Improved volatilization chamber for the Kontes sweep co-destillation apparatus (K-50050)

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Introduction

Sweep co-distillation or forced volatilization is a technique used for cleanup of sample extracts by volatilization in a temperature controlled chamber in the presence of an inert gas, and sweeping the more volatilized materials through the chamber into a collection trap. The less volatile materials are then discarded leaving only the more volatile "cleaned-up" materials for analyses. This device provides a very fast, efficient and economical means for preparing samples, particularly pesticides for analyses (1,2, 3,4,5,6). In 1966 Kontes Glass Company marketed the model K-50050 Sweep Co-Distiller, based on the work of Storherr and Watts (5). An evaluation of this apparatus and some simple modifications are presented in order to provide a more accurate and consistent temperature within the sample tube where the more volatile and less volatile materials are separated. In brief, these modifications included redesigning the volatilization chamber and attaching a preheater oven to the outer chamber to heat the gas prior to entering the sample tubes. Two types of volatilization chamber were tried. One used the marketed model but included a copper jacket to surround the sample tubes. The other replaced the contents of the chamber with an aluminum block designed to contain the heating elements and the sample tubes. The latter was found to be superior in performance and would be the recommended modification particularly when precise control or operation is desired.

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Construction and Operation

Preheater

The preheater assembly was designed to heat the gas prior to its entrance into the sample tube so that at the point of injection the temperature inside the tube would be as high or higher than the rest of the tube, resulting in complete volatilization of the injected sample.

Fabrication was from a aluminum block 5" long x 1" wide x 1 3/8" high. Four 7/16" holes (through which the Storherr tubes would be placed) were centered and drilled at one inch intervals along the length of the block. Cuts, 3/8" wide, were made from the top of the block down to each hole, to allow the arm of the tube to pass through the oven and snap into the retainer clip.

A 3/8" hole, 3 1/2" deep, was drilled from the side of the oven to house the cartridge heater just below the level of the sample tube holes. A 1/8" hole about 3/4" deep was drilled from the front and near the center, just above the location of the cartridge heater, to house the thermocouple when needed. The preheater was attached to the front cover of the volatilization chamber from the inside with two No. 8-3/4" self tapping screws. A piece of 1/4" thick transite was cut to fit and placed in front of the preheater oven with two No. 8-3/4" self tapping screws to protect the operator from possible burning. Independent control of the preheater was provided by connecting heater to a variable autotransformer capable of handling 2 amps.

Volatilization Chamber

A simple method for improving the temperature within the Storherr sample tubes was first attempted by inserting 1/2" I.D. copper tubing the length of the oven to form a heat conductor jacket around the sample tubes. The front cover and transite front wall were removed and the necessary copper tubing inserted. However, in order that this may be accomplished, the chamber housing the sample tubes must be partially disassembled. The round mounting

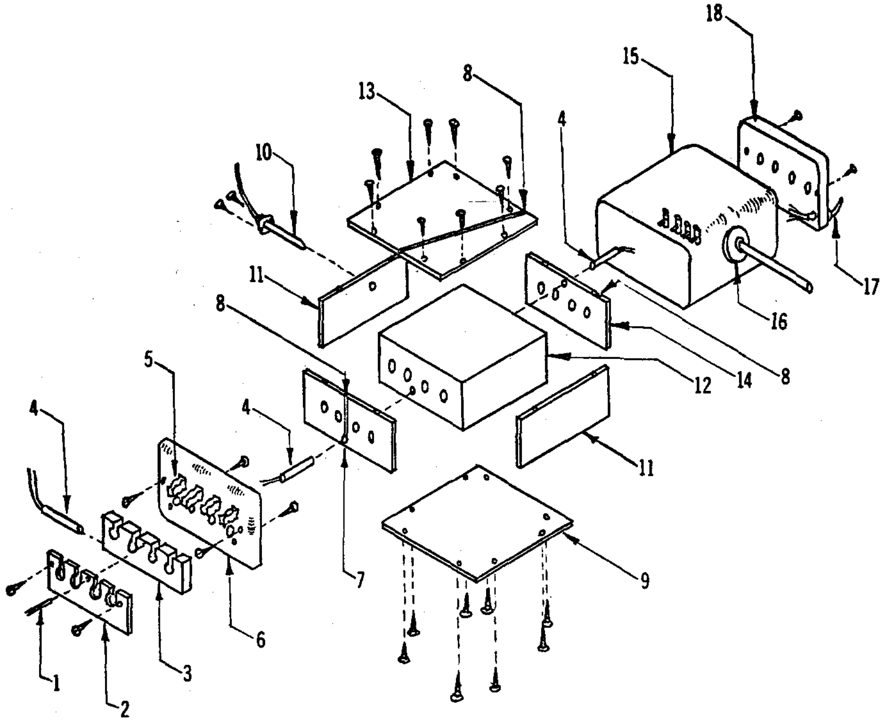

Figure 1. EXPLODED VIEW OF THE MODIFIED KONTES CO-DISTILLATION APPARATUS, MODEL K-50050

Illus. No.	Part Name	Illus. No.	Part Name
1	Thermocouple Wire, 10Ω Iron-Constantan (6 ft. LG)	10	Thermocouple-Thermistor Assy.
2	Transite Protection Plate	11	Insulite Side Walls
3	Aluminum Preheater Oven	12	Aluminum Volatilization Chamber
4	Cartridge Heater, Hotwatt 150 W, 115 V, HiWatt	13	Insulite Top Plate
5	Retainer Clips	14	Insulite Back Wall
6	Front Cover	15	Stainless Steel Cover
7	Insulite Front Wall	16	Mounting Plate & Bracket
8	V - Groove for High Temp. Wire	17	Oven Controller Cord
9	Insulite Bottom Plate	18	Back Cover

bracket plate, the flange holding the thermocouple-thermistor assembly and the front and back metal covers were removed. The stainless steel cover surrounding the chamber could be moved back far enough to expose the two screws that hold the front wall of transite. At this point, the copper jackets were inserted and the chamber reassembled.

A more efficient type of volatilization chamber was fabricated from a 4 3/4" x 4 3/4" x 2 3/4" aluminum block. Four 7/16", sample tube holes were centered and drilled at one inch intervals across the width of the block. These holes were made to line up with the holes in the front and back covers. Two, 3/8" x 3 1/2" holes were drilled, one from the front, and the other from the back, to accommodate the 150 watt cartridge heaters. One was located at the center front about 1/2" below the sample tube holes. The other was located at the center back 1/2" above the sample tube holes. A hole, 3/8" x 2 1/2", was also drilled in the upper left side in position to match the hole in the chamber housing where the thermistor-thermocouple assembly fits.

In order to place the aluminum block into the housing it was necessary to first disassemble the oven. The heating coils and the retainer eyehooks were removed from the top and bottom of the chamber and replaced with the cartridge heaters. The transite surrounding the block was notched in such a way as to provide space for the high temperature lead wires running from each of the heater cartridges to the right rear corner of the chamber. At this junction point, the leads from the two heaters were either soldered or quick connected in parallel to the oven cord connecting the oven controller. The heater cartridges were inserted into the aluminum block and the complete unit assembled.

Thermocouple Addition

The purchase of an additional pyrometer was avoided by attaching the preheater oven thermocouple to the existing pyrometer. The top control unit was removed and a new 6 ft. standard 10Ω iron-constantan thermocouple (Varian Aerograph, Walnut Creek, Calif. -replacement item or equivalent) was fed through the grommeted lead-in hole. A hole was drilled in the front panel to accommodate a toggle switch (SPDT Cutler-Hammer, No. 8282K12 or equivalent). The switch was tightened in place and the two thermocouples were soldered to the switch so that one temperature at a time may be read out on the pyrometer (Fig. 2).

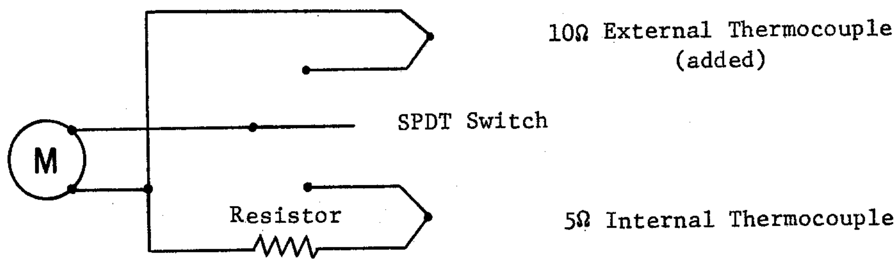

Figure 2. Diagram of SPDT Switch and Thermocouples

Results and Discussion

The volatilization chamber of the commercial sweep co-distiller showed considerable temperature variation within the sample tube itself, as well as between the actual sample tube temperature and the indicated oven temperature. Table I shows the actual temperature within the sample tubes as compared to the indicated temperature on the pyrometer using the three types of chambers. Modification of the volatilization chamber to include the copper tubing jackets showed considerable improvement in consistency of the temperature within the sample tubes, however, the sample temperature was considerably lower than the indicated chamber temperature. Replacement of the commercial volatilization chamber with the aluminum block gives constant front to back sample temperatures that are consistent with the indicated chamber temperature. Temperature variations between the inlet and chamber can be achieved at the discretion of the operator through the separate controls for the

TABLE I.

Comparison of temperature readings (°C) of three different ovens at four locations inside a Storherr sample tube.[a]

Oven[b]	Inside front wall	Sample injection area	Mid-oven	Inside back wall
Commercial K-50050	120	125	170	160
Copper tubing jackets	129	135	135	135
Aluminum chamber	170	170	170	170

a/ Gas flow was maintained at 600 ml/min. The preheater was not operating.
b/ Pyrometer reading was 170° C. in all cases.

TABLE II.

Temperature readings (°C) within the modified aluminum block forced volatilization chamber at different control settings under varied conditions.

Control setting	Empty tube, no gas flow	Empty tube, gas flow[a]	Glass wool, gas flow[a]	Preheater[b], glass wool, gas flow[a]
30	130	110	120	120
40	150	130	140	140
50	190	170	180	180
60	250	235	240	240
70	350	330	340	340

a/ Gas flow through the tube was 600 ml/min.
b/ Preheater temperature was maintained at 200° C.

preheater unit and the volatilization chamber. The preheater facilitates a temperature constant at the point of injection which should be as high or higher than what may be found throughout the sample tube.

The modified instrument with the aluminum block chamber is making possible an improvement in cleanup and recovery for a broader range of pesticides. However, warm up and cool down time has been somewhat lengthened but the two heaters are adequate for an equilibrium time of about one hour.

Table II lists temperature readings at various control settings of the volatilization chamber under varied conditions. As would be expected, temperature midway in the sample tube is somewhat dependent on gas flow and packing.

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