

**2003 LS User's Forum**

**Liquid Scintillation at NPL:  
Practical Applications**

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# Introduction

- ◆ Overview of applications
- ◆ Current projects
- ◆ Neutron-activated thioacetamide discs
- ◆ Impurity assay for  $^{32}\text{P}$  comparison

Primary Standards & Development	Customer & Secondary Standards	Health Physics
<ul style="list-style-type: none"> <li>• TDCR</li> <li>• 4πβ-γ LS</li> <li>• Adsorption tests</li> </ul>	<ul style="list-style-type: none"> <li>• <sup>3</sup>H assay</li> <li>• Alpha assay</li> <li>• Pure beta</li> <li>• Dilution checks</li> </ul>	<ul style="list-style-type: none"> <li>• <sup>3</sup>H screening</li> <li>• Swabs</li> <li>• Waste assay</li> </ul>
<ul style="list-style-type: none"> <li>• Radiochemistry</li> <li>• CIEMAT / NIST</li> </ul>		

# Primary Standards & Development

Funded by DTI:

- ◆ Assay of  $^{32}\text{P}$ ,  $^{90}\text{Y}$ ,  $^{192}\text{Ir}$ ,  $^{204}\text{Tl}$  for international comparisons
- ◆ Enhancement of TDCR system
- ◆ Extension of CIEMAT/NIST technique
- ◆ Organically Bound Tritium (OBT)

# Primary Standards & Development

Other maintenance/development work:

- ◆ HSE  $^3\text{H}$  in water audit
- ◆ NPL environmental level comparison 2002 & 2003
- ◆ Development of new environmental level standards
- ◆ Radon in water rig

# Secondary Standards & Customer

Many projects/services involving LSC, for example:

- ◆ Environmental level radioactivity standards
- ◆ Non-gamma emitting radioactivity standards
- ◆  $^{32}\text{P}$  in neutron-activated Thioacetamide discs

# Impurities in $^{32}\text{P}$ by Liquid Scintillation

In 2002, NPL participated in the International Comparison of  $^{32}\text{P}$  activity organised under *Bureau International des Poids et Mesures* (BIPM)

$^{32}\text{P}$  is used to bind to molecules such as ATP (involved in cellular respiration) for MR spectroscopy

However...



# Impurities in $^{32}\text{P}$ by Liquid Scintillation

... $^{32}\text{P}$  is produced by neutron irradiation of Sulphur

... $^{33}\text{P}$  &  $^{35}\text{S}$  also produced in trace amounts

...the half life of  $^{32}\text{P}$  is shorter than the impurities – if the solution is old, the contaminants become increasingly significant



# Impurities in $^{32}\text{P}$ by Liquid Scintillation

To determine activity of  $^{32}\text{P}$  accurately, the ratio of the contaminants must first be quantified

- ◆ A series of 63 measurements taken on two LS vials containing samples of the solution were assayed over a 2-3 month period
- ◆ Knowledge of the half lives enables the impurity to be determined by a linear least squares technique

# Impurities in $^{32}\text{P}$ by Liquid Scintillation

- ◆ The count rate in the samples is given by:

$$x_1 = e^{-\lambda_1 t}; \quad x_2 = e^{-\lambda_2 t}; \quad x_3 = e^{-\lambda_3 t}$$

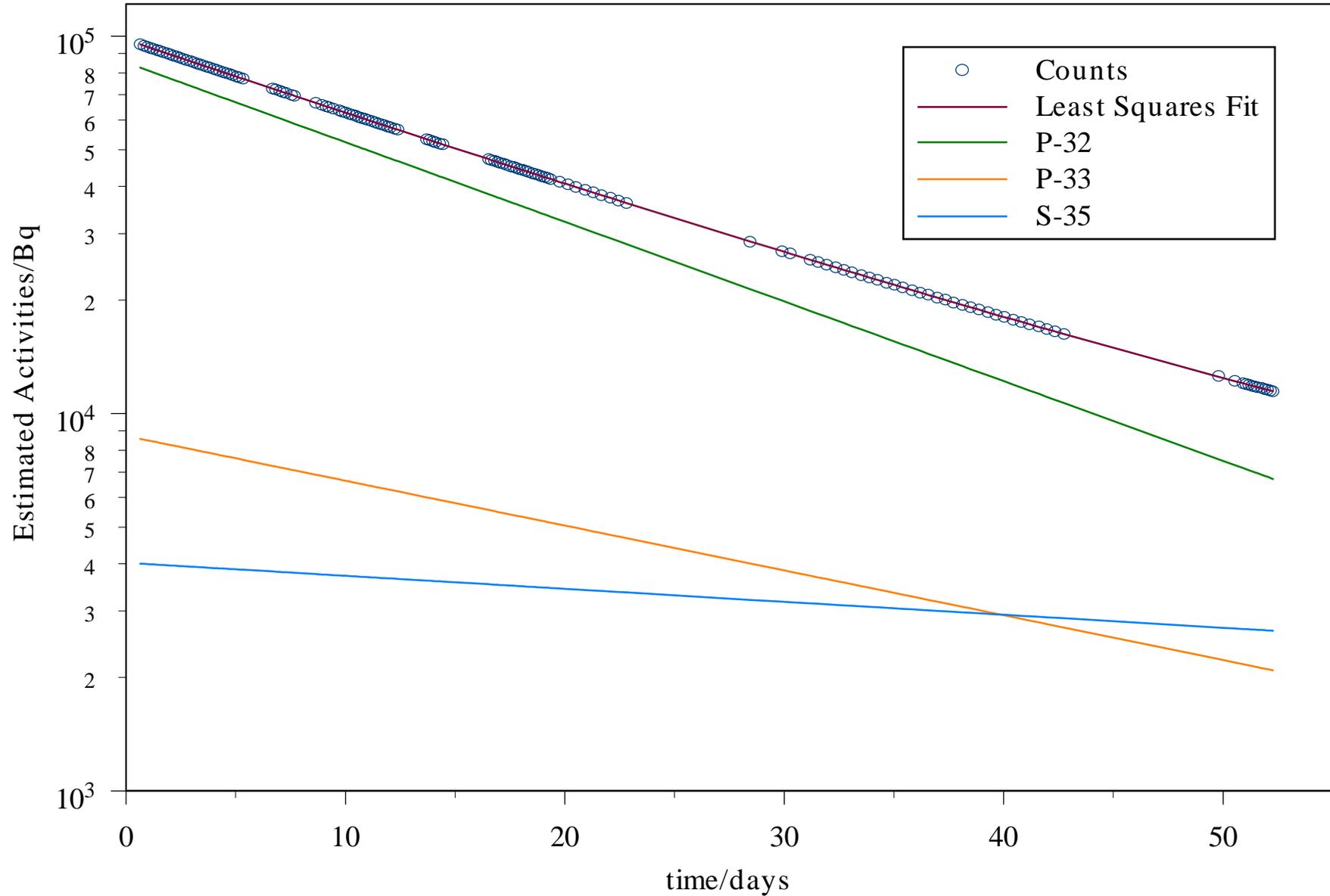
$$N(x_1, x_2, x_3) = A_1 \varepsilon_1 x_1 + A_2 \varepsilon_2 x_2 + A_3 \varepsilon_3 x_3$$

- ◆ The “best fit” is found by minimising:

$$\chi^2 = \sum_i \left[ \frac{N_i - N(x_{1,i}, x_{2,i}, x_{3,i})}{\sigma_i} \right]^2$$

# Estimate of Activities in $^{32}\text{P}$

Fitted values for  $^{32}\text{P}$ ,  $^{33}\text{P}$ ,  $^{35}\text{S}$



# Impurities in $^{32}\text{P}$ by Liquid Scintillation

Some comments:

- ◆ By comparing  $\chi^2$  for models with and without  $^{35}\text{S}$ , can determine if  $^{35}\text{S}$  is really present (we're pretty sure it is)
- ◆ Samples have to be stable over the measurement period. We're dealing with 0.1 g aqueous solution in 10 ml (get the scintillant), and the results seem OK
- ◆ The model fits the data very well and the type A uncertainties are low at around 0.1 %

# Neutron-Activated Thioacetamide Discs

Neutron accident dosimeters (neutron belts) contain sulphur discs which become activated on exposure to large neutron fluxes

These discs are assayed via a calibrated GM counter to determine activity and hence received dose

NPL provides a service to a particular customer of calibrating the  $^{32}\text{P}$  content of the sulphur discs

# Neutron-Activated Thioacetamide Discs

**Problem 1:** Getting large sulphur samples into a scintillation cocktail can be hazardous for your Radiochemist's health

**Solution:** Thioacetamide and Sulphur tablets are irradiated in the same neutron beam. A relative measurement is made of all tablets using a proportional counter and the Thioacetamide tablets are assayed (destructively) to determine the activity by LSC

# Neutron-Activated Thioacetamide Discs

**Problem 2:** How to determine the efficiency of the liquid scintillation counter for the  $^{32}\text{P}$  (and the contaminants) at high quench

**Solution:** The  $^{32}\text{P}$  is usually absolutely standardised and relative measurements made on the LSC; this time however the activity of the  $^{32}\text{P}$  in the cocktail was assayed directly by the CIEMAT/NIST technique using  $^3\text{H}$  as a tracer and thioacetamide as the quenching agent

# Neutron-Activated Thioacetamide Discs

sample crushed under a fume hood



0.1 g Thioacetamide dispensed to each vial



2 g Ethanol dispensed & allowed to dissolve (~2 hours)



10 ml Ecoscint A dispensed to each vial

# Neutron-Activated Thioacetamide Discs

10 ml Ecoscint A dispensed to each of 10 LS vials



0.1 g  $^3\text{H}$  standard added to each vial at 10 kBq/g

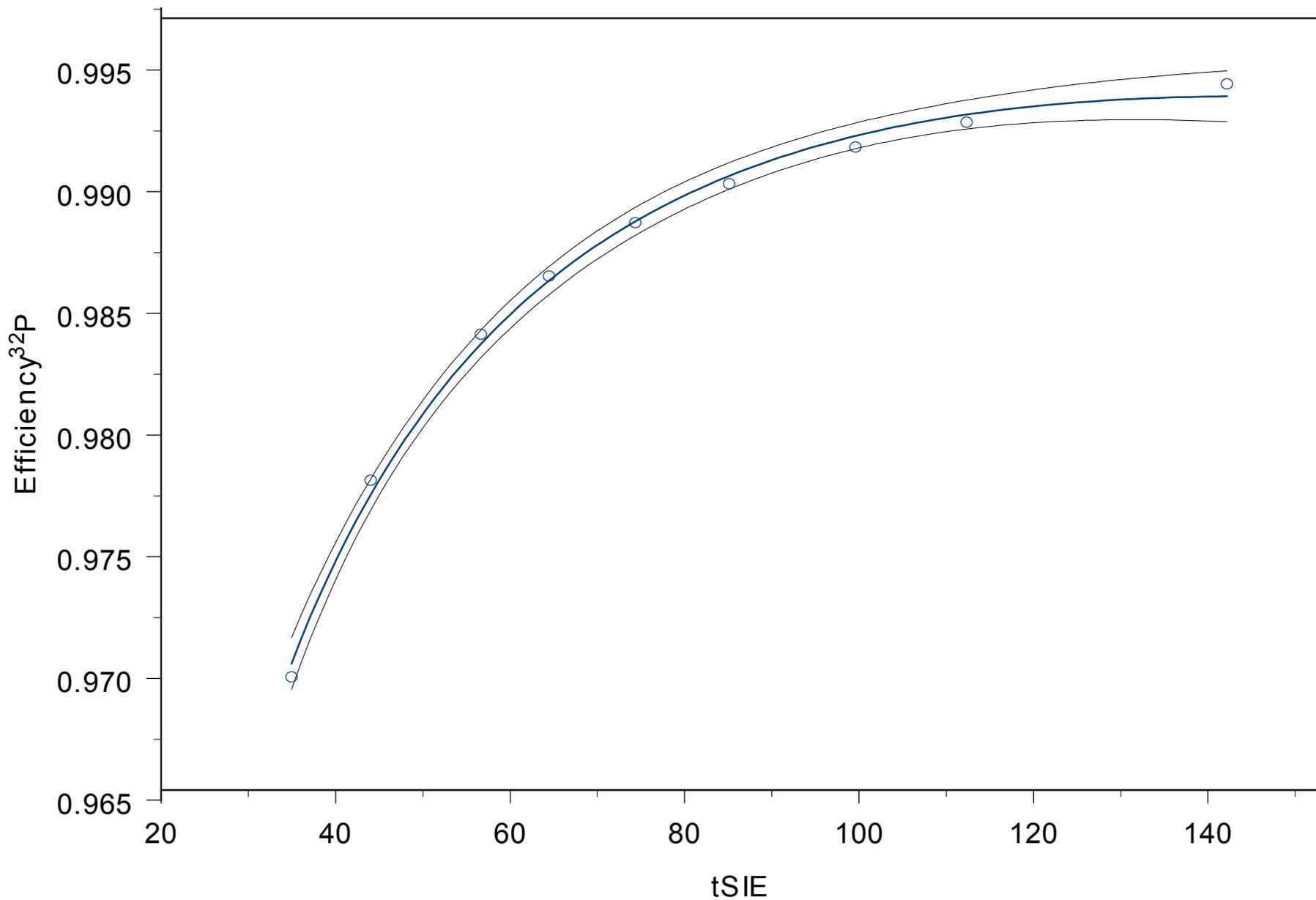


0-3 g 10 % Thioacetamide in Ethanol dispensed to each vial



Vials thoroughly whirlmixed before use!

# Quench Curve $^{32}\text{P}$ Thioacetamide in Ethanol



# Neutron-Activated Thioacetamide Discs

**Results still in preparation** but appears to work as well as the previous technique of using a  $^{32}\text{P}$  standard directly to determine the efficiency

**Has some benefits** particularly as  $^3\text{H}$  is not usually contaminated with secondary nuclides

**Uncertainties** on the activity concentration determined by LSC are around 4 % - not bad considering extremely high quench