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Aspects of Low Temperature Irradiation in Neutron Activation Analysis

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ASPECTS OF LOW TEMPERATURE IRRADIATION IN NEUTRON ACTIVATION ANALYSIS

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SUMMARY

Neutron irradiation of the sample while frozen in a cooling device inserted in a reactor channel has been carried out in the analysis of iodine in aqueous samples as well as of mercury in biological tissue and water.

For the simultaneous irradiation of a large number of aqueous solutions the samples were arranged in a suitable geometry in order to avoid mutual flux perturbation effects.

The influence of the neutron temperature on the activation process has been discussed.

Potential applications of the low temperature irradiation technique are outlined.

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INTRODUCTION

Neutron, photon or charged particle activation of aqueous samples or biological tissues at low temperature offers various advantages in the analysis. Thus, elemental losses due to evaporization effects as well as contamination of the sample from the container surfaces can be eliminated [1]. This technique has been favourably applied in a study of the normal distribution of mercury in human whole blood [2] and in the determination of various elements in water [3, 4].

The present study concerns the determination of iodine in pharmaceutical solutions in the micro-to milligram range, and the analysis of mercury in fish and rainwater at the nano- to microgram level. The low temperature irradiation technique is in this case adequate for an accurate analysis inasmuch as iodine is easily evaporizable, especially from acid solutions. Iodine is also adsorbed to plastic containers. Evaporization effects of mercury are strongly pronounced for water solutions and seem to be dependent on the water quality.

Often, in analyses of this kind, a great number of determinations are carried out simultaneously, which necessitates the irradiation of a large number of samples in one operation. As regards neutron activation of several aqueous - or tissue-samples close together, mutual flux perturbation effects may occur, giving rise to heterogenous activation. Such disturbances can, however, be corrected for [4]. Similarly a large aqueous - or tissue-sample gives rise to flux disturbances [4].

Using a special geometrical arrangement during the multiple sample irradiation process, as here described, the mutual flux perturbation effect can be eliminated.

THE EFFECT OF NEUTRON TEMPERATURE ON THE ACTIVATION PROCESS

Neutron activation processes are generally carried out at neutron temperatures around 50 ^{o}C, whereas in the present study the activation process has been achieved at a temperature about 100 ^{o}C lower.

It is well known that the neutron flux as well as the absorption cross-section varies with the neutron temperature.

For a 1/v absorber (e.g. J^{127}, Hg^{202}) the absorption cross-section temperature dependence is given by the following relation

$$\sigma_a = \sigma_o \left(\frac{T_o}{T}\right)^{1/2} \tag{1}$$

where σ_a denotes the absorption cross-section at temperature T, and, σ_o the absorption cross-section at the temperature T_o corresponding to a neutron velocity of 2200 m/sec.

A temperature decrease of 100 ^oC corresponds to an increase of about 12 per cent in the absorption cross-section value.

The activation rate, R, is proportional to the expression

$$R \sim \int \phi(E)\, \sigma_a(E) dE \tag{2}$$

where $\phi(E)$ expresses the neutron flux per energy unit.

Considering the case of activation of a 1/v absorber, and assuming that the sample does not change the total neutron density, we then have

$$\sigma_a(E) = \sigma_o \frac{v_o}{v} \tag{3}$$

$$\phi(E) = vn(E)$$

$$R \sim \int vn(E)\, \sigma_o \frac{v_o}{v}\, dE = \int n(E)\, \sigma_o v_o dE \tag{4}$$

$$\int n(E)\, dE = n_{tot} \tag{5}$$

$$\underline{R \sim \sigma_o v_o n_{tot}} \tag{6}$$

where v is equal to the neutron velocity (v_o = 2200 m/sec),
n(E) expresses the neutron density per energy unit and
n_{tot} denotes the total neutron density.

Thus the activation rate is proportional to the total neutron density and independent of the neutron temperature.

In practical activation analytical work a flux monitor is sometimes used in order to facilitate the quantitative evaluations. With a monitor possessing a 1/v-dependent activation cross-section, e.g. copper or cobolt, for which the above treatment is valid, the activation rate will be independent of the temperature.

However, in the determination of an element possessing resonances in the thermal region or very close to it, e.g. indium, the activation rate would vary with the neutron temperature. Using a 1/v monitor in such cases instead of a standard, corrections for the temperature effect must be introduced.

The above discussion of the temperature effect also applies to rather complicated irradiation sample arrangements like that used in the present study, assuming the flux perturbation effects to be equal in samples, standard or monitor.

EXPERIMENTAL

The iodine analysis

Iodine in pharmaceutical plasma solutions (containing 0.9 % NaCl) in the concentration range of 50 µg - 50 mg/ml was determined in 0.5 ml samples. A sample arrangement for the simultaneous irradiation of sixty 0.5 ml samples is shown in fig 1. The holder is made of aluminium. The samples were solidified*) in a deep freeze refrigerator before being inserted in the cooling device for irradiation. In one series equal amounts of iodine (0.5 mg) were added to sixty 0.5 ml samples in order to investigate the mutual flux perturbation effect. In the irradiation position used the thermal flux gradient was less than 0.5 per cent per cm.

*) The solidification may also be carried out advantageously by freezing the samples in liquid nitrogen. However, this technique needs special care in order to avoid cracking of the containers.

The analysis was assayed gamma-spectrometrically by means of the nuclide I^{128}, which was measured directly in the solution without the performance of chemical separations.

The mercury analysis

1-100 ml aqueous samples (rainwater etc.) or fish samples (pike) of 1-10 g contained in polyethylene bottles were solidified, like the aqueous iodine samples, in a deep freeze refrigerator before being irradiated in the cooling device.

After the activation process the fish tissue was destroyed by immersion in conc. HNO_3 (about 20 ml/10 g fish) and gentle heating for about 20 min in a flask connected to a reflux condenser. About 1 mg $Hg(NO_3)_2$ was added as carrier. The pH of the solution was then adjusted to the region between 1.5 and 2 by neutralisation with conc. NaOH. The irradiated aqueous samples were also adjusted to the same pH region by acidification with HNO_3. The mercury activities were then separated from the bulk by means of mercury droplets using an isotopic exchange technique previously described by Kim and Silverman [5]. This technique has recently been modified [4]. The exchange time used depended on the volume of the solution [4]. The analysis was assayed gamma-spectrometrically by means of Hg^{203}.

RESULT AND DISCUSSION

Cooling Devices

During these experiments dry ice has been used as cooling medium in a double-walled aluminium cryostat inserted in a central channel of a heavy-water moderated reactor (R1). Operation of this device is simple and fairly inexpensive.

The irradiation time available is, however, limited inasmuch as continuous replenishment of the dry ice material is rather complicated. With the present dry ice cooling arrangement temperatures

of about -40 °C were measured in solidified aqueous samples during the irradiation [3].

If the low temperature irradiation is done with liquid nitrogen, longer irradiation periods can be attained and lower temperatures reached. In this context it may be mentioned that most of the liquid phase in tissues is solidified at or about -20 °C. The animal tissue, however, needs to be cooled to -65 °C [6].

In the liquid nitrogen cooling devices*), nitrogen of extremely pure quality must be used within the radiation field in order to avoid explosions. Such explosions are assumed to originate from the decomposition of ozone formed from oxygen impurities in the nitrogen cooler [7, 8].

Using a liquid helium cooling technique extremely low temperatures can be attained during the irradiation procedure [9-11].

The advantage of carrying out the irradiation of aqueous samples as well as biological tissues while frozen in order to avoid the high pressures arising in the ampoules due to radiolysis, which complicates the opening procedure, has been pointed out previously [1]. In this context it should be mentioned that such pressures can be diminished by cooling the ampoules in liquid nitrogen before opening. This technique has found various applications for samples of biological origin [12, 13].

The iodine and mercury analysis

The geometrical arrangement for low temperature irradiation of sixty 0.5 ml samples simultaneously is shown in fig. 1. With this arrangement no mutual flux perturbation effects were observed. Normalized activities (of I^{128}) in central and peripheral positions in each of 3 layers are given in table 1.

It should be borne in mind that a 30 ml water sample which corresponds to the volume of the sixty 0.5 ml samples gives rise to a flux perturbation effect corresponding to an increase of the ac-

*) Cooling devices for reactor operation are now commercially available.

tivity by about 25 per cent in an irradiation position as used at present [4].

Iodine in the concentration range 25 μg - 25 mg in the pharmaceutical solutions was analysed with standard deviations of the single value less than 5 per cent without the performance of chemical separations.

As regards the mercury analysis, low temperature irradiation combined with the fast isotopic exchange technique revealed an overall yield of (98 ± 2*) per cent (for fish tissue or water).

In a 70 ml rain-water sample a mercury content of 0.33 ng/ml was measured. Correction for the flux perturbation effect was carried out by estimation of the flux advantage factor [4].

Dual analysis was made of the mercury content in 3 pike. The result is given in table 2.

Potential Applications of the Low Temperature Irradiation Technique

The low temperature activation technique seems to be of interest in the analysis of various elements in trace quantities in enzyme samples. Drying of such samples of the order of 0.2 ml often leaves only a thin film on the container surface. The quantitative removal of the induced activities from this surface may be difficult without thorough rinsing of the container surface. Determination of copper at the nanogram level in such samples at the author's laboratory revealed doubt concerning the interpretation of the result inasmuch as about equal copper activities could be extracted from empty quartz containers by rinsing with acids.

Moreover, the irradiation of aqueous samples and wet biological tissues while solidified by freezing finds applications in cases when it is desired that the destruction of the organic molecules in the samples should be minimized. Irradiation under such conditions in a suitable position of a thermal column would strongly suppress the radiation-induced chemical reactions which destroy the organic molecules.

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*) Standard deviation of a single value.

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Table 1

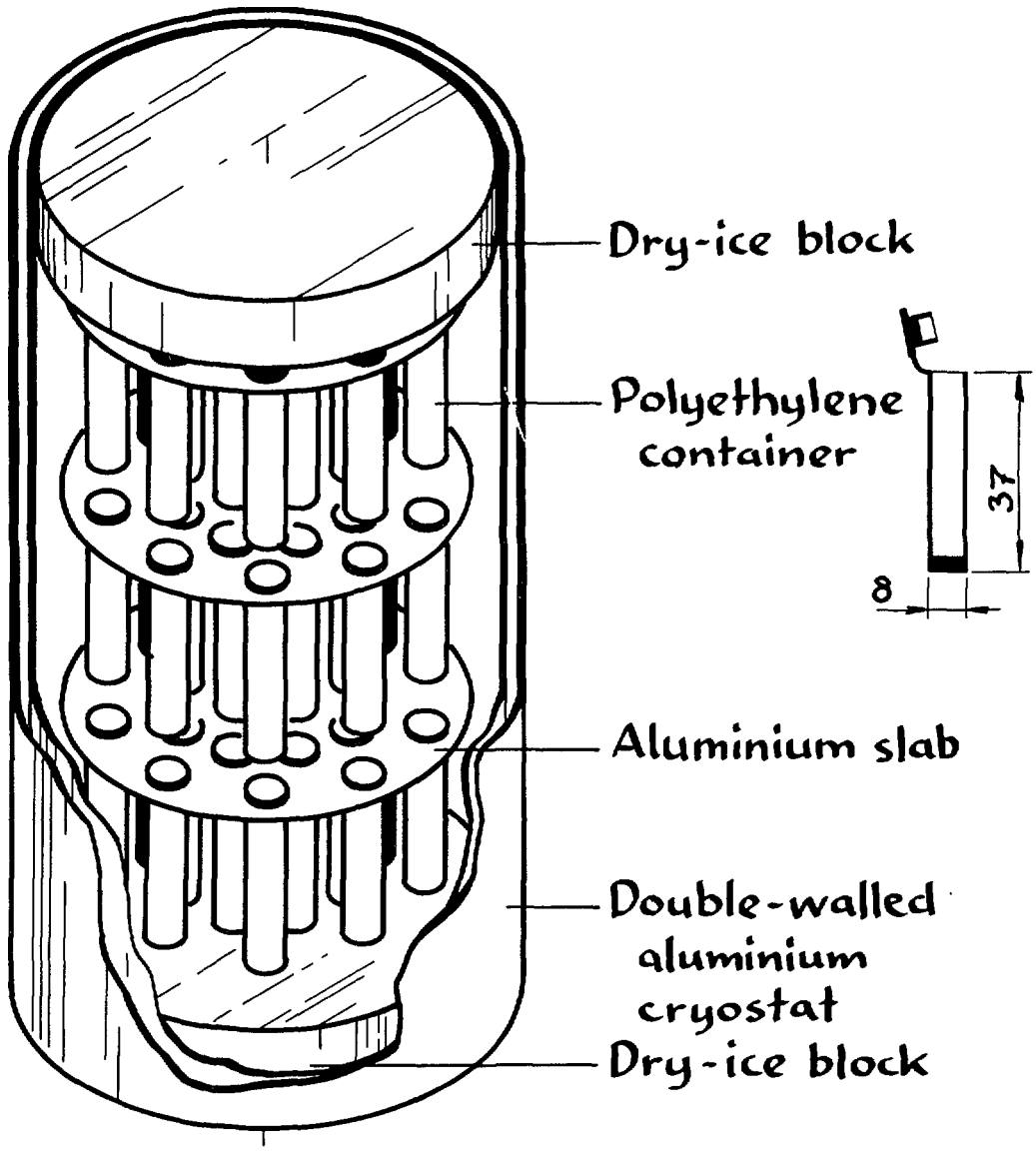
Layer	Central position	Peripheral position
Upper	0.97 ± 0.05	1.02 ± 0.05
Middle	0.98 ± 0.05	1.00 ± 0.05
Lower	1.00 ± 0.05	0.98 ± 0.05

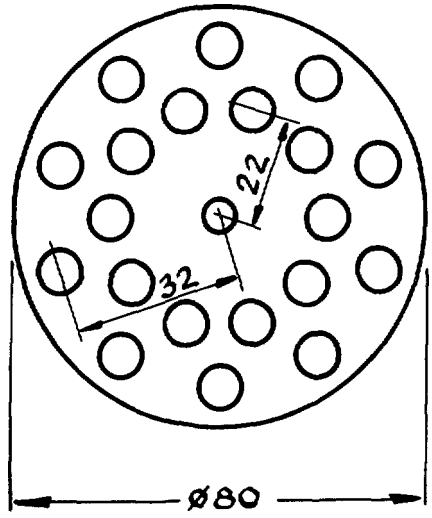
Normalized activities (of I^{128}) in each of the 3 layers. The values are mean values of 10 samples with standard deviation of the single value. The samples were geometrically arranged according to figure 1.

Table 2

Sample No.	Hg content in µg/g (Wet material)	
1	0.124	0.126
2	0.138	0.139
3	0.141	0.154

Dual analysis of Hg in 3 pike.





Sample positions in one layer

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Fig. 1. Arrangement for simultaneous irradiation of 60 samples in 3 layers.

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