

Chapter 6

Preparation of the Quenched Standards and their Durability

K. Hoizumi

*Japan Atomic Energy Research Institute, Tokai Research Establishment,
Tokai-mura, Naka-gun, Ibaraki-ken, Japan.*

INTRODUCTION

A pure material submitted to radioactive measurement should give almost the same dpm irrespective of the difference of both counting apparatus and standardizing method. Most researchers have usually used relative values of radioactivity in their work rather than absolute values. However, interchangeable data will be necessary for future work relating to compounds labelled with C-14 and H-3, because of an increasing use of the liquid scintillation counter and of the increase of joint research among the various research fields.

Formerly, various sets of quenched standard were compared with each other. There were great differences between the characteristics of the commercial quenched standard sets, which caused problems. The author and N. Morikawa have attempted to solve this problem by the use of a reference source in our joint research.

Recently, the various newly obtained quenched standard sets were compared with each other. The data obtained indicated that the quench characteristics of the sets agreed well. This suggests that the manufacturing process of the quenched standard set is good. Nevertheless, it happens sometimes that the deviation among the radioactivity values which are obtained for the same sample by using the various liquid scintillation counters and their quench calibration curves, rises to over 10%. It is considered that such inconsistency is caused by the use of undesirable quench calibration curves. The internal standard and quenched standards prepared by oneself will be convenient for finding the incorrect curve. In our laboratory, the quenched standards were prepared and successfully used for both the standardization of the counting data and the comparison with the commercial quenched standard set. But the quenched standards will not be able to be utilized for a long period due to the incomplete seal of the sample vial. And furthermore, caution must be exercised concerning changes in the quality of even commercial quenched standards.

In order to find the extent and manner of the change, most simple types of quenched standard were prepared, and the counting efficiency and the external standard channels ratio were observed for 5 years.

PREPARATION OF THE QUENCHED STANDARDS

The basic compositions of the scintillator solutions were as follows:

- (I) toluene, DPO 4 g l⁻¹
- (II) dioxane, DPO 4 g l⁻¹, naphthalene

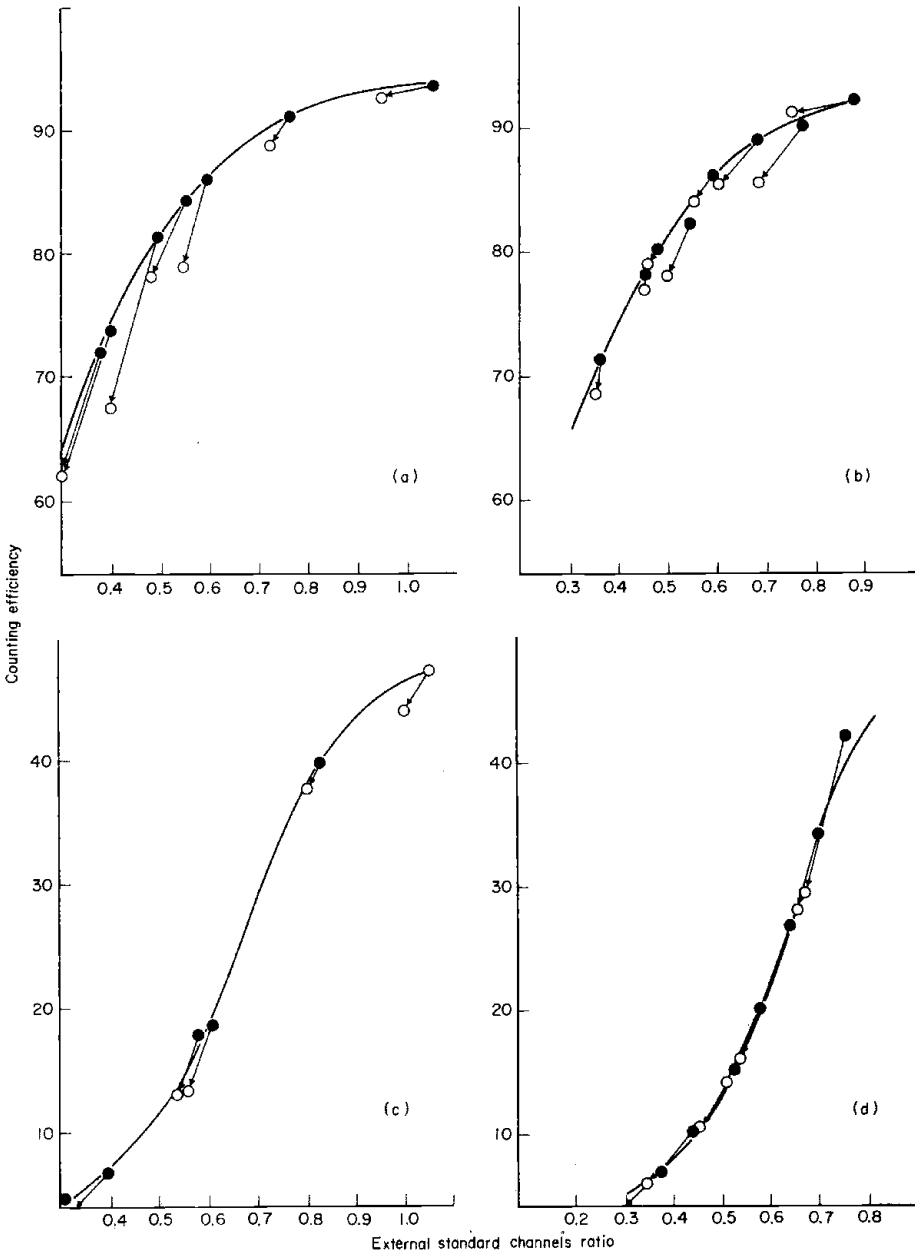


Fig. 1 Plots of the data for the quenched standards showing the relation between the counting efficiency and the external channels ratio. Full lines correspond to the quench calibration curve at 1972. —●—●— determined at 1972, —○—○— determined at 1977. (A) for C-14, toluene scintillator solution; (B) for C-14, dioxane scintillator solution; (C) for H-3, toluene scintillator solution; (D) for H-3, dioxane scintillator solution.

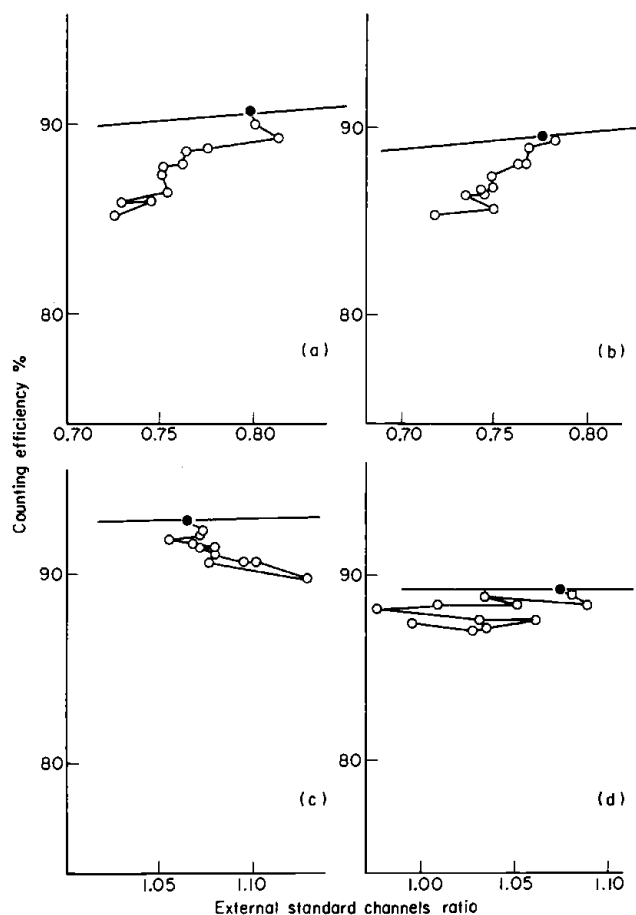


Fig. 2 Plots of the data for some counting samples, which were determined from 1972 to 1977. (A),(B) for C-14, toluene scintillator solution; (C),(D) for C-14, dioxane scintillator solution.

Accurate quantities of triphenylmethane- ^{14}C and ^{-3}H were dissolved in each scintillator solution, and varying amounts of carbon tetrachloride were added to each solution. Many kinds of reference source are commercially obtainable, and these materials are convenient for preparing quenched standards. In the present work, however, C-14- and H-3-labelled triphenylmethane were used for the preparation of the quenched standards since these materials were already in use as the reference sources in joint research work.

These materials were used to prepare quenched standards. A 15 ml portion of the prepared solution was put into a screw-cap glass vial and then the vial was sealed with an adhesive agent without replacement of the air with inert gas.

CHANGES OF THE COUNTING EFFICIENCY AND EXTERNAL STANDARD CHANNELS RATIO

The quenched standards prepared were stored under ambient conditions and their counting efficiency and external channels ratio were checked twice per year.

Both counting efficiency and external standard channels ratio always show some scatter during counting, but the amount of scatter remained within 3% of the average

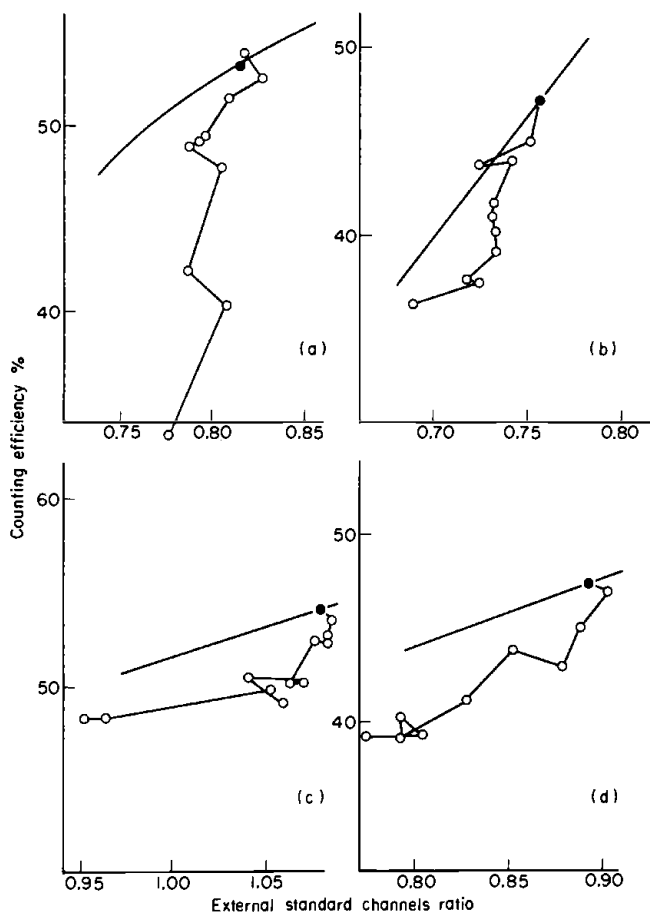


Fig. 3 Plots of the data for some counting samples, which were determined from 1972 to 1977. (A),(B) for H-3, toluene scintillator solution; (C),(D) for H-3, dioxane scintillator solution.

value. During one or two years, no remarkable changes in the calibration curve were observed. However, the plots on the curve were gradually changing from the initial positions.

The data obtained in 1972 and 1977 are shown in Fig. 1 (A,B,C,D). On these figures, the relations between the counting efficiency and the external standard channels ratio were plotted. Any samples of four standard series resulted in a lowering of both counting efficiency and external standard channels ratio. In the data for C-14 (Fig. 1 (A,B)), the drop in counting efficiency is large compared to the increase of the quenched level. The phenomenon appeared to a variable extent for individual counting samples.

In the case of the data for H-3, most of the plots undergo a change on the initial quench calibration curve. As a result, the quench calibration curve obtained at 1977 is almost the same as that of 1972 (Fig. 1 (D)).

The data for some counting samples, which have been determined from 1972 to 1977, are shown in Figs 2-4. The data for C-14 are shown in Figs 2 and 4. The data for H-3 are shown in Figs 3 and 4. The figures (A), (B), (C) and (D) of Figs 2-4 correspond to the data of each counting sample.

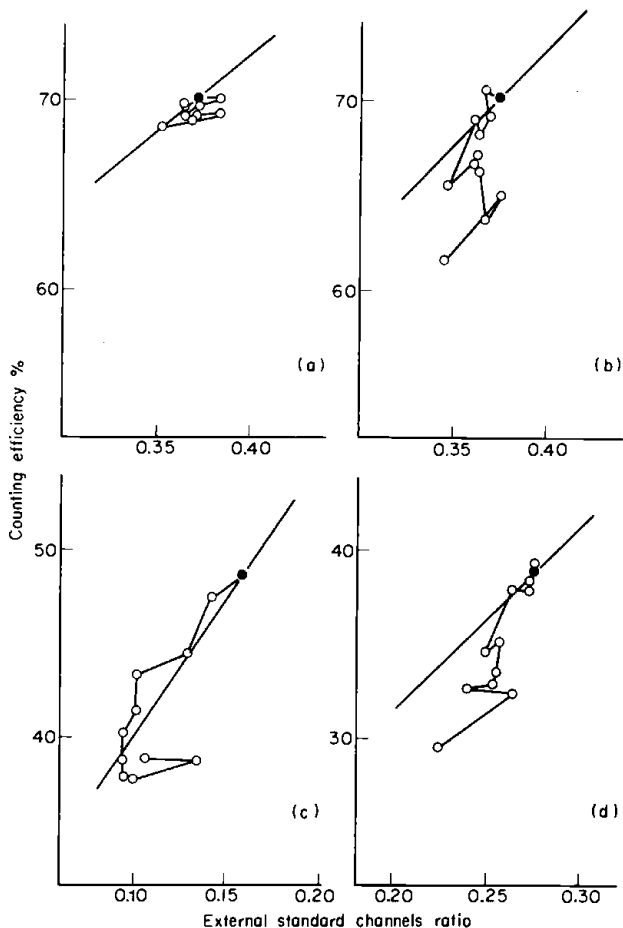


Fig. 4 Plots of the data for some counting samples, which were determined from 1972 to 1977. (A) for C-14, toluene scintillator solution; (B) for C-14, dioxane scintillator solution; (C) for H-3, toluene scintillator solution; (D) for H-3, dioxane scintillator solution.

Most of these examples show a similar tendency. The changes in values during two years were rather small and have gradually increased over 5 years. These plots on the figure shift to the direction which corresponds to the decrease of both counting efficiencies. All of the change modes may be conveniently divided into the following three:

1. They oscillate around the starting positions.
2. They move along the initial calibration curves.
3. They deviate from the initial calibration curves.

A significant change in color and solution volume were observed for some examples. However, the changes in their external appearance did not correlate with changes in observed values. Although some quality changes may happen in the quenched sample and the non-quenched sample after the preparation, cases 1 and 2 will not affect the shape of the quench calibration curve. Case 3 has an effect on the shape of the calibration curve and in its turn influences the accuracy of the liquid scintillation counting, but this tendency was not observed until the second year.

The counting efficiency drop in case 3 is an incomprehensible problem in the present work. It will probably be attributed to reasons other than quenching. The quality change, which increased in the scintillator solution with the elapse of time, may not be a simple mechanism. It is conceivable that some possible cause for this change was already latent in that solution at the time of the preparation. So the preparative conditions are very important for the quenched standards used for long periods. The relation between the individual conditions of this preparation and the changes of the quench characteristics on standing were not elucidated in this work. In such work, much time is needed to clarify the relation between cause and effect.

Prior to this work, the photostability tests were undertaken for the quenched standards by means of UV-irradiation. According to the results, it was found that deterioration was enhanced by the presence of a small amount of carbon tetrachloride. A major effect of the UV-radiation on the scintillation characteristics is the increase of the chemical quenching. The UV-radiation will have a strong effect on all components of the quenched standards. Since the effect was similar to that on standing, UV-radiation can be successfully used for studying the quality change of the quenched sample on standing. If radiation from the source materials has effects similar to UV-radiation, though initially insignificant, on the deterioration of the quenched standard, the effects accumulated for a long period may be significant.

The quenched standards, which were prepared by the use of non-volatile sources, could be used for two years. Since many undesirable effects on the scintillation mechanism will be increased with increases of the period of use, care is required in the use of standards over long periods of time.

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