

Sulphydryl collectors in bulk and selective flotation. Part 2. Covalent dithiocarbamate derivatives

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SYNOPSIS

The effect of varying aliphatic, olefinic, and aromatic groups in the N- and S-positions of dithiocarbamates was investigated in the selective and bulk flotation of a mixed copper-lead-zinc-iron ore. Selective and bulk flotation were found to be dependent on the type of functional group, as well as on the position of substitution.

SAMEVATTING

Die effek van verskillende alifatiese, olefiniese en aromatiese groepe in die N- en S-posisies van ditiokarbamate is ondersoek in die selektiewe- en massaflottasie van 'n gemengde koper-, lood-, sink- en yster-erts. Daar is bevind dat selektiewe- en massaflottasie afhanklik is van die tipe funksionele groep, asook die posisie van die substitusie.

Introduction

The loss of valuable minerals to slimes dams following flotation must be one of the most disturbing factors in flotation-plant operations. Normally, the efficiency of the mineral liberation, and the selectivity and efficiency of the collection, can account for these losses. Pursuant with efficient recovery, the chemicals involved in flotation take on a pivotal position. In particular, the molecular structures of the flotation chemicals become important criteria in the prediction, design, and selection of flotation chemicals.

For the collection of metal sulphides, the collectors are normally sulphydryl. There are many factors that influence the ability of a collector molecule to become attached to a mineral, and there are many factors that influence the ability of a particle to remain attached to a bubble. Eventually these two become, not only the rate-determining, but also the recovery-determining, steps¹.

The molecules of sulphydryl collectors normally contain at least one sulphur atom that is involved in bonding to the mineral surface. In an earlier study¹, the role of sulphur and oxygen atoms in the polar part of ionic collectors was investigated. Another structural element that is commonly present is nitrogen. The non-polar group is generally aliphatic, olefinic, or aromatic. A number of researchers, such as Livshits and Kitaeva², Bogdanov *et al.*^{3,4}, Glembotskii⁵, Ackerman *et al.*⁶, and Jiwu *et al.*⁷, have studied various covalent structures in flotation. Several of these were dithiocarbonate derivatives. Ackerman *et al.*⁶ investigated O- and N-substituted thionocarbamates, and Livshits and Kitaeva² studied dithiocarbonate esters. Jiwu *et al.*⁷ reported on a diethyl dithiocarbamate, and Coetzer and Davidtz⁸ on various trithiocarbonate esters.

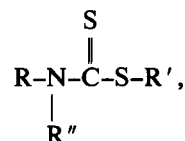
A common route for the synthesis of thionocarbamates

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is from a xanthate and an amine⁹. In the investigation reported here, the xanthate portion was replaced by a trithiocarbonate, and the resultant molecules differed from the trithiocarbonate esters in that a sulphur molecule was replaced by a nitrogen molecule. The general structure is therefore



where R, R', and R'' are aliphatic, olefinic, and aromatic groups respectively. In the investigation, the effects of various N- and S-substitutions of these groups on the selective and bulk flotation of an ore containing copper, lead, zinc, and iron were studied¹⁰.

Experimental

Materials

A mixed sulphide ore containing chalcopyrite, galena, sphalerite, pyrrhotite, and pyrite was used. It was run-of-mill ore from the Black Mountain Mineral Development Company at Aggeneys in the Cape Province. The average mineralogical composition is listed in Table I.

TABLE I
AVERAGE COMPOSITION OF THE ORE

Mineral	Occurrence %
Chalcopyrite	2
Galena	11
Sphalerite	6
Pyrrhotite	5
Pyrite	2
Magnetite	45
Gangue minerals*	29

* Such as quartz, garnet, apatite, amphibole, and biotite¹¹.

The collector molecules were synthesized in the laboratory, and there were five major classes. The basic molecule was the dithiocarbamate molecule $\text{RNHC(S)SR}'$. There are three possible positions for substitution: one on the sulphur and two on the nitrogen atom. One and two organic groups on the nitrogen respectively produce mono- and di-substituted dithiocarbamates (DTCBs).

Class 1. These were monosubstituted with an ethyl group attached to the nitrogen. The R' group varied as isopropyl (iC_3), isobutyl (iC_4), normal butyl (nC_4), allyl, and benzyl.

Class 2. This was the same as Class 1 except that the ethyl group on the nitrogen was replaced by isopropyl.

Class 3. A benzyl group was used in the monosubstitution. The non-polar groups were iC_3 and iC_4 .

Class 4. These molecules were diethyl substituted on the nitrogen, and the S-substitution was iC_3 and iC_4 .

Class 5. The N-substitution was diisopropyl, and the S-substitution was iC_3 and iC_4 .

These molecules are listed in Table II. Mass spectra analysis revealed that all the abovementioned molecules are consistent with their basic formulae. The purities of these molecules were found to be better than 85 per cent.

Flotation Procedure

All the tests simulated the copper rougher circuit and were conducted in a 2,4-litre Denver flotation cell. The 200 kg of ore used as rod-mill feed was crushed to minus $1700 \mu\text{m}$ and then homogenized. Samples of this ore of 1 kg each were milled in a laboratory stainless-steel rod mill for 20 minutes to an average grain distribution size of 66 per cent minus $75 \mu\text{m}$, and $50 \mu\text{mol}$ of collector (i.e. $8,15 \times 10^{-3} \text{ kg/t}$ for N-ethyl-S-isopropyl DTCB or $11,1 \times 10^{-3} \text{ kg/t}$ for N-diisopropyl-S-isopropyl DTCB), and 500 ml of tapwater were added to the mill. After aeration and activation with 0,128 kg/t of sulphurous acid (0,8 per cent solution), a further $100 \mu\text{mol}$ of collector and $10 \mu\text{l}$ of frother were added, followed by further conditioning. All the flotation tests were carried out at a pH value of 6,5 and a slurry density of 33 per cent. Three

concentrates were collected after 0,5, 1, and 2 minutes respectively. Owing to the care that one has to take to remove the same amount of water from as near as possible equivalent froth depth, the liquid and froth level were carefully maintained and the froth was removed by use of a handscraper at regular intervals that were consistent for all experiments. The concentrates and tailings were analysed for copper, lead, zinc, and iron. Three tests were carried out for each collector under investigation, and the recovery results were obtained from the mean of these results for each collector.

Results and Discussion

The results are reported in Table III. The selectivity quotient¹⁰ is defined as the percentage cumulative recovery of a particular mineral divided by the mean of the sum of the percentage cumulative recoveries of the other three minerals.

N-Monosubstitutions

In Classes 1 and 2, short-chain C_2 and iC_3 were monosubstituted in the N-position. The replacement of C_2 by iC_3 in this position consistently increased the recovery of all the minerals, although this was not the case with the allyl and benzyl substituents. However, the copper selectivities were better with N- C_2 substitution. This is to be expected since selectivity arises from particle-to-bubble attachment, and the higher relative density of galena results in the particle falling off the bubble. In other words, critical hydrophobicity was not reached with the C_2 group. These findings are consistent with those of Ackerman *et al.*⁶, in which N-substituted alkyl chains of increasing length resulted in decreased selectivity for thionocarbamates. Compared with the average values for the various substitutions, the selectivity for copper is reduced as a result of increased lead, zinc, and iron collection. The exception is the allyl compound, which will be discussed under double bonds in the S-position.

The N-monosubstituted collectors showed good bulk mineral-recovery characteristics for copper. Again, the extra carbon on the aliphatic group increased the collecting power considerably. In previous studies, iC_4 groups were found to favour copper collection¹.

TABLE II
DTCB COLLECTOR MOLECULES USED

Collector	Formula	Abbreviation
N-ethyl-S-isopropyl DTCB	$\text{CH}_3\text{CH}_2\text{NHC(S)SCH}(\text{CH}_3)_2$	N- C_2 -S- iC_3
N-ethyl-S-isobutyl DTCB	$\text{CH}_3\text{CH}_2\text{NHC(S)SCH}_2\text{CH}(\text{CH}_3)_2$	N- C_2 -S- iC_4
N-ethyl-S-nbutyl DTCB	$\text{CH}_3\text{CH}_2\text{NHC(S)SCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$	N- C_2 -S- nC_4
N-ethyl-S-allyl DTCB	$\text{CH}_3\text{CH}_2\text{NHC(S)SCH}_2\text{CH}=\text{CH}_2$	N- C_2 -S-All
N-ethyl-S-benzyl DTCB	$\text{CH}_3\text{CH}_2\text{NHC(S)SCH}_2\text{C}_6\text{H}_5$	N- C_2 -S-Bzl
N-isopropyl-S-isopropyl DTCB	$(\text{CH}_3)_2\text{CHNHC(S)SCH}(\text{CH}_3)_2$	N- iC_3 -S- iC_3
N-isopropyl-S-isobutyl DTCB	$(\text{CH}_3)_2\text{CHNHC(S)SCH}_2\text{CH}(\text{CH}_3)_2$	N- iC_3 -S- iC_4
N-isopropyl-S-allyl DTCB	$(\text{CH}_3)_2\text{CHNHC(S)SCH}_2\text{CH}=\text{CH}_2$	N- iC_3 -S-All
N-isopropyl-S-benzyl DTCB	$(\text{CH}_3)_2\text{CHNHC(S)SCH}_2\text{C}_6\text{H}_5$	N- iC_3 -S-Bzl
N-benzyl-S-isopropyl DTCB	$\text{C}_6\text{H}_5\text{CH}_2\text{NHC(S)SCH}(\text{CH}_3)_2$	N-Bzl-S- iC_3
N-benzyl-S-isobutyl DTCB	$\text{C}_6\text{H}_5\text{CH}_2\text{NHC(S)SCH}_2\text{CH}(\text{CH}_3)_2$	N-Bzl-S- iC_4
N-diethyl-S-isopropyl DTCB	$(\text{CH}_3\text{CH}_2)_2\text{NC(S)SCH}(\text{CH}_3)_2$	N-di C_2 -S- iC_3
N-diethyl-S-isobutyl DTCB	$(\text{CH}_3\text{CH}_2)_2\text{NC(S)SCH}_2\text{CH}(\text{CH}_3)_2$	N-di C_2 -S- iC_4
N-diisopropyl-S-isopropyl DTCB	$[(\text{CH}_3)_2\text{CH}]_2\text{NC(S)SCH}_2(\text{CH}_3)_2$	N-dii C_3 -S- iC_3
N-diisopropyl-S-isobutyl DTCB	$[(\text{CH}_3)_2\text{CH}]_2\text{NC(S)SCH}_2\text{CH}(\text{CH}_3)_2$	N-dii C_3 -S- iC_4

TABLE III
CHEMICAL ANALYSES OF CONCENTRATES

Flotation agents	Cumulative recoveries %				Selectivity quotient				Cumulative grades %			
	Cu	Pb	Zn	Fe	Cu	Pb	Zn	Fe	Cu	Pb	Zn	Fe
<i>Class 1</i>												
N-C ₂ -S-iC ₃	81,2	37,5	11,2	3,4	4,7	1,2	0,27	0,11	7,6	46,0	4,6	11,0
N-C ₂ -S-iC ₄	88,7	21,5	6,6	4,2	8,2	0,7	0,17	0,13	7,4	36,9	3,9	22,8
N-C ₂ -S-nC ₄	82,2	35,3	15,9	13,8	3,8	1,0	0,36	0,37	4,3	24,2	3,4	25,7
N-C ₂ -S-All	78,7	20,0	8,4	7,3	6,6	0,6	0,24	0,23	10,1	24,1	3,1	24,0
N-C ₂ -S-Bzl	82,8	36,7	10,7	7,9	4,5	1,1	0,25	0,23	6,1	33,3	3,2	18,0
<i>Class 2</i>												
N-iC ₃ -S-iC ₃	92,8	47,9	20,9	11,9	3,5	1,2	0,41	0,28	5,0	33,5	4,9	23,2
N-iC ₃ -S-iC ₄	94,6	58,2	30,3	18,6	2,7	1,2	0,53	0,39	2,5	30,7	4,6	25,8
N-iC ₃ -S-All	74,8	12,5	5,8	6,5	9,1	0,4	0,19	0,22	8,4	19,6	2,8	27,6
N-iC ₃ -S-Bzl	78,1	30,9	13,4	15,2	3,9	0,9	0,32	0,43	4,3	20,2	3,1	28,6
<i>Class 3</i>												
N-Bzl-S-iC ₃	82,6	46,0	24,1	18,3	2,8	1,1	0,49	0,44	3,8	24,0	4,4	26,5
N-Bzl-S-iC ₄	89,9	57,6	38,3	20,7	2,3	1,2	0,68	0,42	3,3	26,2	6,0	24,7
<i>Class 4</i>												
N-diC ₂ -S-iC ₃	81,4	35,8	13,1	13,0	4,0	1,0	0,30	0,36	4,4	24,5	3,0	27,8
N-diC ₂ -S-iC ₄	84,2	84,2	52,7	8,7	1,7	1,7	0,89	0,18	3,3	40,7	10,7	12,8
<i>Class 5</i>												
N-diiC ₃ -S-iC ₃	82,1	40,4	12,3	6,0	4,2	1,2	0,29	0,18	5,8	34,3	4,2	17,1
N-diiC ₃ -S-iC ₄	85,0	30,1	8,0	4,5	6,0	0,9	0,20	0,14	7,1	39,3	3,4	14,8

N-Disubstitutions

The N-diC₂-S-iC₄ DTCB is a good bulk collector and, once again, the iC₄ was a stronger copper collector. Increased bulk collection was also noted with aliphatic substitution in the S-position of TTC esters⁸. Compared with the first two members of Class 2, the added carbon in the N-position in Class 4 did not improve the copper recovery. However, the lead and zinc recoveries were strongly influenced in N-diC₂-S-iC₄ DTCB. The diisopropyl compounds were not better bulk collectors, but there did appear to be less selectivity for zinc and iron. This is possibly a steric effect.

S-Substitutions

In every class, a replacement of iC₃ by iC₄ in the S-position resulted in improved copper recovery. This was also true for most of the other minerals. Ackerman *et al.*⁶ also found that, when iC₃ was replaced by iC₄ in the O-position of thionocarbamates, the recovery improved. This was probably largely due to the increased interaction of the aliphatic group with water, since it is doubtful whether oxygen atoms are involved in surface bonding in sulphide minerals¹.

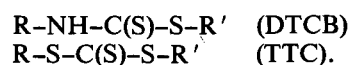
Double Bonds in the S-Position

In Classes 1 and 2, allyl and benzyl substitution occurred in the S-position. There was a general tendency for reduced copper and lead recoveries. There were also frothing problems with the allyl collectors, i.e. they tended to break the froth, and it will be noticed that the copper grades were high. Although mass pulls were not recorded, poor frothing conditions normally lead to high grades. The substitution of a benzyl group in the S-position did not influence the recoveries or selectivities;

however, in the N-position, it showed strong bulk-collecting properties.

Comparison of Covalent Dithiocarbamates and Trithiocarbonates

The basic structural units of the DTCBs and TTCs differ in that the nitrogen in the N-position is replaced by a sulphur atom. The structures are therefore



The effect of variations in R and R' is given in Table IV and Figs. 1 and 2.

In the case of copper recovery, the DTCBs and TTCs were virtually identical. The individual results and the general trends were also similar. Therefore, whether the molecule contained N or S, it did not have an effect on the copper collection. However, the alkyl TTCs were better bulk collectors. Thus, the effect of the alkyl group was more pronounced on the sulphur atom than on the nitrogen atom. The presence of double bonds tended to increase the selectivity for copper, and the N-substituted benzyl groups were less-selective collectors than the corresponding S-substituted collectors. For both copper and lead, the general trend in recovery was iC₄ > iC₃ > benzyl > allyl in the S-substituted position.

The best zinc collectors were the alkyl TTCs and benzyl DTCBs. The general trend for the recovery of zinc followed the same sequence as for copper and lead, viz iC₄ > iC₃ > benzyl > allyl. Although the iron collection was generally low, it appeared that, when both a nitrogen atom and a benzyl group were present, the iron collection improved. As pointed out by Coetzer and Davidtz⁸,

TABLE IV
CUMULATIVE RECOVERIES AND SELECTIVITIES FOR SELECTED
TTCs AND DTCBs

Flotation agents	Cumulative recoveries, %				Selectivity quotient			
	Cu	Pb	Zn	Fe	Cu	Pb	Zn	Fe
S-iC ₃ -S'-R TTC								
R = iC ₃	92,1	80,1	21,5	6,8	2,6	2,00	0,36	0,17
iC ₄	91,6	86,3	52,0	10,1	1,9	1,70	0,83	0,20
Allyl	71,9	14,6	7,8	7,7	7,2	0,50	0,24	0,26
Benzyl	79,1	18,8	12,0	10,6	5,7	0,56	0,33	0,31
N-iC ₃ -S-R DTCB								
R = iC ₃	92,8	47,9	20,9	11,9	3,5	1,15	0,41	0,28
iC ₄	94,8	58,2	30,3	18,6	2,7	1,22	0,53	0,39
Allyl	74,8	12,5	5,8	6,5	9,1	0,43	0,19	0,22
Benzyl	78,1	30,9	13,4	15,2	3,9	0,87	0,32	0,43
S-Bzl-S'-R TTC								
R = iC ₃	79,1	18,8	12,0	10,6	5,7	0,56	0,33	0,31
iC ₄	79,9	17,2	9,4	6,1	7,3	0,54	0,27	0,19
N-Bzl-S-R DTCB								
R = iC ₃	82,6	46,0	24,1	18,3	2,8	1,11	0,49	0,44
iC ₄	89,9	57,6	38,3	20,7	2,3	1,16	0,68	0,42

the total iron present was dominated by the presence of magnetite, which is an iron oxide. It is unlikely that sulphhydryl collectors would bond to this iron oxide. The problem, however, at these high concentrations of magnetite is that entrained particles can influence recoveries and, unless the amount of water carried over by the froth is constant, entrainment of gangue can influence the results. Therefore, less emphasis has been placed here on the iron results, which is also the reason for the low percentage recoveries.

Conclusions

There are a number of consistencies in the results reported, and the following general conclusions are drawn.

- (1) Longer aliphatic chains in the N-position of DTCBs increases recoveries but decreases selectivity.
- (2) The higher selectivities found with the C₂ monosubstitution, in general, does not extend to disubstitution, whereas the reversed situation was found for the iC₃ monosubstitution.
- (3) The iC₄ S-substitutions are better copper collectors than their iC₃ counterparts.
- (4) Allyl and benzyl S-substitutions have a negative effect on the flotation of copper but allyl substitutions improve the selectivity for copper.
- (5) Benzyl substitution in the N-position enhances bulk collection.
- (6) The presence of benzyl and nitrogen groups appears to increase the iron collection.
- (7) For the recovery of copper, the TTCs and DTCBs behaved the same for similar functional-group substitution.
- (8) Copper, lead, and zinc recoveries followed the same general trend of iC₄ > iC₃ > benzyl > allyl, and the selectivities appeared to have been reversed.

Acknowledgements

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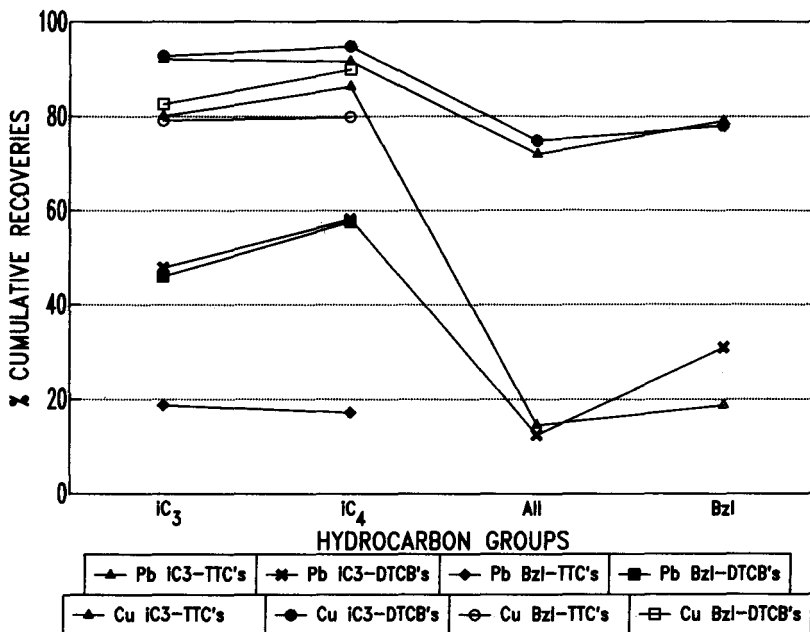
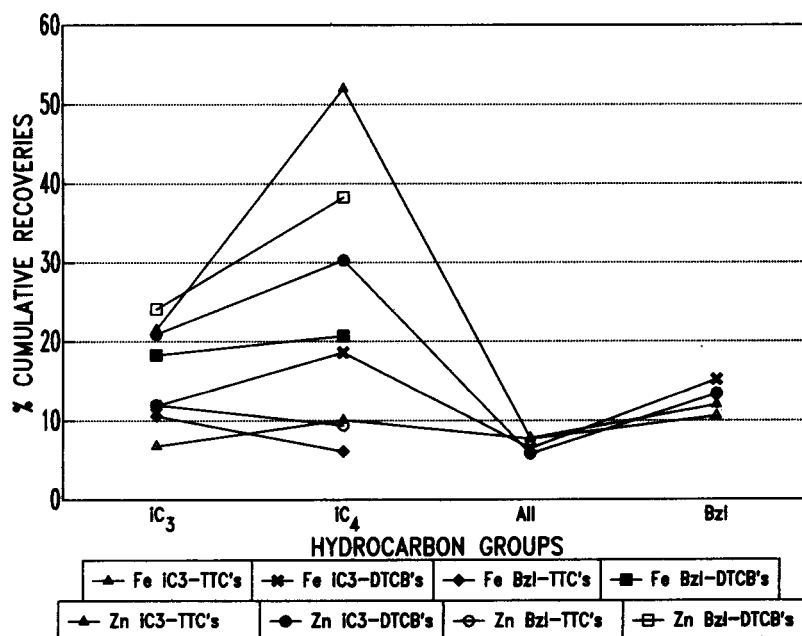


Fig. 1—Copper and lead recoveries as influenced by the hydrocarbon substituents for selected covalent TTCs and DTCBs

Fig. 2—Zinc and iron recoveries as influenced by the hydrocarbon substituents for selected covalent TTCs and DTCBs



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Superconductors

The even-year International Cryogenic Materials Conference, organized jointly with the Deutsche Gesellschaft für Metallkunde e.V., is to be held in Garmisch-Partenkirchen (FRG) from 9th to 11th May, 1990.

The aim of the Conference is to provide an up-to-date comprehensive assessment of the various aspects—recent progress and unsolved problems—of materials research and development in the exciting area of high-temperature superconductors (HTSC).

The Conference topics will be as follows:

- Synthesis, Processing, and Shaping of Bulk- and Thin-film Oxide Superconductors
- Structure and the Effect of Substitutions
- Microstructure of HTSC and Its Control
- Influence of Microstructure and Structure on Physical and Chemical Properties
- Critical Transport Currents.

Those who wish to present papers should so indicate as soon as possible and submit two copies of a camera-ready extended abstract in English to the Conference Secretariat by 15th December, 1989. The abstract (one

page) should begin with the title of the paper, followed by the authors' names and affiliations. It may include a figure and/or a table, but all should fit within a 16 × 24 cm (6-¼ × 9-½ inch) rectangle. As guidance to the Programme Committee, who will make the final decision, authors should state whether they prefer an oral or poster presentation.

Authors will be notified of the acceptance of papers and of further information for the full papers as soon as possible.

For abstracts, papers, poster presentations, and lectures, the language to be used is English. The full-length papers presented at the Conference will be reviewed and published in the Conference proceedings.

Further information is available from the following address, to which abstracts should also be directed:

Deutsche Gesellschaft für Metallkunde
Adenauerallee 21
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West Germany.
Telephone: (06171) 4081 Fax: (06171) 52554.

Forensic engineering and litigation*

The increasing incidence of litigation over liability for product/engineering failures, legal defence strategies, and the role of fracture experts were among the forensic-engineering issues discussed at the Third National Conference on Fracture, which was held in Johannesburg recently.

The Conference was held under the auspices of the Fracture Research Group of the Department of Metallurgy and Materials Engineering at the University of the Witwatersrand in conjunction with the South African Institution of Mechanical Engineers.

According to Dr Neil James, Director of the Fracture Research Group, forensic engineering is a relatively new term in engineering literature but relates to an old problem: the application of the principles of engineering analysis (especially in failure analysis) to the determination of responsibility or culpability for a state of events.

In his keynote address on forensic engineering, Professor Campbell Laird of the Department of Materials Science and Engineering at the University of Pennsylvania in Philadelphia, USA, said that 'in almost any branch of life, potential or kinetic energy suddenly released in uncontrolled fashion by mechanical failure causes immense losses in property damage, personal injury, and death.

'Such failures can result in costly litigation, especially in countries where the economic and legal systems are organized to make litigation potentially rewarding.'

According to Professor Laird, a manufacturer faced with a lawsuit that involves a mechanical failure generally has to choose between three possible lines of defence or a combination of these defences:

- (a) the nonsense gambit, where the defendant takes the position that the allegation of defectiveness by the plaintiff as a cause of the incident is fraudulent or nonsensical;
- (b) the misinterpretation gambit, where the defendant asserts the integrity of his product and assigns another

cause to the catastrophe, arguing that the failure occurred during the incident rather than before it as the initiating event;

- (c) the pointing-finger gambit, where the defendant accepts his product as the causative agent but still maintains the integrity of his product in the as-manufactured condition, and claims that interference or abuse by another party has rendered his product defective after being introduced into service.

Addressing the Conference on the role of the fracture expert in failure analysis, Dr James of Wits said that, in many instances, it was advisable to seek the opinion of an expert in order to obtain definitive information on the causes and mechanisms of fracture.

'In cases of major structural failure it is quite common for each party involved to retain its own expert. The principals in such a case could be the manufacturer, the owner of the equipment, and their respective insurance companies.

'This proliferation of expertise sometimes leads to a diversity in opinions and subsequent litigation. The moral of this is probably that the definitive expert opinion is only that which is accepted by the court. Retaining an independent expert has the additional advantage of eliminating the subjectivity that could creep into an in-house failure investigation.'

Dr James divided the major causes of failure of mechanical equipment or structures into three categories: design inadequacies, material processing or fabrication problems, and environmental or service deterioration. The major factors that should be examined by a failure investigation include loading, material, shape, and environment.

A practitioner in the field of failure analysis required an interdisciplinary background, he said, pointing out that expertise in design, stress analysis, fatigue and fracture (including experience of fractography), welding problems, and physical metallurgy were required to address these questions adequately.

'It may often be necessary to involve other consultants with particular expertise in such areas as non-metallic materials, corrosion, or wear in order to arrive at a complete understanding of the sequence of events which led to the failure.'

Dr James concluded that not the least of the benefits of a failure analysis was that of technology transfer. 'The fracture knowledge gained by workshop and line supervisors as a result of a simple investigation and report is often beneficial in avoiding failures in similar classes of component.'



Dr Neil James

* Press release from Lynne Hancock Communications (P.O. Box 1564, Parklands 2121).