Modified and Improved Procedure for Schöniger Total Chlorine Residue Analysis

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The Schöniger total chlorine method of pesticide residue analysis has been modified and new techniques for preparing and handling the sample holders have been developed. An automatic cone-filling device is used for low chlorine samples. A 1-liter, round-bottomed combustion flask with a magnetic stirrer enables absorption of gases without shaking. The method color reaction may be used to determine up to 175 μg. of chloride.

THE COMBUSTION of solvent extract of plant material prior to determination of residues of chlorinated pesticides has been described (*2*). Several modifications simplify the method.

Experimental

Preparation and Handling of Plastic Cones. The following procedure eliminates the need for rubber gloves.

Wearing cloth gloves, cut out 1-mil cellulose acetate film using the cardboard template. Still wearing gloves, heat and solvent-seal the plastic to form the cone as described in the procedure. Wash and rinse the cone by handling it with forceps rather than rubber gloves. Use forceps to place the dry cone in the funnel.

The funnel consists of a 60°, 60-mm. borosilicate glass powder funnel from which the entire stem is cut off and the outlet reshaped to yield a smooth, boat-shaped opening 3 mm. wide and 15 mm. long. Transfer 5 ml. of solvent extract to the cone. After evaporation, draw the cone through the base of the funnel using forceps. Fold upward with the aid of a second pair of forceps, while drawing the cone through until a small packet is formed. This procedure is most advantageous when the residue left is not excessively oily. Place the packet in the platinum holder and attach the fuse using forceps.

Automatic Filling Device. The plastic cone will conveniently hold 5 ml. of sample extract. To increase sensitivity, it was previously necessary to concentrate the extract or to pipet successive portions of the strip solution into the cone. An automatic filling device (Figure 1), built to facilitate handling larger samples, consists of a 50-ml. graduate with a 24/40 ⅀ female joint sealed on top. Three 4-mm. glass tubes (*A*, *B*, and *C*) are sealed on or through the male section of the joint as shown.

Pipet 10 ml. more than the desired portion (up to 50 ml.) of the strip solution into the graduate. Grease the male joint and place the top section on the graduate. Close off tube *C* (rubber plug or tubing). Place the plastic cone supported in the funnel under tube *B*. The tube should extend about 1 cm. into the cone. Close off either opening at the top of tube *A*, using your finger. Apply air to the other opening of this tube using a squeeze bulb. As the solvent begins siphoning through tube *B*, remove your finger and slowly remove the suction bulb.

The strip solution will fill the cone until its level is at the same height as the bottom of tube *A*. As solvent evaporates from the cone, more will automatically siphon into the cone until the liquid in the graduate reaches the 10-ml. mark (bottom of tube *B* inside the graduate). Heating mantles to fit funnels are now available which may be used to speed evaporation. After the remaining solvent in the cone has evaporated, the cone is burned as described.

Tube *C* is necessary, as a vent when putting the top section on the graduate, to avoid compression of air inside and uncontrolled siphoning of liquid at the start which may flood the cone. The volume of solution which has siphoned is easily determined by difference by recording the liquid level in the graduate after removing the top section.

The combustion flask consisted of a 1-liter, round-bottomed borosilicate flask with a side arm to which the balloon is attached. The 34/28 standard taper, female joint is sealed on the neck at a height, so that the platinum basket is about 0.5 inch above the absorbing solution (30 ml. of distilled water). A $1^5/_8$-inch, egg-shaped magnetic stirring bar is spun rapidly for 10 minutes to absorb combustion gases.

Colorimetric Determination of Residues. Residue levels and chlorine content of many of the common pesticides may vary considerably. The colorimetric method (*1*) can be used to determine chloride over a considerable range of concentrations. Table I gives the necessary information for use of the Bergmann and Sanik procedure for determining up to 175 μg. of chloride.

The determination of small quantities of chloride is most accurate when the chloride reagent blank is at a minimum. A satisfactory standard curve and lower reagent blank in the 0- to 50-μg. chloride range can be obtained by using 1 ml. of ferric reagent.

Table I. Data for Use of Colorimetric Method (*1*) at Low and High Chloride Levels

Range Chloride, μg.	Volume of Absorbing Solution for Analysis, Ml.	Volume of Color Reagents, Ml.		Length of Absorption Cell, Cm.
		Ferric ammonium sulfate	Mercuric thiocyanate	
0–25	25	2.5	2.5	10
0–50	15	1.0	3.0	2
0–125	15	5.0	5.0	2
0–175	20	5.0	5.0	1

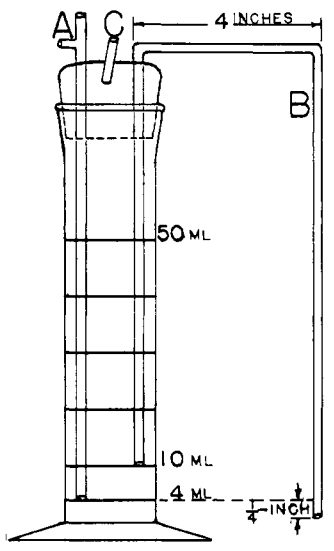

Figure 1. Automatic cone-filling device

Determination of Other Pesticides

DD, lindane, and Thiodan in forage by flask combustion have been determined (*2*). The method has since been used to determine dieldrin, endrin, heptachlor, and methoxychlor in alfalfa and dalapon in bird's-foot trefoil and cherries.

Literature Cited

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(2) Lisk, D. J., J. AGR. FOOD CHEM. **8,** 119–21 (1960).

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