

## TECHNICAL NOTE

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# Structural Determination of the Principal Byproduct of the Lithium-Ammonia Reduction Method of Methamphetamine Manufacture

**ABSTRACT:** One common method of illicit methamphetamine manufacture utilizes an alkali metal, typically lithium, and liquid ammonia to chemically reduce ephedrine or pseudoephedrine to form methamphetamine. This method is often referred to as the lithium-ammonia reduction method or the Birch reduction method. While the hydroxyl group of ephedrine is more reactive than the aromatic ring, excess alkali metal and the presence of a proton source allow the formation of a cyclohexadiene byproduct not found in samples of methamphetamine produced from other manufacturing methods. A sample enriched in this byproduct was generated and characterized using nuclear magnetic resonance (NMR) spectroscopy, gas chromatography-mass spectrometry (GC-MS), infrared (IR) spectrophotometry, and ultraviolet (UV) spectrophotometry. The chemical structure of this byproduct was determined to be 1-(1',4'-cyclohexadienyl)-2-methylaminopropane (CMP).

**KEYWORDS:** forensic science, forensic chemistry, methamphetamine, manufacture, byproduct, nuclear magnetic resonance, reduction

The lithium-ammonia reduction method is one of the most common methods of illicit methamphetamine manufacture in the United States. It is a dissolving metal reduction where an alkali metal, typically lithium, serves as an electron source with ammonia as a solvent that allows the electrons to chemically reduce the hydroxyl group of ephedrine to form methamphetamine (1–5). These conditions are similar to a classical Birch reduction where sodium, ammonia, and an alcohol are used to reduce aromatic rings to form cyclohexadienes (6–12). This similarity results in the use of the term Birch reduction in describing this method. It has also been referred to as the Birch-Benkese reduction and the “Nazi” method (7) in the forensic community.

While the hydroxyl group of ephedrine is preferentially reduced by an alkali metal in the presence of a proton source, such as water absorbed from the atmosphere, an excess of the alkali metal under these reaction conditions can lead to a further partial reduction of the aromatic ring of methamphetamine (Fig. 1) (6–8,12,13). Because this ring reduced byproduct is not formed by other illicit manufacturing methods it serves as a marker of this method of manufacture in forensic casework. With the growing use of this method, this byproduct is encountered in routine drug casework and can be found in blood samples from routine toxicology casework (7,14).

Several names have been used to refer to this byproduct including “the 150 compound,” the “Birch byproduct,” and the “Birch

reduction product.” A tentative structure for this byproduct, matching the theoretically predicted Birch reduction product of methamphetamine, was proposed in an anonymous article in 1997 (1). This article used the notation of (*S*)-*N*-Methyl-1-(1,4-cyclohexadienyl)-2-propanamine for this byproduct, while other references use the notation of 1-(1',4'-cyclohexadienyl)-2-methylaminopropane (Nila Bremer, personal communication, 2003) (7,15). This latter notation and the abbreviation CMP will be used in this article. The authors are not aware of any published confirmation of this structure or of a commercial source for an authenticated standard.

This article describes a simple procedure to generate a sample enriched in CMP and the characterization of this material using techniques frequently used in forensic analysis as well as nuclear magnetic resonance (NMR) experiments. Though not included in the anonymous article, similar experiments were used for structural determination in that study (1).

## Materials and Methods

### *Materials and Instrumentation*

Chemicals were purchased from Sigma-Aldrich Chemical Company with the exception of the anhydrous ammonia purchased from Airgas Inc. and the bulk (1*R*, 2*S*)-ephedrine hydrochloride used to generate the CMP sample. This ephedrine was a seized sample characterized prior to use using authenticated standards.

Fourier transform infrared spectrophotometry (FTIR) analysis was conducted using a Nicolet Nexus 670 bench with an attenuated total reflectance (ATR) accessory with a diamond crystal. Sixteen scans were collected between 4000 and 600 wavenumbers and the Omnic software ATR correction was applied.

Gas chromatography-electron impact mass spectrometry (GC-EIMS) analysis was conducted using an Agilent 6890N Gas

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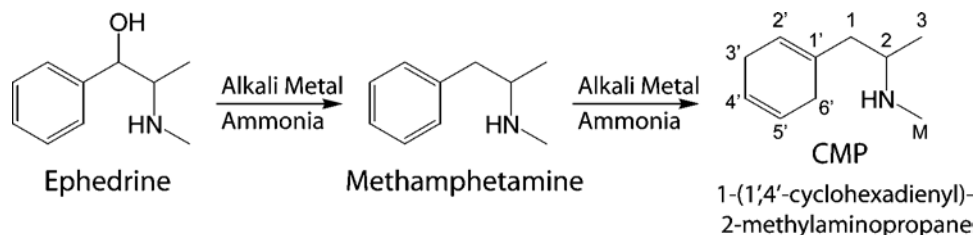


FIG. 1—The lithium-ammonia reduction of ephedrine will produce 1-(1',4'-cyclohexadienyl)-2-methylaminopropane (CMP) if provided with excess lithium and a proton source. The numbering scheme shown is used for peak labeling in Figs. 5–9.

Chromatograph with a 5973N Mass Selective Detector. A 30 m HP-5MS 250  $\mu\text{m}$  ID column with a 0.25  $\mu\text{m}$  film thickness was used with helium carrier gas. After 2 min at 80°C, the oven temperature was ramped to 280°C at 20°C per minute. Samples were prepared by a basic extraction into pentane. Acetyl derivatization was accomplished by adding one drop of acetic anhydride to the extracted sample. (*S*)-(-)-*N*-(Trifluoroacetyl)-prolyl chloride (TPC) derivatization was accomplished by the addition of a drop of 0.1 M TPC in chloroform to the sample and incubation on the top of the GC oven for approximately 6 hours prior to injection.

Gas chromatography-chemical ionization mass spectrometry (GC-CIMS) analysis was conducted using a Varian 3800 Gas Chromatograph and Saturn 2000 Mass Spectrometer with an ion-trap mass analyzer. Methanol was used as the chemical ionization reagent. A 30 m Varian CP-Sil 5CB column was used with helium carrier gas. After two minutes at 80°C, the oven temperature was ramped to 280°C at 20°C per minute. The sample was prepared by a basic extraction into pentane.

Ultraviolet spectrophotometry (UV) analysis was conducted using an Agilent 8453 UV/Vis Spectrometer on a 2.1 mg/mL solution in spectroscopic grade methanol.

Nuclear magnetic resonance (NMR) was conducted using a Varian UNITYINOVA 500 MHz spectrometer. The sample was prepared by dissolving 42 mg of 1-(1',4'-cyclohexadienyl)-2-methylaminopropane (CMP) in 0.7 mL of  $\text{CD}_3\text{OD}$ . Chemical shifts are reported relative to methanol signals at 3.31 ppm in the  $^1\text{H}$  spectrum and 49.15 ppm in the  $^{13}\text{C}$  spectrum. The NMR integrated  $^1\text{H}$ ,  $^{13}\text{C}$ , Distortionless Enhancement via Polarization Transfer (DEPT), gradient Proton-Proton Correlation Spectroscopy (gCOSY), and gradient Heteronuclear Single Quantum Correlation (gHSQC) pulse sequences were used.

#### Generation of Sample Enriched with CMP

In a previous study, 10% isopropyl alcohol in liquid ammonia gave strong CMP production (16). It was hypothesized that by increasing the amount of lithium added to the reaction, a sample with a proportionately large amount of CMP could be generated.

To this end, approximately 200 mL of anhydrous ammonia and 20 mL of isopropyl alcohol were added to a beaker. One gram of (1*R*, 2*S*)-ephedrine hydrochloride was dissolved in this mixture and a large excess of lithium (~4 g) was added portionwise. An additional 200 mL of anhydrous ammonia was added as the solvent evaporated to allow the continued addition of lithium. After addition of the lithium was complete the ammonia was allowed to evaporate. The reaction and excess lithium were quenched with additional isopropyl alcohol and then with water. This basic solution was extracted with pentane. Hydrogen chloride gas was bubbled through the pentane layer, resulting in a precipitate that was filtered and washed with acetone. Seventy milligrams of white powder were recovered from this inefficient purification process (this sample was used for the structural determination). The various extraction and

wash fractions containing the remaining material were discarded. A similar reaction, on a smaller scale, was used to generate CMP from (1*S*, 2*R*)-ephedrine.

#### Results and Discussion

The addition of excess lithium to ephedrine in the presence of approximately 10% isopropyl alcohol allowed the production of a sample enriched in the principal byproduct of the lithium-ammonia reduction method. The simple workup followed by an acetone wash produced a sample of sufficient purity to proceed with the structural determination. The GC retention time and EIMS of the principal component of this sample matched those observed for the byproduct in casework. The EIMS and FTIR matched the data included in the 1997 paper (1). Having established that the sample contained the chemically relevant byproduct, work proceeded using a variety of instrumental techniques to characterize the structure of the byproduct. For clarity, this byproduct will be henceforth referred to as CMP and the derivation of its structure is described. The numbering notation used throughout this discussion and the full chemical name of CMP can be found in Fig. 1.

The gas chromatogram of this sample shows a dominant peak of CMP and several smaller peaks (Fig. 2A). The major impurity in this sample is an unidentified byproduct, possibly a cyclohexene, eluting slightly before the CMP peak with a base peak of  $m/z = 58$  and a parent ion of  $m/z = 152$ . This compound coelutes with methamphetamine under the conditions used in this study. All of the peaks observed had a base peak of mass 58. The purity of this material was estimated as 85% using the peak area of the mass 58-extracted ion chromatogram. The EIMS of the major peak in this material (Fig. 3A) matched the spectrum of CMP published with the original article (1). Like methamphetamine, the EIMS of CMP is dominated by a  $m/z = 58$  ion corresponding to the ethylmethylamine fragment. The parent ion of  $m/z = 150$  corresponds to loss of one proton. Though not as strong as the peak in methamphetamine, a  $m/z = 91$  ion is observed. This ion most likely corresponds to a tropylium ion, (often indicative of an alkyl aromatic) even though the FTIR, UV, empirical formula, and the NMR data (all discussed below) of CMP are not consistent with the presence of an aromatic ring. This ion likely results from loss of  $\text{H}_2$  from the cyclohexadiene ring during fragmentation, although the precise mechanism is not clear.

The FTIR of this sample matches the spectrum published in 1997, but with weaker 700 and 748  $\text{cm}^{-1}$  absorbances (Fig. 4). The major impurity in the sample published in 1997 was methamphetamine, which shows strong absorbances at these frequencies. The pattern of absorbances observed in the 2000 to 3200  $\text{cm}^{-1}$  region of the FTIR are consistent with an amine salt.

The sample was evaluated using the color and crystal tests often used for the forensic analysis of methamphetamine. It gave a very weak orange color when tested with the Marquis reagent (17,18) and

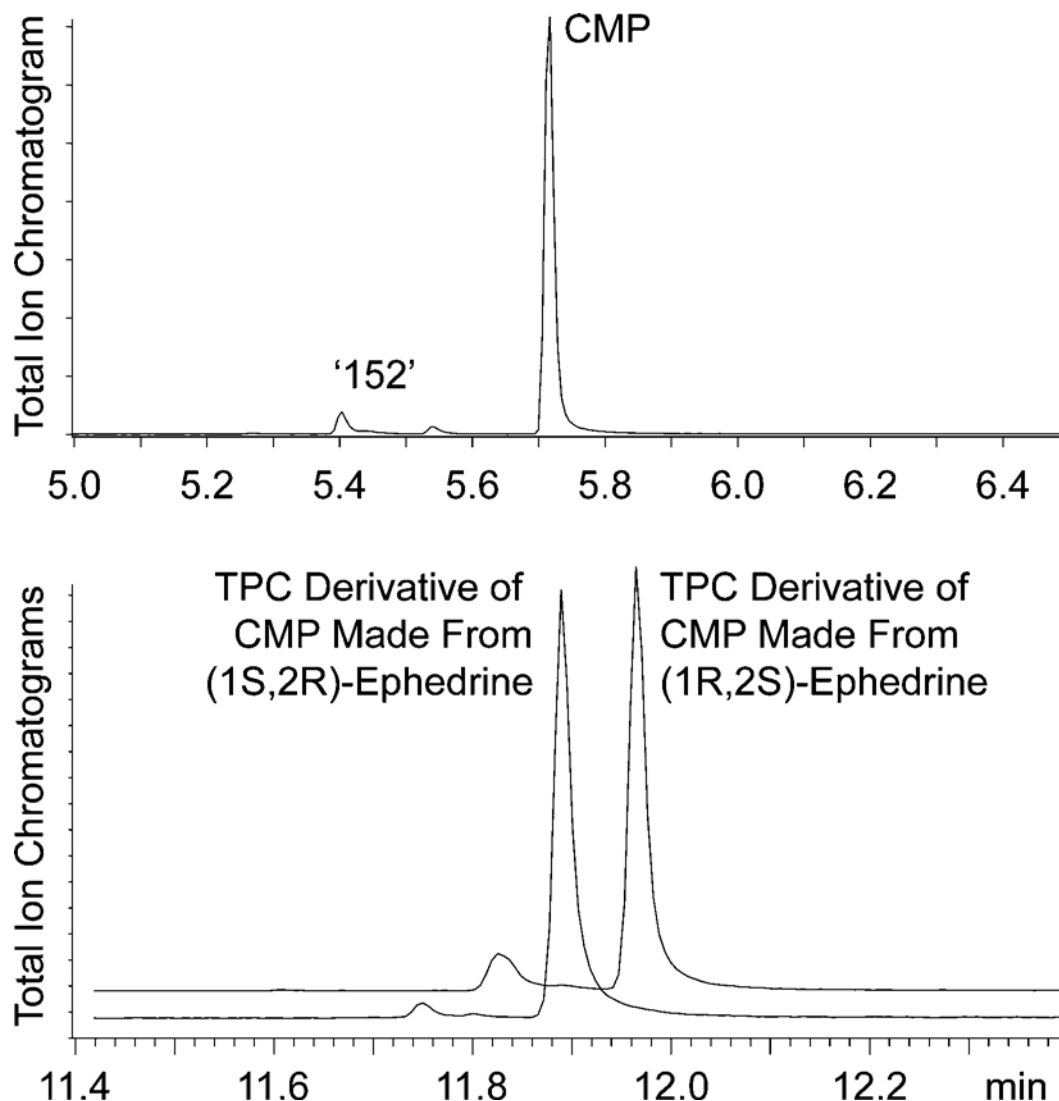


FIG. 2—Total ion chromatograms of A: the CMP enriched sample used to determine the chemical structure and B: the TPC derivatives of CMP samples generated from (1*S*, 2*R*)-Ephedrine and (1*R*, 2*S*)-Ephedrine.

gave a strong blue color with Simon's Test for secondary amines (17). The weak orange observed in the Marquis test may be the result of a trace amount of methamphetamine. The gold chloride in phosphoric acid crystal test for methamphetamine (19) gave numerous rosettes, but did not exhibit any calipers or caliper-like crystals. No blades or other crystals often observed when testing impure methamphetamine samples were noted.

Chemical derivatization using acetic anhydride and TPC followed by GC-MS analysis each yielded samples consistent with reaction with a single nucleophilic site (Fig. 3*B* and 3*C*). This is consistent with the proposed structure, the result of the secondary amine spot test, and the amine characteristics observed in the FTIR. TPC derivatives of separate samples of CMP prepared from (1*R*, 2*S*)-ephedrine and (1*S*, 2*R*)-ephedrine each give distinct, resolved peaks in the gas chromatogram (Fig. 2*B*). While this is not direct evidence of the stereochemical configuration of CMP, it does show that a single enantiomer is formed. As proposed mechanisms for this reduction do not involve the 2 position and racemization is not observed in methamphetamine synthesized under these conditions, it is unlikely that complete inversion of this stereocenter occurs. It is therefore reasonable to conclude that (2*S*)-methamphetamine will reduce to form (2*S*)-CMP.

The parent ion observed for CMP under electron impact mass spectrometry may not be representative of the molecular weight of this species as some small amines are known to show an M-1 peak in place of a molecular ion (20–22). An example is methamphetamine, which has a molecular weight of 149 amu but shows a parent ion of  $m/z = 148$ . Chemical ionization mass spectrometry, a softer ionization technique, offers a way to gain information about the molecular weight of this compound. This spectrum shows peaks at  $m/z = 58$  and 152 (data not shown). The mass 58 peak is the result of the favored fragmentation pathway that leads to the base peak seen in the EIMS spectrum. The  $m/z = 152$  peak corresponds to the  $MH^+$  ion of CMP indicating a molecular weight of 151 amu.

Ten peaks are observed in the  $^{13}C$  NMR spectrum (discussed below), suggesting that this compound contains ten carbons. The nitrogen rule and the observed odd molecular weight indicate an odd number of nitrogen atoms in the structure. The remaining 31 atomic mass units (amu) allows only for a single nitrogen atom. The assignment of a single nitrogen is consistent with the secondary amine spot test, the chemical derivatization experiments, the amine characteristics of the FTIR, and the formation of a precipitate after bubbling hydrogen chloride gas through the base extract of the reaction mixture during sample purification. The remaining

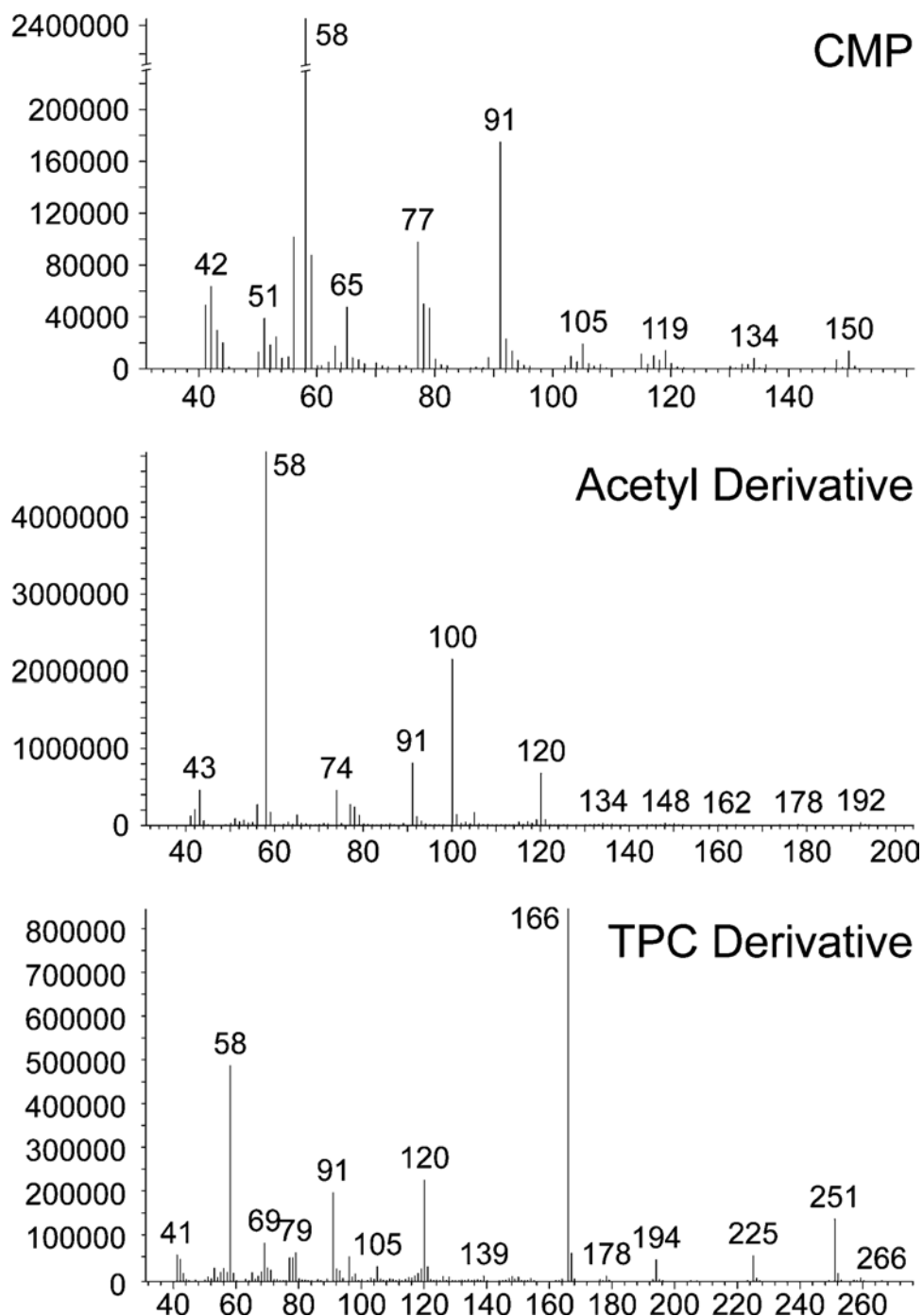


FIG. 3—Electron impact mass spectra of A: CMP (with an expanded vertical scale), B: the acetyl derivative of CMP, and C: the TPC derivative of CMP.

17 amu can be assigned to hydrogen atoms, giving the empirical formula of  $C_{10}H_{17}N$ , consistent with the tentative structure proposed in 1997. This formula gives a degree of unsaturation of 3, precluding the presence of an aromatic ring.

The UV spectrum of this sample shows a strong absorbance at 204 nm but no significant absorbance at 265 nm (data not shown). A weak absorbance at 261 nm would be expected if the aromatic ring was intact (20–22). A homoannular diene, such as a conjugated cyclohexadiene, would be expected to show a strong absorbance at approximately 268 nm (20–22). The lack of a peak at approximately 265 nm in the UV spectrum indicates that there is not a conjugated pi-system in this compound.

#### Nuclear Magnetic Resonance Experiments

The  $^1H$ -NMR spectrum is shown in Fig. 5. Any exchangeable protons, as are expected for the secondary amine previously predicted, will have undergone proton/deuterium exchange with the deuterated methanol solvent and will not appear in these experiments. The singlet at 4.87 ppm and small multiplet at 3.31 ppm correspond to solvent peaks.

The spectrum shows an immediately recognizable ABX pattern including the multiplet at 3.39 ppm and peaks between 2.15 and 2.55 ppm. This complex splitting pattern, expanded in the inset, occurs because the methylene protons are in diastereotopic

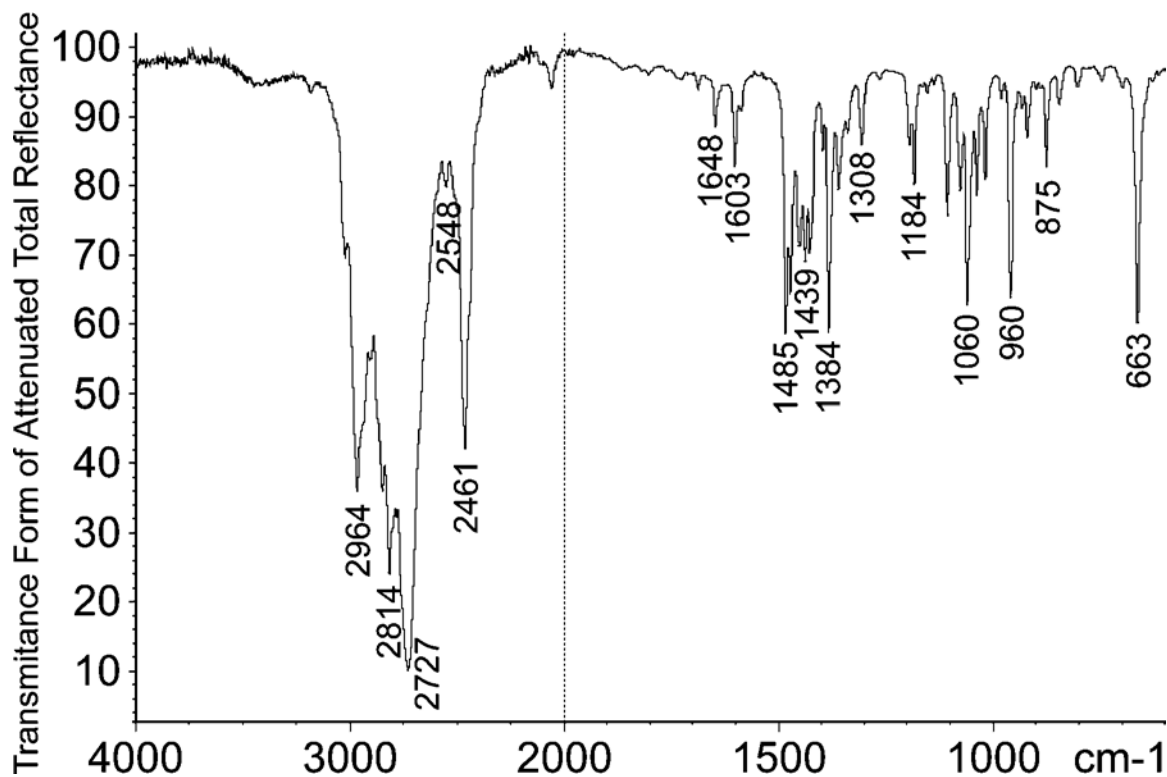


FIG. 4—The Fourier Transform Infrared Spectrum of the attenuated total reflectance data with the ATR correction.

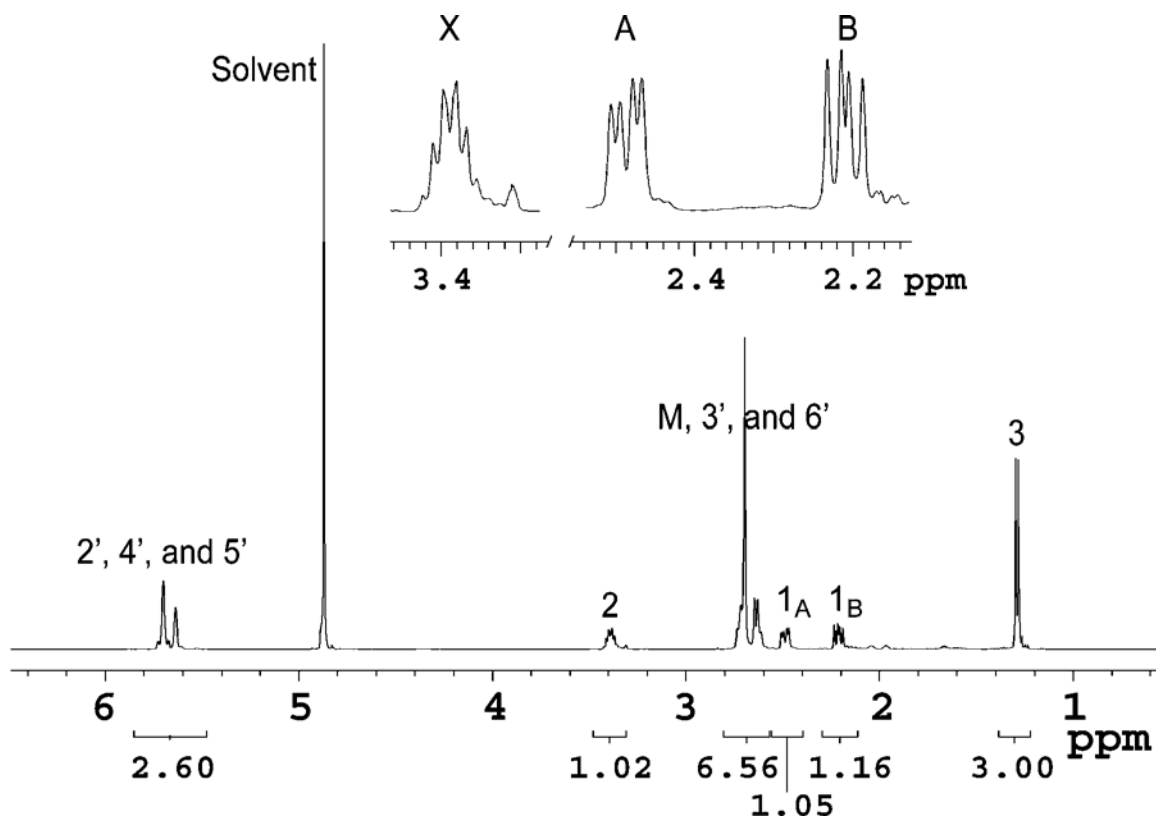


FIG. 5—The  $^1\text{H-NMR}$  (500 MHz, 8 transients) with peaks labeled using the notation presented in Fig. 1. The inset shows an expansion of the ABX pattern created by protons at the 1 and 2 positions.

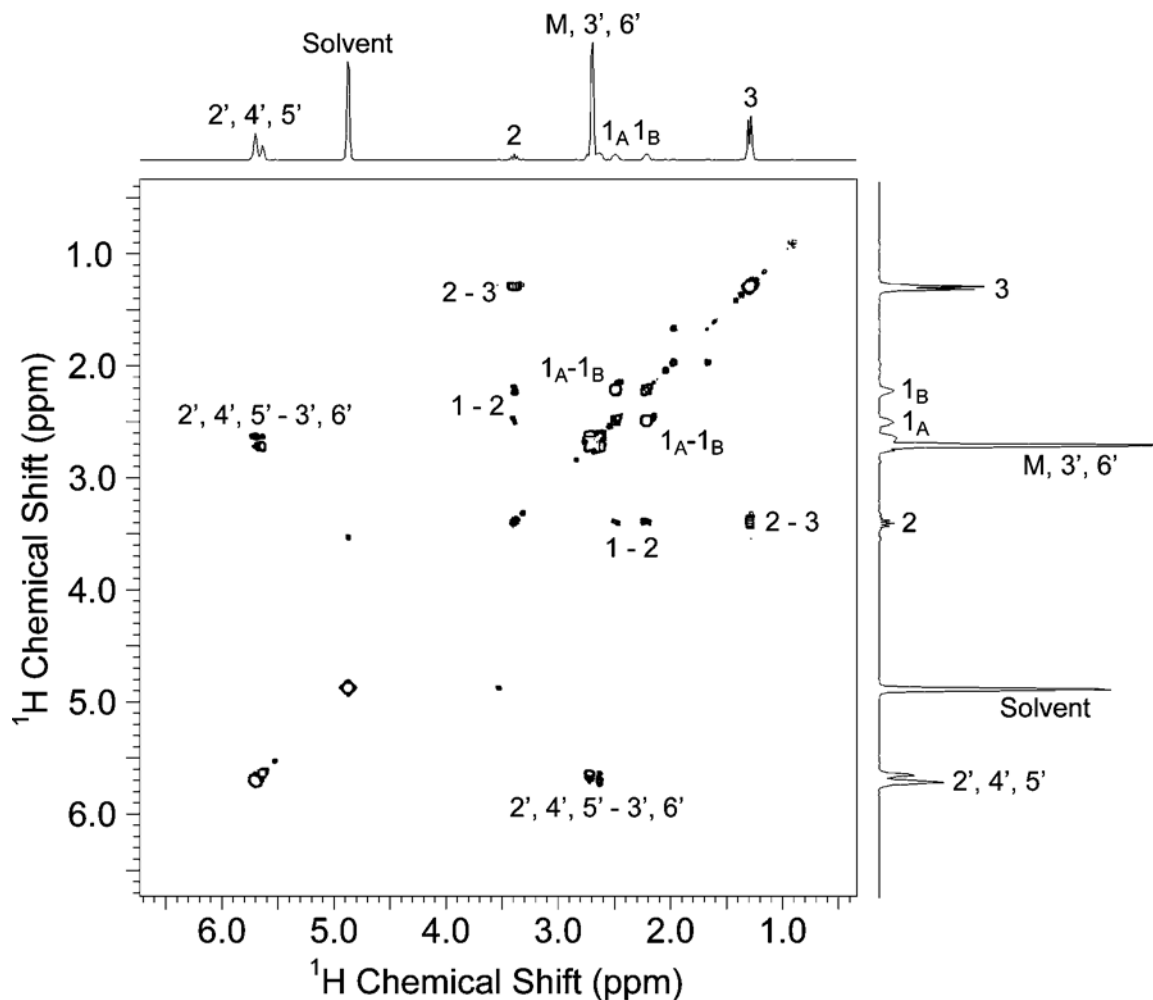


FIG. 6—Gradient Proton-Proton Correlation Spectroscopy (gCOSY, 128 increments with 1 scan per increment) with peaks labeled using the notation presented in Fig. 1.

environments created by an adjacent stereogenic methine carbon. The presence of this diastereotopic methylene group is supported by the resolved peaks observed in the TPC derivatization of CMP generated from the two-ephedrine enantiomers.

The multiplet between 5.63 and 5.72 ppm falls in the chemical shift range typical of vinylic protons. The integration of this peak indicates that there are only three vinylic protons. Coupled with the degree of unsaturation derived from the empirical formula, this suggests a cyclohexadiene group including one quaternary carbon. The UV data discussed previously eliminates the 1',3' conjugated cyclohexadiene, leaving only the 1',4'-cyclohexadiene ring as consistent with the experimental data.

The doublet at 1.29 ppm is consistent with a methyl group adjacent to a methine, and is assigned to the 3 position. The second-order pattern between 2.55 and 2.75 ppm overlaps a strong singlet at 2.70 ppm with an approximate integration of three protons. The singlet peak corresponds to a methylene group with no adjacent protons, and is assigned to the *N*-methyl group. The remaining four protons are assigned to the methylene groups on the cyclohexadiene ring.

The gCOSY NMR experiment (Fig. 6) is a two-dimensional pulse sequence that shows interactions of protons that share a scalar coupling. A  $^1\text{H}$ -NMR spectrum of the sample is shown on both the horizontal and vertical axis. A series of peaks along a diagonal from the lower left to the upper right results from each group of

protons regardless of neighboring groups. The spectrum is symmetrical showing the same crosspeaks on each side of the diagonal. Crosspeaks are observed for the five protons on the cyclohexadiene ring. As the peaks in the proton spectrum for these protons were not resolved, one group of crosspeaks is observed for all of these protons. As expected, crosspeaks are observed between the proton at position 2 with each of the diastereotopic protons at position 1 as well as the methyl doublet at position 3. This is consistent with the proposed structure and confirms the peak assignments made for the proton spectrum.

The  $^{13}\text{C}$  NMR experiment (Fig. 7) is a proton-decoupled single pulse experiment. As a result, single peaks result for each carbon in the structure. The group of peaks centered at 49.15 ppm is from the  $\text{CD}_3\text{OD}$  solvent. Ten distinct carbon peaks are observed, with three of them grouped closely around 125 ppm. This suggests that the structure contains ten carbons. The DEPT experiment (Fig. 8) can assist in assigning these peaks by revealing the carbon multiplicities that are lost when the  $^{13}\text{C}$  spectrum is proton-decoupled. The information from this experiment is conveniently displayed by the number of protons attached to each carbon. The solvent peaks and the carbon peak assigned to the 1' position do not appear in any of the DEPT spectra as they are not protonated. Further support of the 1',4'-cyclohexadiene ring assignment is the presence of only three protonated vinylic carbons. The chemical shifts of these three peaks are too close together to definitively assign. For similar reasons,

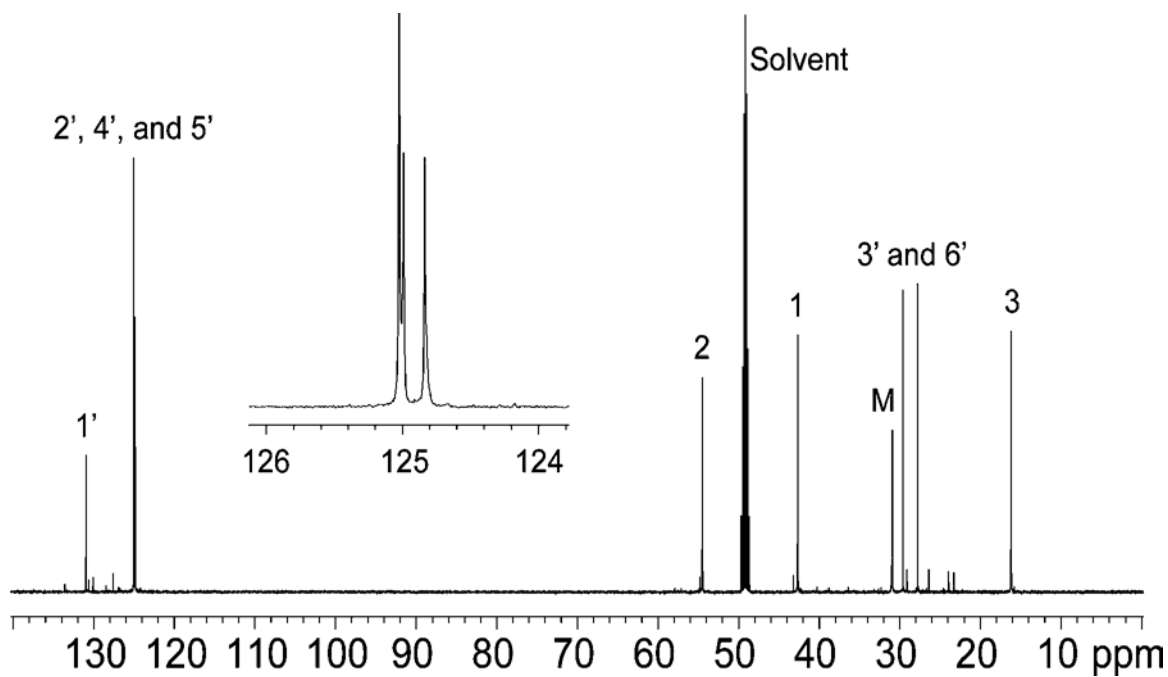


FIG. 7—The  $^{13}\text{C}$ -NMR (125 MHz, 512 transients) with peaks labeled using the notation presented in Fig. 1. The inset shows an expansion of the three aliphatic peaks at 125 ppm.

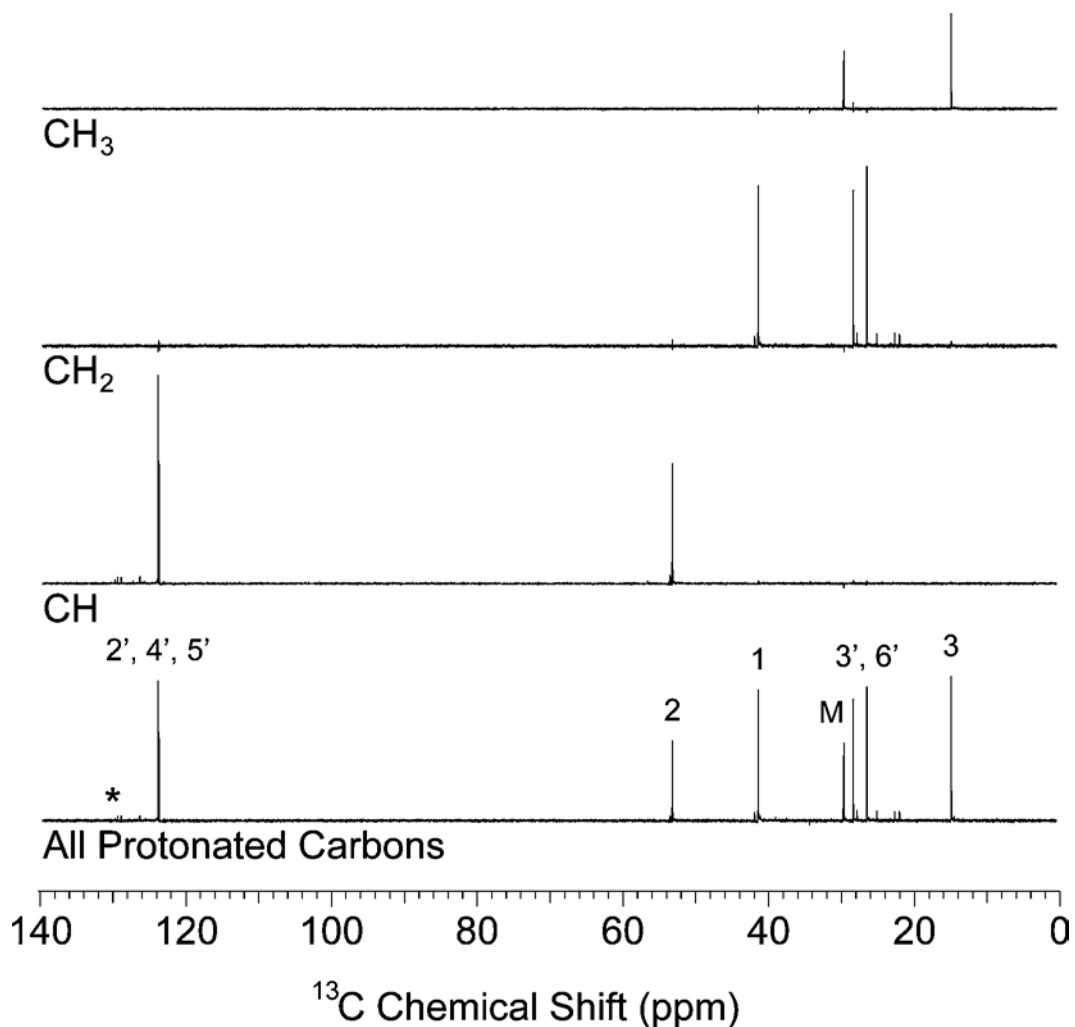


FIG. 8—Distortionless Enhancement via Polarization Transfer (DEPT, 128 transients). Note that the peak at 130 ppm in the  $^{13}\text{C}$  spectrum does not appear, indicating that it has no attached protons.

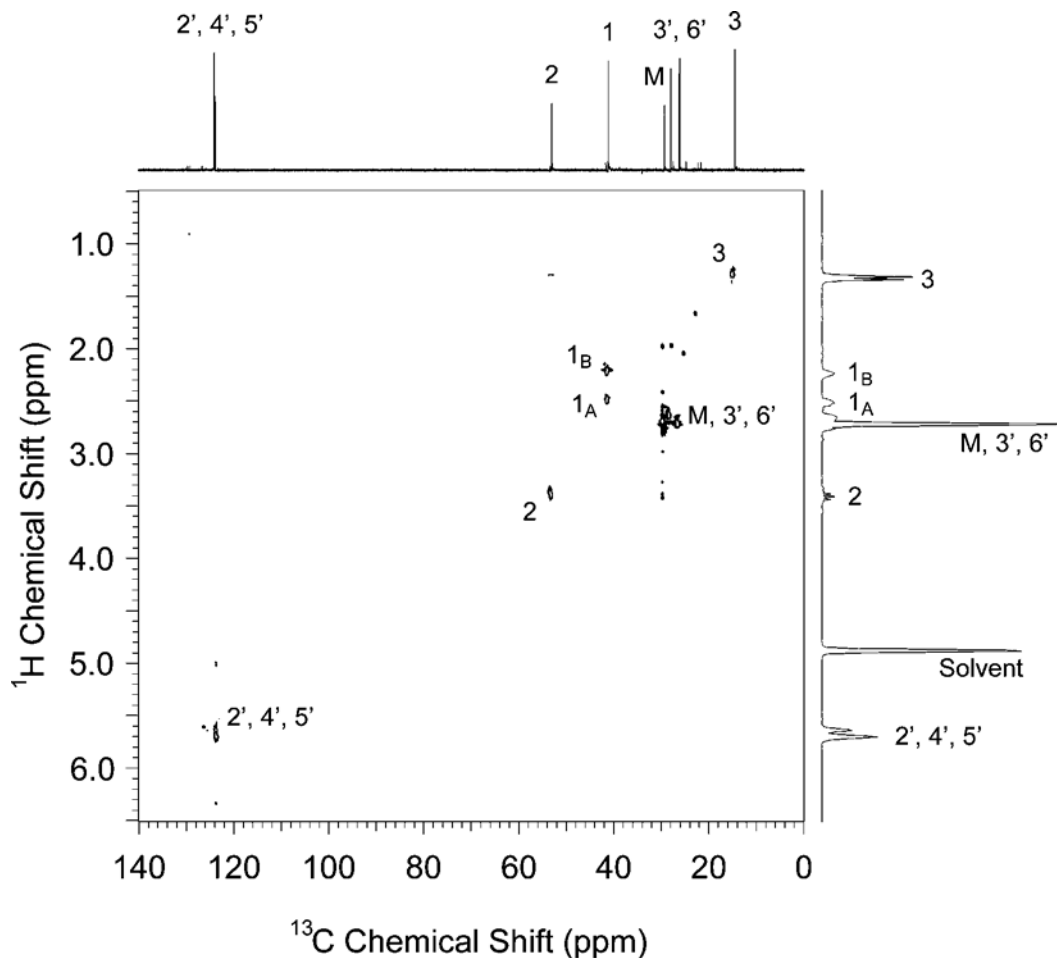


FIG. 9—Gradient Heteronuclear Single Quantum Correlation (gHSQC, 2x200 increments with 4 transients per increment) with peaks labeled using the notation presented in Fig. 1.

individual assignments are not given to the 3' and 6' peaks, though they can be distinguished from carbon 1 using predicted chemical shift values. The two-methyl groups can also be distinguished based on predicted chemical shifts. All of the peaks in the  $^{13}\text{C}$  and DEPT spectra show the expected number of attached protons and fall in the expected regions based on the proposed structure of CMP.

The gHSQC NMR experiment (Fig. 9) is a two-dimensional pulse sequence that shows interactions of protons and the carbons to which they are attached. The gHSQC is similar in concept to the HETCOR experiments that may be more familiar to the reader. It is more sensitive than the HETCOR experiments because the HSQC data is proton-detected rather than carbon-detected. The  $^1\text{H}$ -NMR spectrum is displayed on the vertical axis and the positive and negative projections of the  $^{13}\text{C}$ -NMR spectrum are displayed on the horizontal axis. These projections are the result of special pulse sequences similar to those used to extract the information presented in the DEPT spectrum. In this case, cross peaks are observed for protons and the carbon to which they are attached. All of the expected cross peaks are observed, thus confirming the peak assignments presented above.

## Conclusions

A sample enriched in CMP can be generated during the lithium-ammonia reduction of ephedrine by the addition of excess lithium in

the presence of isopropyl alcohol. With a simple workup, a sample of approximately 85% CMP was generated and used to collect structural data. This data allowed the structure of the major component to be determined from first principles. The determined structure, 1-(1',4'-cyclohexadienyl)-2-methylaminopropane, or CMP, matches the previously proposed structure of the byproduct formed during the lithium-ammonia reduction method.

While these experiments do not provide direct information on the absolute stereochemistry at the 2 position, they do show that a single stereoisomer is formed. Based on proposed reaction mechanisms, it is more likely that the stereochemistry at this position is preserved from the starting material than these conditions result in a total inversion of the stereochemistry at this center.

## Note

Since submission of this manuscript, Zvilichovsky and Gbara-Haj-Yahia published a note on the preparation and use of CMP as an intermediate for further organic transformations (23).

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Tami Kee and Lori Knops of the Washington State Patrol Crime Laboratory assisted in the review of this manuscript. Roger Ely provided scientific advice on the nomenclature used for CMP,

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