Impurity Analysis of MDA Synthesized from Unrestricted Compounds

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2019

CERTIFICATE OF AUTHORSHIP/ORIGINALITY

I, Katherine Cooper declare that this thesis, is submitted in fulfilment of the requirements for the award of Master of Science (Research), in the School of Mathematical and Physical Sciences at the University of Technology Sydney. This thesis is wholly my own work unless otherwise reference or acknowledged. In addition, I certify that all information sources and literature used are indicated in the thesis. This document has not been submitted for qualifications at any other academic institution. This research is supported by the Australian Government Research Training Program.

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Katherine Cooper
16 July 2019

This is a conventional thesis format.

Acknowledgements

I'd like to thank my primary supervisor Scott Chadwick for your no-nonsense, practical, and helpful advice throughout my degree. Thank you for always being available and willing to help someone who started a chemistry degree with very limited organic chemistry knowledge. Your patience and guidance are very much appreciated, and I would not have finished without you.

I'd like to thank my secondary supervisor Andrew McDonagh for sharing your knowledge and apparent love for your job. Thank you for your encouragement and reassurance. Your contribution especially regarding NMR interpretation is much appreciated.

I'd like to thank Erin Heather for your help with mass spectral interpretation and expertise regarding things you'd only just overcome yourself, as well as your friendly and approachable demeanour.

I'd like to thank the laboratory technical staff. Thank you for answering my silly questions while I learn my way around the labs. Thank you for helping me and teaching me with the procedures and interpretation of the various analytical instruments I needed.

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List of Abbreviations

AFP	Australian Federal Police
ATS	Amphetamine type Stimulants
CNS	Central nervous system
DMA	Dimethylamphetamine
FTIR	Fourier transform infra-red spectroscopy
GC	Gas chromatography
GCMS	Gas Chromatography – Mass Spectrometry
HPLC	High Performance Liquid Chromatography
MA	Methamphetamine
MDA	3,4 methylenedioxyamphetamine
MDMA	3,4 methylenedioxymethamphetamine
MS	Mass spectrometry
NMR	Nuclear magnetic resonance spectroscopy
NPS	New Psychoactive Substances
PMA	*p*-methoxyamphetamine
PMMA	4-methoxymethamphetamine
2D DOSY	Two-dimensional diffused-ordered spectroscopy
2D-GC	Two-dimensional gas chromatography

Abstract

Methylenedioxyamphetamine (MDA) is classified as an illegal substance in many countries and jurisdictions around the world. Its popularity for illicit use is due to its stimulant and hallucinogenic effects. MDA can be synthesized from starting materials and reagents that are uncontrolled. These syntheses are attractive to clandestine laboratories as they can source large quantities of reagents without causing suspicion or risk of detection. Procedures for these syntheses require very little synthetic chemistry knowledge and are readily available online. Examination of the chemical profile of products from these syntheses can provide information about the starting material and synthetic pathway. This provides valuable information regarding linking cases, and tracking and limiting the supply of reagents used in clandestine laboratories.

This thesis examines the impurity profile of two synthetic pathways to MDA, from helional and piperonal, both of which involve few restricted compounds and would therefore be ideal to clandestine chemists. Helional is a fragrant oil available for wholesale purchase for home perfume makers and piperonal can be extracted from pepper. This thesis focuses on the organic impurities from side reactions between precursors, intermediates, and reagents, or reaction by-products. The products of each step were analysed using proton nuclear magnetic resonance spectroscopy (^{1}HNMR) and gas chromatography-mass spectrometry (GCMS). The results were examined to determine the identity of the impurities and to determine the presence of route specific impurities.

The results of this investigation show the identity of multiple impurities in the free base product of MDA synthesized from both helional and piperonal. Piperonal and MDP2P were shown to be common impurities between the syntheses. Most of the impurities identified within the helional product had been seen and recorded previously as impurities from other methods and none of the impurities carried through to the HCl product. Therefore, no route specific impurities were identified for the synthesis of MDA from helional.