NOTE

Determination of Iodine in Organic Compounds by the Activation Method with Bremsstrahlung Irradiation[†]

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1. Introduction

The iodine contents in organic compounds are ordinarily determined by the so-called Carius method, Pregl method, silver absorption method or flask combustion method, etc.¹⁾ These methods, however, are rather tedious and, sometimes, more or less erroneous owing to the fluctuation of blank test values, the unfavorable reaction between iodine and the absorbent used and so on.

In this work, the activation method is applied to the determination of iodine in organic substances in the non-destructive way, using the nuclear reaction of $^{127}\text{I}(\gamma, n)^{126}\text{I}$ with the aid of a linear accelerator.

2. Experimental

2.1 Mounting of the sample for irradiation

A small aluminum ring (6.5 mm i.d.) was put on an aluminum target holder in the way as shown in Fig. 1 and a weighed amount (about 10 mg) of the sample was taken in the ring. The target was wrapped tightly together with the target holder in aluminum foil of 4.0 mg/cm² thickness.

2.2 Standard

As the standard material, potassium iodide was used. Potassium iodide has such advantages that (i) it is available in high purity at a low cost, (ii) its melting point is rather high (723°C), (iii) it is not hygroscopic and (iv) the half lives of $^{38\text{m}}\text{K}$ (0.95 s) and ^{39}K

Fig. 1 Mounting of the sample for irradiation.

(7.71 m), the products of (γ, n) reaction on potassium, are much shorter than that of ^{126}I (12.8 d).

A weighed amount of potassium iodide was taken in an aluminum ring on the target holder, and wrapped in aluminum foil as described in 2.1.

2.3 Bremsstrahlung irradiation

The bremsstrahlung irradiation was carried out by the use of Linac at Tokai Research Establishment of the Japan Atomic Energy Research Institute. The bremsstrahlung was produced by shooting the accelerated electron beam of 20 MeV (average electron current was about 50 μA) on a platinum convertor. The samples and standards were irradiated under cooling with dry ice for 2 hr. The outline of the geometric ar-

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[†] 制動放射線照射による有機化合物中のヨウ素の放射化分析。池田長生, 高橋泰子, 木村 幹: 東京教育大学理学部 (東京都文京区大塚3-29-1)

rangement of the convertor and the target container is given in Fig. 2.

Fig. 2 Arrangement of the convertor and the target container assembly for irradiation.

a : accelerating tube
t : target container
s : target stack
c : Pt convertor with a water jacket
d : dry ice

2.4 Measurement of activity

Activities of ^{128}I produced were measured with a well-type NaI (Tl) scintillation counter.

3. Results and Discussion

3.1 Irradiation on potassium iodide

Several portions of 10~20 mg I of potassium iodide were weighed and mounted on the target holders in the way as described in 2.1. Each target holder with potassium iodide on it in aluminum foil was arranged

Fig. 3 Relation between irradiation position and specific activity produced.

being contacted one with another in the order shown at the bottom of Fig. 3. After the photon bombardment, the activity of each target together with the target holder was measured without unwrapping.

The decay curve for each sample all corresponds to disintegration with the half life of ^{128}I (12.8 d). The total activity and specific activity formed in each sample normalized to the end time of irradiation are shown in Table 1. The total activities produced in

Table 1 Relation between amounts of iodine taken, irradiation position and produced activities

Target No.*	Amount of KI taken (mgI)	Total activity produced** (cpm)	Specific activity** (cpm/mgI)
1	20.0	14.78×10^5	7.39×10^4
2	20.0	14.20×10^5	7.10×10^4
3	15.0	10.38×10^5	6.92×10^4
4	15.0	9.84×10^5	6.56×10^4
5	10.0	6.46×10^5	6.46×10^4
6	10.0	6.26×10^5	6.26×10^4

* The order of irradiation position from the convertor side.

** Normalized to the end time of the irradiation.

the pairs of the same amounts of potassium iodide samples, namely in the pairs of (No. 1 and No. 2), (No. 3 and No. 4) and (No. 5 and No. 6), respectively, are apparently in accordance with each other. The specific activity produced in each sample, however, shows the gradual decrease as shown in the last column of Table 1, indicating that the effect of absorption of bremsstrahlung by the sample and aluminum foil can not be neglected.

When we assume that the absorption of bremsstrahlung obeys the exponential law, the specific activity (S_d) at the depth d from the outer surface of the first sample should be expressed as follows;

$$S_d = S_0 e^{-\mu d} \quad (1)$$

where S_0 is the specific activity at $d=0$ and μ is the apparent absorption coefficient.

Here, it is assumed that the effect of scattering of bremsstrahlung would be negligible

and the thickness of the aluminum foil used (4.0 mg/cm^2) could be neglected with respect to that of potassium iodide layers. From Eq. (1),

$$\log S_d = \log S_0 - \frac{1}{2.303} \mu d \quad (2)$$

showing that plotting S_d against d on semi-logarithmic paper should give a straight line. The results of the treatment of the experimental data in this way are shown in Fig. 3, revealing that Eq.(2) holds good in this experiment.

3.2 Determination of iodine in organoiodine compounds

For the activation analysis of iodine in organoiodine compounds, a weighed amount of the sample was taken on a target holder and wrapped in aluminum foil. The pack thus obtained was then sandwiched between two aluminum packs which contain equal amounts (each W_0) of potassium iodide standards, and subjected to the photon bombardment in the direction vertical to the sandwich surface. After bombarding for a definite time (2 hr), the ^{126}I activities produced in the sample (A) as well as in the both standards (A_1, A_2) are measured.

From the linear relationship shown in Fig. 3, the following equation holds between the specific activities of the sample (S) and the both standards (S_1, S_2):

$$\log S = -\frac{1}{2}(\log S_1 + \log S_2)$$

Accordingly,

$$S = \sqrt{S_1 \cdot S_2}$$

Therefore, the amount of iodine in the sample (W_x) can be obtained by the following formula:

$$W_x = \frac{A}{\sqrt{A_1 \cdot A_2}} \cdot W_0 \quad (3)$$

For some examples of this method, the elementary analyses of iodine were tried on 3,5-diiodotyrosine and 2,4,6-triiodophenol. The results are given in Table 2, showing the fairly good concordance between theoretical and observed values for iodine contents.

The method is simple, selective and non-destructive and seems to be convenient for the elementary analysis of iodine in organic compounds.

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References

- 1) For instance, I.M. Kolthoff, P.J. Elving, (Ed.), "Treatise on Analytical Chemistry", Part II, Vol. 7, p. 335, Interscience Publishers, New York (1961); Japan Soc. Anal. Chem. (Ed.), "Organic Quantitative Analysis", (in Japanese), p. 38, Kyoritsu Shuppan K. K., Tokyo (1965)

Table 2 Elementary analysis of iodine

Compound	Molecular formula	Iodine taken (mgI)	Iodine found (mgI)	Iodine content (%)	
				Calc.	Obs.
3,5-Diiodotyrosine	$\text{C}_9\text{H}_9\text{I}_2\text{NO}_3$	10.0	10.1	58.6	59
			9.8		57
			9.4		55
2,4,6-Triiodophenol	$\text{C}_6\text{H}_3\text{I}_3\text{O}$	10.0	10.2	80.7	82
			10.0		81
			9.9		80