The design of equipment for a solvent-extraction plant for the recovery of metal salts

by H.-M. STÖNNER*, Diplom. Ing. and P. WIESNER*, Ing. Grad.

SYNOPSIS

A brief account is given of the influence of sulphuric acid and chloride ions during the recovery of uranium by solvent extraction, followed by a discussion of the settling behaviour of dispersions and models to represent that behaviour. The paper ends with a description of various methods for the recovery of solvent, which is lost in the solvent-extraction process by entrainment and adsorption on solids and by dissolution.

SAMEVATTING

Daar word kortliks verslag gedoen oor die invloed van swawelsuur en chloriedione tydens die herwinning van uraan deur oplosmiddelekstraksie en dit word gevolg deur 'n bespreking van die besinkgedrag van dispersies en modelle om daardie gedrag voor te stel. Die referaat eindig met 'n beskrywing van verskillende metodes vir die herwinning van die oplosmiddel wat in die oplosmiddelekstraksieproses verlore gaan deur saamsleping en adsorpsie op vaste stowwe en deur oplossing.

Influence of Foreign Ions on Solvent Loading

The first design step in a solvent-extraction plant for the recovery of uranium is to establish the material balance and to determine the number of stages to be employed. This is usually done by the use of a McCabe-Thiele plot for the various steps. However, it is often found that the information for the plotting of the equilibrium line – for example, of the extraction step – is relatively scanty, and the available data have to be smoothed out. In addition, when the solvent flow and the number of stages are being determined, attention has to be paid to the influence, during the extraction, on the equilibrium concentration of the uranyl sulphate complex by such competing ions as sulphuric acid and chloride ions.

A correlation formula for the equilibrium line, which, also takes into account the influence of competing ions, was derived by Stönner and Wöhler¹ some years ago. Its usefulness in the correlation of data is demonstrated by the use of equilibrium data published by the Extraction Metallurgy Division of the South African Atomic Energy Board^{2,3}, for the extraction of uranium from solutions containing between 110 and 140 g/l of sulphuric acid and between 0 and 5 g/l of chloride ions.

The equation is basically of the Langmuir adsorption isotherm type:

$$(\mathbf{U})_{\mathrm{org}} = k_{1} \frac{(\mathbf{U})_{\mathrm{aq}}}{(\mathbf{U})_{\mathrm{aq}} + \mathbf{k}_{2} \cdot (\mathbf{Cl}^{-})_{\mathrm{aq}} + k_{3} \cdot (\mathbf{H}_{2} \mathbf{SO}_{4})_{\mathrm{aq}} + k_{4}}$$

The constant k_1 describes the maximum loading capacity of the organic phase, which is for a 5 per cent Alamine 336 solution of about 7 g of U_3O_8 per litre.

This type of equation can be linearized by the plotting of the distribution coefficient for uranium, expressed as U, in the aqueous phase divided by the uranium concentration in the organic phase, versus the uranium concentration in the aqueous phase. Provided the chloride and sulphuric acid contents remain constant over the whole test series, straight lines are obtained. A least-squares method can be applied to the fitting of the constants. For the sake of simplicity, only three constants were fitted for the graphs shown here.

In Fig. 1, which shows equilibrium lines for uranium at different sulphuric acid strengths, the measured data fit reasonably well. It should be noted that, with increasing sulphuric acid strength, the steepness of the curves diminishes, showing a lower loading of the organic

CONSTANTS USED

k ₁	=	6.772	۵	110 g/1	H ₂ SO4
kz	-	0,02864	۰	125 g/l	H ₂ SO4
k4	=	2,510	•	140 g/l	H _Z SO4



Fig. I-Equilibrium lines for U₃O₈ at different acid strengths

 ^{*} c/o Bockenheimer Landstr. 42, D 6000 Frankfurt/Main, West Germany.
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phase and eventually a higher number of extraction stages to achieve a good extraction.

Fig. 2 shows the correlated values of U_{aq}/U_{org} versus the measured values for a sulphuric acid concentration of 110 g/l and chloride contents of between 0.5 and 5 g/l of the aqueous phase; again, the correlation is reasonably good. It should be noted that the value of the constant k_1 denoting the maximum stoichiometric loading of the organic phase with $U_{a}O_{a}$ is again about 7 g/l.

This treatment of the extraction equilibria is considered to be sufficient for the purposes of this paper.

Settling Characteristics

Settling Models

To adapt the settling of dispersions to the design procedures normally used in the design of chemical reactors, Wöhler used the following approach³.

The kinetics of the settling of dispersions can be regarded as a reaction made up of two consecutive steps. The first step is a volume-controlled reaction, i.e.

$$rac{dV_1}{\mathrm{dt}} = -k \cdot F \cdot H rac{V_1}{V}$$
 ,

which can be visualized, for example, as the coalescence of small droplets to large droplets, V_1 denoting the volume of the dispersion containing small droplets, Fthe settling area, H the thickness of the dispersion band, and V the total volume of dispersion. The second step

CONSTANTS USED k₁ = 7,02375 k₂ = 0,77218 k₃(H₂S04) aq + k₄ = 0,63559

H₂SO₄-CONCENTRATION : 110 g/l





an area-controlled reaction, i.e.

$$rac{dV_2}{\mathrm{dt}} = -c \cdot F rac{\mathrm{V_2}}{\mathrm{V}},$$

which can be visualized, for example, as the sinking or rising of the large droplets resulting from step 1 to the active interface, V_2 denoting the volume of dispersion containing large droplets, and c the velocity of these droplets.

These kinetic expressions can be combined with the material balances to describe a certain flow pattern within the dispersion band. As was shown by Robinson⁴, the flow behaviour of practically all the settlers used for the extraction of uranium can be described as a first approximation by the model of a 'continuous stirred tank reactor', neglecting a short timelag. The simple algebraic equation describing the material balance covering this flow behaviour for the two types of dispersion, i.e.



is combined with the kinetic expressions given earlier, and an equation for the specific settling rate is obtained:

$$rac{Q}{F}=B=crac{H}{H+rac{c}{k}}$$
 ,

B denoting the settling rate of dispersion in cubic metres per (square metre per hour). This equation can be linearized to

$$\frac{H}{B} = \frac{H}{c} + \frac{1}{k}$$

This was shown by Stönner and Wöhler¹ and later derived in a different way by Vieler *et al.* ⁵.

Some test results measured in an experimental unit incorporating design features characteristic of modern mixer-settlers, i.e. a vertical premixer arranged directly in front of the overflow weirs and dispersion discharge at the bottom of the premixer via a pipe to the entry of the settler, demonstrate the usefulness of the abovementioned approach (Fig. 3).

In Fig. 3, the constant
$$d = \frac{c}{k}$$
 , so that $\frac{d}{c} = \frac{1}{k}$.

Diagram 1 is a representation of the measured values and shows the usual trend of the settling curves. Diagram II demonstrates the excellent linearization achieved by the above derived equation, and Diagram III shows the values expressed in a double-logarithmic representation according to Ryon *et al.*⁶.

The proposed model can be utilized to demonstrate the dependence of the settling velocity c of the coarse droplets on the phase ratio of the dispersion and the temperature of the dispersion, as is shown in Fig. 4 by the use of data published by Barnea and Mizrahi⁷. As can



be seen, the maximum settling rate c is, within certain limits, directly proportional to the phase ratio of the dispersed phase. Furthermore, the temperature dependence of the maximum settling rate can be illustrated fairly well by an Arrhenius plot, which exhibits a surprisingly high activation energy.

Flooded Settlers

By use of the proposed model of consecutive reactions³, the same parameters can be defined for continuous steady-state settlers as well as for batch-settling processes. The model in its present convenient form does not include the settling region of small droplets, since these calculations are rather complicated. In other words, the proposed model is used only for the dense dispersed zone.

Fig. 5 shows the general case of a settling cell. The feed mixture, Q_0 , may contain 'new' and 'old' dispersion, the distribution being ξ_{10} and $\xi_{20} = 1 - \xi_{10}$. The outlet dispersion, Q_1 , may contain a different distribution of these two types: ξ_{11} and $\xi_{21} = 1 - \xi_{11}$. The difference between the dispersion feed and the dispersion outlet is $\triangle Q = Q_0 - Q_1$, that is the separated, clear dispersed phase. This simple model of the settling dispersion assumes two types of droplets. It is assumed that type 1 droplets react with one another and produce type 2 droplets, with a reaction constant, k, expressed in cubic metres per (square metre per hour), or a reacting disper-

sion rate expressed in (cubic metres per hour) per cubic metre of total volume. It is assumed further that type 2 droplets (whatever they may be) react with the settler area and produce clear dispersed phase with a reaction constant, c, expressed in cubic metres per hour (dispersion rate) per square metre (of settling area).

In the lower section of Fig. 5, the generalized basic equations (1) and (2) for this model are shown. Equation (3) follows from equations (1) and (2) and represents the overall balance of the settler.

Fig. 6 shows the solution of this set of equations for the general case of a flooded settler, being also an element of the total settling area or, for instance, the compartment between two picket fences. The flooded element of a settler is defined by a constant thickness of dispersion band, H, and a constant settling area, F. If these conditions are put into equations (1) and (2) of Fig. 5, a quadratic equation can be derived. Only one of the two solutions has a physical meaning. This one is shown in the middle of Fig. 6 equation (4). One can make a model of the total settler out of a series of flooded settlers (except the last one!) and study how the type 1 droplets change to type 2 droplets until finally all the dispersion is separated in the last element, which must be a nonflooded settler if the total settler is doing its work properly.

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Fig. 4---Maximum settling rates (based on data from reference 7, p. 86)



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Non-flooded Settlers

Fig. 7 shows how a non-flooded settler behaves. It is the settler one normally refers to when talking of a mixer-settler. This settler is characterized by the fact that the complete mixture is separated or, expressed mathematically, in this settler $Q_1 = 0$.

If this is inserted in equations (1) and (2) and the relationship of equation (5) is derived, it is clear that the dispersion band increases with a growing dispersion rate. Equation (5) can be shown in two linearized forms.

- (1) Equation (6) shows the function q_0/H as a function of (q_0) ; (q_0/H) becomes 0 when q_0 equals c. This is the maximum throughput at an infinite thickness of dispersion band (this is only a theoretical explanation).
- (2) The other linearized form, equation (7), shows the same: H/q_0 as a function of (H). Again, when q_0 equals c, H must be infinite.

The linearized equations are very useful in the derivation of the constants c and (\mathfrak{E}_{10}/k) . The expression (\mathfrak{E}_{10}/k) is new and has not been published before. It means that k cannot be determined as long as the quality of the dispersion fed to the settler is unknown. This also explains why in the past values of k were not as constant as was expected.

If one assumes that the feed is a well mixed, fresh dispersion with only type 1 droplets, $\mathfrak{E}_{10} = 1$ and the well-known equation (8) applies. In this equation, k can

<u>CASE 1:</u>

<u>CONDITIONS</u>

- H = CONST (DISPERSION BAND HEIGHT)
- F = CONST (SETTLING AREA)

<u>Solution</u>

 $\Delta Q = f(Q_0)$ SEPARATED PHASE

$$\Delta Q = P - \sqrt{P^2 + R} \qquad EQN(4)$$

WITH

$$P = \frac{1}{2} (k \times F \times H + c \times F + Q_0) \qquad EQN(4a)$$

$$R = -c [k \times F \times H + (1 - \epsilon_{10}) \times Q_0]$$
 EQN (4b)

Fig. 6—The equations for a flooded settler

be determined direct from continuous settling tests. Without going into the details, ξ_{10} can be derived from the initial slope of a batch settling curve, e.g. a batch settling curve with a horizontal initial slope has a value of $\xi_{10} = 1$. This is the case in well-mixed, fresh dispersions.

Old or not well-mixed dispersions have a steep initial slope in the batch settling curve, resulting in $\xi_{10} < 1$ or $\xi_{20} > 0$. In other words, batch *and* continuous tests together allow an easy method for the determination of the parameters ξ_{10} , k, and c.

Settlers Operated below and above Flooding Point

Barnea⁸ published measurements of deep-layer settlers operated below and above the flooding point. He defined flooding at a constant thickness of dispersion band when the dispersed phase flows over the weir for the continuous phase. He measured the amount of unseparated substance while increasing the dispersion feed step by step far over the flooding point.

It is of interest to see whether the present model of consecutive settling reactions correlates with the data of Barnea⁸. The result is shown in the lower part of Fig. 8, which is based on different parameter sets of k, c, and $\boldsymbol{\xi}_{10}$. It was assumed that $\boldsymbol{\xi}_{10} = 1$, or that a well-mixed, fresh dispersion was fed to the settler. A set of k and c

was adopted in which the calculated line was somewhere between the measured points. Barnea⁸ says that it is rather difficult to make exact measurements near the flooding point. This statement is agreed to by all who have conducted experiments in that field. Hence the points must be scattered owing to the nature of the subject, but one can see from the measurements, as well as from the calculated line, that the clarified dispersion rate is less once the settler is operated above the flooding point.

The dependence of the thickness of the dispersion band on the dispersion rate according to equation (4) is shown in the upper part of Fig. 8 for the same set of k, c, and ε_{10} as used for the flooded settler. Owing to the scattered points, it is not possible to determine the parameters individually and exactly; only one suitable set of parameters has been selected in order to hit the flooding point and to run somehow through the cluster of points. This further qualitative evidence shows the usefulness of the proposed simple settling model.

Recovery of Solvent

The operating costs of a plant are directly influenced by the loss of solvent. In many cases the raffinate cannot

) CONDITIONS

SOLUTION

 $H = f(Q_0)$ (DISPERSION BAND)

$$H = \frac{Q_0 \times E_{10} / (k \times F)}{1 - Q_0 / (c \times F)} = \frac{Q_0 \times E_{10} / k}{1 - Q_0 / c}$$
(5)

LINEARIZATIONS OF EQN (5)

$$\frac{q_0}{H} = \frac{k_1}{\epsilon_{10}} \left(1 - \frac{q_0}{c}\right) = f(q_0)$$
(6)

$$\frac{H}{q_0} = \frac{H}{c} + \frac{\varepsilon_{10}}{k} = f(H)$$
(7)

SPECIAL CASE

FOR FRESH DISPERSIONS WITH $\varepsilon_{10} = 1$

$$\frac{H}{q_0} = \frac{H}{c} \div \frac{1}{k}$$
(8)

Fig. 7—The equations for a non-flooded settler



Fig. 8—Application of the settling model—equation (5) below flood point, equation (4) above flood point

be re-used owing to its contamination with solvent, which, in addition creates environmental problems.

The following does not attempt to show why solvent losses occur, but how they can be reduced. Losses due to entrainment and adsorption on solids, and losses due to dissolution are treated separately

Entrainment and Adsorption on Solids

The separation of entrained or adsorbed solvent can be carried out by the following methods: gravity settling, flotation, centrifugal forces, and coalescence aids.

Gravity settling is used, for example in the well-known after-settlers in solvent-extraction plants. A very efficient way to recover solvents is the use of the Shell corrugatedplate interceptor (CPI). The general arrangement is shown in Fig. 9. The advantage of this separator is an improved separation of oil and sludge from water. The plate pack consists of a number of corrugated plates mounted in parallel in a closed frame and arranged at 45° . Sludge is deposited in the troughs of the corrugations and slides down, while lighter material collects under the ridges and flows up in the gutters to the surface. A further advantage is that it needs only approximately 20 per cent of the floor-space required by a conventional aftersettler. Flotation with air for the recovery of solvent is used, for example, in the Chemilite Quadricell. Difficulties such as the generation of foam and the need for surfactants prevents the wider application of this method.

Centrifugal forces could be applied successfully, but the large flows of raffinate prevent the use of complicated centrifuges, which have a limited capacity. Cyclones with high velocities and large shearing forces are not suitable to eliminate the fine haze of solvent.

Coalescence has achieved the best results in the recovery of solvents. (Fine filtration, porous sheets, glass beads, and electrostatic fields are not discussed here because they cannot be applied to raffinate in the presence of slimes and/or crud). Coalescers that are of particular interest include the TPI, FRAM, Lurgi, and Fibre-Film coalescers.

Coalescers

The TPI (tiltable plate interceptor from Pielkenrood– Vinitex) is identical to the Shell CPI mentioned earlier. The FRAM also uses corrugated plates built with oleophilic material, but the direction of flow is across the corrugations, as shown in Fig 10. The coalesced oil weeps through small holes on top of the corrugations. Both the TPI and the FRAM show more or less identical results for the decantation of solvent but there are difficulties with cleaning.

The Lurgi coalescer (Fig. 11), which is similar in design to up-flow filters, has built-in cleaning devices. Classified broken coke is used as the coalescence material with the needed rough surfaces. It is supported on a tray that is equipped with filter jets. The solvent droplets coalesce on the coke surface and rise to the top, where they form an organic layer, which is continuously withdrawn. The raffinate flows to the after-settler. From time to time cleaning is necessary, which is done in the same direction of flow using water, air, and steam if necessary; the muddy liquor is collected for further treatment.

All the results obtained with these three coalescing devices show that a certain amount of solvent remains in the raffinate as secondary haze.

Merichem, Houston, offers the fibre-film technique for the intimate contacting of two immiscible liquids (Fig. 12). The principle of mass transfer is based on the



Fig. 9-The Shell CPI waste-water de-oiler



Fig. 10-The FRAM

difference in solid-surface tension between the two liquids. The solid surface is provided by a bundle of long, continuous, small-diameter fibres contained in a pipe. The type of fibre material is chosen so that the phase of smaller volume selectively coats the fibre with a thin film thereby creating a large mass-transfer surface. The larger volume, i.e. the continuous phase, flows through the bundle parallel to the array of wetted fibres, and actually moves the smaller volume of constrained phase along the fibres by drag forces.

This equipment could be helpful in breaking the secondary haze in the same way as, for example, an additional kerosene stream is recycled from the aftersettler. Tests on the application of the fibre-film technique, also for extraction purposes, are under way.

Dissolution

Such considerations as the solvent wash present another possibility for the recovery of solvent through the removal of dissolved solvent by use of re-extraction, desorption, and adsorption.

Re-extraction can be used for the recovery of dispersed, as well as dissolved, solvent. According to the authors, experience with, for example, phenol extraction from gas liquor, the re-extraction could easily be done. The solvent from the raffinate would be re-extracted by another, low-boiling solvent, which is really insoluble in the first solvent. The separation of both solvents can be achieved by distillation. The consumption of steam can be estimated as approximately 1 per cent (by mass) of the treated raffinate. This figure applies only if the recovery of the low-boiling solvent from the raffinate is not necessary. If other losses occur, for example by adsorption on solids, the re-extraction method is not practicable.

In that case the steam stripping method could be considered in place of re-extraction. In a desorption column equipped with trays or packings, the solvent is driven off with steam. This steam can either be injected as live steam or indirectly generated in a reboiler. Together with the solvent, 1 to 3 per cent of the raffinate has to be distilled off. These overhead products are condensed, cooled, and separated into an aqueous and an oily phase. The heat of condensation could be used for other purposes, for example for the heating of the pregnant solu-



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SOLVENT / KEROSENE



Fig. 13—Treatment with activated carbon

tion before extraction to accelerate the separation velocity or prevent the growth of fungus. The consumption of steam is estimated to be in the range 2 to 4 per cent (by mass) of the treated liquid. The figures for the consumption of steam in these appliances should be confirmed by tests.

As for *adsorption* methods for the recovery of solvent, the application of zeolites does not look promising because the regeneration problem has not been solved. However, some years ago successful tests were performed with activated carbon for the recovery of amine. Bergbauforschung¹⁰ developed a process for the treatment of coke-oven gas liquor with continuous regeneration of activated carbon. In the application of this process to the cleaning of raffinate, the process would operate as follows (Fig. 13). After the filtered raffinate is fed through a bed of activated carbon, where the organic substance



Fig. 14—The recovery of benzene with activated carbon

is adsorbed up to approximately 10 per cent (by mass) of the coke, a small portion of the loaded carbon is ejected with a water stream to the carbon-water separation. In a fluidized-bed retort heated with gas at about 750 to 900°C, the organic material is burnt off and the activated carbon is regenerated and recycled. Fines from coke, slimes, and unburnt crud are separated in a cyclone.

However, this process removes solvent rather than recovering it. For this purpose an additional step must be added, as shown in Fig. 14. This is the backwashing of the loaded carbon with another low-boiling solvent, for example benzene. The benzene as solvent II is distilled off from the diluted solvent I. The benzene is recovered from the carbon by steaming out. The quantity of carbon employed is about 0.5 to 1 kg/m^3 . A recovery of more than 95 per cent of the solvent can be expected. It is estimated that approximately 10 to 20 per cent of the carbon employed should be regenerated by thermal treatment in a fluidized-bed retort. It has to be kept in mind that the minimum capacity for this regeneration is 200 kg/h of carbon and consequently sufficient for 1000 to 2000 m³/h of raffinate. This may be a motive for considering a centrally situated regeneration station. The running cost of approximately R1 per kilogram of solvent appears more reasonable when expressed as 5 to 10 cents per cubic metre of treated raffinate. However, with 15 per cent interest and capital depreciation, the recovery costs would climb to between R5 and R10 per kilogram and could be justified only when full reimbursement is possible by the re-use of the raffinate.

Conclusion

Of the various options available for the design of

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solvent-extraction plant, the best for a particular purpose has to be chosen on the basis of tests on the material to be treated in the plant, for a design that may be best for one type of material may be quite unsuited to another.

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Fluorspar specifications

A technical committee of the International Organization for Standardization, 'ISO TC 175 Fluorspar', of which South Africa holds the secretariat, is meeting at the headquarters of the South African Bureau of Standards in Pretoria to draft a specification that is of great importance to exporters of this strategic mineral.

South Africa has three major fluorspar mines and the third-largest reserve of fluorspar in the world, and is developing into the principal producer of this mineral. Several importing countries are also represented on the committee, which is charged with drawing up a specification that will cover the three technical grades for fluorspar for use in international trading.

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The three grades are acid grade, ceramic grade, and metallurgical grade. Acid-grade fluorspar has a very high calcium fluoride content and is used in the production of hydrofluoric acid. Ceramic grade also has a high calcium fluoride content and is used in the ceramic industry. The metallurgical grade contains less calcium fluoride and is mainly used as a flux in the metal industries.

The grading system has an influence on the price of fluorspar on the world market because the price is fixed according to grade. To smooth the way for trade, it is also advisable that there should be a general acceptance of the definitions of the different grades.

Exploration planning

A seminar on 'Economic Guidelines for Exploration Planning', organized by Rhodes University will be held in Grahamstown from 11th to 19th August, 1982. The attendance will be limited to 30 participants, the seminar being intended for more senior personnel of the mining industry. However, prior experience in economic evaluation practice is not essential.

The seminar has been designed with the practical needs of exploration personnel in mind. It will provide explorationists with an opportunity to study, discuss, and work with economic analysis techniques, and their application to economic planning issues associated with exploration. Emphasis will be placed on small-group workshop sessions. The economics and planning aspects of mineral exploration will be considered against a background of basic economic analytical techniques.

Professor B.W. Mackenzie, who will conduct the seminar, is an internationally recognized expert in the field of mineral economics and is Professor in the Department of Geological Sciences at Queen's University, Kingston, Ontario, and Research Associate at the Centre for Resource Studies at Queen's University. He was formerly Director of the Mining Program at McGill University, where he was responsible for undergraduate and graduate courses in mineral economics as part of the mining engineering curriculum. During the past ten years, Professor Mackenzie has developed and organized professional seminar programmes in mineral economics at McGill and at Queen's and has conducted a series of seminars for other institutions in various parts of the world during the same period. He is also at present responsible for more broadly based mineral economics seminars at the Australian Foundation in Adelaide.

This will be the fourth series of seminars presented by Professor Mackenzie at Rhodes, and it will be an updated and extended version of the very successful seminars presented in 1979, 1980 and 1981. The topics are as follows:

Introduction

The Concepts of Cash Flow and Time Value

Discounted Cash Flow Methods

Mining Taxation Considerations

- Establishing Minimum Acceptable Exploration Target Conditions
- **Exploration Economics and Strategies**
- **Evaluating Exploration Projects**
- Structuring Exploration Agreements
- Economic Guidelines for Finding and Acquiring **Mineral Deposits**

Risk Analysis Techniques

The Decision Tree Concept for Exploration Planning Further details are obtainable from Miss D.V. Turner,

Geology Department, Rhodes University, P.O. Box 94, Grahamstown 6140.