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POSITION PAPER

Liquid–Liquid Extraction: Standard Procedures and International Collaboration

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INTRODUCTION

Because of the importance of liquid–liquid solvent extraction in the nuclear industry, the International Atomic Energy Agency (IAEA) convened a group of experts on the occasion of the International Solvent Extraction Conference (ISEC), Liège, to discuss problems of establishing standard procedures and methods for obtaining solvent extraction distribution data and international collaboration in the field of liquid extraction. An outline of procedural conditions for obtaining and reporting solvent extraction distribution data was established based on the collected views of several practitioners in the field. This is presented herein, as well as a review of international collaboration in the areas of information exchange, data assessments, and

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standard test systems. An earlier IAEA meeting reviewed the status of solvent extraction in the field of nuclear science and current research with new solvent extractants (*1*).

STANDARD PROCEDURES FOR DISTRIBUTION DATA

The distribution ratio is defined as the ratio of the total analytical concentration of a solute in the organic phase (regardless of chemical form) to its total analytical concentration in the aqueous phase (*2*). It is recommended that distribution coefficient be reported rather than percent extraction; if percent extraction is reported, phase–volume ratio must also be reported. Because there are many variables which affect distribution ratio measurements, standard procedures are needed for these determinations. The following outlines suggest reporting aspects for such measurements.

Materials

Provide information on the source, grade, purity, and analysis of all starting reagents. Give details of the purification and pretreatment procedure of both phases including composition, results of analysis (especially impurities in extractant which alter the distribution ratio and any other extractable species in aqueous phase), and storage time and conditions.

Equipment

Describe the mixing and phase disengagment apparatus, including treatment of apparatus to control surface adsorption problems. Provide information of temperature controls (and limits) and time for equilibration and phase disengagement.

Procedure

State phase–volume ratio, physical method of phase equilibration, whether phases were preequilibrated or not, and number of replicate measurements. (Preequilibration, equal phase rations, and triplicate measurements recommended.) State how equilibration and disengagement times were established. State sampling procedure.

Analytical Methods

State number and type of analyses and accuracy and precision of measurements. Recommend analysis of feed prior to equilibration and of both phases after equilibration to check material balance.

Reporting Procedure

Provide numerical data with estimated error limits in all cases, even when graphical presentation is given. State material balance.

It is often very important to validate distribution data with successive extractions and/or back extractions.

INTERNATIONAL INFORMATION EXCHANGE

It is desirable to have continued international exchange of information even prior to publication in order to:

(a) Avoid or minimize wasteful duplication of effort
(b) Provide better communication of research activities and hence stimulate development of the subject
(c) Effect a more orderly development of the subject by eliminating bottlenecks

However, collaboration is only likely to be forthcoming in areas remote from commercial sensitivity, which more or less excludes such topics as process development.

Current Position

One of the most effective avenues of communication in the field at the present time is the system of international conference, the ISEC conferences. These have each been sponsored independently by different organizing bodies. Up to 1974, there was no coordination whatever, but since then a degree of international coordination for these conferences has been provided by the International Committee for Solvent Extraction Chemistry and Technology. The latter is an essentially *ad hoc* body founded at ISEC '74. There are 15 members drawn from different countries and chosen so as to give a balance of interest and expertise over both the chemical and chemical engineering aspects of the subject, and applications in both metals and nonmetals areas. The group includes both industrialists and members of academic institutions. It must be emphasized that this group has no real power and only opeates by advice and recommendation.

The European Federation of Chemical Engineering (EFCE) Working Party on Distillation, Absorption and Extraction which, as the name implies, includes liquid extraction within its terms of reference. Its interests are primarily in chemical engineering aspects. Some years ago it sponsored a project on the recommendation of a limited range of standard test systems for liquid extraction equipment (*3*). This was a useful exercise, and there are

plans to produce a more up-to-date version with an extra system. The Working Party has also had some involvement in the recommendation of nomenclature of chemical engineering terms for separation processes.

IUPAC has an active committee concerned with nomenclature covering much of interest to the application of solvent extraction to metals and a commission on equilibrium data.

Finally, reference should be made to the fact that the Society of Chemical Industry (notionally an international learned society but based in London and strongly oriented to the United Kingdom) has a specialist subject group in Solvent Extraction and Ion Exchange. This Society acted as sponsor for both ISEC '71 and ISEC '74. The subject group continues to be very active and has an international outlook. Approximately 25% of group members are from outside the United Kingdom, and there is an international attendance at most of its meetings.

It will be noted that there is no real coordination between these various bodies except that provided by individuals who happen to be in membership of more than one. Another point of comment might be the apparent lack of any focal point for liquid extraction in North America. A meeting of United States workers interested in forming a liquid extraction group was held during ISEC '80 but no positive action was taken and the outcome is not known. There is a group in Canada (based on the C.I.M.) concerned with the applications of liquid extraction in hydrometallurgy.

Equilibrium Data

A great deal of equilibrium data already exists in the literature, although covering a relatively narrow range of systems, many of them of rather academic interest. Attempts are being made to establish comprehensive data banks. Thus the Warren Spring Laboratory in the United Kingdom has established a data bank of data on metals extraction systems, while Prof. Sørensen and colleagues at the Technical University of Denmark have established a similar facility for nonmetals systems. It is not known how critical the data banks are in respect to the data they contain. It would obviously be desirable to have some informed selectivity about the data included.

An attempt is being made at Oak Ridge National Laboratory, U.S.A., to establish a complete data bank on solvent extraction research (*4*). Again, one of the difficulties must be the selection of the information to be included.

System Performance and Characterization

To a degree, standardized equilibrium data generation will inevitably be applied only to pure systems. There is a real need for agreed methods of

testing the performance of industrial systems for questions such as extraction kinetics, the stability of the reagents under specified conditions (degradation testing), and possibly toxicity. Individual user companies, particularly in the metallurgical field, have developed their own acceptance tests for reagents but it would be very valuable to have some recommended methods available which all workers could be expected to use, thus giving a method of reporting such characteristics in their results.

The above concerns tests which would primarily be sensitive to chemical factors. Another area, system characterization, includes factors of importance in determining the chemical engineering aspects of plant design, e.g., the ease of phase interdispersion and disengagement. The need for standardized methods of characterizing systems was stressed during several sessions at ISEC '80 and a public call was made for development of an agreed method, the results of which could then be published as part of any paper describing the performance of solvent extraction equipment.

Comparison of the performance of liquid–liquid extraction contactors is complicated by the fact that many workers have used different systems, making it difficult to distinguish between performance effects caused by the different characteristics of the equipment and those caused by differences in the chemical systems employed. The EFCE initiative described above was an attempt to overcome this. Limitations have been identified in some of the systems recommended as a result of that study and discussions are now in progress on possible modifications. The Society of Chemical Industry sponsored a meeting in London on December 1, 1980, for a discussion among interested parties.

Future Positions

Quite a strong case can be made for the establishment of some form of international solvent extraction society which would, among other possible activities, organize the ISEC events. This would have the big attraction of a better carry-forward of experience. On the other hand, the establishment of such an international society is no easy matter. It demands people with time and resources. The present system is operating, even though it does have some recognized deficiencies, and a better approach in the short-term at least might be a strengthening of the present International Committee for Solvent Extraction Chemistry and Technology.

In view of the historic importance of the nuclear industry in the development of the science of liquid–liquid extraction, it is very appropriate for the IAEA to take an interest in fostering international collaboration in this field. There is certainly a need for some focal point. As is so often the case, the main limitation at the moment comprises people and finance. One possibility might be for the Agency to provide finance for a research team to work on

one of the above projects, e.g., to establish methods of testing system performance. However, we think the greater immediate need would be the sponsorship of a working discussion meeting to bring together workers from several parts of the world to discuss and define the current needs and subsequently to monitor any work which is carried out. Collaboration with EFCE or IUPAC would be desirable. However, in any initiative we would strongly press the desirability of involving both chemists and chemical engineers. Liquid–liquid extraction is very much of an interdisciplinary subject demanding an interaction between chemists and chemical engineers. Any initiative which leaves room for a polarization between the two groups is not likely to achieve maximum benefit.

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Note added in proof:

The Division of Industrial and Engineering Chemistry of the American Chemical Society has formed a committee on Separation Science and Technology to serve as a focal point for the diverse field of separations (including solvent extraction) within ACS.