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# A Possible Method for the Estimation of Bisphenol-A in a Reaction Mixture of Phenol and Acetone

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To work out a procedure for estimation of bisphenol-A in a reaction mixture with phenol and acetone, synthetic mixtures were used. Bisphenol-A and phenol were separated from acetone by dissolving the mixture in approximately NaOH(2*N*) and then extracting acetone with diethyl ether subsequently boiling the extracted solution to remove any remaining traces of acetone. The solution was cooled, acidified with conc. HCl, drop by drop, till acidic to congo red paper and then re-extracted with diethyl ether. The solvent was removed, the solute dissolved in spectroscopic ethanol and optical densities measured at 230 mμ and 265 mμ respectively. From these, the amount of bisphenol-A was found out (relative error ± 5%). The extinction coefficient at 230 mμ and 265 mμ were found to be 62.5 and 7.085 litre/gm.cm. for bisphenol-A and 8.31 and 12.995 litre/gm.cm. for phenol respectively.

The acid catalysed condensation of phenol with acetone leads to the formation of a compound known as bisphenol-A. During the reaction, the medium would consist of acetone, phenol and bisphenol-A as the principal components. In studying the kinetics and mechanism of this reaction, estimation of the individual components at definite intervals of time was necessary. Filippov and Ivolga[1] determined bisphenol-A in phenol spectroscopically in ethanol solution at 230 mμ and 265 mμ. According to them, the direct use of the absorbance ratio at these wavelengths enabled estimation without a calibration curve with a relative error of 4%. However, we have observed that the method is of limited applicability when acetone is present along with phenol and bisphenol-A. Moreover, when the condensation reaction of phenol and acetone is carried out in solvents such as alcohols, chlorinated hydrocarbons, etc. the sharpness of absorption is lowered. The spectrophotometric method can however be successfully applied by isolating the bisphenol-A from the reaction mixture and estimating it in an ethanolic medium. The present paper describes the experimental procedure.

## EXPERIMENTAL

(a) *Preparation of pure bisphenol-A.*—60 gms. (75 c.c.) of dry acetone was quickly saturated with dry HCl gas at -5 to 0° and added to a mixture of 500 gms. of pure phenol and 0.5 c.c. of hexyl mercaptan at 45° placed in a 3-necked flask. The flask was fitted with a stirrer, a condenser and a thermometer and was placed in a thermostatic bath. The contents of the flask turned, within 10 minutes, into a slurry which was stirred for about 4 hours and then transferred to a 2-litre beaker and neutralised with 10% ammonium carbonate solution. The organic

phase was washed free of electrolytes and then steam-distilled to remove most of the phenol. The product thus obtained (200 gms.) was crystallised from chlorobenzene[2].

The crude bisphenol-A (90 gms.) was taken in 130 c.c. of chlorobenzene and refluxed till it goes in solution. The solution was kept at 60°C for about 15 minutes and the resulting slurry filtered in a buchner funnel (yield, 75 gms.). The process of crystallisation was repeated four times and the final product dried at 150 ± 1°C for 24 hours (m.p. 158.4°C).

(b) *Thin layer chromatography of crude and crystallised bisphenol-A.*— Layers approximately 0.3 mm thick were formed on glass plates (10 cm.×20 cm.) using a laboratory-made applicator, with a well-stirred suspension of 6 gms. of kieselguhr (E. Merck), 0.5 gms. of plaster of Paris (E. Merck) and 13 c.c. of water. The plates were heated at 105–110°C for 45 minutes, cooled in a desiccator, and then impregnated in a 5% v|v solution of dimethyl formamide in acetone. The plates were then kept under a fan to remove acetone. The samples were applied as 0.5% solution in acetone in amounts varying from 10-15 $\mu$g. using glass capillaries along a line 2.5 cm. above the plates and 2 cm. apart. Development was carried out in an ascending manner with the solvent system[3] cyclohexane : ethyl acetate : acetic acid in the volume proportion of 125:25:4.5. When the solvent reached a distance of 14.5 cms. in about 25 minutes, the plates were taken out. The phenols were detected by spraying the chromatogram first lightly with diazotised sulphanilic acid[4] and then with 20% sodium carbonate solution (Fig. 1). Crude bisphenol-A gave 6 spots whereas crystallised bisphenol-A gave only one spot. The R values for phenol and bisphenol-A were 0.66 and 0.30 respectively.


FIG. 1.

Absorbent : kieselguhr inpregnated with 5% dimethyl formamide.
Solvent : Cyclohexane : Ethylacetate : Acetic acid (125:25:4.5)
Detection : sprayed with (1) diazotised sulphanilic acid (2) 20% $Na_2CO_3$ soln.
1 & 5 = Pure phenol.
2 = Pure bisphenol-A.
3 & 4 = Crude bisphenol-A.

(c) *Estimation of Bisphenol-A.*—Bisphenol-A was estimated spectrophotometrically by measuring the optical density (O.D.) of its solution in ethanol at 230 mμ and 265 mμ respectively. Standard solutions of pure phenol and bisphenol-A were prepared in spectroscopic ethanol and their O.D. were measured with the results shown in Table I.

TABLE I

*Optical densities of pure phenol and pure bisphenol-A at varying concentrations.*

| Phenol in mgm/litre. | Optical density 230 mμ. | 265 mμ. | Bisphenol-A in mgm/litre. | Optical density 230 mμ. | 265 mμ. |
|---|---|---|---|---|---|
| 16.36 | 0.131 | 0.211 | 3.92 | 0.249 | 0.028 |
| 20.00 | 0.163 | 0.255 | 7.83 | 0.490 | 0.055 |
| 32.72 | 0.264 | 0.422 | 10.00 | 0.625 | 0.070 |
| 41.00 | 0.338 | 0.632 | 11.75 | 0.730 | 0.082 |
| 49.08 | 0.404 | 0.640 | 15.66 | 0.960 | 0.110 |
| 65.40 | 0.537 | 0.850 | 20.01 | 1.225 | 0.140 |
| 81.80 | 0.678 | 1.003 | 39.16 | — | 0.277 |
| | | | 60.02 | — | 0.425 |
| | | | 78.32 | — | 0.554 |
| | | | 100.00 | — | 0.710 |
| | | | 117.50 | — | 0.832 |

From the data in Table I the extinction coefficients ( $\epsilon$ ) for phenol and bisphenol-A were found to be

$$\epsilon^{230,\ \text{phenol}} = 8.310 \text{ litres/gm.cm.}$$

$$\epsilon^{265,\ \text{phenol}} = 12.995 \text{ litres/gm.cm.}$$

$$\epsilon^{230,\ \text{bisphenol-A}} = 62.500 \text{ litres/gm.cm.}$$

$$\epsilon^{265,\ \text{bisphenol-A}} = 7.085 \text{ litres/gm.cm.}$$

(d) *Method of estimation.*—To standardise the method of estimatiin of bisphenol-A in a reaction mixture of phenol and acetone, synthetic mixtures of bisphenol-A, phenol, acetone and chlorobenzene in varying amounts were made and treated as follows :

Mixtures containing 0.5 to 2.5 gms. of phenols were dissolved in 25 c.c. of approximately NaOH(2*N*) at room temperature and the solution was thrice extracted with diethyl ether. The extracts were rejected. The solution was then boiled gently over a low flame for about 15 minutes, cooled, and transferred quantitatively in a separating funnel with about 20–25 c.c. distilled water and acidified with conc. HCl drop by drop till acidic to congo red paper. The phenol and bisphenol-A set free were extracted 5 times with diethyl ether using about 50 c.c. portions thrice and 30 c.c. portions twice. The extracts were collected together, dried over anhydrous sodium sulfate, filtered and ether distilled off. The contents were then taken up in spectroscopic ethanol, the solution diluted

suitably and the absorbance measured at 230 mμ and 265 mμ using a Beckmann DU spectrophotometer. From these, the amount of bisphenol-A was determined by solving the following equations :

$$O.D._{230\ m\mu} = 8.31 \times C_{phenol} + 62.5 \times C_{bisphenol\text{-}A}$$

$$O.D._{265\ m\mu} = 12.995 \times C_{phenol} + 7.085 \times C_{bisphenol\text{-}A}$$

where $C_{phenol}$ and $C_{bisphenol\text{-}A}$ are the concentrations of phenol and bisphenol-A in gms./litre.

The results are given in Table II and the amount of bisphenol-A found by solving the equations mentioned above with percentage error are given in Table III.

TABLE II

*Synthetic mixtures, which have been treated as in the procedure (d) and their optical densities at different dilution.*

| I* | II | III | IV | V |
|---|---|---|---|---|
| Phenol : 0.2012 gms.<br>Bisphenol-A : 0.2128 gms.<br>Acetone : 1 c.c. | 1 c.c.<br>3 c.c.<br>10 c.c. | 0.153<br>0.445<br>1.48 | 0.047<br>0.127<br>0.417 | 0.2122<br>0.2092<br>0.2094 |
| Phenol : 0.5103 gms.<br>Bisphenol-A : 0.1501 gms.<br>Acetone : 3 c.c. | 1 c.c.<br>3 c.c.<br>10 c.c. | 0.125<br>0.374<br>1.25 | 0.060<br>0.182<br>0.605 | 0.1494<br>0.1481<br>0.1488 |
| Phenol : 0.1042 gms.<br>Bisphenol-A : 1.2041 gms.<br>Acetone : 3 c.c.<br>Chlorobenzene : 1 c.c. | 1 c.c.<br>3 c.c.<br>10 c.c. | 0.101<br>0.310<br>1.00 | 0.049<br>0.141<br>0.473 | 1.201<br>1.264<br>1.2030 |
| Phenol : 1.6931 gms.<br>Bisphenol-A : 1.6011 gms.<br>Acetone : 3 c.c.<br>Chlorobenzene : 3 c.c. | 1 c.c. | 1.09 | 0.321 | 1.635 gms. |
| Phenol : 1.6510 gms.<br>Bisphenol-A : 20.6020 gms.<br>Acetone : 3 c.c.<br>Chlorobenzene : 5 c.c. | 1 c.c. | 0.921 | 0.421 | 2.622 |

*I = Amount of substances taken. These substances were treated in the procedure described above (d) and were collected in a one litre volumetric flask in spectroscopic ethanol.

II = Amount of solution in I diluted to one litre.

III & IV = Optical density of solutions in II at 230 and 265 mμ respectively.

V = Bisphenol-A found from O.D. by use of equations in text.

TABLE III

*Amount of bisphenol-A found by solving the equations in the text with percentage error.*

| Bisphenol-A taken in gms. | Bisphenol-A found in gms. | Percentage error. |
|---|---|---|
| 0.2128 | 0.2122 | −0.28 |
| 0.2128 | 0.2092 | −1.69 |
| 0.2128 | 0.2094 | −1.60 |
| 0.1501 | 0.1494 | −0.47 |
| 0.1501 | 0.1481 | −1.33 |
| 0.1501 | 0.1488 | −0.87 |
| 1.2041 | 1.2010 | −0.26 |
| 1.2041 | 1.2640 | +4.97 |
| 1.2041 | 1.2030 | −0.09 |
| 1.6011 | 1.6350 | +2.12 |
| 2.6020 | 2.6220 | +0.77 |

## DISCUSSION

The ultraviolet spectrophotometric procedure appears to be suitable for estimation of bisphenol-A from a mixture of phenol, bisphenol-A and acetone, after complete removal of acetone by the method described. The method, when standardised against artificial mixtures of bisphenol-A and phenol shows an error of ± 5% in estimation of bisphenol-A.

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