A Quantitative Sample Concentration Procedure

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Preparatory pesticide chromatography frequently militates inclusion of a concentration step following extraction and clean-up procedures. This is particularly pertinent to residue investigations outlining food chains of micro and macro invertebrates where a picogram level of sensitivity is essential because the specimens often weigh less than a milligram.

A rapid technique has been developed by the authors that permits quantitative concentration of a sample whose volume is almost entirely available for chromatographing. By sealing with a propane torch the distal end of the graduated portions of 10, 5, and 1 (0.01 ml. graduations) ml. serological pipets, it was possible to concentrate a 5 ml. volume to 0.01 ml. The internal diameter of the 1 ml. pipet segment does not accommodate the barrel of a Hamilton syringe and consequently is limited in length to the 2 inches of the needle. The 5 and 10 ml. pipet

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vials are not limited by this consideration and, unless injection directly from them is anticipated, could be 4 to 5 inches long. It is necessary to determine volume between the closure and the last calibration because the capacity is altered in the fusing process. This is readily accomplished with a tuberculin and 0.01 ml. Hamilton syringe for the 10 and 5 ml. pipet vials respectively. The vial length and high ratio of depth to surface area limits loss of sample through evaporation prior to injection.

A routine run consists essentially of blowing the sample down with nitrogen, transferring the concentrate and washings (3) to next smaller vial size and repeating the procedure. The concentration can be performed in 5 minutes and has made possible the detection (sodium flame) of 1 ppm. of organophosphate residue in a single marine oligochaete weighing 0.1 mg.

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