*Journal of Chromatography*, 555 (1991) 302–305
Elsevier Science Publishers B.V., Amsterdam

CHROM. 23 349

## Short Communication

# Use of a Sep-Pak cartridge for preparative collection in gas chromatography

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(First received December 3rd, 1990; revised manuscript received April 4th, 1991)

ABSTRACT

Commercial Sep-Pak cartridges (Si or $C_{18}$) with a piece of short PTFE tubing could be used directly without any cooling or heating as a convenient and efficient collection device for preparative gas chromatography. Four sets of experimental results are presented.

## INTRODUCTION

Conventionally, cold-bath (dry-ice–acetone or NaCl–ice–water) cooling or certain types of alternative heating and cooling devices have been used for preparative collections in gas chromatography (GC) [1–5]. However, in our hands these methods suffered several drawbacks, such as the inconvenient supply of dry-ice, the liability to breakage of the long glass collection tubes and the often low recovery yields. We recently found that commercial Sep-Pak cartridges (Si or $C_{18}$) with a piece of short PTFE tubing could be used directly without any cooling or heating as a very convenient and efficient collection device. In this paper, four sets of experimental results are presented.

## EXPERIMENTAL

### *Materials*

Ethyl acetate was of analytical reagent grade. α-Pinene was purchased from TCI (Janpan, Taiwan); all other chemicals were obtained from Fluka, (Buchs, Switzerland). The properties of the test samples and international standards are given in Table I. Both silica and $C_{18}$ Sep-Pak cartridges were products of Waters Assoc. (Milford, MA, U.S.A.).

0021-9673/91/$03.50

TABLE I

PROPERTIES OF TEST SAMPLES (TS) AND INTERNAL STANDARDS (IS)

| Group | Compound | B.p. (°C)[a] | Formula | M.W. |
|---|---|---|---|---|
| I | Isolongifolol (TS) | 113–114 (m.p.) | $C_{15}H_{26}O$ | 222.37 |
| | Globulol (IS) | 87–88 (m.p.) | $C_{15}H_{26}O$ | 222.37 |
| II | Longifolene (TS) | 258–260 | $C_{15}H_{24}$ | 204.36 |
| | Ledene (IS) | 268–270 | $C_{15}H_{24}$ | 204.36 |
| III | Pulegone (TS) | 223–224 | $C_{10}H_{16}O$ | 152.24 |
| | Carvone (IS) | 228–230 | $C_{10}H_{14}O$ | 150.22 |
| IV | α-Pinene (TS) | 154–156 | $C_{10}H_{16}$ | 136.24 |
| | α-Phellandrene (IS) | 171–174 | $C_{10}H_{16}$ | 136.24 |

[a] Taken from Fluka Catalogue.

### *Gas chromatography*

All collections were carried out on a Shimadzu GC-8A gas chromatograph with a 5% SE-30 (methylsilicone) packed column (3 m × 3 mm I.D.) and under the following conditions, unless specified otherwise: injector temperature, 220°C; column temperature, programmed from 50 to 200°C at 5°C/min; detector (thermal conductivity) temperature, 220°C; and carrier gas (nitrogen) flow-rate, 25 ml/min.

All determinations were performed on a Hewlett-Packard HP 5890A gas chromatograph equipped with fused-silica capillary columns of DB-Wax (bonded phase, polyethylene glycol), (30 m × 0.25 mm I.D., 0.25 μm film thickness) (J&W Scientific) or SP-2250 [methyl–phenylsilicone (50:50)] (60 m × 0.25 mm I.D., 0.2 μm film thickness) (Supelco) and a flame ionization detector. The carrier gas (nitrogen) flow-rate was 2 ml/min and the make-up gas flow-rate was 30 ml/min. The splitting ratio was 1:80. The injector and detector temperatures were 250°C. The oven temperature was programmed from 150 (DB-Wax) or 50°C (SP-2250) to 220°C at 5°C/min. A Shimadzu Model C-R3A integrator was used.

### *Collection device*

As shown in Fig. 1, a 6 cm × 4.2 mm O.D. PTFE tube connecting with a Sep-Pak cartridge was used as the collection device. The PTFE tube was snugly inserted into the outlet of the gas chromatograph for about 3–4 cm and fixed with a silicone-rubber septum screw-cap.

### *Sample collection and recovery*

All injected samples were weighed (*ca.* 10 mg) and dissolved in 100–200 μl of ethyl acetate. The same set of Sep-Pak cartridge and PTFE tube was used for collection for the same sample during three or four injections performed on each sample.

The collected material was washed out with ethyl acetate from the bottom side of the cartridge, with the PTFE tube still connected, into a 5-ml calibrated flask. The final volume was precisely adjusted to 5 ml. An internal standard of the same kind as the test sample was chosen for comparison. The weighed standard was also dissolved in a 5-ml calibrated flask. By using a 1-ml pipette, 1 ml was taken from both the

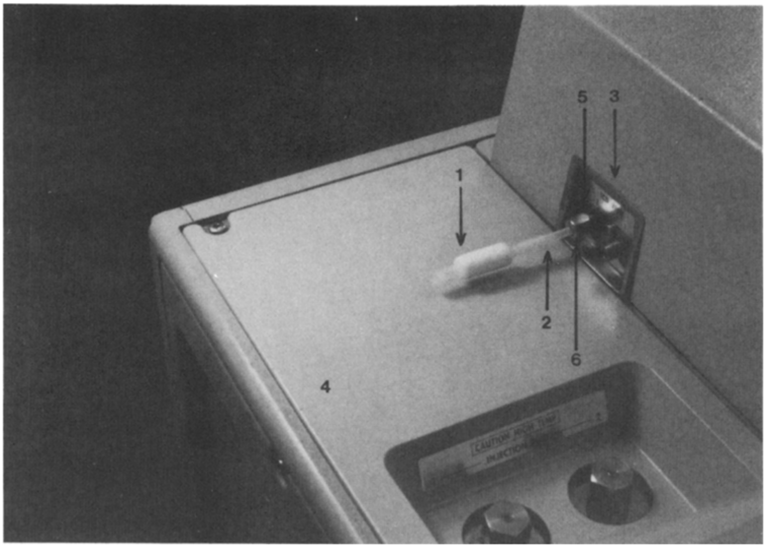

Fig. 1. Preparative GC collection device. 1 = Sep-Pak cartridge; 2 = PTFE tube (ca. 6 cm); 3 = GC thermal conductivity detector outlet; 4 = gas chromatograph; 5 = screw-cap; 6 = silicone-rubber septum (inside the screw-cap).

collected sample and the corresponding standard to make a mixture for GC quantification. The area ratio of the two peaks and the number of moles of the internal standard were used to calculate the recovery of the sample.

TABLE II

RESULTS OF PREPARATIVE GC COLLECTION USING SEP-PAK CARTRIDGES

| Group[a] | Sep-Pak cartridge | Amount injected (mg) | Amount of internal standard (mg) | Calculated recovery (mg) | Recovery (%) |
|---|---|---|---|---|---|
| I | Silica | 13.1 | 7.9 | 12.6 | 96.18 |
| | $C_{18}$ | 10.6 | 6.6 | 10.1 | 95.25 |
| II | Silica | 11.2 | 8.6 | 10.8 | 96.43 |
| | $C_{18}$ | 10.2 | 8.6 | 9.7 | 95.10 |
| III | Silica | 9.7 | 10.9 | 9.1 | 93.81 |
| | $C_{18}$ | 9.4 | 10.7 | 8.5 | 90.43 |
| IV | Silica: | | | | |
| | Run 1 | 17.9 | 15.9 | 14.4 | 80.45 |
| | Run 2[b] | 9.8 | 6.6 | 8.2 | 83.67 |
| | $C_{18}$: | | | | |
| | Run 1 | 17.7 | 15.5 | 5.8 | 32.77 |
| | Run 2[b] | 8.4 | 9.7 | 6.5 | 77.38 |
| | Run 3[b,c] | 10.7 | 9.9 | 8.8 | 82.24 |

[a] See Table I.

[b] Both injector and detector temperature were lowered to 180°C.

[c] Two Sep-Pak cartridges were connected in parallel, but the yield was based on the ethyl acetate wash from the first cartridge only.

## RESULTS AND DISCUSSION

Except for α-pinene, the recoveries were all better than 90%, whichever cartridge (Si of $C_{18}$) was used. As shown in Table II, for the sesquiterpenes and monoterpenes tested, the silica Sep-Pak gave slightly better yields in all instances. For α-pinene, the low recovery from run 1 on the $C_{18}$ cartridge was improved considerably when both the injector and detector temperatures were lowered from 250 to 180°C (run 2) and improved still further when two cartridges were connected in parallel (run 3). In run 3, however, nothing could be washed out from the second cartridge, and the recovery was based on the ethyl acetate wash from the first cartridge only.

During nearly all collections, either white solid or oil droplets were observed in the PTFE tube near the cartridge end. In run 1 on the $C_{18}$ cartridge for α-pinene, however, nothing could be seen in the PTFE tube and the recovered material (32.77%) was obviously trapped in the cartridge. It seems that the Sep-Pak cartridge works mainly as a very efficient stopping device to slow the fast gas stream from the outlet of the gas chromatograph, but if this stopping function fails, the packing in the cartridge may also work as an adsorbent to trap the material. The lower recoveries for α-pinene illustrate that both the stopping and absorbing functions of the packing in the cartridge do not work as efficiently for small and non-polar molecules. However, adding a third cartridge in parallel may further improve the recovery for such compounds. The slightly better recovery with the silica cartridge in all instances is probably due to the stronger adsorbing property of silica particles.

In conclusion, for reasonably sized molecules ($\geqslant C_{10}$), Sep-Pak cartridges can be used for very convenient and efficient preparative GC collections.

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