

Problems in the hydrometallurgical extraction of uranium in the U.S.A. and South Africa*

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SYNOPSIS

A variety of processing problems in the hydrometallurgical extraction of uranium in the U.S.A. are contrasted with those in South Africa. In U.S. plants, for example, problems are often encountered in the separation of vanadium, molybdenum, and uranium, and in the contamination of the circuit with organic acids. South African plants often experience problems with dissolved silica in the feed, clarification of feeds, and problems associated with synthetic polyelectrolytes. Individual situations are examined, along with the analytical techniques that assist in solving specific problems. Successful approaches that have been applied to mechanical and chemical problems in uranium solvent-extraction plants are discussed.

SAMEVATTING

in Verskeidenheid verwerkingsprobleme wat in verband met die hidro-metallurgiese ekstraksie van uraan in die V.S.A. ondervind word, word met dié in Suid-Afrika vergelyk. In die Amerikaanse aanlegginge word daar byvoorbeeld dikwels probleme ondervind met dié skeiding van vanadium, molybdeen en uraan en met die besmetting van die kring met organiese sure. Suid-Afrikaanse aanlegginge ondervind weer dikwels probleme met opgeloste silika in die toevoer, die verheldering van die toevoer en in verband met sintetiese poliëlektroliete. Individuele gevalle word ondersoek tesame met die analitiese tegnieke wat help om spesifieke probleme op te los. Geslaagde benaderings wat toegepas is op meganiese en chemiese probleme in aanlegginge vir die oplosmiddel-ekstraksie van uraan word bespreek.

Practice in the U.S.A.

To avoid confusion, some explanation regarding terminology is appropriate. In the U.S.A., the term *scrubbing* refers to the removal of accumulated contaminants after the uranium has been stripped. The South African term for this operation is *regeneration*, while *scrubbing* refers to the treatment of the organic phase after loading but before stripping. This paper uses the locally accepted terms when discussing each country's plants.

Uranium plants in the U.S.A. treat secondary uranium deposits, which have a uranium grade of 0,09 to 0,12 per cent. A typical operation involves crushing, grinding, and agitation leaching of the ore, followed by 6 to 8 stages of countercurrent decantation (CCD) and 1 to 2 stages of clarification to produce a pregnant liquor. Polyacrylamides are normally used for the liquid-solid separation in CCD and the clarifiers because they easily liberate the uranium from ore that has been coarsely ground (to minus 28 mesh). After washing, the underflow from CCD has a solids content of approximately 50 to 55 per cent, and these fine suspended solids passing through the clarifiers are removed by sand or pre-coat pressure polish filters. The pregnant liquors have a uranium content of 0,1 to 0,5 g/l, from which the uranium is recovered by solvent extraction or by resin treatment followed by solvent extraction (ELUEX).

A typical uranium solvent-extraction plant on the Colorado Plateau (which stretches across Wyoming, New Mexico, Colorado, Utah, and Arizona) consists of four extraction stages, three to four stripping stages and,

when required, one scrubbing stage. In the extraction, the aqueous-to-organic ratio is usually between 3/1 and 5/1, which allows for a reduction in the size of the stripping stages. Unless there are extenuating circumstances, gaseous ammonia-ammonium sulphate is used for stripping. Ammonia gas is usually introduced in number one and number three stripping mixers through sparge rings located in the bottom of the mixer. The concentration of ammonium sulphate - $(\text{NH}_4)_2\text{SO}_4$ - in the stripping solution is controlled at approximately 120 to 150 g/l by the bleeding of ammonium sulphate from the barren stripping solution. The organic-to-aqueous ratio in stripping is adjusted to produce a pregnant stripping liquor with a uranium content of 25 to 35 g/l. To minimize the localized precipitation of yellow-cake in the strip mixers at these uranium concentrations, the pH value during stripping is maintained at approximately 3,8 in the first stripping stage and 4,4 in the third. The uranium contained in the pregnant strip liquor is precipitated with ammonia gas at pH 7,0. The precipitated ammonium diuranate is thickened, filtered, dried, and often calcined at the plant site, the calcine being packaged in drums for shipment and subsequent conversion into nuclear fuels.

Problems Encountered in the U.S.A.

Removal of Organic Matter

The majority of the uranium mined in the U.S.A. occurs in sedimentary sandstone and siltstone deposits called roll fronts. These deposits are believed to have been formed by the precipitation of uranium in ancient river beds, and, because they are sedimentary in nature, they often contain high concentrations of organic contaminants, which are primarily vegetable matter that has been partially carbonized. This peat-like material is usually high in uranium, and in humic and fulvic acids. The chemically diverse organic compounds making up these peaty layers are readily extracted during the leaching of uranium, and report along with the uranium

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to the solvent-extraction circuit. During the extraction of the uranium, these organic contaminants are extracted and/or solubilized by the kerosene diluent. They are often extracted preferentially to the uranium, and block the reaction sites available for uranium loading. Invariably, with uranium ores that contain a great deal of organic matter, the resins in ELUEX circuits become contaminated, or emulsions are produced in solvent-extraction circuits.

Because this partially carbonized vegetable material normally contains high concentrations of uranium, partially carbonized ore cannot be isolated and left in the pit. However, several approaches can be followed in the treatment of such ores. In general, if the organic contaminants are present in concentrations that do not allow for their removal after the uranium has been recovered, they must be reduced in the pregnant liquor prior to solvent extraction. There are several routes that can be followed to achieve this, including the following.

1. The level of organic matter can be reduced by the blending of contaminated ore with run-of-mine ore.
2. The ore can be roasted prior to milling.
3. Activated carbon can be added to the CCD circuit, or a carbon column can be installed to remove the contaminants from the pregnant liquor.
4. A Tricellator* or similar device can be installed to float off the organic contaminants from the pregnant liquor.
5. The pregnant liquor can be contacted with kerosene or an aromatic diluent prior to solvent extraction.
6. Additional oxidant can be added during leaching to convert the hydrophobic organic contaminants into hydrophilic non-organic soluble contaminants.

Obviously, the best solution to the problem is the blending of run-of-mine ore with contaminated ore to reduce the overall content of organic matter in the mill feed. The roasting of ore is very costly from both a capital and an operating standpoint, and the addition of granulated carbon to the CCD circuit not only increases the operating costs but slightly reduces the uranium content of the pregnant liquor. The same comments can be made in regard to the installation of a carbon column or a Tricellator. The use of slightly more oxidant during leaching to oxidize the organic material represents a relatively low-cost functional solution to the problem, but extreme care must be exercised when additional oxidant is used to minimize the leaching of silica. The presence of excessive amounts of oxidant during leaching greatly increases the degradation rate of Alamine 336 and isodecanol, which in turn contributes to poor phase disengagement.

If contaminated ore can be blended with run-of-mine ore to the extent that at least one extraction and one step cycle can be achieved, the organic matter can be scrubbed from the organic phase after stripping. In the scrubbing stage, the stripped phase can be contacted with an aqueous solution of 5 per cent NaOH or Na₂CO₃, and 5 per cent methanol. If the uranium was not com-

pletely stripped before the scrubbing stage, the Na₂CO₃ will minimize the tendency for it to precipitate. Methanol is often required to help solubilize the carboxylate salts and speed the phase disengagement.

Contaminated aqueous scrubbing solution must be continuously bled from the scrubbing stage to control the build-up of stripped contaminants. Where this aqueous bleed contains appreciable uranium values, it can be acidified and returned to the extraction feed, provided that provision has first been made to skim or extract the tramp organic acids, which will now be in their water-insoluble hydrogen form.

The amount of organic phase can be measured by a number of different techniques, those most commonly used being titration procedures. Prior to titration, however, all the metals and acidic impurities must be removed from the sample by scrubbing with 10 per cent Na₂CO₃ solution, which converts the amine to the free reagent.

Although simple titration with acid indicates the total amount of amines present, it does not give any information about the relative proportions of tertiary, secondary, and primary amines. However, each type of amine can be determined separately. If the sample is reacted with acetic anhydride, the primary and secondary amines are converted to neutral amines, and these are titrated as tertiary amines. The difference between the amount of tertiary amines and the total amines must be equal to the primary plus the secondary amines. The primary amines can then be masked by being converted with salicylaldehyde to the less basic amine, and titration of the secondary and tertiary amines potentiometrically. From these results, one can calculate the amounts of the individual primary, secondary, and tertiary amines.

Normally, the tertiary amines are only 5 per cent or so less than the total value of amines. As the 5 per cent represents virtually only secondary amines, the value of primary amines is not usually required. However, in circuits where severe degradation has occurred, the primary amines may constitute a significant proportion of the non-tertiary amines. But, in steady-state circuit operation, the amount of primary amine tends to be low owing to its relatively high aqueous solubility.

The level of alcohol modifier, usually isodecanol, can be determined if the solution is reacted with excess acetic anhydride and if the acetic acid generated is titrated. As the acetic anhydride also reacts with the primary and secondary amines, the alcohol value must be corrected for these components.

Another helpful tool for the measurement of organic material in the circuit is gas chromatography, optionally coupled with mass spectroscopy. Fig. 1 shows the chromatograph of a kerosene solution of Alamine 336 spiked with dioctylamine. The kerosene elutes first (far left), followed immediately by the dioctylamine, and then by the evenly spaced peaks of Alamine 336. Since Alamine 336 is made from a mixture of n-octanol and n-decanol, the tertiary amine contains all combinations of those alkyl groups. Mass spectra of each peak indicate their respective molecular masses. Isodecanol, as well as its occasional substitute, tridecanol, is not resolved from the numerous kerosene peaks, and thus cannot be

*The Tricellator is a patented device, manufactured by Pollution Control Engineering, Inc., that removes organic matter from aqueous solutions.

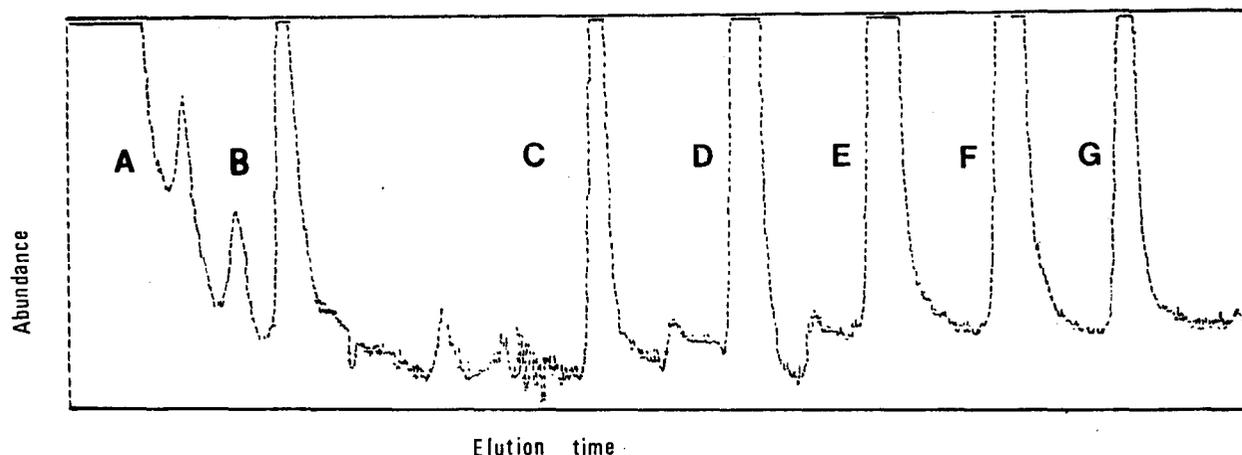


Fig. 1—Spectrum of Alamine 336 and dioctylamine in kerosene, obtained by a combination of gas chromatography and mass spectroscopy

A	Kerosene	E	Dioctyldecylamine
B	Dioctylamine	F	Dioctyloctylamine
C	Dioctylhexylamine	G	Tridecylamine
D	Trioctylamine		

determined by this technique. However, other organic components can sometimes be detected if they are of higher molecular mass.

The most frequently encountered tramp organic components, as already mentioned, are carboxylic acids. Their presence can be detected in at least three ways. In the first approach, the circuit is scrubbed with aqueous 10 per cent sodium carbonate, which converts the carboxylic acids to their sodium salts. These salts either report to the aqueous phase or, if the solubility is low, they form a third phase. In either event, they are isolated from the organic components in the circuit and, after being acidified, are recovered by extraction with ether. After being dried and the ether evaporated, the residue is examined by infrared spectroscopy for the presence of acids. Although the spectrum is often complicated by the absorption of amines and kerosene (phase separation in the carbonate scrub is frequently very poor), this technique has the advantage of being able to show many other functionalities, such as humic and fulvic acids, which have characteristic spectra.

The second method for the detection of carboxylic acids is thin-layer chromatography. The plate is first spotted with a small amount of organic phase, which is then eluted with toluene and visualized with bromeresol green, which gives a yellow colour with carboxylic acids. This technique is very quick, but provides only qualitative values.

The third method is much more complex, but can give valuable information regarding the source of the acids. The acids from a carbonate scrub are recovered in the hydrogen form as with the infrared method. Then, to eliminate the interference due to entrained kerosene, the acid sample is placed in a column of silica gel and is first eluted with pentane. The acids are then eluted with acidified methanol. When the methanol has been stripped, the acids are converted to their methyl esters, and the residue is analysed by the combined technique of gas chromatography and mass spectroscopy. The esters

are thus resolved according to molecular mass and boiling point, and their mass spectra can be compared with those of reference compounds.

Thus, for ore from an orebody that contains a high level of organic matter, a carbonate scrub followed by this type of analysis showed a range of fatty acids predominantly in the C_{14} to C_{18} range with a substantial amount of isodecanoic acid, but very low levels of C_8 and C_{10} fatty acid. From this it was concluded that the contaminating acids were derived from the ore and from oxidation of the isodecanol, but only to a slight degree from degradation of the amines.

In another instance, where the e.m.f. had been maintained at a very high level, the major acid components were shown to be mainly n-decanoic acid with substantial amounts of isodecanoic acid and lesser amounts of higher fatty acids up to C_{17} . It was concluded that degradation of the isodecanol and of the tertiary amine had been the main pathway of acid formation, although there had also been significant contamination from the ore itself. Apparently, the expected n-octanoic acid from the degradation of amine is of low enough molecular mass to be continuously washed out of the organic phase. The conclusion was reinforced by the analysis of the amine, which showed that over 40 per cent of the amine in the sample had degraded to secondary or primary amine.

Removal of Molybdenum

Another contaminant commonly found in the uranium ores of the Colorado Plateau is molybdenum. When the uranium from such an ore is leached, a portion of the molybdenum present is also extracted. Molybdenum in uranium leach liquors presents a separation problem, for not only does it poison the resin in ELUEX circuits but it also precipitates from solvent-extraction circuits as a tertiary amine organo-molybdenum complex. The tolerance of Alamine 336 for molybdenum varies with several factors, including the concentration of the amine

and the concentration of isodecanol and/or aromatic constituents in the organic phase. Table I illustrates the increasing tolerance of Alamine 336 for molybdenum contained in a typical uranium pregnant liquor.

TABLE I
TOLERANCE OF ALAMINE 336 FOR MOLYBDENUM

Reagent	Concn in organic phase % (v/v)	Molybdenum g/l
Alamine 336 (Henkel Corp.)	2,5	0,2 to 0,30
Isodecanol	3,0	
Napoleum 470 (Kerr McGee)	94,5	
Alamine 336	5,0	0,40 to 0,50
Isodecanol	3,0	
Napoleum 470	92,0	
Alamine 336	10,0	1,10 to 1,30
Isodecanol	10,0	
Napoleum 470	80,0	

In addition to forming an insoluble amine complex with organic matter, molybdenum loads preferentially to uranium, and silica, when present, further reduces the solubility of the molybdenum-amine complex. Since relatively minor amounts of molybdenum in uranium leach liquors can cause severe operating problems, Henkel Corporation has developed several approaches for the treatment of uranium ores containing molybdenum, including the following.

1. The molybdenum can be extracted from uranium leach liquors by the use of activated carbon.
2. The molybdenum can be extracted selectively with XI-8A, an experimental reagent produced by Henkel Corporation (which is not currently available in commercial quantities).
3. The tolerance of Alamine 336 to molybdenum can be increased by the addition of Alamine 304 to the circuit. Alamine 304 has a high tolerance for molybdenum and is being used commercially for the extraction of molybdenum.

If the molybdenum concentration of the uranium pregnant liquor is high, the best approach to the problem is the extraction of the molybdenum prior to that of the uranium. The recommended reagent is XI-8A because the loaded organic phase can be stripped with ammonia-ammonia chloride at pH 9,5, and the molybdenum in the XI-8A pregnant strip liquor can then be precipitated with calcium chloride to produce calcium molybdate.

If the concentration of molybdenum in the uranium pregnant liquor is so low that extraction with XI-8A cannot be justified, selective stripping of the uranium from Alamine 336 can be carried out with an acidified chloride solution. This stripping procedure involves contacting of the loaded organic phase with a chloride solution (approximately 80 g/l) at pH 2,0. Ammonium chloride is normally used as the source of chloride ion since stripping with sodium chloride produces a yellow-cake high in sodium that requires further treatment to meet the sodium specifications.

After the uranium has been stripped with acidified

chloride, the molybdenum that was co-extracted with the uranium remains on the amine and does not strip. Consequently, an organic bleed or the total organic flow must be contacted with a solution of soda ash or ammonia-ammonium chloride solution to remove the molybdenum. If a 1M solution of soda ash is used to strip the molybdenum, any trace quantities of uranium remaining in the organic solution will remain soluble in the molybdenum strip liquor. Although stripping with soda ash is desirable because this process eliminates the precipitation of uranium, the recovery of molybdenum from a sodium carbonate solution presents a problem. To precipitate the molybdenum and minimize the co-precipitation of calcium carbonate, a solution of calcium chloride (approximately 300 g/l) must be added to the well-agitated molybdenum liquor as a fine spray over an extended period of time. Care must be exercised to avoid overdosing, since excessive amounts of calcium chloride promote massive precipitation of the calcium carbonate.

As indicated previously, a solution of ammonia-ammonium chloride at pH 8 to 9 can be used to strip molybdenum from Alamine 336. However, before that can be done, the uranium must have been completely stripped from the organic phase, or any uranium remaining in the organic phase will precipitate immediately upon contact with this solution and cause phase-disengagement problems. The advantage in the use of ammonia-ammonium chloride solution in the stripping of molybdenum and the regeneration of amine is the ease with which the molybdenum can be recovered from this strip liquor by precipitation with calcium chloride.

Removal of Vanadium

Uranium deposits in the U.S.A. often contain vanadium as well as molybdenum. When uranium, vanadium, and molybdenum occur together, the uranium and molybdenum can be separated as indicated previously. Since the e.m.f. of typical uranium leach liquors is maintained in the range -400 to -450 mV, vanadium exists in the non-extractable tetravalent state. As the e.m.f. is increased to -600 mV, all the ferrous iron in the solution is oxidized to ferric iron, and the vanadium begins oxidizing to the pentavalent state. At an e.m.f. of -680 mV, the vanadium is almost totally converted to the extractable pentavalent state. After that, the vanadium ($(H_2V_6O_{17})^{-2}$) can be extracted in four stages with Alamine 336. Since vanadium (like molybdenum) loads as a polymeric complex, 2,5 per cent (v/v) Alamine 336 will easily load the V_2O_5 present in concentrations of 10 g/l. The recovery of vanadium is enhanced if the fourth extraction stage is maintained at pH 2,0 to 2,3 and a temperature of 25 to 40°C.

The loaded organic phase leaving the first extraction stage is usually washed at pH 2,0 with a weak solution of sulphuric acid before reporting to a two-stage sodium carbonate stripping section. Although red cake ($NaVO_3$) can be precipitated if the sodium carbonate strip liquor is acidified, the addition of 1,2 pounds of ammonium chloride per pound of V_2O_5 precipitates ammonium metavanadate (NH_4VO_3), which has a sodium content of only 0,1 to 0,3 per cent. Precipitation with NH_4Cl is usually carried out over a 6 to 8 hour period, after which the ammonium metavanadate is filtered and washed with

a weak ammonia solution to remove the sodium. The final product has a V_2O_5 content of 75 to 77 percent.

The South African uranium industry is fortunate in being based on ore-bodies that contain only trace quantities of vanadium and molybdenum, and the separation problems associated with these metals are not encountered in South Africa at the present time.

Practice in South Africa

Most South African uranium plants treat a pregnant liquor that has been derived by intensive atmospheric leaching of finely ground Witwatersrand gold ore.

Molybdenum and vanadium are absent and therefore present no problems. However, in the future, as in the U.S.A., ores may have to be treated that contain such metals.

Three of the most commonly encountered technical problems arising from the operation of solvent-extraction plants in South Africa in 1979 are discussed below.

Problems Encountered in South Africa

Removal of Dissolved Silica

In South Africa, the ratio of dissolved silica to dissolved U_3O_8 in the pregnant liquor is normally between about 3:1 to 10:1, but it is possible that, in some of the circuits currently under construction, the ratio will be even higher. This dissolved silica can become loaded on the tertiary amine extractant and, if not removed in the scrubbing section of the solvent-extraction plant, will precipitate in the stripping section as the pH value is raised from around 3,0 to 5,0. This precipitated crud can cause physical problems in the stripping and regeneration sections. There are two main methods to solve these problems. The first is to reduce the amount of dissolved silica present in the pregnant liquor by careful control of the leaching process. Excessive additions of acid, too high a leaching temperature, and too long a leaching time can all lead to a high concentration of dissolved silica in the leach liquor. Obviously, these parameters cannot be reduced to a level at which the uranium dissolution would be affected adversely, but equally obviously they should not be maintained at high levels that yield no improvement in the uranium dissolution but increase the silica dissolution significantly. The second method is to design and operate the scrubbing section so that most of the silica is removed before the organic phase enters the stripping and regeneration stages, where the pH values are high. The silica is best scrubbed when the pH value of the aqueous phase in the final scrubbing section is controlled within the range 2,5 to 3,0. The exact range varies from one operation to another, and must not be so high as to result in significant stripping of uranium in the scrubbing section. In pilot-plant scrubbing tests, re-cycling of the aqueous phase has been found helpful to maintain the organic-to-aqueous ratio in the scrub mixer close to 1:1, a ratio that results in more effective scrubbing than the higher ratios often used in practice.

Problems of Phase Disengagement

Flocculants are used in South African uranium plants to assist in solid-liquid separation and in clarification of the leach liquor. The following are the major types used:

- for filtration: Guar-based flocculants and minor amounts of non-ionic polyacrylamides,
- for CCD: Guar-based flocculants or non-ionic polyacrylamides,
- for clarification: cationic polyacrylamides, non-ionic polyacrylamides, and minor amounts of guar-based flocculants.

The presence of certain flocculants in the leach liquor can lead to phase-separation problems in the solvent-extraction circuit. Field observations and laboratory tests on phase disengagement have shown that organic continuous emulsions of guar-based flocculants, and of non-ionic and slightly anionic polyacrylamides have no detrimental effect on phase separation and may slightly enhance the separation, but that organic continuous emulsions of cationic polyacrylamides can increase the phase-separation time appreciably.

These effects are illustrated in Figs. 2 and 3. The mechanism by which cationic polyacrylamide influences the rate of phase separation is not understood, but it appears to be a consistent effect no matter what type of cationic polymer is used.

The effect of flocculants in aqueous continuous emulsions on the phase separation is much more severe. When present in that form, all the non-ionic and anionic flocculants listed above have a markedly detrimental effect on the phase separation, as illustrated in Figs. 3 and 4.

The reason for this more marked effect may be related to the nature of the film, which has to be thinned before coalescence can take place. Polymer flocculants are all water-soluble and, therefore, in an aqueous continuous emulsion, they are present in the very thin film of aqueous phase that must rupture to allow coalescence. Molecules of the size present in polymer flocculants can structure water and greatly increase the viscosity of the film when they are present in extremely thin films of

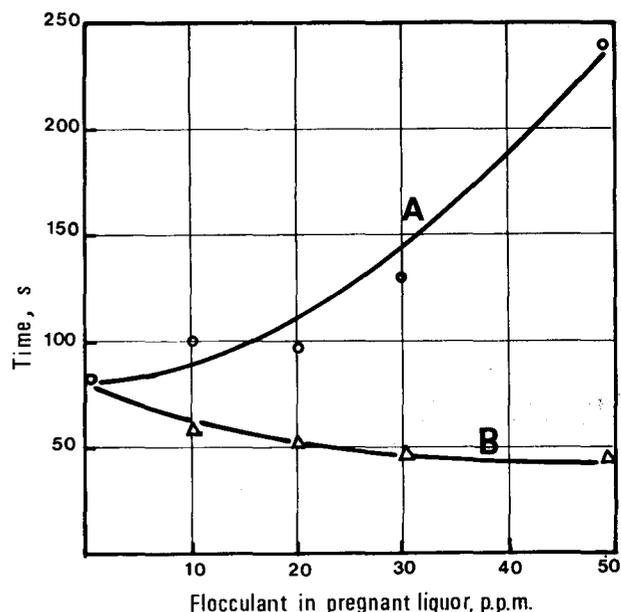


Fig. 2—The rate of phase separation in organic continuous emulsions of cationic polacrylamide (A) and Guarfloc (B)

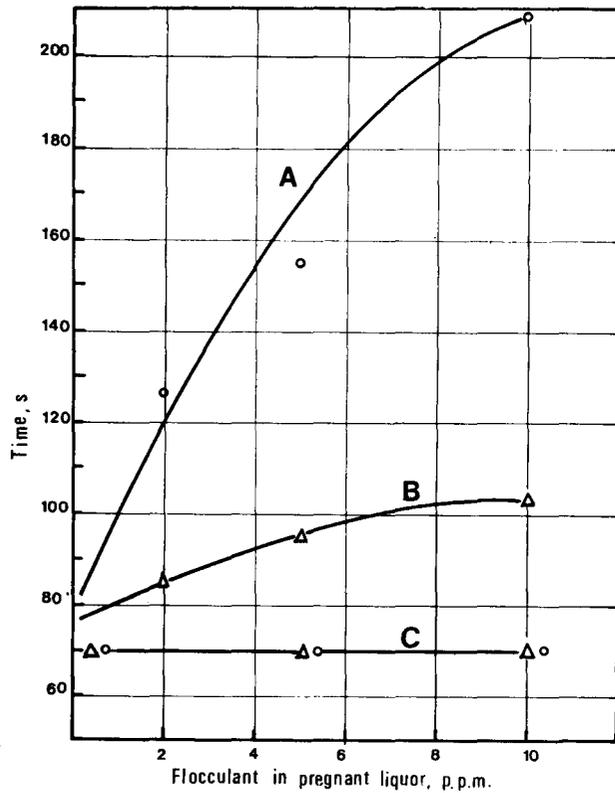


Fig. 3—The rate of phase separation of a non-ionic polyacrylamide aqueous continuous emulsion (A), a Guarfloc aqueous continuous emulsion (B), and a Guarfloc or non-ionic polyacrylamide organic continuous emulsion (C)

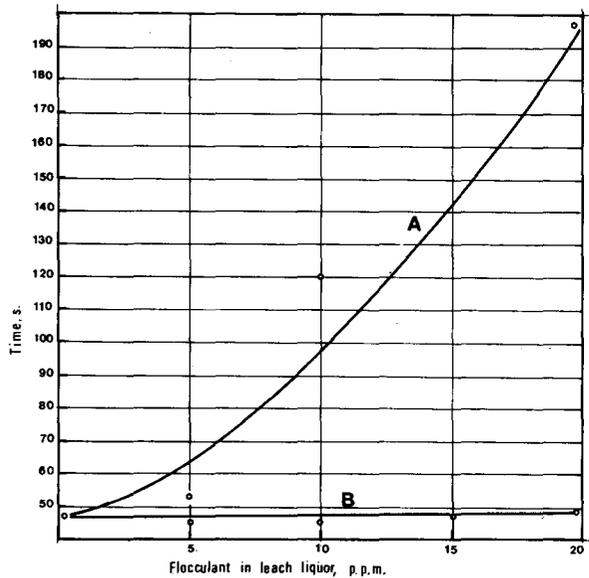


Fig. 4—Rate of phase separation in an anionic polyacrylamide aqueous continuous emulsion (A) and an anionic polyacrylamide organic continuous emulsion (B)

water. In organic continuous emulsions, the polymer molecules are located within the aqueous droplet and are not able to control the thinning of the organic film.

In the evaluation of the effect of flocculants on phase-disengagement rates in laboratory tests, it is essential that the rate of phase disengagement should be measured by a standard and reproducible technique. The mere mixing of organic phase with aqueous flocculant, visual observation of a shake-out test, and reporting of the results as 'emulsion formed' or 'no emulsion formed' are inadequate. Flocculants rarely form emulsions; rather, they reduce the phase-separation rate, with the result that mixed phases overflow the weirs of settlers and pass through the pump impellers or turbines. This action produces the emulsion; therefore, flocculants indirectly cause emulsions to be formed.

All the results reported in Figs. 2 to 4 were measured by a standard technique used at Henkel Corporation. The question of how much of the polymer added to a pulp reports in the aqueous phase and how much is adsorbed onto the solids is a vexing one; assumptions are often made that more than 90 per cent of the polymer added is adsorbed onto the solids. However, the quantity of solids in clarification circuits is small, and too much flocculant in the clarification circuit could result in appreciable concentrations of flocculant in the pregnant liquor. Unfortunately, there is no means for the direct analysis of part-per-million levels of flocculants in aqueous feeds; instead, one has to rely on physical manifestations and try to correlate them with flocculation practice.

Problems Arising from Inadequate Clarification

Pregnant solutions containing more than 30 p.p.m. of suspended solids can give rise to high losses of solvent from a solvent-extraction circuit. Clarification circuits have, in the past, involved high capital costs, but the recently developed hopper clarifier may provide a low-cost method for the effective clarification of pregnant liquors.

The Future

New ores in the U.S.A. and South Africa may contain organic contaminants that lead to the formation of 'tar' during leaching, which would require a new approach to the extraction of uranium. The processes used in existing plants may also change. The use of pressure leaching may result in pregnant liquors that contain dissolved rare-earth elements, while the leaching of pyrite concentrates on their own may alter the chemical composition of the pregnant liquor. Under these circumstances, the scope of the technical problems, as well as the approaches and techniques needed to solve them, will be broadened significantly.