# *SPECTROSCOPIC CHARACTERIZATION OF A NEW HALLUCINOGEN : 1-(2,5-DIMETHOXY-4-NITROPHENYL)-2-AMINOPROPANE (DON).*

J.S.Gómez-Jeria [1*], B.K.Cassels [2], R.Clavijo [1], V.Vargas [1], R.Quintana [1] and J.C.Saavedra-Aguilar [3].

1. Departamento de Química, Facultad de Ciencias, Universidad de Chile. Casilla 653, Santiago, CHILE.
2. Université de Paris Sud, Faculté de Pharmacie, Laboratoire de Pharmacognosie, 92296 Chatenay-Malabry, FRANCE.
3. Departamento de Ciencias Neurológicas, Facultad de Medicina, Universidad de Chile, Santiago, CHILE.

* To whom all the correspondence should be addressed.

ABSTRACT.

We present the IR, UV, $^{1}H$- and $^{13}C$-NMR spectra of 1-(2,5-dimethoxy-4-nitrophenyl)-2-aminopropane (DON), whose hallucinogenic properties have been recently discovered. The data presented here should prove useful for the identification of DON in forensic specimens.

## INTRODUCTION.

1-(2,5-dimethoxy-4-nitrophenyl)-2-aminopropane (DON,Fig.1) is readily accessible by direct nitration of the photographic chemical 1-(2,5-dimethoxyphenyl)-2-aminopropane (2,5-DMA) (1). When this compound was firstly synthetized, it was found to be equipotent with the reference hallucinogen DOM (STP) in a simple rat behavioral assay (2), and it has also been shown to have a strong affinity for the rat stomach fundus serotonergic receptor, a model which correlates rather well with hallucinogenesis in humans (1).

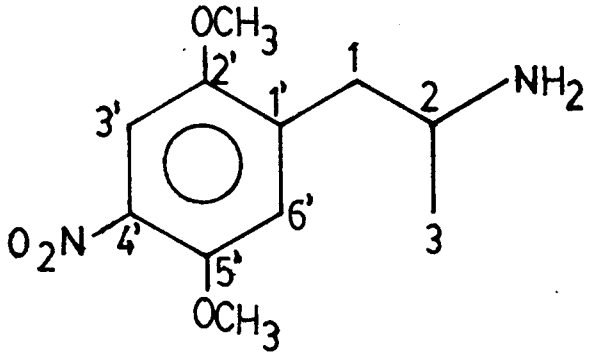


Figure 1. 1-(2,5-dimethoxy-4-nitrophenyl)-2-aminopropane (DON).

The application of a new formal QSAR method (3,4) to the study of the relationship between electronic stucture and serotonin receptor binding affinities of 1-phenyl-2-aminopropanes (5) and indolealkylamines (6,7), has led to the conclusion that DON can be expected to be hallucinogenic at low doses in spite of the fact that, contrary to the case of the potent hallucinogenic amphetamines such as DOM and DOB, the 4-substituent is hydrophilic. The study leading to this conclusion and the pharmacological testing of this compound will be published elsewhere (8, 9). Here we can say that a dose of 4.5 mg of DON nitrate produces the standard effects described in the literature (10,11). The only qualitative difference between DON and its 4-Br analog (DOB) is that DON has a very strong stimulating action reminiscent of amphetamine. This action seems to reduce the incidence of insightful, and therefore potentially unpleasant experiences, and thus seems likely to appear on the market as an illicit recreational drug.

As a contribution to the task of forensic chemists and law enforcement agencies, we report here the UV-VIS, IR, 1-H- and 13-C-NMR spectra of DON.

## EXPERIMENTAL.

DON was prepared by direct nitration of 2,5-DMA (1,2). As the nitrate salt crystallizes quite readily, it can be purified very well by recrystallization from propan-2-ol and normally gives larger, cleaner crystals than the hydrochloride, the nitrate was retained for our spectroscopic and pharmacological studies.

The UV spectrum was recorded with a CARY 17 spectrophotometer. The temperature was 20 °C, 10 mm quartz cells were used, and the concentration of the DON aqueous solution was 0.00012 M. A spectral band of 10 nm was chosen with a sweep velocity of 0.2 nm/sec.

The IR spectrum was obtained with a Perkin-Elmer 621 IR spectrophotometer over the range 4000 to 300 (1/cm). The sample was prepared in the form of 13 mm KBr disc containing about 1 mg of DON nitrate in 100 mg of Merck KBr of spectroscopic quality.

The $^{1}$H-NMR spectrum was recorded with a Varian T-60 spectrometer (60 MHz) for a solution of DON nitrate in $D_2O$. The internal reference used was the sodium salt of 3-(trimethylsilyl)-propane sulfonic acid.

The $^{13}$C-NMR spectrum was recorded at 50.1 MHz using a Bruker WA-200 instrument. The solution was prepared in $D_2O$ and the internal reference used was the same as above.

## RESULTS AND DISCUSSION.

The UV-Vis spectrum is displayed in Fig.2. The wavelengths and molar absorption coefficients for the maxima and minima are shown in Tables

I and II, respectively. The absorption maximum at 375 nm corresponds to a band wich is highly characteristic of the yellow DON, as the colorless 1-phenyl-2-aminopropanes like DOM and DOB (the 4-methyl- and 4-Bromo-analogues of DON) only show absorption maxima below 300 nm. The long-wavelength band of DON can be interpreted as an intramolecular charge-transfer band (benzene ring to nitro group).

The IR spectrum is presented in Fig.3. Table III shows the frequencies and assignments for the more characteristic vibrational bands of DON nitrate.

The $^{1}$H-NMR spectrum is shown in Fig.4, with the tentative assignments in Table IV. The proton-decoupled 13-C NMR spectrum is reproduced in Fig.5, with the corresponding assignments given in Table V. The downfield $^{1}$H and $^{13}$C methoxyl resonances were assigned to the group located at C-5', which is assumed to be more strongly deshielded than the C-2' methoxyl due to the proximity of the magnetically anisotropic nitro function.

Taken in conjunction, these spectroscopic data can lead to the rapid identification of DON in black market samples. In particular, the UV absorption at 375 nm should prove useful for the quantitative assay of this drug.

ACKNOWLEDGMENTS.

This work has received financial support from the DIB (Q-2742 Project) and from Fondo Nacional de Ciencia (Project 1075).

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TABLE I. Wavelengths and molar absorption coefficients of the absorption maxima.

| λ(nm) | ε (lt/mol cm) |
|---|---|
| 245 ± 0.4 | 4670 ± 50 |
| 279.7 | 3560 |
| 375.1 | 2940 |

TABLE II. Wavelengths and molar absorption coefficients of the absorption minima.

| λ(nm) | ε(lt/mol cm) |
|---|---|
| 237.0 ± 0.4 | 4460 ± 50 |
| 265.0 | 2970 |
| 318 | 1120 |

TABLE III. Frequencies and assignments for the main IR bands of DON nitrate.

| Wavelength ($cm^{-1}$) | Assignment |
|---|---|
| 3230 and 3180 | Asymmetric and symmetric N-H stretching. |
| 2600-2400 | Quaternary N atom. |
| 1605 and 1575 | C=C stretching in the aromatic ring. |
| 1510 and 1345 | Symmetric and asymmetric stretching of the 4-$NO_2$ group. |
| 1385 | Bending of the C-H bonds of the isopropyl group. |
| 1280 and 1220 | Stretching of the O-$CH_3$ group. |
| 1305 | C-O stretching |

TABLE IV. Chemical shifts (δ),multiplicities and assignments for the $^1$H-NMR signals of DON nitrate in $D_2O$ at 60 MHz.

| Chemical shift (δ,ppm) | Multiplicity | Assignment |
|---|---|---|
| 1.41 | d, J=6 Hz | H-3 |
| 3.07 | d, J=6 Hz | H-1 |
| 3.83 | m | H-2 |
| 3.94 | s | C-2' O-$CH_3$ |
| 4.02 | s | C-5' o-$CH_3$ |
| 7.20 | s | H-6' |
| 7.50 | s | H-3' |
| 4.90 | s | $H_2O$ |

TABLE V. Chemical shifts ($\delta$) and assignments for the $^{13}C$ NMR signals of DON nitrate in $D_2O$ at 50 MHz.

| Chemical shift ($\delta$,ppm) | Assignment |
|---|---|
| 20.5 | C-3 |
| 37.9 | C-1 |
| 50.6 | C-2 |
| 59.0 | C-2' O-$CH_3$ |
| 60.0 | C-5' O-$CH_3$ |
| 111.3 | C-3' |
| 120.6 | C-6' |
| 136.2 | C-1' |
| 140.0 | C-4' |
| 150.4 | C-5' |
| 153.8 | C-2' |

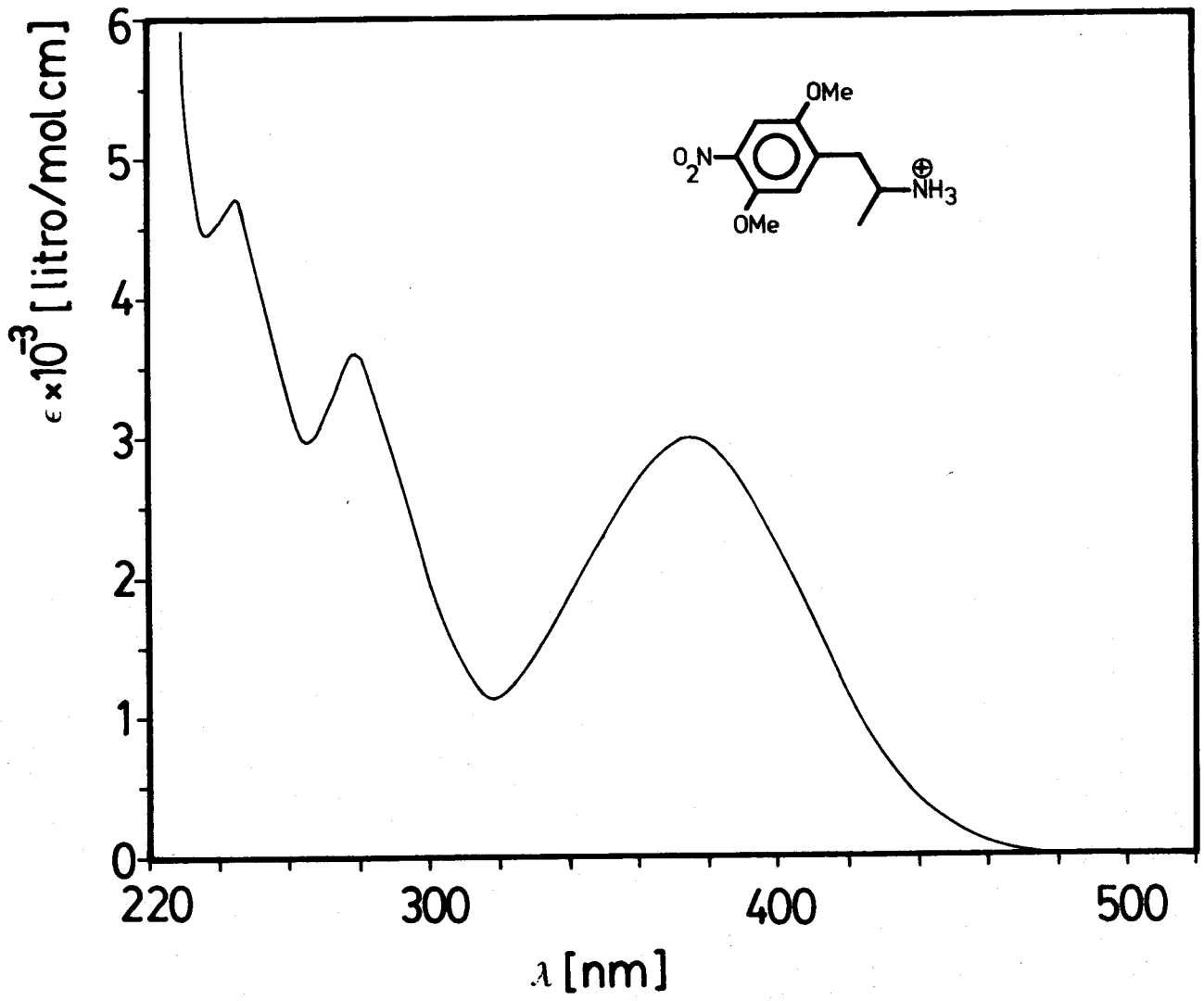


FIGURE 2. UV-Vis spectrum of DON nitrate.

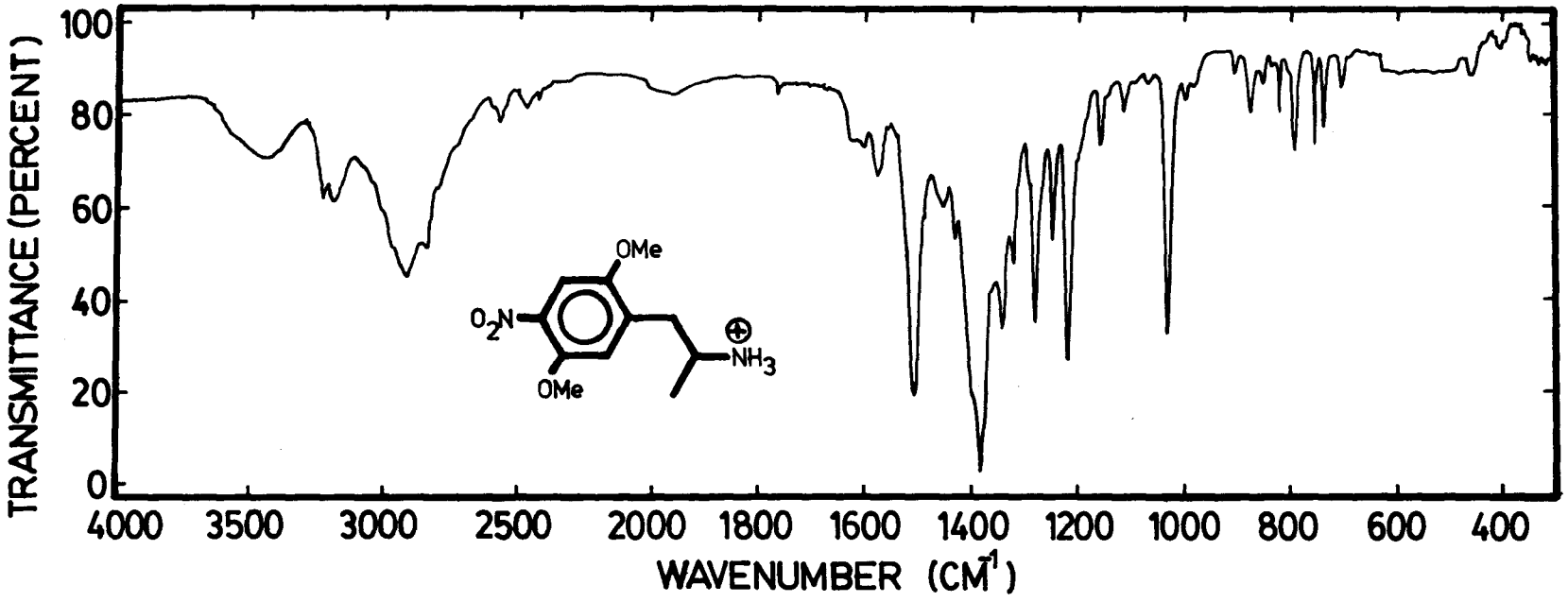


*Figure 3.* IR spectrum of DON nitrate.

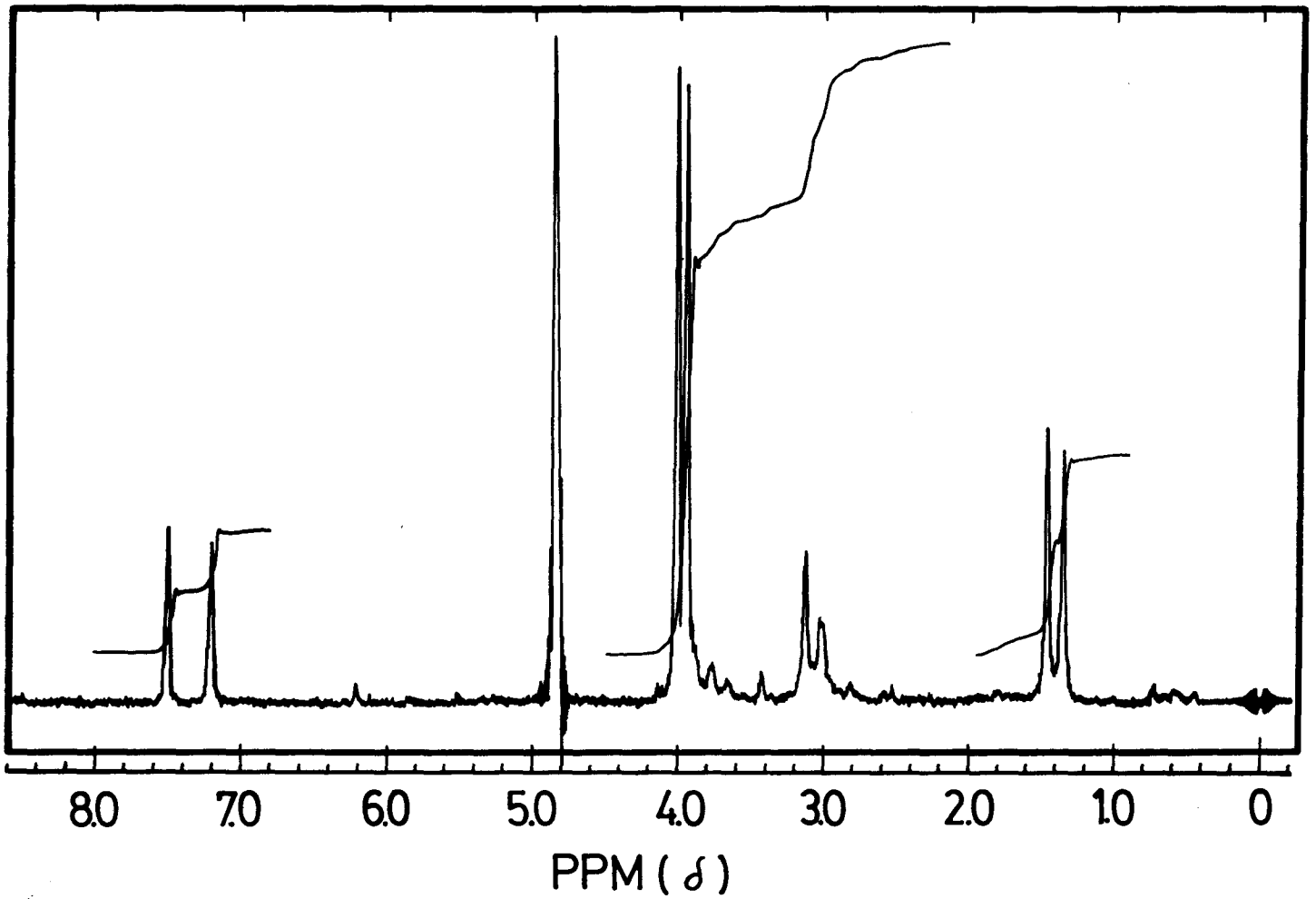


Figure 4. $^{1}H$ NMR spectrum of DON nitrate in $D_2O$ at 60 MHz.

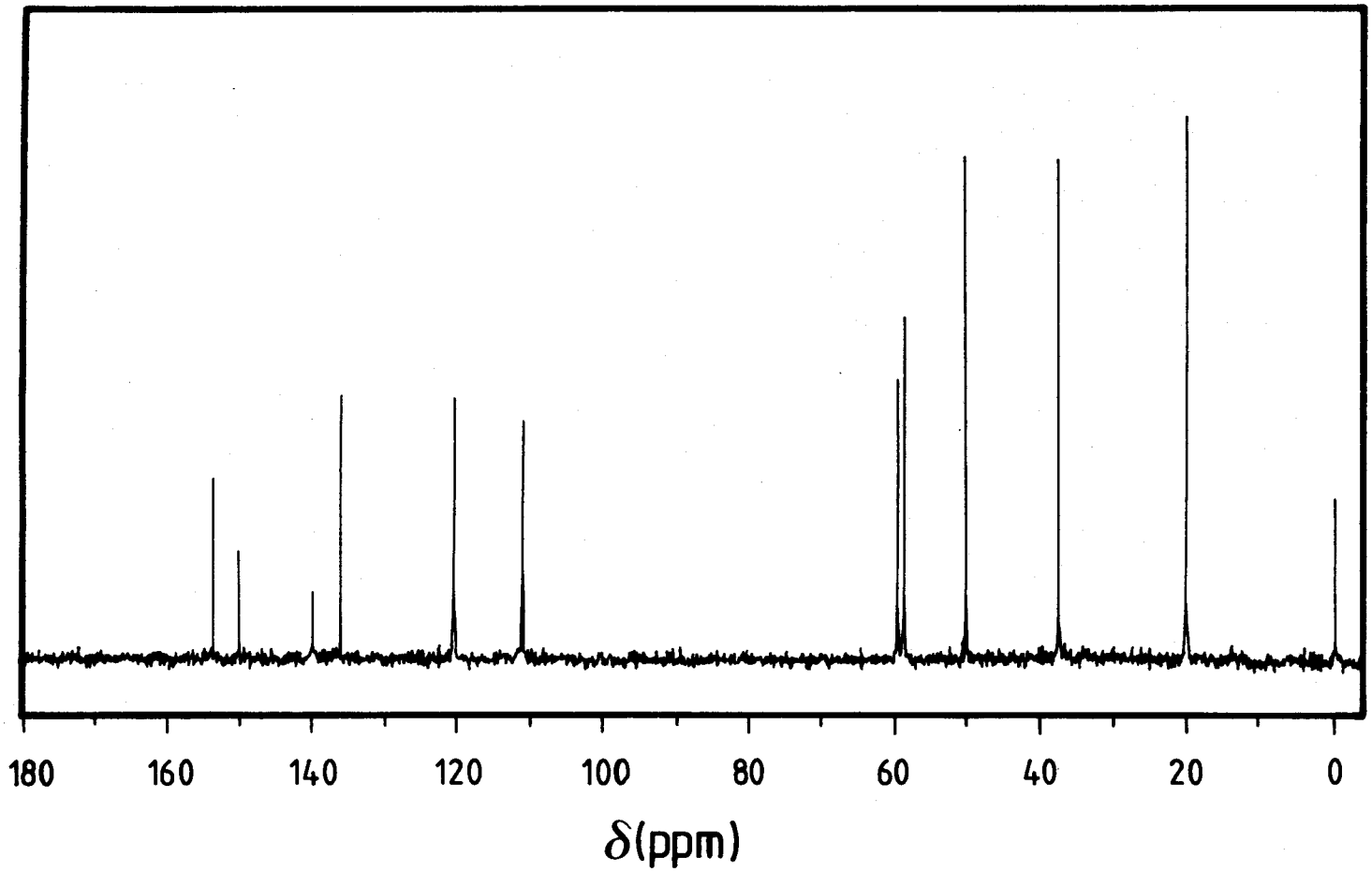


*Figure 5.* $^{13}C$ NMR spectrum of DON nitrate at 50 MHz.

* * * NOTICE * * *

The infrared spectrum of methamphetamine HCl shown below and identified as Figure #3 is an addendum to the September 1986 issue of Microgram. Therefore, you may wish to either copy or remove this page and place it at the end of the September issue (Vol. XIX, No. 9).

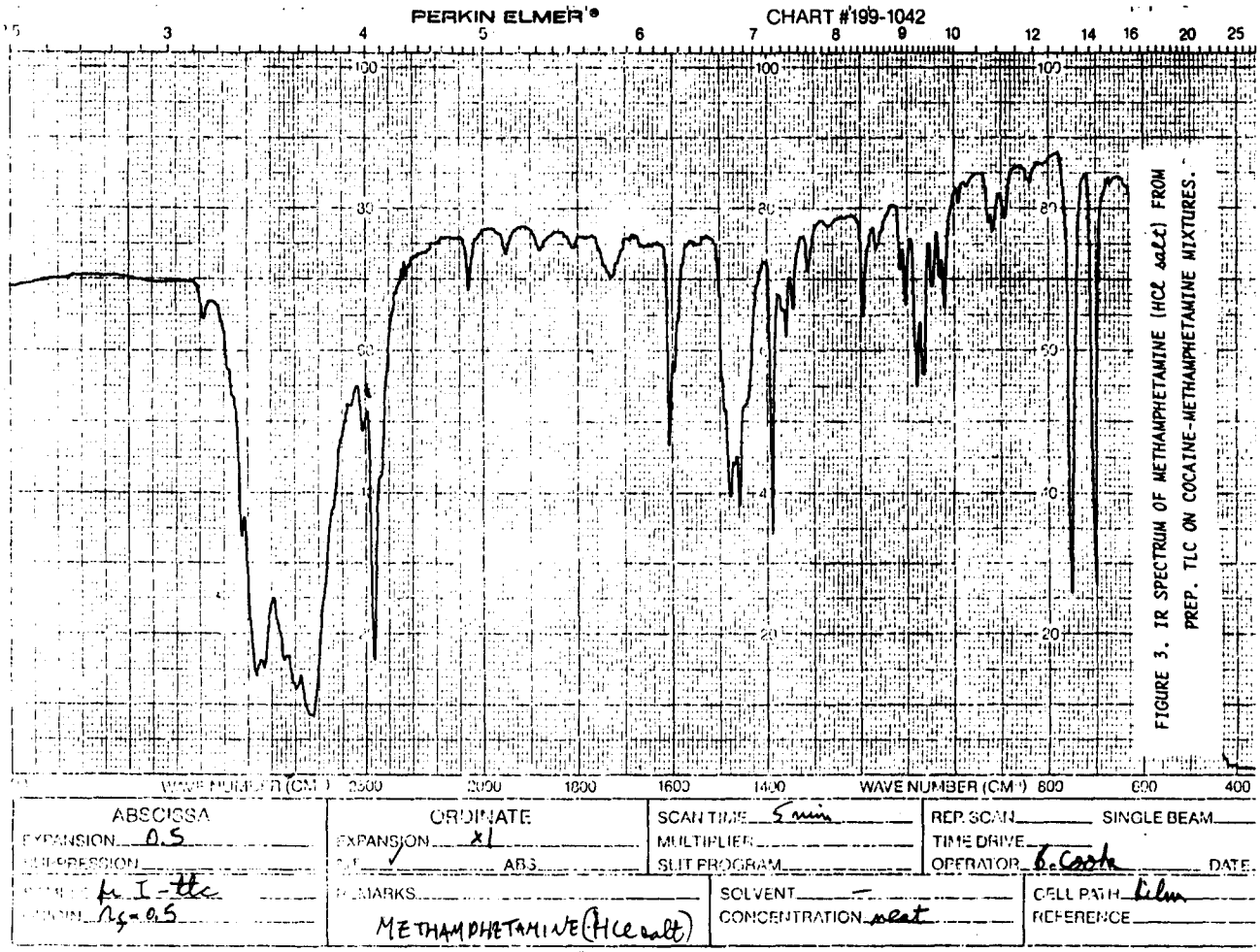


FIGURE 3. IR SPECTRUM OF METHAMPHETAMINE (HCl salt) FROM PREP. TLC ON COCAINE-METHAMPHETAMINE MIXTURES.