Neutron Activation Analysis for Traces of Sodium in Polycarbonate

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The determination of microgram quantities of sodium in polycarbonate has received particular attention, because of its importance in an investigation of the properties, as well as in the development of an improved process for the production of this polymer.

Although the p.p.m. level of sodium in the polymer may be detemined by a combination of preliminary chemical concentration and subsequent flame-photometry, acid-base titration or polarography, these analytical methods are either difficult or troublesome, time-consuming, and apt to be inaccurate, therefore reliable results are rarely expected.

The authors have applied neutron activation and γ-ray spectrometry to the determination of sodium in bisphenol A-Polycarbonate, following the successfull use of this method in the determination of traces of sodium in aluminum,[1,2] diamond,[3] polyethylene[4] and polypropylene[5].

Experimental and Results

A half to two grams of the accurately weighed polycarbonate sample was sealed in a polyethylene pouch and, together with standard sample, irradiated for 2 hr. in the JRR-1 which produces neutron flux of about 10^{11} n./cm^2·sec. The irradiated sample was washed with nitric acid and water, and transferred to a test tube. No activity was found in the washings. The standard sample was prepared by impregnating a filter paper (Toyo Roshi, No. 5B, diam. 5.5 cm.) with 1 mg. of sodium (1 mg.-Na/ml. sodium oxalate solution), then drying the paper under an infrared lamp, and sealing it in a polyethylene pouch. After irradiation, the sodium was extracted with water, diluted to a suitable volume, and an aliquot of the resulting solution was transferred to a test tube.

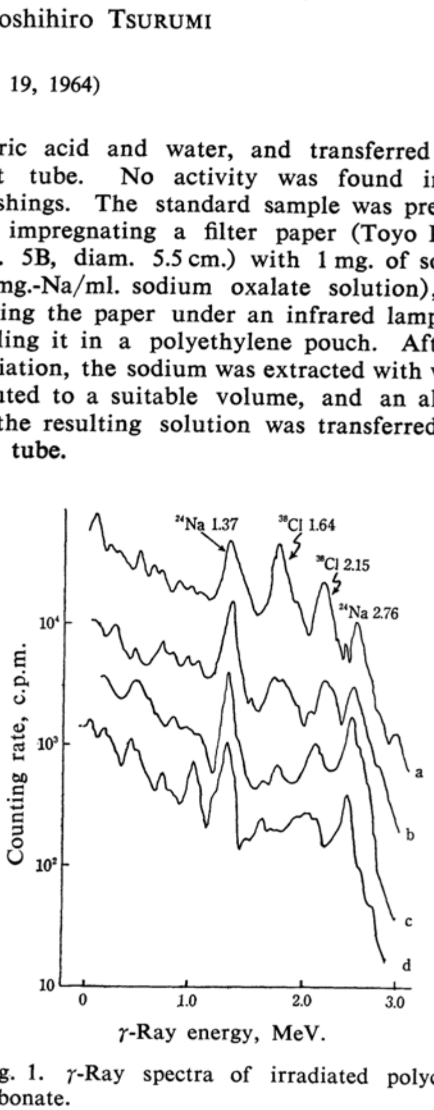

Fig. 1. γ-Ray spectra of irradiated polycarbonate.
(Sample II) (Na 25 p.p.m., Cl 700 p.p.m.)
Cooling a: 2.5, b: 4.5, c: 22.0, d: 46.0 hr.

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1) H. A. Das, *Nucl. Sci. Abstr.*, **17**, 14171 (1963).

2) R. Chauvin and P. Leveque, *Int. J. Appl. Rad. Isot.*, **1**, 115 (1956).

3) E. C. Lightowlers, *Anal. Chem.*, **34**, 1285 (1963).

4) Y. Suzuki, O. Saito, N. Sairenji and N. Saito, "Kiki Ni Yoru Kobunshi-Bunseki II," (Instrumental Methods in High Polymer Analysis, II) Hirokawa Publ. Co., Tokyo (1964), p. 172.

5) T. Takeuchi and D. Ishii, ibid., p. 180.

The γ-ray spectra of the polycarbonate and of the standard samples were obtained intermittently using a 3×3 inch^2 NaI(T1) crystal attached to the TMC[*1]-256 channel pulse height analyzer, the spectral imformation being recorded on an electric paper printer.

A representative γ-ray spectrum of the irradiated polycarbonate is shown in Fig. 1. The activity of chlorine-38 (half-life 37 min.) was dominant shortly after the irradiation. This activity would have come from a ^{37}Cl (n, γ) ^{38}Cl reaction, since methylene chloride had been employed as solvent in the production of the polycarbonate. After the chlorine-38 activity had decayed away, only the activity of sodium-24 remained and this was counted at convenient intervals.

The sodium contents were calculated from the 1.37 MeV photopeak height (above broken line) of sodium-24, which decayed with a half-life of about 15 hr.

As spectrochemical studies had already established the absence of magnesium and aluminum,[*2] interference by ^{24}Mg (n, p) ^{24}Na or ^{27}Al (n, α) ^{24}Na reactions would be negligible.

No appreciable self-shielding effect was observed in this experiment. The purity of the 1.37 MeV. photopeaks was verified by measurements of the half-life.

Typical results of the analyses are shown in Table I, where the samples shown in column B were obtained by treating the samples indicated in column A in order to decrease sodium content.

The proposed analytical method was applied to an investigation of the efficiency with which sodium is extracted into water from a methylene chloride solution of the polycarbonate.

The extraction procedure was as follows: 100 g. of the polycarbonate sample was dissolved in 500 ml. of methylene chloride, and this solution was shaken for 1 min. with 500 ml. of water in a separating funnel.

A repetition of the above procedure with additional amounts of water gave the samples which had been repeatedly washed. After separation of the water layer, isopropyl alcohol was added to the organic layer with stirring until polycarbonate powder precipitated; the precipitate was then dried in vacuo.

The results are shown in Fig. 2 and indicate that the repeated washing of the polymer solution with water does not effectively remove the sodium.

TABLE I. RESULTS OF ANALYSES FOR SODIUM IN VARIOUS POLYCARBONATE SAMPLES

(I—III pellet, IV—VI powder, sample taken ~1 g.)

Sample	Sodium content p.p.m.		Chloride content p.p.m.
	A	B	
I	30	—	113
II	25	—	700
III	0.7	—	147
IV	11	1.8	—
V	4.5	0.8	—
VI	5.1	1.0	—

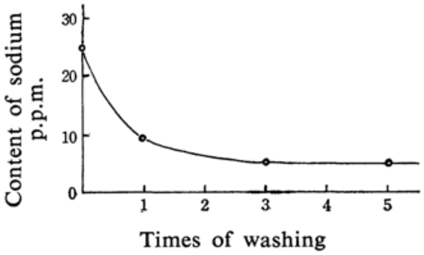

Fig. 2. Washing of the polymer with water.

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[*1] Technical Measurement Company.

[*2] 800 μg. of magnesium or 10 mg. of aluminum is equivalent to 1 μg. of sodium.