METHOD FOR THE DETERMINATION OF LINDANE

The Schechter-Hornstein method for benzene hexachloride[1/] has been found to give accurate and reproducible results for air samples and other materials. However, the method as published is tedious and the special apparatus relatively expensive and complicated. Modifications of the apparatus and method of isolation have been made which simplify the method and reduce the number and quantities of reagents needed.

Modified Shechter-Hornstein Method

Reagents -

1. Glacial Acetic Acid (A.C.S.)
2. Malonic Acid (C.P.)
3. Zinc Dust
4. Nitrating Acid. This is a 1:1 mixture by volume of C.P. fuming nitric acid (sp.gr. 1.49-1.50) and C.P. concentrated sulfuric acid (sp.gr. 1.84).
5. Sodium Hydroxide, 40 percent aqueous solution
6. Ferrous Sulphate (C.P.)
7. Potassium Hydroxide, 40 percent aqueous solution (470 g./liter)
8. Adsorption Alumina, 80-200 mesh
9. Activated Alumina, Grade F-20[2/]

1/ Anal. Chem. 24: 544-548 (1952).

2/ Aluminum Ore Company, East St. Louis, Ill.

10. Mineral Oil (U.S.P.)

11. o-Phosphoric Acid (C.P.), 85 percent

12. Diethyl Ether (A.C.S.). The ether is purified before use: One liter volumes are extracted twice with 50 ml. portions of 10 percent ferrous sulphate in a separatory funnel. The ether is then passed through 30 grams of adsorption alumina (packed by tapping) in a 25 mm. O.D. glass column with a restricted end. A glass wool plug is used to retain the alumina in the column.

13. Methyl Ethyl Ketone, white label[3/]. This reagent is purified by passing 500 gram portions through 35 grams of activated alumina using the glass columns described above.

14. Distilled Water

15. Lindane

Apparatus -

1. Columns for purification of reagents (described above)

2. Special fractionating columns and nitration tubes (figure 1)

3. Powerstats[4/] (or other adjustable voltage controls)

4. Heaters, electric (as shown in figure 2 or equivalent, such as Glas-Col mantles[5/])

5. Stainless steel protruded packing[6/]

6. Flasks, round or flat bottom, 300 ml., S 24/40

3/ Eastman Kodak Company, Rochester, N. Y.

4/ Superior Electric Company, Bristol, Conn.

5/ Glas-Col Apparatus Co., Terre Haute, Ind.

6/ Scientific Development Company, State College, Pa.

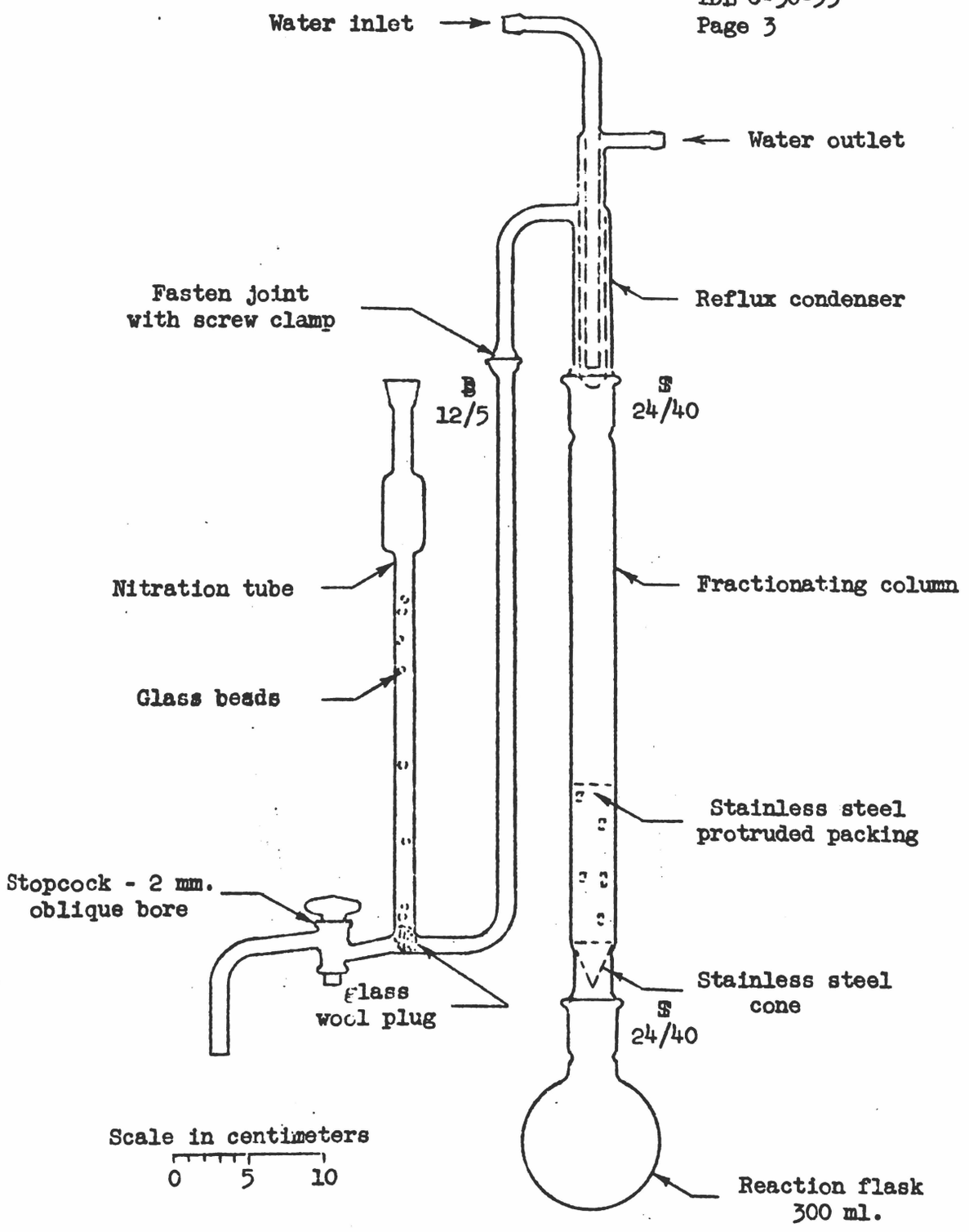
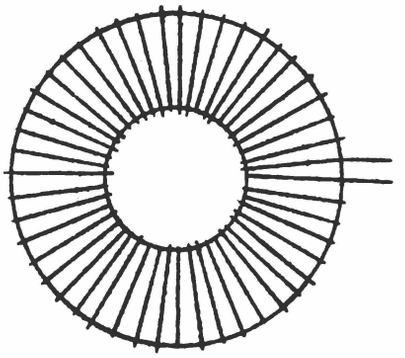


Figure 1: Modified S-H apparatus for dechlorination and nitration



Detail of Aluminum Ring Heater

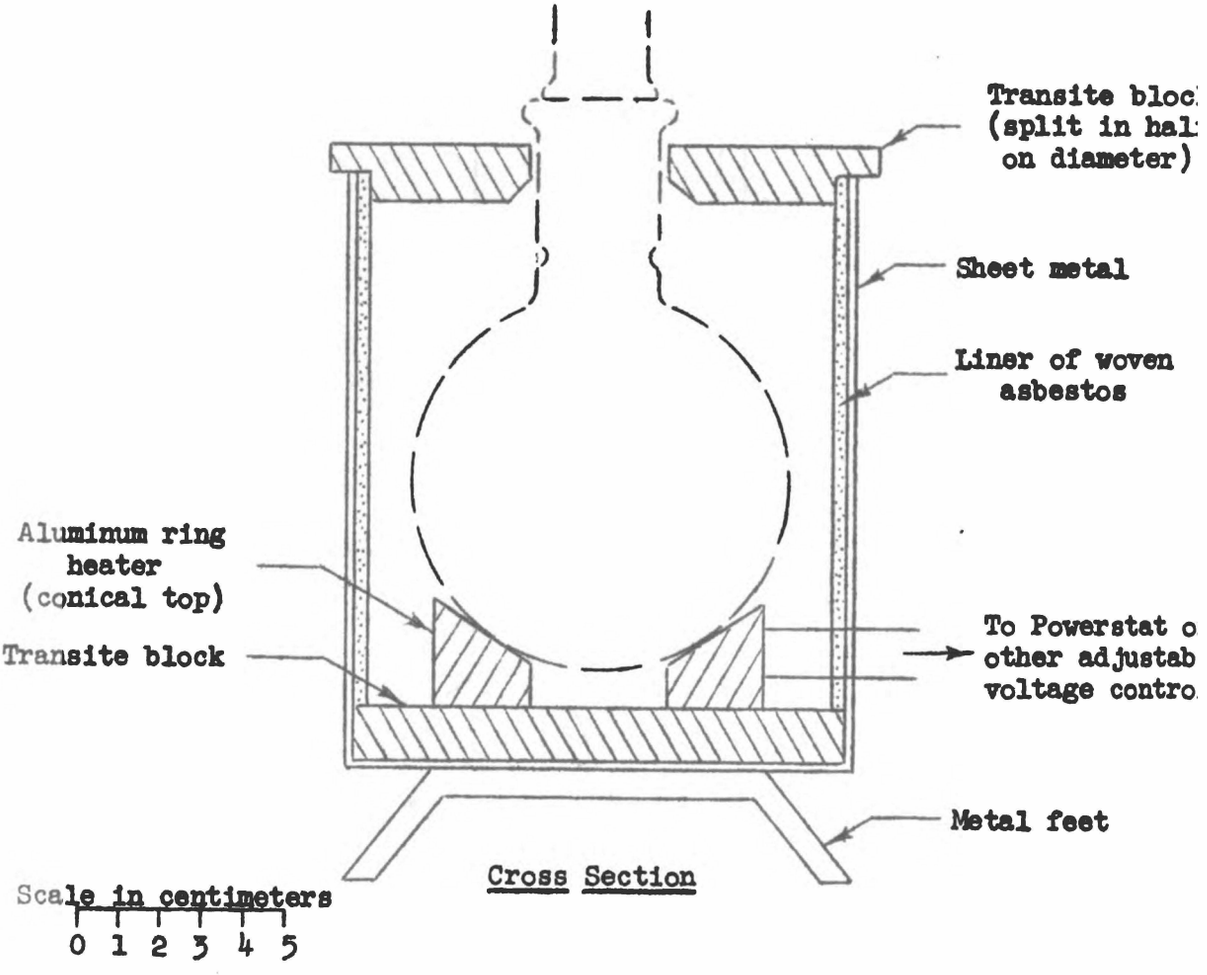


Figure 2: Electric heater for modified S-H apparatus

7. Flasks, Erlenmeyer, 300 ml., G.S.

8. Joints, ground glass, S, fitting the 300 ml. G.S. Erlenmeyer flasks.

9. Bottles, pyrex, 125 ml., G.S.

10. Chromatography tubes. They are made from 20 x 175 mm. pyrex test tubes joined to 3" of 7 mm. pyrex tubing.

11. Ice water bath

12. Steam bath

13. Glass beads

14. Glass wool

15. Ring stand with clamps

16. Aspirating tube

17. Spectrophotometer

Method -

1. For preparation of a standard curve, standard solutions of lindane containing 10.0 micrograms per ml. are prepared in glacial acetic acid. Various amounts of the standard are put into 300 ml. flat or round bottom flasks equipped with S 24/40 joints.

2. One hundred ml. of glacial acetic acid are then added to each flask. Four grams of zinc dust and 4 grams of malonic acid are added to each sample, taking care not to get any zinc on the necks of the flasks.

3. Each flask is then connected to a fractionating column, greasing all joints well with phosphoric acid. Five ml. of the nitrating acid are now added to each nitration tube.

4. The 300 ml. reaction flasks are placed in electric heating units (figure 2) and the Powerstats are set at 60 volts and kept there for 10 minutes. Then the voltage is increased gradually until 120 volts is reached. The samples are allowed to nitrate for 1/2 hour from the time the acetic acid begins to reflux.

5. During the half-hour reaction period an ice water bath is prepared into which 125 ml. U.S. pyrex bottles containing 5 ml. of distilled water are placed to cool. At the expiration of the reaction time the nitrating tubes are removed from the fractionating columns and placed in clamps on a ring stand.

6. The nitrating mixture is then drained into the 125 ml. bottles while they are kept in the bath. The nitration tubes are rinsed with a total of 30 ml. of ice-cold distilled water which is also run into the 125 ml. bottles. The samples are neutralized with 40 percent NaOH and a few drops in excess are added. A small piece of litmus dropped into the bottle serves as the indicator.

7. The nitration tubes are then rinsed with a total volume of 35 ml. of ether, which is run into the 125 ml. bottles. The samples are removed from the ice bath, stoppered, and shaken by hand for three 1-minute periods, allowing the layers to separate between shaking periods. The bottles are returned to the bath when not being shaken. After the last shaking the samples are allowed to stand for 15 minutes and then the water layer is aspirated off.

8. The chromatography tubes are prepared by packing 5 grams of adsorption alumina in the 20 x 175 mm. glass columns using a small glass wool plug to retain the alumina. The ether solution is poured very carefully into the column, collecting the eluate in a 300 ml. glass stoppered Erlenmeyer flask. Each bottle is rinsed with 35 ml. of ether and this is also poured gently onto the column. A glass bead and one drop of mineral oil are added to each sample in the 300 ml. flask.

9. The flasks are then fitted with S ground glass joints and placed on a steam bath. At intervals during the evaporation the flasks are swirled to prevent overheating on the bottom. When about 6 ml. remains the flasks are removed from the steam bath and carefully

tilted on their sides to complete the evaporation. This usually takes about 15-20 minutes.

10. When the evaporation is complete, 10 ml. of methyl ethyl ketone and 1 ml. of 40 percent KOH are added to each sample. The flasks are stoppered and shaken by hand for 2 minutes. Then they are put in a dark place for 15 minutes.

11. The optical density readings are taken at 565 millimicrons on a spectrophotometer. Each sample is read separately to prevent the fading which occurs due to loss of contact with the KOH.

12. The extinction coefficient (absorptivity) is calculated from the following equation:

$$K = \frac{D}{C}$$

Where:

K = extinction coefficient (absorptivity)

D = optical density (absorbance)

C = concentration of lindane per 10 ml. of methyl ethyl ketone added to develop the color.

An average value of K equal to 0.0065 with a variation of $\pm$ 5 percent has been found for a number of standard determinations.

13. In analyzing air samples for lindane, sampling and preparation of the samples has been done successfully by the method of Hornstein and Sullivan[7/].

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7/ Anal. Chem. 25: 496-498 (1953).