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Synthesis by-products from the *Wacker* oxidation of safrole in methanol using ρ-benzoquinone and palladium chloride

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Abstract

This paper reports the identification of a number of by-products, which are produced during the *Wacker* oxidation of safrole to 3,4-methylenedioxyphenyl-2-propanone (MDP2P) using ρ-benzoquinone and palladium chloride when methanol is utilised as the solvent. Also described is the retrieval of these compounds from illicit samples from a clandestine laboratory, which was uncovered in South Australia in September 2003.

Keywords: Safrole; MDP2P; 3,4-Methylenedioxymethamphetamine; By-products; *Wacker* oxidation

1. Introduction

Most of the 3,4-methylenedioxymethamphetamine (MDMA) available in Australia is produced in clandestine laboratories in The Netherlands and Belgium [1]. We have recently observed a resurgence of clandestine laboratories engaged in the manufacture of MDMA within South Australia, with four ecstasy laboratories having been uncovered in the years 2003 and 2004. *Wacker* oxidation of safrole to 3,4-methylenedioxyphenyl-2-propanone (MDP2P) using palladium chloride, ρ-benzoquinone and methanol was being utilised at one of the laboratories (Scheme 1).

Many unknown components were encountered in the gas chromatograms (GCs) of samples taken from the scene and we have since attempted to unambiguously identify some of these unknown compounds.

It was thought that some of these compounds may be route specific and as such, their identification may aid in the determination of the method by which an illicit drug might be manufactured. For example linking or discriminating information might be required to establish a connection between a drug seizure found in possession of a suspect with another batch of drugs or with the source laboratory. Manufacturing information also gives law enforcement agencies an indication as to which chemicals should be subject to sales control and monitoring. A number of papers have been devoted to the route specific markers associated with the various different synthetic routes to 3,4-methylenedioxyamphetamine and 3,4-methylenedioxymethamphetamine but at this stage it appears that the production of the precursor MDP2P via the *Wacker* oxidation of safrole has received little attention [2–11].

The goals of this work included the synthesis of the anticipated by-products and their comparison with the unidentified by-products from the *Wacker* oxidation of safrole and then followed by screens for the presence of these compounds in illicit preparations.

2. Experimental procedure

2.1. Chemicals and reagents

All solvents used in this work were of analytical grade and were purchased from Aldrich Chemical Company. All of the reagents used in this work were acquired from Aldrich Chemical Company.

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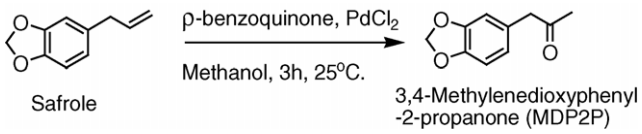


Scheme 1.

2.2. Instrumentation

Sample analysis was effected with gas chromatography–mass spectrometry (GC–MS), viz. a Hewlett Packard 6890 plus gas chromatograph equipped with a Hewlett Packard 5973 mass selective detector (MSD) and electronic pressure programming. Helium was used as a carrier gas; the column was a 15 m × 0.25 mm × 0.25 μm DB-1 capillary.

The mass spectrometer operated from 30 to 450 amu in electron impact (EI) mode with an ionization energy of 70 eV. A solvent delay of 1.50 min was applied. The injector temperature was 300 °C. The initial column temperature was 90 °C for 4 min and then ramped at 45 °C over 4.67 min.

2.3. Synthesis procedures

2.3.1. Wacker oxidation of safrole in methanol using ρ-benzoquinone

This conversion was done by roughly following the procedure of *Methyl Man* [12]: a solution containing safrole (17.8 g, 162.1 mmol) and methanol (10 mL) was added dropwise over a period of 60 min to a solution containing palladium chloride (0.2 g, 1.1 mmol), ρ-benzoquinone (15 g, 138.8 mmol), methanol (40 mL) and distilled water (5 mL). The reaction mixture was stirred for 3 h, by which time GC analysis indicated that all of the safrole had been consumed. After this time the insoluble solid material was filtered from the reaction mixture and 10% hydrochloric acid solution (170 mL) was added. The aqueous mixture was extracted with dichloromethane (3 × 30 mL) and the organic extracts were combined. The organic phase was then washed with saturated sodium bicarbonate solution (2 × 30 mL), saturated sodium chloride solution (3 × 30 mL) and 5% sodium hydroxide solution (3 × 30 mL). The organic phase was then dried ($MgSO_4$), filtered and evaporated to give a straw yellow oil. Analysis by GC indicated a product mixture represented by Fig. 1.

2.3.2. Wacker oxidation of safrole in ethanol using ρ-benzoquinone

The above procedure was duplicated but ethanol replaced methanol as the solvent.

3. Results and discussion

MDP2P was synthesised in our laboratory by following the procedure of *Methyl Man* [12]. After a 3 h reaction time and work-up, GC analysis indicated that all of the safrole starting material had been consumed (Fig. 1).

The oxidation of safrole to MDP2P is accompanied by the reduction of ρ-benzoquinone to hydroquinone and this compound is observed in the crude reaction mixture prior to alkaline work-up. Significantly, 4-methoxyphenol was also noted as a reduction product of ρ-benzoquinone, but was also lost to the aqueous phase during alkaline work-up. The presence of this compound has been implicated in the synthesis of *para*-methoxyphenyl-2-propanone [13].

Isosafrole (A) and the major product MDP2P (B) were recognisable products amongst a number of unknown minor products from the reaction (unknowns 1–5, Fig. 1). Unknown compounds 1–5 are the subjects of this communication.

3.1. Unknown 1

The retention time of unknown compound 1 was 4.90 min. The base peak (165 amu) and the molecular ion (194 amu) suggested that unknown 1 was consistent with a reduced safrole structure with a methoxy group positioned at the benzylic position (Fig. 2).

1-(3,4-Methylenedioxyphenyl)-1-methoxypropane (1) was synthesised for comparison with unknown 1 (Fig. 3).

1-(3,4-Methylenedioxyphenyl)-1-methoxypropane (1) was synthesised by reaction of ethyl magnesium bromide with

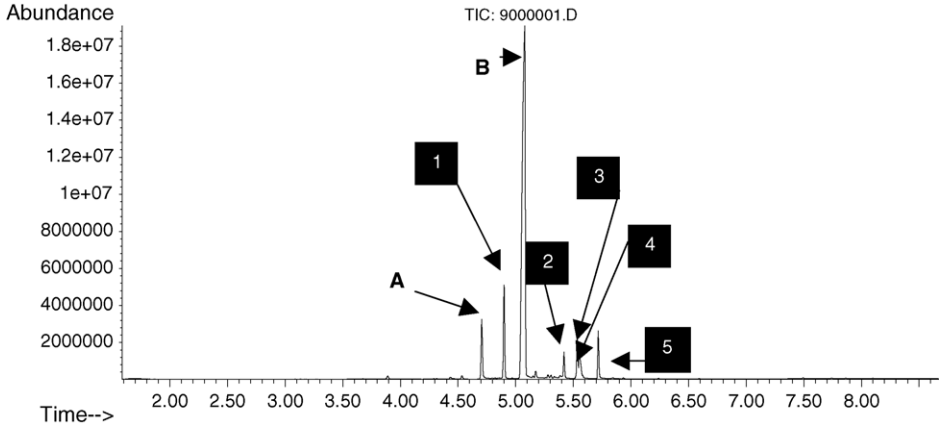


Fig. 1. GC of the *Wacker* oxidation of safrole after work-up and a 3 h reaction time. Peak identities; (A) isosafrole, 1: unknown 1, (B) MDP2P, 2: unknown 2, 3: unknown 3, 4: unknown 4, 5: unknown 5.

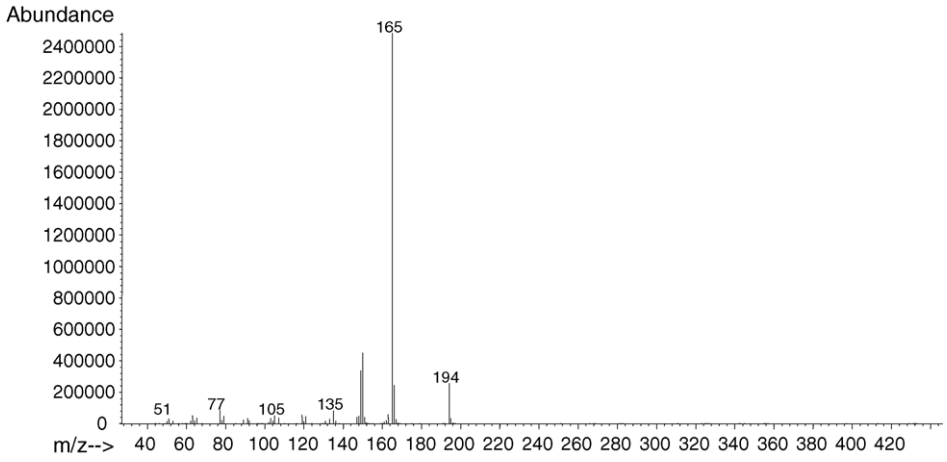
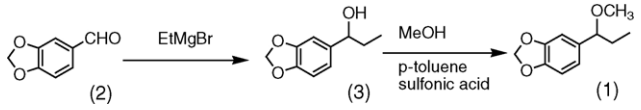


Fig. 2. Mass spectrum of unknown compound 1, at retention time = 4.90 min.



Scheme 2.

piperonal (2), which gave 1-(3,4-methylenedioxyphenyl)-1-hydroxypropane (3), which was then converted to the methoxy derivative by solvolysis of (3) in methanol with ρ-toluene-sulfonic acid (Scheme 2).

The mass spectrum of synthesised (1) was identical with the mass spectrum of unknown 1. The retention time was checked by co-injection of synthesised (1) with the product mixture from the *Wacker* oxidation of safrole and this resulted in the symmetrical enhancement of the peak eluting at 4.90 min. This co-injection procedure was applied to all of the unknown compounds mentioned in this report. We concluded that the identity of unknown 1 was 1-(3,4-methylenedioxyphenyl)-1-methoxypropane (1).

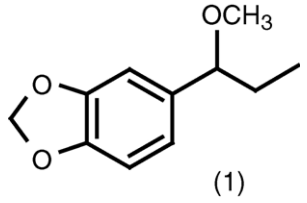


Fig. 3. Proposed structure of unknown compound 1.

3.2. Unknown 2

The retention time of unknown 2 was 5.42 min. The mass spectrum of this compound contained a base peak of 165 amu and a molecular ion of 208 amu, which suggested that this compound contained a methoxy substituent at the benzylic position as well as a carbonyl group (Fig. 4).

1-(3,4-Methylenedioxyphenyl)-1-methoxypropan-2-one (4) was synthesised for comparison with unknown 2 (Fig. 5).

Bromination of MDP2P (5) with bromine resulted in the predominant formation of the singly brominated species, 1-(3,4-methylenedioxyphenyl)-1-bromopropan-2-one (6) and 1-(3,4-methylenedioxyphenyl)-3-bromopropan-2-one (7). Initially it was thought that direct substitution of (6) with sodium

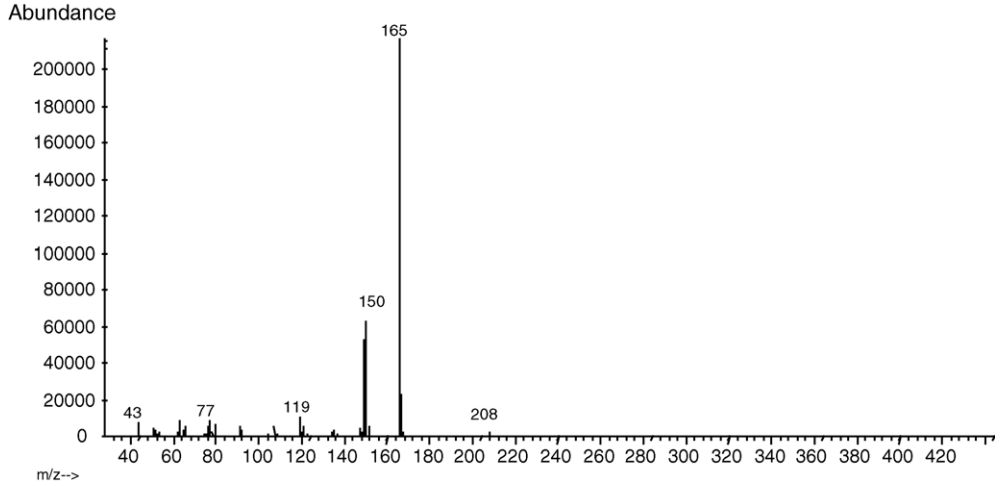


Fig. 4. Mass spectrum of unknown compound 2, at retention time = 5.42 min.

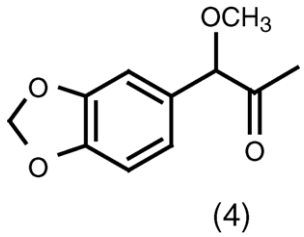
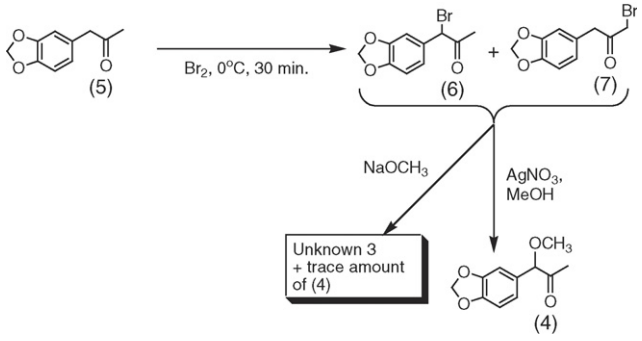


Fig. 5. Proposed structure of unknown compound 2.

methoxide might give (4). When a mixture of (6) and (7) was refluxed with sodium methoxide a compound was predominantly formed with identical mass spectral fragmentation and retention time to the third unknown compound which eluted at 5.54 min as well as trace amounts of (4). Solvolysis of (6) in methanol produced larger amounts of (4) and unreacted (7), but high yields of (4) (along with unreacted (7)) were obtained when (6) was treated with silver nitrate in methanol (Scheme 3).

The mass spectrum and retention time of synthesised (4) was identical with unknown 2. We concluded that the identity of unknown 2 was 1-(3,4-methylenedioxyphenyl)-1-methoxypropan-2-one (4). Compound (4) has recently been identified in MDP2P prepared by the per-acid oxidation of isosafrole [14].



Scheme 3.

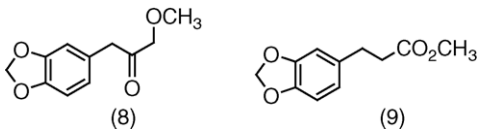


Fig. 7. Proposed structures of unknown compound 3.

Therefore, (4) cannot be considered as route specific for this oxidation process.

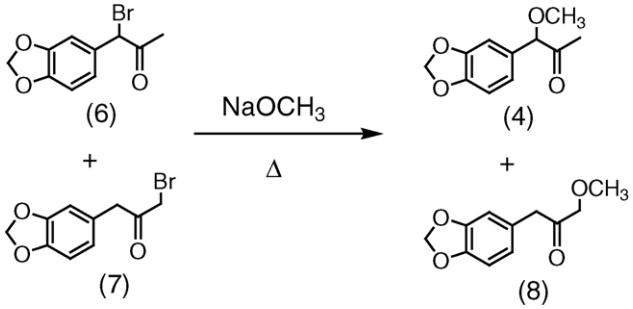
3.3. Unknown 3

The base peak of 135 amu of unknown 3, which eluted at 5.54 min suggests a vacant benzylic position but the molecular ion of 208 amu suggests the presence of a methoxy group and a carbonyl group in the molecule (Fig. 6).

Possible structures of the third unknown compound are shown in Fig. 7.

As mentioned above when a mixture of (6) and (7) was refluxed with sodium methoxide a compound was produced with identical mass spectral fragmentation and retention time as unknown compound 3. Direct substitution of (7) with methoxide may produce (8) (Scheme 4).

An alternative mechanism involving *Favorskii* re-arrangement of (6) or (7) with methoxide through the cyclopropanone



Scheme 4.

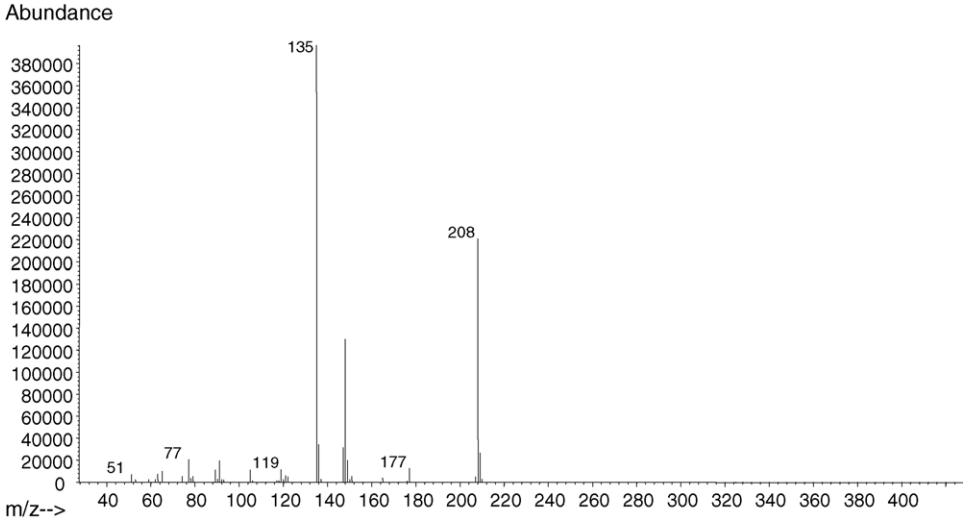
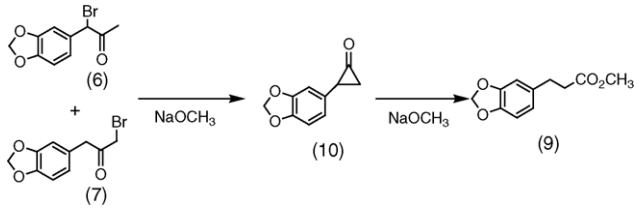


Fig. 6. Mass spectrum of unknown compound 3, at retention time = 5.54 min.

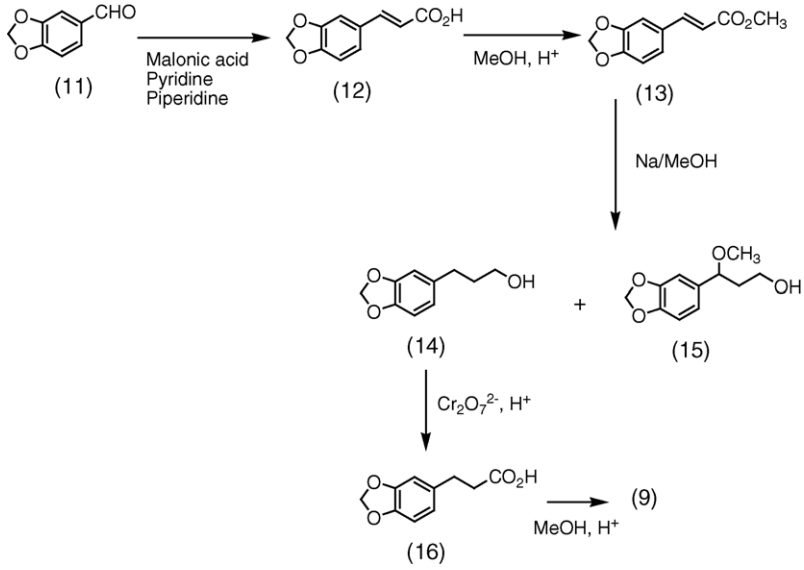


Scheme 5.

intermediate (10) to (9) could also have occurred [15] (Scheme 5). Since (8) or (9) could both theoretically be produced from (7) (when heated with sodium methoxide) then more work was required to unambiguously identify the structure of unknown compound 3.

Further identification of unknown compound 3 was required despite the production of a compound with identical features during the reaction of sodium methoxide with a mixture of compounds (6) and (7) (above). Methyl 3-(3,4-methylenedioxyphenyl)propanoate (9) was synthesised as per Scheme 6. The conjugated ester (13) was produced by *Knoevenagel* condensation of piperonal (11) with malonic acid, followed by esterification of the conjugated acid (12). Reduction of the conjugated ester (13) with sodium in methanol resulted in the formation of 3-(3,4-methylenedioxyphenyl)propanol (14) and the *Michael* type addition product 3-(3,4-methylenedioxyphenyl)-3-methoxypropanol (15). The alcohol (14) was then oxidised with acidified dichromate to the acid (16), which was then esterified to give (9).

The spectral features and elution time of synthesised (9) was identical with the third unknown compound. We concluded that the third unknown compound was methyl 3-(3,4-methylenedioxyphenyl)propanoate (9).



Scheme 6.

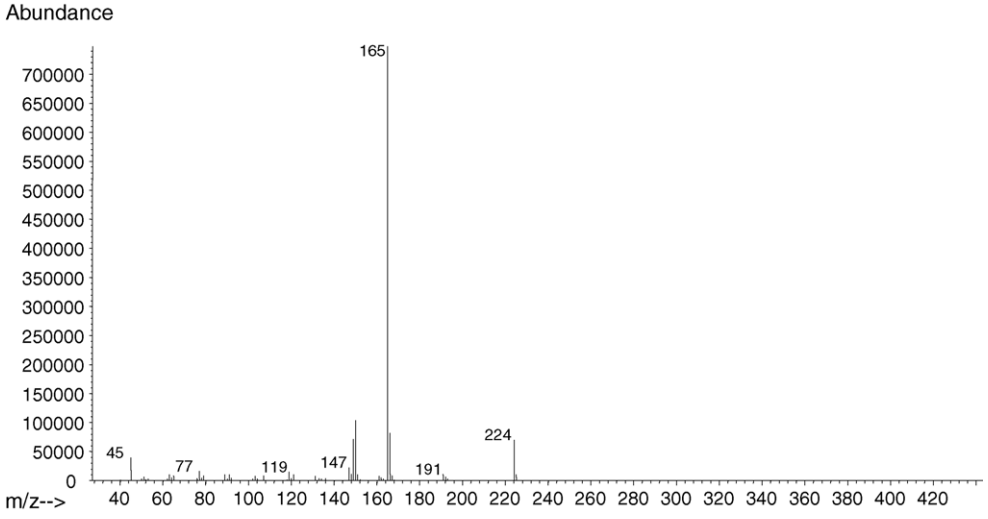


Fig. 8. Mass spectrum of unknown compound 4, at retention time = 5.56 min.

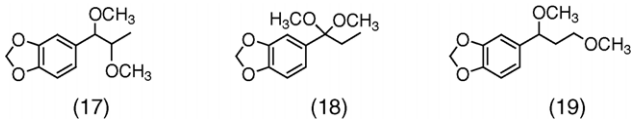
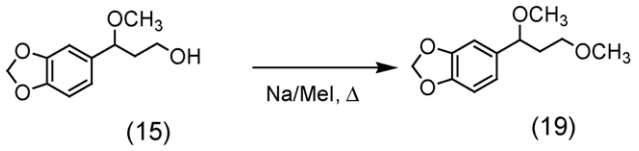


Fig. 9. Proposed structures of unknown compound 4.



Scheme 7.

3.4. Unknown 4

Unknown compound 4 eluted at 5.56 min and contained fragment ions consistent with a methoxy group at the benzylic position, but in this case the parent ion suggested that another methoxy group was present (Fig. 8).

Possible structures of the fourth unknown compound are shown in Fig. 9.

Both diastereomers of (17) were synthesised and found to have very similar mass spectral fragmentation with unknown compound 4, but neither diastereomer of (17) matched the retention time. The dimethylacetal (18) of 3,4-methylenedioxyphenylpropan-1-one (MDP1P) was synthesised in trace amounts but neither the mass spectrum nor the elution time matched the fourth unknown compound. 1-(3,4-Methylenedioxyphenyl)-1,3-dimethoxypropane (19) was synthesised from (15) by methylation of the sodium salt with methyl iodide in anhydrous 1,2-dimethoxyethane (Scheme 7).

The mass spectrum and retention time of synthesised (19) were identical with those of the fourth unknown compound, which eluted at 5.56 min and we concluded that the fourth unknown compound was 1-(3,4-methylenedioxyphenyl)-1,3-dimethoxypropane (19).

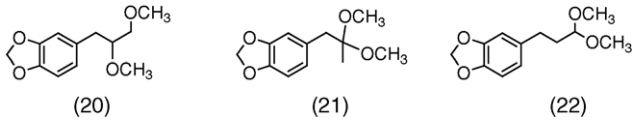


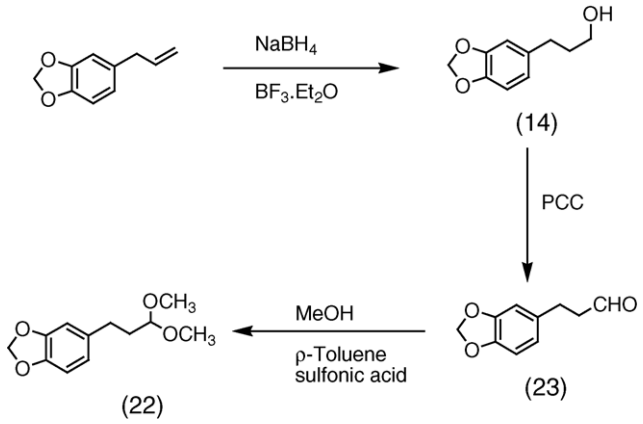
Fig. 11. Proposed structures of unknown compound 5.

3.5. Unknown 5

Unknown compound 5 eluted at 5.71 min and contained a base peak of 135 amu and a parent ion of 224 amu, which suggested that the structure of the final unknown compound contained an unsubstituted benzylic position but the molecular ion suggested that the unknown compound contained two methoxy groups (Fig. 10).

Possible structures of the fifth unknown compound are shown below in Fig. 11.

Dimethyl safrole glycol (20) was synthesised by permanganate oxidation of safrole to give safrole glycol followed by methylation with sodium hydride and methyl iodide, but neither retention time nor the mass spectrum of (20) matched unknown compound 5. The dimethyl acetal (21) of MDP2P was



Scheme 8.

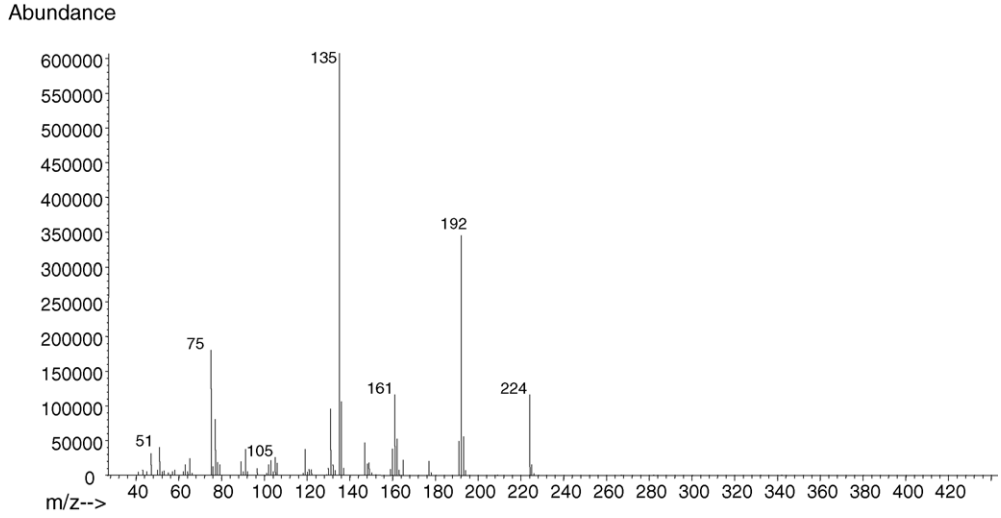


Fig. 10. Mass spectrum of unknown compound 5, at retention time = 5.71 min.

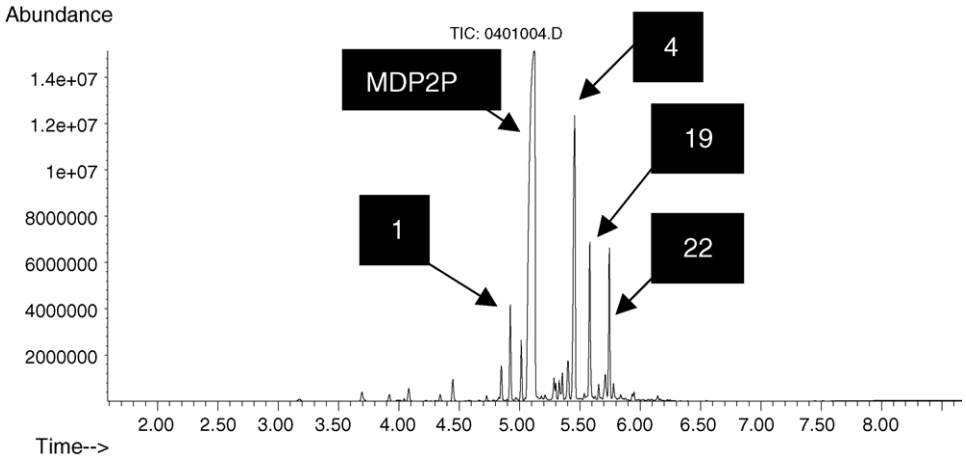


Fig. 12. GC plot of crude MDP2P located at the MDMA laboratory.

Fig. 13. Mitsubishi design tablets, which were being produced at the clandestine laboratory in Willunga (2003).

synthesised and neither the retention time nor the mass spectrum of (21) matched those of unknown compound 5.

Compound (22) was synthesised by initial hydroboration of safrole to give 3-(3,4-methylenedioxyphenyl)propanol (14) which was then selectively oxidised to 3-(3,4-methylenedioxyphenyl)propanal (23) with pyridinium chlorochromate followed by the formation of the dimethylacetal (22) in methanol with ρ-toluenesulfonic acid (Scheme 8).

The mass spectrum and retention time of synthesised (22) were identical with those of the fifth unknown compound, which eluted at 5.71 min and we concluded that the fifth unknown compound was 3-(3,4-methylenedioxyphenyl)-1,1-dimethoxypropane (22).

The next phase of this study involved the investigation of the retrieval of some of these by-product chemicals from samples taken from a clandestine laboratory, which was employing the *Wacker* oxidation of safrole to manufacture MDP2P. Police uncovered the laboratory in September 2003 at a rural property in Willunga, about a 1-h drive south of Adelaide, South Australia. A large array of chemicals and equipment was present at the scene. The vast majority of the chemicals had nothing to do with drug production, but some of the chemicals, including *sassafras oil*, pointed to MDMA manufacture. A sample of crude MDP2P from the laboratory was analysed for the presence of any of the neutral by-products mentioned previously; the chromatogram is presented in Fig. 12.

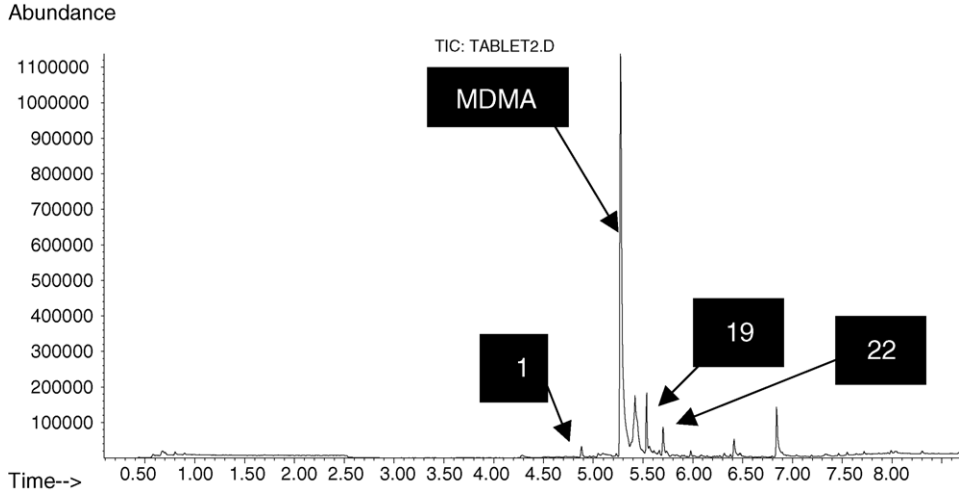


Fig. 14. GC SPME profile of a *Mitsubishi* tablet using a 100 μm polydimethylsiloxane fibre.

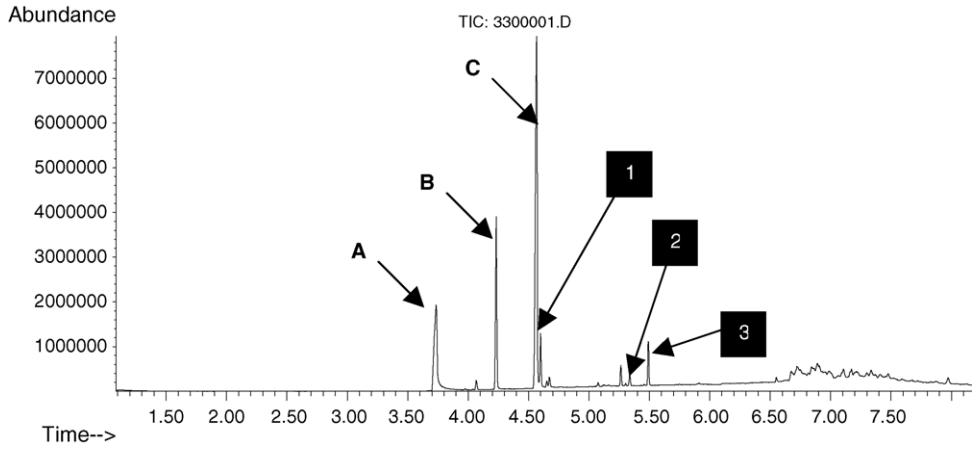


Fig. 15. GC plot of crude MDP2P manufactured by the *Wacker* oxidation of safrole in ethanol. Peak identities; (A) piperonal, (B) isosafrole, (C): MDP2P, 1: unknown 1, 2: unknown 2, 3: unknown 3.

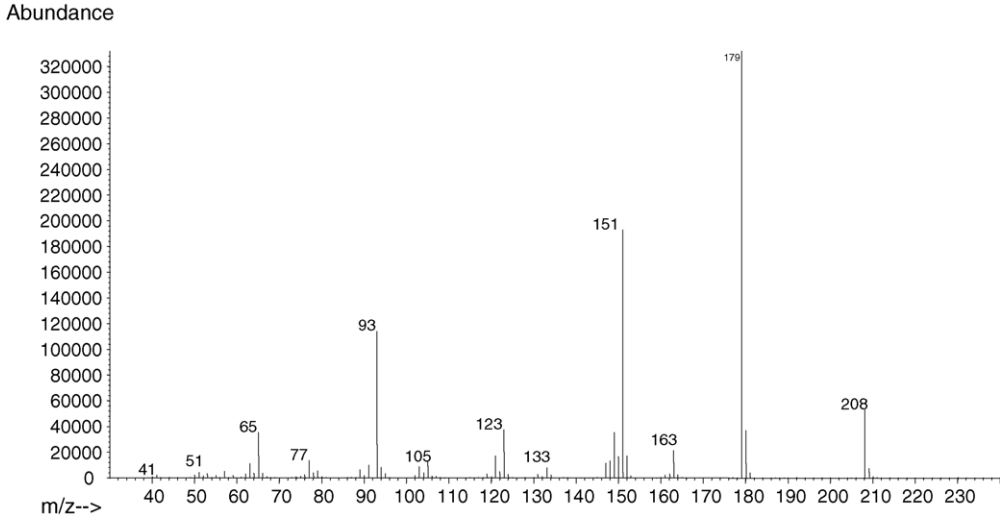


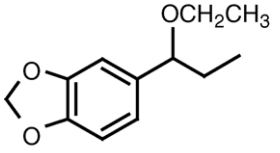
Fig. 16. Mass spectrum of unknown compound 1 from the *Wacker* oxidation of safrole in ethanol.

The chromatographic result shows the presence of 1-(3,4-methylenedioxyphenyl)-1-methoxypropane (1), 1-(3,4-methylenedioxyphenyl)-1-methoxypropan-2-one (4), 1-(3,4-methylenedioxyphenyl)-1,3-dimethoxypropane (19) and 3-(3,4-methylenedioxyphenyl)-1,1-dimethoxypropane (22). The presence of these compounds in this combination may be considered as markers for the *Wacker* oxidation of safrole to MDP2P.

In addition to crude MDP2P, MDMA powder and MDMA tablets were located. The MDMA had been synthesised using the alumina amalgam method where nitromethane was employed as the nitrogen source. The MDMA tablets were impressed with the *Mitsubishi* logo (Fig. 13).

The tablets were investigated for the presence of the compounds described here. It was expected that they could be present due to their neutral character, but this would be dependent on the purification processes, which had been used to prepare the MDMA tablets. The laboratory operator had used vacuum distillation for purification of safrole (from sassafras oil), MDP2P and MDMA free base. The ultimate test of the utility of these by-products as markers for the *Wacker* oxidation of safrole would be to retrieve these materials from the final MDMA tablets, which were being produced in this laboratory. Solid-phase microextraction (SPME) was investigated as a tool for non-destructive recovery of the drug manufacturing impurities and the chromatographic result of the sampled *Mitsubishi* design tablet is presented in Fig. 14.

The presence of (1), (19) and (22) in the final MDMA tablets indicates that trace amounts of these materials carry through to the final MDMA even after purification by vacuum distillation. The ultimate fate of ketone (4) during the reductive amination



(24)

Fig. 17. Proposed structure of unknown compound 1 from the *Wacker* oxidation of safrole in ethanol.

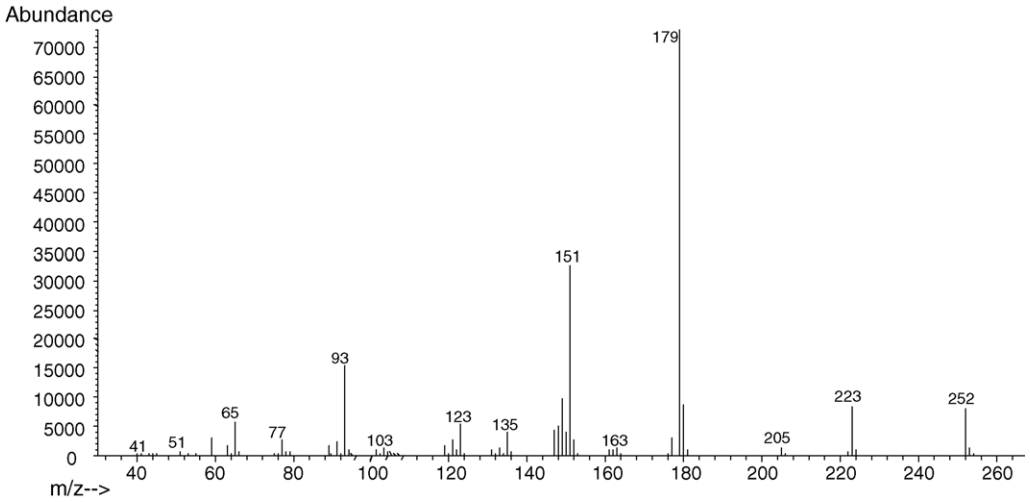
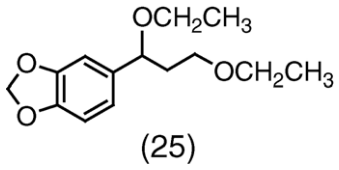


Fig. 18. Mass spectrum of unknown compound 2 from the *Wacker* oxidation of safrole in ethanol.

step is not known at this stage, but it is conceivable that this material may also be reductively aminated, but we were unable to detect *N*-methyl-2-methoxy-1-methyl-2-(3,4-methylenedioxyphenyl)-ethanamine in the final MDMA tablets. This material has been reported as a reductive amination product of (4) [14].

3.6. Ethanol mediated Wacker oxidation of safrole

We have also investigated the use of an alternative solvent in the *Wacker* oxidation. Ethanol is widely available as methylated spirits and although we have not detected the use of this solvent for the *Wacker* oxidation we thought that ethanol might be a convenient alternative for some underground chemists. Whereas *Wacker* oxidation of safrole in methanol is complete within 3 h we observed the reaction was sluggish in the case of ethanol and the reaction required 24 h for completion. The chromatographic result of the *Wacker* oxidation of safrole in ethanol is presented in Fig. 15.



(25)

Fig. 19. Proposed structure of unknown compound 2 from the *Wacker* oxidation of safrole in ethanol.

The MDP2P produced during the *Wacker* oxidation of safrole in ethanol was accompanied by the formation of the known compounds isosafrole and piperonal. Piperonal was not detected in the methanol mediated *Wacker* oxidation but this may be due to a shorter reaction time than when using ethanol. The extra reaction time may allow oxidative cleavage of the produced isosafrole. By-product formation appeared in general to be less, but we have tentatively assigned the structures of some of the trace by-product components as having ethoxy substitution rather than the corresponding methoxy substitution.

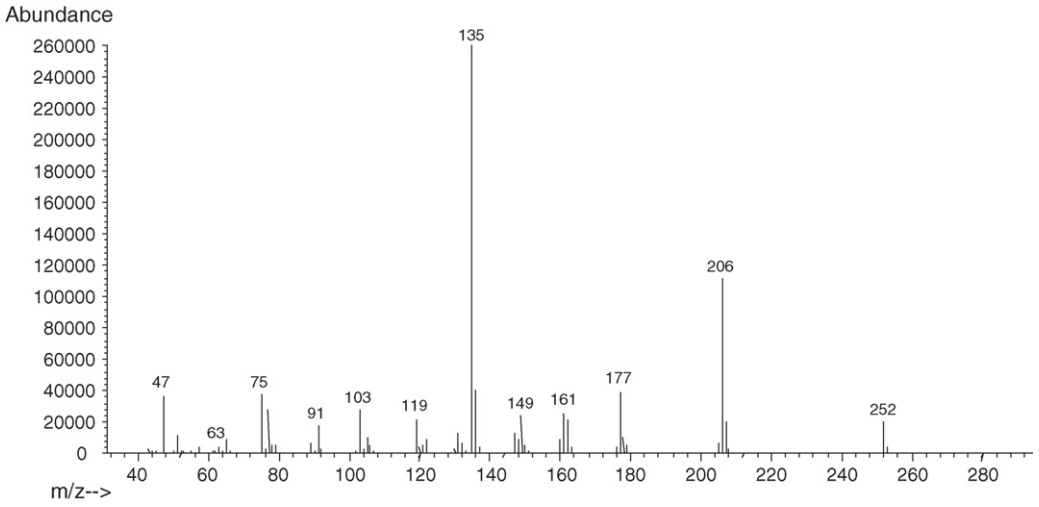
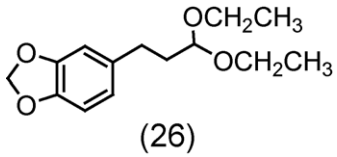


Fig. 20. Mass spectrum of unknown compound 3 from the *Wacker* oxidation of safrole in ethanol.



(26)

Fig. 21. Proposed structure of unknown compound 3 from the *Wacker* oxidation of safrole in ethanol.

3.6.1. Unknown 1

Unknown compound 1 from Fig. 15 has a base peak of 179 amu and a molecular ion of 208 amu and is consistent with an ethoxy group positioned at the benzylic position (Fig. 16).

This mass spectrum may represent the structure of 1-(3,4-methylenedioxyphenyl)-1-ethoxypropane (24) (Fig. 17).

3.6.2. Unknown 2

Unknown compound 2 from Fig. 15 has a base peak of 179 amu and a molecular ion of 252 amu and is consistent with two ethoxy groups in the molecule with one of them positioned at the benzylic position (Fig. 18).

This mass spectrum may represent the structure of 1-(3,4-methylenedioxyphenyl)-1,3-diethoxypropane (25) (Fig. 19).

3.6.3. Unknown 3

Unknown compound 3 from Fig. 15 has a base peak of 135 amu and a molecular ion of 252 amu and is consistent with two ethoxy groups in the molecule with none of them positioned at the benzylic position (Fig. 20).

This mass spectrum may represent the structure of 3-(3,4-methylenedioxyphenyl)-1,1-diethoxypropane (26) (Fig. 21).

4. Conclusions

It has been found that 1-(3,4-methylenedioxyphenyl)-1-methoxypropane (1), 1-(3,4-methylenedioxyphenyl)-1-methoxypropan-2-one (4), methyl 3-(3,4-methylenedioxyphenyl)-propanoate (9), 1-(3,4-methylenedioxyphenyl)-1,3-dimethoxypropane (19) and 3-(3,4-methylenedioxyphenyl)-1,1-dimethoxypropane (22) are produced during the *Wacker* oxidation of safrole to MDP2P using ρ-benzoquinone and palladium chloride with methanol as the solvent. Furthermore we have demonstrated the retrieval of (1), (4), (19) and (22) from clandestinely produced MDP2P and (1), (19) and (22) from final MDMA tablets (having been synthesised by this route). Ethanol is a viable alternative to methanol for the *Wacker* oxidation of safrole but similar by-products with ethoxy substitution are likely produced instead of the methoxy substituted by-products. It should be noted, however, that the presence of these impurities depends a great deal upon the exact technique by which purification of the intermediary and final products is performed i.e., solvent extraction and/or vacuum distillation etc.

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