Sanford A. Angelos,[1] *M.Sc., M.Ed.; Thomas J. Janovsky,*[2] *B.Sc.; and Jack K. Raney,*[1] *M.Sc.*

The Identification and Quantitation of Pharmaceutical Preparations by Nuclear Magnetic Resonance Spectroscopy

REFERENCE: Angelos, S. A., Janovsky, T. J., and Raney, J. K., **"The Identification and Quantitation of Pharmaceutical Preparations by Nuclear Magnetic Resonance Spectroscopy,"** *Journal of Forensic Sciences,* JFSCA, Vol. 36, No. 2, March 1991, pp. 358–365.

ABSTRACT: The identification and quantitation of pharmaceutical preparations is often the most time-consuming analysis performed in the forensic science laboratory. Because of their hygroscopic and polymorphic properties, matching infrared spectra are frequently difficult to obtain. Moreover, many of these compounds also fragment in such a manner that their mass spectra are difficult to interpret. Nuclear magnetic resonance offers an attractive alternative analysis since it avoids these problems and allows simultaneous identification and quantitation of dosage forms. Typical procedures are described for pharmaceutical forms of barbiturates, phenethylamines, quinazolones, benzodiazepines, and ethchlorvynol. This method has been successfully used and in most cases does not require a preliminary separation of the pharmaceutical preparation's components.

KEYWORDS: toxicology, chemical analysis, drug identification, nuclear magnetic resonance

At the 1978 International Symposium on Instrumental Applications in Forensic Drug Chemistry, the opinion was expressed that, because of its limited sensitivity, nuclear magnetic resonance (NMR) has had very little impact in the field of forensic chemistry [*1*]. Although there have been considerable improvements in its sensitivity in the past ten years, there may still be forensic science areas where NMR is still not the analytical spectroscopic method of choice [*2–5*]. An exception, however, is the analysis of pharmaceutical preparations [*6–11*], where sensitivity is generally not a crucial factor. Although the literature coverage for the NMR quantitation of barbiturates and phenethylamines, in particular, is excellent, the analysis of many preparations in the presence of excipients and multiple drug preparations is not sufficiently covered. This is especially true for the multiple barbiturates such as SBP®.

While the components of most pharmaceutical preparations submitted for analysis can be nominally identified by some type of reference material, such as the *Physicians' Desk Reference* (PDR), the forensic chemist must still conduct an analysis of the material to confirm the presence (if any) and amount of the controlled substance. This is significant

Received for publication 16 Feb. 1990; revised manuscript received 5 April 1990; accepted for publication 9 April 1990.

[1]Senior forensic chemist and forensic chemist, respectively, U.S. Drug Enforcement Administration, North Central Laboratory, Chicago, IL.

[2]Supervisory chemist, U.S. Drug Administration, Special Testing and Research Laboratory, McClean, VA.

with the increase of counterfeit phenethylamines. There is usually a sufficient amount of the controlled substance in a single-dosage unit to produce a satisfactory NMR spectrum. Where one dosage unit is insufficient, not more than one or two extra units are usually required for a viable sample. With the addition of an internal standard, identification and quantitation are obtained in a single analysis. The benefits of using NMR as opposed to other spectroscopic techniques become evident when considering some of the typical problems encountered in the analysis of pharmaceutical preparations.

Obtaining an infrared (IR) spectrum of substituted phenethylamines (that is, methamphetamine, amphetamine) is often complicated by their hygroscopic and polymorphic properties. This can result in several different IR spectra for a single compound. The mass spectra of these phenethylamines exhibit weak parent peaks and very similar fragmentation patterns and are not as definitive as NMR. Similarly, barbiturates form clathrates (that is, chloroform adducts) and polymorphs, which makes their analysis by IR difficult. Mass spectral (MS) differences between most of the barbiturates are usually minimal and not easily distinguished. Benzodiazepines, tranquilizers such as diazepam (Valium®), flurazepam (Dalmane®), and oxazapam (Serax®), break down in such a manner that they also display similar mass spectra. In cases where the IR and MS are suitable for use in identifying the compound of interest, quantitation may require separation from the formulation material or noncontrolled companion drugs (for example, Placidyl® and Darvocet®). In almost all cases, a combination of IR, gas chromatography/mass spectrometry (GC/MS), gas-liquid chromatography (GLC), or high-performance liquid chromatography (HPLC) is needed both to identify and to quantitate the controlled substance.

The NMR spectra of these and other compounds are, however, unique and often result in a clearly identifiable spectrum. Adding an appropriate internal standard during sample preparation makes simultaneous quantitation possible. The savings in time from using NMR instead of two different techniques for identification and quantitation can be significant [*12–22*].

This paper presents an NMR method that can be used for simultaneous identification and quantitation of a number of pharmaceutical preparations commonly encountered in forensic science laboratories. No effort is made to reconfirm the accuracy of the quantitations since the use of an internal standard produces values which are accurate to 1.5 to 2.0% [*23–25*]. The values obtained, however, will be compared with the manufacturer's declared potency for informational purposes. The data can be useful, particularly in cases in which the preparation is of suspected illicit origin.

Experimental Procedure

All spectra were obtained on a Varian EM-390 NMR continuous-wave (CW) spectrometer operating at 90 MHz. The instrumental parameters are listed in Table 1. The deuterated solvents were purchased from the Aldrich Chemical Co. The samples are prepared by accurately weighing an amount of powder from the ground-up pharmaceutical preparation, equivalent to approximately 15 to 25 mg of the pure drug, into a disposable test tube. To this is added accurately weighed internal standard (approximately 15 to 20 mg of the internal standard). With the small amount of the internal standard used in the analysis, transfer losses can greatly affect the quantitation results; therefore, it is extremely important to determine the masses by measuring mass differences accurately.

To the test tube with the weighed sample and internal standard, approximately 0.6 mL of a suitable deuterated solvent is added. The mixture is then placed in an ultrasonic vibrator for up to 5 min to ensure that the sample and standard are properly dissolved in the solvent. The mixture is then centrifuged to separate any undissolved formulation material. The clear liquid is withdrawn and transferred into an NMR tube. In some

TABLE 1—*Instrumental parameters of the CV spectrometer.*

Field strength	90 MHz
Spectrum amplitude	1000–9000
Filter	0.05 sec
RF power	0.03 milligauss
Sweep time	5.0 min
Sweep width	10 ppm
End of sweep	0 ppm
Nucleus	H^1
Zero reference	tetramethylsilane
Sample temperature	34°C
Lock power	off
Decoupling power	off

instances, the amount of the liquid portion recovered may not be enough to fill the NMR tube properly. In these cases, additional solvent can be added to the test tube, after which the tube is re-sonicated and centrifuged. Tetramethylsilane (TMS) is added to the solution as a chemical shift reference.

The NMR spectrum is then obtained in the usual manner with an integration of the required peaks [*2,26,27*]. The integrated area of the proton resonance peak of the standard is then directly compared with the area of the resonance peak of the sample being analyzed. From the molecular weight considerations and the relative peak areas, the following relationship may be used for the assay:

$$\text{Composition in } \% = \left(\frac{Wi}{Ws}\right) \times \left(\frac{As}{Ai}\right) \times \left(\frac{Ms}{Mi}\right) \times 100$$

Ws and *Wi* are the weights of the total pharmaceutical sample and the internal standard, respectively. *As* and *Ai* are the measured integral areas of the analytical peaks of the pharmaceutical sample and the internal standard, respectively. *Ms* and *Mi* are the proton equivalent weights of the pharmaceutical sample and the internal standard, respectively. The proton equivalent weight is the molecular weight of the compound divided by the number of protons generating the absorption signal [*23,28*].

The internal standard of choice for most of the pharmaceutical preparation analyzed is maleic acid. The resonance peak of the two protons for maleic acid appears at 6.2 ppm. Since the NMR spectra of most pharmaceutical preparations have no double bonding, they are free of absorption resonances in the 6 to 7-ppm region. Maleic acid avoids the solubility problems encountered with sulfate salts in deuterated methanol or dimethylsulfoxide (DMSO). Any deuterated solvent in which both the sample and the internal standard are readily soluble can be used.

Pharmaceutical Preparations Analyzed

Barbiturates

Phenobarbital is probably the simplest barbiturate to quantitate, since three of the four peaks may be chosen. The triplet at 0.85 ppm is due to the terminal methyl group and has a proton density of three. Similarly, the methylene quartet at 2.25 ppm has a density of two protons, and the phenyl resonance peak at 7.42 ppm has a density of five protons. The amine protons are not appropriate for quantitation because of the possibility of exchange with any hydroxyl protons in the solution, as well as with the deuterons of the

solvent. The companion drugs most frequently encountered in phenobarbital preparations and formulating material have NMR spectra whose major resonance peaks appear between the methylene and the phenyl resonances of the phenobarbital. Therefore, in all cases which have been investigated to date, any of the three specified resonance peaks could be used for quantitation.

Butalbital is another commonly encountered barbiturate. One resonance peak seems to be best for quantitation. This is the doublet due to the geminal dimethyl group at 0.65 ppm. Its proton density may be determined from the structure shown in Table 2 to be six. Butalbital preparations have been encountered in extremely complex mixtures of companion drugs. Two such mixtures are (1) aspirin, phenacetin, and caffeine (APC) and (2) caffeine, phenacetin, and salicylamide. Even with spectra as complicated as these, the internal standard resonance peak and the gem dimethyl doublet of the barbiturate are well separated and may be used for quantitation. For a better identification of butalbital, it may be advisable to increase the intensity, thus bringing out the butalbital spectra and allowing the spectra of the companion drugs to go off scale.

With some barbiturate preparations, it may be advantageous to add trifluoroacetic acid to the solution prior to obtaining the NMR spectrum. The addition will shift the amine resonance peak downfield to the 8 to 10-ppm region. The actual amount of trifluoroacetic acid added to the solution will vary with the sample; usually two to five drops will be sufficient. Therefore, a spectrum will be obtained in which the amine resonance peak will not interfere with or overlap the maleic acid or the aromatic region resonances [*29*].

The identification and quantitation of multiple barbiturate preparations can also be accomplished by NMR. There are several such pharmaceutical preparations that have as many as three barbiturates. Sedrite® and SBP® both contain secobarbital and phenobarbital. In addition, Sedrite has pentobarbital, while SBP has butabarbital. The most common multiple barbiturate encountered in forensic science laboratories is Tuinal®, a mixture of secobarbital and amobarbital. It also presents one of the most difficult pharmaceutical preparations for quantitation. The secobarbital resonance peak at 2.6 ppm is well separated, allowing an easy quantitation; however, the amobarbital resonance peaks are all coincident with the secobarbital resonances. The resonance cluster between 1.7 and 2.3 ppm represents one proton from the secobarbital and five protons from the amobarbital. It is, therefore, possible to quantitate the amobarbital by subtracting the area represented by the protons of secobarbital from the area of the entire cluster [*30*].

Phenethylamines

The first two compounds investigated were methamphetamine and phentermine. Both compounds have the same molecular weight of 149; the only structural difference is the position of a methyl group. The mass fragmentation patterns of these compounds are very similar and, without additional spectral data, very difficult to differentiate. However, the NMR spectrum of methamphetamine is quite different from the spectrum of phentermine. Quantitation of these compounds and other phenethylamines can be accomplished by integrating the suitable interference free resonance peaks (Table 3).

The analysis of a sample by NMR even allows the identification and quantitation of isomeric compounds. Because of similar fragmentation patterns, the mass spectra of the three trimethoxyamphetamines are difficult to differentiate. NMR, however, displays very clear and interpretable differences. The first isomer investigated was 2,4,6-trimethoxyamphetamine. The spectrum of the isopropylamine moiety displays the same resonance patterns found in other amphetamines and is essentially the same for each of the three trimethoxyamphetamine compounds. Since the two *ortho*-methoxy peaks (Positions 2 and 6) are equivalent, a single resonance peak representing six protons is found. The third methoxy group has a peak intensity of three protons and is observed at a

TABLE 2—*Proton resonances for barbiturates in ppm.*[a,b]

	N—H[c]	CH_3	CH_2	CH	CH_2=	CH=	C_6H_5
Allobarbital	3.3 (2)		2.5d (4)		5.1m (4)	5.6m (2)	
Amobarbital	3.3 (2)	0.8t (9)	1.8q (6)	1.4m (1)			
Aprobarbital	3.3 (2)	1.0d (6)	2.7d (2)	2.4m (1)	5.1m (2)	5.6m (1)	
Barbital	3.3 (2)	0.7t (6)	1.8q (4)				
Butalbital	3.3 (2)	0.7d (6)	1.8d (4)	1.5m (1)	5.1s (2)	5.5m (1)	
Pentobarbital	3.3 (2)	1.0m (6)	2.2q (4)	1.3m (1)			
Phenobarbital	3.3 (2)	0.8s (3)	2.2s (2)				7.4 (5)
Secobarbital	3.3 (2)	0.8d (6)	1.8m (6)	1.3m (1)	4.9t (2)	5.4m (1)	

[a]Resonance peak types:
s = single peak.
d = doublet.
t = triplet.
q = quartet.
m = multiple peaks.
[b]Numbers in parentheses are the numbers of protons causing the resonance.
[c]General position if not shifted with trifluoroacetic acid.

TABLE 3—*Proton resonances for phenethylamines in ppm.*[a,b]

	N—H[c]	CH_3	CH_2	CH	CH_3—N	C_6H_5
Phentermine	1.6 (2)	1.2s (6)	2.7s (2)			7.3s (5)
Methamphetamine	1.8 (1)	1.1d (3)	2.8d (2)	2.8m (1)	2.5s (3)	7.3s (5)
Amphetamine	1.7 (2)	1.0d (3)	2.7d (2)	2.4m (1)		7.4s (5)
Ephedrine	1.5 (1)	1.2d (3)		2.5m (2)	2.5s (3)	7.3s (5)

[a]Resonance peak types:
s = single peak.
d = doublet.
t = triplet.
q = quartet.
m = multiple peaks.
[b]Numbers in parentheses are the numbers of protons causing the resonance.
[c]General position if not shifted with trifluoroacetic acid.

chemical shift of 3.8 ppm. The remaining two phenyl protons (equivalent) have a resonance at 6.2 ppm. The 3,4,5-trimethoxyamphetamine spectrum is similar to that of the 2,4,6 isomer. The difference is that the high-field methoxy resonance peak now represents the single *para*-methoxy group and the low-field resonance peak is the two equivalent *meta*-methoxy groups (Positions 3 and 5). The third isomer, 2,4,5-trimethoxyamphetamine, shows three nonequivalent methoxy peaks of equal intensity. In addition, the two phenyl protons are also nonequivalent and display two resonance peaks of equal intensity at 6.6 and 6.8 ppm [*31*,*32*].

Quinazolones

The NMR spectra of methaqualone and mecloqualone are similar. The notable difference between the two spectra is the additional methyl group of methaqualone. The quantitation of either can be accomplished by measuring the resonance peak areas. In the case of methaqualone, both methyl peaks at 2.1 ppm and 2.2 ppm should be integrated and used for quantitation. Mecloqualone has one methyl group whose resonance peak is at 2.2 ppm.

Propoxyphene

The use of the deuterated dimethyl sulfoxide allows the quantitation of both the napsylate and hydrochloric salts of propoxyphene. The integration of the entire aromatic region is the most suitable choice.

When the companion drug, acetaminophen, is present, as in Darvocet, its phenyl protons contribute to the resonance in the aromatic region. In this case, quantitation of the propoxyphene can be accomplished by using the resonance peaks from the two non-nitrogen methyl groups in the region of 0.9 to 1.3 ppm. Acetaminophen can be quantitated by using the acetamide methyl at 2.3 ppm. Both compounds can be identified and quantitated with a single NMR spectrum. It is important to note that in doing the calculations for this propoxyphene quantitation, it is necessary to evaluate a standard spectrum of propoxyphene. The M_r propoxyphene is not simply six protons as expected. The methine proton *beta* to the nitrogen has a very complicated splitting pattern, which is partially coincident with the two non-nitrogen methyl resonance peaks. It has been determined that overlapping resonance contributes an area equivalent to one third of a proton. Therefore, for the 90-MHz system, the M_r is determined to be 6.33 [*33*].

Benzodiazepines

Oxazepam—The resonance signals for this compound are very weak, and as a result, when a sample is analyzed, the eight aromatic protons are used for both identification and quantitation.

Diazepam—In Valium preparations, the tableting material causes considerable interference and a good identification is not directly possible. Therefore, a preliminary extraction is required. The preparation is extracted with three equal 15-mL portions of chloroform. (If one or two tablets are used for the extraction, the volume of chloroform may be reduced to 5 mL.) Methylene chloride may be substituted for chloroform. The solution is evaporated to dryness, and the internal standard is added to the residue. The resulting spectrum is then suitable for identification and quantitation. The seven aromatic protons can be used for quantitation even if the sample is not extracted.

Flurazepam—The identification of flurazepam from a Dalmane preparation is similarly difficult. Extraction of the preparation in the same manner as diazepam is required. The resulting spectrum can be used both to identify and to quantitate. The seven aromatic protons are the most suitable for quantitation.

Ethchlorvynol

The alkene protons of ethchlorvynol are in the 7 to 6-ppm region. Their resonance would interfere with the maleic acid resonance peak. Therefore, another internal standard must be chosen—in this case, benzoic acid is used. The quantitation from a spectrum of a Placidyl preparation with benzoic acid can be accomplished by using the five aromatic protons for the standard and the alkene protons for the sample.

Conclusions

NMR is a nondestructive technique that has proven to be very versatile. It is possible to analyze and identify pure compounds as well as mixtures of compounds.

This work has shown that identification and quantitation can often be accomplished in a single NMR spectrum. This procedure is equally applicable to legitimate and illicit pharmaceutical preparations, although tableting formulations sometimes require a preliminary extraction. Maleic acid has been found to be an excellent choice for the internal

standard, since only a few drugs of interest to forensic science interfere in its absorption region.

References

[1] Becker, E. D., "Nuclear Magnetic Resonance: Recent Development in Techniques and Their Potential Application to Forensic Research," *Instrumental Applications in Forensic Drug Chemistry, Proceedings of the International Symposium*, U.S. Drug Enforcement Administration, Washington, DC, May 1978, pp. 70–79.

[2] Freeman, R., *A Handbook of Nuclear Magnetic Resonance*, Longman Scientific & Technical, New York, 1988.

[3] Akitt, J. W., *NMR and Chemistry: An Introduction to the Fourier Transform-Multinuclear Era*, Chapman and Hall, London, 1983.

[4] Memory, J. D. and Wilson, N. K., *NMR of Aromatic Compounds*, John Wiley & Sons, New York, 1982.

[5] Levy, G. C., *NMR Spectroscopy: New Methods and Applications*, American Chemical Society, Washington, DC, 1982.

[6] Pariff, R. T., "Nuclear Magnetic Resonance Spectroscopy: Part 1," *Pharmaceutical Journal*, Vol. 203, No. 5524, 1969, pp. 300–303.

[7] Pariff, R. T., "Nuclear Magnetic Resonance Spectroscopy: Part 2," *Pharmaceutical Journal*, Vol. 203, No. 5525, 1969, pp. 320–324.

[8] Pariff, R. T., *Instrument News (Perkin Elmer)*, Vol. 20, No. 4, 1970, pp. 8–9.

[9] Rackham, D. M., "Quantitative Analysis in Pharmacy and Pharmaceutical Chemistry by Nuclear Magnetic Resonance Spectroscopy," *Talanta*, Vol. 17, 1970, pp. 895–906.

[10] Rackham, D. M., "Recent Applications of Quantitative Nuclear Magnetic Resonance Spectroscopy in Pharmaceutical Research," *Talanta*, Vol. 23, 1976, pp. 269–274.

[11] Kasler, F., *Quantitative Analysis by NMR Spectroscopy*, Academic Press, New York, 1973.

[12] Rucker, G., "Über die Quantitativ Bestimmung von Barbituraten durch Kernresonanzspektroskopie," *Zeitschrift für Analytische Chemie*, Vol. 229, 1967, pp. 340–343.

[13] Turczan, J. W. and Kram, T. C., "Determination of Meprobamate in Tablets by NMR," *Journal of Pharmaceutical Sciences*, Vol. 56, No. 12, 1967, pp. 1643–1645.

[14] Kram, T. C., "Analysis of Illicit Drug Exhibits by Hydrogen-1 Nuclear Magnetic Resonance Spectroscopy," *Journal of Forensic Sciences*, Vol. 23, No. 3, 1978, pp. 456–469.

[15] Liu, J. H., Ramesh, S., Tsay, J. T., Ku, W. W., Fitzgerald, M. P., Angelos, S. A., and Lins, C. L., "Approaches to Drug Sample Differentiation: II. Nuclear Magnetic Resonance Determination of Methamphetamine Enantiomers," *Journal of Forensic Sciences*, Vol. 26, No. 4, 1981, pp. 656–663.

[16] Turczan, J. W. and Kram, T. C., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—I," *Journal of Pharmaceutical Sciences*, Vol. 56, No. 12, 1967, pp. 1643–1645.

[17] Kram, T. C. and Turczan, J. W., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—II," *Journal of Pharmaceutical Sciences*, Vol. 57, No. 4, 1969, pp. 651–652.

[18] Kram, T. C. and Turczan, J. W., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—III," *FDA By-Lines*, Vol. 2, No. 3, 1971, pp. 105–130.

[19] Turczan, J. W. and Medwick, T., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—IV," *Journal of Pharmaceutical Sciences*, Vol. 61, No. 3, 1972, pp. 434–443.

[20] Turczan, J. W. and Goldwitz, B. A., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—V," *Journal of Pharmaceutical Sciences*, Vol. 61, No. 4, 1972, pp. 613–615.

[21] Turczan, J. W. and Goldwitz, B. A., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—VI," *Journal of Pharmaceutical Sciences*, Vol. 61, No. 8, 1972, pp. 1309–1314.

[22] Turczan, J. W., Goldwitz, B. A., and Nelson, J. J., "Nuclear Magnetic Resonance Analysis of Pharmaceuticals—VII," *Talanta*, Vol. 19, 1972, pp. 1549–1554.

[23] Leyden, D. E. and Cox, R. H., *Analytical Application of NMR*, Vol. 48, *Chemical Analysis Series*, P. J. Elving and J. D. Winefordner, Eds., John Wiley & Sons, New York, 1977.

[24] Casy, A. F., *PMR Spectroscopy in Medicinal and Biological Chemistry*, Academic Press, London, 1971.

[25] Jackman, L. M. and Sternhell, S., *Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry*, Vol. 10, *International Series in Organic Chemistry*, D. H. R. Barton and W. Doering, Eds., Pergamon Press, Oxford, 1969.

[26] Gunther, H., *NMR Spectroscopy: An Introduction*, translation by R. W. Gleason, John Wiley & Sons, New York, 1980.

[27] Jackman, L. M. and Sternhell, S., *Applications of Nuclear Magnetic Resonance Spectroscopy in Organic Chemistry*, Pergamon Press, Oxford, 1978.

[28] Paudler, W. W., *Nuclear Magnetic Resonance: General Concepts and Applications*, John Wiley & Sons, New York, 1988.

[29] Angelos, S. A. and Raney, J. K., "Identification and Quantitation of Barbiturates by Nuclear Magnetic Resonance," presented at the 32nd Annual Meeting of the Orleans, LA, American Academy of Forensic Sciences, Feb. 1980.

[30] Angelos, S. A. and Raney, J. K., "Identification and Quantitation of Multiple Barbiturates' Preparations by Nuclear Magnetic Resonance," presented at the 33nd Annual Meeting of the American Academy of Forensic Sciences, Los Angeles, CA, 17–20 Feb. 1981.

[31] Janovsky, T. J. and Raney, J. K., "A Comparison of Spectroscopic Techniques Used in the Identification Phenethylamines," presented at the 32nd Annual Meeting of the American Academy of Forensic Sciences, New Orleans, LA, 20–23 Feb. 1980.

[32] Dawson, B. A. and Avdovich, H. W., "Identification of 4-Ethoxy-2,5 Dimethoxyamphetamine by an NMR Shift Reagent Study," *Canadian Society of Forensic Science*, Vol. 20, No. 1, 1987, pp. 29–34.

[33] Angelos, S. A., Janovsky, T. J., and Raney, J. K., "Quantitation and Identification by Nuclear Magnetic Resonance," presented at the 34th Annual Meeting of the American Academy of Forensic Sciences, Orlando, FL, 8–11 Feb. 1982.

Address requests for reprints or additional information to
Sanford A. Angelos, Senior Forensic Chemist
U.S. Drug Enforcement Administration
North Central Laboratory
610 S. Canal St.
Chicago, IL 60607