO<sub>3</sub>. The conditioning of the suspension was carried out at least 24 hours before measurement to ensure the stability of the suspension.

In conducting adsorption tests, 0.05% (w/v) gm of the mineral powder was taken and added to a fixed amount of 10<sup>-3</sup>M KNO<sub>3</sub> and D2EHPA solution of known concentration. The pH of the solution was then fixed by the addition of a small amount of acid or alkali. The entire solution was placed in a 250 ml conical flask and shaken in a Julabo Metabolic Shaker at a predetermined temperature and time. After adsorption, the suspension was centrifuged for 5 minutes and then filtered. The clear solution was then analyzed to determine the concentration of D2EHPA in solution. The

adsorption of D<sub>2</sub>EHPA in the mineral surface was then calculated by the difference between the concentration before and after adsorption by spectrophotometric method. The method is based on the formation of a yellow complex formed between the Fe (III) thiocyanate and D<sub>2</sub>EHPA which is extracted into a non-polar solvent, namely carbon tetrachloride<sup>9</sup>.

The dissolution studies of hematite as a function of time and pH in the presence and absence of D<sub>2</sub>EHPA was carried out taking 1 gm of mineral sample in 100 ml of distilled water. The suspension was agitated for a given period. The suspension was then centrifuged filtered and analysed for Fe in an Atomic Adsorption Spectrophotometer (AAS).

Flotation tests were carried out in a Denver sub-aeration cell of one-litre capacity. For the flotation tests a 100 gm of -150+45 or -75 micron size mineral sample was conditioned with a known concentration of D<sub>2</sub>EHPA solution at a desired pH for five minutes. Initially the sample was conditioned at higher consistency of solids and then diluted with water to the level of cell. The pH of the suspension was monitored throughout the experiments. After conditioning, the air was released slowly and both the concentrate and the tailing fraction were collected separately, dried and weighed. Experiments were also carried out on the synthetic mixture of hematite and quartz and the natural occurring iron ore.

## Results and discussion

### Electrokinetic studies

The variation of zeta potential of hematite with pH in the presence of two different concentration of D2EHPA at constant electrolytic concentration of 0.001M KNO<sub>3</sub> is shown in Figure 1. The point of zero charge of natural hematite is found to be at pH 4.2. This value is slightly lower than the reported value (4.8–6.7), which may be due to impurities present in the sample<sup>10</sup>. It has been observed that in the presence of D2EHPA (32.2 ppm) the PZC of hematite is lower than 2.9. The value has been further shifted to 2.3 at the higher concentration of D2EHPA (64.4 ppm). Figure 1 shows that the adsorption of D2EHPA is not uniform. However, the decrease in electrophoretic mobility by the addition of the reagent is due to the specific adsorption of a D2EHPA anion on the surface of hematite.

### Dissolution studies

The dissolution test of hematite as a function of time and pH was carried out in the absence and presence of D2EHPA to ascertain the dissolution of metal ions from the mineral surface (Figures 2 and 3). From Figure 2, it is evident that, in the absence of D2EHPA, the highest dissolution occurs in the first 10 min followed by a sharp decrease, attaining minimum at 30 minutes. After that the Fe concentration more or less remains the same. In the presence of D2EHPA the corresponding Fe concentration is always lower and follows the trend of the Fe concentration curve in the absence of D2EHPA. The higher the D2EHPA concentration, the lower the dissolution of iron. The decrease in Fe concentration is due to the adsorption of D2EHPA on the hematite surface, hindering the dissolution of iron from the mineral surface. With the increase in D2EHPA concentration dissolution of the

# Electrokinetic and flotation studies of hematite using di(2-ethyl hexyl) phosphoric acid

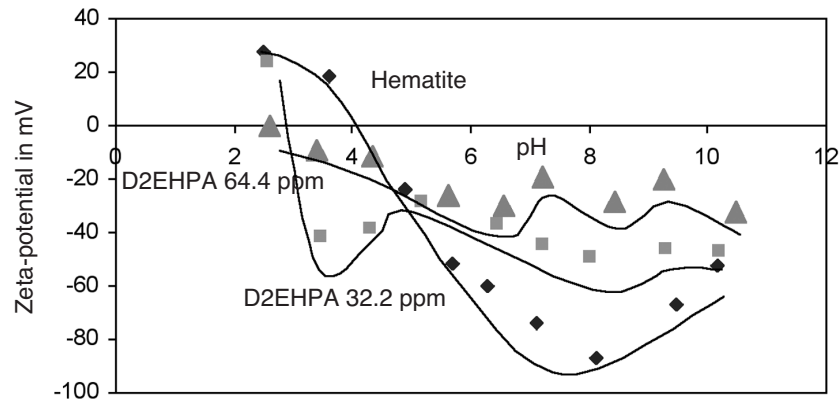


Figure 1—Variation of zeta potential of hematite as a function of pH, in the absence and presence of D2EHPA,  $10^{-3}$  M  $KNO_3$

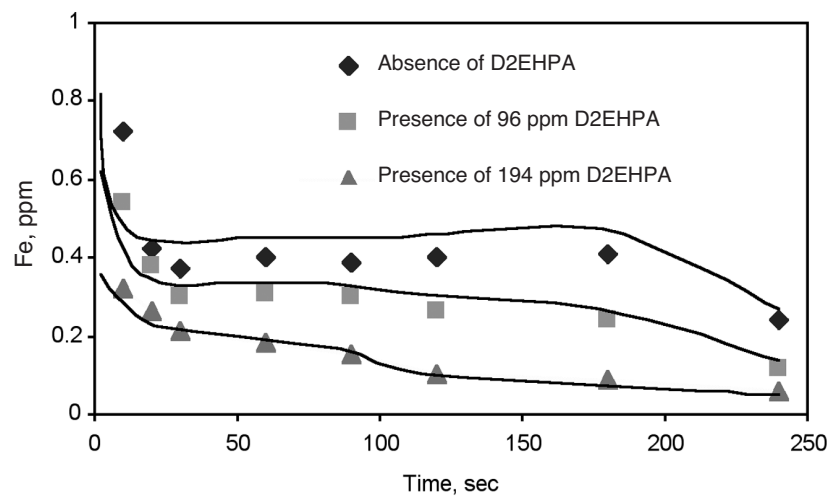


Figure 2—Dissolution of iron from hematite as a function of time in the presence and absence of D2EHPA

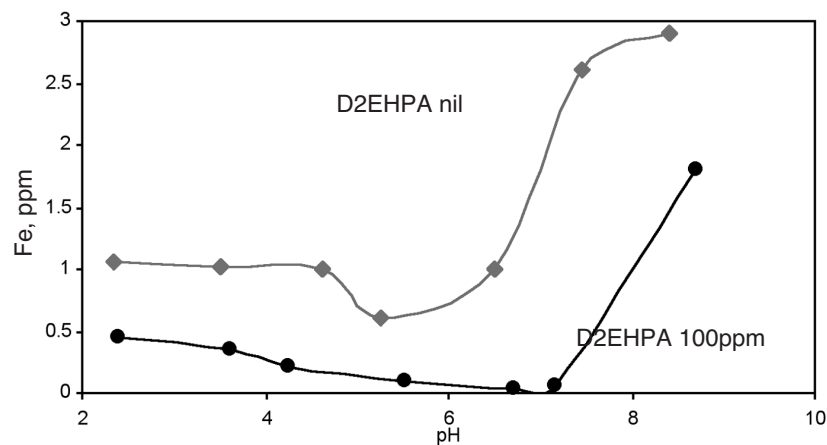


Figure 3—Dissolution of iron from hematite as a function of pH in the presence and absence of D2EHPA, agitation 60 min

Fe concentration further decreased because of co-adsorption of D2EHPA molecule and multilayer formation on the hematite surface.

The dissolution of Fe at different pH values without D2EHPA (Figure 3) shows that there is a decrease in Fe ion concentration up to pH 8 and thereafter there is a sharp increase. This increase may be due to hydrolysis of an Fe ion

at alkaline pH value. At all pH, there is a corresponding decrease in Fe ion concentration in the presence of D2EHPA due to adsorption of D2EHPA on the hematite surface.

## Flotation studies

The flotation of two different sizes of hematite particles as a

# Electrokinetic and flotation studies of hematite using di(2-ethyl hexyl) phosphoric acid

function of pH at 128 g/t is given in Figure 4. The pH of optimum flotation was at 4.7. At this pH the recovery for -75  $\mu\text{m}$  and -150+45  $\mu\text{m}$  was 70% and 42%, respectively. The recovery of hematite dropped sharply between pH 4.7 and 8.6 and after that it remained more or less the same. The recovery of -75  $\mu\text{m}$  particles beyond pH 8.6 was around 40%. This indicated that D2EHPA interacted with the hydrophilic hematite surface in a wide range of pH. D2EHPA anion adsorbs on the hematite surface with their non-polar group oriented towards bulk suspension imparting a high degree of hydrophobicity. The low floatability at low pH (<4.0) is due to the low solubility of D2EHPA and at higher pH (>4.0) is due to low availability of D2EHPA anion ( $P_{k_a}$  value 3.95) and possible interaction of the D2EHPA molecule causing multilayer formation. The floatability of hematite even at pH 12 supported the role of the D2EHPA molecule along with its anion on flotation.

The effect of reagent concentration on flotation of -75  $\mu\text{m}$  particles at pH 4.7 is given in Figure 5. It is evident that the recovery increases sharply with the increase in D2EHPA concentration up to 193.2 gm/tonne; after that the increase is very slow. The maximum recovery of 94% was obtained at 510 gm/tonne of D2EHPA. The flotation kinetics of -75

micron particles at pH 4.7 with 193.2 gm/tonne of D2EHPA concentration is given in Figure 6. It was found that the kinetics are of first order.

The flotation studies of a synthetic mixture of -75  $\mu\text{m}$  hematite and quartz particle with 193.2 gm/tonne of D2EHPA, at pH 4.7 is given in Figure 7. The recovery decreased with the increase in hematite and quartz ratio, whereas the grade increased.

## Conclusions

The electrokinetic studies indicated that the PZC of hematite is at pH 4.2 and it shifted to below 2.9 in the presence of D2EHPA. The shift in electrophoretic mobility was maximum around pH 3.5 with lower concentration of D2EHPA but less with higher concentration. This is due to charge neutralization by a species other than D2EHPA anion. Dissolution studies indicated that D2EHPA adsorbed on the hematite surface at all pH hindering the dissolution of iron. This supported flotation of hematite in a wide range of pH. The optimum pH for flotation of hematite at 10% pulp density was 4.7. At this pH hematite could be selectively floated from quartz with 193.2 gm/tonne of D2EHPA.

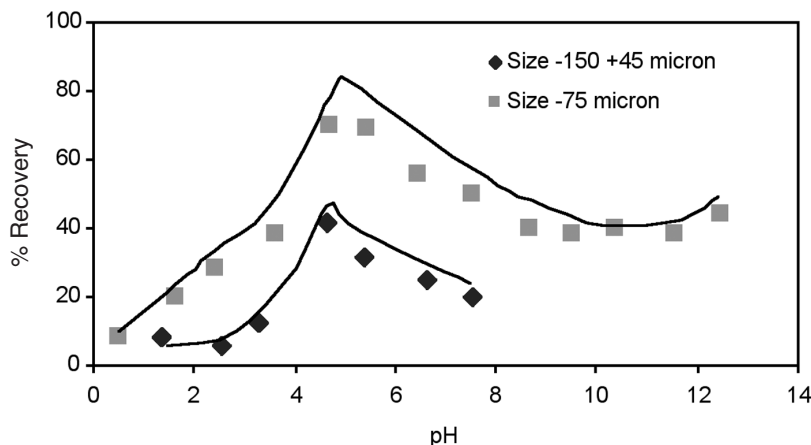


Figure 4—Recovery of hematite as a function of pH at 128.8 g/tonne of D2EHPA

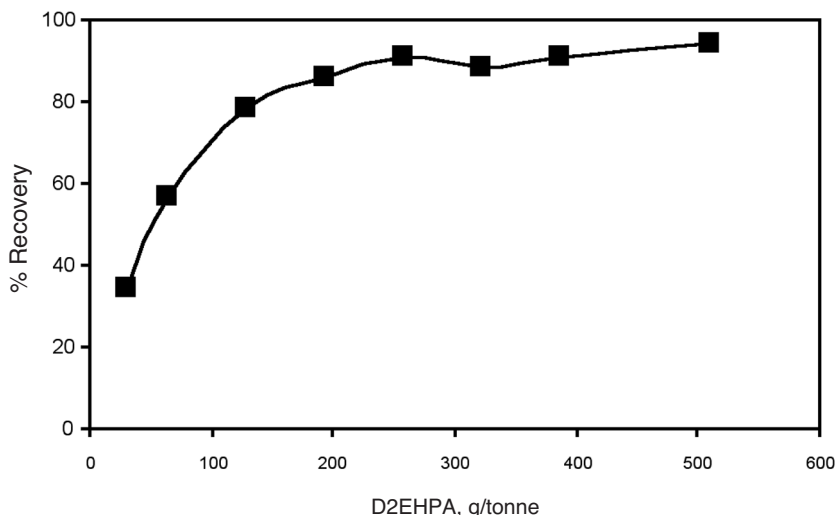


Figure 5—Recovery of hematite as a function of reagent concentration, size -75 microns, pH 4.5

# Electrokinetic and flotation studies of hematite using di(2-ethyl hexyl) phosphoric acid

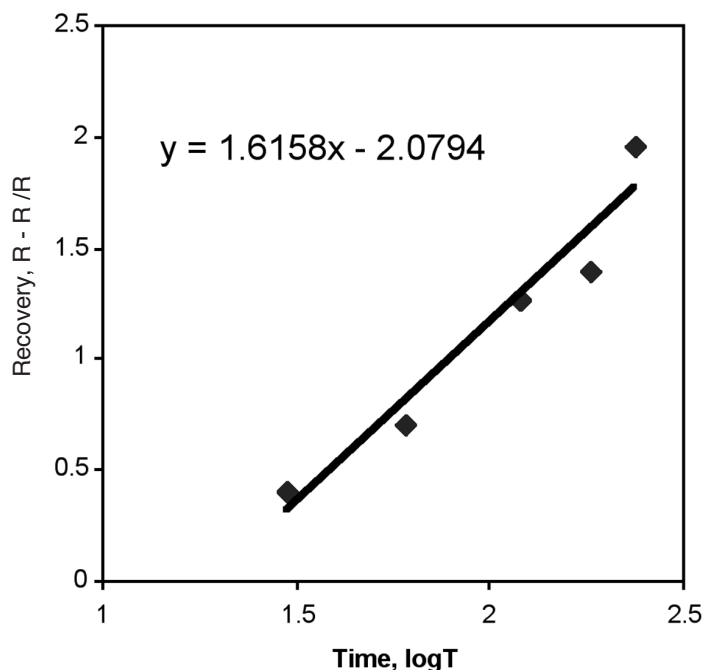


Figure 6. Flotation kinetics of hematite with D2EHPA

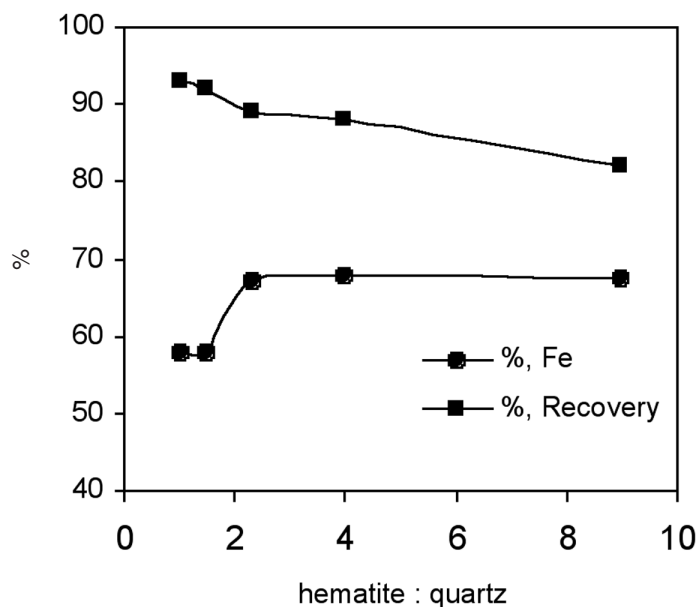


Figure 7. Flotation studies on a synthetic mixture of hematite and quartz

## Acknowledgement

The authors are thankful to Dr V.N. Misra, Director, Regional Research Laboratory, Bhubaneswar for his encouragement and kind permission to publish this paper.

## References

- NUMMELA, W. and IWASAKI, I. Iron ore flotation, *Advances in Mineral Processing, Arbitr Symposium*, P. Somasundaran (ed.), SME, 1986 pp. 309-342.
- Pradip, Beneficiation of alumina rich Indian iron ore slimes, *Metals Materials and Processes*, 6, 3, 1995, pp. 179-194.
- HOUOT, P. Beneficiation of iron ore by flotation, *IJMP* 10, 1983, p. 183
- MEECH, J.A. Feasibility of iron recovery from Mount Wright tailing material, *CIM Bulletin* 74, 1981, p. 115.
- Recovery of iron ore ultra fines by column flotation techniques, Regional Research Laboratory, Bhubaneswar, India, Internal Report, 2001.
- DAS, B. and RAVIPRASAD, A. Enhancing the flotability of Malanjkhand (India) mixed sulphide oxide copper ore with LIX-65NS, *Scandinavian Journal of Metallurgy*, 24, 1995, pp. 1-6.
- DAS, B. and RAVIPRASAD, A. Flotation of Rakha (India) copper ore with LIX-65NS, *Minerals and Metallurgical Processing*, May, 1996, pp. 61-64.
- ASHBROOK, A. W. Spectrophotometric determination of small amount of reagents used in solvent extraction processes, *Anal. Chem. Acta* .58 1972, pp. 123-129.
- PRESTON, J. S. Solvent extraction of base metals by mixtures of organophosphoric acids and non-chelating oximes, *Hydrometallurgy* 10(2), 1983, pp. 187-200.
- FUERSTEANAU, D.W. and CHANDER, S. Thermodynamics of flotation, *Advances In Mineral Processing, Arbitr Symposium*, P. Somasundaran (ed.), SME, (1986), pp. 121-136. ◆

## World first in information solutions for mining industry\*

Barloworld Equipment and Optron have formed a joint venture company known as Barloworld Optron Technologies to supply selected Caterpillar information technology products to the mining industry.

Barloworld Equipment is the sole dealer for Caterpillar equipment in southern Africa and Optron is a leading edge provider of technology based positioning and integrated information solutions and sole dealer for Trimble survey equipment in southern Africa.

The core competency of the new company will be the provision of machine positioning technology using GPS and wireless communications, linking machines in the field with business and operating systems to provide accurate, real-time operational and on-board machine information.

Barloworld Optron Technologies will supply the mining industry with leading edge technology solutions including sales, installation, service, support and training. These include the MineStar suite of integrated mining information systems and Computer Aided Earthmoving Systems (CAES) developed by Caterpillar and alliance partners, as well as Aquila drill management systems. The MineStar range incorporates MineStar Fleet Commander, MineStar Machine Health, MineStar Infrastructure, MineStar Machine tracking, MineStar Office and MineStar Production.

Barloworld Optron Technologies is headed by Dennis Kattowitz, manager of the Machine Technology Systems (MTS) Division at Optron. This division, which has had considerable success in the supply of location based technology to the mining industry, will be absorbed together with its highly qualified staff complement into Barloworld Optron Technologies.

Dennis Kattowitz says the formation of the company is a world first in terms of formally combining the strengths of a

Caterpillar dealer with a Trimble dealer to supply Caterpillar developed information technology under a single banner.

Says Chris Gibb, managing director of Barloworld Equipment Mining: 'Our strategy of providing the mining industry with total solutions is based on the supply and support of Caterpillar equipment to help the mines achieve lowest cost per tonne. Barloworld Optron Technologies brings technology that meets the same demands for optimum productivity based on complete information and data solutions that provide 24/7 availability and reliability.'

'This venture is a smart partnership that holds major benefits in terms of single source solutions and support for the mining industry and also ultimately for other sectors in which we operate.'

'The technology we supply enables a mine to develop and execute its mining strategy with absolute efficiency,' adds Optron managing director Trevor Venter. 'Through Barloworld Optron Technologies, a mine can ensure that its fleet is working optimally and productively with the use of on-board and centralized information on where machines should be working, how they should be working and their productivity levels. 'Optron has the skills and expertise required to fully support the technology, while Barloworld Equipment has the marketing strength, geographic footprint throughout southern Africa and constant presence on mining sites required to ensure the success of Barloworld Optron Technologies.' ◆

---

\* Issued by: Lynne Hancock Communications  
PO Box 180, Witkoppen 2068  
Tel: (011) 709 8471, Cell: 082 441 5813  
E-mail: lynne@lhcom.co.za

## Metallurgical equipment costs\*

Mintek has published an updated version of its guide to estimating metallurgical equipment costs.

The handbook provides formulae and variables for calculating costs for commonly used items of metallurgical process equipment in different sizes and capacities. Apart from the cost data, which have been updated to a base of March 2002, the revised guide includes several new categories of equipment, and the ranges of some items have been expanded.

The information will be useful to those involved in the costing of metallurgical processes, plant design, and

feasibility studies, by providing a means of obtaining indicative capex estimates.

Metallurgical Equipment Costs (March 2002) is published at a price of R500.00 (including VAT, South Africa) or US\$250 (overseas). ◆

---

\* Further information: Minnesh Kaliprasad,  
minneshk@mintek.co.za  
Orders: Karl Kowar, karlk@mintek.co.za