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Methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate: An Ecstasy Precursor Seized in Sydney, Australia

ABSTRACT: Five 44 gallon drums labeled as glycidyl methacrylate were seized by the Australian Customs Service and the Australian Federal Police at Port Botany, Sydney, Australia, in December 2004. Each drum contained a white, semisolid substance that was initially suspected to be 3,4-methylenedioxymethylamphetamine (MDMA). Gas chromatography–mass spectroscopy (GC/MS) analysis demonstrated that the material was neither glycidyl methacrylate nor MDMA. Because intelligence sources employed by federal agents indicated that this material was in some way connected to MDMA production, suspicion fell on the various MDMA precursor chemicals. Using a number of techniques including proton nuclear magnetic resonance spectroscopy (^{1}H NMR), carbon nuclear magnetic resonance spectroscopy (^{13}C NMR), GC/MS, infrared spectroscopy, and total synthesis, the unknown substance was eventually identified as methyl 3-[3′,4′(methylenedioxy)phenyl]-2-methyl glycidate. The substance was also subjected to a published hydrolysis and decarboxylation procedure and gave a high yield of the MDMA precursor chemical, 3,4-methylenedioxyphenyl-2-propanone, thereby establishing this material as a "precursor to a precursor."

KEYWORDS: forensic science, MDMA, ecstasy, precursor, glycidyl ester, 3,4-MDP-2-P

In December 2004 five 44 gallon drums labeled "glycidyl methacrylate" were seized by the Australian Crime Commission and Australian Customs. The material was believed to conceal 3,4-methylenedioxymethamphetamine (MDMA) for the purpose of international transport. However, the Marquis presumptive color test for MDMA produced a negative result. Custody of the material was transferred to the Australian Federal Police (AFP), who coordinated all subsequent investigations.

Glycidyl methacrylate is a monomer used in the production of coatings and resins (1,2). Initial GC–MS analyses of core samples taken from each drum indicated that the unknown material was not glycidyl methacrylate. The partial similarity between the name on the seized drums and a rarely encountered precursor of 3,4-methylenedioxyphenyl-2-propanone (3,4-MDP-2-P) described by Dal Cason (3), i.e., ethyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate (Fig. 2) caught the attention of one author. No mass spectral library data were available for this compound and the molecular weight was incorrect for the ethyl ester. It was, however, consistent with the methyl ester. To prove that the seized material was methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate (see Fig. 3), it was decided to carry out the hydrolysis and decarboxylation reaction steps shown in Fig. 2 using a sample of the seized material to verify that 3,4-MDP-2-P was produced. Proton nuclear magnetic resonance spectroscopy (^{1}H NMR) and carbon nuclear magnetic resonance spectroscopy (^{13}C NMR) studies were performed to confirm the suspected structure. Final proof of structure was the total synthesis of methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate and comparison of its spectral properties with those of the seized material.

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Experimental Section

Reagents and Standards

All reference standards, internal standards and surrogate standards used in the chemical profiling at the National Measurement Institute (NMI) were obtained from the NMI reference collection. Piperonal was also obtained from this reference collection. Sodium ethoxide was prepared in-house. Analytical grade methanol was obtained from Mallinckrodt Chemicals (Phillipsburg, NJ) and analytical grade dichloromethane and acetonitrile from Merck, Kilsyth, Vic., Australia. Sodium hydroxide and hydrochloric acid were obtained from APS Fine Chemicals, Seven Hills, NSW, Australia. Infrared spectroscopy grade potassium bromide was obtained from Aldrich, St. Louis, MO. Methyl-α-bromopropionate was obtained from Aldrich Chemicals, Castle Hill, NSW, Australia.

Reactions

1. Production of 3,4-MDP-2-P from the seized material by the method of Elks and Hey (4).

The seized material (35 g) was refluxed for 5 h with a solution of sodium hydroxide (10 g) in a 90% aqueous methanol solution (150 mL). The methanol was removed on a rotary evaporator and reaction mixture was poured into water (600 mL), acidified with concentrated hydrochloric acid and extracted with ether (2× 100 mL). The ether extracts were combined, dried over anhydrous sodium sulfate and the ether removed on a rotary evaporator leaving a light yellow liquid (21 g). To the light yellow liquid obtained was added a trace of copper powder and the mixture heated to 180°C for 18 h.

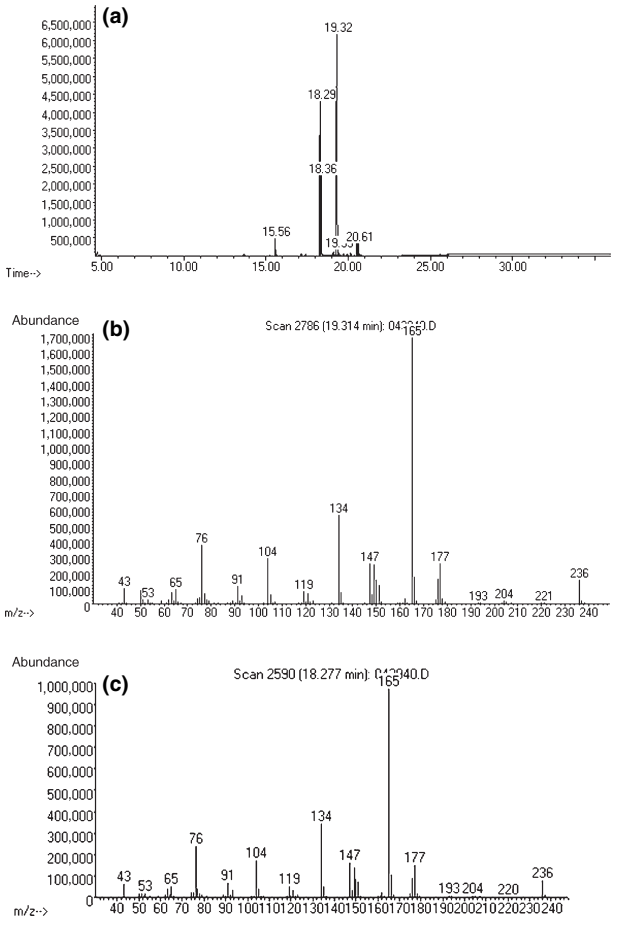


FIG. 1—*Total ion chromatogram of the seized material (a) and mass spectra obtained from chromatographic peaks at 18.29 (c) and 19.32 min (b).*

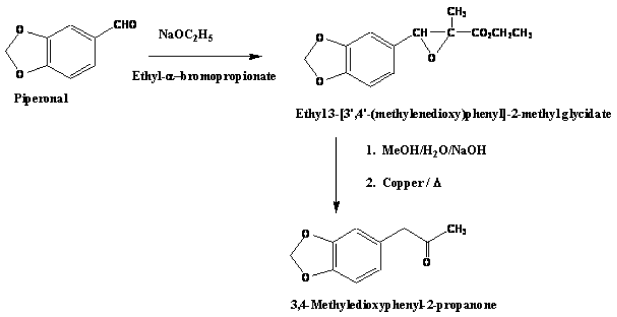


FIG. 2—*Reaction pathway from piperonal to 3,4-MDP2P via ethyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate.*

2. Preparation of methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate using the method of Elks and Hey (4).

Sodium ethoxide (23 g) was added over 4 h to a mixture of piperonal (50 g) and methyl α-bromopropionate (61 g). The mixture was stirred and cooled in an ice bath during the addition and stirred overnight. The reaction mixture was added to ice water, acidified with dilute acetic acid, and extracted with ether. The ether solution was washed with sodium carbonate solution and dried over sodium sulfate, and the ether was removed on a rotary evaporator leaving a yellowish viscous liquid.

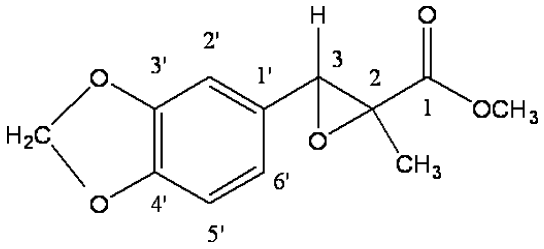


FIG. 3—*Proposed structure of the seized material.*

Infrared Spectroscopy

All infrared spectra were recorded on a Biorad 3000MX Excalibur Series Fourier transform infrared spectrometer (Bio-Rad Informatics Sadtler, Philadelphia, PA). The sample (5 mg) was mixed with potassium bromide in a mortar. Background scans: 10, scans: 10, resolution: 4, scan range: 500–4000/cm, scan type: absorbance.

Nuclear Magnetic Resonance Spectroscopy

All ^{1}H NMR and ^{13}C NMR were acquired on a Bruker DMX500 spectrometer (Bruker-Biospin, Rheinstetten, Germany) in $CDCl_3$. Approximately 10 mg of sample was dissolved in ~0.7 mL of deuterated chloroform and placed into a 5 mm NMR sample tube. For ^{1}H NMR the field strength was 500.133 MHz, receiver gain was 128, and the number of scans taken was 16. The spectra were acquired at 298 K with a sweep width of 10 ppm and a prescan delay of 5.5 μsec. For ^{13}C NMR spectra the field strength was 125.8 MHz, the receiver gain 4096, and the number of scans taken was 1024. The spectra were acquired at 298 K with a sweep width of 210.3 ppm and a prescan delay of 4.5 μsec. Nuclear Overhauser Effect SpectroscopY experiments were performed to distinguish the *trans* from the *cis* diastereomer.

Melting Point

All melting points were acquired on a Perkin-Elmer Differential Scanning Calorimeter Pyris (Perkin-Elmer, Waltham, MA) and are uncorrected.

Gas Chromatography

All analyses were performed on an Agilent Technologies 6890N gas chromatograph interfaced with an Agilent 5973 MSD (Agilent J&W, Santa Clara, CA). A 30 m × 0.32 mm × 1 μm DB5-MS column (Agilent) was employed using helium carrier gas in the constant-flow-rate mode. Injection port temperature was 280°C and the MS interface temperature was 300°C. Oven temperature program was 50°C (1 min) to 300°C (10 min) at 10°C/min. Injections (1 μL) were made in the split mode (25:1) and a mass range of 50–500 was scanned.

Preparative Liquid Chromatography

Preparative liquid chromatography was performed using a Shimadzu LC-10AT liquid chromatograph equipped with a SIL-10AD autoinjector and a SPD-10A UV–VIS detector (Shimadzu Corporation, Columbia, MD). A Waters SymmetryPrep C_{18} 150 mm × 19 mm × 7 μm column (Waters, Millford, MA) (P/N: WAT066240) was used with a mobile phase of 65% methanol/water. A portion of seized material (1 g) was dissolved in 10 mL of the mobile phase and a series of 500 μL injections were

made. Fraction 1 (*cis*-diastereomer) was collected between 10 and 15 min and fraction 2 (*trans*-diastereomer) between 17 and 23 min. Methanol was removed from each fraction leaving cloudy, aqueous suspensions. The suspensions were extracted with ether, which was dried over anhydrous sodium sulfate and then removed on a rotary evaporator to leave pale yellow oily residues that solidified on standing to give off-white crystals.

Results and Discussion

The total ion chromatogram (TIC) from the gas chromatography–mass spectroscopy (GC/MS) analysis of the seized material is shown in Fig. 1 and exhibits two major chromatographic peaks at 18.29 and 19.32 min and a number of smaller peaks. The mass spectra corresponding to the chromatographic peaks at 18.29 and 19.32 min are also shown in Fig. 1. These mass spectra are very similar indicating that they may be diastereomers. It was also obvious that the seized material could not be glycidyl methacrylate, which has a molecular weight of 142 and a mass spectrum with a base peak of 39. The apparent molecular weight of the as-yet-unknown substance was 236. Retrospective searching of mass spectral libraries revealed no matches. Two of the smaller peaks had mass spectra consistent with piperonal and 3,4-MDP-2-P.

Dal Cason's paper on MDA analogs and homologs (3) discusses the interrelationship of various precursors used in the synthesis of MDMA (4–9). One synthetic route described in this paper involves the formation of ethyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate from piperonal and its subsequent transformation into 3,4-MDP2P following the procedure of Elks and Hey (4) as shown in Fig. 2. The structure of this precursor has a molecular weight of 250 amu, while the molecular weight of the seized substance appeared to be 236 amu. This discrepancy is explicable if the seized material was the methyl ester depicted in Fig. 3 rather than ethyl ester. Furthermore, a molecule containing the glycidoate moiety should exist as a mixture of *trans* and *cis* stereoisomers, which would explain the presence of the two chromatographic peaks having the same mass spectra.

It was hypothesized that the seized material was the methyl analogue, i.e., methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate. When the seized material (35 g) was subjected to the hydrolysis and decarboxylation method described by Elks and Hey (4), 3,4-MDP2P was obtained, and confirmed by GC–MS. The TIC obtained from the GC/MS analysis of the liquid, shown in Fig. 4*a*, revealed only one major peak at 15.57 min. The mass spectrum corresponding to the chromatographic peak at 15.57 min is shown in Fig. 4*b*. The retention time and mass spectrum of the peak matches that of a certified reference standard of 3,4-MDP2P shown in Fig. 4*c*.

The next step in proving that the seized substance was methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate was its total synthesis from piperonal using Elks and Hey's method (4) shown in Fig. 2, substituting methyl α-bromopropionate for ethyl α-bromopropionate. When this was done as described in Step 2 reactions, a yellowish viscous liquid was obtained. The TIC of this liquid is shown in Fig. 5. Also shown are the mass spectra corresponding to the chromatographic peaks at 18.27 and 19.26 min. The retention times of these peaks match those in the TIC of the seized material shown in Fig. 1. The mass spectra obtained from these peaks also match the mass spectra of the same peaks in the TIC of the seized material shown in Fig. 1. The infrared spectra of the synthetic methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate and the seized material are shown in Fig. 6. The two infrared spectra match closely. Each spectrum demonstrates a strong absorbance at 1730/cm consistent with the ester carbonyl group stretching. Strong absorbances are also seen from 1440 to 1500/cm, which is consistent with the carbon–carbon stretching of aromatic rings. The strong absorbance at 1240/cm is consistent with the ring breathing frequency of the epoxide group and the absorbance at 1040/cm is consistent with the symmetric C–O–C stretching of the methylenedioxy group.

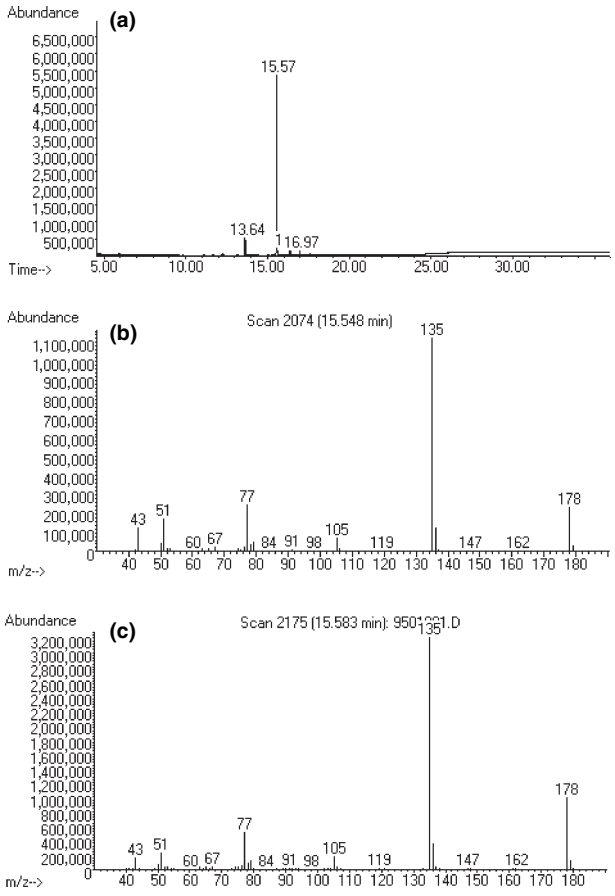


FIG. 4—*Total ion chromatogram (a) of the hydrolysis and decarboxylation product obtained from the seized material; mass spectrum (b) of the chromatographic peak at 15.57 min; mass spectrum (c) of a certified reference standard of 3,4-MDP2P.*

Preparative liquid chromatography was used to separate the two diastereomers represented by the two chromatographic peaks in both the seized material and the synthetic methyl 3-[3′,4′-(methylenedioxy)phenyl]-2′-glycidate. The characterization data for each diastereomer are as follows:

trans-diastereomer (corresponds to GC peak at RT 19.32 min Fig. 1)

m.p. 59–61°C, (found C 60.8%, H 4.9%. $C_{12}H_{12}O_5$, requires C 61.0%, H 5.1%). υ_{max} (KBr/cm) 3072, 3000, 2955, 2923, 1742, 1496, 1449, 1293, 1252, 1197, 1166, 1033, 922, 814, 739, 560. δ_H (500 MHz, $CDCl_3$) 6.81–6.77 (3H, m), 5.97 (2H, s), 4.24 (1H, s), 3.81 (3H, s), 1.33 (3H, s). δ_C (125 MHz, $CDCl_3$) 171.3, 147.7, 147.6, 127.5, 120.3, 108.2, 107.0, 101.2, 62.3, 59.9, 52.8, 12.6. *m/z* (GC/MS, EI) 236 (8), 165 (100), 134 (39), 104 (24), 76 (46).

cis-diastereomer (corresponds to GC peak at RT 18.29 min in Fig. 1).

The *cis*-diastereomer was obtained as an oil which later solidified. A sharp melting point could not be obtained. Found C

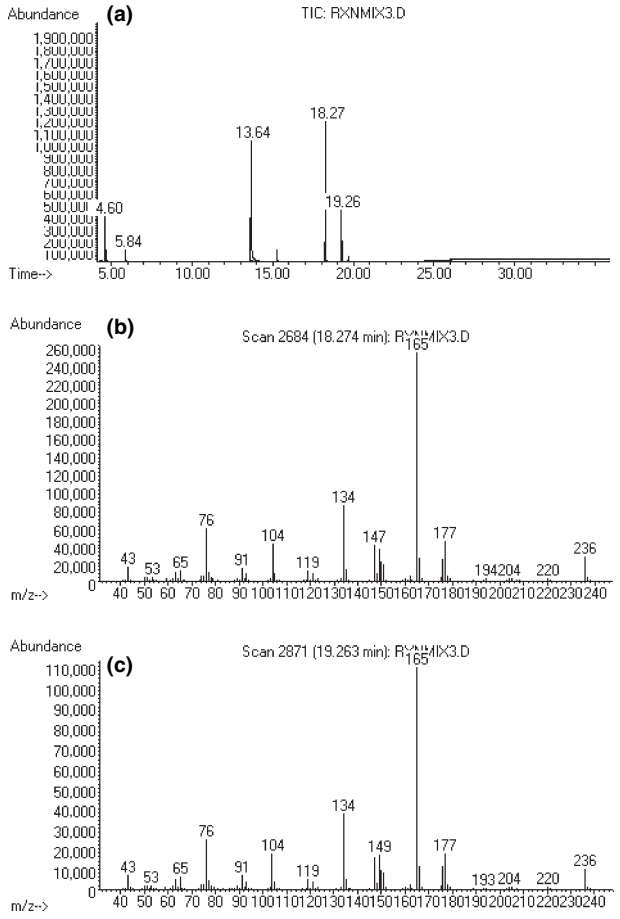


FIG. 5—*Total ion chromatogram (a) and mass spectra (b and c) of the reaction mixture resulting from the synthesis of methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate using the method of Elks and Hey (4).*

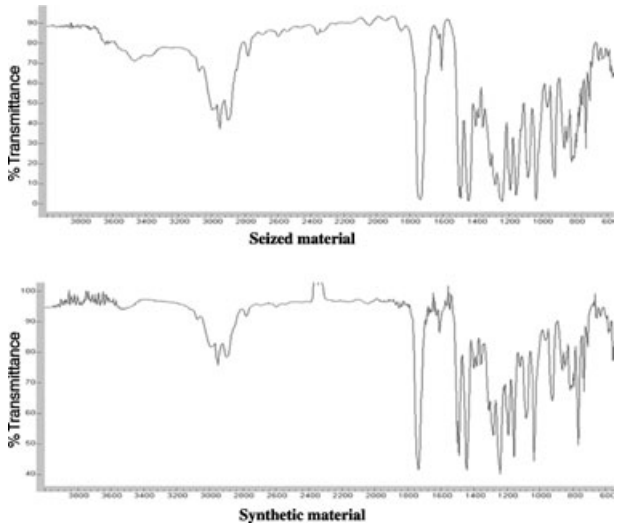


FIG. 6—*Infrared spectra of the seized material (a) and the synthetic methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate (b).*

61.0%, H 5.1%. $C_{12}H_{12}O_5$, requires C 61.0%, H 5.1%). υ_{max} (KBr/cm) 3072, 3000, 2955, 2923, 1742, 1496, 1449, 1293, 1252, 1197, 1166, 1033, 922, 814, 739, 560. δ_H (500 MHz, $CDCl_3$) 6.74–6.83 (3H, m), 5.94 (2H, s), 3.92 (1H, s), 3.55 (3H, s), 1.70 (3H, s). δ_C (125 MHz, $CDCl_3$) 169.0, 147.6, 147.5, 127.5, 120.0, 108.0, 106.7, 101.1, 63.8, 63.2, 52.1, 19.5. *m/z* (GC/MS, EI) 236 (8), 165 (100), 134 (39), 104 (24), 76 (46).

TABLE 1—*Proton nuclear magnetic resonance spectroscopy data for the stereoisomers detected in the seized material. The bracketed figures represent the chemical shifts for the two diastereomers in the synthesized methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate.*

Chemical shift (ppm)			
GC peak at 18.29 min	GC peak at 19.37 min	No. of protons	Assignment
1.70 (1.70)	1.33 (1.33)	3	–C–**CH_3**
3.55 (3.55)	3.81 (3.81)	3	–CO–O–**CH_3**
3.92 (3.92)	4.24 (4.24)	1	Ph–**C–H**
5.94 (5.94)	5.97 (5.97)	2	–O–**CH_2**–O
6.74–6.83 (6.74–6.83)	6.77–6.81 (6.77–6.81)	3	Aromatic protons

The ^{1}H NMR data for both diastereomers in both the seized material and in the synthesized methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate are given in Table 1. It is apparent from this data that the seized material matches the synthetic product.

The initial structural elucidation of the diastereomer of methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate that eluted at a retention time of 19.32 min under the stated GC conditions (Fig. 1) was based on a series of 1D and 2D NMR experiments comprising ^{1}H NMR, ^{13}C NMR, heteronuclear multiple quantum correlation (HMQC) and heteronuclear multiple bond correlation (HMBC) and the data is presented in Table 2. The ^{1}H NMR spectrum of the material isolated as a white crystalline solid, confirmed the presence of nine aliphatic protons at δ 1.33, 3.81, 4.24 and 5.97 ppm and three aromatic protons at δ 6.77–6.81. The singlet at δ 1.33 ppm, a singlet that integrated for three protons, was assigned as the 2-methyl group. The singlet at δ 3.81 ppm, a singlet integrating for three protons, was assigned as the ester OMe group. The singlet at δ 4.24 ppm was assigned as H3. The singlet at δ 5.97 ppm, a singlet integrating for two protons, was assigned as the methylenedioxy group. The coupling pattern of the three aromatic protons at δ 6.77–6.81 is typical of a 1,3,4-trisubstituted phenyl

TABLE 2—*Proton nuclear magnetic resonance spectroscopy and carbon nuclear magnetic resonance spectroscopy data obtained on the stereoisomer trans-methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate present in the seized material.*

Assignment	Proton (ppm)	Carbon (ppm)	Heteronuclear multiple bond correlation
Me	1.33	12.6	171.3
OMe	3.81	52.8	171.3
2		59.9	
3	4.24	62.3	59.9, 107.0, 120.3, 127.5, 171.3
CH_2O_2	5.97	101.2	147.6, 147.7
2′	6.77	107.0	147.7, 147.6, 127.5, 120.3, 107.0, 62.3*
5′	6.81	108.2	147.7, 147.6, 127.5, 120.3, 107.0, 62.3*
6′	6.77	120.3	147.7, 147.6, 127.5, 120.3, 107.0, 62.3*
1′		127.5	
3′ or 4′		147.6	
3′ or 4′		147.7	
C=O		171.3	

*Individual correlations of protons 2′, 5′, and 6′ (6.77, 6.81, and 6.77 ppm, respectively) cannot be distinguished due to the similarity of chemical shifts.

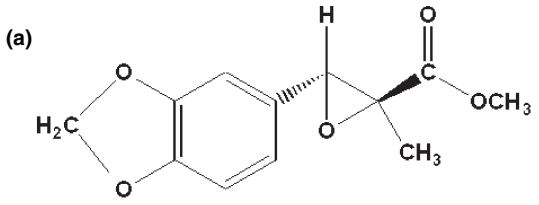
group. The pattern observed for the methylenedioxy proton together with the aromatic protons is very diagnostic for a 3,4-methylenedioxyphenyl group. The ^{13}C NMR spectrum confirmed the presence of five aliphatic carbons at δ 12.6, 52.8, 59.9, 62.3, 101.2 ppm as well as six aromatic carbons, δ 107.0, 108.2, 120.3, 127.5, 147.6, 147.7 ppm and one carbonyl at δ 171.3 ppm. The resonance at δ 101.2 ppm is diagnostic of the carbon in the methylenedioxy group. The final structure was verified by $^{2-3}J_{CH}$ correlated 2D NMR experiments (Table 2). From the HMBC results, there is a strong correlation between the proton at δ 4.24 ppm and three aromatic carbons. This result confirms that the resonance at δ 4.24 ppm is a benzylic proton. The correlation between this proton and one aliphatic carbon and one carbonyl, together with the absence of any coupling in the aliphatic region, confirm the structure as methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate.

The diastereomer eluted at 18.29 min under the stated GC conditions and did not crystallize was nevertheless obtained pure by preparative liquid chromatography. It was also examined by 1D and 2D NMR (^1H, ^{13}C, HMQC, and HMBC) and this data is similarly presented in Table 3. The ^1H NMR spectrum of this material also confirmed the presence of nine aliphatic protons at δ 1.70, 3.55, 3.92, and 5.94 ppm and three aromatic protons at δ 6.74–6.83. The singlet at δ 1.70 ppm, a singlet that integrated for three protons was assigned as the 2-methyl group. The singlet at δ 3.55 ppm, a singlet integrating for three protons was assigned as the ester OMe group. The singlet at δ 3.92 ppm was assigned as H3. The singlet at δ 5.94 ppm, a singlet integrating for two protons was assigned as the methylenedioxy group. The coupling pattern of the three aromatic protons at δ 6.74–6.83 is typical of the 1,3,4-trisubstituted phenyl group. The pattern observed for the methylenedioxy proton together with the aromatic protons is very diagnostic for a 3,4-methylenedioxyphenyl group. The ^{13}C NMR spectrum confirmed the presence of five aliphatic carbons at δ 19.5, 52.1, 63.2, 63.8, 101.1 ppm as well as six aromatic carbons, δ 106.7, 108.0, 120.0, 127.5, 147.5, 147.6 ppm, and one carbonyl at δ 169.0 ppm. The resonance at δ 101.1 ppm is diagnostic of the carbon in the methylenedioxy group. The final structure was verified by $^{2-3}J_{CH}$ correlated 2D NMR experiments (Table 2). From the HMBC results, there is a strong correlation between the proton at δ 3.92 ppm and three aromatic carbons. This result confirms that the resonance at δ 3.92 ppm is a benzylic proton. The correlation between this proton and one aliphatic carbon and the one methyl group, together with the absence of any coupling in the aliphatic region, also confirms the structure as methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate.

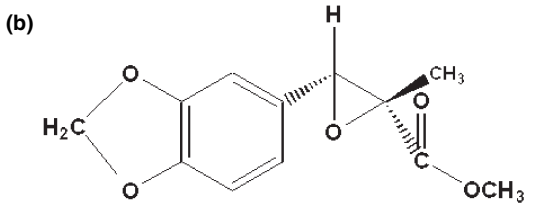
TABLE 3—*Proton nuclear magnetic resonance spectroscopy and carbon nuclear magnetic resonance spectroscopy data obtained on the stereoisomer cis-methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate present in the seized material.*

Assignment	Proton (ppm)	Carbon (ppm)	Heteronuclear multiple bond correlation
Me	1.70	19.5	63.2, 169.0
OMe	3.55	52.1	169.0
2		63.2	
3	3.92	63.8	19.5, 63.2, 106.7, 120.0, 127.5
CH_2O_2	5.94	101.1	147.6, 147.7
2′	6.83	106.7	108.0, 147.6, 147.7, 120.0, 106.7, 63.8*
5′	6.74	108.0	108.0, 127.5, 147.7, 147.6
6′	6.82	120.0	108.0, 147.6, 147.7, 120.0, 106.7, 63.8*
1′		127.5	
3′ or 4′		147.5	
3′ or 4′		147.6	
C=O		169.0	

*Individual correlations of protons 2′ and 6′ (6.83 and 6.82 ppm, respectively) cannot be distinguished due to the similarity of chemical shifts.

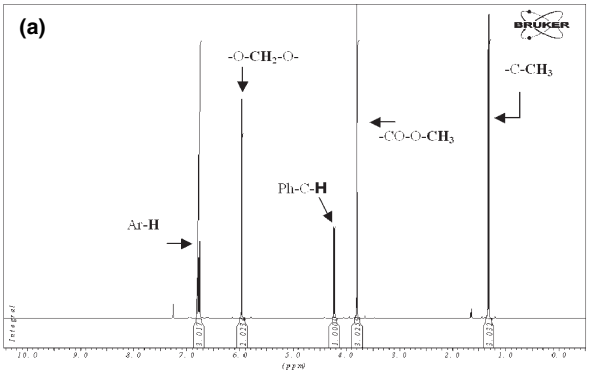


Trans - methyl 3-[3',4'-(methylenedioxy)phenyl]-2-methyl glycidate

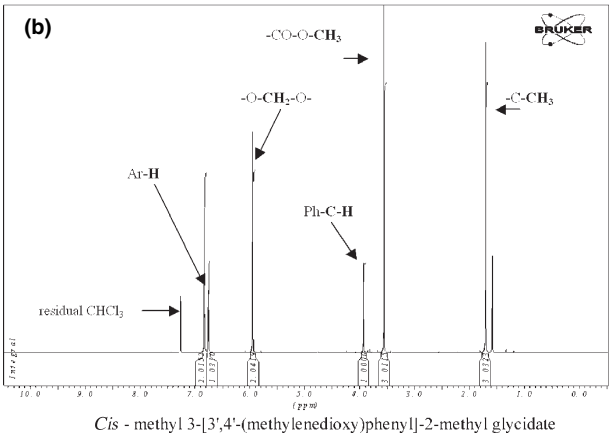


Cis - methyl 3-[3',4'-(methylenedioxy)phenyl]-2-methyl glycidate

FIG. 7—*(a) trans-methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate (b) cis-methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate*



Trans - methyl 3-[3',4'-(methylenedioxy)phenyl]-2-methyl glycidate



Cis - methyl 3-[3',4'-(methylenedioxy)phenyl]-2-methyl glycidate

FIG. 8—*(a)^1H NMR spectrum of the trans-methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate. (b)^1H NMR spectrum of the cis-methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-methyl glycidate.*

Nuclear Overhauser Effect SpectroscopY experiments were performed to determine which diastereomer was the *trans* form (Fig. 7*a*) and which was the *cis* form (Fig. 7*b*). The results of these experiments proved that the diastereomer eluting at 18.29 min in the TIC (Fig. 1) was the *cis* isomer while the *trans* isomer eluted at 19.32 min. The ^{1}H NMR spectra for both the *cis*- and the *trans*-methyl 3-[3′,4′(methylenedioxy)phenyl]-2-glycidate are presented in Fig. 8.

Conclusion

The identity of the unknown material in the seizure was established as methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate. This material was synthesized in our laboratory using a published method. ^{1}H NMR and ^{13}C NMR spectra, mass spectra and infrared spectra of the synthetic material, and the seized material all match. Although the ethyl analog is described by Dal Cason (3) its use is a comparatively unusual route to MDMA. It is believed that this is the first time that methyl 3-[3′,4′-(methylenedioxy)phenyl]-2-glycidate has been found in Australia.

Acknowledgments

The authors are indebted to the Australian Customs Service and the AFP for providing the samples.

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