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Note

Gas chromatographic separation of allylbarbital and butabarbital

N. C. JAIN*

Departments of Pharmacology and of Community Medicine and Public Health, University of Southern California School of Medicine, Los Angeles, Calif., and Rancho Los Amigos Hospital, 7601 E. Imperial Highway, Downey, Calif. 90242 (U.S.A.)

and

T. SNEATH, R. BUDD, D. CHINN, W. LEUNG and B. OLSON

Rancho Los Amigos Hospital, 7601 E. Imperial Highway, Downey, Calif. 90242 (U.S.A.)

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Allylbarbital (5-allyl-5-isobutylbarbituric acid) and butabarbital (5-ethyl-5-*sec.*-butylbarbituric acid) are among the commonly abused barbiturates. The gas–liquid chromatographic (GLC) columns used in many toxicology laboratories do not differentiate between these two barbiturates either as free drugs or as their methylated derivatives[1–5]. We have investigated a number of columns to obtain their best separation.

EQUIPMENT

A Varian gas chromatograph equipped with two columns, dual differential electrometer and two flame ionization detectors was used in this study. The columns used were 6 ft. × 2 mm I.D. glass, packed with the various materials described.

RESULTS AND DISCUSSION

The results of our study are summarized in Table I. Data show that a number of columns commonly in use for free and methylated barbiturates[1–5] cannot differentiate between allylbarbital and butabarbital. Analysis procedures relying on these columns cannot possibly be accurate for the two drugs.

Of the columns tested for free acid barbiturates, only 4% OV-210 could separate free allylbarbital from butabarbital. Fig. 1 shows that this column also gives clear separation of other commonly used barbiturates such as amobarbital, pentobarbital, phenobarbital, secobarbital and ibomal, which was used as an internal standard.

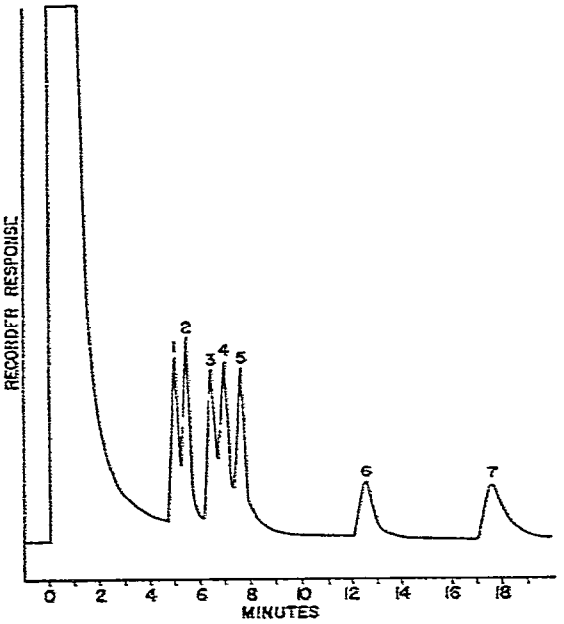
Four columns were found that could clearly separate methylated derivatives of allylbarbital and butabarbital: 3% OV-17, 3% OV-25, 4% OV-210, and 3% Poly-

* Reprint requests to: Dr. N. C. Jain, Rancho Los Amigos Hospital, 7601 E. Imperial Highway, Downey, Calif. 90242, U.S.A.

TABLE I
SEPARATION OF ALLYLBARBITAL AND BUTABARBITAL

Column	*Free acids**	*Methylated derivatives***
3% SE-30 on Chromosorb W, 80–100 mesh	no	no
3.8% UC W-98 on Chromosorb W, 80–100 mesh	no	no
10% UC W-98 on Chromosorb Q, 80–100 mesh	no	no
10% DC-200 on Chromosorb Q, 80–100 mesh	no	no
5% OV-1 on Chromosorb W, 80–100 mesh	no	no
3% SE-30–3% trimesic acid on Chromosorb W, 80–100 mesh	no	no
3% OV-17 on Chromosorb W, 80–100 mesh	no	yes
3% OV-25 on Chromosorb W, 80–100 mesh	no	yes
4% OV-210 on Chromosorb W, 80–100 mesh	yes	yes
3% Poly-A-103 on Chromosorb Q, 100–120 mesh	no	yes
5% QF-1 on Chromosorb W, 80–100 mesh	no	no
3% XE-60 on Chromosorb W, 80–100 mesh	no	no
5% Hi-Eff 8B on Chromosorb Q, 80–100 mesh	no	no
0.5% Versamide on Chromosorb W, 80–100 mesh	no	no
3% Poly-S-179 on Chromosorb Q, 100–120 mesh	no	no

* Column temperatures ranging from 165–210°.
** Column temperatures ranging from 140–190°.



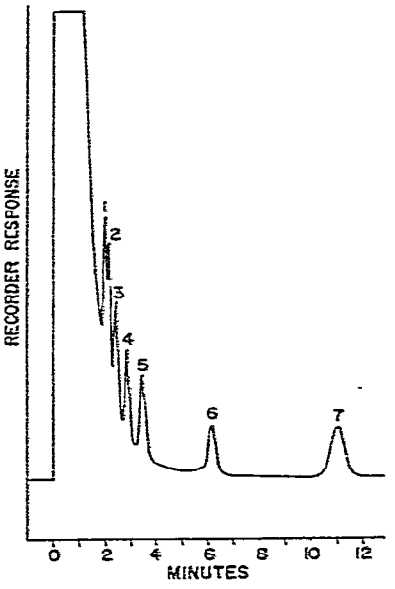


Fig. 1. Separation of barbiturates on 4% OV-210. Column, glass, 6 ft. × 2 mm I.D., column temperature, 180°; injector temperature, 220°; detector temperature, 230°; range, 10^{-11}; attenuation, ×32; injection, 1 μl containing 1 μg/ml each of (1) allylbarbital, (2) butabarbital, (3) amobarbital, (4) pentobarbital, (5) secobarbital, (6) ibomal (I.S.), and (7) phenobarbital.

Fig. 2. Separation of methylated barbiturates on 3% OV-17. Column, glass, 6 ft × 2 mm I.D.: column temperature, 175°; injector temperature, 250°; detector temperature, 260°; range, 10^{-11}; attenuation, ×32; injection, 1 μl containing 1 μg/ml each of (1) allylbarbital, (2) butabarbital, (3) amobarbital, (4) pentobarbital, (5) secobarbital, (6) ibomal (I.S.), and (7) phenobarbital.

TABLE II

RELATIVE RETENTION TIMES OF METHYLATED DERIVATIVES OF BARBITURATES
Column temperature, 185°; injector temperature, 225°; detector temperature, 250°.

Barbiturate	*3% OV-17*	*3% OV-25*	*3% Poly-A-103*	*4% OV-210*
Allylbarbital	0.35	0.32	0.30	0.42
Butabarbital	0.38	0.35	0.36	0.48
Amobarbital	0.42	0.38	0.37	0.56
Pentobarbital	0.50	0.44	0.45	0.61
Secobarbital	0.58	0.52	0.51	0.63
Ibomal	1.00	1.00	1.00	1.00
Phenobarbital	1.73	1.84	1.79	1.67
Retention time of ibomal, min	6.0	6.3	6.4	5.8

A-103. The relative retention times of common barbiturates on these columns are shown in Table II. The table illustrates that although 4% OV-210 and 3% Poly-A-103 give the greatest separation of allylbarbital and butabarbital, they do not give good separation of some of the other common barbiturates. Poly-A-103 does not clearly separate butabarbital from amobarbital and OV-210 does not separate pentobarbital from secobarbital. The best separation of all the common barbiturates is accomplished by OV-17, as shown in Fig. 2.

In conclusion, 4% OV-210 is found to separate most free barbiturates, including allylbarbital and butabarbital, whereas 3% OV-17 and 3% OV-25 are most suitable to distinguish their methylated derivatives.

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