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#### ANALYSIS OF SOME CHLORINATED HYDROCARBONS IN WATER BY

## HEADSPACE TECHNIQUE



Mr. Wutichai Yentongchai

A thesis Submitted in Partial Fulfillment of the Requirements for the Degree of Master of Science

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เทคนิคเฮดสเปส ได้รับการพัฒนาขึ้นเพื่อใช้วิเคราะห์ สารประกอบคลอริเนตเตด ไฮโดร-คาร์บอน ที่ระเหยได้ง่ายในตัวอย่างน้ำ เช่น เมธิลลีนคลอไรด์ คลอโรฟอร์ม คาร์บอนเตตระคลอไรด์ 1,1,1-ไตรคลอโรอีเทน และ ไตรคลอโรเอธิลีน โดยทำการศึกษาและประเมินค่าปัจจัยต่างๆ ที่มี และ ประสิทธิภาพของการสกัด (percent ผลต่อความไว ( sensitivity ) ได้แก่ เวลาที่ระบบเข้าสู่สมดุล อุณหภูมิ อัตราส่วนของปริมาตรระหว่างน้ำต่อ อากาศ ปริมาณของเฮดสเปสแก้สที่ใช้ในการวิเคราะห์และการเติมเกลือโซเดียมคลอไรด์ โซเดียมซัลเฟต และ แคลเชียมคาร์บอเนต ผลการศึกษาพบว่า การใช้อุณหภูมิ 60 °C เป็นเวลา 60 นาที ด้วย อัตราส่วนน้ำต่ออากาศ 30 : 30 ในขวดขนาด 60 มิลลิลิตร ปริมาตรของเฮดสเปสแก๊สที่น้ำเข้า สู่เครื่องแก๊สโครมาโตกราฟ 1.50 มิลลิลิตร และใช้เกลือโชเดียมซัลเฟต 13.00 กรัม ที่เหมาะที่สุดของการวิเคราะห์โดยเทคนิคเฮดสเปส ซึ่งสามารถที่จะนำมาใช้กับการวิเคราะห์ สารประกอบ ตลอริเนตเตดไฮโดรคาร์บอน ที่ระเหยได้ง่ายในตัวอย่างน้ำได้ เทคนิคนี้สามารถตรวจวัดสารประเภทนี้ใน ตัวอย่างน้ำได้ที่ระดับต่ำที่สุดซึ่งต่ำกว่า 0.50 ไมโครกรัมต่อลิตร (ppb) โดยมีประสิทธิภาพการสกัด สารประกอบคลอริเนตเตด ไฮโดรคาร์บอนนี้ อยู่ในช่วง 54.22 ถึง 90.81 เปอร์เซ็นต์ และมีค่าเบี่ยง เบนมาตราฐานสัมพันธ์ ( %RSD ) ระหว่าง 1.04 ถึง 4.81 เปอร์เซ็นต์ นอกจากนี้ยังได้ศึกษา ความถูกต้องในการวิเคราะห์ด้วยเทคนิคนี้โดยวิธีอินเทอร์นัลสแตนดาร์ดไดเซชัน ( internal standerdization method ) พบว่า เปอร์เซ็นต์ของความผิดพลาดอยู่ในช่วง 0.31 ถึง 6.44 เปอร์เซ็นต์ จากการเก็บตัวอย่างน้ำประปาและน้ำจากสระน้ำ 6 แห่ง มาตรวจวิเคราะห์ด้วยเทคนิคนี้พบว่า ตัวอย่าง น้ำ ที่เก็บมาทั้งหมด มีสาร เมธิลลีนคอลไรด์ และ คลอโรฟอร์ม อยู่ในช่วง 10.22 - 448.68 0.22 - 31.86 ส่วนในพันล้านส่วน ตามลำดับ

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WUTICHAI YENTONGCHAI : ANALYSIS OF SOME CHLORINATED HYDROCARBONS IN WATER BY HEADSPACE TECHNIQUE. THESIS ADVISER :

Dr. SITTICHAI LEEPIPATPIBOON, Ph.D. 179 pp. ISBN 974-582-090-3

In this study, a headspace technique was developed for the determination of some volatile chlorinated hydrocarbons i.e., methylene chloride, chloroform , carbon tetrachloride , 1,1,1-trichloroethane and trichloroethylene in water samples. Various factors having effect on the sensitivity and the percent recovery i.e., equilibration time, temperature, liquid to gas phase volume ratio, injection volume and salting out with sodium chloride, sodium sulfate and calcium carbonate were studied and evaluated. The temperature of 60 °C, equilibration time of 60 minutes, the liquid to gas phase volume ratio of 30:30 in 60 mL serum vial, 1.50 mL of injection volume, and salting out with 13.00 g of anhydrous sodium sulfate were chosen as the optimal headspace analysis condition for the analysis of the volatile chlorinated hydrocarbons in water samples. The minimum detectable level of this technique was lower than 0.50 ppb (Mg/L) for all studied compounds and the percent recoveries of the chlorinated hydrocarbons were in the range of 54.22 - 90.81 % with  $\pm 1.04 - 4.81$  % RSD. The accuracy of this technique was also studied by means of internal standardization method and the percent errors were in the range of 0.31 - 6.44 % at the ppb level of concentration. Moreover, the developed technique was also applied to analyze tap water and pool water collected from six different places. Results showed that all water samples appeared to have methylene chloride and chloroform in the range of 10.22 - 448.68 and 0.22 - 31.86 ppb, respectively.

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