

공기중에 오염되어 있는 BHC의 정량에 관한 연구

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Study on the Determination of Benzene Hexachloride in Contaminated Air

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요 약. 공기중에 오염되어 있는 미량의 (benzene hexachloride : BHC)를 정량하는 간단하고 예민한 방법을 연구하였다.

시료를 *n*-hexane에 통과시켜 BHC를 녹인 다음 *n*-hexane 용액의 흡광도를 245 nm에서 측정하고 고체 BHC 표준시료를 기화시킨 후 *n*-hexane에 통과시키고 같은 방법으로 흡광도를 측정하여 얻은 점량선으로부터 농도를 계산하였다.

ABSTRACT. A simple and sensitive method of determining trace amounts of benzene hexachloride (BHC) in air has been investigated. The sample is passed through the *n*-hexane solution and the absorbance of it was measured at 245 nm spectrophotometrically and the concentration of benzene hexachloride was calculated using a working curve obtained from the vaporized standard benzene hexachloride passed into *n*-hexane.

INTRODUCTION

Several methods have been devised for the quantitative determination of benzene hexachloride. Methods previously reported was spectrophotometry,¹⁻⁴ polarographic analysis⁵ and gas chromatography,⁶⁻⁸ etc. Benzene hexachloride in contaminated air could be determined by those methods, but those methods are tedious and have little sensitivity to determine trace amounts of the benzene hexachloride in air sample.

In this work a simple and sensitive analytical method was investigated. The sample is passed

through the *n*-hexane solution and the absorbance of it was measured at 245 nm spectrophotometrically and the concentration of benzene hexachloride was calculated using a working curve.

EXPERIMENTAL

Benzene hexachloride used in this experiment was given by Hankook Agricultural Pharmaceutical Company. This composition were α -BHC 74%, β -BHC 6.8%, γ -BHC 13% and δ -BHC 6.0% and other reagent were analytical grade. Demineralized water is used in this experiment. Spectrophotometer was Perkin-Elmer, model

202 and absorption cell, 10 mm in width.

RESULTS AND DISCUSSION

Selection of Solvent. Ethyl alcohol was selected as solvent to determine benzene hexachloride in the air sample by dissolving it in a solvent and measuring the absorbance of the solution. A definite amount of solid benzene hexachloride was weighed and dissolved in a known volume of ethyl alcohol and the absorbance of it was measured in the wavelength region from 200 nm to 400 nm spectrophotometrically.

The maximum absorption of benzene hexachloride was appeared at 220 nm but ethyl alcohol itself absorbs the above wavelength significantly, therefore ethyl alcohol is avoided in this experiment.

Secondly *n*-hexane solvent, which has no absorption peak of itself in the UV region, was selected to dissolve benzene hexachloride and the absorbance of it was measured.

The absorption spectrum obtained by 200 mg of standard benzene hexachloride dissolved in 100 ml *n*-hexane is shown in Fig. 1. The maximum wavelength in this solvent appears at 245 nm, which is shifted toward longer region by 25 nm than in ethyl alcohol. This seems to be due to solvent effects. The working curve made in this solvent is given in Fig. 2. The curve is obtained plotting absorbance versus the concentration in series of benzene hexachloride dissolved in definite volume of *n*-hexane. To apply this working curve of Fig. 2 for determination of benzene hexachloride in air sample, two hundred milliliters of air sample contained benzene hexachloride was passed into 10 ml *n*-hexane at the rate of 200 ml per minute and the absorbance of this 10 ml *n*-hexane was measured at such a condition as the working curve. Its spectrum is curve A in Fig. 3. The spectr-

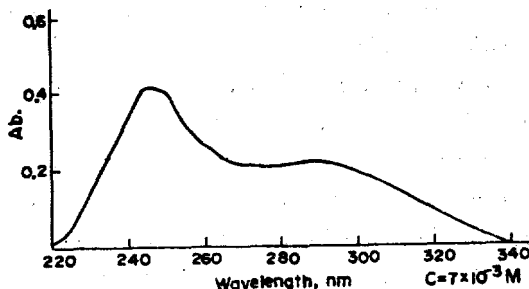


Fig. 1. Absorption spectrum of unvaporized BHC.

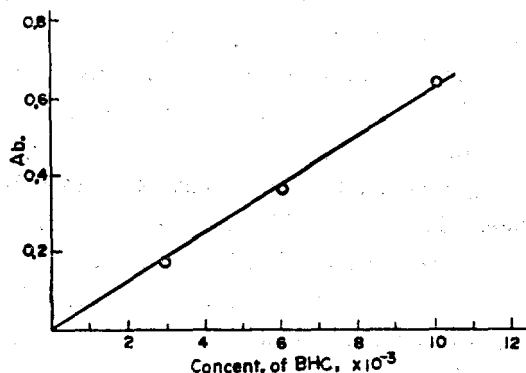


Fig. 2. Standard curve of unvaporized BHC at 10 °C.

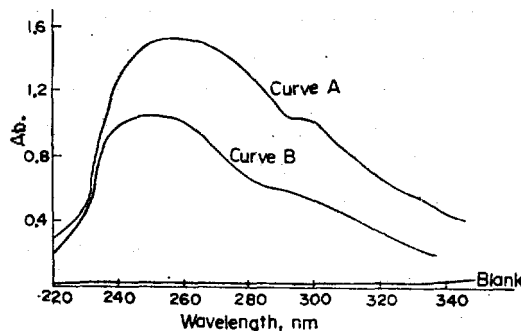


Fig. 3. Absorption spectrum of air sample. sample taken=200 ml in 10 ml *n*-hexane.

ophotometer used here has 1.5 absorbance scale as maximum. Absorbance of curve A in Fig. 3 at 245 nm is too intense to be measured. So this solution was diluted to half concentration with pure *n*-hexane and the absorbance of the dilute solution was measured and curve B in Fig. 3 was obtained. From the absorbance of curve B and its dilution factor and volume of

sample taken, concentration of benzene hexachloride in air sample could be calculated using working curve of Fig. 2. Calculated value was about 400 ppm.

To confirm the value 5×10^{-6} l of 10 ml *n*-hexane solution from which curve A in Fig. 3 was obtained was injected into column of gas chromatography⁶ and the peak height of elution curve was measured and the concentration of benzene hexachloride in this sample was calculated by comparing the peak height of standard sample. The value was 4ppm. This difference demonstrated the defect of the present method.

Vaporization of Solid Benzene Hexachloride. The defect of this method seems to come from working curve of Fig. 2, obtained by dissolving solid benzene hexachloride directly into *n*-hexane. The hint that benzene hexachloride in air sample would be existed as vaporized state made a consideration that the working curve was achieved from the vaporized benzene hexachloride, that is, solid benzene hexachloride was vaporized and this vapor was passed into *n*-hexane. So a series of known amount of solid benzene hexachloride was vaporized individually in vaporizing flask as like Fig. 4 for one hour at 90 °C and each vapor was passed into *n*-hexane contained in tall test tube.

At this time the entrance stem in tall test tube had sparger and the end of entrance tube was reached to the bottom of the tall test tube. After the vaporization is over, heating of the vaporizing flask was stopped and the flask was taken off from the water jacket. The pressure in the flask was reduced because the temperature in the flask was reduced. So the *n*-hexane flowed into the flask from the tall test tube. The flask was shaken several times to dissolve the benzene hexachloride vapor left in the flask.

After dissolving enough of benzene hexachloride vapor in the flask, the *n*-hexane solution was transferred into photometric cell and the absorbance was measured at 245 nm against pure *n*-hexane solvent as a blank.

Working curve of Fig. 5 was obtained from this process. These curves show that the absorbance of the unvaporized benzene hexachloride (Fig. 2) and that of the vaporized benzene hexachloride (Fig. 5) for identical amount of solid benzene hexachloride differed in great deal. The absorbance measured from the vaporized benzene hexachloride was extremely larger than that measured from the unvaporized benzene hexachloride.

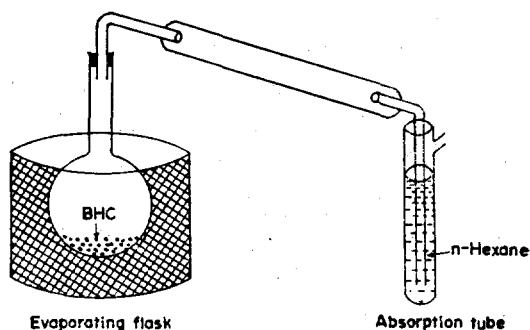


Fig. 4. Vaporization apparatus.

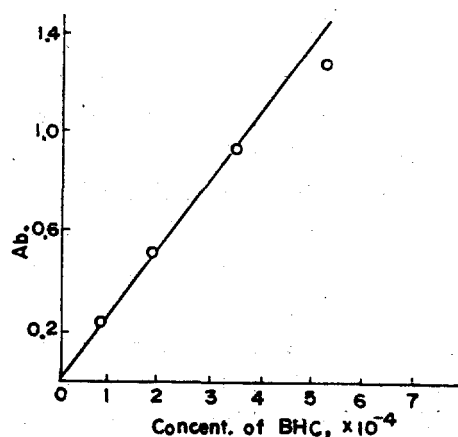


Fig. 5. Standard curve of vaporized BHC.

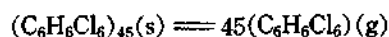
Absorbance Variation According to Vaporization Temperature.

To see the dependence of absorbance on the vaporization temperature, an identical amount of solid benzene hexachloride was added in the vaporizing flask shown in

Fig. 4 and 10 ml of *n*-hexane was filled in the tall absorbing test tube. After the apparatus was set up, the temperature of oil jacket was heated to various temperature and maintained for one hour at each temperature. After one hour heat source was removed and the vaporized flask was taken off above the oil jacket. Now at each temperature vaporized benzene hexachloride must have passed into *n*-hexane and unvaporized benzene hexachloride must be left in the vaporizing flask. *n*-Hexane in the absorbing tube flowed into the vaporizing flask. With shaking the flask several times, both the vaporized benzene hexachloride left in flask and the unvaporized benzene hexachloride was dissolved. The absorbance of *n*-hexane obtained in each temperature was measured. At this time, the higher the temperature rose, the larger the absorbance was. So *n*-hexane which dissolved the vapor of benzene hexachloride was diluted with pure *n*-hexane with known ratio and the absorbance of dilute solution was measured.

Absorbance variation curve obtained by plotting the absorbance versus temperature is shown in Fig. 6. Fig. 6 indicates that the absorbance at temperatures from 10 to 20°C are alike and starts to increase rapidly at 30 until 60°C and maintains almost constant absorbance at above 90°C. Such results show that the absorbance of unvaporized benzene hexachloride is smaller than that of vaporized benzene hexachloride. The results of absorbance increase in the vaporized state could be expressed on the assumption that benzene hexachloride exists as polymer in the solid state and as monomer in the vapor state.

If the absorbance at about 10°C is due to polymer state and the absorbance at above 90°C is due to monomer state, the following relationship will be achieved since the absorbance increase is 45 times.



Investigation of Benzene Hexachloride in the Atmosphere of Inha University Area.

Hankook Agricultural Pharmaceutical Factory placed before Inha university manufactures benzene hexachloride from May to October every year. This benzene hexachloride contaminated the air around Inha University area. To investigate the contamination of air, sampling place was selected and one liter of air sample was passed through 10 ml *n*-hexane at the rate of 200 ml per minute and the absorbance of *n*-hexane was read and then the concentration was calculated by using working curve of Fig. 5. The list was shown in the Table 1. The

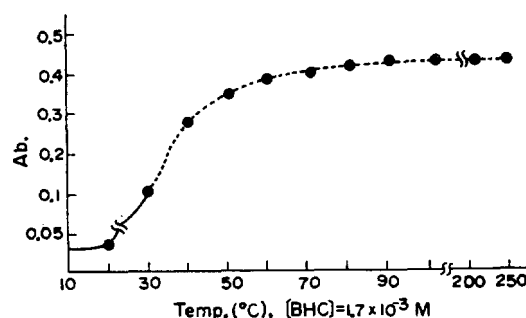


Fig. 6. Absorbance variation according to vaporized temperature.

Table 1. Analytical results of benzene hexachloride in air sample (ppm).

date	Place No.		
	1	2	3
77. 4. 27	3.2	4.0	51.0
5. 27	30.2	32.0	35.0
	30.3*	33.0*	34.0*
5. 31	5.3	12.2	11.0

sample with high concentration was rechecked by gas chromatographic method. These data in the table was marked with star and the two value are well close within tolerance error.

CONCLUSION

(1) Benzene hexachloride contaminated in air was determined by passing the sample into *n*-hexane and the absorbance of the *n*-hexane was measured at 245 nm spectrophotometrically.

(2) The absorbance of solid benzene hexachloride and that of vaporized benzene hexachloride are different. The absorbance of vaporized state is 45 times as large as that of solid state.

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