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**SMART-LEVEL SOLVEX:  
A NEW INSTRUMENT FOR QUANTIFYING PHASE DISENGAGEMENT**

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**ABSTRACT**

A variety of methods for the measurement of phase disengagement are in common use. These typically rely on visual observation of the time required for separation of the two phases, and are therefore dependent on operator experience and perception. The Smart-Level Solvex instrument electronically detects the rate of phase disengagement and separation time. Comparisons of the reproducibility and precision of the Smart-Level Solvex with three widely used conventional techniques are reported for selected copper, zinc, and uranium solvent-extraction systems.

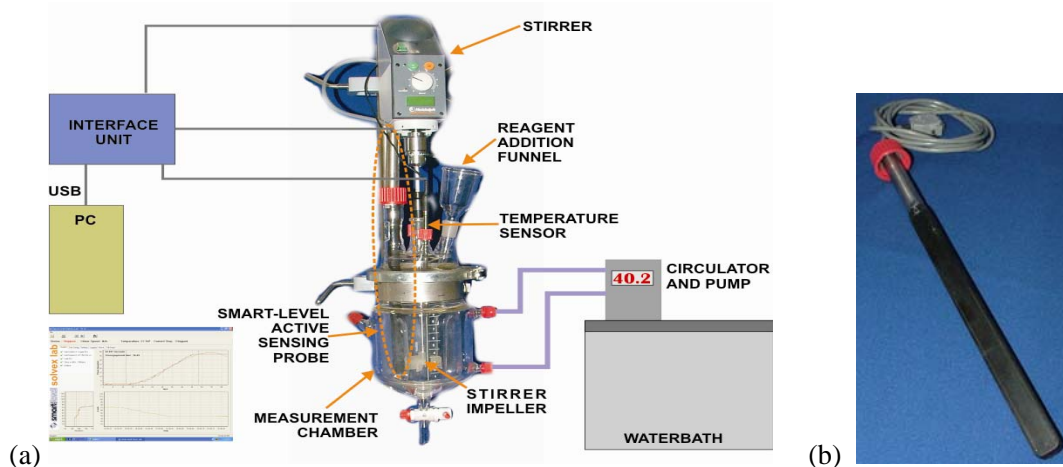
**INTRODUCTION**

The rate of phase disengagement is a reliable indication of the health of an organic phase, and can provide early warning of impending physical or chemical problems in an operating solvent-extraction (SX) circuit. Phase separation is dependent *inter alia* on aqueous-phase acidity, presence of surfactants (such as flocculants, coagulants, or electrowinning additives added in up- or downstream processes), degradation products of the extractant or diluent, changes in extractant concentration, and the presence of crud or suspended solids. Although phase separation is routinely measured on operating plants, there is lack of conformity in the methods used. These range from visual observation of phase separation in a measuring cylinder to more sophisticated systems which involve a fixed reactor geometry, controlled temperature, and set stirring time and speed before settling. Camera or video recording have been tested, but most methods rely on an operator to visually monitor the process and record the separation time or position of an interface. Reproducibility can vary widely, not only between operators, but also with a single operator from one measurement to the next.

The Smart-Level Solvex overcomes inherent problems of this measurement by electronic detection of the interfaces between the aqueous, organic, and emulsion phases, and algorithmic determination of the phase-separation time (PST). Comparison of the reproducibility and precision of the Smart-level Solvex with three widely used conventional techniques for this measurement is reported for selected SX systems.

**DESCRIPTION OF SMART-LEVEL SOLVEX**

The unit comprises a water-jacketed reactor with integrated water bath, variable speed stirrer, sensing probe, computer-based display and control software, and dedicated control panel or computer interface (Figure 1). The unique feature is the chemically resistant resistivity probe (150 mm length), with 128 sensors enabling a reading every 100 ms at 1.2 mm vertical resolution. Experimental parameters are fully programmable. A real-time phase disengagement profile is generated and data are recorded graphically or transformed to *Excel* spreadsheet format. The measurement principle relies on differences in conductivity between the phases: in addition to detection of aqueous and organic phases, this allows identification of emulsions, crud, and third-phase formation.



**Figure 1.** (a) Schematic of Smart-Level Solvex equipment. (b) Close-up of the detection probe.

## EXPERIMENTAL

### Solvent-Extraction Systems

For copper-oxime systems, the aqueous phase contained 6 g/L Cu, 2 g/L Fe (as sulfate salts) at pH 2, while the organic comprised 10 vol.% LIX<sup>®</sup> 984N or ACORGA<sup>®</sup> M5640 in the diluents SSX 210, ShellSol<sup>®</sup> 2325, or ShellSol A150. The zinc system used an aqueous phase containing (g/L) 31.5 Zn, 0.5 Ca, 0.5 Cu, 0.2 g/L Cd, 0.2 g/L Ni (as sulfates) at pH 4 and organic phase of 40 vol.% di(2-ethylhexyl)phosphoric acid (D2EHPA) in Escaid<sup>®</sup> 100. The uranium system employed a 4 g/L U plant solution and organic phase of 10 vol.% Alamine<sup>®</sup> 336, 5 vol.% isodecanol in ShellSol A150.

### Standard Methods for Determination of Phase Separation Times

The standard methods of Cognis [1], Cytec [2], and Técnicas Reunidas (TR) [3] were employed. Cognis employ a baffled glass beaker, TR use an open polyvinylchloride reactor with a tapered base and tap, while Cytec has a graduated water-jacketed glass vessel with a tapered base and tap to facilitate removal of the phases. The latter is also used for the Smart-Level Solvex. In all methods, prescribed volumes of each phase are

stirred under preset conditions, then allowed to separate and the time required for phase separation measured. In the Cytec and TR methods, the position of the interface is additionally recorded at regular intervals to obtain information regarding the rate of separation. The main differences between the methods are summarised in Table 1.

**Table 1.** Comparison of differences between the four experimental methods

Parameter	Cognis	Cytec	TR	SL Solvex
Volume of phases (mL)	350	400	1000	400
Temperature control	No	Yes	No	Yes
Mixing time (min)	5	3	3	3
Stirring speed (rpm)	1750	600	580	variable
PST measurement	stopwatch	stopwatch	stopwatch, ruler	electronic
Time profile data	No	Yes	Yes	Yes

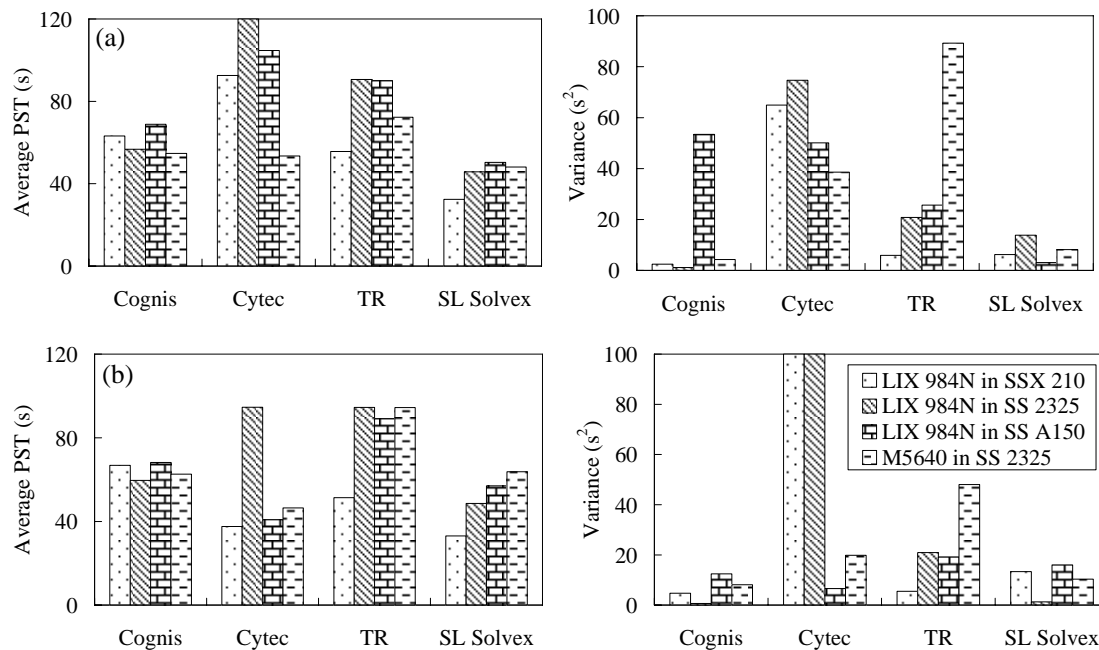
## Experimental Procedures

Clean dry glassware is essential for reproducible results. Each reactor was washed with laboratory detergent and water, and then sequentially rinsed with deionised water, ethanol, acetone, and deionised water again before each experiment. The four methods were compared for each SX system, under aqueous- and organic-continuous mixing conditions. In the first evaluation, the same aqueous and organic phases were recycled ten times under identical conditions and the PSTs recorded. In the second evaluation, fresh phases were employed each time and the experiment repeated six times. Statistical analysis of the results (using standard *Excel* functions) was undertaken to calculate the precision and reproducibility of each method.

## RESULTS AND DISCUSSION

### Copper-Oxime Systems

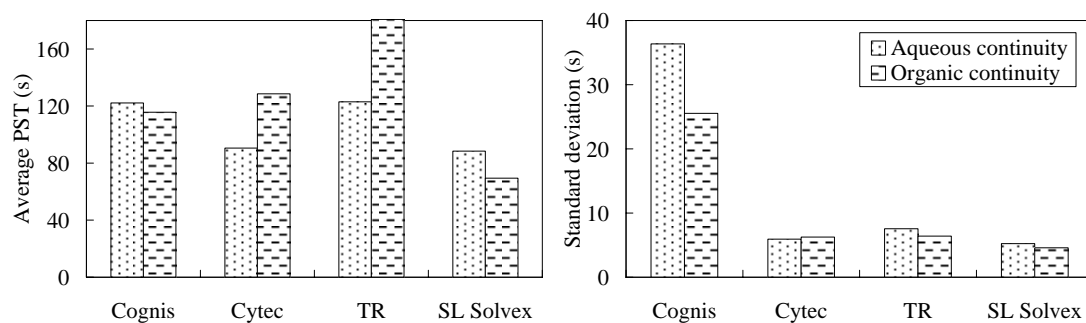
Figure 2 shows the average PST results and variances (square of the standard deviation) of (a) six replicates on fresh phases and (b) ten replicates on recycled phases measured for copper-oxime systems following aqueous-continuous mixing. Significant differences in PST were measured for the same systems using the different techniques, confirming the inherent problems with accurate measurement of this parameter. For copper-oxime systems, a PST of ~60 s would typically be expected. PST is expected to increase with decreasing aromaticity of the diluent, *i.e.*, Shellsol A150 (fully aromatic) < Shellsol 2325 (~ 20% aromatic) < SSX 210 (aliphatic). This was not observed in the system studied. There was also little difference in PST between LIX 984N (ketoxime-aldoxime blend) and ACORGA M5640 (ester-modified aldoxime). Based on the PST values and calculated variances, it is evident that the Smart-Level Solvex and Cognis methods gave best reproducibility for PST measurement in these systems.



**Figure 2.** Average PST and calculated variances on the copper-oxime systems for (a) fresh phases and (b) recycled phases following aqueous-continuous mixing.

### Zinc-D2EHPA System

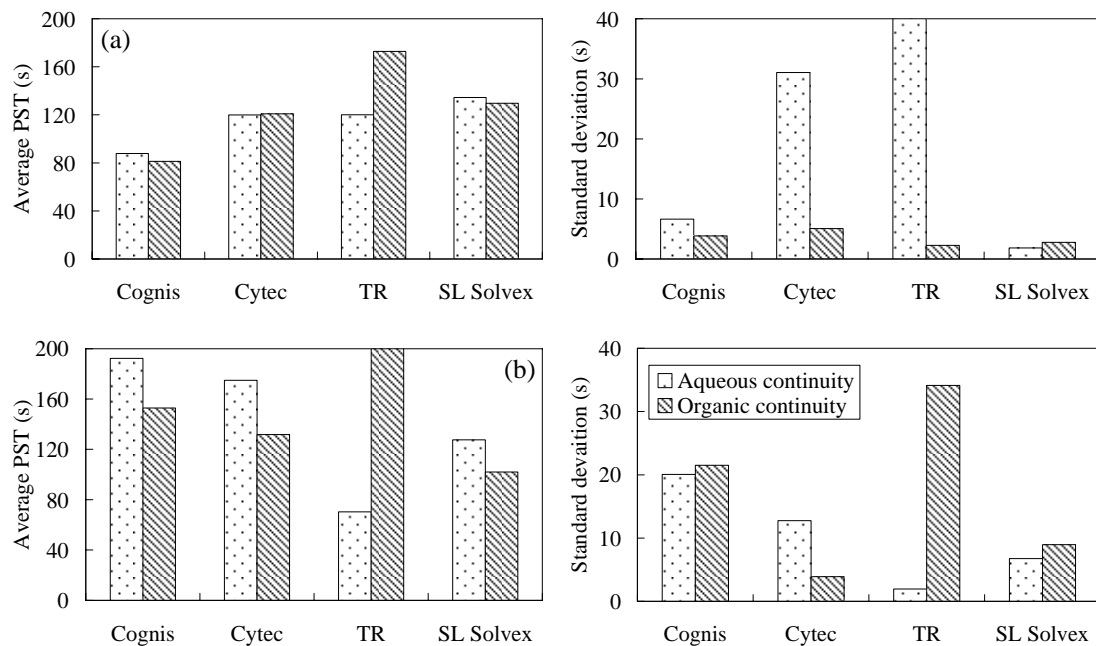
The TR method was specifically established for the Zn-D2EHPA system, while the Cognis and Cytec methods are generally used for copper systems. Figure 3 shows PSTs and standard deviations measured for recycled phases in the Zn-D2EHPA (10 replicates) system following aqueous- and organic-continuous mixing for all four methodologies. As with the copper system, there is lack of agreement between the three methods with respect to PSTs measured. Reproducibility was slightly better using the Smart-Level Solvex compared to the Cytec and TR methods, while the Cognis method was poor for this application.



**Figure 3.** Effect of measurement technique on average PST and standard deviation for the Zn-D2EHPA recycled system following aqueous and organic-continuous mixing.

## Uranium-Amine System

Figure 4 shows results obtained under aqueous-and organic-continuous mixing conditions for the uranium system. The Smart-Level Solvex demonstrated the best reproducibility of PST of the four methods and the closest correlation between results for the fresh and recycled phases. The TR method showed the opposite trend for phase continuity compared to the other three methods and poorest reproducibility. This may be attributed to the difficulties of adequately cleaning the plastic reaction vessel, and possible interfacial activity of the organic phase with the reactor walls.

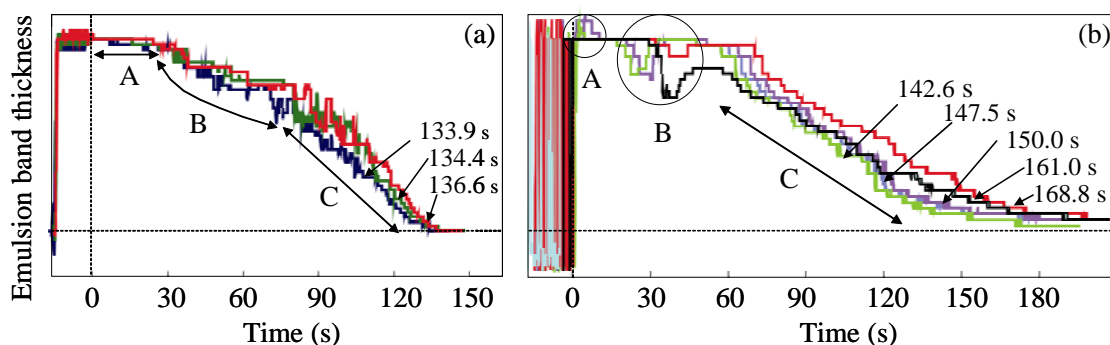


**Figure 4.** Average PST and calculated standard deviations for PST measurements on the uranium-amine system for (a) fresh phases (6 repeats) and (b) recycled phases (10 repeats).

## Phase Separation Characteristics

One advantage of the electronic detection technique is that more detailed information regarding the phase separation process can be obtained. In Figure 5(a) (aqueous-continuous phase separation in uranium system), three distinct processes can be identified: an initial phase break (A), primary separation (B), and secondary separation (C). The profiles under organic-continuous conditions are different (Figure 5(b)), showing an initial increase in the emulsion band thickness (A) and “bounce” (B), before the main separation (C) occurs. Speculation on the reasons for these reproducible effects is beyond the scope of this paper.

The influence of the presence of a phase modifier, crud, or alternative diluents on the rate of phase disengagement can be similarly evaluated and better understood.



**Figure 5.** Overlaid replicate profiles of phase separation in the uranium-amine system following (a) aqueous- and (b) organic-continuous mixing conditions, showing comparison of final phase separation times and different processes detected during phase separation.

## CONCLUSIONS

A general lack of conformity in the measurement of phase separation exists. The methods in most widespread use rely heavily on operator experience and objectivity. Even for identical systems, considerable variation in results is measured using different techniques. The introduction of the Smart-Level Solvex, an instrument with electronic detection of phase disengagement and tight control of environmental and operating variables, allows better reproducibility of measurement to be achieved. Furthermore, information on the phase disengagement profile with time, and the behaviour of emulsions, third phases, crud, and other interfacially active phenomena can be obtained.

## ACKNOWLEDGEMENTS

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