

**STATE OF THE ART  
AND  
FUTURE DIRECTIONS IN SOLVENT EXTRACTION**

**PROCEEDINGS OF  
THE FOURTH INTERNATIONAL  
SOLVENT EXTRACTION WORKSHOP 2007**

**AUGUST 21- 24, 2007  
NIAGARA FALLS, ONTARIO,  
CANADA**

**Organized by Dr. Gordon M. Ritcey**

**Sponsored by the International Committee for Solvent Extraction  
Technology (ICST)**

Workshop Web Site: <http://www.solvent-extraction.com>

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## State of the Art and Future Directions in Solvent Extraction - 2007

### PROGRAM

TUESDAY AUG. 21            1630 – 1800 REGISTRATION HOTEL LOBBY  
   1830 – 2000 RECEPTION

WEDNESDAY AUG. 22       0700 – 0800 BREAKFAST  
   0830 – 1230 WORKSHOP  
   1000 – 1015 REFRESHMENT BREAK  
   1230 – 1900 AFTERNOON BREAK  
   1900 – 2130 WORKSHOP  
   2015 - 2030 REFRESHMENT BREAK

THURSDAY AUG. 23        0700 – 0800 BREAKFAST  
   0830 – 1230 WORKSHOP  
   1000 – 1015 REFRESHMENT BREAK  
   1230 – 1900 AFTERNOON BREAK  
   1900 – 2130 WORKSHOP  
   2015 - 2030 REFRESHMENT BREAK

FRIDAY AUG. 24            0700 – 0800 BREAKFAST  
   0830 – 1200 WORKSHOP  
   1000 – 1015 REFRESHMENT BREAK  
   1200 – 1300 LUNCH BUFFET AT "A CUT ABOVE"

1300 – 1700 WORKSHOP  
1530 – 1545 REFRESHMENT BREAK  
1700                    WORKSHOP CONCLUDES

## **INTRODUCTION**

The 4<sup>th</sup> Solvent Extraction Workshop began with a welcome by Gordon Ritcey to the 4<sup>th</sup> Solvent Extraction Workshop, followed by a brief greeting by Geoff Stevens on behalf of the International Committee for Solvent Extraction. A summary of the accomplishments of the first 3 Workshops was next presented by Gordon Ritcey.

## **SUMMARY OF PREVIOUS 3 WORKSHOPS**

Gordon Ritcey (G.M. Ritcey & Associates Inc.)

The Summary of the first 3 Workshops is shown in the Power Point presentation which is provided in the following pages.

# INTRODUCTION

## SX WORKSHOPS

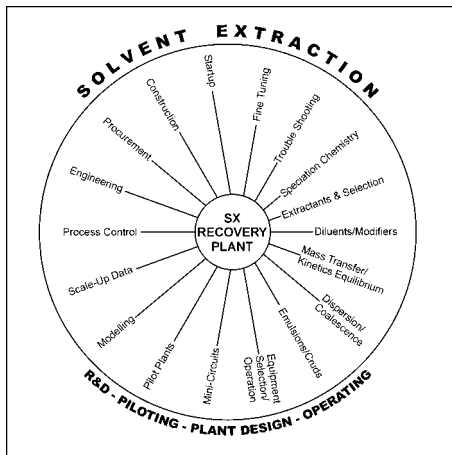
G.M. RITCEY

## OBJECTIVES

**Objectives:**

**Critique** the various areas of the SX process:

- a) Fundamental research in chem.& chem. Eng.
- b) Flowsheet development (chemistry, chemical engineering, metallurgy) through to piloting to obtain preliminary design and cost data
- c) Engineering design, control, plant design and plant construction
- d) Plant operation (problems and solutions)



## WORKSHOPS SUMMARY

1997 BANFF  
2000 BANFF  
2003 DIGBY

## CHEMISTRY

- REAGENTS (GENERAL)
- PURITY OF REAGENTS
- REAL SOLUTIONS
- SPECIATION (MASS TRANS., Eh-pH)
- NEW REAG'TS (MARKET, PROCESS)
- POT. APPL'NS (VARIOUS WASTES)
- SYNERGISM ( MIX'D EXT'TS OR COMP'DS)
- DIL.& MOD. (LEAST UNDERSTOOD)

## CHEMICAL ENGINEERING

- INTERFACIAL PHENOMENA-KINETICS, DISP/COAL
- CHEM. INFLUENCE (COALESCENCE)
- MICELLES (SURFACTANTS)
- MASS TRANSFER (KINETICS, MULTI-COMPONENT SYSTEMS)
- DESIGN MODELS (PREDICTION FOR MXS, COL., DROPLET SIZE)
- PROCESS DYNAMICS (CONTROL, START-UP)

## CHEMICAL ENGINEERING (CONT'D)

- SCALE-UP (VALID'N MODELS, CONTACTOR TYPES, COALESCENCE)
- EQUIPMENT SELECTION (ALL PARAMETERS)
- MXS, COLUMNS, IN-LINE MIXERS
- RECYCLE MINOR PHASE
- PHASE RATIO

## CHEMICAL ENGINEERING (CONT'D)

### COLUMN TESTS

- SIZING (EQUIL. DATA, STAGES, PHASE CONTINUITY)
- CONTINUOUS TESTING (CHEMISTRY, DEGRADATION, PRODUCT SPECS, TRAINING)
- DEMONSTRATION PLANT

## BENCH-SCALE & PILOTING

### REQUIRE:

- FEED CHARACTERISTICS
- PROCESS CHEM., EQUIL. DATA
- KINETICS FOR DESIGN
- SCRUBBING AND STRIPPING
- SELECTION OF CONTACTOR

## BENCH-SCALE & PILOTING

### PHASES

- BENCH TESTS
- CONTINUOUS TESTING & PILOT
- SIZE & DURATION
- ASSESS METALLURGY, EMULSIONS, CRUD, DISP/COAL, LOSSES

## BENCH-SCALE & PILOTING

### PHASES (CONTINUED)

- OTHER FLOWSHEETS; OTHER CONTACTORS
- CONFIRM MODELS BASED ON DATA
- MATERIALS FOR EACH STREAM

## ANALYSIS & MONITORING

- IN-LINE (ENTRAINMENT, SOLUTION; SOLVENT LOSSES)
- OFF-LINE (ELEMENTS, ORGANICS)
- TECHNIQUES (XRF, AA, DENSITY, VISCOSITY, SPECTROSCOPY)
- MONITORING (ORG. DEGRAD'N)
- CONTROL SYSTEMS (pH, levels, flows)

## MODELLING

### REQUIRED FOR:

- EQUIPMENT DESIGN
- PROCESS OPTIMIZATION
- FUNDAMENTALS

## MODELLING

### SHORT-FALLS OF EXISTING MODELS

- PRACTICALITY OF ACADEMIC MODELS
- DISP/COALS MUST BE CONSIDERED
- LACK OF SETTLING MODELS
- LACK OF FUNDAMENTAL DATA
- VALIDATION OF MODEL REQUIRED

## PLANTS & OPERATION

- LITERATURE INFORMATION
- TRAINING
- EQUIPMENT & CAPITAL COSTS
- OPERATING COSTS
- PLANT ENGINEERING

## PLANTS & OPERATION

### OPERATION

- START-UP PROBLEMS
- INFORMATION REQ'D TO OPERATE
- MEASUREMENTS OF PERFORMANCE



## PLANTS & OPERATION

- NOVEL PROCESS EQUIPMENT
- FUTURE SX DESIGN & OPERATION
  - EXPERT SYSTEMS
  - COLUMNS

## SOLVENT LOSSES

- SOLUBILITY
- ENTRAINMENT
- EVAPORATION
- MISTING
- DEGRADATION
- CRUD
- SAMPLING & SPILLS

## NOVEL SYSTEMS

- MICROEMULSIONS
- MEMBRANE SYSTEMS
- SUPERCRITICAL EXTRACTION
- ELECTRICALLY-ASSISTED SX & MEMBRANE-ASSISTED

## NOVEL PROCESSES

- SOLVENT-IN-PULP (EXTRACTION DIRECTLY FROM LEACH SLURRY)
- IONIC LIQUIDS (REPLACEMENT OF ORGANIC WITH H<sub>2</sub>O-IMMISCIBLE IONIC LIQUID)

## ENVIRONMENTAL

### SOURCES:

- SOIL, WATER, AIR, METALS, ORGANICS
- CONTAINMENT (WITHIN PLANT; WASTE DUMP DISPOSAL).
- DISPOSAL (CRUD, INCINERATE, BACKFILL)

## ENVIRONMENTAL

### CONCERNS

- RISK ASSESSMENT
- ENVIRONMENTAL IMPACT
- DECOMMISSIONING
- SAFETY DESIGN IN PLANT
- WASTE REDUCTION
- OXID'N & BIODEGR'N OF ORGANICS
- RECOVERY TOXIC METALS

## FIRES

- MATERIALS
- INSULATION
- ELECTROSTATIC DISSIPATION
- HIGH FLASH POINT DILUENT
- EXPLOSION PROOF MOTORS
- EARTHING OF TANKS & LINES
- SYNTHETIC CLOTHING
- FIREWALLS & BUNDED AREAS

### WORKSHOP AGENDA

Based on the information received by the participants, the following was the order of discussions. However the order of presentation in this document more closely follows the actual process of solvent extraction.

- 1) Solvent losses--identify causes by tests and analyses, and then minimize by plant operational control and solvent recovery; minimize losses caused by crud, entrainment, misting. Address the aspect of oxidation effects on components of the solvent and the ensuing solvent degradation and thus losses. What can be done to prevent?
- 2) Crud formation and crud treatment with phosphine type reagents (Cyanex 272, 301, 302, etc) e.g. in Ni / Co SX
- 3) Interfacial effects and plant performance
- 4) Chemistry of highly concentrated solutions and the interaction of diluents and modifiers.
- 5) Development and modification of solvents using sophisticated thermodynamic equilibrium and kinetic models vs. simply a laboratory technique for equilibrium isotherms.
- 6) Prediction of Plant Design--McCabe Thiele, kinetics and design. Influence of contactor type (e.g., shear, axial and back mixing). Should there be a latitude in design given for the subsequent construction that does not consider the influences such as kinetics, shear, contactor type, solvent concentration, and phase continuity? Also, what are the considerations and advantages /disadvantages of minor phase recycle? Is it always necessary?
- 7) Mixer settler configuration (series, series-parallel). Prediction by testing and criteria for decision.
- 8) Materials of construction in SX plants
- 9) Pulsed and reciprocating columns: The gap between the experimental approach used by the industrial engineers vs. the mathematical models developed in academia, but hardly applied in the field.
- 10) Advantages/disadvantages of "breaking" the circuit in recovering more than one metal by SX.IX vs SX "What are the selection criteria?"
- 11) Safety
- 12) Prevention of Fires

### TOPICAL PRESENTATIONS

In order to provide important background information to the major areas for discussion, and thus set the scene for debate, several participants of this 4<sup>th</sup> Workshop were asked to make short presentations.

Chemistry / Reagents	Gary Kordosky, Cognis
Process Design	Indje Mihaylov, CVRD-Inco
Column Operation & Interfacial Effects	Geoff Stevens, Melbourne University
Pulsed & Reciprocating Columns	Baruch Grinbaum, Bateman
Plant Design	Mark Vancas, Bateman
Plant Operation	Mark Vancas, Bateman
Solvent Losses	Gordon Ritcey

While the above participants were identified for those specific areas, others were encouraged to contribute in their particular areas of expertise and to provide short summaries where possible.

### DISCUSSIONS

#### CHEMISTRY OF METAL EXTRACTION

Gary Kordosky (COGNIS)

The Power Point presentation which accompanied the keynote paper on the Chemistry of Metal Extraction is shown following this section. Following the presentation, the following points were discussed:

- Possibly more reagents may be required in the future because of decreasing resources, ore grades and impurities
- Perhaps no new reagents are required if selective scrubbing and selective stripping are further developed.
- Kinetics can be accelerated by the addition of phase transfer catalysts, e.g. protonated amines.
- Synergists are likely to be the future for "new reagents" instead of actually developing expensive reagents that must then go through the regulatory process
- Synergism optimization is possibly difficult to control because of the differential losses, and the incentive will be to develop and optimize a mix for a particular deposit
- Zinc is a prime element for recovery by solvent extraction, such as in the recovery of secondary zinc.
- Bulk extraction followed by selective separation in smaller circuits will be more popular. An example of bulk extraction is that of Goro, using Cyanex 301 for Co + Ni co-extraction, followed by stripping with chloride and selective separation of Co from Ni using an amine.

## **State of the Art and Future Directions in Solvent Extraction - 2007**

- Reverse micelles have been found to be a possible source of contamination in the tank house. The organic, if clean (containing no impurities and surfactants) will be less susceptible to micelle formation and therefore problems in the tankhouse (see Fouling under Solvent Losses).
- Direct stripping of the product from the organic phase by hydrolytic stripping or hydrogen reduction has been proven technically feasible, and these options could be considered for application in the future.
- A thermodynamic model using a computer program has been developed to determine the extraction and stripping isotherms for copper. Developed by reagent suppliers, the program is distributed to their customers. Similar programs are used by Cognis and Cytec.
- If the viscosity is maintained low (by control of surfactants-see Fouling under Solvent Losses), then the solvent concentration can be raised to a higher level for operational purposes.
- As regards the tenor in the PLS, and the feasibility of good plant performance, uranium plants have been up to 12 g U/L. However a 3rd phase results in an aliphatic diluent and is prevented by the addition of a high molecular weight alcohol or the use of an aromatic diluent. Some plants (e.g. Cu) are running at >30 % extractant, even at room temperature.
- As the extractant concentration increases, the viscosity increases which ultimately impacts on an economic operation. For example, at 40% extractant concentration, the economic limit may be reached in one system.
- Although the extractant concentration will govern the recovery, nevertheless the stripping efficiency will limit the extraction efficiency—not the reagent.
- Extraction from high salt solution (90-100 g/L Cl) can operate well at certain plants (e.g. Michella)
- In the case of using a 40% LIX reagent, the operation temperature required would be 40°C. Start-up would be at low flows.
- For many operations the use of mixer settlers as the contactor was stated as “forgiving”.
- Highly concentrated solutions can be treated by the use of columns, in-line mixers, and the RTL contactor—all low shear in operation—to treat highly viscous solutions.
- “Breaking the circuit” (e.g. first stage SX circuit followed by precipitation, re-solution and SX separation as in the Murrin Murrin Nickel process for Co & Ni recovery) provides flexibility of the SX process, and therefore a “comfort zone” in the design. The bankers like this approach (conservative but expensive) as compared to the one flow system of successive extraction plants for Co and Ni as used at Bulong Nickel, for example.

# Chemistry of Metal Extraction by SX Reagents

Gary Kordosky

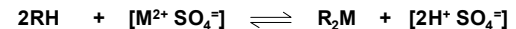
## Extractable Metal Species

1. **Metal cations:**  $\text{Cu}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Co}^{2+}$ , etc.
2. **Complex metal anions**, for example,  $[\text{UO}_2(\text{SO}_4)_3]^{4-}$ ,  $[\text{Mo}_8\text{O}_{26}]^{4-}$  and others
3. **Complex metal cations** for example,  $[\text{MoO}_2]^{2+}$
4. **Neutral metal species** such as  $\text{UO}_2(\text{NO}_3)_2$

## Reagent Types

- **Chelation Extractants**
- **Organic Acid Extractants**
- **Ligand Substitution Extractants**
- **Neutral or Solvating Extractants**
- **Ion-Pair Extractants**

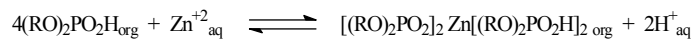
## Chelation Extractants



1. They operate on a hydrogen ion cycle.
2. Some operate well with both acid and ammonia leach solutions.
3. They tend to be more selective than either ion-pair or organic acid extractants.
4. They tend to be kinetically slower than organic acid and ion-pair extractants.
5. They have good operating characteristics with respect to phase separation.
6. They are usually more difficult to produce.

Most common application, Cu (over 3 million tonnes annually)  
Also Ni and Pd

## Organic Acid Extractants



1. They show less selectivity than chelating extractants.
2. They can promote stable emulsions with basic solutions.
3. They operate on a hydrogen ion cycle.
4. Their kinetics of extraction are usually quite rapid.
5. Some of these reagents are easy to produce and some are difficult to produce. Highly variable selling prices depending on the reagent.

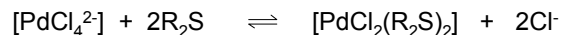
Most significant applications, Zn and Co

Table 1. Metal Extraction by D<sub>2</sub>EHPA (0.5M) in Xylene at 20°C, 50% Extraction pH (a)

Metal	pH 50	Metal	pH 50
Cu <sup>+2</sup>	2.90	Fe <sup>+3</sup>	0.32
Ni <sup>+2</sup>	4.12	Cr <sup>+3</sup>	3.12
Co <sup>+2</sup>	3.70	V <sup>+3</sup>	1.90
Fe <sup>+2</sup>	3.56	Al <sup>+3</sup>	1.53
Mn <sup>+2</sup>	2.82	Bi <sup>+3</sup>	0.29
VO <sup>+2</sup>	1.25	La <sup>+3</sup>	1.96
Mg <sup>+2</sup>	3.81	Ce <sup>+3</sup>	1.73
Ca <sup>+2</sup>	2.85	Nd <sup>+3</sup>	1.46
Zn <sup>+2</sup>	1.42		
Cd <sup>+2</sup>	2.98	Ag <sup>+</sup>	3.25
Sn <sup>+2</sup>	0.15	Cu <sup>+</sup>	4.07 (b)
Pb <sup>+2</sup>	2.50	VO <sub>2</sub> <sup>+</sup>	1.17

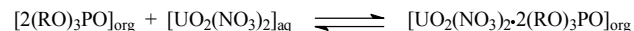
- a. Taken from Product Information Bulletin, DEHPA Metal Extractant, Albright & Wilson Americas, P.O. Box 26229, Richmond, VA 23260-6229.  
 b. Aqueous phase contained 5 volume % CH<sub>3</sub>CN.

## Ligand Substitution Extractants



1. Metal extraction is not normally sensitive to pH.
2. At times remarkable selectivity can be achieved.
3. Kinetics can be rapid or slow depending on the metal and the ligand.
4. These reagents can be relatively inexpensive or very expensive depending on the complexity.

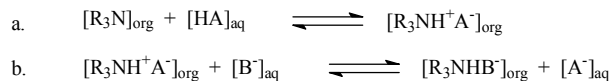
## Neutral or Solvating Extractants



R<sub>3</sub>PO > (RO)<sub>3</sub>PO > R<sub>2</sub>CO > ROH > R<sub>2</sub>O  
 trialkylphosphine oxides > trialkylphosphates > ketones > alcohols > ethers

1. Normally exhibit fast extraction and stripping kinetics.
2. Extract neutral metal complexes.
3. In general are not selective.
4. In combination with other extractants, can act as synergists, antagonists or phase modifiers.

## Ion-Pair Extractants



1. Amines extract anionic metal complexes.
2. Extraction with amines usually takes place from acidic to near- neutral solutions with the best pH range dependent upon the specific system.
3. Amines generally require the use of aromatic diluents and/or require that a phase modifier, normally a long chain alcohol, be added to the hydrocarbon diluent in order to prevent the formation and precipitation of organic insoluble amine acid or amine metal complexes.
4. The kinetics of extraction and stripping are usually rapid.
5. Quaternary and tertiary amines are more selective than secondary or primary amines.
6. There are a wide variety of amines which are available and/or can be synthesized.

## Modifiers

Organic compounds with the ability to donate and / or accept a hydrogen bond which are soluble in the diluent.

Alcohols, Esters, Ketones, Alkylphenols, Phosphine oxides, Phosphate esters, Ethers, and Nitriles come to mind

When used with amines modifiers help solubilize the amine acid and amine metal ion pair complexes

When used with oximes for copper extraction modifiers allow the oxime to be stripped with less acid. Modifiers which only accept a hydrogen bond tend to increase stability of the oxime and also increase selectivity over iron. Modifiers that donate a hydrogen bond to an acceptor have the opposite affect.

## Synergistic Mixtures

A mixture of D<sub>2</sub>EHPA with the oxime Reagent LIX 63 gives a blend that extracts certain metals at lower pH than either extractant by itself. This is only one example of many reagent combinations which show a synergistic behavior. For many other examples see work by John Preston and others.

The LIX 63 / D<sub>2</sub>EHPA combination was investigated as a Ni extractant, but the poor stability of the LIX 63 to hydrolysis made the combination to costly.

## Diluents

1. Solubilize the extractant and metal-extractant complex.
2. Have a low viscosity and density in order to assist phase separation and reduce entrainment.
3. Should be free of objectionable components to minimize crud formation.
4. Should be chemically stable to circuit conditions.
5. Should have a flash point 25 °C or higher above the highest operating temperature in the SX circuit.
6. Should be insoluble in the aqueous phase.
7. Must have low evaporation losses.
8. Should not be detrimental to metal extraction/stripping chemistry.
9. Should be readily available at a low cost.



## **New Extractant Molecules**

**Chances of new extractant molecules being synthesized, developed and commercialized are not good. The reasons are:**

- 1. Today's commercially available copper extractants are very good and a better, less costly to use, molecule is not likely.**
- 2. The cost to develop and commercialize a new molecule is high for a variety of reasons not the least of which is registration of any new molecule and the accompanying toxicity testing.**
- 3. There are not a lot of metals that could be recovered in sufficient amounts to justify the expense of a new molecule.**
- 4. Over the years many molecules have been made and investigated by reagent suppliers, but the results have not been made public. Thus there are fewer new molecules to investigate than the public realizes.**

**As a result new reagents will likely come from combinations of existing reagents and new SX applications will come from fitting flow sheets to the properties of existing reagents.**

**PROCESS DESIGN**

Indje Mihaylov (CVRD – INCO)

The Power Point presentation which accompanied the keynote paper on Process Design is shown following this section. Following the presentation, the following points were discussed:

- The extractant concentration vs. the O/A ratio, are sufficient to cope with variation in the feed tenor, and to minimize solvent entrainment losses.
- In the development of one process, the pilot plant duration was 6 months in order to obtain sufficient mass transfer data.
- It was stated that 200 cycles in a pilot plant were required to determine organic performance and to assess the effect on the coalescence rate and extractant loss.
- The importance of analysis of components of the lost solvent was stressed.

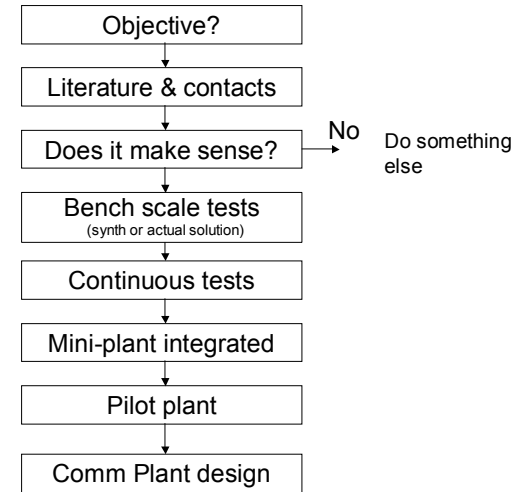
# SX Process Design (and importance of piloting)

GM Ritcey 4<sup>th</sup> SX Workshop  
Niagara Falls, Ontario

Indje Mihaylov  
Inco Technical Services Limited, Mississauga



Aug 23, 2007

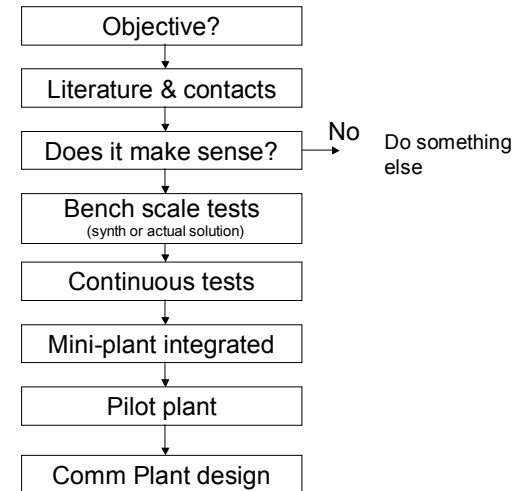


Aug 23, 2007

- Organic – extractant concentration, modifier, diluent
- Extraction, wash/scrub, strip -- stages & res time; O:A ratios; phase-continuity; pH profile; temperature; base/acid addtn and requirements
- Metal displacement/accumulation in stages; gypsum?
- Phase-disengagement, viscosity, s.g., interfacial tension
- Entrainment (A in O, O in A) and measurement; soluble loss and TOC; organic recovery
- Extractant stability; organic treatment; diluent oxidation; air ingress; ORP control
- Crud formation and treatment; organic recovery; underappreciation of susp solids problem
- Evaporative losses – organic recovery and meeting discharge regulations
- Mixer efficiency and settler performance
- Feeds and raffinate diluent wash
- Silica and other nasties
- Organic cross-contamination
- Equipment selection and testing



Aug 23, 2007



Aug 23, 2007

**... and importance of piloting**

- Effect of Fe on extractant stability
- Cobalt catalysed diluent oxidation

**COLUMN OPERATION & INTERFACIAL EFFECTS**

Geoff Stevens (University of Melbourne)

The Power Point presentation which accompanied the keynote paper on Column Operation and Interfacial Effects is shown in the following pages.



## Solvent Extraction Models and Practice

Professor Geoff W Stevens  
Director  
Particulate Fluids Processing Centre  
(an ARC Special Research Centre)  
Department of Chemical & Biomolecular Engineering  
The University of Melbourne, Australia



## Equipment

Mixer Settlers:

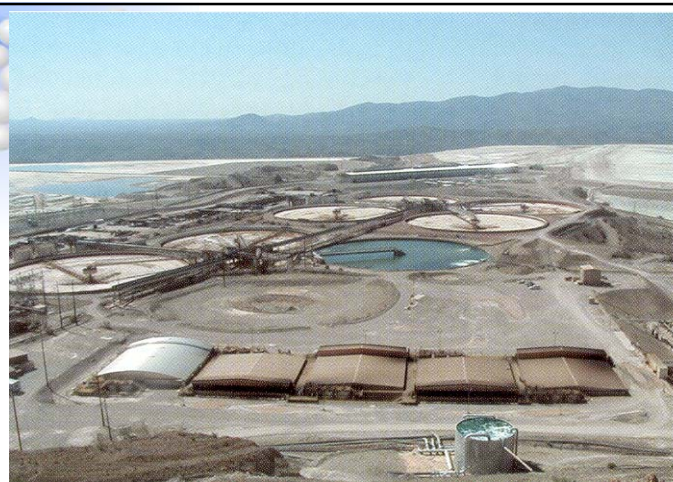
- ❖ Workhorse of the industry
- ❖ Other industries moved on from these in the 1940s



Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council



Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council



Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council





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# Equipment

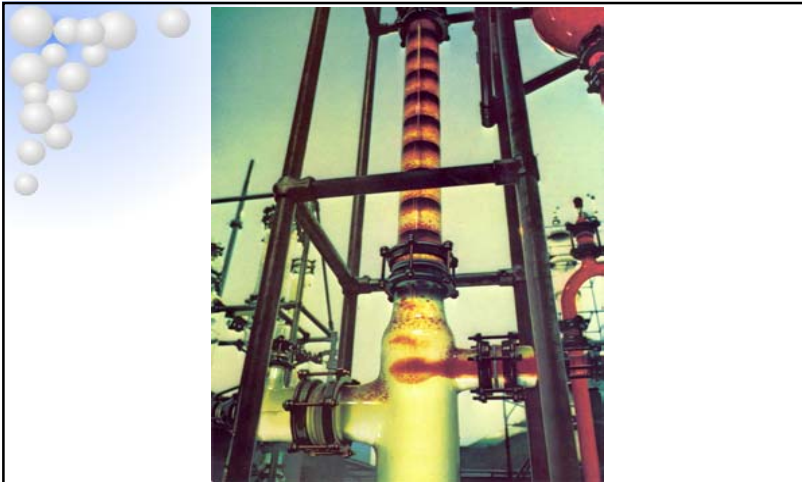
## Columns, in-line mixers, membrane contactors

- ❖ Advantages:
  - ❖ Better control of dispersion
  - ❖ Reduced loss by entrainment
  - ❖ Reduced loss by evaporation
  - ❖ Higher turndown, more flexible
  - ❖ Better crud control
  - ❖ Reduced organic inventory
- ❖ Disadvantage:
  - ❖ Simple equilibrium stage models not accurate for scale-up. Need more realistic model
  - ❖ Decrease residence time in contactor

} = CHEAPER



Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council




Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council



Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council





## Mass Transfer Performance


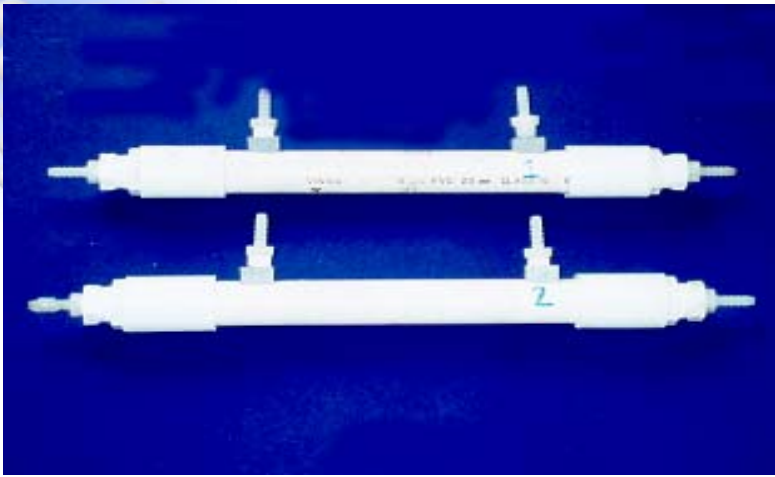
Flux =  $k a \Delta c$

$k$  = Mass Transfer Coefficient  
[function of intensity of mixing]


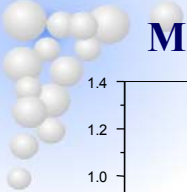
$a$  = Interfacial Area

$\Delta c$  = Concentration Driving Force

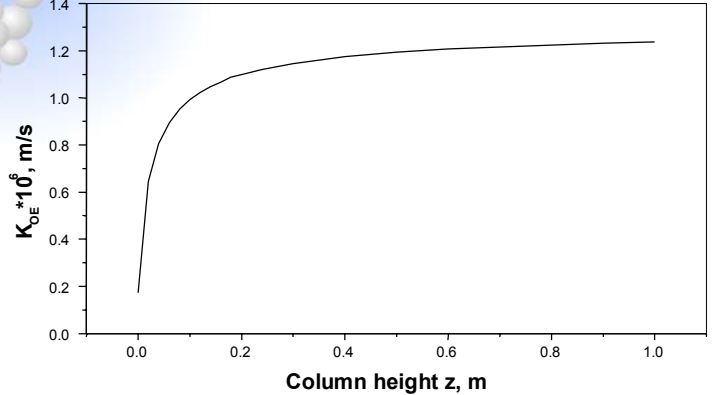
Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council

Particulate Fluids Processing Centre  
A Special Research Centre of the Australian Research Council






## Mass Transfer Performance



Column height $z$ , m	$K_{OE} \cdot 10^6$ , m/s
0.0	0.0
0.05	0.7
0.1	0.9
0.2	1.1
0.4	1.18
0.6	1.22
0.8	1.24
1.0	1.25


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## Mass Transfer Performance

Mass transfer coefficient is not constant in most metal extraction processes because of changes in kinetics

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## Operational Problems in Solvent Extraction Plants

- ❖ Crud
- ❖ Carry over of organic in Raffinate
- ❖ Carry over of feed in extract
- ❖ Poor recoveries

Control of dispersion and coalescence is the key to good design and operation of a solvent extraction plant



## Kinetics, Coalescence and Dispersion Depend on Interfacial Properties

To date, there is no agreed method of incorporating interfacial effects in the prediction of kinetics of extraction or coalescence of drops

- ❖ Issues with kinetics
- ❖ Issues with coalescence



## Models for Prediction of Size of Contactors

- ❖ **Plug Flow Model [McCabe-Thiele]**
  - ❖ Used for M/S design
  - ❖ Used for columns with empirical parameters
- ❖ **Axial Dispersion Models**
  - ❖ Accounts for non uniform continuous phase flow
  - ❖ Need model for axial dispersion
- ❖ **Numerical simulation of two phase flow in contactors**
  - ❖ Usually based on Computational Fluid Dynamics Modeling
  - ❖ Interfacial phenomena not well understood
  - ❖ Only useful for considering changes to internal geometry at present

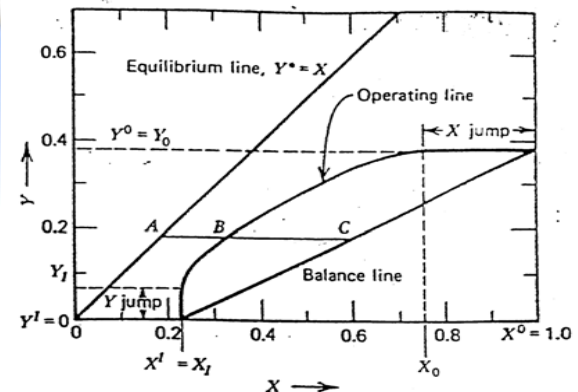


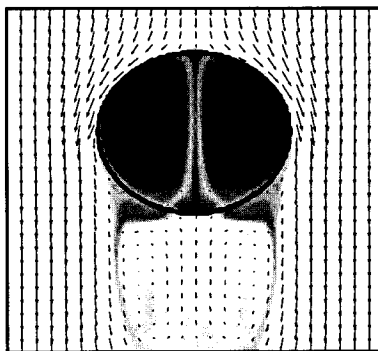
Figure 3. Operating diagram on X-Y coordinates. The parameter values are the same as those for Fig. 2.

McCabe Thiele Design



## Finite Element Analysis

- ❖ Good flow field visualisation around droplets
- ❖ Predict mass transfer from single drops
- ❖ Not yet applied to total contactor



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## What is an Interface?



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## Molecular Mechanics and Dynamics

### Definitions: MM & MD

#### 1. Molecular Mechanics (MM)

- ❖ molecular statics
- ❖ representation of molecular system with “force field”  
➔ potential energy minimization

#### 2. Molecular Dynamics (MD)

- ❖ many particle system interacting via “force field”
- ❖ numerical solution of classical equations of motion  
➔ creation of trajectory in phase space of system



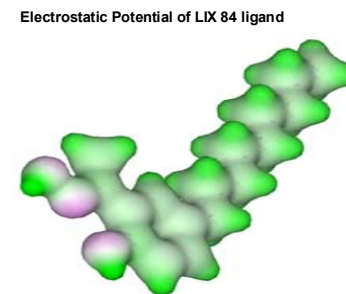
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## Extraction Ligand Properties

### Semi-empirical Quantum Mechanics Calculations:

Approximate Solution of the Schrödinger Equation  
 $H\Psi = E\Psi$



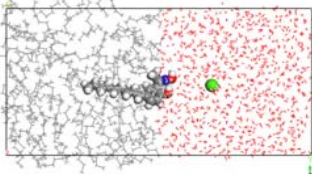
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## Solvent Extraction System

### Charge Dependent Interfacial Adsorption Energy

Charged LIX 84 at interface with counter ion



	Neutral LIX 84 at interface (kcal/mol)	LIX 84 cation at interface (kcal/mol)	LIX 84 anion at interface (kcal/mol)
$\Delta E_{\text{LIX8}}$	-190.03	-287.57	-225.94

➡ Charged species show a preferred adsorptivity!

➡ Preferential adsorptivity of cationic species!



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## Conclusion

- ❖ Molecular modeling of solvent extraction issues is still in its infancy
- ❖ Simulation strategies have to be tailored to the property of interest
- ❖ Increase in computer capacity will promote the use of molecular modeling in a range of applications



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## Kinetics of Extraction

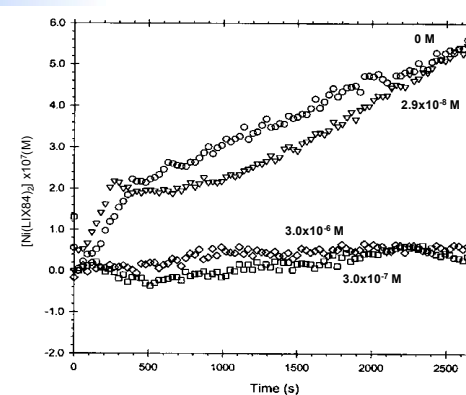
- ❖ Many studies of the kinetics of extraction in pure systems with relatively low concentrations
- ❖ Need to extend this to industrially relevant systems to help in developing reliable scale up models



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## Interfacial Competition Effect of $C_{12}E_8$



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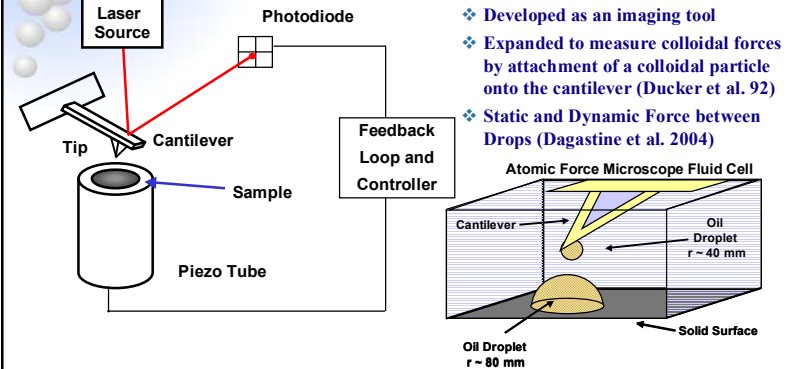
## Kinetics of Extraction

### Conclusions:

- ❖ Modeling only fits if interfacial concentrations are used, not bulk
- ❖ Small concentrations of surface active agents influence the kinetics significantly

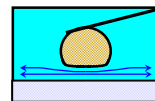
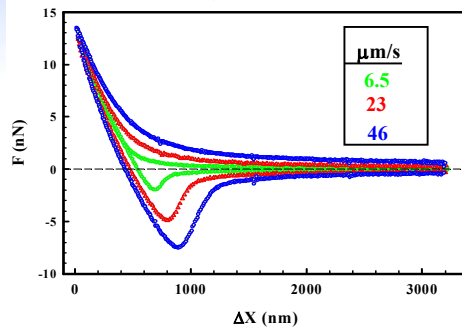


## Atomic Force Microscopy Force Measurement



## Drop Impact on a Flat Surface

Oil Drop  $R_s = 40 \text{ nm}$   
Electrolyte: 10 mM SDS and 1 mM  $\text{NaNO}_3$



## Coalescence of Two Drops

- ❖ Modelled based on electrostatic and van der Waals forces
- ❖ Surface mobility?
- ❖ Surface deformation?
- ❖ Effect of polymers?
- ❖ Need new ways of measuring these



## Preliminary Results

Teflon plate at 120 RPM  
Phase ratio 0.7



Stainless Steel plate at 120 RPM  
Phase ratio 0.7



Nylon Plate at 120 RPM  
Phase Ratio 0.7



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## Conclusion

- ❖ **Interfacial properties are important in solvent extraction processes**
- ❖ **We have a number of new techniques that allow us to probe the interface in more depth**
- ❖ **Kinetic expression should include interfacial concentrations not bulk concentrations**



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### **PULSED & RECIPROCATING COLUMNS**

Baruch Grinbaum (Bateman-Litwin)

The Power Point presentation which accompanied the keynote paper on Pulsed & Reciprocating Columns is shown following this section. Following the presentation, the following points were discussed:

- The drop-size is important, particularly when on flat surfaces, where there is an increase in the rate of coalescence. Organic continuous may be preferred.
- The detrimental effect of interfacial tension and the organic fouling on the coalescence and kinetics was discussed.
- The direction of mass transfer, e.g. in extraction the transfer from aqueous to organic, and therefore organic continuous. In a several-stage system when using mixer settlers, the last stage is usually aqueous continuous to minimize the solvent entrainment losses.
- Antic-static agents are known to have been used in at least one plant.
- The effect of surfactants on the phase separation was demonstrated by a slide showing the positive effect of washing the PLS with diluent prior to the extraction stage in order to maintain good coalescence.
- Washing of a contaminated solvent using a caustic solution showed positive results. This wash (which was first introduced into the Canadian uranium operations in about 1980) will be used in the new CVRD plant in Brazil.
- Clay treatment has been in practice in most of the Cu plants in the past 10-15 years to maintain a reasonable coalescence rate by removing "contaminants". The completeness and rate of treatment varies with each operation.

## The existing models for simulation of pulsed and reciprocating columns

- how well do they work in the real world?

by

Baruch Grinbaum

Bateman Solvent Extraction Division

SX workshop

Niagara Falls 2007

## The Challenge:

- Designing a column for a new application
- Predict the influence of various operational and process parameters on its performance

*without a pilot plant study*

*Similar to distillation*

## BASIC PROCESS REQUIREMENTS

Type of column and internals configuration

Usually done a-priori

(neither model nor experiment applied)

For design of a process with given flow rates and recovery the main parameters are:

- Diameter of the column (or number of parallel columns for huge flow rates).
- Height of the column

Determine the cost and the layout of the column

### Two additional parameters:

- Phase continuity – determines the solvent inventory
- Energy input – determines the size of the blowers/agitator's motors.

**That is all that the designer needs**

### Factors needed for understanding of the process

- Holdup
- Axial dispersion
- Drop size distribution  
(or the Sauter diameter of the drops)
- Flow regime

### Two basic approaches to design

1. The experimental or the "black box" approach.
2. Design based on theoretical considerations and modeling.

### The black box model

1. Find in the lab phase ratio, residence time and (optionally) NTU
2. Run a pilot and find the configuration that meets process criteria: phase continuity, flux and height of the column
3. Scale up

### NTU and HTU not essential!!!!

$$H = NTU \cdot HTU$$

- Scale up is done from H
- If experimental H is known  
NTU and HTU are immaterial for the designer  
(still very important for process engineer)
- Calculation of NTU relatively easy  
HTU – very complicated



### **Primitive but efficient**

The easiest, fastest and most accurate method to supply data to the designers.

*The most common one  
(not to say the only one)*

*in use for design of industrial columns*

Tells nothing about the process beyond the very conditions that were tested.

### **THE THEORETICAL MODELING APPROACH**

**3 types of theoretical approaches:**

**Correlations**

**Drop size distribution**

**CFD**

### **Correlations to estimate single variable**

- **Holdup (flooding, flux)**
- **Axial mixing**
- **Mass transfer coefficient (HTU)**
- **Drop size (HTU, energy input)**
- **Flow regime (flux, energy input)**
- **Phase inversion**

- **Relatively easy for application (except from the drop size)**
- **A lot of data in the literature**
- **Give a good insight to processes even if not accurate enough for design**
- **For troubleshooting easy to obtain accurate parameters, incorporate in a simple Excel program and use in a plant.**

## Drop population balances

Try to replace small scale piloting  
Enable excellent prediction of outlets as a function of physical parameters  
At present limited to well studied systems, physical extraction and small columns  
The lab work to obtain the drop sizes very difficult and time consuming

## CFD

The most sophisticated approach  
Used to solve parts of processes  
Applied in industry  
Looooong time before a model will simulate any new SX process in a column like in distillation

## How useful the models are?

- a. Can a process be developed and scaled up without piloting at all?  
No
- b. Can the existing models predict *a-priori* what dispersion to use, what flux, HTU, holdup and drop size to expect?  
May help, but must be verified
- c. Can the models predict what happens in the industrial column if some physical properties change without additional experiments?
- YES

*At the current level of knowledge  
A pilot is a must anyway*

*So*

*Why to invest in modeling?*

The design engineer does not need theoretical or empirical correlations for holdup, drop size, or axial-dispersion

Practically he may disregard these properties.

Who needs the models?  
(besides the academics)

- The R&D engineers who make process updates and troubleshooting
- The plant engineers who are going to run the columns
- Column manufacturers who want to improve their equipment

**And in the bottom line this is the majority of the work on SX.**

How the models may be applied?

- Incorporation in a user friendly simulators (similar to ASPEN)
- Cooperation between academia and industry is essential for the application

**IN-LINE MIXERS**

Gordon Ritcey (G.M. Ritcey & Associates Inc.)

The Power Point presentation which accompanied the keynote paper on In-Line Mixers is presented in the following pages.

## **IN-LINE MIXER UNITS**

**G.M. RITCEY**

## **IN-LINE MIXER UNITS**

- ❖ TYPE: LIGHTNING
- ❖ ELEMENTS: 220 mm LENGTH x 25 MM DIAMETER
- ❖ GLASS PIPE: 25 MM DIAM TO ACCEPT MIXERS
- ❖ CONFIGURATION: HORIZONTAL

**G.M. Ritcey & Associates**

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## **SETTLER**

- ❖ DIMENSIONS: 275 mm W x 1215 mm L x 650 mm D
- ❖ CONSTRUCTION: GLASS SIDES, STAINLESS STEEL ENDS REINFORCED WITH STAINLESS STEEL
- ❖ PICKET FENCES: 2 OFFSET ROWS of 25 mm OD POLY PIPE; ALSO SIMILAR SS 316 PIPE, EXTENDING THE DEPTH OF SETTLER

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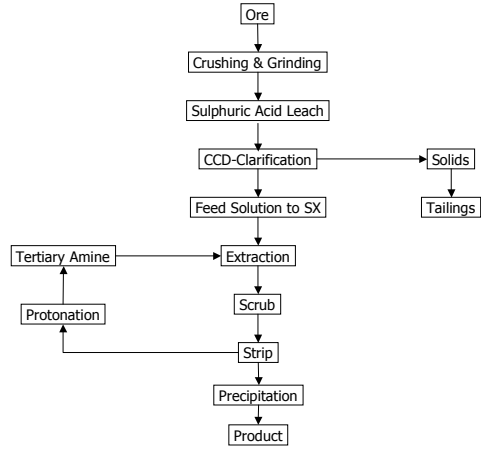
## **IN-LINE MIXERS**

- ❖ For the extraction of uranium by a tertiary amine from ore leach sulphuric acid solutions

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## URANIUM ORE PROCESSING FLOWSHEET



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## EXTRACTION SYSTEM

- ❖ PLS (TYPICAL) 1 g U/L, pH 1.5
- ❖ SOLVENT 2.5% Alamine 336 in ShellSol 2046 Diluent

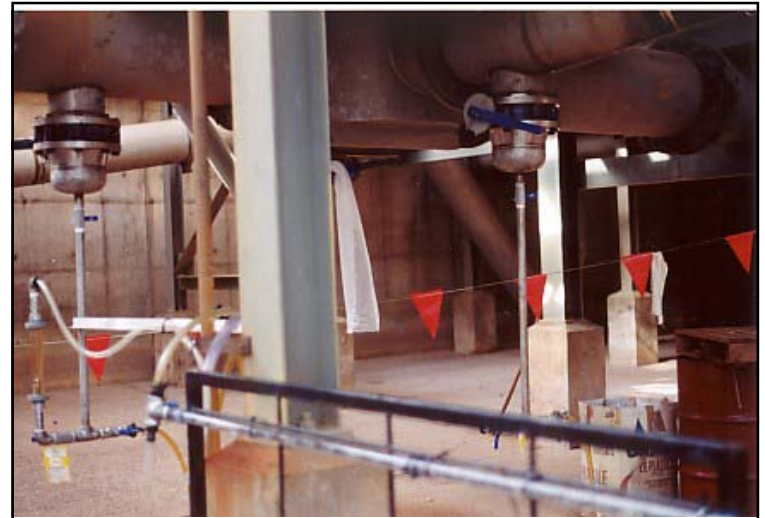
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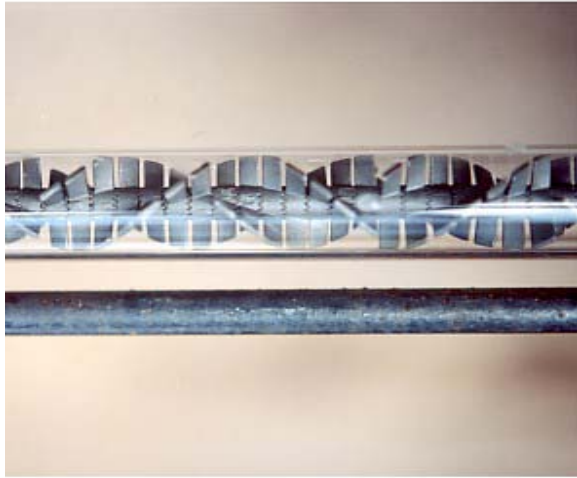
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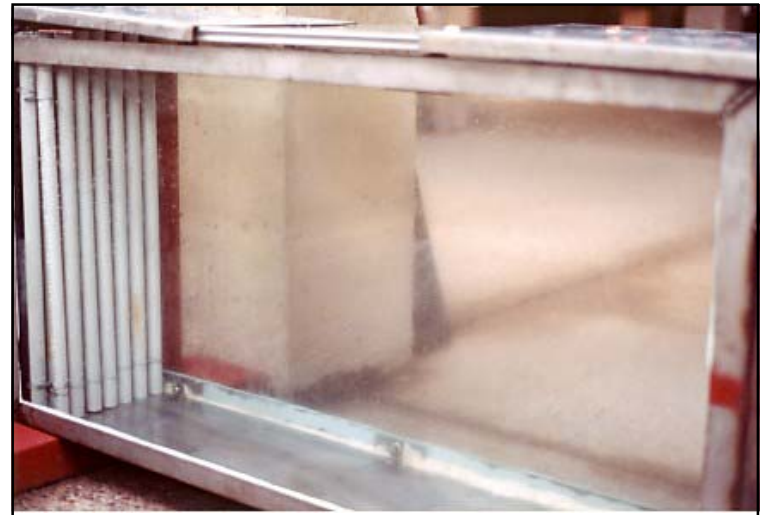
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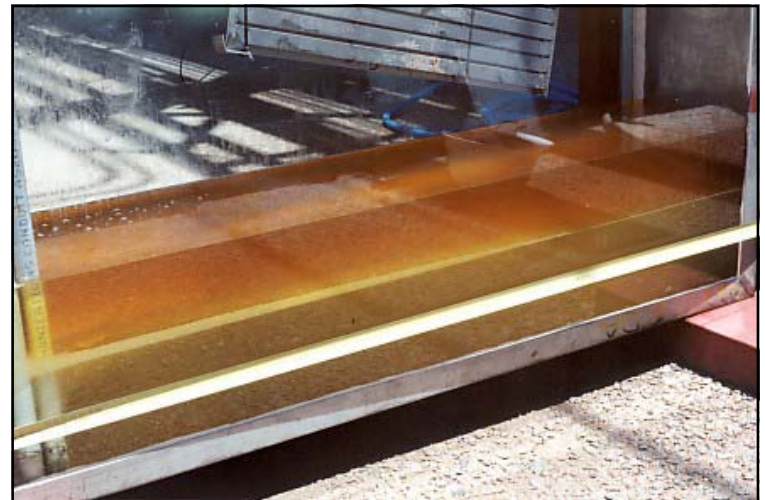
### PRELIMINARY TESTS

#### FLOODING

- ❖ FOR EXTRACTION ----- 17.2 l/M TOTAL, A/O 2/1
- ❖ 9-Element and 1215 Length of Settler
- ❖ Subsequent Tests Run at 15% Below Flooding

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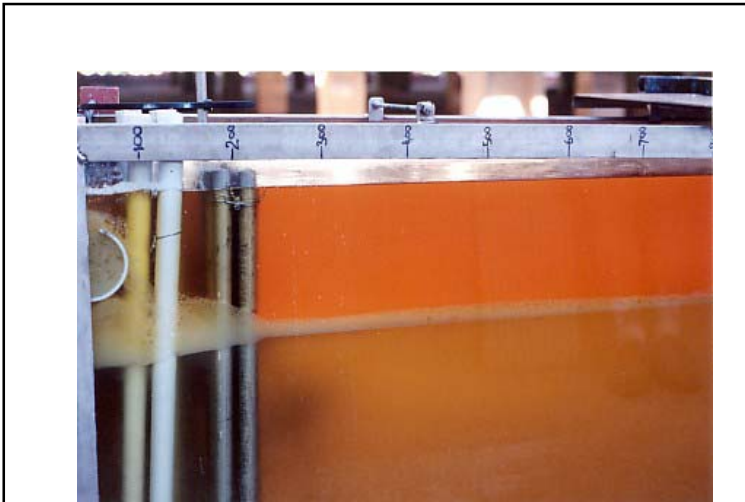
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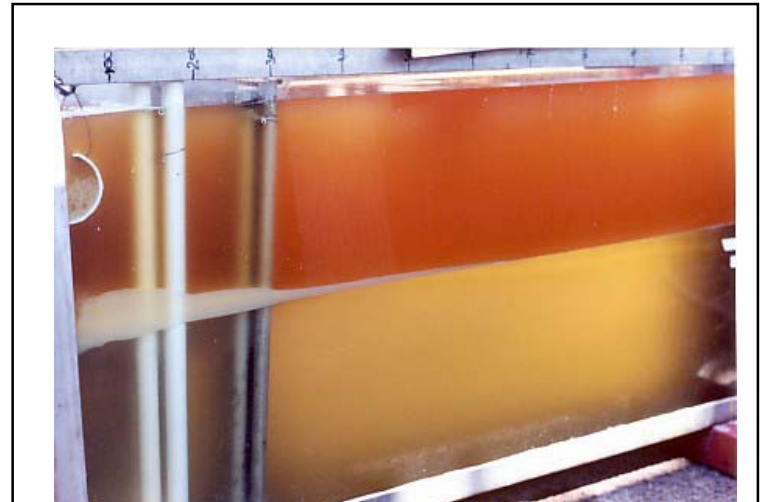
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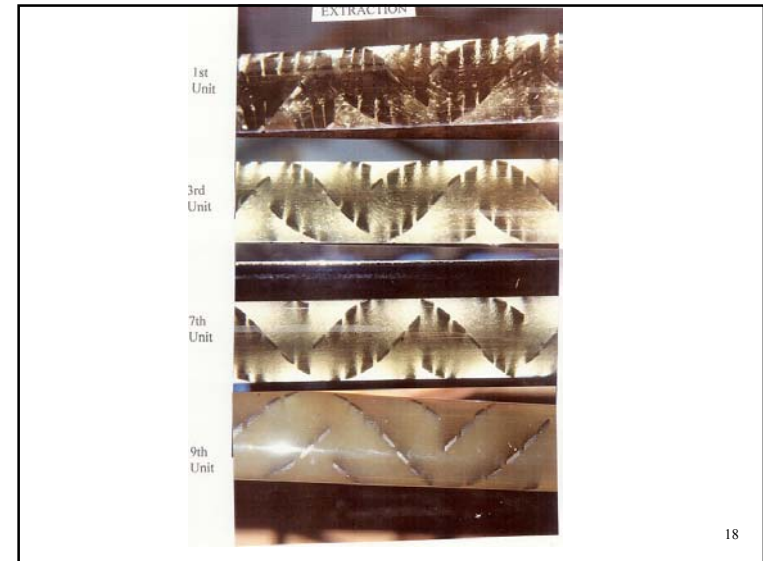
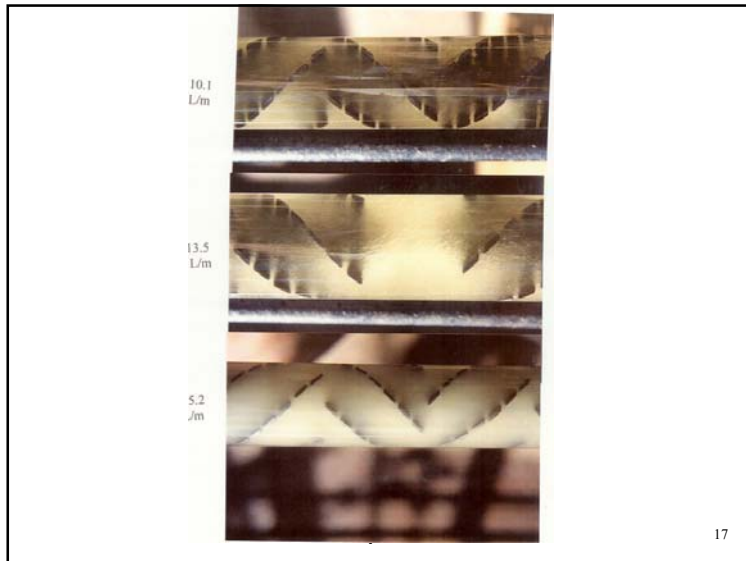


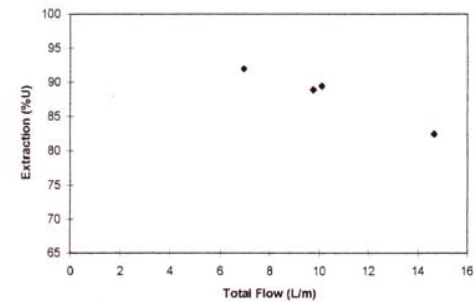
Table 1: Comparison of Pilot Plant Results with Plant and Bench Scale Shake-out

Extraction Units Number	Flow L/m	Extraction Bench	Efficiency Plant	(%U) Pilot Plant
9	6.95	89.6	90.5	91.9
9	6.95	80.5	82.1	82.3
9	9.75	82.4	81.9	88.9
9	14.65	77.7	80.0	82.9
9	6.95	80.1	95.1	76.3
9	6.95	84.4	81.2	83.1
9	6.95	79.4	80.3	77.9
9	6.95	77.7	80.0	82.4

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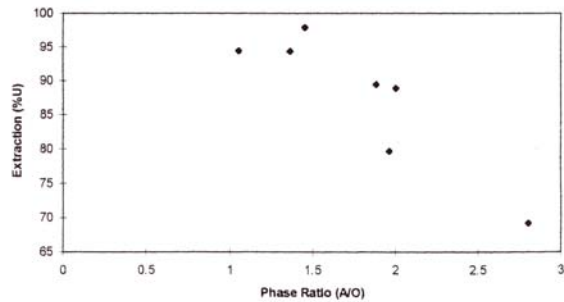
Figure 1: Extraction vs Flow Rate  
A/O 2/1 6 Extraction Units



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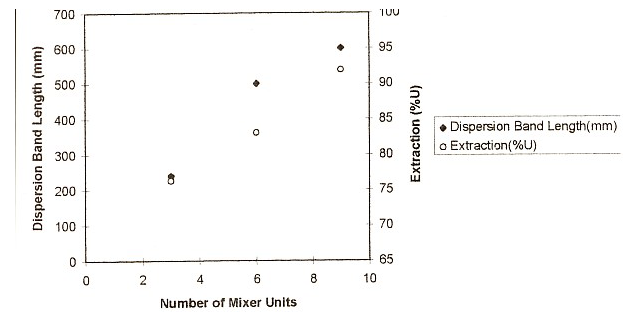
Figure 2: Phase Ratio vs Extraction  
9 Extraction Units



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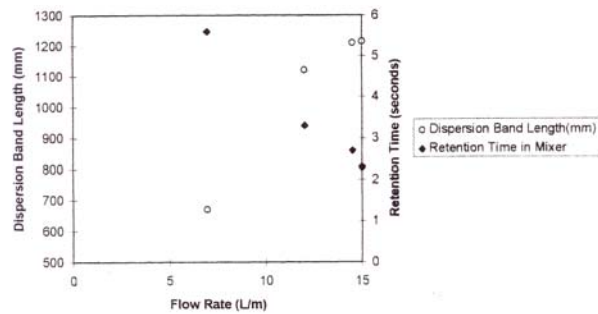
Figure 3: Mixer Units vs Dispersion Band Length & Extraction  
A/O 2/1 Flow 6.95 L/m



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Figure 4: Flow vs Mixer Time & Dispersion Band Length  
A/O 2/1 6 Extraction Units



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Drop Size Measurements vs. Number of Units and Total Flow

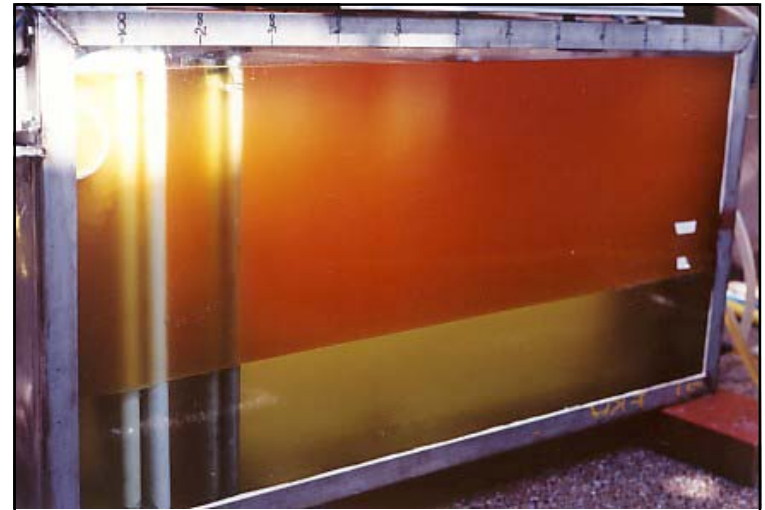
Unit No.	Total Flow L/m	U Extraction %	Drop Size mm
1	6.95		0.338–1.35
3	6.95		0.176–0.704
5	6.95		<0.032–0.324
7	6.95		<0.035–0.352
9	6.95	79.7	<0.032–0.324
9	10.1	89.5	<0.032–0.658
9	13.5	91.9	<0.032–0.305
9	15.2	89.5	<0.032

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WASH STAGE:

- ❖ PHASE SEPARATION RAPID
- ❖ < 1 MIN SEPARATION TIME
- ❖ NO DISPERSION BAND
- ❖ A SMALL SETTLER THEREFORE REQUIRED



PROTONATION STAGE

- ❖ HIGH O/A RATIO
- ❖ PHASE SEPARATION < 1 MIN
- ❖ NO DISPERSION BAND
- ❖ AT LOW 10 g/L ACID – ORGANIC CLOUDY
- ❖ CLARITY IMPROVED AS ACID INCREASED TO 40 g/L

OPTIMUM CONDITIONS:

- ❖ O/A 4/1 OR HIGHER
- ❖ 3 ELEMENTS
- ❖ PROTONATION WITH 40 g/L H<sub>2</sub>SO<sub>4</sub>
- ❖ TOTAL FLOW = 15 L/m
- ❖ AT OPTIMUM CONDITIONS: 95% U EXTRACTION ACHIEVED



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## CONCLUSIONS

IN-LINE MIXER TESTS DEMONSTRATED: HIGH STAGE EFFICIENCY ON U PROCESS SOLUTION

### ADVANTAGES INCLUDED

- ❖ NO REQUIREMENT TO MAINTAIN A SPECIFIC PHASE CONTINUITY IN MIXING
- ❖ DECREASED SOLVENT INVENTORY AS NO RECYCLE REQUIREMENTS
- ❖ FINE DROPLETS PRODUCED AND HIGH MASS TRANSFER EFFICIENCY WITHOUT HIGH SHEAR AND TURBULENCE

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- ❖ NO POSSIBILITY TO DRAW AIR INTO MIXING WHICH PRODUCES SERIOUS PROBLEMS IN THE CONVENTIONAL HIGH SHEAR PUMP-MIX DESIGN
- ❖ IMPROVED SETTLING RATES , AND THEREFORE INCREASED SETTLER UTILIZATION
- ❖ DECREASED STABLE EMULSIONS AND CRUD FORMATION, AND THUS DECREASED SOLVENT LOSSES
- ❖ DEMONSTRATED VIABLE FOR EXTRACTION, PROTONATION, AND WASHING SECTIONS

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- ❖ DROPLET SIZE DECREASES WITH INCREASED FLOW AND WITH INCREASED MIXER UNITS, INCREASING SETTLER DISPERSION BAND LENGTH
- ❖ SETTLER REQUIREMENTS APPEAR TO BE AT LEAST ONE HALF THAT REQUIRED COMPARED TO CONVENTIONAL PUMP-MIXING
- ❖ IN BOTH THE PROTONATION AND WASH STAGES, THE DISPERSION BANDS WERE FLAT, WITH RAPID COALESCENCE: THUS SETTLER REQUIREMENTS WOULD BE MINIMAL
- ❖ SETTLER REQUIREMENTS ARE REDUCED WHEN THE MIXED FLOWS ARE ORGANIC CONTINUOUS, AT HIGH O/A RATIOS AS IN THE PROTONATION AND WASH SECTIONS

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- ❖ NO TESTS WERE PERFORMED IN STRIP CIRCUIT, ALTHOUGH KINETICS OF STRIPPING MAY PROVE IN-LINE MIXING SUITABLE
- ❖ THEN pH CONTROL WOULD BE FACILITATED BY INTRODUCTION OF THE REAGENT DIRECTLY INTO THE PIPE
- ❖ DISSIMILAR PICKET FENCE MATERIAL BETTER THAN A SINGLE MATERIAL

THIS CONTINUOUS TEST DATA INDICATES

- ❖ THE IN-LINE MIXER COULD REPLACE THE AGITATED PUMP MIXER; USUALLY DESIGNED FOR 1.5 – 2 MIN COMPARED TO < 10 SEC FOR IN-LINE MIXERS IN THE URANIUM SX PROCESS
- ❖ THE PRESENT OR MODIFIED SETTLER COULD STILL BE UTILIZED WITH HIGHER CAPACITY

THIS IS AN ALTERNATIVE TO CONVENTIONAL CONTACTING. COULD BE ATTRACTIVE TO:

- ❖ A MILL PLANNING AN EXPANSION
- ❖ A NEW MILL SO AS TO REDUCE SHEAR, SETTLER REQUIREMENTS, ETC...

**SOME CALCULATIONS**

Vol of 1 Mixer Unit = 0.000107 m<sup>3</sup>

Vol of 6 Mixer Unit = 0.00064 m<sup>3</sup>

Vol of 9 Mixer Unit = 0.00096 m<sup>3</sup>

Time of contact of mixers: At about 85% of flooding flow of 14.25 L/m, or 855 L/h or 0.885 m<sup>3</sup>/h

9 EXTRACTION UNITS,

Then 0.00097/0.885 = 0.0011 H, or 0.0011 H x 3600 sec

= 4.0 sec / 9 mixer Elements

= 2.7 sec / 6 mixer Elements

Velocity through mixer at 9.75 L/M = 32 cm/sec

Flow capacity in the mixer at a flow of 14.25 L/M = 1716 M<sup>3</sup>/M<sup>2</sup>/H at a flow of 14.25 L/M = 1716 M<sup>3</sup>/M<sup>2</sup>/H

*(Based on pilot plant settler design and performance.)*

Settling Capacity – Based on 50 % of Settler Length and 14.25 L/M Total flow in this settler design: 5.2 M<sup>3</sup>/M<sup>2</sup>/H

• IN-LINE MIXERS

- LIMITED USE TO DATE AS A SX CONTACTOR (U)

- NOT VERY SUITABLE FOR SLOW KINETICS OTHER THAN AS A PRE-MIX

- GENERAL ADVANTAGES OF IN-LINE MIXERS

:

- NO REQUIREMENT TO MAINTAIN A SPECIFIC PHASE CONTINUITY OR PHASE RATIO
- DECREASED SOLVENT INVENTORY
- NO RECYCLE REQUIREMENTS
- FINE DROPLETS PRODUCED AND HIGH MASS TRANSFER EFFICIENCY WITHOUT HIGH SHEAR AND TURBULENCE
- NO POSSIBILITY TO DRAW AIR INTO MIXING
- IMPROVED SETTLING RATES
- DECREASED STABLE EMULSIONS AND CRUD FORMATION, AND
- DECREASED ENTRAINMENT LOSSES

### PLANT DESIGN CONSIDERATIONS

Mark Vancas (Bateman Litwin)

The Power Point presentation which accompanied the keynote paper on Plant Design Considerations is shown following this section. Following the presentation, the following points were discussed:

- The design is often based on a flux ratio of  $5\text{m}^3/\text{m}^2/\text{sec}$ .
- The general “rule of thumb” in the design (e.g. Cu) for the linear velocity is 3 cm/sec.
- In the case of the Bulong Nickel plant (and several other operations), deep settler tests were used to obtain the design.
- Measurement of the amount of entrainment will assist in the design.
- As regards materials of construction, FRP and metals have been used for most construction, while PVC on occasion for internals.
- The static electricity criteria is  $< 1$  m/sec into the tank, while Shell state  $< 7$  m/sec. Dissipation of the charge will occur through proper grounding and allowing sufficient time in the organic tank.
- Mists are generated, in part, by the overflow (and drop) into the launder.
- As regards the use of stainless steel in construction, grades have included 316 (or better), 2507, 225 etc.
- Mixer settlers within buildings are covered and vented to atmosphere.
- For safety purposes in the case of a fire, the design is for the organic to be discharged from all settlers to external ponds. The discharge in a short time from very large settlers could be a problem.
- Large settlers of 30 m x 30 m are covered by a roof.
- For fire control, foam and sprays can be used. Some foams (Triple F seems the best) do not adversely affect the solvent extraction process, but the foam has to first be removed by washing prior to running the SX circuit.
- Between circuits there should be a distance of 6 m space to isolate in case of fire.
- In the discharge from the settlers, only the organic phase should be discharge, in full lines.
- Linear velocity and flux are important in the design—an increase in these parameters results in a decrease in performance.
- A shallow dispersion band width results in higher entrainment.
- Picket fences decrease the linear velocity, and the positioning of the fences will be dictated by the particular system.

## **State of the Art and Future Directions in Solvent Extraction - 2007**

- The Holmes & Narver settler design, in which there were vertical baffles to slow the linear velocity, were probably a good design and perhaps this “old” design should be again considered.
- There should be an increased focus on the mixer in order to decrease the high shear and therefore the extremely small droplets that are produced. The pumping should be separate from the mixing.
- In-line mixers should be considered.
- Perhaps a shaft containing 2 impellers should be considered in the design.
- In Cu circuits, the arbitrary mix time is 2-3 minutes in extraction and 2 minutes for stripping.
- Also in Cu extraction, at ratios in the mixer box of 1:1 and 2:1 A:O, kinetic tests have been performed in which the mixing was varied over the range of 100-1000 rpm in a rectangular box. In extraction, the extraction efficiency increased with increase in rpm.
- Discussion on the phase ratio that should be maintained in the mixer indicated a range of about 1:1 to 2:1
- In some mixers in Chile a straight blade was changed to a curved impeller and the latter showed improved reduction on solvent losses. At Chino (Phelps Dodge) there was no reduction noted with the change, but that may be due to increased flows at Chino.
- Lightning mixers appear easier to maintain compared to the Spirox of Outokumpu which require a plant shut-down for maintenance.
- There was a good discussion on the possible use of in-line mixers and a power point presentation amplified that discussion. There appeared to be good agreement on the positive aspects of the in-line mixers to replace the conventional high shear mixers.
- In addition to the use of in-line mixers to provide mass transfer, it was noted that they have been successfully used in plant operation for the mixing (pre-equilibration of DEHPA) with ammonia in the extraction of Co and separation from Ni process.
- As regards to circuit configuration, most of the variations have been implemented in Cu operations. The circuits were originally designed as series, and subsequently many plants incorporated series-parallel configurations. Prediction of design has been by computer programs. By making a change to series-parallel there is a decrease in capital costs but an increase in operating costs. Such a design may provide some additional flexibility. Multiple trains have been designed. The design was originally used for dump leach operations.
- With increasing restrictions imposed by insurance companies, the design and development of a plant has become increasingly more difficult.

## Gordon Ritcey SX Workshop 2007

### Plant Design Considerations

Presented by:  
Mark F. Vancas

### Plant Design Considerations

#### Design Criteria

- Production Rate
- PLS Grade
- PLS Flowrate

### Plant Design Considerations

#### Design Criteria

- Mixing times
- Flux Rates
- Velocities
- Advance Ratios
- Mixing Ratios
- Phase Depths
- Maximum Organic Loading
- Maximum Organic Transfer
- Cu/Fe Selectivity
- Organic Entrainment in Aqueous

### Plant Design Considerations

#### Design Criteria

- Aqueous Entrainment in Organic
- Circuit Configuration
- Number of Mixing Stages
- Impeller Types and Tip Speeds
- Materials of Construction
- Static Electricity Reduction and/or Elimination
- Fire Protection



## **Plant Design Considerations**

### **Other Considerations**

- Crud
- Operability
- Maintenance
- Client Desires
- Niceties and Necessities
- Client Knowledge
- Operating staff knowledge & capabilities
- Location and Plant Layout
- Piping Design particularly around Pump-Mixers
- Level of Automation
- Cross contamination of series circuits

### PLANT SETTLER COMMISSIONING

Mark Vancas (Bateman Litwin)

The Power Point presentation which accompanied the keynote paper on the Plant Settler Commissioning is shown following this section. Following the presentation, the following points were discussed:

#### On start-up:

- 1) Fill the circuit with water and test for leaks throughout the system.
- 2) Bleed in the PLS.
- 3) Add copper sulphate to the strip circuit to provide the electrolyte.
- 4) Add diluent.
- 5) Extractant is added to the launder to the loaded organic tank.
- 6) Control of the plant:
  - i) flowmeters types: ultrasonic, pressure, orifice meters and others
  - ii) phase continuity, measure by conductivity in the mixer. If the phases invert (flip) then the situation can be returned to the desired phase continuity by shutting off the flow of one phase for a few minutes.
  - iii) O/A ratios
  - iv) pH – operator control in some plants; control room in others. Control is best obtained by scheduled sampling so there is a “walk-around” to check the plant at the same time.
  - v) phase separation times – tests should be performed each shift, and certainly at least daily.
  - vi) Loading tests – daily and more frequent depending upon PLS tenor, flow control, etc. Low loading does not necessarily indicate reagent degradation, but possible solvent fouling.
- 7) Analysis for control – every couple of hours
- 8) A set of “standard samples” often can be useful to visually monitor the circuit.
- 9) Shut-down – often when one mixer shuts down, all mixers are shut down

## BATEMAN SETTLER® COMMISSIONING

Presented By

**Mark F. Vancas**  
BSX Process Specialist

bsxtucson@aol.com



BATEMAN SETTLER® COMMISSIONING



Piedras Verdes

Mantos de la Luna



BATEMAN SETTLER® COMMISSIONING

### Basic Criteria

Plant	Mantos de la Luna	Piedras Verdes
Location	Tocopilla, Chile	Alamos, Sonora, Mexico
Rated Production	25,000 t/a	32,000 t/a
Leach Material and Method	Crushed, Agglomerated Ore on a dynamic Pad	ROM ore on permanent pad of approximately 100 m ultimate height
Leaching Media	Acidified Sea Water	Acidified Potable Water
Materials of Construction	FRP, HDPE	Stainless Steel
PLS Grade	4.2 g/l design, 3 g/l at present	3.1 g/l design, 2 g/l at present
PLS Flowrate	750 m <sup>3</sup> /h, 700 m <sup>3</sup> /h at present	1350 m <sup>3</sup> /h design, 2000 m <sup>3</sup> /h at present
Design Unit Flowrate	3.66 m <sup>3</sup> /h/m <sup>2</sup>	5 m <sup>3</sup> /h/m <sup>2</sup>
Circuit Configuration	2 extract, 1 wash, 2 strip	2 extract, 1 strip
Control Philosophy	Fully automatic	Manual



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**Let's now go on a photographic tour of both sites.**

**In the process we will:**

**Look at some Client/site specific details**

**Discuss some of the Lessons Learned**

**Have some fun, and**

**Hopefully generate some discussion**



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ALTA 2007



MDL uses pumped PLS feed



PV uses gravity flow of PLS



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ALTA 2007

Mantos de la Luna

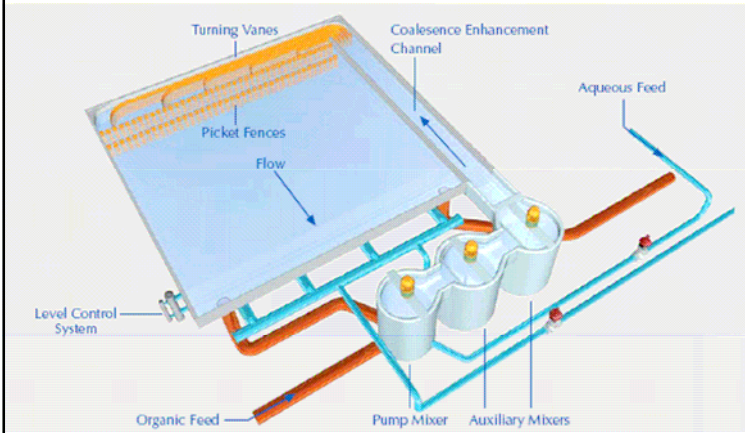


The settlers for MDL can be called the genuine **BATEMAN SETTLER®**



BATEMAN SETTLER® COMMISSIONING

ALTA 2007



BATEMAN SETTLER® COMMISSIONING

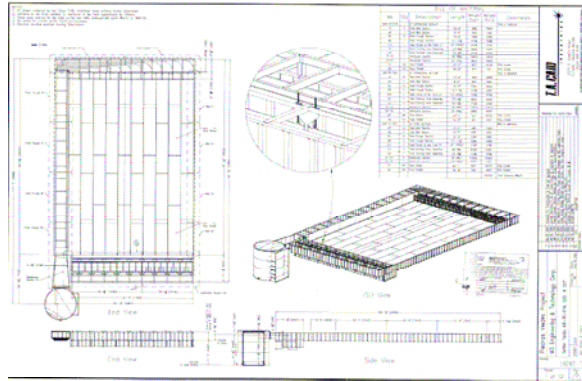
ALTA 2007

MDL control circuit includes interface and organic advance controls



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ALTA 2007

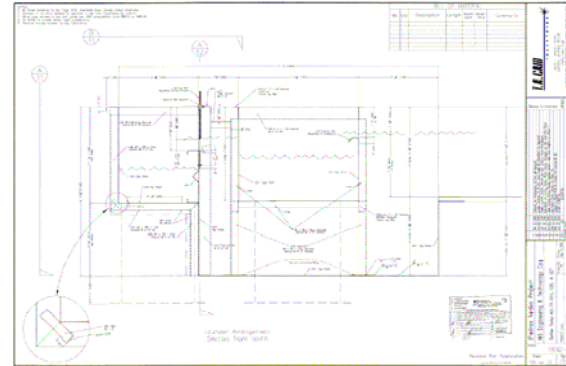


PV on the other hand uses traditional organic and aqueous discharge weirs however I did insist on retaining the submerged aqueous launder.



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ALTA 2007



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ALTA 2007



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MDL has automatic valves in many locations including bypasses and manual valves



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ALTA 2007

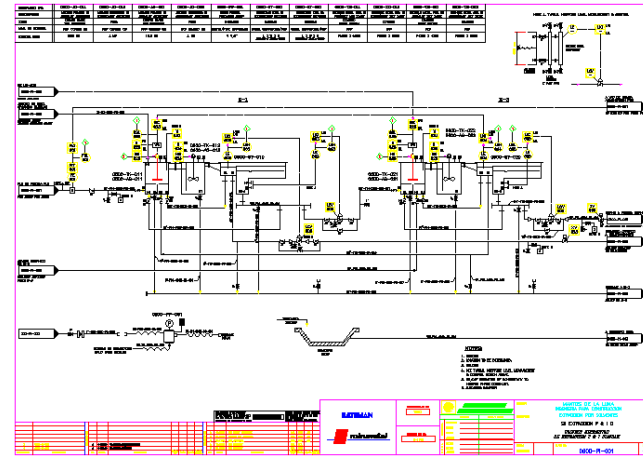


Mantos de la Luna was the first fully automatic BATEMAN SETTLER® installation.



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ALTA 2007



BATEMAN SETTLER® COMMISSIONING

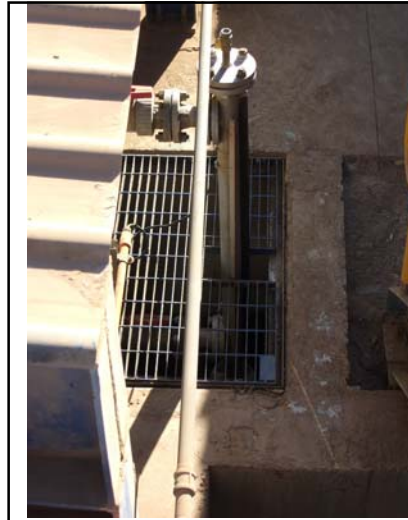


ALTA 2007

This is what I consider perfect launder control, but it was not this way at the start.



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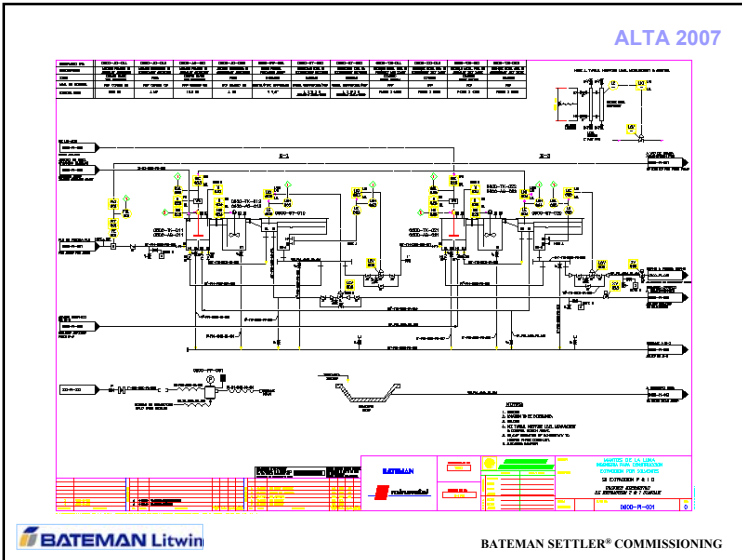
ALTA 2007

Control of the interface in the settlers was problematic initially.

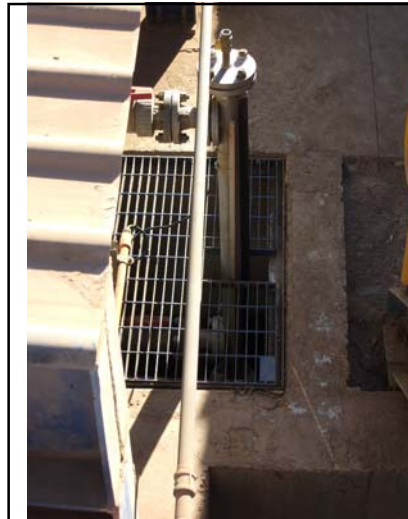
Fluctuations in organic flow were compounded by instrumentation that was not installed correctly.



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ALTA 2007

Control of the interface in the settlers was problematic initially.

Fluctuations in organic flow were compounded by instrumentation that was not installed correctly.



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ALTA 2007

Solenoid valves were installed to avoid reagent loss in case of an automatic valve failure during shut down but, inappropriately applied, thin walled pipe collapsed under vacuum.



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ALTA 2007

What about performance of this automatic system?

Entrainment of organic in the raffinate is generally lower than 30 ppm and entrainment of aqueous in the loaded organic has consistently been less than 100 ppm.

This is particularly beneficial because the leach solution is sea water at more than 20,000 ppm Cl but yet the Cl levels in the electrolyte are approximately 15 ppm and pose no danger to the stainless steel cathodes used in EW.



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ALTA 2007

The biggest headache at PV was getting the pump-mixer impellers set within specifications of the false bottoms in the primary mixers.



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ALTA 2007



 BATEMAN Litwin

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ALTA 2007

To date the impellers have been able to move 4000 m<sup>3</sup>/h of combined flow with impeller speeds of 95% of maximum and the organic advance valves less than 100% open.

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Piedras Verdes has a fire fighting foam system that was tested before the diluent was introduced to the circuit.

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ALTA 2007

To avoid agitation and air entrainment as much as possible gravity lines into tanks were piped such that the discharge was below the surface.



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ALTA 2007



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ALTA 2007

In the electrolyte tanks we outsmarted ourselves. We turned down the inlet but we also turned down the overflow so as to stop organic (if present) from flowing from one tank to the other.



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ALTA 2007

Well, we turned down the other end of the overflow as also and in doing so created a siphon.



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We tried to reduce capital costs by using gaskets of EPDM surrounded by a teflon envelope.

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ALTA 2007



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BATEMAN SETTLER® COMMISSIONING

Commissioning is where one sees the good and bad features of a particular design.

Commissioning is where one learns how to improve plant design.

For me, commissioning is the most exciting time of a project.

### OPERATING ASPECTS

Mark Vancas (Bateman Litwin)

The Power Point presentation which accompanied the keynote paper on Operating Aspects is shown following this section. Following the presentation, the following points were discussed:

- 1) As regards crud, the generation of crud is greater when the circuit is running aqueous continuous.
- 2) Often 1-person is dedicated to crud removal in a plant using mixer settlers.
- 3) In column operation, the crud generation is considerably less, and in one plant the amount of crud generated in a week that accumulated at the interface was about 10 litres.
- 4) Following a rain period, there is more crud generated from a heap leach operation due to the increase in fines. Clarification prior to the mixer settler plant is often not economically feasible. The aim is generally to achieve 20 ppm suspended solids in the feed PLS to solvent extraction.
- 5) In the presence of the PLS + diluent the corrosion can be greater than the single solutions in contact with the material (synthetics, metals) so the choice of materials can be site specific.
- 6) The use of polymeric concrete was discussed for application to ponds and settler construction.
- 7) Columns have ranged from up to 3 m diameter. Any solids in the system are discharged with the raffinate or may sit at the interface. The control of the phase continuity is easy. Columns have been used in many situations and industries, and in the metals area have been successfully applied to several metal recovery systems, including: U, Zr, Co, Ni. Construction materials vary and may be site specific. FRP and stainless steel columns have been used and the internals have included stainless steel and Teflon.
- 8) Solvent can be degraded in several situations, and these degradation products will adversely affect the solvent extraction process.
  - i) when exposed to sunlight,
  - ii) bacteria
  - iii) concentrated H<sub>2</sub>SO<sub>4</sub> in some reagents
  - iv) high alkalinity in some reagents

All result in gradual degradation of the constituents.

- 9) Treatment of plant organic – pond organic and sump organic - requires treatment (clay is mostly used, although a caustic wash can be effective) before re-use to remove undesirable surfactants that would adversely affect the phase separation characteristics, the loading, discrimination and stripping.

## **OPERATING ASPECTS OF SX-EW**

(How to screw up an SX-EW Plant)

**Flowrates**

**Crud**

**Extractant Concentration**

**Organic Depth in the Settler**

**Crystallization**

**Launder Adjustments**

**Air**

**Organic Entrainment**

**Cobalt**

**Rectifier Adjustments**

**Mist Suppression**

**Details**

**Dealing with Management**

### SOLVENT LOSSES

Gordon Ritcey (G.M. Ritcey & Associates Inc.)

The Power Point presentation which accompanied the keynote paper on Solvent Losses is shown following this section. Following the presentation, the following points were discussed:

Constituent reagent losses are not equal, and the loss may be dependent on the molecular weights.

#### Fouling (poisoning)of the Solvent

- Fouling of the solvent by organic acids, e.g. humic and fulvic acids
- The poisoning may often be seasonal, particularly in the case where open pit operations exist and the runoff is high
- Prevention of loading the solvent of undesirable contaminants (organics as well as organic-metallic species) can be accomplished by a diluent wash of the aqueous phase to solvent extraction
- A caustic wash, at controlled alkaline pH, can be successful in the removal of the accumulated organic acids.
- Fouling is also due to the preferential loading and difficulty in stripping of some elements, such as the amphoteric elements. In such as case the control and removal from the solvent is usually accomplished by a caustic wash or a highly acidic wash.

#### Crud

- Some crud can be tolerated in certain circuits, otherwise a physical/mechanical problem results.
- The presence of large amounts of chloride ion (in a sulphate system) appears to inhibit interfacial problems
- Silica is a problem because of its many species of colloids and precipitates, due in part to the acidity and the amphoteric nature of the element. Can the use of zeta potential measurements on solids be of assistance in the management of a process that is generating crud?
- Treatment of crud may be accomplished by:
  - a) 3-phase centrifuges (newer ones are superior)
  - b) horizontal centrifuges appear to be excellent
  - c) tricanters
  - d) washing of the crud (e.g. raffinate)
- Can we design for crud in a plant?

#### Emulsions

- Emulsions are difficult because of the nature of the constituents and the type of formation (e.g. shear, surfactants)

#### Phase Continuity

- Control of phase continuity can minimize emulsions/cruds
- Recycle of the minor phase in the mixer, usually to maintain a desired phase continuity is accomplished at a ratio in the mix box of 1:1 of O:A. The control can be a problem. Can sufficient intimate mixing be achieved at high ratios (e.g. 1:10) in the mix box?

## **State of the Art and Future Directions in Solvent Extraction - 2007**

### Misting

- Minimize the distance the solution has to drop, maintain a full launder, and the design of the discharge are all important in reducing mist formation
- In addition to the loss of solvent and the adverse impact to the worker, the generation of mist becomes a flash point issue. The dielectric constants may be of use in prediction
- With mist generation, there are visible signs appearing in the settler, such as "fish eggs". These are often predominant in the extraction stage.

### Recovery of Solvent from Raffinate

-Some of the methods & equipment that have been used with varying success are:

- Coalescers
- Activated carbon & filtration
- Spintex filters
- Flotation treatment of the raffinate before discharge
- Flotation treatment in the pond
- After settlers
- Electrical coalescence has been used in pharmaceutical but not in mining because of the variation in PLS

A value of 0.7 kg reagent/T Cu produced was stated as a reasonable amount of loss to expect.

One of the main problems of analysis and the design of recovery systems has been the sampling of streams when a "grab" sample is taken. A composite sample gathered "over time" would be preferable.

## SOLVENT LOSSES

G.M. RITCEY

### LOSSES DUE TO:

- SOLUBILITY
- ENTRAINMENT
- EVAPORATION
- MISTING
- DEGRADATION
- CRUD
- SAMPLING & SPILLAGE

## SOLVENT LOSS

### RELATED TO:

- REAGENT CHOICE
- EQUIPMENT SELECTION
- METHOD OF OPERATION
- NATURE OF FEED

## SOLVENT LOSSES CAUSES

- HIGH SHEAR,
- DROPLET SIZE VS. COALESCENCE
- EMULSIONS
- 3<sup>RD</sup> PHASE FORMATION
- DUAL CIRCUITS & CROSS-CONTAMINATION

## SOLUBLE LOSSES

### RECOVERY FROM RAFFINATE:

- RE-EXTRACTION WITH LOW BOILING SOLVENT
- STEAM STRIPPING
- ADSORPTION ON ACTIVATED CARBON

## MISTING

- HIGH SOLVENT LOSSES OF ALL COMPONENTS + METAL
- POSSIBLY CAUSED BY:
  - SURFACTANTS,
  - ORGANIC DEGRADATION,
  - SOLVENT POISONING
  - OVER-MIX,
  - EXCESSIVE AIR

## ENTRAINMENT

### MINIMIZED BY:

- SKIMMERS
- CENTRIFUGES
- COALESCERS
- SETTLING (AFTER-SETTLERS)
- FLOTATION
- ACTIVATED CARBON
- IN-LINE FILTERS, ETC----





## DEGRADATION

### CAUSED BY:

- HIGH TEMPERATURE
- OXIDATION (METALS, AQUEOUS SOLUTION)
- EXCESSIVE ACIDITY / ALKALINITY
- BIODEGRADABILITY

## RECYCLING SOLVENT

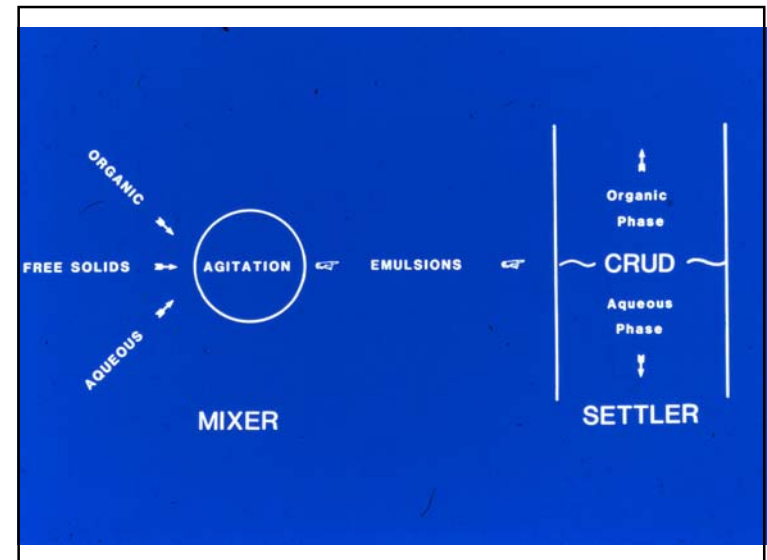
### PROBLEMS THAT MAY OCCUR:

- ACCUMULATION OF NON-STRIPPABLE METAL
- EXTRACTION SITES OCCUPIED BY A "POISON" WHICH MAY REACT IN STRIPPING (HYDROLYSIS)
- DEGRADATION OF THE EXTRACTANT
- SOLUBILITY OF DEGRADATION PRODUCTS IN AQUEOUS (PROBLEMS)

## CRUD

C. R. U. D.

Chalk River Unidentified Deposit





## CRUD

CRUD IS THE SLIMY MATERIAL THAT ACCUMULATES IN SETTLERS

### DERIVED FROM:

- PARTICLES IN FEED
- DUST FROM THE AIR
- PRECIPITATES
- BACTERIAL ACTION

## CRUD

### COMPOSITION

- Si, Al, Fe, Mg, P, SO<sub>4</sub>, CLAY, GYPSUM, FINES

## SOME CRUD SAMPLES MAJOR CONSTITUENTS

- URANIUM
  - Zr 8-10 %
  - Bi 35 %
  - Mo 5 %
- COPPER
  - Si 10-20, Cu 1-2, Fe 1, Pb 0.5 %
- COBALT
  - Si 10-25, Fe 15, Al 4 Mn 0.6, Mg 1.3 %
- NICKEL
  - Si 3.5, Ni 18

## CRUD

### FACTORS CAUSING CRUD

- SOLIDS
- HIGH SHEAR
- CARBOXYLIC ACIDS, HUMIC ACIDS
- SILICA
- PHOSPHORUS
- HYDROLYSIS OF EXTRACTED SPECIES
- BACTERIA, FUNGUS

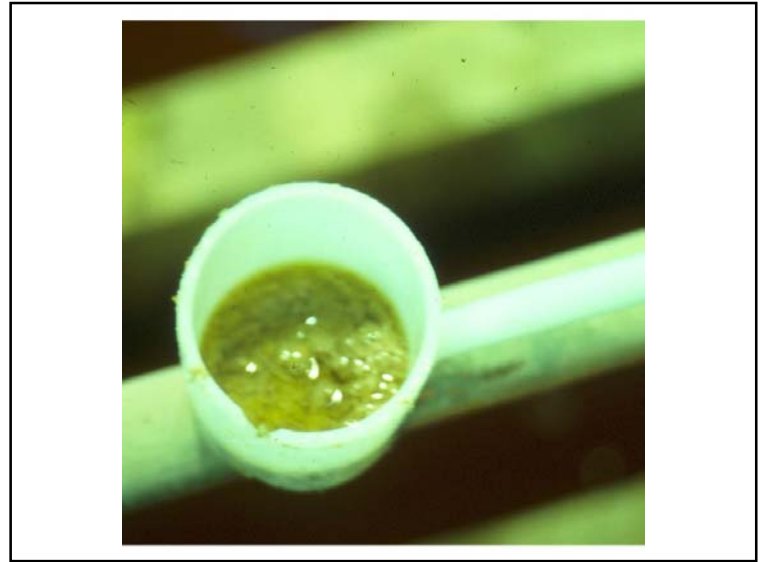
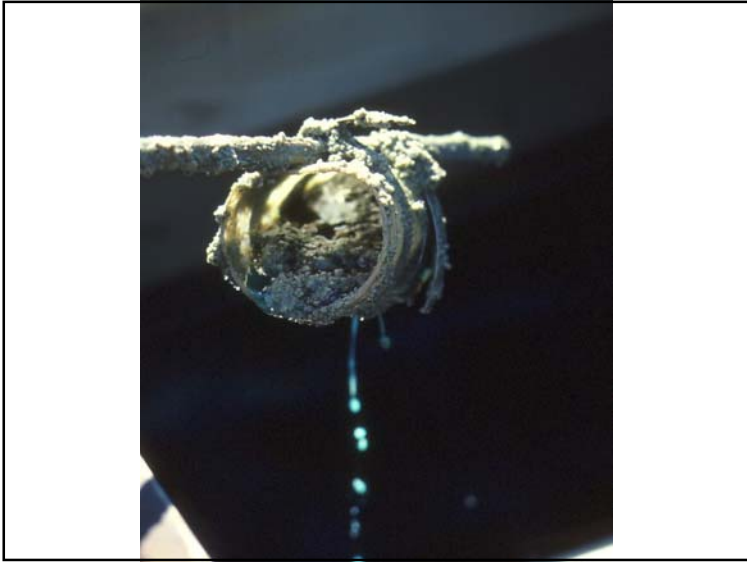
## SOME CRUD DESCRIPTIONS

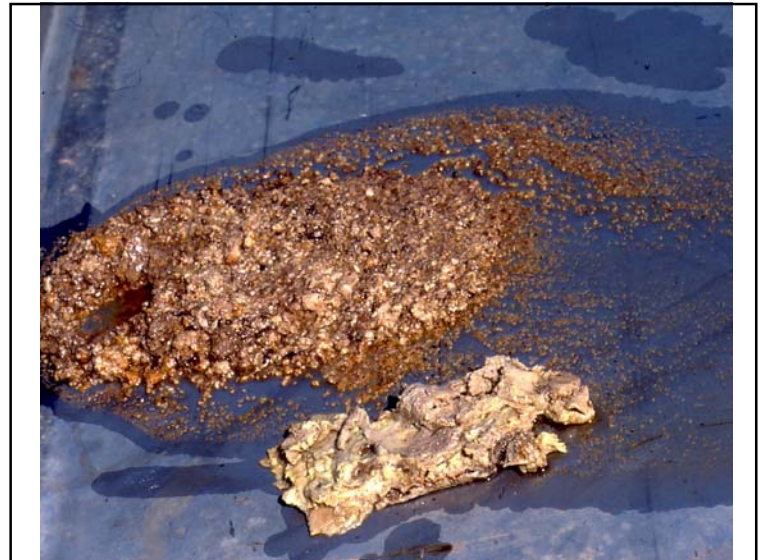
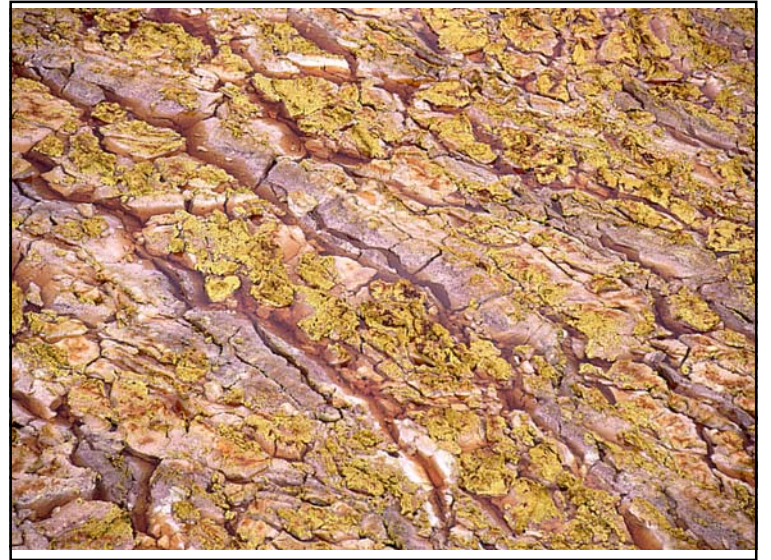
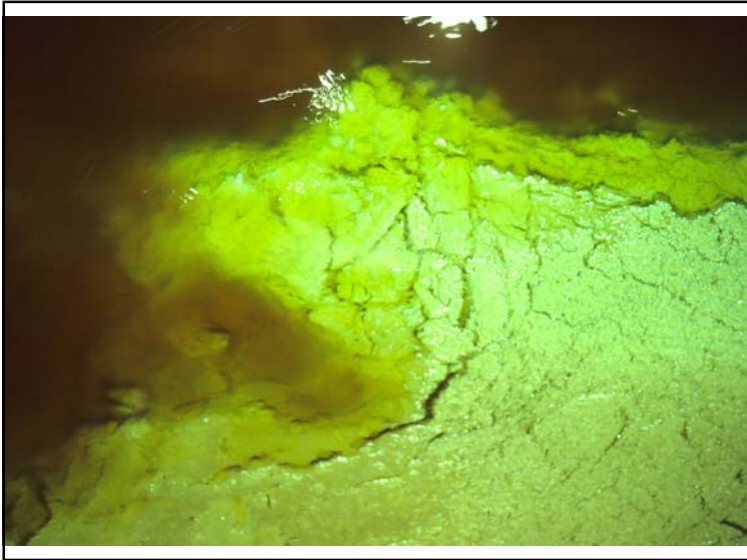
- COTTON-LIKE WHITE / YELLOW
- PORRIGE-LIKE SLIMES
- THICK VISCOUS BROWN MUD
- BROWN, GUMMY MATERIAL
- YELLOWISH, PLASTIC-LIKE
- BLACK-BROWN MATERIAL

## CONSEQUENCES OF CRUD

- LOSS OF SETTLER AREA AND INCREASED ENTRAINMENT LOSSES
- ADSORPTION LOSS SOLVENT ON CRUD SURFACES
- POOR EXTRACTION & STRIPPING
- WASH LIQUOR TO CCD INCREASES SOLUBLE METAL LOSSES
- IF EXTREME—MILL SHUT-DOWN REQUIRED

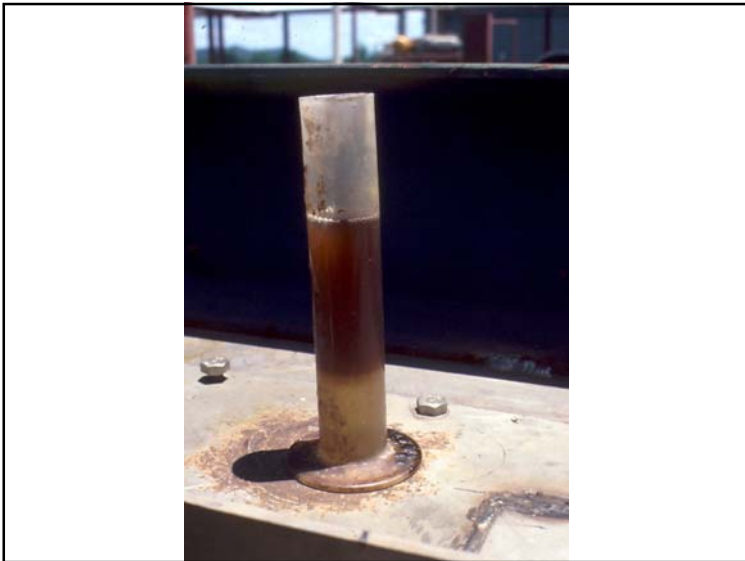








	NO HUMIC ACID				HUMIC ACID ADDED			
	FRESH	FEED	AGED	FEED	FRESH	FEED	AGED	FEED
	CLAR- IFIED	UNCLAR- IFIED	CLAR- IFIED	UNCLAR- IFIED	CLAR- IFIED	UNCLAR- IFIED	CLAR- IFIED	UNCLAR- IFIED
FRESH ORGANIC	CLEAN	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	CRUD	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
USED MILL ORGANIC	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>



## CRUD

### CRUD

- FORMATION
- TREATMENT
- PREVENTION

## CRUD TREATMENT

### TREAT CRUD FOR SOLVENT RECOVERY:

- WASH WITH RAFFINATE
- WASH WITH ACID, BASE, DILUENT
- CENTRIFUGE

## CRUD PREVENTION

- IMPROVE FEED CLARIFICATION
- AVOID OVERGRINDING (SLIMES)
- REMOVAL OF ORGANIC CONTAMINANTS (HUMIC ACIDS, CARBOXYLIC ACIDS) BY ACT. C ; OR DILUENT WASH OF AQUEOUS
- MINIMIZE OXIDANTS IN LEACHING
- AROMATIC DILUENT USE

(CONTINUED)

## CRUD PREVENTION

- CAREFUL SELECTION & USE OF FLOCCULENTS TO CCD (POLYACRYLAMIDES)
- O/A RATIOS & SATURATION IN EXTR'N TO SCRUB IMPURITIES
- WASH STAGE BETWEEN EXTRACTION AND STRIPPING
- SELECTION OF OTHER CONTACTORS

CONTINUED

## CRUD PREVENTION

- SCRUB LOADED SOLVENT (PARTICLES & AMPHOTERICS)
- HIGH MOL. WT. AMINES (EG ALAMINE 304 FOR U CIRCUITS)
- CAREFUL DESIGN, WARNING DEVICES, EFF. CONTROLS, & CLOSE ATTENTION TO OPERATION

**PARTICIPANTS LIST AND E-MAIL CONTACTS**

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