# Final Report
**(May 2021)**

## Proficiencytesting@forensicfoundations

# Drug Analysis (Clandestine Laboratory) Inter-laboratory Collaborative Trial 2020-13

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# Introduction

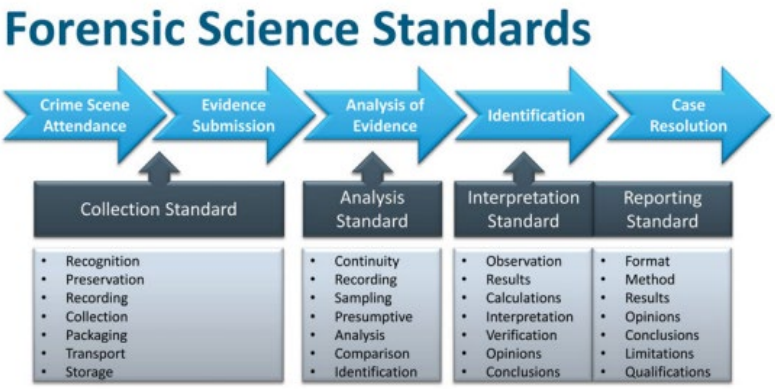
## Design

There are a number of parts to this trial:

1. Participants were provided with:
   a. A brief case scenario
   b. Observations from the scene and electronic analytical data files relating to the analysis of certain items taken from the scene, all provided by a clandestine laboratory investigating chemist.
2. It was not feasible in this trial to allow participants to examine the scene and to provide them with actual samples taken from the scene, especially for international participants. Instead, observations made at the scene by a clandestine drug laboratory investigating chemist and electronic data files obtained from analysis of certain items taken from the scene have been provided to participants. In this way the trial replicates the results they would obtain if they had the opportunity to analyse the items in their own laboratory. There are a number of issues that may occur when using the proprietary electronic data files provided, therefore we included a number of printouts of the analytical data. Furthermore, it is acknowledged that it is common practice for analysts to analyse reference materials in order to identify unknown compounds such as the products, precursors and by-products of manufacture in this exercise. Data for reference materials were not provided with this exercise as to do so would have provided participants with hints as to the identity of unknowns, which would have defeated the purpose of the exercise. As a consequence, this issue has been taken into account when providing responses to participants.

This trial has been designed to allow participants to evaluate the quality of their interpretation of the scene and analytical findings against those from other laboratories and observe how other laboratories express their opinions or advice to clients. To enable this, we requested that participants submit their opinion(s) in the format that they would provide to court.

We also anticipate that it will enable participants to evaluate the quality of their analytical results against those from other laboratories and observe the terminology used in other laboratories / countries, how other laboratories express their opinions or advice to clients.




Forensic Foundations' Proficiency Tests are designed to test the end-to-end forensic examination process. The AS5388 and the ISO21043 series of Standards describe the forensic examination process from collection to reporting. This figure[1] illustrates the inter-relatedness of all steps in this process and was used as the basis of the Australian Standards' development. The figure is also used as the basis of the development of Forensic Foundations' Proficiency Tests.

Thus, all Forensic Foundations' Proficiency Tests commence with item collection and/or receipt and all the subsequent examination / analysis steps, culminating in the reporting, therefore reflecting actual forensic casework.

[1] James Robertson, Karl Kent & Linzi Wilson-Wilde (2013) The Development of a Core Forensic Standards Framework for Australia, Forensic Science Policy & Management: An International Journal, 4:3-4, 59-67

# Drug Analysis Inter-lab Collaborative Trial 2020-13

This collaborative trial was distributed to ten laboratories and all the laboratories submitted results during this round of testing.

In order to minimise contextual bias in the interpretation, the information relating to the 'offence' was minimal.

This test provides a mechanism for participating laboratories to review their results and those of other laboratories to facilitate[2]:

- An evaluation and appraisal of their performance
- Continuous improvement
- Corrective action (where required).

As a collaborative trial we also asked questions on how the labs thought we could improve.

**Disclaimer:**

**The data contained in this report and any observations made are based on the material provided by the participants; however, it is understood that the laboratories may hold additional material that supported the findings reached.**

**Some results were prepared in languages other than English and the contents translated for review. It is acknowledged that, whilst the translation process may be accurate, on occasions, the context of the information may change, particularly where technical terms are used. It is assumed that the translated information is an accurate reflection of the original report.**

[2] ISO17025 (2017) General requirements for the competency of testing and calibration laboratories.

# Laboratory Responses

## Continuity, receipt, and description of items

Laboratories were requested to provide information with respect to the continuity, receipt, and description of the items received using a supplied proforma table. This facilitated both consistent responses and the collation and comparison of responses. Laboratories were requested to respond to each statement with a 'Yes' or 'No' and 'Comments' where appropriate. Where images of the items were included with the response, this was noted in the text below. Where no information was provided the cells of the table are shaded.

| Laboratory ID | Submission to the laboratory undertaken correctly, in accordance with laboratory procedures | Sample of chromatographs corresponds to accompanying paperwork | Security /continuity maintained | Chromatographs fully described in case notes | Description of any deviations from the expected |
|---|---|---|---|---|---|
| 16473 | No,<br>Submission of evidence was supposed to come with:<br>- Accompanying letter<br>- Police Report<br>- Letter of Investigation Order<br>- Evidence packing official report<br>- Suspect examination official report | Yes | No<br>No information on how the evidence was packed and wax-sealed. | Yes | No deviation (only received data) |
| 39947 | No,<br>All evidence collected at the crime scene that are subject to laboratory analyses should have been sent to the laboratory accompanied with formal requirements e.g. a Letter of Request, a Police Report, an Evidence Packing Report, and Suspect Examination Report (if any). | No,<br>Several compounds that were being used at the clandestine lab were missing their chromatograms e.g formic acid | No,<br>The evidence sent should have been wax-sealed. | Yes | All items confiscated at the crime scene should be sent to the laboratory, to make it easier in determining the relations of each chemical material, and should be accompanied with the formal requirements as described above. |
| 49026 | Yes | Yes | Yes | Yes | No deviation |
| 54624 | Yes | Yes | Yes | Yes | |

| Laboratory ID | Submission to the laboratory undertaken correctly, in accordance with laboratory procedures | Sample of chromatographs corresponds to accompanying paperwork | Security /continuity maintained | Chromatographs fully described in case notes | Description of any deviations from the expected |
|---|---|---|---|---|---|
| 55205 | Yes | Yes | Yes | Yes | No Deviation |
| 84132 | | | | | |
| 86790 | Yes,<br>Submission of evidence should have been accompanied by:<br>Accompanying letter<br>Police report<br>Letter of Investigation Order<br>Evidence packing report<br>Suspect examination report | Yes | Yes,<br>No information on evidence being packed and wax-sealed. | Yes | No deviation (only received data) |
| 90421 | Yes | Yes | Yes | Yes | No Deviation |
| 98179 | | | | | |
| 96150 | No,<br>In our jurisdiction we do not directly receive items from a clandestine laboratory. If we did, each item would need to be received sealed in a drug bag, which are uniquely numbered (exhibit number). If they were received as described they would be rejected.<br>The receipt form should have a unique identifier, the submitter and receiver should be identified and signed and dated by both parties. | No,<br>If there is an initial DCM blank followed by a DCM blank after each sample, there should be five blanks, but six are provided.<br>Normally the chromatograms would include our case reference number.<br>I would include the item number in each chromatogram title.<br>The infrared title on the spectrum for item 3b implies it is a hexane extract of 3a (not submitted) not 3b. | No,<br>As indicated above, the exhibits should be submitted sealed and have unique identifiers.<br>No chain of custody is provided from receipt.<br>If items were received at our facility the chain of custody would start from receipt, describe where the items were stored prior to analysis, identify who took possession of the items for analysis and who did the processing steps (e.g. extractions, undertook analysis, reported). | Yes,<br>For this exercise we processed the GCMS data files using Agilent software. The data was processed the same way we would record our results in the case file with the results reported as we would in a Certificate. We didn't rely on the infrared provided for 3b as it is a mixture. | As indicated above, receipt is a deviation from our standard practice.<br>This exercise doesn't follow our standard procedures. For item 3b we would have done a second GCMS and for all GCMS analysis we would have run standards: APAAN, P2P, methylamphetamine to determine retention times (I understand why this wasn't included with the test).<br>In providing an assessment of the scene we would have also had access to photos of the items in situ. |

Forensic Foundations' comments

The comments provide by the participants highlight the issues encountered providing data only. As discussed in the section outlining the design of the trial, Forensic Foundations was constrained by the legality of shipping illicit drugs and their precursors. However, the suggestion to provide photographs of the scene and items of interest in the appropriate packaging material has been noted and the feasibility of providing this information in further trials will be explored.

## Case Analysis & Interpretation

### Part 1. Interpretation of analytical results

Raw laboratory results are provided in Appendix A.

**Item 2**

Five of the ten participating laboratories correctly determined that Item 2 is crude alpha-phenylacetoacetonitrile, also known as APAAN or α-acetyl-benzeneacetonitrile.

Five of the ten participating laboratories did not indicate the presence of APAAN.

Only two participants (98179 and 96150) used the infrared data in their examination; one of those participants (96150) correctly proposed the contents of Item 2.

**Item 3b**

Item 3b data was collected from crude phenyl-2-propanone (also known as phenyl acetone or benzyl methyl ketone) prepared from APAAN, a residual trace of which is evident in the data.

Nine laboratories proposed that item 3b was phenyl-2-propanone (the expected response) and one laboratory did not mention the presence of phenyl-2-propanone in this Item.

**Item 4**

Item 4 data was collected from crude methylamphetamine that had been prepared from the crude phenyl-2-propanone (which in turn was prepared from the crude APAAN) using sodium cyanoborohydride and methylamine hydrochloride in methanol.

The presence of methamphetamine, phenyl-2-propanol (also known as α-methyl-benzeneethanol, benzeneethanol or -α-methyl compound), phenyl-2-propanone, and N-cyanomethyl-N-methyl-1-phenyl-2-propylamine in Item 4 are expected answers.
The presence of N-acetyl-N-(2-cyanoethyl)methamphetamine (also known as metamphemine,N-acetyl-N-(2-cyanoethyl)), benzphetamine (also known as benzamphetamine), N, α,α'-Trimethyldiphenethylamine, 7,8-Dimethyl-3-phenyl-6H-imidazo[1,2-a]pyrrolo[3,2- E]pyridine, N,α,α-Trimethyldiphenethylamine, and bis Pentadionato copper(II) are not the expected answers.

All participants proposed the expected, critical finding, that methamphetamine was likely to be present in Item 4.
All participants proposed the expected, critical finding, that phenyl-2-propanol was likely to be present in Item 4.

None of the participating laboratories reported the possible presence of N-cyanomethyl-N-methyl-1-phenyl-2-propylamine, which is the compound eluting at 10.154 minutes in the 1:4 dilution data for Item 4.

Forensic Foundations' comments –

Due to the format of this trial, participants could not carry out further analysis using reference materials in order to verify the identity of compounds responsible for the signals in the data that they were given. Therefore it is not surprising that several laboratories reported the presence of compounds that the test providers did not expect in Items 2, 3b, and 4. This test reinforces the importance of using analytical reference compounds to identify unknown compounds rather than relying upon mass spectral library search results as evidence. In the case of item 2, the infrared spectrum for the product was provided and that was sufficient to rule-out the presence of 2-methyl benzonitrile in Item 2.

None of the participants reported the possible presence of N-cyanomethyl-N-methyl-1-phenyl-2-propylamine, which is the compound eluting at 10.154 minutes in the 1:4 dilution data for Item 4. Whilst the presence of this compound (together with the presence of phenyl-2-propanol) is of significance to the proposition of the likely synthetic route used in the production of methamphetamine in this "case", this compound is not present in readily available mass spectral libraries therefore it is not surprising that this compound was not mentioned by participants. This test highlights the value of user-generated spectral libraries as a supplement to commercial and SWGDRUG libraries.

## Part 2. Interpretation of analytical results and scene observations

Interpretations provided by participants are given in Appendix B

Items 1, 2 and 5
Participants 16473 and 39947 did not discuss the non-analytical information provided relating to Item 1, 2 and 5. The hazard pictograms on Item 1 and the instruction to add the contents slowly to the oil and the salt in methanol were provided as hints to participants that perhaps the clandestine laboratory had access to a reagent capable of carrying out a reductive amination of phenyl-2-propanone to yield methamphetamine.

The words on Item 2 “oil precursor, boil with acid” were provided as a hint that the solid may be a precursor to an oily compound. As the solid in the jar was APAAN it is possible that the oily substance referred to is phenyl-2-propanone and therefore the words represent a suggestion that hydrolysis of APAAN was being carried out in the clandestine laboratory. This inference is corroborated by the analytical results in relation to Item 3. Participants 54624 and 96150 discussed the significance of these chemicals.

Item 5 describes a range of chemicals that the clandestine laboratory had access to that would enable hydrolysis of APAAN to be carried out and some chemicals that would enable a reductive amination to be carried out (with the exception of the reducing agent, which is alluded to in Item 1). Participants 96150, 54624, and 84132 discussed the significance of these chemicals.

Items 2, 3b, and 4
Three participants (16473, 39947, 86790) indicated that the Leuckart method was being used, three participants (49026, 55205, 90421) indicated an unspecified process involving phenyl-2-propanone was being used, participants 96150, 54624, 84132 indicated that an unspecified reductive amination process involving phenyl-2-propanone was being used, and participant 98179 indicated than an unspecified process involving phenyl-2-propanone or an unspecified reductive amination was being used.

Forensic Foundations’ comments

The presence of phenyl-2-propanone and phenyl-2-propanol in the GC-MS data for Item 4 should allow participants to propose that the production of methylamphetamine was being carried out using phenyl-2-propanone as precursor. The analytical results relating to Items 2 and 3 and the instructions on the bottle in Item 2 should allow participants to propose that phenyl-2-propanone had been produced *via* the hydrolysis of APAAN. The presence of phenyl-2-propanol in Item 4 should allow participants to rule out the involvement of a Leuckart reductive amination, which is a proposition supported to some extent by the instructions and labels on Item 1 and the list of chemicals in Item 5. The presence of N-cyanomethyl-N-methyl-1-phenyl-2-propylamine (see Salouros et al.) in Item 4 and the hints provided in Item 1 would allow participants to propose that the clandestine laboratory was engaged in the production of methylamphetamine using a cyanoborohydride reductive amination of the phenyl-2-propanone

## Conclusion and Summary of the Test

The aim of this test was to examine the end-to-end forensic examination, analysis, and reporting process. To minimise extraneous elements influencing the interpretation, limited contextual information was provided to the participating laboratories.

This test provides a mechanism for participating laboratories to use their results and those of other laboratories to facilitate[3]:

- An evaluation and review of their performance
- Continuous improvement
- Corrective action (where required)

The Forensic Science laboratories were provided with a series of chromatographs labelled item 2, 3b, and 4 along with a dilution series.

NOTE: The following observations are based on the material provided; however, it is understood that the laboratories may hold additional material.

### Continuity, receipt, and description of items

As the receipt and chain of custody for items, subject to forensic examination and analysis, is significant to the final outcome, information pertaining to receipt, continuity and a description of the items formed part of this test.

The remaining details, regarding continuity, receipt and item description was provided by all participants in full or in part.

### Examination / Analysis

All of the participants undertook analysis of the items of interest.

### Interpretation and Conclusions

All laboratories correctly deduced that the product being synthesised was methamphetamine *via* phenyl-2-propanone and all but one participant concluded that the laboratory had access to APAAN. One participant (55205) did not report the presence of phenyl-2-propanone in Item 2 in the clandestine laboratory.

The presence of phenyl-2-propanol, which was determined by all laboratories, is a strong suggestion that the Leuckart method was not being used in the laboratory, nor were any of the methods that involve the use of ephedrine or pseudoephedrine. The apparent absence of key ingredients for the Leuckart reaction or reduction of ephedrine/pseudoephedrine and the hints in Item 1 also help to rule out the involvement of these methods. Despite this, three participants indicated that the Leuckart method was being used (16473, 39947, 86790).

Seven participants indicated other methods of synthesis:
an unspecified phenyl acetone route (49026, 55205, 90421)
an unspecified reductive amination (96150, 54624, 84132) and
either a reductive amination or an unspecified phenyl acetone route (98179).

[3] ISO17025 (2017) General requirements for the competency of testing and calibration laboratories.

The presence of N-cyanomethyl-N-methyl-1-phenyl-2-propylamine in Item 4 is a strong suggestion that the laboratory was using sodium cyanoborohydride in a reductive amination. This compound has been reported in the literature, but it is not present in SWGDRUG or NIST mass spectral libraries. As participants did not have the opportunity to positively identify the compound eluting at 10.154 minutes in the provided chromatogram for Item 4, nor could they analyze items to establish whether boron or other elements were present, it is understandable that participants did not attempt to propose the exact reductive amination route used.

# APPENDIX A

## Laboratory Results

### Part 1. Interpretation of analytical results

**Item 2**

| | |
|---|---|
| 16473 | According to the wavenumber, the infrared spectral data for item 2 showed the Alkene and aromatic compound groups. It is estimated that item 2 is hydrocarbon and organic aromatic compounds.<br>Based on the chromatogram interpretation at 9.042 minutes, item 2 is Benzonitrile, 4-methyl compound. |
| 39947 | According to the wavenumber, the infrared spectral data for **item 2** indicated Alkene and aromatic compound groups. It is estimated that **item 2** is hydrocarbon and organic aromatic compounds. Based on the chromatogram interpretation at minute 9.042, **item 2** is Benzonitrile, 4-methyl compound. |
| 49026 | Item 2 – Item 2 min 9.051,from the electronic file of GC-MS data collected from fawn coloured solid, the Chromatogram interpretation was Benzeneacetonitrile, α-acetyl- compound |
| 54624 | From the electronic file of GCMS data obtained from fawn coloured solid, the APAAN (alpha-phenylacetoacetonitrile) compound was identified at retention time 9.042 minutes, which is a pre-precursor compound for synthesizing phenyl-2-propanone (P2P) precursor compound or Benzyl Methyl Ketone (BMK) which is the main precursor for producing Methamphetamine. |
| 55205 | The fawn coloured powder in MeOH scan 649, minute 9.051 from the interpretation of the chromatogram, was Benzeneacetonitrile, α-acetyl- compound. |
| 84132 | From **Pictures 1** and **2**, peaks from the chromatogram and Mass Spectroscopy were identified, **Benzonitrile / Benzyl cyanide** compound, the basic material for the production of Benzyl Methyl Ketone |
| 86790 | According to the wavenumber, the infrared spectral data for **item 2** indicated Alkene and aromatic compound groups. It is estimated that **item 2** is hydrocarbon and organic aromatic compounds. Based on the chromatogram interpretation at minute 9.042, **item 2** is Benzonitrile, 4-methyl compound. |
| 90421 | Item 2 min 9.051 from the electronic file of GC-MS data from a fawn colored solid, from the Chromatogram interpretation, Benzeneacetonitrile, α-acetyl- was identified |
| 98179 | On item 2, namely fawn coloured powder diluted in MeOh, the result of the GC-MS analysis was obtained with a Gas Chromatography retention time result at 9.402 minutes, and this resulted in a 4 dominant peaks on the Mass Spectrometry, namely 43.1 m/z, 88.1m/z,117 m/z dan 159.1 m/z. From the data, a base peak of 117 m/z was obtained, which was **Benzonitrile, 2 Methyl** compound or commonly known as **Benzocyanide** (**C6H5CN2CN**) with a 117 BM. The identification of each peak in the chromatography was performed by matching the m/z spectrum of each peak with NIST's GC-MS data base. |

| | |
|---|---|
| | While based on the IR reading, the identified frequency was 3122.44 cm-1, an Aromatic Ring (C-H) vibration type; 2213.39 cm-1 frequency, a Nitrile Compound (C ≡ N), and 1635.80 cm-1, a (C=C) compound. Therefore, it could be concluded that the compound was **Benzonitrile, 2 Methyl.** |
| 96150 | *A fawn coloured powder (based on scene notes).*<br>Contains Alpha-phenylacetoacetonitrile (APAAN).<br>Reported as "contains" as compound identity was confirmed using two A-class techniques: GCMS and FTIR. |

**Item 3b**

| | |
|---|---|
| 16473 | Based on the chromatogram interpretation of item 3b at 7.626 minutes, item 3b is 2-Propanone phenyl.<br>Based on the chromatogram interpretation of item 3b at 9.014 minutes, item 3b is Benzeneacetonitrile. |
| 39947 | Based on the chromatogram interpretation of **Item 3b** at minute 7.626, it is 2-Propanone phenyl.<br>Based on the chromatogram interpretation of **Item 3b** at minute 9.014, it is Benzeneacetonitrile. |
| 49026 | Item 3b –electronic file of GC-MS data obtained from yellow liquid<br>Item 3b min 7.626, from the Chromatogram interpretation was Benzyl methylketone compound<br>Item 3b min 9.014, from the Chromatogram interpretation was Benzeneacetonitrile, α-acetyl- compound |
| 54624 | From the electronic file of GCMS data obtained from yellow liquid, phenyl-2-propanone (P2P) precursor compound was identified at retention time 7.608 minutes and APAAN pre-precursor compound at retention time 9.014 minutes. Item 3b displayed the synthesis step of APAAN pre-precursor into P2P precursor. |
| 55205 | Item 3b Yellow oil in dichloromethane, scan 494, minute 7.626 from the interpretation of the chromatogram, was Benzeneacetonitrile, α-acetyl- compound.<br>Item 3b Yellow oil in dichloromethane, scan 645, minute 9.014 from the interpretation of the chromatogram, was Benzeneacetonitrile, α-acetyl- compound. |
| 84132 | From **Picture 3,** there were chromatogram peaks of Benzyl Methyl Ketone / P2P at Rt : 7,608 minutes and Benzeneacetonitrile at Rt : 9,017 minutes<br>From **Picture 4**, mass spectroscopy of Benzyl Methyl Ketone / Phenyl-2-Propanone was identified, one of the chemical compounds used in the synthesis of methamphetamine<br>From **Picture 5**, the mass spectroscopy of **Benzeneacetonitrile** compound is identified, basic material for the production of Benzyl Methyl Ketone |
| 86790 | Based on the chromatogram interpretation of **Item 3b** at minute 7.626, it is 2-Propanone phenyl.<br>Based on the chromatogram interpretation of **Item 3b** at minute 9.014, it is Benzeneacetonitrile |
| 90421 | The electronic file of GC-MS data from yellow liquid.<br>Item 3b min 7.626, from the Chromatogram interpretation, was Benzyl methyl ketone compound<br>Item 3b min. 9.014 from the Chromatogram interpretation, was Benzeneacetonitrile, α-acetyl- compound |
| 98179 | On item 3b, namely yellow oil diluted in dichloromethane, the GC-MS analysis result was obtained with a gas chromatography retention time results at 7.608 and 9.014 minutes. At retention time of 7.608 minutes, 3 dominant peaks were obtained, with a base peak of 136 m/z, a **Benzyl Methyl Ketone** compound or **Phenyl-2-Propanone.** |

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|---|---|
| | While at retention time 9.014 minutes, the same result as item 2 was obtained i.e.**Benzonitrile, 2 Methyl.**<br>From the IR reading, the following frequencies were identified: 3031.19 cm-1, an Aromatic Ring type of vibration; 1711.79 cm-1, a ketone compound (C=O); 2207.31 cm-1, a Nitrile Compound (C≡N); and 1638.06 cm-1, a (C=C) compound |
| 96150 | *Yellow oil in dichloromethane (based on scene notes).*<br>Indicated to contain P2P and APAAN.<br>Compound identities could not be confirmed due to lack of reference standard data for retention time matching, analysis of second sample portion, and use of confirmatory GC column.<br>FTIR visually similar to P2P reference spectrum, with additional absorbances present. Not confirmatory enough to report as “contains”. |

**Item 4**

| | |
|---|---|
| 16473 | a) Item 4, 1:100 dilution, min 5.687 was Methylene chloride compound<br>b) Item 4, 1:100 dilution, min 7.672 was Benzeneethanol, α-methyl- compound<br>c) Item 4, 1:100 dilution, min 8.086 was Methamphetamine compound<br>d) Item 4, 1:100 dilution, min 10.210 was Fenproporex $$ Propanenitrile compound<br>e) Item 4, 1:4 dilution, min 7.672 was Benzeneethanol, α-methyl- compound<br>f) Item 4, 1:4 dilution, min 7.617 was 2-Propanone compound<br>g) Item 4, 1:4 dilution, min 7.728 was Benzeneacetonitrile compound<br>h) Item 4, 1:4 dilution, min 10.154 was Fenproporex $$ Propanenitrile compound<br>i) Item 4, 1:4 dilution, min 11.385 was Benzylamphetamine compound<br>j) Item 4, 1:4 dilution, min 11.955 was N,.α.,α.’Trimethyldiphenethylamine compound<br>k) Item 4, 1:4 dilution, min 12.423 was 5 α –Estran-2-one compound |
| 39947 | a) **Item 4**, 1:100 dilution, min 5.687 was Methylene chloride compound<br>b) **Item 4**, 1:100 dilution, min 7.672 was Benzeneethanol, α-methyl- compound<br>c) **Item 4**, 1:100 dilution, min 8.086 was Methamphetamine compound<br>d) **Item 4**, 1:100 dilution, min 10.210 was Fenproporex $$ Propanenitrile compound<br>e) **Item 4**, 1:4 dilution, min 7.617 was Phenyl-2-propanone compound<br>f) **Item 4**, 1:4 dilution, min 7.728 was Benzeneacetonitrile compound<br>g) **Item 4**, 1:4 dilution, min 10.154 was Fenproporex $$ Propanenitrile compound<br>h) **Item 4**, 1:4 dilution, min 11.385 was Benzylamphetamine compound<br>i) **Item 4**, 1:4 dilution, min 11.955 was N,.α.,α.’Trimethyldiphenethylamine compound<br>j) **Item 4**, 1:4 dilution, min 12.423 was 5 α –Estran-2-one compound |
| 49026 | Item 4 – Electronic file of GC-MS data obtained from contents of conical flask. The following Chromatogram interpretations were obtained:<br>Item 4 min 7.617 was Benzyl methylketone compound<br>Item 4 min 7.672 was Benzeneethanol,α-methyl- compound<br>Item 4 min 7.728 was Benzene,(isocyanomethyl)- compound<br>Item 4 min 8.086 was Methamphetamine compound |

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| | Item 4 min 8.582 was Methamphetamine compound<br>Item 4 min 10.154 was Metamphemine,N-acetyl-N-(2-cyanoethyl)- compound<br>Item 4 min 10.200 was Metamphemine,N-acetyl-N-(2-cyanoethyl)- compound<br>Item 4 min 11.385 was Benzamphetamine compound<br>Item 4 min 11.955 was N, α,α'-Trimethyldiphenethylamine compound<br>Item 4 min 12.423 was 7,8-Dimethyl-3-phenyl-6H-imidazo[1,2-a]pyrrolo[3,2- E]pyridine compound |
| 54624 | From the electronic file of GCMS data obtained from basified strongly acid solution dichloromethane 1 in 100, several compounds were identified, among others were 1-phenyl-2-propanol at retention time 7.672 minutes, Methamphetamine at retention times 8.076 minutes and 8.582 minutes, unidentified peak at retention time 5.687 minutes and Methamphetamine, N-Acetyl-N-(2-Cyanethy) at retention time 10.200 minutes. For the electronic file of GCMS data obtained from basified strongly acid solution dichloromethane 1 in 4, these compounds were identified: 1-phenyl-2-propanol at retention time 7.672 minutes; Benzene, (isocyanomethyl) at retention time 7.728 minutes; phenyl-2-propanone (P2P) at retention time 7.617 minutes; Methamphetamine,N-Acetyl-N-(2-Cyanethy) at retention time 10.200 minutes; Benzphetamine at retention time 11.385 minutes; N,N-di (β-phenylisopropyl)-methylamine, diastomer or diphenylisopropylmethylamine (DPIMA) at retention time 11.955 minutes and an unidentified peak at retention time 12.423 minutes. Item 4 displayed the synthesis stage of P2P precursor into Methamphetamine and there were impurities N,N-di (β-phenylisopropyl)-**methylamine**, diastomer or diphenylisopropylmethylamine (DPIMA) compound as a marker of synthesis method used i.e. "reductive amination" with methylamine reagent |
| 55205 | Item 4 Basified acid solution dichloromethane, scan 283, minute 5.687 from the interpretation of the chromatogram, was Benzyl methyl ketone compound.<br>Item 4 Basified acid solution dichloromethane, scan 499, minute 7.672 from the interpretation of the chromatogram, was Benzeneetanol, α-methyl compound.<br>Item 4 Basified acid solution dichloromethane, scan 544, minute 8.086 from the interpretation of the chromatogram, was Methamphetamine compound.<br>Item 4 Basified acid solution dichloromethane, scan 598, minute 8.582 from the interpretation of the chromatogram, was Methamphetamine compound.<br>Item 4 Basified acid solution dichloromethane, scan 774, minute 10.200 from the interpretation of the chromatogram, was Methamphetamine, N-acetyl-N-(2-cynoethyl)- compounds<br>Item 4 Basified acid solution dichloromethane, scan 505, minute 7.728 from the interpretation of the chromatogram, was benzene, (isocyonometyl)-compound<br>Item 4 Basified acid solution dichloromethane, scan 493, minute 7.617 from the interpretation of the chromatogram, was Benzyl methyl ketone compound<br>Item 4 Basified acid solution dichloromethane, scan 769, minute 10.154 from the interpretation of the chromatogram, was Methamphetamine, N—acetyl-N-(2-cyanoethyl)- compound<br>Item 4 Basified acid solution dichloromethane, scan 903, minute 11.385 from the interpretation of the chromatogram, was Benzphetamine compound<br>Item 4 Basified acid solution dichloromethane, scan 965, minute 11.955 from the interpretation of the chromatogram, was N,α,α-Trimethyldiphenethylamine compound |

| | |
|---|---|
| | Item 4 Basified acid solution dichloromethane, scan 1016, minute 12.423 from the interpretation of the chromatogram, was 7,8-Dimethyl-3-phenyl-6H-imidazo [1,2-a]pyrolo[3,2-E]pyridine compound |
| 84132 | From **Picture 6**, there are chromatogram peaks of 1-Phenyl-2-Propanol (Rt: 7,672 minutes), Deoxyephedrine (Rt; 8,086 minutes), Benzeneethanamine (Rt: 8,582 minutes), Methamphetamine (Rt : 10,200 minutes) compounds<br>From **Picture 7,** the mass spectroscopy of **1-Phenyl-2-Propanol** compound is seen, a key impurity in reductive amination route<br>From **Pictures 8, 9 & 10**, the mass spectroscopy of **Methamphetamine** compound are seen, which is the end product of the synthesis<br>**Picture 12**. Mass Spectroscopy of Item 4 Rt : 7,617 minutes, Benzyl Methyl Ketone compound<br>**Picture 13**. Mass Spectroscopy of Item 4 Rt : 7,672 minutes, Phenyl-2-Propanol compound<br>**Picture 14**. Mass Spectroscopy of Item 4 Rt : 7,728 minutes, Benzyl Nitrile compound<br>**Picture 15**. Mass Spectroscopy of Item 4 Rt : 10,154 minutes, N-Acetylfenproporex compound<br>**Picture 16**. Mass Spectroscopy of Item 4 Rt : 11,385 minutes, Benzeneethanamine compound<br>**Picture 17**. Mass Spectroscopy of Item 4 Rt : 11,955 minutes, N-alpa Trimethyldephenylamine compound<br>**Picture 18** is a mass spectroscopy of ***Cooper Bis Pentadionat*** compound, a catalysator of methamphetamine synthesis reaction using reductive amination route |
| 86790 | **A)** **Item 4**, 1:100 dilution, min 5.687 was Methylene chloride compound<br>**B)** **Item 4**, 1:100 dilution, min 7.672 was Benzeneethanol, α-methyl- compound<br>**C)** **Item 4**, 1:100 dilution, min 8.086 was Methamphetamine compound<br>**D)** **Item 4**, 1:100 dilution, min 10.210 was Fenproporex $$ Propanenitrile compound<br>**E)** **Item 4**, 1:4 dilution, min 7.672 was Benzeneethanol, α-methyl- compound<br>**F)** **Item 4**, 1:4 dilution, min 7.617 was Phenyl-2-propanone compound<br>**G)** **Item 4**, 1:4 dilution, min 7.728 was Benzeneacetonitrile compound<br>**H)** **Item 4**, 1:4 dilution, min 10.154 was Fenproporex $$ Propanenitrile compound<br>**I)** **Item 4**, 1:4 dilution, min 11.385 was Benzylamphetamine compound<br>**J)** **Item 4**, 1:4 dilution, min 11.955 was N,.α.,α.'Trimethyldiphenethylamine compound<br>**K)** **Item 4**, 1:4 dilution, min 12.423 was 5 α –Estran-2-one compound |
| 90421 | The electronic file of GC-MS data from the conical flask, the interpretation of the Chromatogram are:<br>Item 4 min 7.617 was Benzyl methyl ketone compound<br>Item 4 min 7.672 was Benzeneethanol, α-methyl- compound<br>Item 4 min 7.728 was Benzene, (isocyanomethly)- compound<br>Item 4 min 8.086 was Methamphetamine compound<br>Item 4 min 8.582 was Methamphetamine compound<br>Item 4 min 10.154 was Metahmphetamine, N-acetyl-N-(2-cyanoethyl)- compound<br>Item 4 min 10.200 was Metahmphetamine, N-acetyl-N-(2-cyanoethyl)- compound<br>Item 4 min 11.385 was Benzamphetamine<br>Item 4 min 11.955 was N, α, α'-Trimethyldiphenethylamine<br>Item 4 min 12.423 was 7,8-Dimethyl-3-phenyl-6H-imidazo[1,2-a]pyrrolo[3,2-E]pyridine |

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| 98179 | On item 4, namely the basified acid solution diluted in dicloromethane with 1:100 dilution, 4 gas chromatography retention times were obtained i.e. 7.672 minutes; 8.086 minutes; 8.582 minutes; and 10.200 minutes.<br>At retention time 7.672 minutes, **Benzenetanol,-Alpha -Methyl** spectrum was identified<br>While at retention times 8.086 minutes; 8.582 minutes and 10.200 minutes, the following peaks: 58.1 m/z; 91.1 m/z; and 134.1 m/z were identified, the characteristics of **Methamphetamine** spectrum.<br>While for comparison, a 1:4 dilution resulted in a GC-MS analysis with retention times of 7.672 minutes; 7.728 minutes; 7.617 minutes; 10.154 minutes; 11. 385 minutes; 11.955 minutes and 12.423 minutes.<br>- At retention time 7.672 minutes, **Benzenetanol,-Alpha -Methyl** spectrum was obtained (see picture 1 item 4)<br>- At retention time 7.728 minutes, a base peak spectrum of 117 m/z was obtained, which is **Benzonitrile, 2 Methyl** compound (see picture 1 item 2)<br>- At retention time 7.617 minutes, similar spectral result as item 3 was obtained, namely **Benzyl Methyl Ketone or Phenyl-2-Propanone** (see picture 1 item 3)<br>- At retention time 10.154 minutes, **Methamphetamine** spectrum was obtained (see picture 2 item 4)<br>- At retention time 11. 385 minutes**, Benzeneethanamine** spectrum was obtained<br><br>At retention time 11.955 minutes, **N, α, α'-Trimethyldiphenethylamine** spectrum was identified<br><br>At retention time 12.432 minutes, **Copper Bis (2,4 Pentanedionato**) spectrum was obtained |
| 96150 | *Basified acid solution in dichloromethane (based on scene notes).*<br>Indicated to contain methylamphetamine.<br>Also indicated to contain P2P-ol, benzyl cyanide, P2P and N,N-di(beta-phenyl-isopropyl)methylamine.<br>Compound identities could not be confirmed due to lack of reference standard data for retention time matching, analysis of second sample portion, and use of confirmatory GC column. |

# APPENDIX B

## Part 2. Interpretation of scene observations

Where no additional information was provided the cells of the table are shaded.

| | |
|---|---|
| 16473 | |
| 39947 | |
| 49026 | In conclusion, from the observation of the Crime Scene and the results of the Chromatogram interpretations of the GC-MS analyses of the evidence, it was concluded that this Clandestine Lab was a Lab for producing Methamphetamine with Phenyl Acetone Route method. |
| 54624 | Several observable things at the Crime Scene were:<br>There was a phenyl-2-propanone (P2P) precursor synthesis activity from APAAN or alpha-phenylacetoacetonitrile chemicals using acid namely concentrated sulfuric acid. This is supported by the mark on the bottle that says “oil precursor, boil with acid”.<br>The separatory funnel at the Crime Scene was used to separate water and oil layers. Phenyl-2-propanone (P2P) and APAAN were identified after the yellow oil liquid was analyzed, therefore it could be deduced that this step was the initial step for synthesis from APAAN pre-precursor compound into P2P precursor.<br>Item 4 namely liquid with a pH of approximately 3 in the large conical flask was the result of a intial process of the mixing of P2P precursor, Methylamine and catalyst, where it would result in base Methamphetamine.<br>The analysis of item 5 namely reagent bottles, both sealed and opened, e.g. isopropanol, dichloromethane, methanol were used as solvents; concentrated sulphuric acid was used in the synthesis process of APAAN compound into P2P; concentrated hydrochloric acid was used in the crystallization process and methylamine (was used) as a reducing agent in the “reductive amination” method. |
| 55205 | The conclusion from the observation of the crime scene and the result of interpretation of chromatogram from the GCMS analysis results of the evidence, it was concluded that this Clandestine Lab was a laboratory for the manufacture of Methamphetamine by Phenyl Acetone Route method. |
| 84132 | At the crime scene, reagent bottles with methanol, dichloromethane, isopropanol, concentrated hydrochloric acid, methylamine hydrochloride, concentrated sulphuric acid labels were found.<br>Methylamine found at the crime scene in principle will be reacted with phenyl-2-propanone in a reactor with cooler, catalyzed with copper (Cu). This synthesis is performed in acid by adding concentrated $H_2SO_4$, then the substance formed is crystallized with concentrated HCl. |
| 86790 | The conclusion from the observation of the crime scene and the result of the interpretation of the chromatogram of the GC-MS instrument analysis of the evidence, it was concluded that the Clandestine Lab was an illegal laboratory for the manufacture of Methamphetamine with Leuckart method.<br>Further details were provided in participant’s results’ sheet |
| 90421 | The conclusion from the Crime Scene observation and the interpretation of the Chromatogram of the GCMS analysis on the evidence, it was concluded that this Clandestine Lab was a lab for producing Methamphetamine by Phenyl Acetone Route method. |
| 98179 | From the observation of the crime scene and the interpretation of the chromatogram result from the GCMS analysis of the evidence, Benzyl cyanide compound was identified, a synthesis from Phenyl-2-Propanone. Phenyl-2Propanone is one of the very popular chemical compounds in Methamphetamine synthesis, as the synthetic route of methamphetamine with this compound is very easy due to the relatively simple compound structure, and also because of its popularity. Aside from benzyl cyanide, Phenyl-2-Propane can also be synthesized from phenyl acetic acid chloroacetone or benzyl chloride and acetonitrile.<br>As such, it may be concluded that this Clandestine Lab is a Lab for producing Methamphetamine with Phenyl Acetone Route method or reductive amination route. The basis is the identification of methylamine in the reagent that could be reacted with Phenyl-2-Propanone which could produce methamphetamine. |

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| 96150 | The following is formatted as a statement we would prepare for court.<br><br>1. In this expert certificate the following abbreviations are used:<br>**APAAN** = *alpha*-Phenylacetoacetontrile<br>**P2P** = 1-Phenyl-2-propanone<br><br>2. On Thursday, 11 February 2021, our facility received an email request for an expert certificate in relation to samples submitted by the Eastern Australian Forensic Services (Our reference: 2021001118). The expert certificate request was allocated the same reference number: 2021001118.<br><br>3. This expert certificate has been prepared using the following resources:<br>• Eastern Australian Forensic Services Examination Request and Item Submission form.<br>• Certificate of Analysis 2020001118, signed by S. BURGE on 02 March 2021.<br><br>4. Methylamphetamine is listed as a prohibited drug in Schedule 1 in our jurisdiction.<br><br>5. A less common method encountered in our jurisdiction for the manufacture of methylamphetamine involves the reaction of 1-phenyl-2-propanone with methylamine followed by reduction of the resulting intermediate. 1-Phenyl-2-propanone in turn can be manufactured from **APAAN**.<br><br>**1) APAAN + acid → P2P**<br><br>**2) P2P + methylamine + reducing agent → methylamphetamine**<br><br>**<u>Manufacture of 1-phenyl-2-propanone</u>**<br>6. Alpha-phenylacetoacetonitrile is not listed as a prohibited drug nor as a drug precursor in our jurisdiction.<br><br>7. 1-Phenyl-2-propanone can be manufactured from **APAAN** by being warmed in the presence of an acid, such as sulfuric acid or hydrochloric acid, and water. Where the reaction had not gone to completion it will still contain some of the starting precursors.<br><br>8. Alpha-phenylacetoacetonitrile was identified in the following sample:<br>**Item 2** a plastic bottle containing a small amount of a fawn coloured powder, which contains **APAAN**.<br><br>9. According to the Eastern Australian Forensic Services' Examination Request and Item Submission form both sulfuric acid and hydrochloric acid were recorded as being present at the location of interest, through labelling. I am not aware if any presumptive testing at the location of interest was undertaken, though the provided notes indicate some quantities of these acids were sealed. |

10. Evidence for the manufacture of **P2P** includes, but not limited to:
    **Item 3** a separatory funnel containing a two phase liquid, one phase (water insoluble) of which was indicated to contain **P2P** and **APAAN**.

11. According to the Eastern Australian Forensic Services' Examination Request and Item Submission form, **item 2** was a 100mL bottle marked "oil precursor, boil with acid". The note on the bottle is consistent with knowledge that the substance it contains is a precursor and that the precursor can be formed by heating the substance in the presence of an acid. **P2P** is a liquid and may be referred to as an oil.

**Manufacture of methylamphetamine**

12. Methylammmonium salts are listed as drug precursors in our jurisdiction.

13. Methylamphetamine can be manufactured from **P2P** by the addition of methylamine, typically as the free-base form, along with the addition of a reducing agent. The reaction can be undertaken in methanol and reducing agents can include, but limited to: sodium borohydride or aluminum amalgam that is preformed by the actions of mercuric chloride on aluminium in water. The reaction is stirred and initially chilled to prevent over-heating.

14. According to the Eastern Australian Forensic Services' Examination Request and Item Submission form methylamine hydrochloride, being a salt of methylammonia, was recorded as being present at the location of interest, through labelling. I am not aware if any presumptive testing at the location of interest was undertaken, though the provided notes indicate some quantities of methylamine hydrochloride were sealed.

15. There was no evidence of any reducing agents recorded on the Eastern Australian Forensic Services' Examination Request and Item Submission form at the location of interest.

16. Methylamphetamine was indicated to be present in the following exhibit:
    **Item 4** a conical flask containing liquid (1.2L, pH ~ 3), which is indicated to contain methylamphetamine. It is also indicated to contain 1-phenyl-2-propanol, benzyl cyanide, **P2P** and *N*,*N*-di(beta-phenylisopropyl)methylamine.

17. In the reaction mentioned in paragraph **13**, where the reaction had not gone to completion it will still contain some of the starting precursors. In the reductive mixture, **P2P** can be reduced before it reacts with methylamine to give 1-phenyl-2-propanone. Benzyl cyanide is a by-product from the degradation of **APAAN**. If the methylamine reacts with two equivalents of **P2P** and is reduced it will give *N*,*N*-di(beta-phenylisopropyl)methylamine.

18. Also noted in the Eastern Australian Forensic Services' Examination Request and Item Submission form, **item 1** was recorded as a 50mL plastic bottle with a tight-fitting screw-cap with three chemical hazard labels and the instructions "Add all contents of this bottle, slowly to oil and salt in methanol".

| | |
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| | The three symbols shown on the Eastern Australian Forensic Services' Examination Request and Item Submission form are the same as, but not unique to, the chemical warning symbols for methylamine (free-base in water) [source: https://www.sigmaaldrich.com/australia.html search term = methylamine; Accessed 02 March 2021].<br><br>19. It should be noted that methylamine hydrochloride was recorded as being present at the location of interest through labelled containers. No caustic substances, such as caustic soda, were indicated to be present which would be required to convert the methylamine hydrochloride to methylamine (free-base). This would typically be conducted in water. Caustic soda has many legitimate uses and can be readily purchased from supermarkets and hardware stores.<br><br>20. The instructions on the bottle (**item 1**) are consistent with the actions required to manufacture methylamphetamine, where the oil may be a reference to **P2P** and the salt may be a reference to the reducing agent.<br><br>Based wholly or substantially on the above knowledge, I am of the opinion that the items examined relate to the manufacture of **P2P** from **APAAN** and methylamphetamine from **P2P** |

# APPENDIX C
## Conclusions

| | |
|---|---|
| 16473 | After a Criminalistics Laboratory examination was performed, it was concluded that the following evidence:<br>= item 2 : as mentioned on (I), was Benzonitrile, 4-methyl, which did not contain Narcotics, Psychotropics, or Dangerous Drugs.<br>= item 3b : as mentioned on (I), was 2-Propanone phenyl, which did not contain Narcotics, Psychotropics or Dangerous Drugs.<br>= item 4 : as mentioned on (I), contained Methamphetamine, listed on group I, number 61, of the Appendix I to the xxx Law No. 35 year 2009 on Narcotics.<br>The police requested an opinion as to what was being produced in this clandestine laboratory and what possible method was being used.<br>The method that might have been used is leukart. |
| 39947 | From the criminalistics laboratory interpretation of electronic data, it could be concluded that:<br>In the Clandestine Lab, Methamphetamine was being produced by Leuckart method.<br>The precursor that was being used was Benzyl Methyl Ketone/P2P.<br>The Pre-Precursor that was being used was Alpha Phenyl Aceto Acetonitrile (APAAN)<br>APAAN was hydrolized with concentrated sulphuric acid to produce BMK/P2P. BMK/P2P was reacted with formic acid to form n-formyl metamphetamine, and crystallization process was performed with concentrated hydrochloric acid, producing crystal Metamphetamine. |
| 49026 | From the Crime Scene observation and the results of the interpretation of the Chromatogram from GCMS analyses on the evidence, it was concluded that this Clandestine Lab was a Lab for producing Methamphetamine with Phenyl Acetone Route method. |
| 54624 | From the result of evidence examination and Crime Scene observation, it could be concluded that the chemicals and equipment at the Crime Scene have been used to produce Methamphetamine by “reductive amination” method. |
| 55205 | From the crime scene observation and the result of the interpretation of chromatogram from the GCMS analysis of the evidence, it was concluded that the Clandestine Lab was a Laboratory for the manufacturing of Methamphetamine by Phenyl Acetone Route method. |
| 84132 | From the result of the comparing the chromatogram and the spectroscopy with the GCMS analysis data (library) in the lab, we concluded that this clandestine lab was producing Methamphetamine by reductive amination. |
| 86790 | The conclusion from the observation of the crime scene and the result of the interpretation of the chromatogram of the GC-MS instrument analysis of the evidence, it was concluded that the Clandestine Lab was an illegal laboratory for the manufacture of Methamphetamine with Leuckart method. |
| 90421 | From the Crime Scene observation and the result of the interpretation of chromatogram from the GCMS analysis results of the evidence, it was concluded that this Clandestine Lab is a lab for producing Methamphetamine with Phenyl Acetone Route method |
| 98179 | Based on the GC-MS results of the three items, it could be concluded that the Clandestine Laboratory was producing **Methamphetamine**. |
| 96150 | Court report wording: That the items examined relate to the manufacture of 1-phenyl-2-propanone (P2P) from alpha-phenylacetoacetontrile (APAAN) and methylamphetamine from P2P. |

# APPENDIX D

## Proficiencytesting@forensicfoundations

### PROGRAM PLAN

| | | | | |
|---|---|---|---|---|
| **Program** | Drug Analysis Inter-Laboratory Collaborative Trial | | | |
| **Round** | 2020-13 | | | |
| | | | | |
| **Advisory Group** | | | | |
| **Program Coordinator /Technical Manager** | Mrs Anna Davey<br>Director<br>Forensic Foundations<br>PO Box 2279<br>North Ringwood, 3134 | | | |
| **Discipline specific expert(s)** | Prof K. Paul Kirkbride<br>Professor of Forensic Science<br>School of Chemical and Physical Sciences<br>Flinders University<br>GPO Box 2100<br>Adelaide, SA 5001 | | | |
| **Provider(s)** | Management, distribution & results collation | Test design & production. Results interpretation | NA | NA |
| | Forensic Foundations<br>PO Box 2279<br>North Ringwood,<br>Victoria 3134 | Prof K. Paul Kirkbride<br>Professor of Forensic Science<br>Flinders University | NA | NA |
| Test set up location | Flinders University | | | |
| | Sample distribution to government facilities within Australia & NZ by ANZPAA-NIFS | | | |
| **Aims/Objectives** | The aim of the program is to assess the facility's ability to assess the facility's case management system from receipt to reporting including the data collection and analysis | | | |
| **Purpose** | To assist the facilities to ensure their methods / procedures are performing adequately | | | |
| **Program Dates** | | | | |
| **Invitation letter** | August 2019 | | | |
| **Sample distribution** | November 2020 | | | |
| **Results due** | January 2021 | | | |

| | |
|---|---|
| **Manufacturing Information to be sent** | February 2021 |
| **Final report due date** | March 2021 |
| | |
| **Program Design** | |
| Number of Rounds | 1 |
| Number and type of samples | Participants will be provided with a digital copy of a number of traces produced from the examination of precursor material in drug manufacture |
| Hazards involved | NA |
| Scenario | A clandestine laboratory, potentially manufacturing illicit drugs has been located. They notified the Clandestine Laboratory Specialists. |
| Sample size/ volume | TBA |
| Range of values/assigned values | Expected result range |
| Traceability/origin of assigned values | Description of sample source |
| Design and Methods | How samples will be created |
| | |
| Selection Criteria | How samples will be selected/ created from batch/ source material |
| Potential Major Sources of Error | How it could go wrong during development and/or execution |
| | |
| **Participants** | |
| Criteria for participation | Who the test is aimed at |
| Expected number of participants | |
| **Reporting Criteria, Accuracy** | Special reporting conditions |
| **Analysis** | How lab should analyse samples (normally their own methods and any results we may want which might not be normally supplied (ie refractive index for glass). |

| | |
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| **Pre-testing** | |
| Homogeneity Testing and acceptance criteria | How this is done |
| Stability Testing and acceptance criteria | As above |
| | |
| **Technical Review (internal)** | |
| | |
| Participant Instructions | Provide evidence of technical review, may be emails |
| Results Sheet | Sample in file |
| Report | Sample in file, include review in file |

| | |
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| **Sample Preparation** | |
| Special conditions | Special sample preparation conditions, ie Cleaning before and after biological sample preparation. |
| Storage requirements | Special storage requirements |
| Use by Date | If applicable |

| | |
|---|---|
| Distribution requirements | Special distribution requirements |
| Packaging requirements | If applicable |
| Sample checks | Evidence in file |
| | |
| **Statistical Analysis** | |
| Homogeneity Testing and acceptance criteria | How it is done |
| Stability Testing and acceptance criteria | As above |
| Measurement Uncertainty | As above |
| Data Entry | Include evidence of data entry checks in file |
| | |
| Review by Statistician | As above |
| | |
| **Reporting** | |
| Report No: | 2020-13 |
| Master copy | Reports folder |
| Availability | Website (available to the public |
| Additional Comments | |

**Program Coordinator signature: KAD**

**Date: 18/3/2020**

# APPENDIX E

forensic FOUNDATIONS™
ISO 9001 & ISO 17043

**Proficiencytesting@forensicfoundations**

## Inter-laboratory Collaborative Trial
## Drug Analysis
## 2020-13

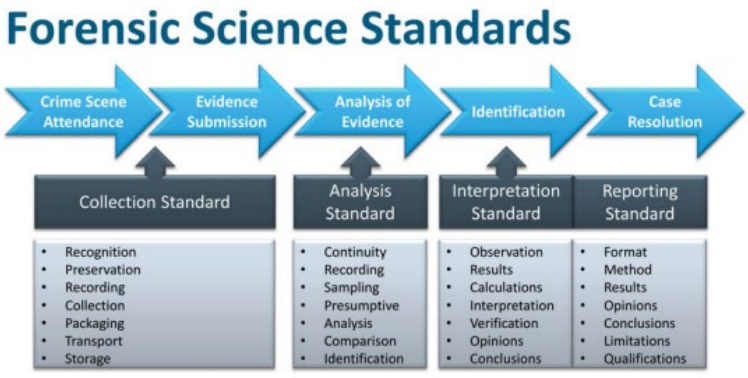
Thank you for participating in this Inter-laboratory Collaborative Trial. We hope that you find this trial useful and welcome any feedback which can be used in the design of further Collaborative Trials and subsequent Proficiency Tests, particularly relating to the feasibility of providing Trials in this manner.

There are a number of parts to this trial:

1. You have been provided:
   a. A brief case scenario
   b. Observations from the scene and electronic analytical data files relating to the analysis of certain items taken from the scene, all provided by a clandestine laboratory investigating chemist.
2. It is not feasible in this trial to allow participants to examine the scene and to provide them with actual samples taken from the scene, especially for international participants. Instead, observations made at the scene by a clandestine drug laboratory investigating chemist and electronic data files obtained from analysis of certain items taken from the scene have been provided to participants. In this way the trial replicates the results they would obtain if they had the opportunity to analyze the items in their own laboratory. There are a number of issues that may occur when using the electronic data files provided, therefore we have included a number of printouts of the analytical data in this document.

This trial has been designed to allow participants to evaluate the quality of their interpretation of the scene and analytical findings against those from other laboratories and observe how other laboratories express their opinions or advice to clients. To enable this, we request that participants submit their opinion(s) in the format that they would provide to court.

Forensic Foundations' Collaborative Trials and Proficiency Tests are designed to test the end-to-end forensic examination process. The AS5388 and the ISO21043 series of Standards describe the forensic examination process from collection to reporting. This figure[4] illustrates the inter-relatedness of all steps in this process and was used as the basis of the Australian Standards'




[4] James Robertson, Karl Kent & Linzi Wilson-Wilde (2013) The Development of a Core Forensic Standards Framework for Australia, Forensic Science Policy & Management: An International Journal, 4:3-4, 59-67

development. The figure is also used as the basis of the development of Forensic Foundations' Proficiency Tests. Thus, all Forensic Foundations' Proficiency Tests commence with item collection and/or receipt and all the subsequent examination / analysis steps, culminating in the reporting, therefore reflecting actual forensic casework.

Attached you will find the case 'Examination Request and Item Submission' form, the test commences with the receipt of the items followed by your routine processes.

The information submitted to the laboratory on the examination request form will direct what needs to be undertaken. Please use the attached results sheets. Additional pages may be added if required. An electronic copy of the results sheet can be downloaded from https://www.forensicfoundations.com.au/download/

The results sheets should be returned to Forensic Foundations by **29th January 2021**. Hardcopy can be returned to PO Box 2279, Ringwood, Victoria, 3134, Australia or a soft copy can be uploaded to https://www.forensicfoundations.com.au/upload/

Qualitative feedback will be provided to participants.

Following the conclusion of the testing participants will be advised of the expected results and details regarding the production of the test.

# APPENDIX F

| EXAMINATION REQUEST AND ITEM SUBMISSION | EASTERN AUSTRALIAN FORENSIC SERVICES |
|---|---|

| OFFENCE: | Manufacture of prohibited substance |
|---|---|
| DATE OF OFFENCE | 12 August 2020 |

BRIEF STATEMENT OF FACTS

The Eastern Australian Police located a clandestine laboratory, potentially manufacturing illicit drugs in a small room located at the rear of 43 Fann St, Eastville. They notified the Clandestine Laboratory Specialist from Eastern Australian Forensic Services. The Police and the Clandestine Laboratory Specialist attended between 12:10 -16:20 pm on August 12, 2020.

The Clandestine Laboratory Specialist located, amongst other items, the following:

**ITEM**

1) An empty 50 mL bottle with a tight-fitting screw-cap. The bottle had adhesive labels as depicted on the right and some hand-written instructions as follows:

"Add all contents of this bottle, slowly to oil and salt in methanol"

2) A 100 mL bottle with a tight-fitting screw cap marked "oil precursor, boil with acid". A small amount of fawn-coloured solid was present in this bottle: it was analyzed using ATR IR spectrometry and a solution in methanol was analyzed using GC-MS.

3) A separatory funnel next to a sink of dirty glassware was found, inside which were residues of two kinds of liquid. One liquid was clear, water soluble and was found to have a pH of approximately 3. The other liquid was yellow, insoluble in water but soluble in dichloromethane and hexane. A droplet of a hexane solution was applied to the crystal of a horizontal ATR accessory, allowed to evaporate, and the IR spectrum of the residue collected. A sample of the dichloromethane solution, diluted with additional dichloromethane, was then analyzed by GC-MS.

4) A large conical flask (2 L) contained approximately 1.2 L of liquid with a pH of approximately 3. The liquid was transferred to an evidence bottle (2 L) and the emptied flask was washed twice with water (100 mL). The washings were added to the evidence bottle and the contents were then diluted to exactly 2 L with water and an aliquot (1 mL) was taken for further analysis. The 1 mL aliquot was treated with sodium hydroxide solution (2 M) until it attained a pH of 11 and then extracted with dichloromethane (1

mL). The organic extract was dried with anhydrous sodium sulphate and two aliquots (50 µL) were diluted in dichloromethane (0.2 mL and 5 mL) for analysis by GC-MS.

**ITEM**

5) The following sealed reagent bottles were found: methanol (4 x 2.5 L), isopropanol (1 x 2.5 L), dichloromethane (4 x 2.5 L), concentrated hydrochloric acid (1 x 2.5 L), concentrated sulphuric acid (4 x 2.5 L), methylamine hydrochloride (2 x 100 g). The following opened reagent bottles were also found, apparently all contents were true to label: methanol (1 x 2.5 L), dichloromethane (1 x 2.5 L), concentrated hydrochloric acid (1 x 2.5 L), concentrated sulphuric acid (1 x 2.5 L), methylamine hydrochloride (1 x 100g).

ITEMS PROVIDED FOR EXAMINATION

Item 1 – Labelled bottle. Not submitted

Item 2 – Electronic file of GC-MS data collected from fawn coloured solid

Item 3a – Clear liquid, water soluble and found to have a pH of approximately 3. Not submitted

Item 3b – Electronic file of GC-MS data obtained from yellow liquid as described above

Item 4 – Electronic file of GC-MS data obtained from contents of conical flask treated as described above.

Items 5 – Not submitted

The electronic files can be downloaded from:
https://www.forensicfoundations.com.au/download

EXAMINATION REQUESTED

Police request an opinion as to what was being produced in this clandestine laboratory and what possible method was being used. .

# APPENDIX G

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**PROFICIENCYTESTING@FORENSICFOUNDATIONS**
**DRUG ANALYSIS (CLANDESTINE LABORATORY)**
**INTER-LABORATORY COLLABORATIVE TRIAL**
**2020-13**
**MANUFACTURER'S INFORMATION**

## Introduction

The aim of the program is to assess the laboratory's ability to examine and understand the methods used in illicit drug manufacture and apply their knowledg existing chromatographs to determine what and how a substance was made.

## Scenario

A clandestine laboratory, potentially manufacturing illicit drugs was located. A number of items were located. The participants are asked to provide an opinion a what was being produced and what possible method was being used.

## Test production

α-Phenylacetoacetonitrile (APAAN) was prepared by treating sodium metal (0.45 in toluene (7.4mL) with absolute ethanol (2.3mL) in a flame dried flask under nitrogen atmosphere. The mixture was then boiled under reflux until all the sodiu had reacted, cooled to 70°C and treated with phenylacetonitrile (15mmol, 1.7mL) anhydrous ethyl acetate (2.2mL). The mixture was heated under refluxed at 100° for a further 3 hours.

Once complete, the reaction was cooled on ice and diluted with enough water (25mL) to dissolve the precipitate formed. The mixture was washed with chilled toluene (3 x 15mL) and the aqueous phase acidified with a mixture of ice and gla acetic acid. Over 1 hour a precipitate formed, which was then isolated by filtratior and dried under vacuum.

The APAAN thus produced was converted into phenyl-2-propanone using the method of Hauser et al. (briefly, APAAN (2.12 g) was treated with water and sulphuric acid at 100° C). Phenyl-2-propanone was converted into methylamphetamine without further purification in a reductive amination using sodium cyanoborohydride and methylamine using a method based upon that of Salouros et al. Briefly, crude phenyl-2-propanone (1.34 g) was added to a stirred solution of methylamine hydrochloride (0.67g ) in methanol (20 mL). Sodium cyanoborohydride (0.62 g) was added portion-wise over a period of 30 min.

After that time the mixture was stirred overnight, quenched by adding it to dilute hydrochloric acid (0.5 M, 100 mL) and thrice extracted with dichloromethane (20 mL). The aqueous phase was made basic with solid sodium hydroxide and extracted thrice more with dichloromethane (20 mL). The combined organic extracts were dried over anhydrous sodium sulphate.

Test samples

Five ‘test items’ were provided to participants.

Item 1 was a fictional description of bottle said to have been found at the site of a clandestine laboratory. The description was designed to provide hints to participants with regards to the procedure that was being used at the laboratory.

Item 2 was a set of GC-MS and IR spectrometry data said to relate to a fawn colored solid found at the clandestine laboratory; these data were obtained from the crude APAAN.

Item 3 was a set of GC-MS and IR spectrometry data said to relate to a yellow liquid found in a separatory funnel at the clandestine laboratory; these data were obtained from the crude phenyl-2-propanone.

Item 4 was GC-MS spectrometry data said to relate to an extract of a basified acid solution that was found in a conical flask found at the clandestine laboratory; these data were obtained from the extract of the reductive amination procedure.

Item 5 was a fictional description of other bottles said to have been found at the site of a clandestine laboratory. The description was designed to provide hints to participants with regards to the procedure that was being used at the laboratory.

The GC-MS and IR conditions are listed in the Appendix.

Hauser, F. M., et al. (2018). "Identification of specific markers for amphetamine synthesised from the pre-precursor APAAN following the Leuckart route and retrospective search for APAAN markers in profiling databases from Germany and the Netherlands." Drug Testing and Analysis 10(4): 671-680.

Salouros, H., et al. (2008). "N-Cyanomethyl-N-Methyl-1-(3,4-methylenedioxyphenyl)-2-propylamine: An MDMA Manufacturing By-Product." Journal of Forensic Sciences 53(5): 1083-1091(1089).

Final results

Participants should be able to propose that methylamphetamine (Item 4), APAAN (Item 2) and phenyl-2-propanone (Item 3) were present at the clandestine laboratory.

Furthermore, it is expected that:

1) the presence of APAAN in Items 2 and 3, the instructions marked on the bottle in Item 2, and the presence of strong mineral acids described in Item 5 would allow participants to propose that the phenyl-2-propanone found at the lab was prepared by hydrolysis of APAAN.

2) the presence of phenyl-2-propanol in the GC-MS data for Item 4 would allow participants to propose that the production of methylamphetamine was being carried out using phenyl-2-propanone as precursor, and together with the detail in Items 1 and 5 the presence of phenyl-2-propanol would also allow participants to rule out the involvement of a Leuckart reductive amination. The presence of N-cyanomethyl-N-methyl-1-phenyl-2-propylamine (see Salouros et al.) in Item 4 and the hints provided in Item 1 would allow participants to propose that the clandestine laboratory was engaged in the production of methylamphetamine using a cyanoborohydride reductive amination of the phenyl-2-propanone found at the crime scene.

END OF DOCUMENT

# Drug Analysis 2020-13 Feedback

Forensic Foundations prides itself in providing flexible fit-for-purpose forensic programs. The manufacture, distribution and assessment and reporting of this test has provided and will provide the basis for continuous improvement for both Forensic Foundations and the forensic laboratories. To this end we would appreciate your comments to assist us to improve the tests.

Please tick the appropriate box and make any relevant comments.

| | Strongly Agree | Agree | Disagree | Strongly Disagree | NA |
|---|---|---|---|---|---|
| 1. The test was too basic for our facility | ☐ | ☐ | ☐ | ☐ | ☐ |
| ……<br>……<br>……<br>…… | | | | | |
| 2. The samples supplied were suitable | ☐ | ☐ | ☐ | ☐ | ☐ |
| ……<br>……<br>……<br>…… | | | | | |
| 3. The results required were not outlined sufficiently | ☐ | ☐ | ☐ | ☐ | ☐ |
| ……<br>……<br>……<br>…… | | | | | |
| 4. The final report provided suitable detail | ☐ | ☐ | ☐ | ☐ | ☐ |
| ……<br>……<br>……<br>…… | | | | | |
| 5. The tests involved should be more challenging | ☐ | ☐ | ☐ | ☐ | ☐ |
| ……<br>……<br>……<br>…… | | | | | |

Please comment briefly on the following:

1. Are there additional aspects which could be included in the test?

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2. Any additional comments

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3. Facility (optional)

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4. Would you like us to contact you to discuss your feedback?

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Forensic Foundations' Proficiency Tests are required to be fit-for purpose. To assist us to provide the relevant tests, please use the following form to suggest further tests for development.

## Recommendation for Proficiency Test development

| | | |
|---|---|---|
| Contact | Name | |
| | Email | |
| | Phone | |
| Discipline/ subdiscipline | | |
| Specific issues(s) to be addressed*. Note: The tests can be designed to be multidisciplinary. | | |
| Suggested technical advisor (if known) | | |
| Suggested manufacturer (if known) | | |

* All Proficiency Tests will include the end to end process (receipt & continuity, triage, description, examination, analysis, data generation, interpretation, reporting) but one aspect may be of particular interest/focus.

This form can be emailed to quality@forensicfoundations.com.au or you can discuss your suggestions on either 03 9018 8919 or 0429 966 012.