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Note

Gas chromatography of drugs on a Carbowax 20M deactivated support

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Rapid and reliable identification of drugs in the crime and chemistry laboratory is commonly accomplished by gas chromatography (GC) [often coupled with mass spectrometry (GC–MS)]. The ideal chromatographic column would be capable of separating all of the drugs of interest in a reasonable length of time without decomposition of the drug or the column. Column decomposition during the analysis can lead to high levels of continuous contamination in the carrier gas making normal temperature programming and/or mass spectral interpretation difficult. Silicone phases such as OV-17 and SE-30 are most commonly used for drug analysis because of their temperature stability and their ability to deactivate many catalytic sites on support material. Recently, a chromatographic phase consisting of a very thin film of liquid coated on a commonly used support has been developed and appears promising as an ideal stationary phase for drug analysis.

GC on very thin (near monomolecular) liquid films was first introduced by Aue *et al.*[1], in 1973. They produced a 0.2% non-extractable film of Carbowax 20M on acid washed Chromosorb W by coating the Chromosorb with 6% Carbowax 20M and then heat treating this material at 260° for 16 h under nitrogen. This period of heat treatment presumably allows 0.2% of the Carbowax polymer to adsorb onto the surface of the support in the optimal configuration. Excess non-adsorbed polymer is removed from the material by an exhaustive Soxhlet extraction with methanol. The final product exhibited superior chromatographic properties of support deactivation, high resolution, and low bleed. Subsequent investigations produced a series of similar materials from Apiezon L, SE-30, AN-600, stabilized ethylene glycol adipatè, Dexsil 300, linear polyethylene, and linear Carbowax 20M[2]. These phases can also be used as deactivated supports for other liquid phases[3].

Primary test compounds used in the evaluation of the non-extractable thin liquid films were mixtures of *n*-alkanes and *n*-alcohols[1,2]. Separations of various sterols[3], free aldehydes[4], polynuclear hydrocarbons[4], chlorinated hydrocarbons[5], and organometallics[6] have also been reported. In all cases the chromatography appeared

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excellent. This phase is used on a routine basis to separate a complex mixture of organics extracted from air-borne particulate matter[7]. Although one would predict that these phases would be ideal for drug analysis, they have not been evaluated specifically for that purpose.

Ultra-Bond 20M (UB 20M) is a commercially available support material (RFR Corp., Hope, R. I., U.S.A.) similar to that prepared by Aue *et al.*[1]. Modified McReynolds constants of 121, 317, 172, 280, and 180 for benzene, butanol, 2-pentanone, nitropropane, and pyridine, respectively, have been reported[8]. If these values are compared with those of the two GC phases most commonly used for drug analysis, UB 20M is expected to be more polar than SE-30 (15, 53, 44, 64, 41) and OV-17 (119, 158, 162, 243, 202) but yet much less polar than normally prepared Carbowax 20M (322, 536, 368, 572, 510). Use of the McReynolds system to compare these modified supports to other coatings is hindered by the fact that comparisons must be made for different loads and that the modified McReynolds data were obtained at a temperature where UB 20M has been shown to be atypical[9]. It is therefore difficult to predict separation behavior of this modified support using the McReynolds system, and direct comparisons of the retention data of various drugs is necessary to evaluate the usefulness of this modified support in drug analysis.

This note reports relative retention data for a number of commonly analyzed drugs on a Carbowax 20M modified support and evaluates the use of this material as a support for traditional liquid phases of OV-17 and SE-30.

EXPERIMENTAL

Conditions

The gas chromatograph used throughout these experiments was a Hewlett-Packard 5830A with flame ionization detection and digitally timed retention data. The Hewlett-Packard 18850A GC terminal provided retention times matched to chromatographic peaks that were reproducible within 1%. The terminal also controlled and monitored the injection temperature at 250°, the oven temperature at either 200° or 230°, and the detector temperature at 300°. Flame ionization detector (FID) gas flow-rates were maintained at 250 ml/min for air and 30 ml/min for hydrogen.

Columns

Coiled glass columns (6 ft. × 6 mm O.D. × 2 mm I.D.) were used throughout this study. Each column was acid washed and treated with dichlorodimethylsilane before packing. Five stationary phases were compared: Ultra-bond II (UB II) 80–100 mesh (RFR Corp.), 3% OV-17 (Supelco, Bellefonte, Pa., U.S.A.) on UB II 80–100 mesh, 3% SE-30 (Supelco) on UB II 80–100 mesh, 3% OV-17 on Chromosorb W HP 80–100 mesh (Supelco) and 3% SE-30 on Chromosorb W HP 80–100 mesh (Supelco).

Ultra-Bond II (UB II) was used in this study as the Carbowax 20M modified stationary phase. It is produced similarly to UB 20M, but the specific modifications of the method are not available. According to RFR Corp., UB II is the same as UB 20M but with *ca.* 0.4% coating of Carbowax 20M[10].

Compounds

Amobarbital, pentobarbital, meperidine, procaine, methadone, cocaine, codeine, morphine, and heroin were chosen as test compounds in this study since they are representative of drugs often analyzed by GC and since they contain functional groups that require deactivated columns for optimum separation. Heroin was a U.S.P. reference standard. The other drugs were U.S.P. grade.

Approximately 10 mg/ml of each drug was isolated in an organic solvent for gas chromatography. Amobarbital and pentobarbital were extracted into chloroform from acid solutions. Meperidine, procaine, methadone, cocaine, codeine and heroin were extracted into chloroform from basic solutions, and morphine was extracted into a mixed solvent of chloroform–butanol from a basic solution. A mixture of procaine, methadone, cocaine, and heroin was prepared in addition to the individual standards.

RESULTS AND DISCUSSION

Retention data relative to cocaine for the various drug standards are reported in Table I. Absolute retention times may be calculated for other test compounds by multiplying their relative retention value by the retention time reported for cocaine.

TABLE I

RELATIVE RETENTION DATA

	3% SE-30 on Chromosorb W (200°)	*3% SE-30 on UB II (200°)*	*UB II (200°)*	*3% OV-17 on UB II (200°)*	*3% OV-17 on Chromosorb W (200°)*
Amobarbital	NR	0.73	1.36	0.39	0.13
Pentobarbital	0.25	0.80	1.46	0.43	0.15
Meperidine (Demerol)	0.26	NR	NR	0.14	0.14
Procaine	0.56	1.00	1.49	0.76	0.50
Methadone	0.88	0.66	0.46	0.55	0.59
Cocaine (standard)	6.03 min*	7.05 min*	7.22 min*	14.04 min*	25.52 min*
Codeine	1.71	2.46	3.01	2.30	1.99
Morphine	1.69	2.49	3.04	2.33	2.02
Heroin	3.76	5.81	7.63	4.92**	4.70**

* Absolute retention time.

** Column temperature = 230°.

From this table a number of observations can be made that help characterize the use of UB II as a chromatographic phase for drug separation. As expected, the order of elution of the nine drugs from SE-30 on Chromosorb W was amobarbital, pentobarbital, meperidine, procaine, methadone, cocaine, codeine, morphine, and heroin. With OV-17 on Chromosorb W, retention times were considerably longer but the elution pattern was the same except for the reversal of pentobarbital and meperidine. For procaine, cocaine, codeine, morphine, and heroin the columns increased in polarity, as indicated by retention data, in the following order: SE-30 on Chromosorb W, SE-30 on UB II, UB II, OV-17 on UB II, and OV-17 on Chromosorb W. The UB II phase selectively retarded the elution of amobarbital and pentobarbital when com-

pared to SE-30 and OV-17 but eluted meperidine and methadone more rapidly than did these silicone phases.

Figs. 1 and 2 illustrate the chromatography for a mixture of meperidine, methadone, cocaine, procaine, and heroin. In all five chromatograms the solvent does not appear as a peak because the attenuation was programmed high at the beginning of the run so that the response for the solvent peak would not peg the electrometer. In chromatograms 1a and 1b meperidine was not retained and eluted with the solvent. In chromatograms 1c and 2b, heroin did not elute within the time frame provided by the program.

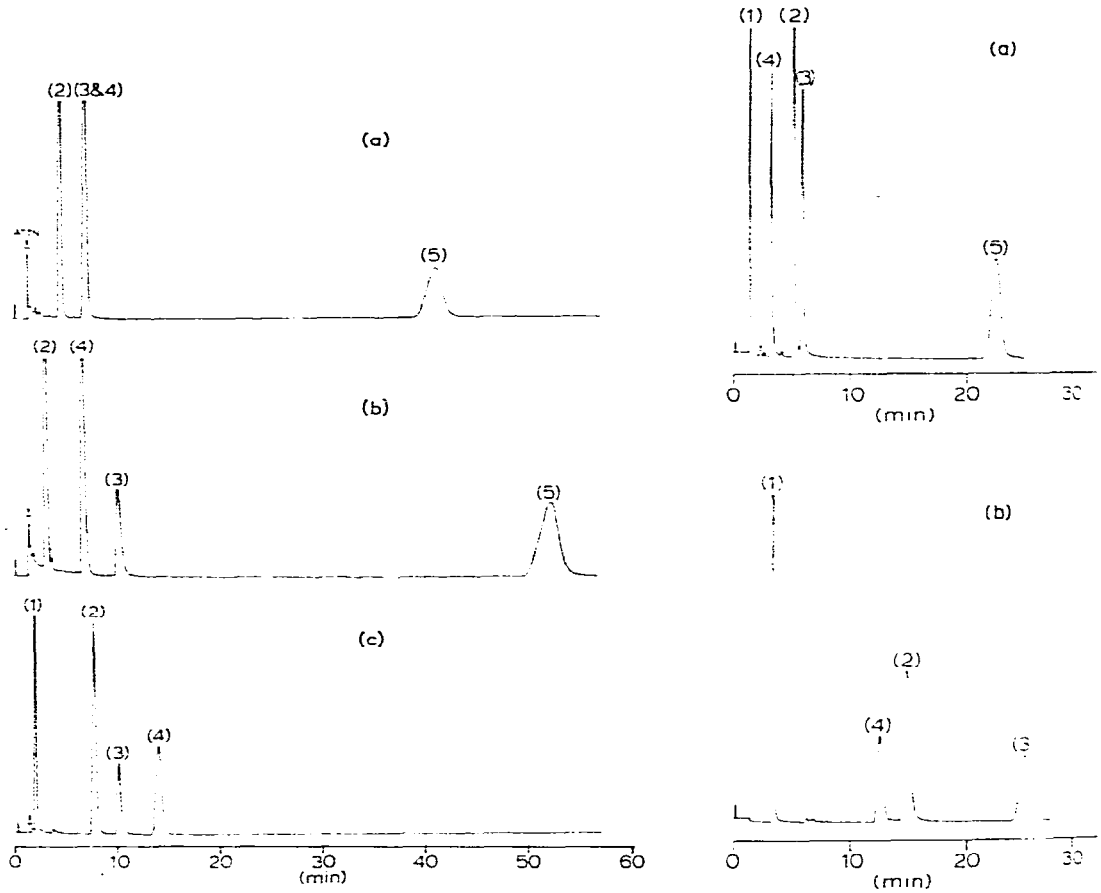

Fig. 1. Comparison of a drug mixture chromatographed under identical conditions on three different columns with UB II as the support material. (a) 3% SE-30 on UB II; (b) UB II; (c) 3% OV-17 on UB II. 1 = Meperidine; 2 = methadone; 3 = cocaine; 4 = procaine; 5 = heroin.

Fig. 2. Comparison of the drug mixture used in Fig. 1 but chromatographed on standard GC phases. (a) 3% SE-30 on Chromosorb W HP; (b) 3% OV-17 on Chromosorb W HP.

Deactivation of support material has always been a major concern when preparing chromatographic phases. This is especially true in drug analysis when compounds chromatographed contain active functional groups that can interact with the

support as well as the liquid coating. The symmetrical peaks in chromatograms 1a, 1b, and 1c indicate that there is little interaction between drugs and active sites.

UB II and Chromosorb W were used as support materials for SE-30 and OV-17 liquid phases to compare performance of the deactivated supports. Completely deactivated supports are believed to have no affect on separation patterns which are determined solely from the characteristics of the outer liquid phase[3]. In this study significant differences for relative retention data were observed between SE-30 coated on Chromosorb W and SE-30 on UB-II and between OV-17 on Chromosorb W and OV-17 on UB-II. Yet, efficiencies of all columns were moderately good, ranging between 600 and 700 plates per foot, with apparently little interaction from active sites on the support material. One explanation for this unexpected variation in retention pattern may be that all of the Carbowax 20M on the UB-II is not adsorbed on the surface and is free to change the chromatographic characteristics by dissolving into the silicone phase.

No distinct advantages (or disadvantages) in efficiency were noted for the commercially prepared UB-II phase compared to the traditional drug separation material, but the novel phase does offer an additional choice in chromatographic selectivity which is often of considerable importance when developing a specific analytical procedure. For drug analysis, UB-II may be considered a compromise between SE-30 and OV-17 with greater selectivity than SE-30 but less retention than OV-17.

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