

SAMPLE PREPARATION IN LIQUID SCINTILLATION  
REMARKS OF THE SESSION CHAIRMAN

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From the early days of liquid scintillation, sample preparation has been the primary nemesis of this elegant analytical tool. One is faced with the reciprocal effects of augmenting sample volume to increase the total radioactivity in the vial (hence, to improve sensitivity and precision) while at the same time the quenching effect of the larger sample size reduces counting efficiency. Thus, workers have adopted sample figure of merit as a means of optimizing sample preparation conditions.

Sample Figure of Merit = Sample Size x Counting Efficiency

One attempts to balance the sample system to achieve the maximum counting rate in the vial. Sample figure of merit is not to be confused with instrument figure of merit (efficiency<sup>2</sup>/background) which is used to monitor instrument performance.

From a historical perspective it is interesting to compare aqueous tritium counting data from early workers with data obtained by state of the art methodology. These data are presented in Table 1. Due to limitations of incorporating aqueous samples into primary organic solvents early workers utilized 60-85ml counting bottles to achieve reasonable counting rates and the first commercial counters were designed to accommodate this configuration. By the late 1950's improvements in instrument design, which simultaneously reduced background and improved counting efficiency, and novel cocktail formulations allowed the use of 20ml counting containers which became standard.

The use of tri-functional surfactants of the polyethoxy-alkylphenol series developed in the late 1960's is primarily responsible for the considerable progress which has been made toward increasing aqueous sample volumes while minimizing the effects of quenching. Our suggestion several years ago that

Table 1 A Chronology of Progress in Liquid Scintillation Sample Preparation

Method	Tritium Counting Efficiency	Sample Volume	Figure of Merit	Secondary Solvent
Chen 1958 <sup>1</sup>	3.7%	1.5%	6	Ethanol
Davidson 1958 <sup>2</sup>	6.0%	20.0%	120	Polyether
Bray 1960 <sup>3</sup>	10.0%	22.0%	220	Dioxane
Patterson 1965 <sup>4</sup>	10.0%	23.0%	230	Triton X-100
Turner 1969 <sup>5</sup>	22.0%	21.0%	462	Triton X-100
Commercial 1972 <sup>6</sup>	27.0%	40.0%	1080	Unknown
Commercial 1975 <sup>7</sup>	30.0%	50.0%	1500	Unknown

<sup>1</sup>Proc. Exp. Bio. Med., 92, 546 (1958).

<sup>2</sup>See reference 1, p.88

<sup>3</sup>Anal. Biochem., 1, 279 (1960).

<sup>4</sup>Anal. Chem. 37, 854 (1965).

<sup>5</sup>Int. J. Appl. Radiat. Isotopes, 20, 499 (1969).

<sup>6</sup>PCS<sup>TM</sup>, Amersham Searle.

<sup>7</sup>Merit<sup>TM</sup>, Isolab.

## SAMPLE PREPARATION

improvement in sample preparation techniques was rendering large volume liquid scintillation counters obsolete has recently been proven with the introduction of the first compact, table-top, mini-vial counter by a major instrument manufacturer.

### LIQUID SCINTILLATION LITERATURE

Since sample preparation is one of the most troublesome areas of liquid scintillation counting, sundry recipes and remedies have been described for solving particular problems. In that regard it is helpful to have at one's fingertips a bibliography of liquid scintillation literature. Since no previous symposium has presented a comprehensive bibliography, one is provided here with commentary.

### SYMPOSIUM PROCEEDINGS

The first liquid scintillation symposium was held in August of 1957 at Northwestern University. The proceedings entitled Liquid Scintillation Counting were published by Pergamon Press (1). It is interesting to review many of the original papers presented at the symposium in an era where instrumentation and sample preparation techniques were very crude. One of our distinguished participants in the workshop session, Dr. Chin-Tzu Peng, presented a paper on Sulphur-35 Liquid Scintillation Counting at that very first symposium.

In 1969 a symposium was held at MIT in Boston and the proceedings were published by Grune and Stratton in a volume entitled The Current Status of Liquid Scintillation Counting (2). We are also very fortunate to have with us in our workshop session, Dr. Edwin D. Bransome, who edited this volume and played a major role in the 1969 symposium.

In 1970 a symposium was held at the University of California in San Francisco and the proceedings entitled Organic Scintillators and Liquid Scintillation Counting were published by Academic Press (3). Dr. Chin-Tzu Peng and Dr. Donald L. Horrocks, who is a plenary lecturer at this symposium, edited the proceedings.

Following the first three symposia in the United States the first of four British symposia organized by the Society for Analytical Chemistry was held in 1970 at the University of Salford. The first United Kingdom symposium was published

by Heyden entitled Liquid Scintillation Counting-Volume I (4). The following year a symposium was held in Brighton, England which resulted in Liquid Scintillation Counting-Volume II (5) and in 1973 a third symposium was held in Brighton which resulted in Liquid Scintillation Counting-Volume III (6). In 1975 a fourth symposium was held in Bath, England and I believe that the proceedings will shortly be published by Heyden as Liquid Scintillation Counting-Volume IV.

In 1973 a symposium was organized in Australia by Dr. B. A. Scoggins and Dr. P.E. Stanley, who is not only presenting a plenary lecture at this symposium, but is also participating in the workshop session on instrumentation. The Sydney Symposium was published by Academic Press in a volume entitled Liquid Scintillation Counting - Recent Developments (7).

And last but by no means least we have our International Symposium in Canada in 1976. The proceedings of this symposium are scheduled to be published later this year by Academic Press.

### TEXTS

In contrast to symposium proceedings, textbooks are presentations by a single author or a single group of authors which attempt to cover theory and applications of scintillation counting. The first of these was published in 1963 by Elsevier and was authored by Dr. E. Schram. This book entitled Organic Scintillation Detectors (8) was an excellent, comprehensive text on scintillation counting. We are fortunate to have Dr. Schram as a member of our scientific advisory committee for this symposium.

The following year Dr. J.B. Birks authored his monumental treatise entitled The Theory and Practice of Scintillation Counting (9). This text is still recognized as one of the foremost authoritative sources on the theory of scintillation counting.

Nearly ten years passed before another comprehensive text on liquid scintillation counting was produced. Evidently four authors simultaneously saw the need for an updated text in the area of liquid scintillation counting and books were published by A. Dyer (An Introduction to Liquid Scintillation Counting - Heyden) (10), D.L. Horrocks (Applications of Liquid Scintillation Counting - Academic Press) (11), Kobayashi and Maudsley (Biological Applications of Liquid

Scintillation Counting - Academic Press) (12), and Neame and Homewood (Liquid Scintillation Counting - Halsted) (13). All of these texts were published in 1974.

### JOURNALS

Scientific papers relating to liquid scintillation counting are apt to appear in almost any analytically oriented journal. Many of the older journals, particularly those relating to instrumentation developments, are no longer being published. Currently articles on liquid scintillation counting appear in the International Journal of Applied Radiation and Isotopes, Clinical Chemistry, Health Physics, Analytical Chemistry, Clinica Chimica Acta, and Analytical Biochemistry.

A number of review articles are particularly worthy of note. The first by Davidson and Feiligson appeared in 1957 and it is an excellent review of the literature to that date (14).

A second review covering the period of time between 1957 and 1964 by Rapkin appeared in the International Journal of Applied Radiation and Isotopes in 1964 (15).

A third review by Parmentier and Ten Haaf in the International Journal of Applied Radiation and Isotopes encompasses the literature from 1964 through 1969 (16).

Perhaps one of the most practical and useful series of articles on liquid scintillation counting appeared in a British publication Laboratory Practice in 1973. The series of at least seven installments by L.W. Price describes clearly and concisely theory and applications of liquid scintillation counting. The series is highly recommended to both novices and experts as a very practical guide to the liquid scintillation technique.

### COMMERCIAL LITERATURE

A final source of information on sample preparation and counting techniques can be found in the literature published by commercial manufacturers. While one must bear in mind that the purpose of this literature is to guide one to the products of the manufacturer, the recipes and the methods described are generally quite good for the particular application. At one time Beckman, Packard, and Searle Analytic had available rather extensive applications manuals

with particular emphasis on sample preparation. One should also not forget that these companies have various experts on liquid scintillation counting on their staffs and most are quite willing to discuss with you on an individual basis particular liquid scintillation problems. On instrumentation one could contact Bart Laney of Searle Analytic and Don Horrocks of Beckman. With regard to sample preparation one could contact Roger Ferris of Amersham Searle, Dr. Y. Kobayashi of New England Nuclear, Mr. Robert A. Chudy of Research Products International and Dr. Murray Vogt of Isolab. These gentlemen have spent countless hours on the telephone with hundreds of scientists to help resolve problems in liquid scintillation.

Regrettably the preceding paragraph may lead one to believe that all of the problems in sample preparation for liquid scintillation counting have been quite well resolved. Unfortunately this is not true, as a number of rather serious problems still plague users of the liquid scintillation technique. The ones which I see as most prominent are standardization, both in terms of radioactivity standards and a standard definition for the term "liquid scintillation grade", the problem of color quenching, the problem of chemiluminescence and the problem of dealing with samples which are difficult to dissolve in scintillation cocktails. In our workshop session here this afternoon the panel of liquid scintillation sample preparation experts will attempt to address these and other questions in their introductory remarks. Following the introductory remarks we will entertain from the floor any and all questions regarding the sample preparation.

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