Radioactivation Analysis of Bromine and Iodine in Organic Compounds Using a Low-level Neutron Source

By Haruo Tsuji, Yuzuru Kusaka and Yasuo Namikawa

(Received June 6, 1962)

This paper describes some experimental results on the activation analysis of bromine and iodine in various organic compounds using a 50 mg.-radium-plus-beryllium-neutron source.

The sample solution to be analyzed was put in a polyethylene capsule (length, 86 mm.; diameter, 16 mm.; thickness, 1 mm.) and irradiated with neutrons at the sample hole of the irradiation apparatus shown in Fig. 1. The thermal neutron flux at the sample position,

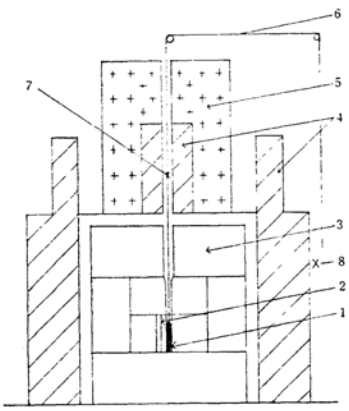

Fig. 1. Irraditation apparatus.

1 Neutron source
2 Sample hole
3 Paraffin
4 Lead
5 Paraffin containg boric acid
6 String
7 Position A
8 Position B

TABLE I. RADIOACTIVATION ANALYSIS OF BROMINE BY 1.0 hr. IRRADIATION

Sample	Weight g./5 ml.	Solvent	Bromine content in sample, %	
			Determined value	Theoretical value
Bromoform	0.527	Benzene	93.96±2.85	94.85
Bromoform	0.527	Methyl alcohol	93.59±2.85	94.85
Ethylene dibromide	0.763	Benzene	86.06±2.10	85.07
Ethylene dibromide	0.748	Methyl alcohol	84.04±2.14	85.07
Acetylene tetrabromide	0.469	Benzene	93.25±3.20	92.47
n-Butyl bromide	0.710	Benzene	60.99±2.11	58.32
Bromoacetic acid	0.826	Methyl alcohol	55.20±1.82	57.51
Bromobenzene	1.176	Benzene	51.64±1.36	50.90
Bromobenzene	1.204	Methyl alcohol	49.56±1.33	50.90
α-Bromonaphthalene	1.063	Benzene	35.34±1.32	38.59

TABLE II. RADIOACTIVATION ANALYSIS OF BROMINE BY 16.0 hr. IRRADIATION

Sample	Weight g./5 ml.	Solvent	Bromine content in sample, %	
			Determined value	Theoretical value
Ethylene dibromide	0.956	Benzene	84.32±0.84	85.07
Acetylene dibromide	0.481	Benzene	93.56±1.25	92.47
Bromoacetic acid	0.869	Methyl alcohol	55.04±0.69	57.51
Bromobenzene	1.204	Benzene	48.39±0.58	50.90
α-Bromonaphthalene	1.017	Benzene	38.74±0.59	38.59

TABLE III. RADIOACTIVATION ANALYSIS OF IODINE BY 1.0 hr. IRRADIATION

Sample	Weight g./5 ml.	Solvent	Iodine content in sample, %	
			Determined value	Theoretical value
Iodoform	0.411	Benzene	95.44±3.41	96.69
Methyl iodide	0.783	Benzene	90.65±2.04	89.41
Ethyl iodide	0.616	Benzene	81.17±2.44	81.37
n-Propyl iodide	0.415	Benzene	77.51±3.13	74.66
Iodoacetic acid	0.954	Water	65.20±1.68	68.25
o-Iodobenzoic acid	0.828	Methyl alcohol	53.30±1.69	51.17
p-Iodoaniline	0.578	Benzene	59.23±2.23	57.94

where the paraffin thickness between the neutron source and the sample hole was 7 mm., was approximately 1×10^3 neutrons/cm^2/sec. (The accuracy of this value will be estimated in another report). When the source was at position A in Fig. 1, the radiation dosage at position B, which was just in front of the lead shield, was about 2 mr./hr. When the source was set at the irradiation position, it was about 1 mr./hr.

A known amount of the sample to be analyzed was dissolved in an appropriate solvent, such as benzene, methyl alcohol or water, adjusted to 5.00 ml. in 30°C, and then irradiated. The irradiation times were 1.0 and 16.0 hr. in the bromine analysis and 1.0 hr. in the iodine analysis. After the end of the irradiation, the neutron source was pulled up to position A in Fig. 1 and the sample capsule was taken out rapidly. Then, after a 2.5 min. interval, the radioactivity induced in the sample was measured by a well-type NaI scintillation counter (1.75''$\phi\times$2''h. hole; 0.75''$\phi\times$1.5'' deep) for 30.0 min.

The induced radioactivities in the bromine analysis were due to ^{80}Br, ^{80m}Br and ^{82}Br, whose counting rate ratio at the beginning of the counting was 1000 : 80 : 66 in the 1.0 hr. irradiation. In the iodine analysis, the induced radioactivity was due only to ^{128}I.

Among the irradiation samples of a water, a methyl alcohol and a benzene solution of a compound to be analyzed, and even of a solid mixture of the compound and cane sugar, no appreciable difference in the induced radioactivity strength could be found.

Using the solution of *p*-dibromobenzene in benzene or hydrogen bromide in water as a standard substance in the bromine analysis, the bromine content in various organic compounds was determined. The analytical results obtained are shown in Tables I and II, along with the

theoretical values. Iodine analysis was done using a solution of iodobenzene in benzene as a standard. The results are shown in Table III.

As is shown in these tables, the results of the non-destructive activation analysis of bromine and iodine in various organic compounds were satisfactory. Although the sensitivities of both the analyses in this report were low, but it is expected that the use of a stronger neutron source will improve the sensitivities and will also make possible the use of smaller amounts of the sample.

Department of Chemistry
Faculty of Science
Konan University
Higashinada-ku, Kobe