Karl Pfleger/Hans Maurer/Armin Weber

Mass Spectral and GC Data of Drugs, Poisons and Their Metabolites

Part I

Introduction, Tables, GC Data

VCH

Karl Pfleger/Hans Maurer/Armin Weber

Mass Spectral and GC Data of Drugs, Poisons and Their Metabolites

Part II

Mass Spectra and Indexes

VCH

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First Edition 1985

Editorial Director: Dr. Hans F. Ebel

Library of Congress Card No. 85-11282

Deutsche Bibliothek Cataloguing-in-Publication Data

Pfleger, Karl: Mass spectral and GC data of drugs, poisons and their metabolites / Karl Pfleger; Hans Maurer; Armin Weber. – Weinheim; Deerfield Beach, Fl.: VCH
ISBN 3-527-26303-9 (Weinheim)
ISBN 0-89573-430-3 (Deerfield Beach, Fl.)

NE: Maurer, Hans:; Weber, Armin:

Pt. 2. Mass spectra and indexes. – 1985.

Composition and Printing: Zechnersche Buchdruckerei, D-6720 Speyer
Bookbinding: Wilhelm Osswald + Co., D-6730 Neustadt/Weinstraße

Printed in the Federal Republic of Germany

Distribution:
VCH Verlagsgesellschaft, P.O. Box 1260/1280, D-6940 Weinheim (Federal Republic of Germany)
USA and Canada: VCH Publishers, 303 N.W. 12th Avenue, Deerfield Beach FL 33442-1705 (USA)

ISBN 3-527-26303-9 (VCH Verlagsgesellschaft)
ISBN 0-89573-430-3 (VCH Publishers)

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1 Explanatory Notes

1.1 Arrangement of Spectra

Part 2 contains one thousand five hundred and fifty different mass spectra. They are arranged primarily in ascending mass of typical fragment ions, whose accurate masses are rounded off to the nearest integer. For each nominal mass value the spectra of underivatized compounds come first and are followed by those of derivatives both in order of ascending retention index.

Various criteria have been selected to aid the search for a spectrum. Because the molecular ion (M^+) normally contains the most important information the spectrum can be found under it. But in many cases the M^+ is too low or hidden by the background so that it cannot be detected. Therefore in many cases the spectrum can also be found under the next highest predominant ion. In order to avoid accumulation of data these ions were chosen sparingly. Finally the spectrum can be found under the base peak. If there are two or more large fragment ions (>80%), the spectrum can be found under both, because it is possible that their relationship could vary (7.4). Hence the reference spectra are reproduced more than once. The ion under which the spectrum is arranged on a particular page is underlined.

The search for reference spectra is illustrated in the example in 2.4.4 in Part 1.

1.2 Lay-out of Spectra

For easier visualization of the data the mass spectra are presented as bar graphs, in which the abscissa represents the mass to charge ratio (*m/z*) in atomic mass units (AMU) and the ordinate indicates the relative intensities of the ion currents of the various fragment ions. Predominant ions are labelled with their *m/z* value. The ion under which the spectrum is arranged on a particular page is underlined.

Some spectra are expanded so that molecular ions with a relative intensity of less than 1% are shown. In our experience the detection of these low intensity M^+ is often necessary for the identification of the compound, when the other fragment ions are not typical. In these cases the unknown spectra should be expanded by the data system. Fig. 1-1 explains the information given with each spectrum and the abbreviations used are listed in Table 2-1.

Compound name: The international non proprietary names for drugs (INN), the common names for pesticides and the chemical names for chemicals were used. If necessary, a synonym index (e.g. ABDA, 1984; Negwer, 1978; Perkow, 1983; Windholz et al., 1983) should be used. Additional information from the CAS is accessible through the list of common names (Part 2, 5.1). If the compound is a common metabolite or derivative of several parent compounds all parent compounds were given.

Structure: The formulas were plotted by a computer plotter to fit the available space in the spectrum.
Formulas of metabolites or artifacts are those of their probable structures (3, 4 in Part 1).

Empirical Form.: The empirical formulas are given to facilitate the identification of new metabolites or derivatives.

Molecular mass: The molecular masses were calculated from the atomic masses of the most abundant isotopes (5 in Part 1).

RI: The retention indices (RI) were measured on OV-101 in a temperature program (2.3.1 in Part 1).

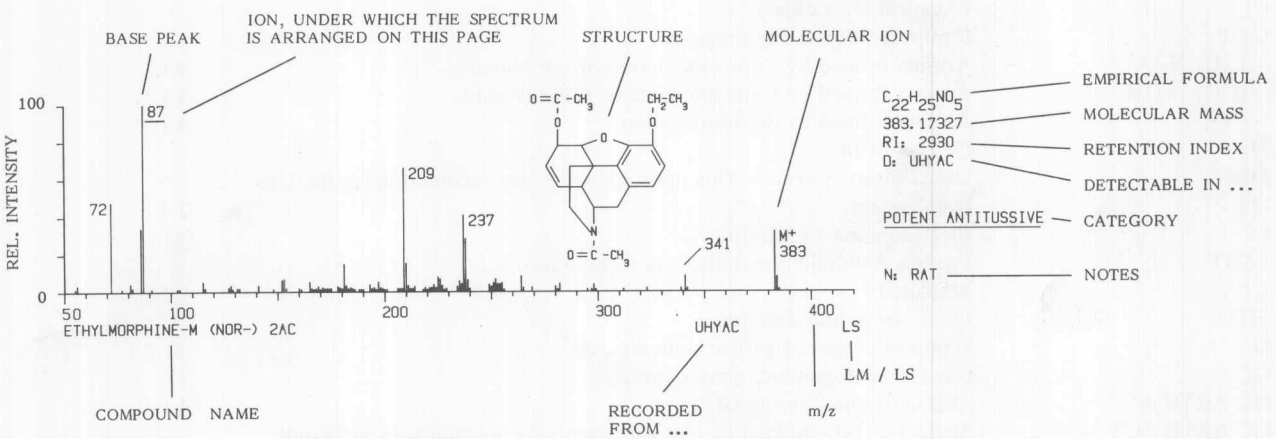


Fig. 1-1: Sample spectrum with explanations

The RI's of metabolites were determined by comparing the gas chromatogram with the mass chromatogram. The RI's of compounds with an asterisk (*) are not detectable by N-FID and FID must be employed.

D: The compound can be detected (D) in the given samples (cf. abbreviations in 2). These data will be completed.

Category: The major category is given.

N: If necessary notes (N) were added (cf. abbreviations in 2).

LM or LS: This indicates whether the low resolution mass spectrum (LM) was background subtracted (LS).
The relative ion intensities can be altered by background subtraction. This should be taken into account when comparing the spectra. Such variations does not compromise the use of the library in our experience. With experience it is possible to decide whether the variation is acceptable within two spectra of the same compound. If in doubt investigators should record a reference spectrum of the suspected compound on their own GC-MS.

Recorded from: A statement of the type of sample from which the spectrum was recorded (cf. abbreviations in 2).
If the spectrum was recorded from samples of biological origin, it should be remembered that fragment ions from sample impurities may be present in the spectrum.
With experience it is possible to decide whether these ions can be ignored.

2 Abbreviations

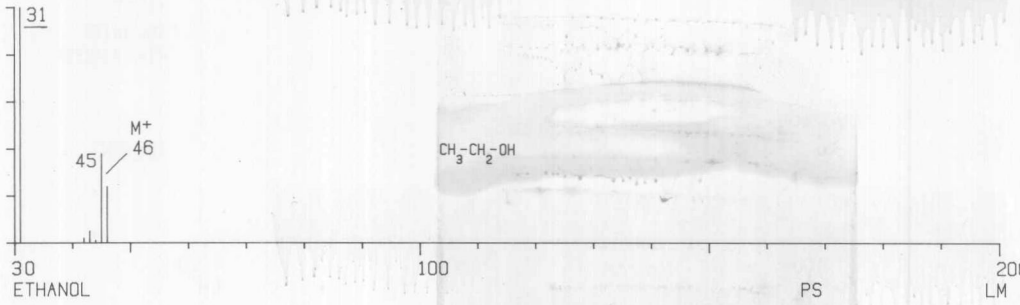
The abbreviations used in this book are listed in Table 2-1.

Tab. 2-1: Abbreviations

Abbreviation	Meaning	see
AC	Acetylated	2.2.3.1
(AC)	Possibly acetylated	
ALTERED DURING HY	The altered compound can be detected in UHY	4.3
AMU	Atomic mass unit = 1/12 of the mass of the ^{12}C isotope	
ARTIFACT ( )	( ) artifact	4
BP	Base peak = The most intense fragment ion in a mass spectrum	
CAS	Chemical Abstract Service	
CI	Chemical ionization	
CMP	Computer monitoring program	2.4.3
$-C_6H_{14}N_2O$	Artifact formed by Cope elimination of the N-oxide	4.1.3
$-(CH_3)_2NOH$	Artifact formed by Cope elimination of the N-oxide	4.1.3
$-CO_2$	Artifact formed by decarboxylation	4.1.1
D:	Detectable in	
DIS	Direct insert system = This mass spectrum was recorded using the DIS	
DS	Data system	2.4.1
EI	Electron impact ionization	3.1
EMIT	Enzyme multiplied immunoassay technique	
ET	Ethylated	2.2.3.3
FID	Flame-ionisation detector	2.3.1
G	Standard extract of gastric contents	2.2.2.1
GC	Gas chromatographic, -graph, -graphy	2.3
GC ARTIFACT	Artifact formed during GC	4.1
GC ARTIFACT IN METHANOL	Artifact of beta-adrenergic blocking agents by reaction with methanol during GC	4.1.4

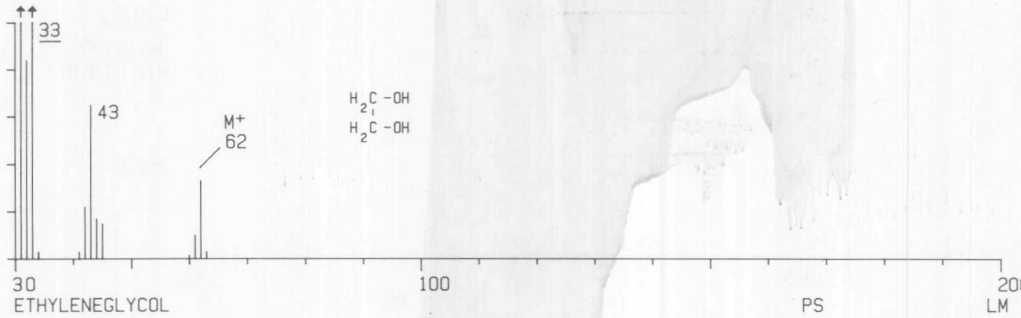
Abbreviation	Meaning	see
$+H_2O$	Artifact formed by hydration of an alkene	4.3.5
$-H_2O$	Artifact formed by dehydration of an alcohol or	4.2.1
	by rearrangement of an amino oxo compound	4.1.5
HPLC	High performance liquid chromatographic, -graph, -graphy	
HY	Acid hydrolyzed or acid hydrolysis	2.2.2.3
HY ARTIFACT	Artifact formed during acid hydrolysis	4.3
– I	Intoxication = This compound is detectable after a toxic dosage	
I. D.	Internal diameter	
INN	International non proprietary name (WHO)	
LM	Low resolution mass spectrum	
LS	Background substracted low resolution mass spectrum	
M^+	Molecular ion	
– M	Metabolite	
– M ( )	( ) metabolite	
– M (HO-)	Hydroxy metabolite	
– M (HOOC-)	Carboxylated metabolite	
– M (NOR-)	N-desmethyl metabolite	
– M (RING)	Ring compound as metabolite e. g. of phenothiazines	
– M ARTIFACT	Artifact of a metabolite	
– M/ARTIFACT	Metabolite or artifact	
m/z	Mass to charge ratio	3.1
ME	Methylated	2.2.3.2
(ME)	Methylated by methanol during GC	4.1.2
ME IN METHANOL	Methylated by methanol during GC	4.1.2
MS	Mass spectrometric, -meter, -metry, mass spectrum	2.4
N:	Notes	
N-FID	Nitrogen-sensitive flame-ionisation detector	2.3.1
NOT DETECTABLE AFTER HY	This compound is destroyed during acid hydrolysis	
P	Standard extract of plasma	2.2.2.1
PC	Paper chromatography	
PS	Pure substance	
RAT	This compound was found in the urine of rats	2.1
RI	Retention index (Kovats, 1958) on OV-101	2.3.1
RIA	Radio immunoassay	
STED	Solvent transfer and evaporation device	2.2.1
TLC	Thin layer chromatography	
TMS	Trimethylsilylated	2.2.3.4
U	Standard extract of urine	2.2.2.1
UA	Extract of urine for detection of amphetamines	2.2.2.2
UGLUC	Extract of urine after cleavage of conjugates using glucuronidase and arylsulfatase	2.2.2.4
UHY	Extract of urine after acid hydrolysis	2.2.2.3
*	This compound contains no nitrogen. Therefore it cannot be detected by a N-FID but by a FID	
----	This RI was not determined	
OOOO	This compound was not volatile and could not be detected by GC	

3 Mass Spectra



C_2H_6O
46.04186
RI: <1000*

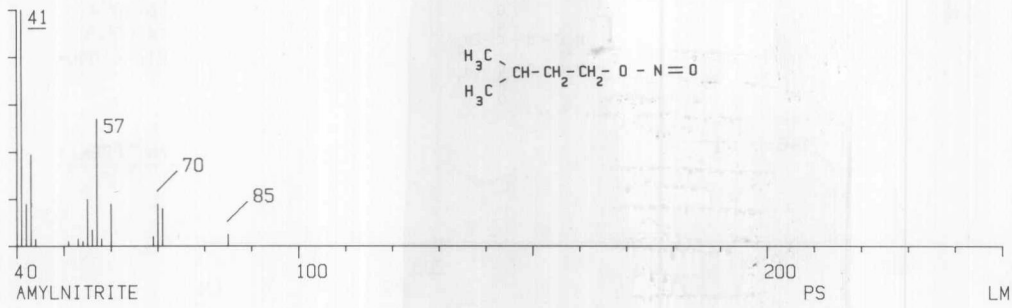
SOLVENT



$C_2H_6O_2$
62.03678
RI: <1000*

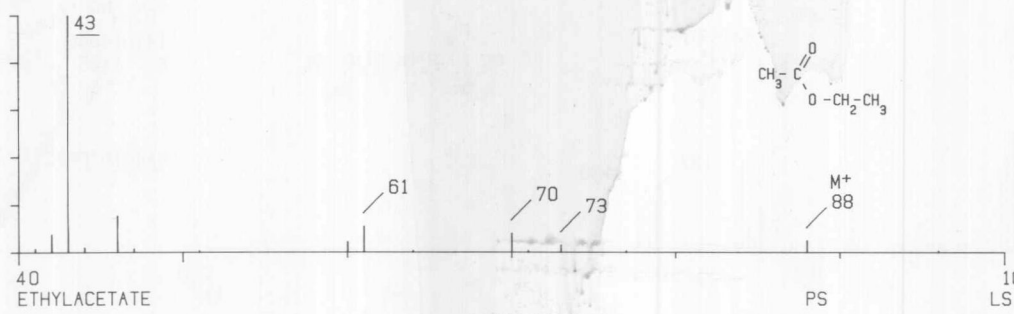
ANTIFREEZY

N: DIS



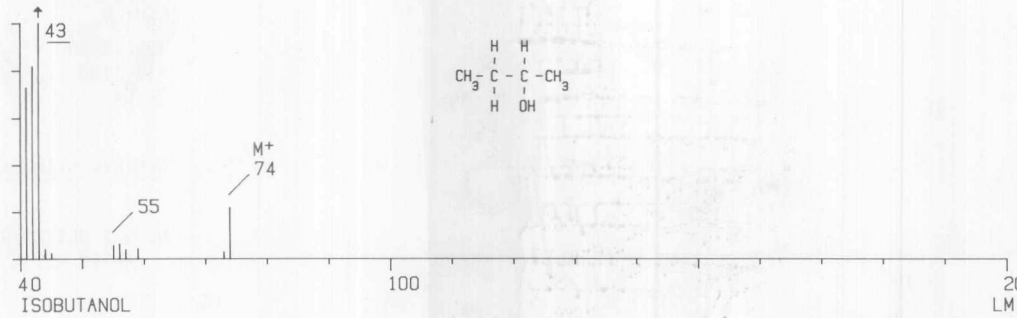
$C_5H_{11}NO_2$
117.07897
RI: <1000

CORON. DILAT.



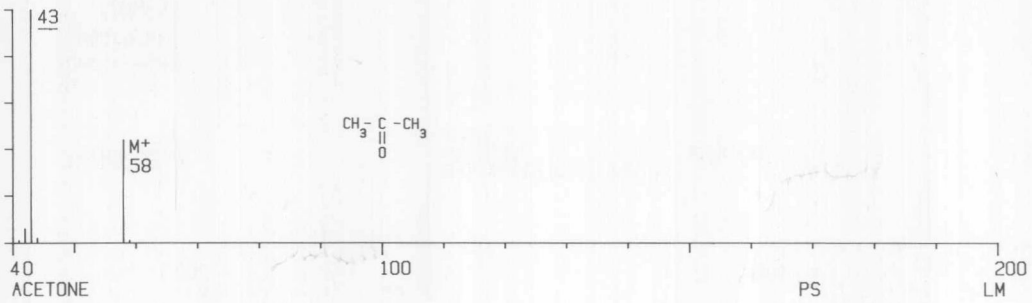
$C_4H_8O_2$
88.05243
RI: <1000*

SOLVENT



$C_4H_{10}O$
74.07316
RI: <1000*

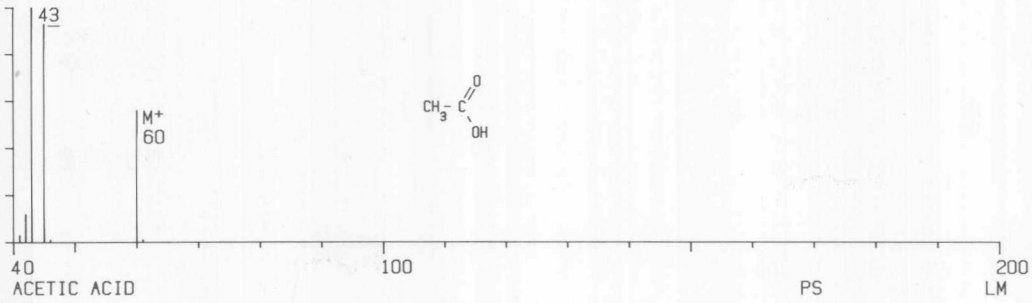
SOLVENT



ACETONE

C_3H_6O
58.04186
RI: <1000*

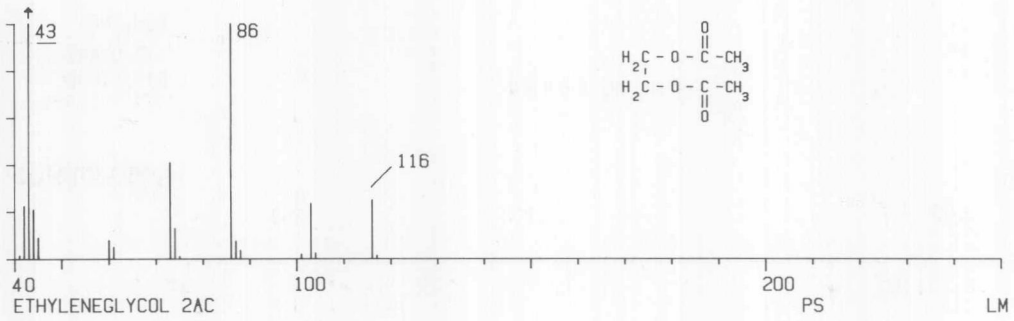
SOLVENT



ACETIC ACID

$C_2H_4O_2$
60.02113
RI: <1000*

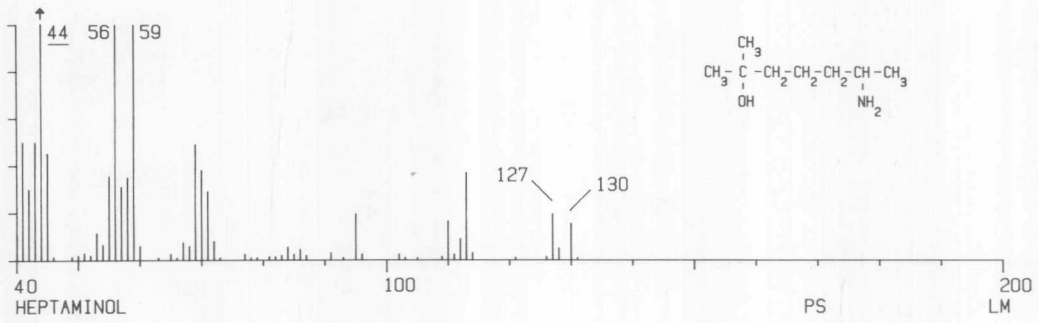
ACID



ETHYLENEGLYCOL 2AC

$C_6H_{10}O_4$
146.0579
RI: <1000*

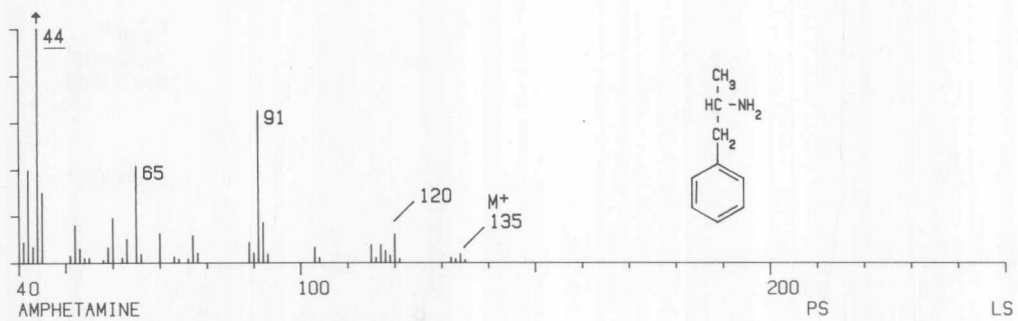
ANTIFREEZY



HEPTAMINOL

$C_8H_{19}NO$
145.14666
RI: 1125

CARDIOTONIC

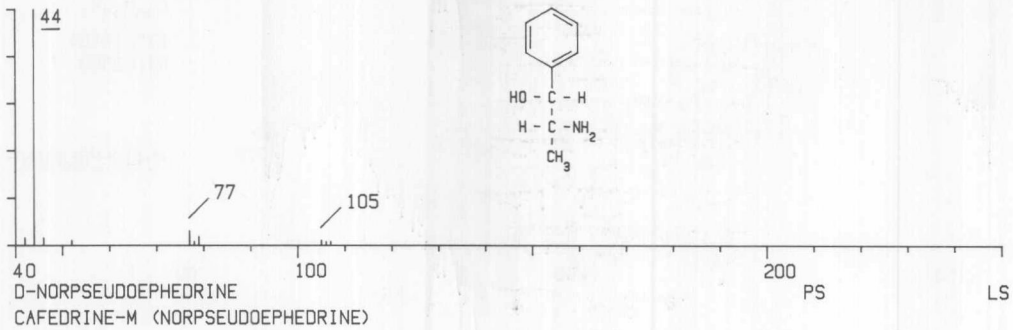


AMPHETAMINE
AMPHETAMINIL-M/ARTIFACT (AMPHETAMINE)
FENETHYLLINE-M (AMPHETAMINE)

$C_9H_{13}N$
135.10479
RI: 1160
D: UA

CENTRAL STIMULANT

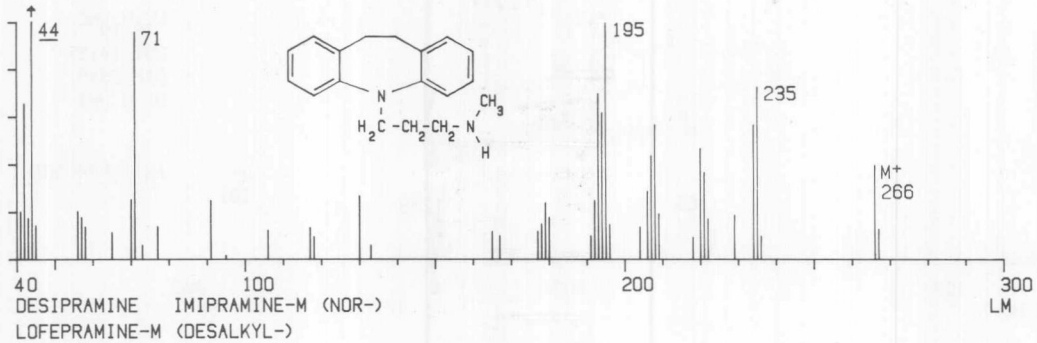
N: NOT DETECTABLE AFTER HY



D-NORPSEUDOEPHEDRINE
CAFEDRINE-M (NORPSEUDOEPHEDRINE)

$C_9H_{13}NO$
151.09971
RI: 1360
D: UA UHY

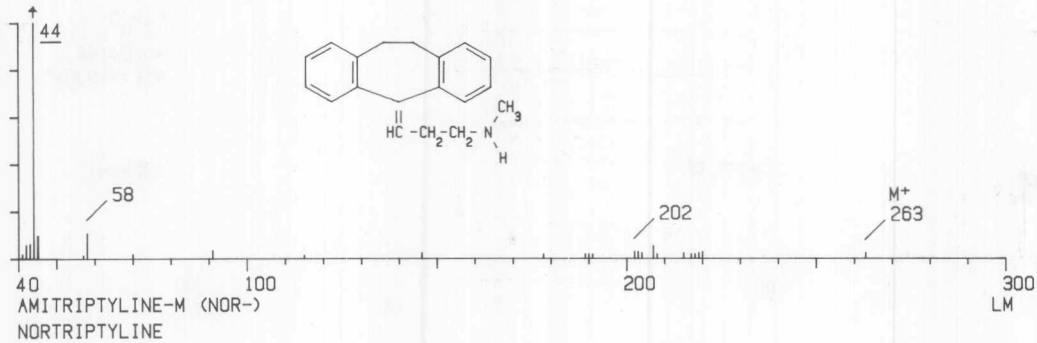
ANOREXIC



DESIPRAMINE IMIPRAMINE-M (NOR-)
LOFEPRAMINE-M (DESALKYL-)

$C_{18}H_{22}N_2$
266.17829
RI: 2225
D: UHY

ANTIDEPRESSANT



AMITRIPTYLINE-M (NOR-)
NORTRIPTYLINE

$C_{19}H_{21}N$
263.16739
RI: 2255
D: P U UHY

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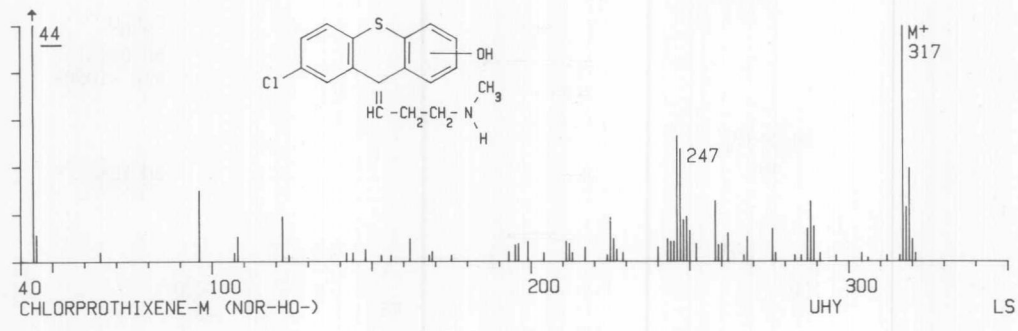
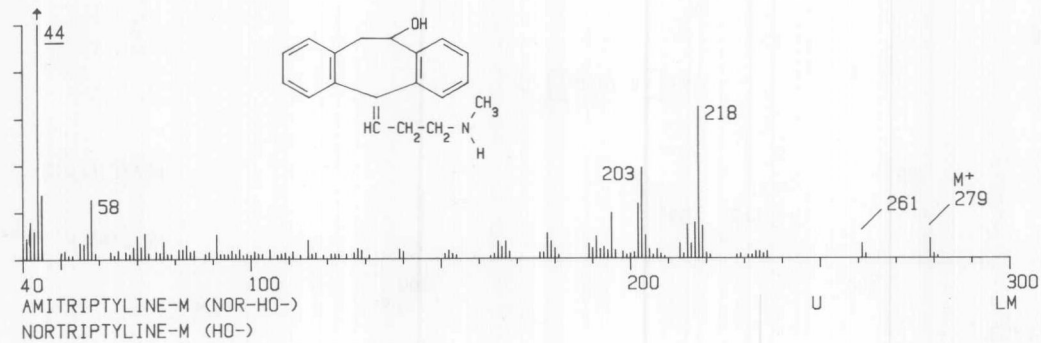


CHLORPROTHIXENE-M (NOR-HO-)

$C_{17}H_{16}NOCLS$
317.06411
RI: 2280
D: UHY

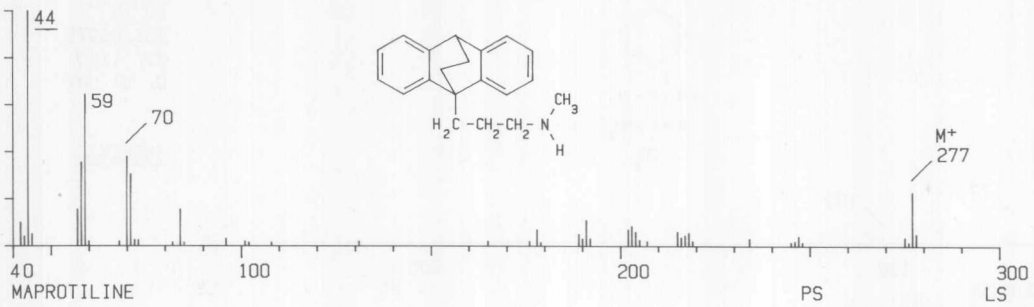
NEUROLEPTIC



AMITRIPTYLINE-M (NOR-HO-)
NORTRIPTYLINE-M (HO-)

$C_{19}H_{21}NO$
279.16231
RI: 2390
D: U-I UGLUC

ANTIDEPRESSANT



$C_{20}H_{23}N$
277.18304
RI: 2390

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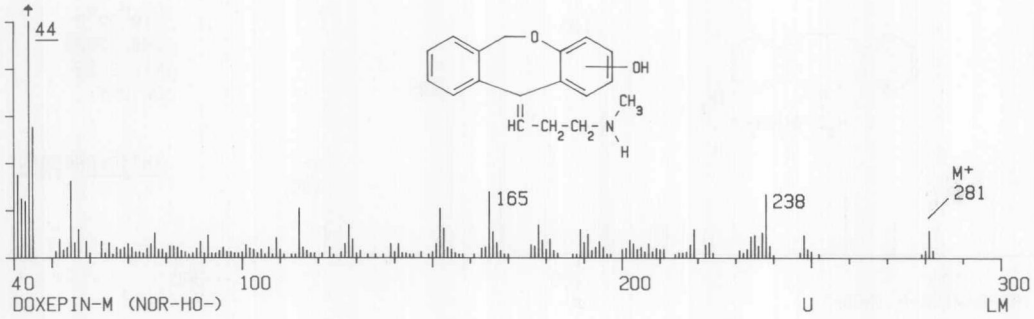

$C_{18}H_{19}NO_2$
281.14157
RI: 2540
D: U UHY

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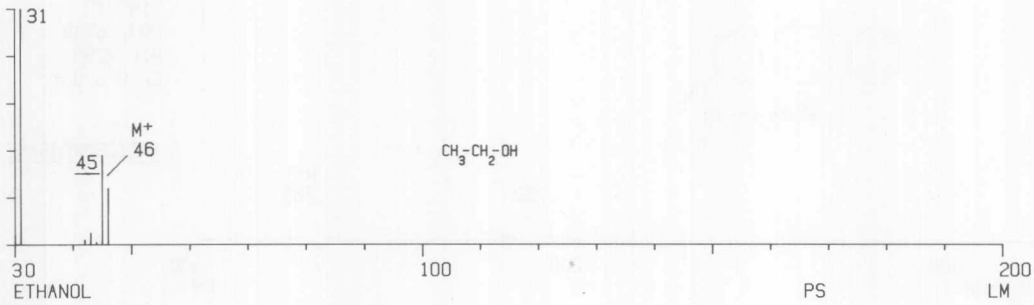
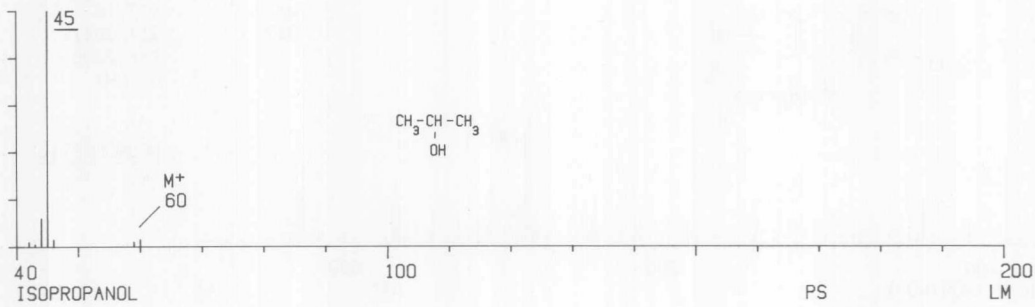


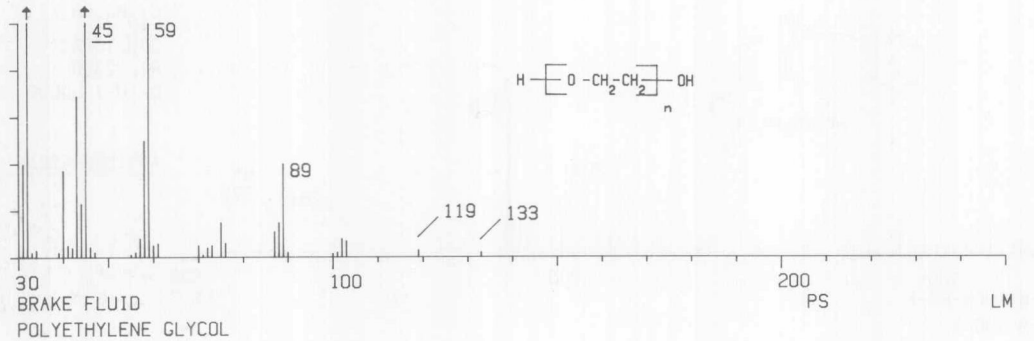
C_2H_6O
46.04186
RI: <1000*

SOLVENT



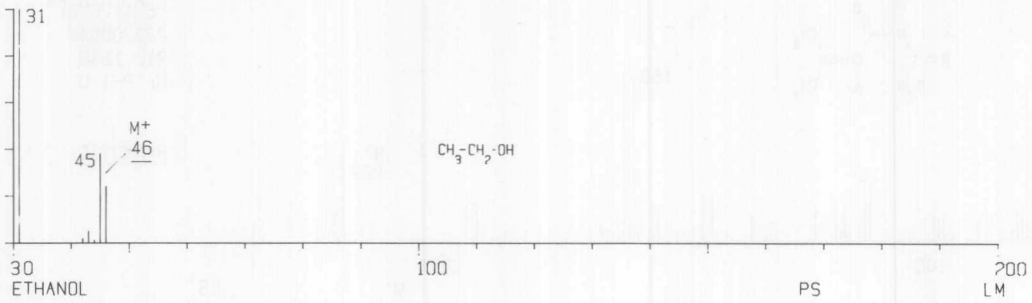
C_3H_8O
60.05751
RI: <1000*

SOLVENT



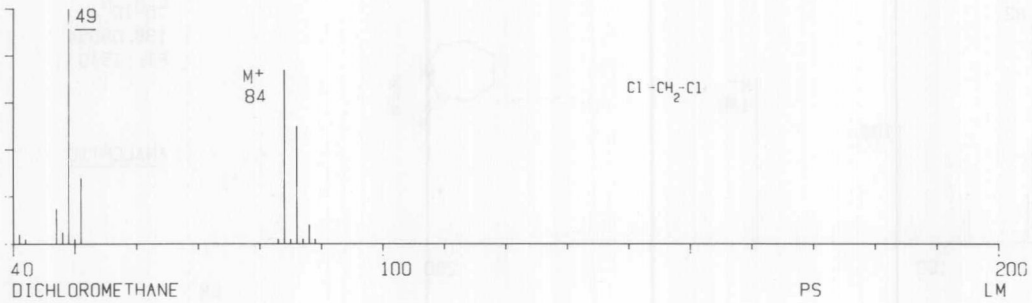
BRAKE FLUID

N: DIS



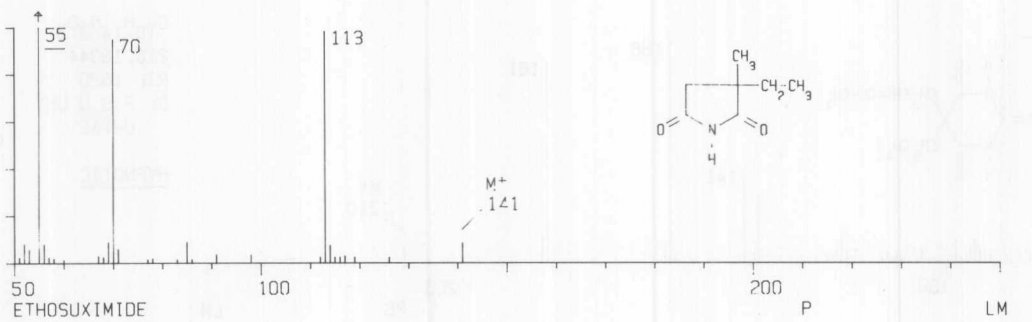
C_2H_6O
46.04186
RI: <1000*

SOLVENT



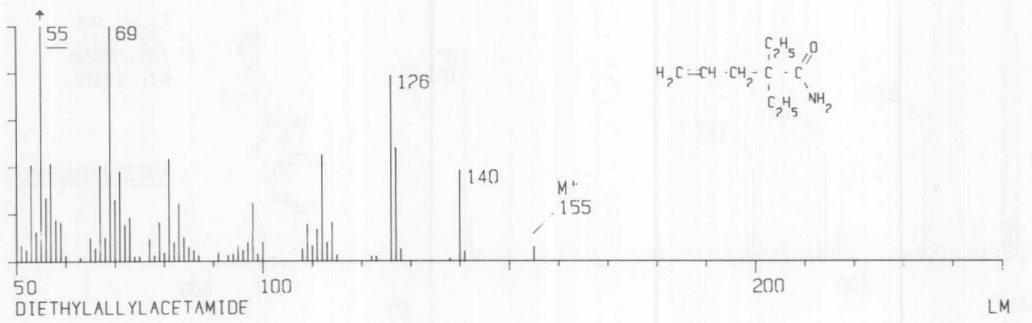
CH_2Cl_2
83.95335
RI: <1000*

SOLVENT



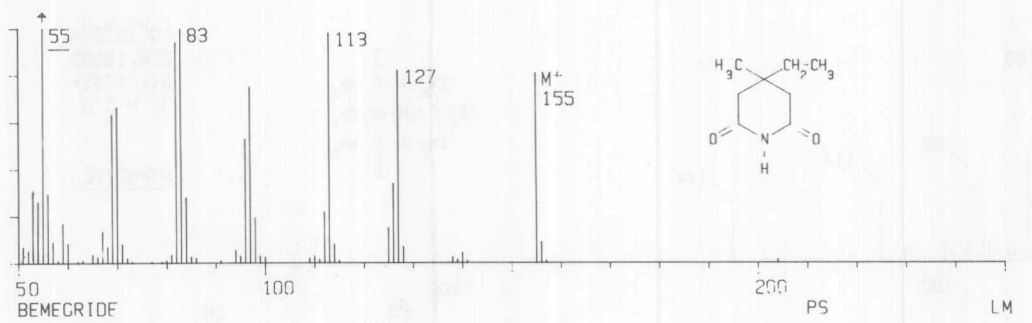
$C_7H_{11}NO_2$
141.07897
RI: 1225
D: P C U UHY
UHYAC

ANTICONVULSANT



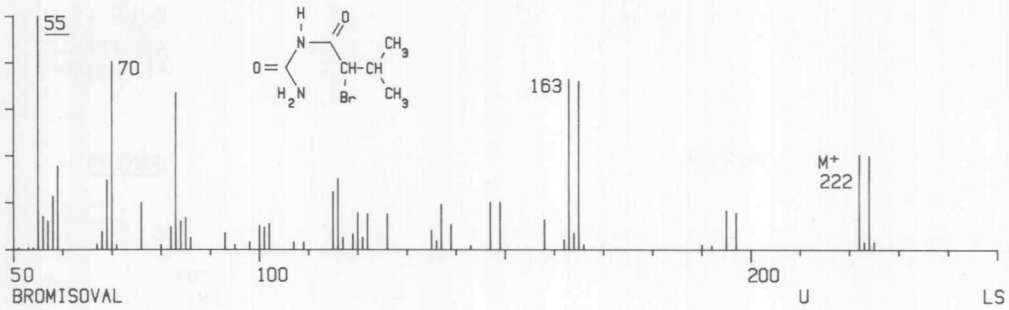
$C_9H_{17}NO$
155.13101
RI: 1285
D: P C U

HYPNOTIC



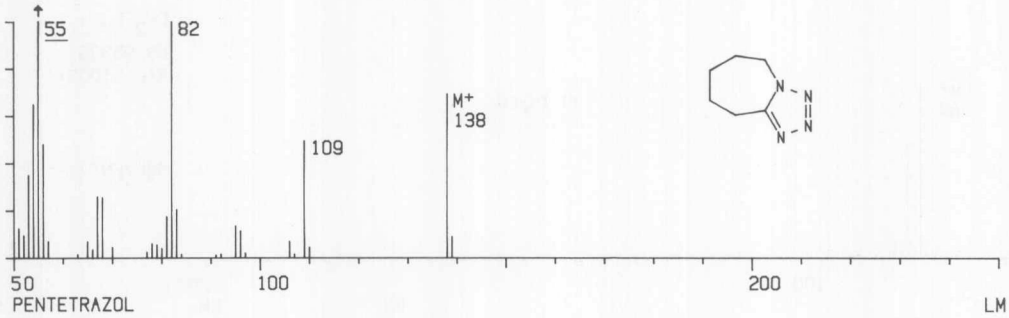
$C_8H_{13}NO_2$
155.09462
RI: 1350

ANALEPTIC



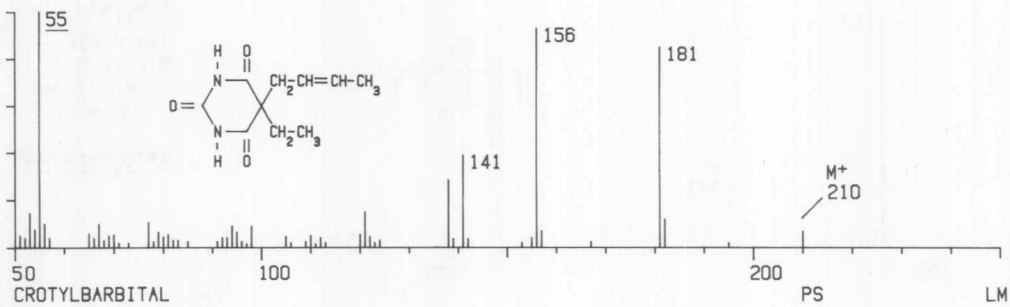
$C_6H_{11}N_2O_2BR$
222.0004
RI: 1540
D: P-I U

HYPNOTIC



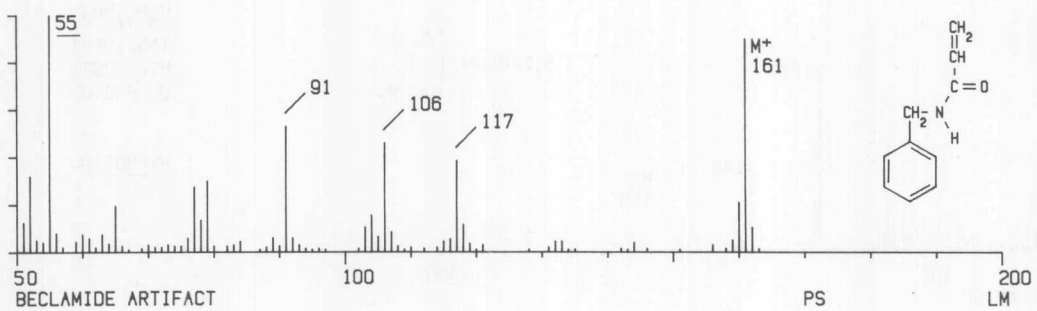
$C_6H_{10}N_4$
138.09054
RI: 1540

ANALEPTIC



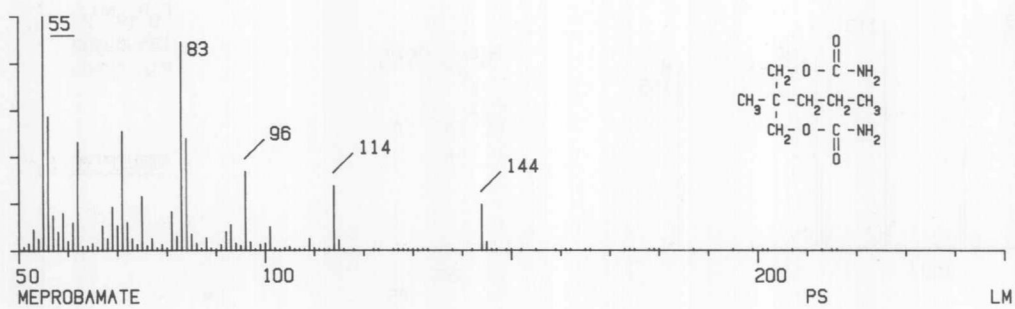
$C_{10}H_{14}N_2O_3$
210.10044
RI: 1620
D: P G U UHY
UHYAC

HYPNOTIC



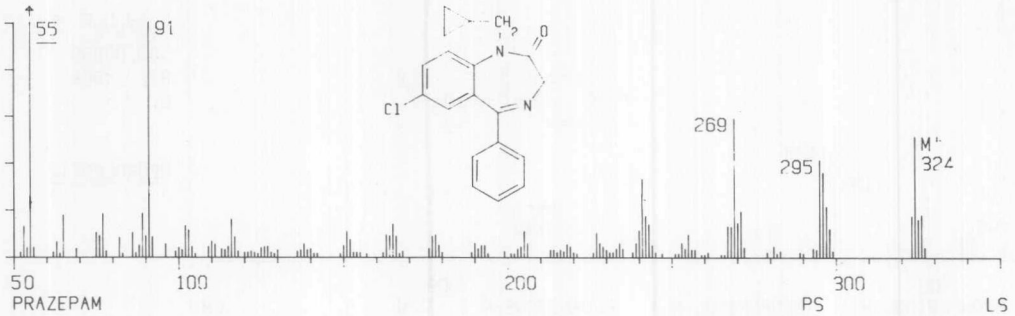
$C_{10}H_{11}NO$
161.08406
RI: 1680

ANTICONVULSANT



$C_9H_{18}N_2O_4$
218.12665
RI: 1785*
D: P G U

HYPNOTIC

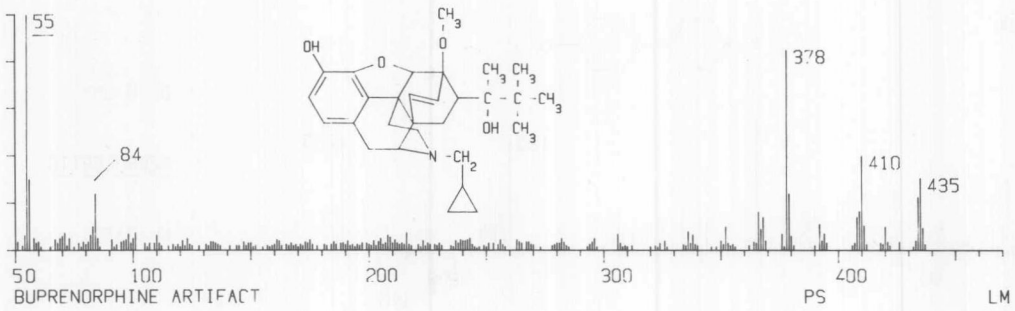


PRAZEPAM

$C_{19}H_{17}N_2OCL$
324.10294
RI: 2650
D: P-I C UGLUC
UGLUCAC

TRANQUILIZER

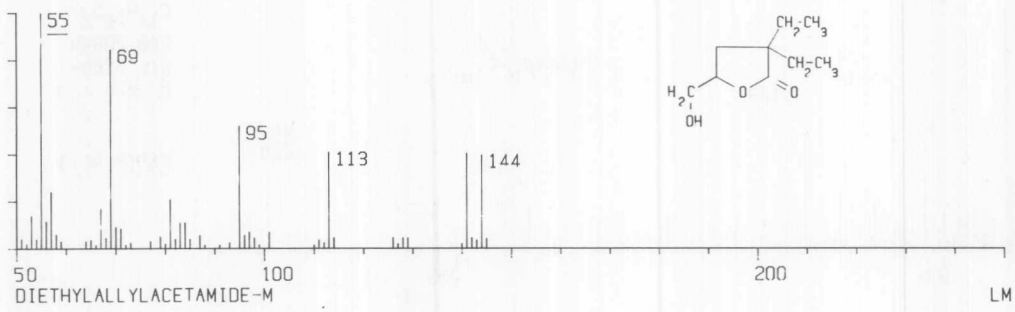
N: ALTERED DURING HY



BUPRENORPHINE ARTIFACT

$C_{29}H_{41}NO_4$
467.30355
RI: 3360

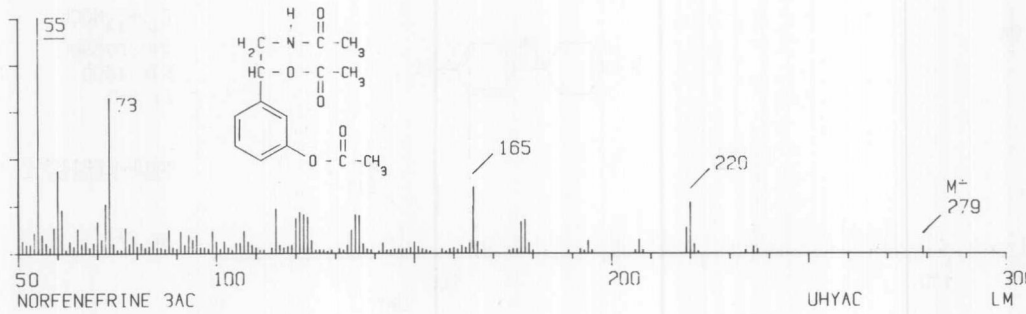
POTENT ANALGESIC



DIETHYLALLYLACETAMIDE-M

$C_9H_{16}O_3$
172.10994
RI: ----*

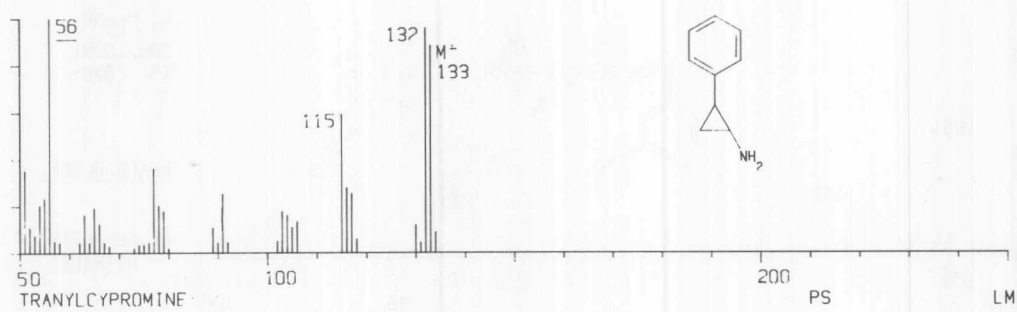
HYPNOTIC



NORFENEFRINE 3AC

$C_{14}H_{17}NO_5$
279.11067
RI: 2085
D: UHYAC

SYMPATHOMIMETIC



TRANYLCYPROMINE

$C_9H_{11}N$
133.08914
RI: 1230

ANTIDEPRESSANT