

Sulphydryl collectors in bulk and selective flotation. Part 1. Covalent trithiocarbonate derivatives

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SYNOPSIS

The substitution of functional groups in covalent trithiocarbonate collector molecules influences the selective and non-selective bonding of collector molecules to mineral surfaces, as well as flotation efficiency in a mixed sulphide ore. These effects were concluded to be related to the metal-sulphide solubility products and electron densities on the sulphur atoms in the collector molecules.

SAMEVATTING

Funksionele groep-substitusie in kovalent gebinde tritiokarbonaat versamelaar-molekule beïnvloed die selektiewe en nie-selektiewe binding van versamelaar-molekule met mineraal-oppervlakke asook die flottasie-effektiwiteit in 'n gemengde sulfied-erts. Hierdie effekte kan in verband gebring word met die oplosbaarheidsprodukte van die metaal-sulfiede en die elektrondigtheid op die swawelatome in die versamelaar-molekule.

Introduction

In the development of new chemicals for flotation, consideration should be given to the many structural features of molecules that influence flotation behaviour. Some of these are the structural elements, such as oxygen, nitrogen, and sulphur, and whether the molecule is ionic or covalent, whether it contains double bonds, whether it contains aromatic or aliphatic groups (which affect electron densities on the sulphur atoms), and to which elements these groups are bonded. It is a combination of these structural properties of the molecule, as well as the nature of the mineral, that eventually determines whether a collector molecule is to behave as a selective or a bulk collector.

The solid-state properties that affect the metal-sulphide (M-S) interactions in the mineral can, as a first approximation, be used in the prediction of collector-surface interactions. The mineralogical data required for the prediction and understanding of collector-surface interactions are not well understood. In some cases, because of preferred cleavage planes, metal cations are not necessarily exposed at surfaces, and these cations are essential for the collector to bond to. Such an extreme example is valleriite, where the exposed surfaces of this layered-lattice copper sulphide mineral are the octahedral oxide and hydroxide basal planes¹, and sulphydryl collectors do not interact with these. If the minerals are simple M-S compounds, then the solubility products are an index of the M-S bond, which is, in turn, an indication of collector-mineral bonding. As a first approximation, the minerals with the lowest metal-sulphide solubility products have the greatest affinity for the molecules of sulphydryl collectors². It is known that mineralogical composition determines hardness, as well as the eventual

particle-size distribution after milling. Very small particles are distorted in structure, and can be amorphous subsequent to milling. In larger particles, the first few external atomic layers may also be amorphous and therefore not reflect the physical properties of the crystalline interior.

Another observation is that molecules containing all the sulphur atoms in covalent structures, for example Minerec 2030, i.e. N-ethyl-O-isopropyl thionocarbamate, are inclined to exhibit selectivity more readily than those molecules containing ionic sulphur atoms³⁻⁵. In a previous study⁶, ionic trithiocarbonates (TTCs) were investigated to determine the role of one, two, and three sulphur atoms associated with a terminal carbon atom on flotation kinetics and recovery. The general structure of the molecules was RSC(S)SNa. The mechanisms by which these TTCs become bonded to the surface of minerals are not understood, but all three sulphur atoms appear to be involved in bonding to the surface. Compared with xanthates, which contain one fewer sulphur atom, lower molar dosages are required to achieve the same rates and recoveries.

The efficiency of sulphide-mineral collection is dependent on whether one is dealing with selective and/or bulk flotation. Therefore, structural features of the sulphydryl collector molecules are pivotal to the effective recovery of sulphide minerals. Here, *selectivity* refers to the preferential collection of one sulphide mineral in the presence of others. *Bulk flotation* refers to the indiscriminate recovery of more than one sulphide mineral.

A number of methods have been employed to report flotation and selectivity data. The following are some of these: grade-recovery relationships⁷, collection and selection efficiencies⁸, release curves^{9,10}, selectivity parameters¹¹, competitive recoveries¹², selectivity indexes¹³, selectivity factors¹⁴, and selectivity quotients¹⁵.

In the kinetics and mechanisms involving selectivity or bulk collection, two rate-determining steps are possible: the first involves the attachment of the collector to the mineral surface; and the second is the attachment of the

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activated particle to the bubble carrier. Selectivity by surface bonding of the collector molecule arises from specific interactions between the collector and the surface. Therefore, specific interactions between the polar part of the molecule and the mineral surface determine this type of selectivity. In the second type of selectivity, the principal rate-determining step for collectors already attached to mineral surfaces is that of particle to bubble attachment⁶. When the hydrodynamics in the flotation cell are always kept constant, the collision frequency between particle and bubble is constant. It is the return to the aqueous phase of an entrained particle from residing at the interface between the aqueous phase of the slurry and the gaseous phase of the bubble that requires the rupture of hydrogen bonds in the bulk water phase, and the restructuring of this water into ordered clathrates around the hydrophobic organic groups of the collector molecules. These latter interactions are the energy-demanding reactions, and they are the activated processes that determine both rate and recovery.

In this study, the relative selectivities of various covalent trithiocarbonate (TTC) derivatives or esters were investigated¹⁵. Three different classes of collectors of covalent bond character were synthesized. They are commonly known as trithiocarbonate derivatives, or thio-xanthate derivatives or esters, with the general formula $RSC(S)SR'$, R and R' being aliphatic, olefinic, and aromatic hydrocarbon groups.

Experimental

Materials

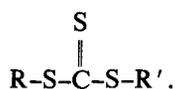
The minerals used were obtained from the Black Mountain Mineral Development Company at Aggeneys, Cape Province. The deposit is a mixed orebody of copper, lead, zinc, and iron sulphides, with an average mineralogical composition as shown in Table I.

TABLE I
AVERAGE COMPOSITION OF 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 biotite¹⁶.

The three classes of collector molecules synthesized had the following general structure (R and R' were varied):



- (1) The R hydrocarbon group contained an allyl group that furnished the double-bond character. The allyl group ($\text{CH}_2 = \text{CHCH}_2-$) was held constant, and the R' group was varied as isopropyl ($i\text{C}_3$), isobutyl ($i\text{C}_4$), normal butyl ($n\text{C}_4$), and benzyl ($\text{C}_6\text{H}_5\text{CH}_2-$).

This class of molecules has the general formula $\text{CH}_2 = \text{CHCH}_2\text{SC(S)SR}'$.

- (2) The R hydrocarbon group contained a benzyl group, thus having aromatic character. The other substituted non-polar group, R', was also $i\text{C}_3$, $i\text{C}_4$, $n\text{C}_4$, and benzyl. This class of molecules has the general formula $\text{C}_6\text{H}_5\text{CH}_2\text{SC(S)SR}'$.
- (3) For the aliphatic $i\text{C}_3$ -TTC derivatives, the substituted non-polar group, R', was $i\text{C}_3$ and $i\text{C}_4$, and it had the general formula $i\text{C}_3\text{SC(S)SR}'$. For the aliphatic $i\text{C}_4$ -TTC derivative, only the $i\text{C}_4$ group, with the formula $i\text{C}_4\text{SC(S)SiC}_4$, was synthesized.

The above-mentioned molecules were analysed by mass spectroscopy, and were found to be consistent with their basic formulae. The purities of these molecules were found to be better than 85 per cent. The various TTC esters are listed in Table II.

Flotation Procedure

All the tests simulated the copper rougher circuit as used by Black Mountain Mineral Development Company, and were conducted in a 2,4-litre Denver flotation cell. The 200 kg of ore used as rodmill feed was crushed to minus 1700 μm and then homogenized. Samples of this ore of 1 kg each were milled in a laboratory stainless-steel rodmill for 20 minutes to an average grain distribution size of 66 per cent minus 75 μm , together with 50 μmol of collector (i.e. 96×10^{-3} kg/t for S-allyl-S'-isopropyl TTC or $14,5 \times 10^{-3}$ kg/t for S-benzyl-S'-benzyl TTC) and 500 ml of tapwater. After aeration and activation with 0,128 kg/t of sulphurous acid (0,8 per cent solution), a further 100 μmol of collector and 10 μl of frother were added, followed by further conditioning. All the flotation tests were carried out at pH 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 levels were carefully maintained and the froth was removed by means of a handscraper at regular intervals that were consistent in all the 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 taken as the mean of these results for each collector.

Results and Discussion

The results are reported in Table III. The selectivity quotient¹⁵ 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; for example, the copper, lead, zinc, and iron cumulative recoveries were 71,9, 14,6, 7,5, and 7,7 per cent respectively for the S-All-S'- $i\text{C}_3$ TTC derivative. The copper selectivity quotient was then calculated as follows: $\text{Cu (SQ)} = 71,9 / [(14,6 + 7,5 + 7,7) / 3] = 7,2$.

Class I

As a group of chemicals, the allyl derivatives of the TTCs had the highest selectivities for copper. This is seen from the selectivity quotients and from Fig. 1. The higher selectivity for copper is actually the result of a lower

TABLE II
TTC COLLECTOR MOLECULES USED

Collector	Formula	Abbreviation
S-allyl-S'-isopropyl TTC	$\text{CH}_2 = \text{CHCH}_2\text{SC}(\text{S})\text{SCH}(\text{CH}_3)_2$	S-All-S'-iC ₃
S-allyl-S'-isobutyl TTC	$\text{CH}_2 = \text{CHCH}_2\text{SC}(\text{S})\text{SCH}_2\text{CH}(\text{CH}_3)_2$	S-All-S'-iC ₄
S-allyl-S'-nbutyl TTC	$\text{CH}_2 = \text{CHCH}_2\text{SC}(\text{S})\text{SCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$	S-All-S'-nC ₄
S-allyl-S'-benzyl TTC	$\text{CH}_2 = \text{CHCH}_2\text{SC}(\text{S})\text{SCH}_2\text{C}_6\text{H}_5$	S-All-S'-Bzl
S-benzyl-S'-isopropyl TTC	$\text{C}_6\text{H}_5\text{CH}_2\text{SC}(\text{S})\text{SCH}(\text{CH}_3)_2$	S-Bzl-S'-iC ₃
S-benzyl-S'-isobutyl TTC	$\text{C}_6\text{H}_5\text{CH}_2\text{SC}(\text{S})\text{SCH}_2\text{CH}(\text{CH}_3)_2$	S-Bzl-S'-iC ₄
S-benzyl-S'-nbutyl TTC	$\text{C}_6\text{H}_5\text{CH}_2\text{SC}(\text{S})\text{SCH}_2\text{CH}_2\text{CH}_2\text{CH}_3$	S-Bzl-S'-nC ₄
S-benzyl-S'-benzyl TTC	$\text{C}_6\text{H}_5\text{CH}_2\text{SC}(\text{S})\text{SCH}_2\text{C}_6\text{H}_5$	S-Bzl-S'-Bzl
S-isopropyl-S'-isopropyl TTC	$(\text{CH}_3)_2\text{CHSC}(\text{S})\text{SCH}(\text{CH}_3)_2$	S-iC ₃ -S'-iC ₃
S-isopropyl-S'-isobutyl TTC	$(\text{CH}_3)_2\text{CHSC}(\text{S})\text{SCH}_2\text{CH}(\text{CH}_3)_2$	S-iC ₃ -S'-iC ₄
S-isobutyl-S'-isobutyl TTC	$(\text{CH}_3)_2\text{CHCH}_2\text{SC}(\text{S})\text{SCH}_2\text{CH}(\text{CH}_3)_2$	S-iC ₄ -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>												
S-All-S'-iC ₃	71,9	14,6	7,5	7,7	7,2	0,5	0,24	0,26	7,8	18,7	3,6	27,9
S-All-S'-iC ₄	79,3	14,8	8,9	7,8	7,6	0,5	0,26	0,24	6,4	18,3	3,5	23,6
S-All-S'-nC ₄	67,4	16,0	8,2	6,1	6,7	0,6	0,27	0,22	7,6	18,9	3,0	21,2
S-All-S'-Bzl	67,0	13,5	6,7	6,3	7,6	0,5	0,23	0,24	8,6	18,9	2,8	20,9
<i>Class 2</i>												
S-Bzl-S'-iC ₃	79,1	18,8	12,0	10,6	5,7	0,6	0,33	0,31	5,3	16,0	3,7	25,3
S-Bzl-S'-iC ₄	79,9	17,2	9,4	6,1	7,3	0,5	0,27	0,19	6,3	16,1	3,4	17,1
S-Bzl-S'-nC ₄	68,6	20,5	10,2	9,0	5,2	0,7	0,31	0,31	6,2	18,8	2,9	23,9
S-Bzl-S'-Bzl	72,1	22,9	8,1	7,2	5,7	0,8	0,20	0,25	7,5	20,9	2,8	22,6
<i>Class 3</i>												
S-iC ₃ -S'-iC ₃	92,1	80,1	21,5	6,8	2,6	2,0	0,36	0,17	4,1	52,1	4,3	10,7
S-iC ₃ -S'-iC ₄	91,6	86,3	52,0	10,1	1,9	1,7	0,83	0,20	4,1	34,9	7,3	10,8
S-iC ₄ -S'-iC ₄	80,9	29,4	12,7	7,3	4,9	0,9	0,32	0,22	7,1	24,6	3,5	15,9

affinity of the collector for lead, since the absolute lead, zinc, and iron recoveries were moderate. The allyl derivatives, and to a lesser extent the benzyl TTC derivatives, reduced the froth stability. Generally, poor froth stability implies that not all the collector molecules are attached to the mineral surface but are free in the aqueous phase. Since minerals with the lowest solubility products (Table IV) normally have the greatest bonding energy between mineral-metal cations and anions, in this case sulphide, it should be possible for the selectivity to be manipulated by modification of the nature of the sulphur atoms in the collector molecules. By alteration of the polarity or electron density on the covalently bonded sulphur atoms, it should be possible to selectively attach certain molecules to the most insoluble minerals only. In these cases, one has to conclude that collector-to-particle attachment is the selectivity-determining step.

TABLE IV
SOLUBILITY PRODUCTS OF SULPHIDE MINERALS¹⁷

Metal sulphide	K _{Sp}
Cu ₂ S	-48
PbS	-27
ZnS	-24
FeS	-17

Unfortunately, the total iron present was dominated by the presence of magnetite, which is an iron oxide. It is unlikely that sulphhydryl collectors will become attached to magnetite. However, at these high concentrations, 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 is placed on the iron-recovery results.

Class 2

The benzyl-substituted TTCs do not significantly differ from the corresponding allyl TTCs in copper recovery. However, the selectivity for copper is reduced. This is primarily due to the increased lead collection. As in the case of copper, the functional groups do not influence the lead collection, with the exception of the benzyl group in S-Bzl-S'-Bzl TTC. In ionic collectors, it has been found that an increase in the number of aliphatic groups and in the chain length⁶ results in a continuous reduction in the selectivity for copper. In these cases, the ionic sulphhydryl group caused no obvious selective interaction to occur between the collector and a particular mineral. The selectivity was therefore due to particle-bubble interactions.

Class 3

When the substituted groups R and R' were both

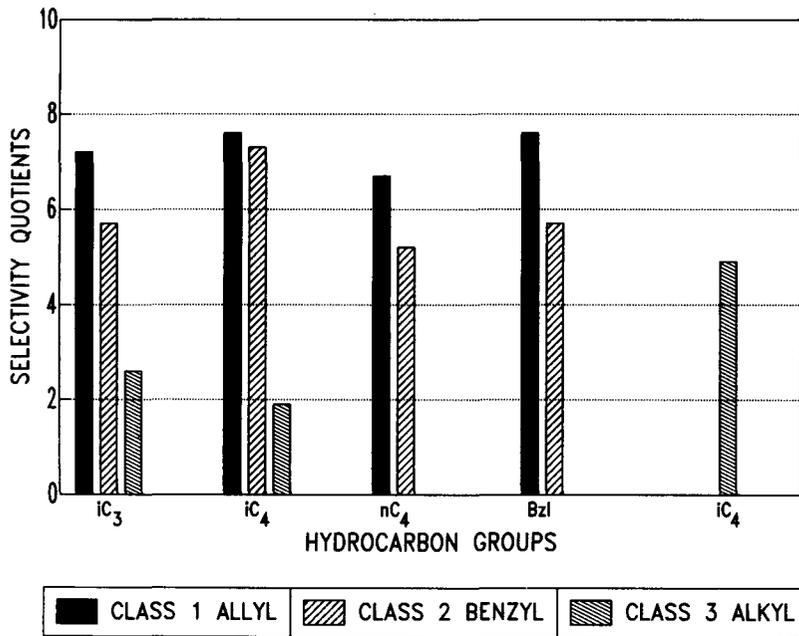


Fig. 1—Copper selectivity as influenced by hydrocarbon substituents for covalent trithiocarbonates

aliphatic and, in this case, iC_3 and/or iC_4 , unusually high recoveries of copper, lead, and even zinc were obtained (Figs. 2 and 3). Therefore, selectivity had disappeared, and typical bulk collection occurred. This means that the collector did not discriminate between copper and lead. In the previous cases where benzyl and allyl groups were associated with covalent sulphur atoms, their higher electron withdrawal could have influenced the bonding power of the collector to the surface. With aliphatic groups present, higher electron densities would occur on the sulphurs and thereby permit a stronger interaction with the metal cations.

Conclusions

The extraordinarily high bulk-collecting properties of the iC_3 and the iC_4 TTC esters imply that there is good collector-particle and particle-bubble attachment. There-

fore, one would conclude that the polar group interacts strongly with the mineral surface, and also that the non-polar group interacts weakly with water in the sense that it is excluded from the water phase.

The copper selectivities of both the allyl and the benzyl TTC derivatives show no significant change for the different hydrocarbon substituents. The allyl derivatives had higher copper selectivities than the benzyl derivatives, but both possessed high copper selectivities. Since the benzyl and allyl had strong froth-breaking tendencies, it has to be concluded that some of these chemicals remained unabsorbed and were present in the water phase of the draining lamellae. Because of the low solubility product of copper in chalcopyrite, it had enough affinity for the covalent sulphur collectors and the collector was therefore absorbed. In the case of lead, the benzyl derivatives were more selective than the allyl derivatives.

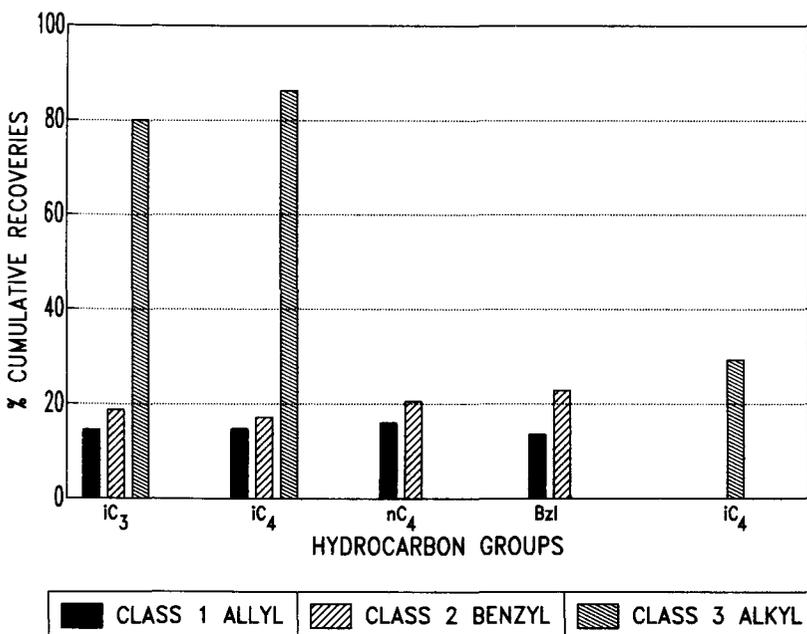
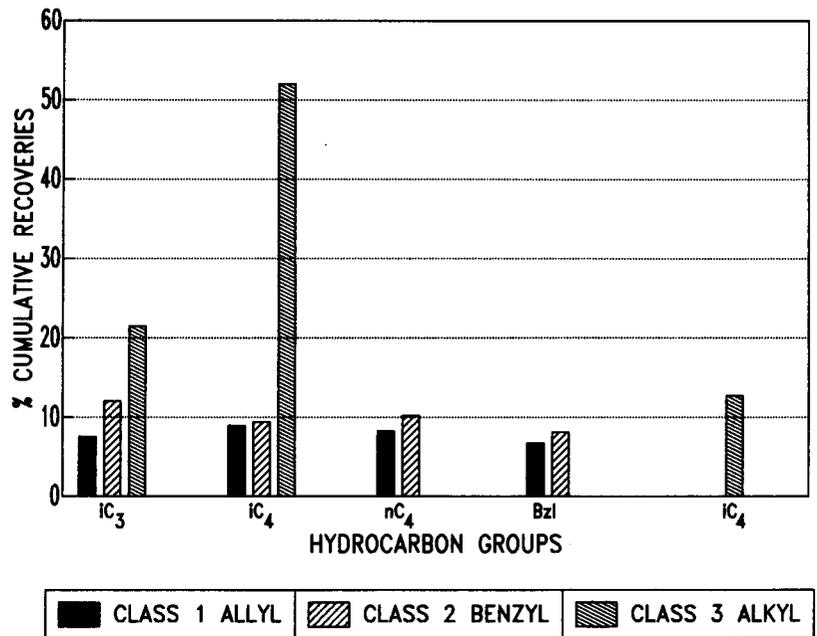


Fig. 2—Lead recoveries as influenced by hydrocarbon substituents for covalent trithiocarbonates

Fig. 3—Zinc recoveries as influenced by hydrocarbon substituents for covalent trithiocarbonates



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Compressed air in the mining industry*

'Compressed-air consumption in the South African mining industry has virtually doubled over the past 25 years, although the tons broken per rockdrill have decreased from over 600 per month to less than 350', was the disquieting conclusion of Mr Paul Deglon's Presidential Address to members of the Mine Ventilation Society on 21st June, 1989.

The topic chosen by Mr Deglon, of Rand Mines, was the role of compressed air in the mining industry, which is of interest, not only to those working in the ventilation departments on mines, but also to mine management and personnel integrally involved in production. The speech was the result of months of painstaking and methodical research, and covered the history of the use of compressed air, leading finally to current practices both legitimate and illegitimate.

Mr Deglon emphasized that, ignoring leakage and illegitimate use for ventilation, all of the compressed air consumed underground is, or should be, for the sole purpose of operating rockdrills. However, his calculations showed that the average consumption at present is 4,3 times higher than the ideal consumption, and that this is a very real cause for concern given recent technological advancements that allow for higher drilling pressures and correct thrusting.

The disturbing factors that emerged from his study of current consumption patterns indicated that the off-shift rate is often little less than the day-shift rate and, on average, accounts for about 60 per cent of the consumption over a 24-hour period. Also, the calculated drilling pattern showed that little more than 20 holes are being achieved per shift by the drilling crew. This, in turn, means that additional drills are being used to achieve the production rates, which increases the peak compressed-air requirements. More ominously, the analysis showed fairly conclusively that, during the drilling shift, the compressed air consumed by rockdrills is only a small pro-

portion of the overall consumption. The obvious conclusion is that the balance can be attributed only to wastage and illegitimate use for ventilation.

This message is particularly significant in the light of the mining industry's desperate need to find ways of reducing production costs to compensate for the low grade of the ore being recovered, the uncertainty of the gold price on the international market, and the ever-increasing cost structure on local mines. Mr Deglon warned that 'when compressed-air usage exceeds legitimate requirements, additional costs are incurred over and above the direct cost of producing compressed air, and capital has to be expended on additional compressors—not to mention a reticulation system that is of larger diameter and therefore more expensive than it need be'.

He continued by saying that 'drilling costs are perhaps more alarming. Typically, the present cost of a drilling crew can amount to approximately R1800 per month. Thirty-thousand rockdrills were in use during 1987 and, on this basis, it is estimated that the monthly cost to the industry of operating these drills was R54 million or R5,67 per ton mined. It is estimated that, at present, the overall cost of supplying compressed air underground and operating rockdrills exceeds R8,00 per ton mined, which amounts to approximately 8 per cent of the total mining costs'.

In conclusion, he referred to the need for a return to the efficiencies and air-distribution pattern of the late 1950s which, he believed, should not be difficult to achieve considering the significant decrease in the amount of equipment powered by compressed air that is legitimately used underground. The savings, he added, could be as much as R500 million per annum. He acknowledged that the long-term future of compressed air is difficult to predict because of competition from hydro- and hydraulic power, but said that 'in the short and medium term, there is little doubt that the existing compressed-air infrastructure, with its huge capital investment in compressors, reticulation systems, and rockdrills, as well as the lack of a fully proven alternative to the pneumatic drills, will ensure the continued use of compressed air underground for the foreseeable future'.

This is a summary of Mr Paul Deglon's Presidential Address to The Mine Ventilation Society of South Africa, which was delivered on 21st June, 1989. The full speech is given in the September and October issues of the *Journal of Mine Ventilation*.