THE PURITY OF COMMERCIAL LIQUID SCINTILLATION FLUORS AND THE EFFECTS OF IMPURITIES ON PERFORMANCE

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ABSTRACT

As a large user of liquid scintillation chemicals, we have noted considerable variation in the quality of liquid scintillation fluors supplied by commercial manufacturers. We have examined in detail the purity of two common fluors, PPO and Bis-MSB. The limitations of conventional methods of analysis are discussed along with suggestions for analyzing fluors to determine scintillation efficiency and to detect color quenching impurities. Examples of results of several quenched and unquenched cocktails demonstrate the effects of impurities on optimum performance.

INTRODUCTION

In a critical experiment the research chemist should never presume that ordinary laboratory reagents are pure. Likewise in the liquid scintillation laboratory the analyst should ensure that all chemicals meet the purity criteria established for the experiment.

In 1957 Hayes noted that "In any liquid scintillator system, purity of the components is a very important consideration, because of the possible quenching or light absorption due to contaminants" (1). Eisenberg (2) has noted the variability in PPO quality, and Radin (3) repurified a batch of PPO to remove impurities which darkened in solution with Hyamine*. Schram (4) has stressed the importance of purity of components in scintillator systems. Horrocks (5) has described impurities in a commercial supply of Butyl-PBD.

*Trademark Rohm and Haas

Our interest in the purity of commercial fluors stems from our massive use in the laboratory and for manufacture of highly unquenched liquid scintillation standards which are used to determine optimum performance of liquid scintillation spectrometers. In our purchases over the last several months we have received fluors containing bits of wood, a ground-up rubber glove and nearly 5% by weight glass wool. Several batches of fluor when dissolved in toluene gave turbid solutions which could not be clarified.

On one occasion when preparing a concentrated solution of 250 g/l PPO which was supplied by a reputable manufacturer, the resultant solution was so heavily color-quenched it could have been mistaken for urine. After several weeks' delay, the supplier reluctantly checked a sample in his quality control laboratory and discovered to his amazement that the batch contained a very pronounced yellow-colored impurity.

On another occasion we analyzed a batch of Bis-MSB which was found to be grossly impure and heavily color-quenched. As a result of our complaint, the manufacturer recalled the batch and is no longer preparing this fluor. We know of another manufacturer who relies solely on melting point (with a 3°C. range) and visual inspection of dissolved samples for "foreign matter" in quality control of fluors.

It should be emphasized at the outset that the average liquid scintillation analyst would incur, at most, perhaps a 5-10% variation in efficiency due to fluor impurity. However, those of us who study liquid scintillation, who prepare calibration standards, must carefully test not only the fluors, but also the solvents and vials. Based upon our experience with impure commercial fluors, we find it quite astonishing that a recent survey of new liquid scintillators to determine optimum relative pulse heights could be undertaken using chemicals which were neither analyzed nor repurified (6).

MATERIALS AND METHOD

Bis-MSB: A five-liter batch of Triton X-100* scintillant was prepared using freshly distilled toluene: Triton X-100 (2:1 v/v) which contained 6 g/l PPO. To one-liter samples of scintillant was added 1.5 g Bis-MSB

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from each supplier. The samples were stirred simultaneously under identical conditions until all solutes had dissolved. From each batch ten samples were prepared by adding 10.0 ml of each scintillant and 100 µl Toluene-^{3}H (350,000 dpm) internal standard to a counting vial which contained 5.0 ml distilled water. The samples were shaken, allowed to equilibrate four hours at room temperature and placed in the counter for four hours prior to counting. Average results of ten samples from each batch are shown in Table I.

PPO: A six-liter batch of Triton X-100* scintillant was prepared using toluene: Triton X-100 (2:1 v/v). To one-liter samples of the batch was added 2 g PPO. The samples were further prepared as described above.

DISCUSSION

Farmer and Bernstein (6) have previously demonstrated that infrared spectral analysis is an unreliable measure of scintillator performance. Our results indicate that neither fluorescence nor ultraviolet absorption alone are good measures of liquid scintillation fluor performance. In the former case the presence of quenching impurities could give a lower apparent fluorescence yield, even though the ultraviolet absorption spectrum indicates a pure sample. Conversely, the presence of ultraviolet-absorbing non-fluorescent impurities could result in falsely high absorption values. A ratio of absorbance/fluorescence appears to correlate well with counting efficiency. However, the ultimate test is the application for which the materials are to be used as compared to a standard known to be pure.

We observed tremendous variation in appearance and dissolution time of fluor crystals, particularly with Bis-MSB. It required only 30 minutes to dissolve one Bis-MSB sample, but 3 hours for another which, incidentally, was found to be the worst performer. Bis-MSB samples showed a significant 7% difference in performance.

The seven commercial PPO samples varied only 1.8% in tritium counting efficiency, even at 2.0 g/1 where small variations in purity affect efficiency greatest (8); this was not a significant difference in the experiments we performed. Both fluorescence emission and ultraviolet absorption spectra appeared normal, but one sample ex-

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TABLE I: Evaluation of Commercial Bis-MSB

Supplier	Relative Tritium Counting Efficiency*	Relative Fluorescence**	Relative Absorb-ance***	Relative Absorbance/ Fluorescence
B	100	95.7	100	100
A	99.6	95.1	92.8	93.8
C	97.2	100	94.1	90.5
D	96.8	96.8	89.8	89.1
E	93.2	95.1	78.2	79.0

*Nuclear-Chicago Mark II at tritium balance point.
**Exitation 374 mμ; Emission 424 mμ (Perkin Elmer MFP-2A).
***Absorbance 353 mμ (Cary 14).

hibited a rather pronounced yellow-colored impurity which absorbed in the region of 670-770 mμ.

SUMMARY

The term "Scintillation Grade" so often used by suppliers has never been defined for specific chemicals. One supplier defines "Scintillation Grade" nebulously as meeting "rigorous quality standards established after careful consideration of the actual requirements of the individual reagents, and their role in the total system".

We have shown that there can be significant variation in the purity of commercial liquid scintillation fluors and that the impurities affect performance.

Standards of "Scintillation Grade" should be established for industry to follow. I propose that at our next conference we consider standards for the purity of toluene, xylene, p-xylene, dioxane, PPO, Butyl-PBD, POPOP and Bis-MSB.

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