

Temperature Programmed Drug Identification
(Prazepam - internal standard)

Introduction:

This gas chromatographic method is designed to help qualitate various basic drugs using a temperature program from 150° C to 270° C. The temperature program facilitates qualitation by reducing the time required to run samples at various temperatures.

References:

Wayne O. Pierce, Terry C. Lamoreauz, Francis M. Curry, Ladislav Kopjak, and Bryan S. Finkle. A new, rapid gas chromatography method for the detection of basic drugs in postmortem blood, using a nitrogen phosphorus detector. part 1 qualitative analysis, Journal of Analytical Toxicology 2:26-31, (1978).

Materials and Methods:

1. Gas chromatograph fitted with dual NP detectors.
Columns: 4' X 1/8" i.d. glass column containing 3% OV-17 on Chromosorb W, 100-120 mesh, 4' X 1/8" i.d. glass column containing 3% OV-1 on Chromosorb W, 100-120 mesh.
Operational Parameters: helium gas flow at a rate of 30 ml/min; hydrogen flow was at 3 ml/min and air flow at 50 ml/min.; inlet injection port temperature, 250° C; detector temperature, 300° C.
Temperature Programs were as follows:

	3% OV-17	3% OV-1
Initial Temp. (1)	150° C	150° C
Holding time at temp. (1)	2 min.	2 min.
Program rate	10° C/min.	10° C/min. 8° C/min.
Final Temp. (2)	270° C	270° C
Holding time at temp. (2)	12 min.	8 min.

2. Prazepam-Internal Standard.

Stock standard was made by dissolving the appropriate amount of the drug in methanol to make a solution equivalent to 1 mg/ml. The internal standard of 10 ug/ml was prepared by diluting 100 ul of stock solution to 10 ml with methanol.

Sample Preparation:

1. Prepare the basic urine extraction as outlined in Basic Urine Extraction Method. Extract a quality control sample with each batch of samples to which a 20 ul aliquot of the internal standard solution has been added.
2. Add 50 ul methanol to the final extraction and inject a 3.0 ul aliquot into the gas chromatograph.

3. Compare the unknown chromatograms with the relative retention values established.

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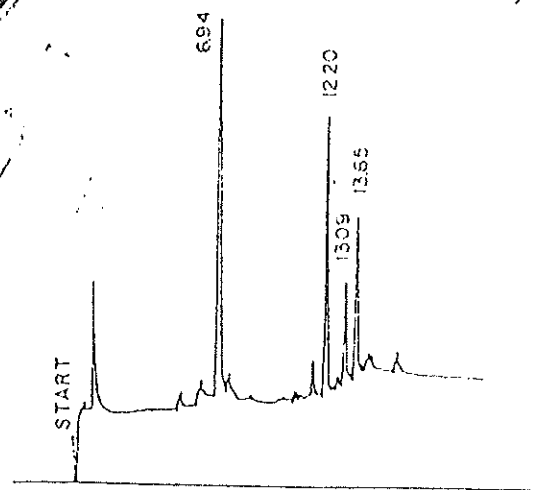


Figure 10. Chromatogram of actual forensic blood specimen analyzed by method: 6.94 min, caffeine; 12.20 min, diazepam; 13.09 min, desmethyldiazepam; 13.65 min, prazepam internal standard at 200 ng/ml.

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Table II. Relative Retention Times of Some Common Drugs. Reference Internal Standard: Prazepam at 13.78 min on 3% OV-17, and 12.85 min on 3% OV-1

DRUG	Rel. Rt. Time 3% OV-1	Rel. Rt. Time 3% OV-17	DRUG	Rel. Rt. Time 3% OV-1	Rel. Rt. Time 3% OV-17
Aminopyrine	0.48	0.54	Diazepam	0.86	0.89
Amiriptryline	0.70	0.76	Diethylarpton	0.14	0.11
Anileridine	1.14	1.15	Diethyltryptamine	0.47	0.44
Atropine	0.70	0.70	Dimethyl tryptamine	0.37	0.41
Benzphetamine	0.41	0.39	Biphenhydramine	0.44	0.49
Benztropine	0.79	0.76	Diphenoxylate	1.67	--
Bexrodiphenhydramine	0.67	0.65	Doxepin	0.72	0.71
Bromocetramine	0.63	0.62	Doxepan	1.14	1.16
Bucizine	1.40	1.49	Ephedrine	0.03	0.03
Bupivacaine	0.77	0.72	Etizolamiprine	0.84	0.86
Caffeine	0.40	0.51	Fenacetamide	0.46	0.40
Chlorazepoxide	1.11 (0.92)	0.77	Flunitrazepam	0.97	1.01
Chlormezanone	0.69	0.73	Flurazepam	1.09	1.04
2-Chloroprocaine	0.72	0.76	Haloperidol	1.18	1.19
Chlorpheniramine	0.56	0.54	Hydroxyzine	1.21	1.12
Chlorpromazine	0.86	0.88	Hyoscyamine	0.71	0.70
Clenzole	0.99	1.01	Imipramine	0.70	0.70
Cocaine	0.69	0.72	Ketamine	0.41	0.46
Codeine	0.81	0.85	Lidocaine	0.44	0.45
Cyclometnycalne	1.04	0.99	Luzepam	0.90	0.95
Demoxepam	0.91	0.96	Meclozine	1.24	1.21
Desalkylflurazepam	0.87	0.93	Mephazepam	0.72	0.75
Desipramine	0.74	0.76	Meperidine	0.33	0.34
Desmethyldiazepam	0.93	0.96	Meprobamate	0.58	0.61
Desmethyldiazepam	0.77	0.82	Mesoridazine	1.54	--
Dextromethorphan	0.64	0.63	Methadone	0.67	0.63
			Methylphenidate	0.59	0.55
			Mebutolone	0.66	0.71
			Methoxyphenamine	0.09	0.08
			Methylchlorazepam	1.06	1.11
			Methylflurazepam	1.01	1.05
			Miltirone	0.91	0.93
			Narcotine	1.31	1.64
			Nitrazepam	1.09a	1.16a
			Narcoproxyphene	0.83 (0.84)	0.83 (0.85)
			Narcoproxyphene Amide	0.54	0.54
			Nortriptyline	0.72	0.70
			Orphenadrine	0.49	0.49
			Oxazepam	0.91	0.88
			Papaverine	1.11	1.19
			Pentazocine	0.77	0.73
			Phencyclidine	0.46	0.41
			Phendimetrazine	0.33	0.11
			Phenindamine	0.66	0.69
			Pheniramine	0.38	0.41
			Phenretazine	0.12	0.12
			Phenyltolozamine	0.49	0.50
			Pilocarpine	0.66	0.70
			Procaine	0.56	0.61
			Proazirine	0.79	0.78
			Prochlorazine	0.74	0.74
			Propoxyphene	0.69	0.65

DRUG	Ret. Rt. Time 31 UV-1	Ret. Rt. Time 31 UV-17
Protriptyline	0.74a	0.73a
Quinidine	1.09	1.11
Quinine	1.09	1.11
SKF-525A	0.80	0.74
Strychnine	1.29	1.55
Tetracaine	0.73	0.72
Thenylidamine	0.53	0.56
Thioethylperazine	1.41	1.53
Thuridazine	1.30	1.33
Trifluoperazine	1.02	0.95
Trihexyphenidyl	0.73	0.68
Trimecaine	0.54	0.52
Tripelennamine	0.54	0.54

a = Tailing

References

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