

Forensic intelligence: Applications in illegal drug trafficking

by

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Certificate of original authorship

I, Harmonie Michelot, declare that this thesis is submitted in fulfilment of the requirements for the award of Doctor of Philosophy, 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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Abbreviations

- **ACBPS** : Australian Customs and Border Protection Services
- **ACT** : Australian Capital Territory
- **AFP** : Australian Federal Police
- **AIDDC** : Australian Illicit Drug Data Centre
- **AIDIP** : Australian Illicit Drug Intelligence Program
- **ATR-FTIR** : Attenuated Total Reflectance - Fourier Transform Infrared
- **APCI-ITMS-MS** : Ion trap tandem Mass Spectrometry with Atmospheric Pressure Chemical Ionization
- **AUC** : Area Under the Curve
- **CE-DAD** : Capillary Electrophoresis with Diode Array Detector
- **Corr** : Correlation
- **DEA** : Drug Enforcement Administration
- **DTB** : Direct Transfer experiment with Benchtop
- **DTF** : Direct Transfer experiment with Fingers
- **ELSD** : Evaporative Light Scattering Detector
- **ENIPID** : Enhanced National Intelligence Picture on Illicit Drugs
- **FASS** : Forensic and Analytical Science Service

- **FN** : False Negative
- **FP** : False Positive
- **GC-MS** : Gas Chromatography coupled to a Mass Spectrometer
- **HPLC** : High-Performance Liquid Chromatography
- **HS-GC-FID** : Head Space Gas Chromatography coupled to a Flame Ionisation Detector
- **ICPMS** : Inductively Coupled Plasma Mass Spectrometry
- **IMS** : Ionisation Mobility Spectrometry
- **IRMS** : Isotopic Ratio Mass Spectrometry
- **LC-MS** : Liquid Chromatography coupled to a Mass Spectrometer
- **LOD** : Limit of detection
- **LT** : Linear transformation
- **MA** : methylamphetamine
- **MDMA** : 3,4- Methylenedioxymethamphetamine
- **MEK** : Methyl ethyl ketone
- **MIBK** : Methylisobutylketone
- **N** : Normalisation
- **NMI** : National Measurement Institute
- **NPS** : New Psychoactive Substances

- **NSW** : New South Wales
- **PEDIT** : Physical Examination Data Input Template
- **PROMIS** : Police Real Time Online Management Information System
- **PTHIT** : Phenyltetrahydroimidazothiazole
- **ROC** : Receiving Operator Curve
- **SIM** : Single Ion Monitoring mode
- **Sqrt** : Square Root
- **STRL** : Special Testing and Research Laboratory
- **THC** : Tetrahydrocannabinol
- **TN** : True Negative
- **TP** : True Postive
- **UNODC** : United Nations Office on Drug and Crime
- **3-MAM** : 3-MonoAcetylMorphine
- **6-MAM** : 6-MonoacetylMorphine
- **10min A** : 10 minutes of Activity experiment
- **30min A** : 30 minutes of Activity experiment
- **1h A** : One hour of Activity experiment
- **12h P** : 12 hours Persistence experiment

- **24h P** : 24 hours Persistence experiment
- **2nd T** : Secondary transfer experiment

Abstract

This research aimed at getting a better understanding of illicit drug trafficking, especially from an Australian point of view, by looking at different approaches of getting valuable information in a timely fashion for forensic intelligence purpose. The study was conducted in collaboration with the Australian Federal Police (AFP) who provided appropriate data. In return, the study was expected to provide findings to grow their knowledge about such criminal phenomenon that is illegal drug trafficking.

Two distinct approaches were undertaken. The first one was an analysis of chemical results of cocaine and heroin border seizures performed by the AFP during 2008 and 2013. Trends regarding the purity as well as added compounds over time and per geographic location were discovered. Moreover, statistical methods were applied on the provided datasets to assess the feasibility to develop an automatic triage of those chemical results and highlighting links between seizures based on their chemical data. Promising results with few error rates were obtained, as cocaine seizures could be discriminated with 9.36 % of false positives and 2.45 % of false negatives, and heroin seizures could be discriminated with 4.82 % of false positives and 2.94 % of false negatives. Therefore, the automatic statistical model could be implemented for routine use at the AFP.

The second approach was a proof of concept study investigating the possibility to use currently deployed portable instruments for intelligence purpose instead of the traditional identification and case-specific aim that they are designed for. Three different technologies were tested, Attenuated Total Reflectance - Transform Infrared spectroscopy (ATR-FTIR), Ion Mobility Spectroscopy (IMS) and Ion trap tandem Mass Spectrometry with Atmospheric Pressure Chemical Ionization (APCI-ITMS-MS) for the detection of remnants of drugs present on the surface of passports, using various parameters including transfer, activity and persistence. An experimental design was developed and different scenarios were trialled. Promising results were obtained especially with APCI-ITMS-MS, as drugs' residues could be detected even after an activity of thirty minutes in quantities less than 0.05 µg. The findings demonstrate that a routine use at customs would be feasible to obtain a better overview of trafficking flows instead of targeting specific individuals.

The different projects conducted within this research emphasise the need for data triangulation and using various source of information to get a more holistic view of the criminality, in this case illegal drug trafficking.

Chapter 1:

Theoretical framework

Chapter 1: Theoretical framework

1.1 Context

1.1.1 Forensic intelligence principles

1.1.1.1 *Forensic science and origin of forensic intelligence*

Forensic science stems from the well-known principle that any crime leaves traces [1], as summarised by Margot [2]: *“the trace, which, by definition, is a pattern, a signal or material transferred during an event (often unknowingly by the actors of the event). It is the remnant (the memory) of the source (identity – who, with what?) and of the activity (what, how, when, why?) that produced it”*. These traces, when detected, analysed and correctly exploited can answer the central six questions (who, what, how, where, when and why) [3]. To address these questions, forensic science is currently accepted as an integral part of an investigation [4, 5], with forensic scientists assisting Police in detecting the remnants of a crime [6].

The main limitation to forensic science's contribution is that it is mainly employed for the court rather than for crime control [7, 8]. Consequently, crucial information conveyed by forensic case data (i.e. traces [9]) that may not support court proceedings is not entirely exploited or even not considered at all [10]. Indeed, the percentage of traces that are detected and collected at crime scenes then analysed and used to be presented as a valuable evidence at court is very low; for instance less than 1 % of DNA collected at crime scenes is used for prosecution [9]. It depends on many factors, starting from the decision to attend or

not a crime scene, to analyse or not a trace, etc. [11]. But in essence, it implies that information obtained from criminal activity is often lost in the judicial system. This is where forensic intelligence comes into play, by exploiting all information from traces, not for the court but to study the criminality and getting an holistic overview of criminal phenomena [9].

Forensic intelligence gained in importance with the emergence of computer-based technology from the 1990's, as it enabled police to deal with mass data related to investigations [12]. The tipping point for most of intelligence developments occurred with the terrorist attacks of September 11, 2001. This event led to the development and fusion of initially separate agencies for intelligence purposes, initiated with fusion centres in the United States including collaboration of state and federal police [13]. These fusion centres responded to the problem of *"information sharing vertically between federal, state and local police, and horizontally between peer agencies within each region"* [13]. Indeed, information sharing is considered as a pillar in prevention and risk management of criminal activities, from a national to a local scale [13]. Intelligence units have been gradually developed internationally with the realisation that an 'Intelligence Community' is essential [14]. These units nowadays are present in many police department worldwide [15]. As a result, intelligence is employed by multiple organisations and in different contexts [16, 17]. Nevertheless, forensic intelligence is not fixed; it is still being shaped [18], and its understandings differ from one country to another, from one jurisdiction to another, or even within the same agency [19]. The proposed definition of forensic intelligence for this study, already recognised and employed internationally, comes from Ribaux et al. [20]:

[Forensic intelligence]

"is the accurate, timely and usable product of logically processed forensic case data" [20].

From this definition, as forensic intelligence is using 'forensic case data', each sub-discipline of forensic science can benefit from forensic intelligence [21]: the latest can therefore be presented as transversal [22, 23].

1.1.1.2 Forensic intelligence: concept and process

The primary aim of forensic intelligence is to provide a better understanding of the criminality in a timely fashion [24, 25], by assisting law enforcement agencies to prioritise their activity and build strategies in a proactive manner depending on the situation rather than seeking reactions to problems [3, 8, 26, 27]. It is ultimately expected to fight more efficiently against the different types of crimes and address related societal problems [18].

Forensic intelligence can be divided into a pyramid model, from a reactive to a more proactive use, as presented by Marclay et al. (Figure 1):

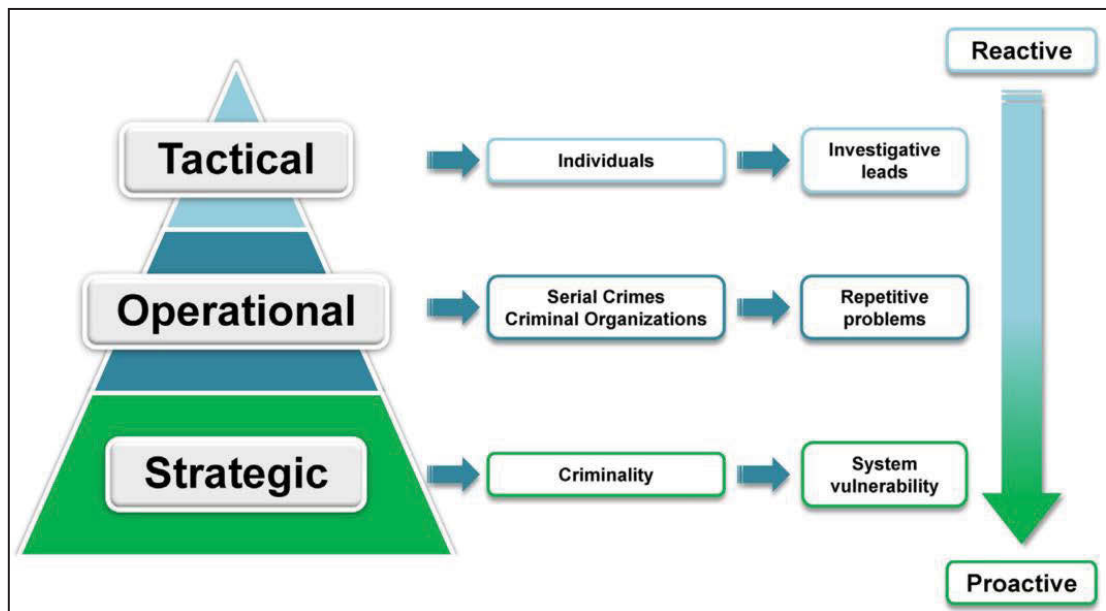


Figure 1: The pyramid model of forensic intelligence [28]

Tactical intelligence is based on real-time decision-making for investigations at a case level [28-30]. It is a case-to-case strategy that front-line officers are in charge of which aims to feed the judicial system [31]. As quoted by Ratcliffe, *“they do not necessarily have access to the broader and more holistic levels of intelligence that can provide a greater understanding of long-term problems, problems that exist at scales greater than that of the behaviour of individual offenders”* [32].

Operational intelligence covers a larger organisation level, aiming to provide an understanding of criminal activity, to address actions and planning crime reduction geographically. It intends to impact on repetitive problems such as serial crimes [29, 30, 32].

Strategic intelligence operates at a third and more global level of organisation [28-30]. It aims at understanding patterns and having an impact on long-term problems [32].

As a result, intelligence produced is different depending on the demand, and the different levels of the pyramidal model are interconnected. Indeed, *“collating information from numerous tactical events contributes to an overall strategic view, while, in an administrative sense, a certain strategic outlook can guide future efforts towards certain types of tactical events”* [33].

The process to get intelligence from criminality, whether tactical, operational or strategic, can be summarised in different steps, which are illustrated in Figure 2:

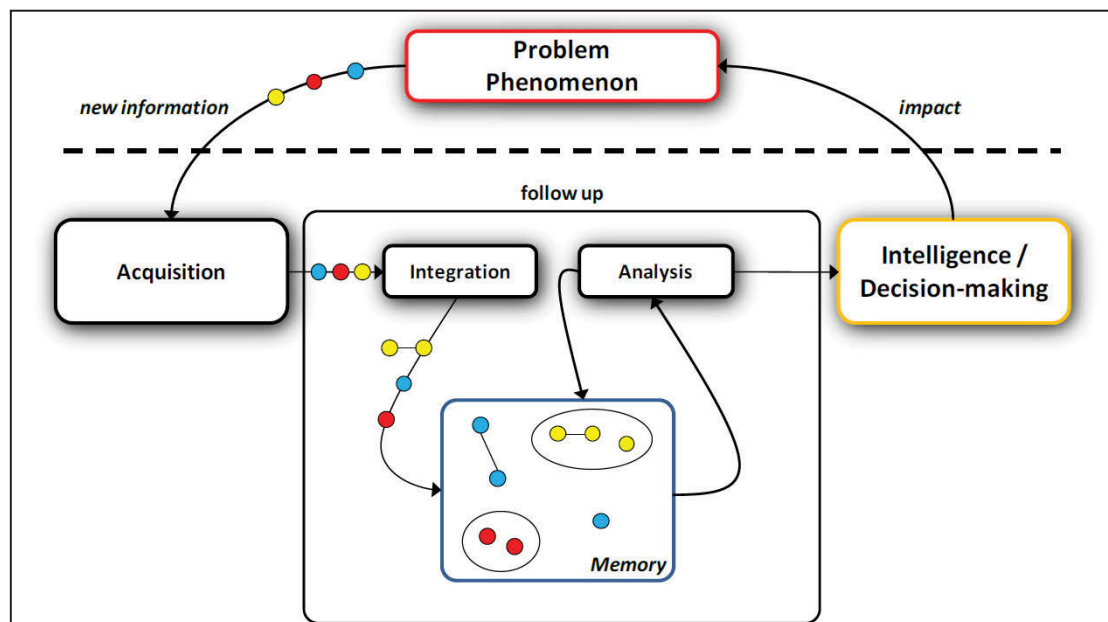


Figure 2: Forensic intelligence cycle process [34]

When an activity occurs, traces are engendered [1]. The investigation starts when a crime has been reported, and scientists are deployed to attend the crime scene where the search of

traces may lead to their detection. Detected traces, or forensic case data, are collected: whatever the type of trace, it is characterised by specific information. Information from all traces, which can be from multiple sources (such as physical traces, witness reports, etc.), is integrated in the memory [35]. The term memory is distinct from database, as it is organised in a structured manner, which is not necessarily the case of a database [10], as defined by Morelato [10]: a memory is *“an organised structure representing the knowledge the organisation operating the process has, at a certain time, about criminal activities (e.g. trends, linked cases, patterns, serial crimes, etc.). In contrast to the database, the memory contains information that is systematically structured to help form meaningful outcomes”*. The memory is designed thoroughly as the organisation and structure of the information it contains is specific to the exploited traces and the expected outcome of law enforcement agencies [28].

Once the information is stored in the memory, it is analysed to detect potential patterns: links or trends may be obtained [36]. For both types of patterns, the assumption is that if traces from different cases share similar characteristics (information from traces entered in the memory), they may describe a repetition from a common source or cause [37]. This source (or cause) could be a person or the trace itself, and it might thus be possible to associate trace to person, person to person, person to trace or trace to trace [2]. To assess and interpret the meaning of detected patterns, the expert knowledge is required [38]: this step is crucial as intelligence is produced.

Visualisation is important to communicate the results of the analysis and avoid any ambiguity or subjectivity [39, 40]. The critical decision is to define an appropriate visualisation to the problem studied and is often related to its dominant dimension (temporal, spatial, relational or quantitative) [39]. If inappropriate, the visualisation might lead to biases, and results will not be correctly interpreted.

Finally, outcomes are communicated to decision-makers [10], and action may be undertaken [41]. The action can have different forms, such as having an impact on social control, crime reduction, harm prevention, etc. [42].

The process is referred as an ‘intelligence cycle’: new information entering in the memory may influence outcomes. Thus, all information contained in the memory and detected

patterns are iteratively reassessed [39]. The added value to an iterative system is that it minimises the problem of linkage blindness [43].

Overall, this forensic intelligence process is based on a balanced interaction between a human and a computer [44]. The digitalisation facilitates the storage of information [41, 45], but it is also increasing the amount of data and agencies are currently facing backlog and information overload [46, 47]. It can be illustrated with the ease of taking pictures nowadays with smart phones when witnessing an event, or the increased number of CCTV cameras installed in cities: if employed for investigations, the information pictures and videos represent a massive dataset. The challenge when being confronted with a massive amount of data is to determine the useful and relevant ones [20, 48]. This is why analysts are essential [49], as it is their role to identify and interpret the relevant information from the massive dataset [12, 50, 51], unlike the common belief that patterns are automatically extracted: a deeper analysis requires a human involved in the process [12, 52, 53].

Police and more broadly law enforcement agencies' ultimate aim is to disrupt criminality. Thus, forensic science services (and more precisely forensic intelligence agencies) measure their 'efficiency' looking at the resolution of crimes [24]. It is often recommended to find solutions to remove the *"issue of unconnected cases"* [54] (i.e. when no link or trend has been established yet). This reasoning is flawed, as unconnected cases are not due to 'non-efficiency' [55]. Isolated cases can provide valuable information, and they might be linked later on with a new case integrated in the memory [8, 37]. The primary aim is therefore not to ultimately obtain a link or trend, but first to identify relevant information and structure it in the memory to simplify the comparison process, which may in turn lead to a pattern. The problem should be analysed from the opposite way and using the information of these apparently unrelated cases to (maybe) identify new links or trends. As highlighted by Margot [2], a pattern *"may not be detected, unless one has an indication of the type of event that is being investigated. It is the result of an activity, which is in the past and cannot be reproduced, only inferred"*. Thus, links or trends may not be identified at first, nevertheless with a growing memory and additional information, patterns may be detected later on [55].

A study conducted by Ribaux et al. [56] demonstrated the importance of the iterative assessment of the information contained in the memory to detect series of crimes. Indeed,

burglaries were reported in Switzerland in a certain period and crimes had been linked based on same DNA detected on different crime scenes. However when adding new information which was shoe marks collected on crime scenes from new burglaries, new links were made between crimes and they were added to the already detected series. Isolated cases were originally not attributed to the identified series as no DNA had been retrieved on crime scenes, but the iterative process enabled to link those cases thanks to new information (shoe marks) entered in the memory.

Besides, the forensic intelligence process may be deployed in a general to the particular approach as described by Morelato et al. [37]:

1. **Surface level:** An overview of the criminal phenomenon may be obtained, allowing detecting general patterns without consuming many resources: mainly strategic intelligence is produced.
2. **Modus operandi level:** this is developed once a pattern has been detected. Different modus operandi may be identified, so more resources may be engaged to detect related criminal activities.
3. **Series level:** The attention is focused on selected cases which have raised interest for investigators, so many resources may be deployed to identify series (i.e. time invested, individuals involved in the investigation process, identification methods, etc.). Tactical intelligence is usually produced at a series level.

Starting with a general point of view, the criminal phenomenon is studied from a surface level, where general trends of the problem may be obtained. As soon as a particularity is noted, which may be attributed to similar modus operandi between cases, more resources may be deployed. If specific cases are of interest and should be scrutinised, even more resources are invested to arrest offenders. However, focusing on particular cases should not stop from having a general overview of the problem which is in constant evolution. Thus, the forensic intelligence process should be deployed simultaneously from a surface to a series level to stay as proactive as possible [57].

In the study from Ribaux et al. [56] described above, the reporting of burglaries was analysed geographically (only the geographic location of the crime and the type of offence was utilised which did not necessitate much resources to be deployed) and it was possible to determine hot spots where burglaries were occurring (i.e. trends obtained at surface level). Moreover, when looking at the information from those crimes, a number cases were linked based on the *modus operandi*, as doors' locks were broken in the same manner using a pair of pliers [20]. The information could be obtained as investigations were carried out with the search of tool marks requiring experts' knowledge (thus more resources deployed than to determine hot spots). Those cases with similar *modus operandi* were scrutinised and similar shoe marks were identified. It implies ACE-V¹ process by shoe marks experts, search warrant to find shoes corresponding to shoe marks at suspects' houses. Cases were finally linked to the same perpetrator through a case to case comparison (tactical intelligence was thus produced).

As seen in this section, forensic intelligence is transversal, i.e. it can be applied to any sub-discipline in forensic science: it can be applied to illicit substances, which will be the focus of this thesis. The next section expands on the concepts of forensic intelligence when applied to illicit drug trafficking.

¹ The ACE-V corresponds to the scientific methodology applied when examining forensic traces, which is divided into four consecutive steps: Analysis, Comparison, Evaluation and Verification [58].

1.1.2 Forensic drug intelligence

1.1.2.1 *Illicit drug trafficking and the use of drug profiling*

Illicit drug trafficking is considered as organised crime. The European Union's Council Framework Decision 2008/841/JHA defines organised crime as *"a structured association, established over a period of time, of more than two persons acting in concert with a view to committing offences which are punishable by deprivation of liberty or a detention order of a maximum of at least four years or a more serious penalty, to obtain, directly or indirectly, a financial or other material benefit"* [59]. In this case, the offence is the trafficking of illicit substances. Legislations are different from one country to another but this crime is internationally recognised and forbidden by the United Nations convention against transnational organized crime [60].

The trafficking is organised in widespread networks internationally that control supply and demand of illicit substances. The starting point of those trafficking networks is the production of the drugs [61], and many steps and intermediates are involved until the end user (i.e. consumers), which makes illicit drug trafficking a very complex multi-layered organisation, as illustrated in Figure 3. The proposed structure may be applied to every trafficking group, with more or less intermediates depending on the size of the trafficking network.

Different roles are attributed within each level of the network, from the manufacture, to the wholesale, to the retail and finally the consumer of the drug. For instance, at the manufacture level, many roles are assigned: operators are leading operations and are in charge of accounting, cooks are working on the actual manufacture of the drug, workers are in charge of the distribution of the product to the wholesaler, and other professions may be involved such as pharmacists for instance to provide manufacturing compounds [61].

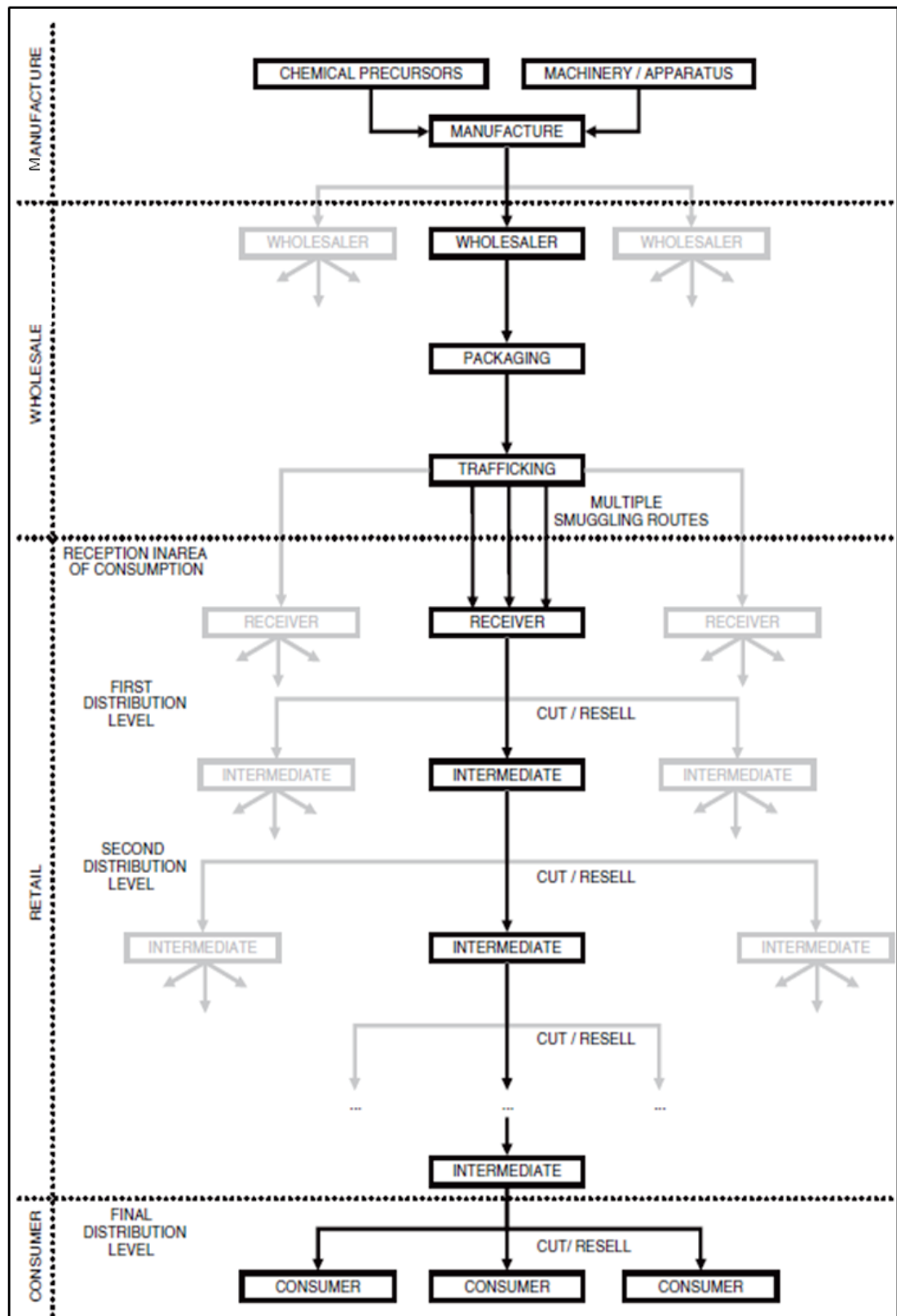


Figure 3: The structure of illicit drug trafficking [33]

Illicit drug trafficking is the number one trafficking commodity of international organised crimes, more widespread than terrorism or illegal immigration [62, 63]. Thus, law enforcement is particularly focused on the dismantling of trafficking networks at international, national, regional and local scale [64]. In many countries now a policy of interdiction has been implemented, with intensified border controls and deployment of resources to arrest offenders within the territory [64]. The goal for law enforcement agencies is to find 'proofs' of the trafficking through the seizure of illicit substance, either via random controls (travellers, baggage, but also mails at the Post) or following an investigation (warrant search or offenders' arrests).

Once a seizure occurred, it may undergo a drug profiling procedure. Esseiva et al. [65] define drug profiling as *"the extraction of a drug sample's chemical and/or physical profile, to be used in the application of policies against the illegal use of drugs (law enforcement, legislation, public health, etc.). The profile of a drug sample is a subset of the sample's characteristics specifically chosen with respect to the purpose of the process"*. This definition implies several concepts developed hereafter.

In a traditional Court driven model, different types of information from a seizure are expected:

1. The identity and composition of the substance, conditioning the innocence or guilt of a suspect;
2. The purity of the substance, determining the type of offence and / or sentence.

To answer these types of questions, the procedure starts with a sampling of the seizure. It would not be efficient and would require too much time and cost invested to utilise all seized product. For this reason, a sampling of seizures is performed. Numerous standardised procedures exist, which are different for qualitative [66, 67] (i.e. substance illicit or not and its identity) or for quantitative purposes [68-71] (i.e. purity of the drug and chemical composition) and differ from one country to another. The overall aim of the sampling is to try to stay as close as possible from the original composition of the seizure [72], regarding its concentration and homogeneity (i.e. composition) [73, 74]. To do so, the size of the seizure has to be taken into account, as well as the physical characteristics and similarities between different items within the seizure (i.e. visually differentiable or not). A **sample** is finally

obtained, which is different from a **specimen**. A specimen can be any unit of a seizure (or the entire seizure). Its representativeness of the whole unit is not assured. Conversely, a sample is a representative unit of the seizure, because a standardised sampling procedure was applied to the seizure to obtain the sample [10].

Different analytical techniques are applied to the sample to extract relevant characteristics (physical or chemical) to form its **profile** (or profiles if more than one method is used) [75].

Information relative to the shape, colour, packaging, etc., corresponds to a **physical profile**. Physical characteristics may be used for intelligence purpose. For instance, Marquis et al. [76] studied physical characteristics of 3,4- Methylenedioxymethamphetamine (MDMA) tablets seized in different countries, such as their logo, shape, colour, etc. They identified different trends in processes to produce pills of MDMA depending on the country (strategic intelligence) and they established operational links between different seizures based on their physical profiles.

Moreover, the **chemical profile** can be used to highlight the major or minor chemical compounds of the sample, or other compounds added during the manufacturing process of illicit drugs, which may leave traces in the final product [35, 77, 78]. Accredited analytical laboratories employ standardised methods to get chemical information from samples. This chemical information obtained from samples could help to identify illegal production processes [79], and they may vary depending on:

- Co-extracted compounds from starting material for plant-based drugs, for instance, the coca plant for cocaine and poppy seeds for heroin;
- Manufacturing chemicals (solvents for example);
- Manufacturing process (i.e. synthesis pathway);
- Chemical modification due to environmental conditions (i.e. geographic region of the growth of the plant-based drugs implying varying climatic conditions, the soil used to grow the plant, UV light, heat, humidity, etc. [80]);
- Ageing of the drug itself [81, 82].

The information may be used for example to classify samples depending on their geographic cultivation site. Indeed, isotope analysis has been successfully employed to discriminate respectively cocaine and heroin samples, by identifying countries of origin and more specifically a potential mapping of cultivation sites per regions within countries based on seized drugs during a certain period [83].

Furthermore, the final illegal product to be sold is usually mixed with other compounds, called **cutting agents**, to increase its mass and its 'drug capacities'. Increasing the mass permits to sell a higher quantity of product and obviously make better profits from the same quantity of pure material [84]. Cutting agents, also part of the chemical profile, can be divided in two sub-categories:

1. The **diluents** are pharmacologically inactive, easy to obtain legally on the market [85];
2. The **adulterants** are pharmacologically active and may or may not be controlled substances [86]. For example, caffeine can be added due to its stimulatory effects on the central nervous system [87]; paracetamol is added as it permits a faster absorption into the brain.

Cutting agents may be added at different levels of the trafficking network and their analysis is also interesting information to get a better knowledge of the distribution chain of illicit products (strategic intelligence). They are directly correlated to the purity of a sample (the more cutting agents added, the less pure the final product will be) [85], and their analysis may allow for example to establish trends over time of the ones the most employed [88].

This thesis will focus on the use of chemical characteristics obtained from drug seizures for intelligence purposes: their usage and usefulness to fight against illegal drug trafficking is presented in the section hereafter.

1.1.2.2 Chemical profiling for intelligence purpose

Intelligence can be obtained from drug profiling, including chemical profiles, as illustrated in Figure 4:

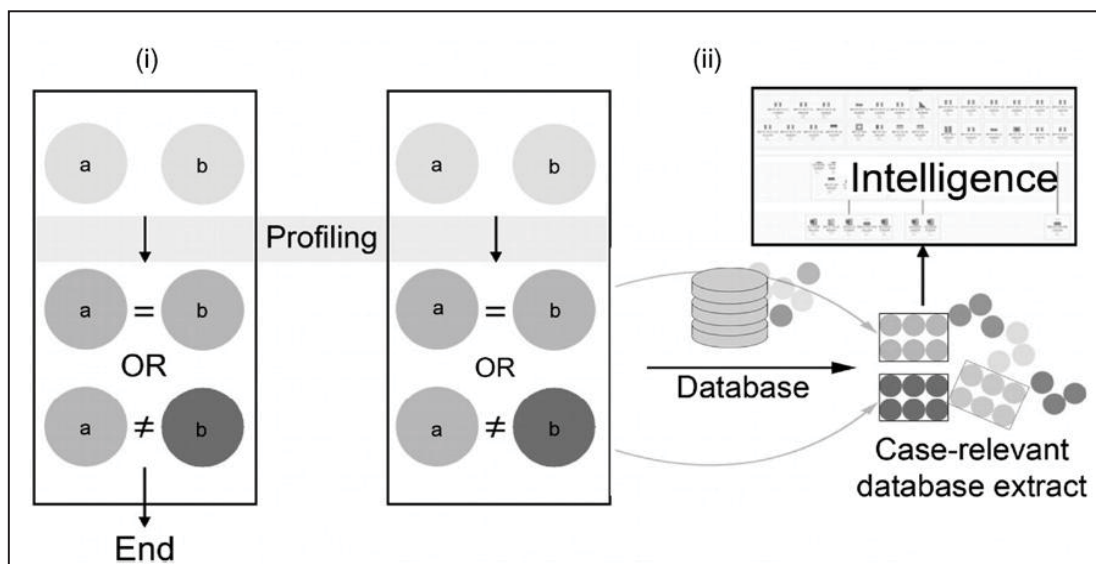


Figure 4: Different conceptions of drug profiling [65]

- “(i) Punctual comparison of two specifically selected samples of drugs,
(ii) Systematic analysis, classification and comparison of samples of drugs.”

A standardised routine procedure is required to acquire the chemical profiles, in order for them to be comparable over a long period [89]. Chemical profiles are stored in the database (or memory) and an automatic process is implemented to extract relevant information [65, 78, 90]. Case-to-case comparisons are made for investigation and court purposes and correspond to tactical intelligence. If the process is performed systematically, with each new specimen entering the memory being compared to all previously integrated ones, operational links and strategic intelligence may be obtained [10, 65]. Consequently, using chemical profiling in an intelligence-led manner provides a better understanding of the illegal drug trafficking situation at local, regional, national and international levels.

Different types of relationships within the trafficking network can be established using this drug intelligence process, depending on the desired outcome of the police (i.e. tactical, operational, or strategic) [91-93], as illustrated in Figure 5:

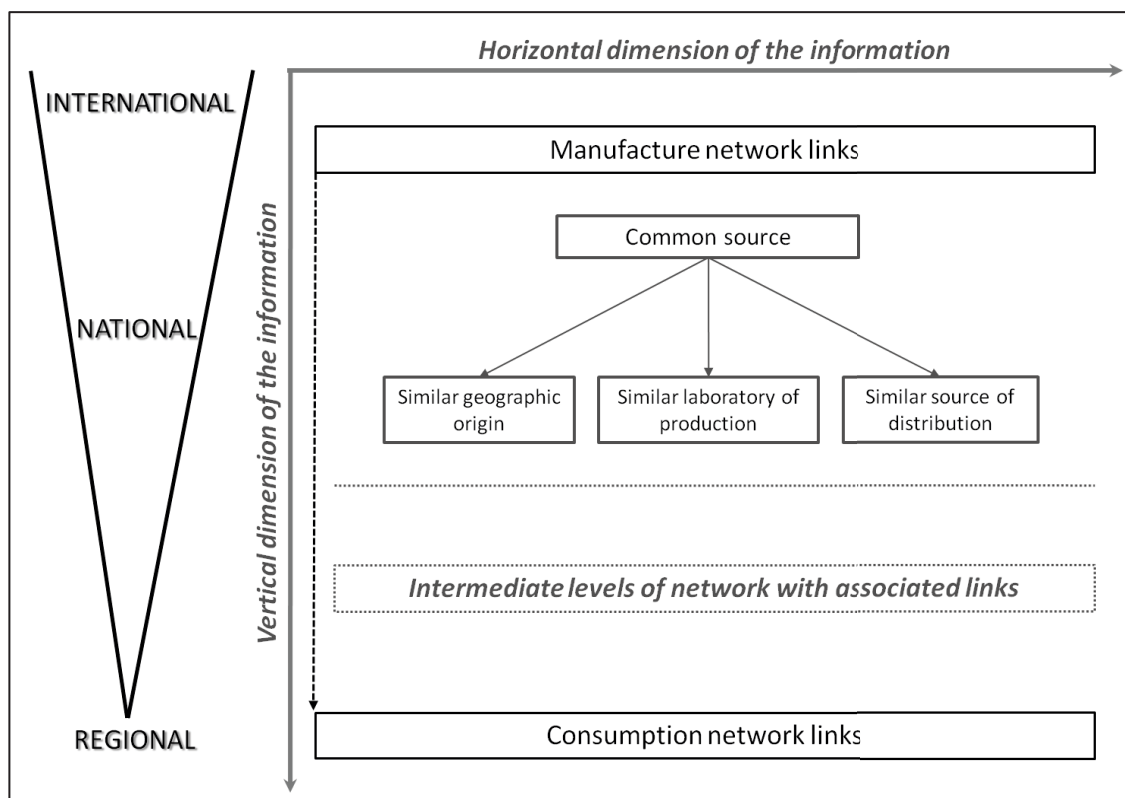


Figure 5: Two-dimensional representation of links' inference depending on the trafficking network's level

At the same level of the trafficking network (i.e. horizontal dimension of the information), links may be identified for instance if different seizures come from a 'common source' [93]. A 'common source' could be related to similar geographic origin, or the laboratory of production of the drug, or the source of distribution, or the unit considered before distribution, etc. [35]. As no rigorous quality control is undertaken in clandestine laboratories² [94, 95], specimens may be related to the same source because they contain the same manufacturing impurities, due to poor chemical handling during synthesis [91].

The establishment of a common source for intelligence purpose based on chemical profiles of drugs is well recognised as it has been studied internationally for many years [22, 65, 75, 96-

² Clandestine laboratories are defined as "facilities operated by organized criminal groups that are used to covertly manufacture illicit drugs or precursors" [61].

102]. Concretely, the evolution of the market and trafficking networks may be identified, if focusing on the temporality of an illicit substance [93]. Patterns over time were for instance established with a study conducted in Switzerland using chemical profiles of cocaine and heroin seizures stored in the database for eight years [97]. Extracting relevant chemical information from all specimens allowed the detection of links between similar chemical profiles. Linked seizures were coming from different jurisdictions, which highlighted the *“transjurisdictional nature of the market”* [97] and thus the need for further collaboration between law enforcement agencies in the different jurisdictions. Moreover, it appeared that different trafficking networks existed in the territory and their evolution in time could be determined based on the established links between specimens.

Additionally, links may be established at different levels of the network, from the manufacturer level to the consumer level (i.e. vertical dimension of the information). For instance, a common source may be determined at intermediate levels of the network (links attributed to a wholesaler or retailer) [35]. Specimens may also be related to the same distribution network as containing the same chemical compounds (for example cutting agents), even if not coming from the same manufacturer [103]. This information is directly related to the distribution network and reinforces the knowledge about the structure of trafficking networks. As an illustration, Broseus et al. [86] studied cutting agents found in seizures of cocaine in Switzerland which provided a relationship between presence of particular chemical compounds and the level of distribution. More specifically, if diluents like glucose, lactose or mannitol were not found in seizures, this indicated that they were actually added to cocaine product after it had been imported into the country.

Finally, intelligence produced from the profiling of seized drugs may assist police and other stakeholders to take actions. It is well summarised by Huttunen et al. [33]: *“an increase in seizures for a certain drug in a particular geographical area (an increase in ‘tactical level events’) can promote the ‘strategic level view’ that the area is important to the distribution of that drug. This in turn may cause increased resources to be directed into that area (a ‘strategic decision’), which consequently allows more investigations and, presumably, more seizures (‘tactical events’) to take place”*.

1.1.3 Forensic drug intelligence's implementation in Australia

According to the Australian Criminal Intelligence Commission report of 2014-2015 [104], illicit drug seizures have considerably increased in term of number and weight over the last decade: resources deployed to fight against drug trafficking seem efficient. However, the increasing number of arrests combined with a continuous consumption recorded on the territory is a concern for law enforcement agencies, which consider this criminality as a priority [104]. Nevertheless, even if the suppression is known to be efficient in stopping the detected and known criminality, it does not eradicate the whole problem [105, 106].

Law enforcement agencies have to face those limitations, while trying to provide accurate results in a timely fashion with as few resources as possible. This was explicitly stated in the Australian Criminal Intelligence Commission report on intelligence management of 2017 [107] where factors conditioning a 'proper success' of intelligence implementation were identified for the next few years. They included a *"more efficient use of limited resources and better risk management practices; (...) established best practice doctrine with emphasis on consistency in guiding principles and a program of continuous improvement; (...) contemporary technology to enable more effective collection, analysis, storage and sharing of national intelligence assets"*.

The intelligence approach appeared in Australia from the late 1990's [108]. Nowadays, intelligence-led policing has been developed and is employed by law enforcement agencies at federal and state levels [10, 32, 80, 104, 109, 110]. Moreover, the Australian Journal of Forensic Science published a forensic intelligence issue in 2015 (Volume 47 Issue 1) covering all approaches and challenges in the forensic intelligence field already identified throughout the years, especially from an Australian perspective [2, 18, 27, 62, 111]. Thus, there is no doubt that initiatives are undertaken to gain more implication in a more intelligence-driven approach.

This thesis will focus on the illicit drug problem from a federal point of view in Australia. The Australian Federal Police (AFP) manages all seizures performed at customs across the territory as well as seizures made in the Australian Capital Territory (ACT). The division at the AFP responsible for drug intelligence is the Australian Illicit Drug Data Centre (AIDDC) [10, 47].

When a seizure is performed at custom and in the ACT, physical information is recorded, such as dimensions, weight, colour, shape, etc. Colourimetric tests are performed to obtain a first indication on the nature of the seized compound. The information is recorded into a Physical Examination Data Input Template (PEDIT), and it is stored in the Australian Illicit Drug Intelligence Program (AIDIP). Its aim is to provide forensic drug intelligence based on chemical and physical characteristics of drug seizures [53].

The Police Real Time Online Management Information System (PROMIS) is another database used by AFP grouping all investigative information regarding seizures (this information is not found in AIDIP but can be accessed easily using the case number of seizures).

Moreover, a sampling procedure is applied to the seizure, according to the United Nations Drug Control Program [109] (see Figure 6). The seizure is separated into different subgroups, these subgroups representing visually differentiable objects (i.e. different colours, shapes, properties, etc.). Each subgroup may comprise several items.

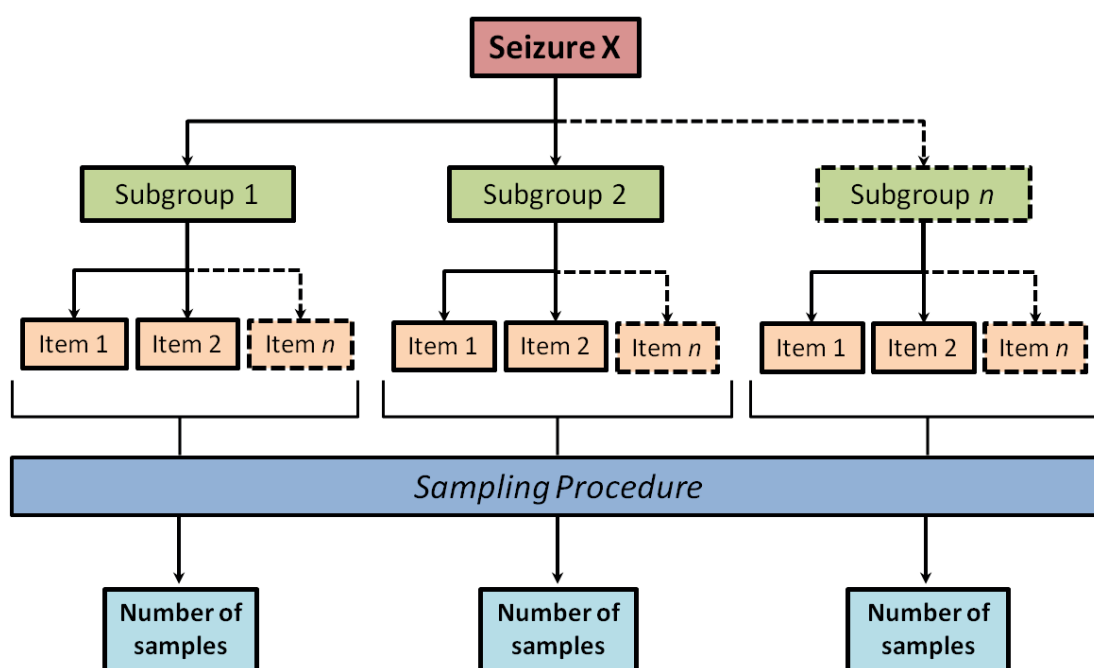


Figure 6: Australian Federal Police standardised sampling procedure

The number of samples for each subgroup is calculated precisely according to the following process³ (Table 1):

Table 1: Sampling procedure in Australia

Number of items per subgroup	Number of samples to be obtained per subgroup
1 - 10	n
11 - 100	10
> 100	\sqrt{n}

Subsequently, the samples are sent to a centralised laboratory, the National Measurement Institute (NMI), to establish their chemical profiles [80]. NMI performs different types of analyses on each sample depending on the suspected illegal nature of the substance (i.e. different analytical techniques depending on the nature of the drug; Detailed explanations on the NMI procedure are provided in Chapter 2 for analysis of cocaine and heroin samples). Results, which constitute the chemical profile of the samples, are then stored into AIDIP.

The whole procedure is summarised in Figure 7:

³ Personal communication from AFP.

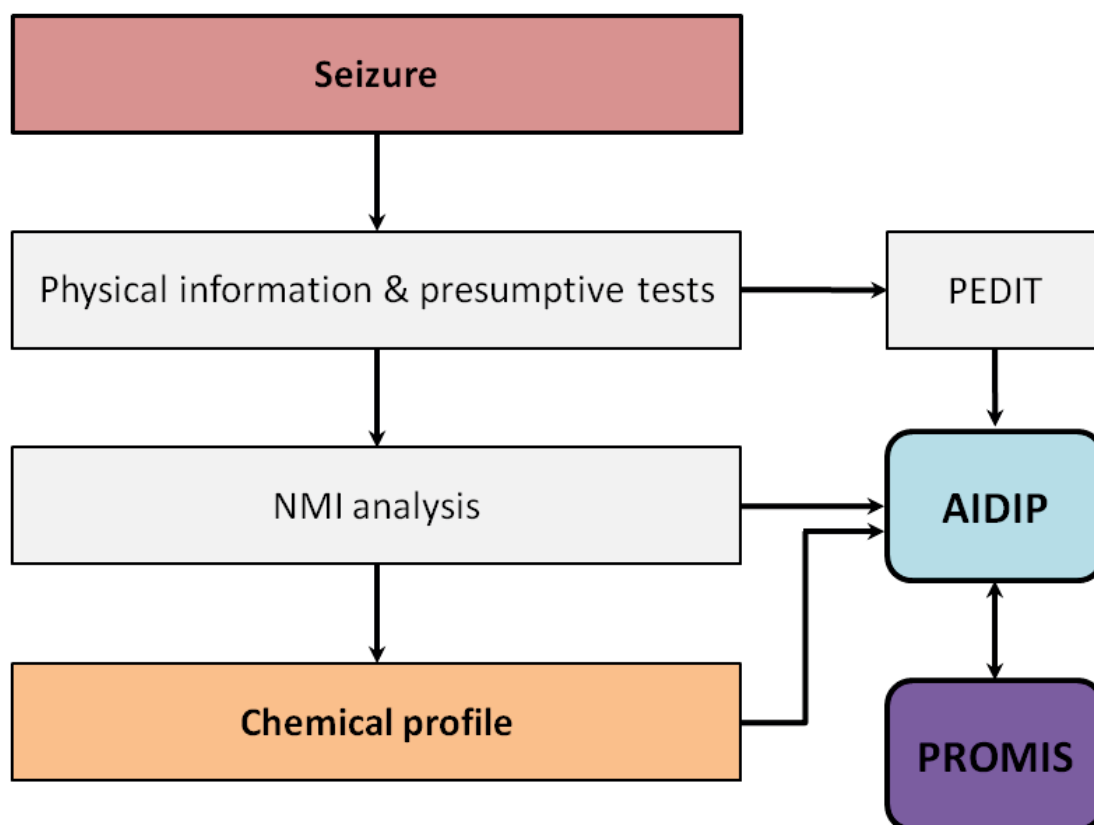


Figure 7: Australian Federal Police procedure regarding drug seizures

The work conducted at the AFP in the fight against drug trafficking is part of the Australian criminal intelligence model in which objectives are defined by the Australian Criminal Intelligence Commission [107], in particular providing tactical, operational and strategic decision-making.

Collins et al. [112] published in 2017 an article looking at the outcomes of the intelligence procedure developed since 2003 (year of the creation of AIDIP). Strategic intelligence was given as an illustration: looking at chemical information extracted from methylamphetamine (MA) border seizures over the years stored in a centralised database allowed to detect trends for the manufacturing of this drug [112]. Precursors were detected in seizures with similar quantities and ratios as MA produced in Mexico. As a result, MA seized in Australia was identified as being imported from Mexico, which was further confirmed with information from American law enforcement agencies.

Moreover, a Ph.D. was conducted at the University of Technology of Sydney in collaboration with the AFP with the aim of getting intelligence from chemical profiles of MA and MDMA

border seizures stored in the database [10]. Operational intelligence was produced: links were detected which allowed the AFP to link cases based on chemical information [102], in addition to cases which had been previously linked based on investigative information. The study demonstrated that chemical information may bring new knowledge about the market and links between seizures if employed in a systematic manner, instead of the traditional case-to-case approach.

Both studies emphasise how chemical profiles of drug seizures may be used for intelligence purpose in Australia.

1.2 Where to from here?

Collaborative works have been developed over the years and have proven their efficiency, notably to counteract the “*justice silo effect*” [14, 32, 113-118]. Projects such as the Collaborative Harmonisation of Methods for Profiling of Amphetamine Type Stimulants have been developed to obtain a harmonised profiling method and database for amphetamine type stimulants over European countries [76, 119]. Operational links from chemical and physical characteristics were possible for MDMA tablets [76, 120, 121], which underlines the benefit of a centralised memory where all stored information is extracted at once for intelligence purposes.

Moreover, collaboration implemented internationally allows to inform each other (government or international agency) about ongoing operations [80, 122]. Numerous law-enforcement agencies have developed coordinated centres internationally⁴, as described by Cockayne and Williams in their 2009 report, to fight against illegal drug trafficking, to increase the exchange of information inter-country and gaining more proactivity [122]. Consequently, the understanding of trafficking routes may be facilitated, and it may allow a better representativeness of drug consumption and associated health harms in geographic regions at international scale.

Despite all those collaborative achievements, the forensic intelligence approach implies that actors in the fight against illegal drug trafficking are aware of the opening of new possibilities in acquiring data not only for prosecution but also for long term planning, notably for strategic intelligence. From an operational point of view, the United Nations Office on Drugs

⁴ These initiatives include, but are not limited to, UNODC’s Operational Plan, ECOWAS’ Regional Action Plan, CARICC, EUROJUST, DPKO-DPA-UNODC-INTERPOL assistance program.

and Crimes (UNODC) stated that law enforcement being focused on groups of traffickers instead of drug markets was inhibiting the development of effective strategies [123-125]. This emphasises the fact that, in an intelligence-led driven approach, attention should be focused on the overall picture of criminal phenomena instead of targeting individuals.

Procedures are already implemented to arrest offenders and instruments are already deployed to detect and identify illicit substances. Technology is nowadays reaching its height with minimised instrumentation that can be easily transported, as well as great selectivity and sensitivity for analytical instruments giving accurate results in small magnitudes (LODs in ng ranges are common nowadays with laboratory-based instruments [126]). Besides, computer-based tools designed and deployed for every application are omnipresent. The goal is not to change those procedures and replacing instruments currently used and functioning for their end-purpose [5]. The idea is to find manners to utilise information that is not required for court purpose, because resource, time and money have been invested to collect the information. Moreover, the goal is also to find ways to utilise information usually employed for prosecution to obtain more knowledge about criminal phenomena. All of this may be achieved if studying the problem as a whole: the fight should be undertaken from different points of view [127, 128].

Analysing results from chemical profiles of seizures to determine if cases could be related is the approach traditionally employed. But it is not the only one and other orientations are currently undertaken that should be used in a holistic manner, whereas they currently tend to be looked at individually. Many fields could be cited; For instance, research is currently conducted in the field of the darknet [98, 129-133], providing a better understanding of illegal drug trafficking, such as the distribution and consumption networks, through a virtual environment (i.e. Internet). Furthermore, studies are conducted to analyse wastewater, to inform on the daily consumption of illegal substances in a particular city or region [96]. Early outcomes demonstrate that conducting these analyses routinely would allow to provide data in real time and also monitoring geographic region on the evolution of drug consumption [134].

Moreover, using jointly results from chemical analyses and from criminological studies, such as consumer surveys, would add knowledge about drug trafficking especially once the drug is already imported in a country. Indeed, sociological and criminological studies have proven that the consumption of illicit drugs is related directly or indirectly to crime (referred as the

Goldstein framework) [135]. People may commit crime under the effect of a drug, they may also commit crime to get money to pay for the drug they use [135]. In addition, the profit generated by drug trafficking may prompt individuals to get involved in crime [135]. As emphasised by the UNODC 2016 report, drug trafficking is a crime reinforcing conflicts locally or nationally [125]. As a result, if drug consumption is reduced, it may lead to a reduction in this direct or indirect criminality [17, 135].

Furthermore, the understanding of the drug trafficking problem can be expanded using intelligence to identify and dismantle, for example, doping products trafficking networks to which athletes could be related [136]. Research is currently being undertaken in this field [29, 130], since anti-doping presents the same problem as drug profiling due to the illegality of the substances, generating international trafficking networks. Thus, these different fields are comparable when orientated in an intelligence perspective [28, 30]. Studies have already been carried out to emphasise the transversality of forensic intelligence within two apparently unrelated fields, such as false identity documents and drug profiling [22, 37]. It has been demonstrated that the main issues are similar, such as the determination of relevant data to collect, extraction of information, and also the integration of the outcomes depending on the context [37, 137].

As a result, the triangulation of data from various sources has great potential in a forensic drug intelligence perspective to expand the knowledge of the phenomenon, which is the central question this research will focus on.

1.3 Aims and objectives of the research

Research may complement police work by investing time in looking at possibilities to facilitate the extraction of relevant information in police's databases and the detection of patterns.

One Ph.D. was conducted in Australia to assist the AFP in getting a better understanding of illicit drug trafficking [10], and to date no other collaborative work between research and law enforcement agencies was conducted for this purpose. Moreover, the study conducted with the AFP was focusing on MA and MDMA chemical profiles. Further research for forensic drug intelligence purposes is needed, focusing on other drugs to get a better understanding of illicit drug trafficking in Australia.

Those reasons lead to the aim of this research which is to **obtain a better knowledge of illegal drug trafficking**. Two different approaches (study 1 and study 2) are adopted following the same framework with a view of using various source of information for intelligence purposes.

Study 1 is based on the study previously mentioned which investigated the use of MA and MDMA chemical profiles in an intelligence perspective in Australia [10]. The aim of this study is to investigate chemical data of cocaine and heroin seizures performed at Australian borders, to gain a better understanding of trafficking from a chemical point of view. It is intended to streamline cocaine and heroin chemical⁵ profiling processes currently conducted

⁵ This research only focused on chemical profiling. Indeed, for confidentiality and access constraints, physical characteristics were not studied.

at the AFP and provide guidelines for developing a more effective capability to get timely results.

The objectives are:

- To gain information about chemical composition of cocaine and heroin seizures. This is presented in **Chapter 2**.
- To develop and optimise a statistical model enabling the establishment of links and trends of cocaine and heroin seizures based on their chemical composition. This is presented in **Chapter 3**.

Study 2 deviates from traditional analysis and chemical profiling methods. The main aim of this study is to investigate a new possibility to use chemical information from drugs for intelligence purposes. The feasibility for an optimised non-destructive methodology to detect remnants of illegal substances present on the surface of passports is assessed, using rapid instruments currently deployed at customs: this is presented in **Chapter 4**.

Both studies are complementary as using the same intelligence approach, as illustrated in Figure 8. The benefit of using complementary information obtained from the two studies is investigated and critically assessed in a forensic intelligence perspective in **Chapter 5**. This chapter also concludes the overall research and provides some reflection regarding future directions in the field.

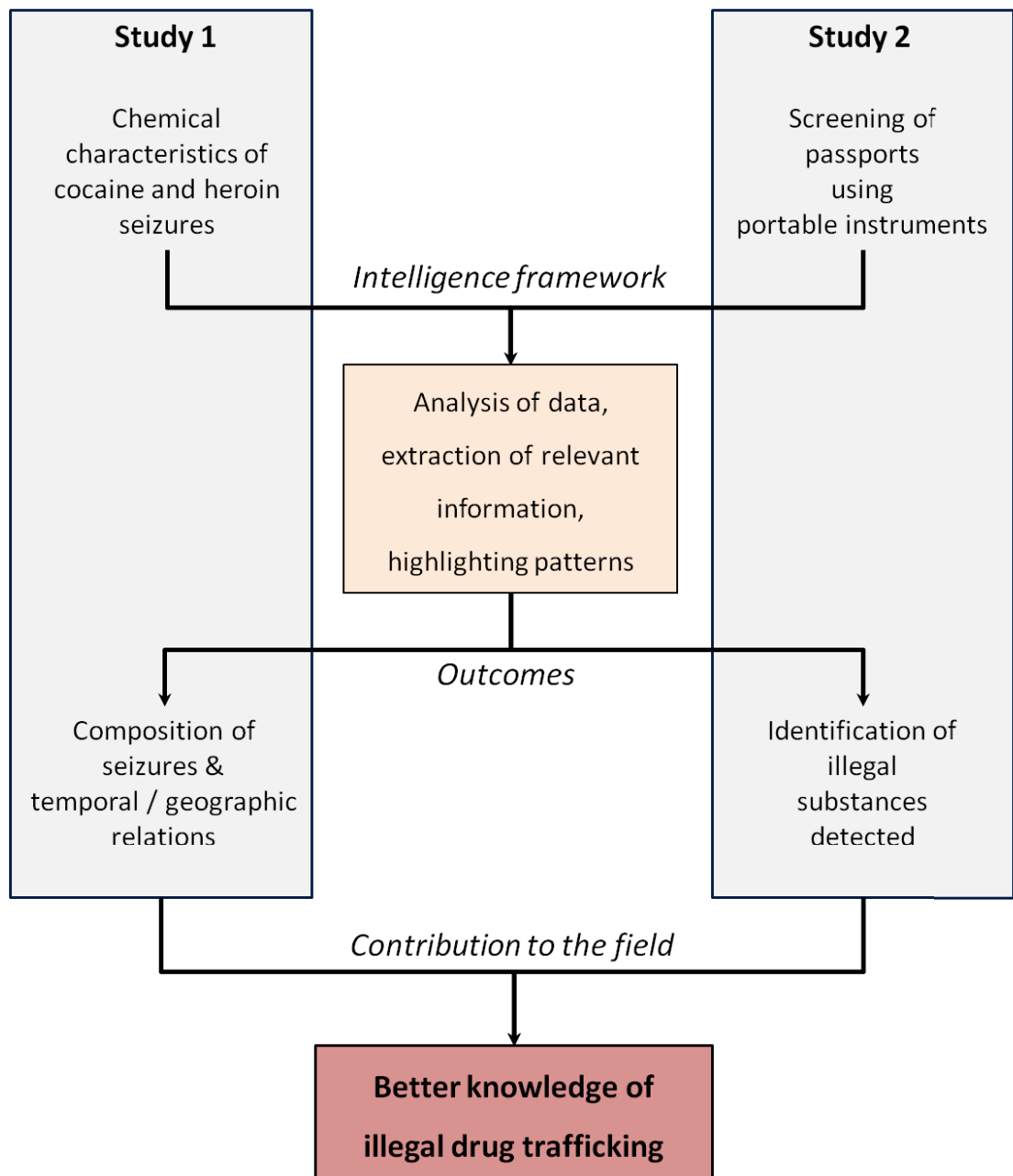


Figure 8: Summary of the research

Chapter 2:
Chemical trends of cocaine and
heroin border seizures

Chapter 2: Chemical trends of cocaine and heroin border seizures

2.1 Introduction

2.1.1 Background concepts: from cultivation to consumption

2.1.1.1 Cocaine

Cocaine is a plant based drug, the starting material being extracted from the *coca* plant. The stimulating properties of cocaine have been known for a long time. Historically, leaves of the coca plant were chewed by Incas to eliminate fatigue. It was not until 1860 that the first extraction of cocaine and its isolation from the coca leaves occurred [138]. It was later used in many commercialised products, such as Coca-Cola, cigarettes and chewing gums. However, the realisation of the harmful effects and highly addictive ability of cocaine led to the progressive prohibition of this compound from the early 1920's [138].

Two hundred types of coca plants exist, but only two species provide the best cocaine yield: the *Erythroxylum coca* and *Erythroxylum novogranatense* [139]. These plants require a lot of heat, as well as a certain altitude (500-1500 meters), and a specific type of soil to grow properly [140]. As a result, this plant exclusively grows in the region of the Andes in South America. The starting point for cocaine cultivation is thus always located in this geographic region.

The process of production of cocaine is quite long: the amount of coca leaves required to obtain 1 kg of cocaine (hydrochloride) is around one ton (see Figure 9). For *Erythroxylum coca*, 0.3 to 1.5 % of the total leaf's weight is actual cocaine [141].

The production of cocaine is a synthesis based on successive extractions of miscible / non-miscible chemicals, involving three steps⁶:

1. Extraction of crude coca paste from the coca leaves;
2. Purification of coca paste to coke base;
3. Conversion of coke base to cocaine hydrochloride [140].

After the extraction from coca leaves, the crude product is usually refined to remove two major impurities, cis and trans-Cinnamoylcocaine, eliminating colouration to give a whiter product⁷. The purification is done by adding potassium permanganate to the acidic solution of cocaine: this is the oxidation step, producing coke base [82, 144].

Liquid-liquid extractions using various solvents allows the conversion from the coke base to the cocaine hydrochloride [140, 146].

⁶ Cocaine synthesis has already been described precisely in the literature, and it can be found in [95, 140, 142-144] for detailed explanations.

⁷ A study released that if aged, the cocaine specimen may go back to a brown colouration depending on the storage conditions [145].

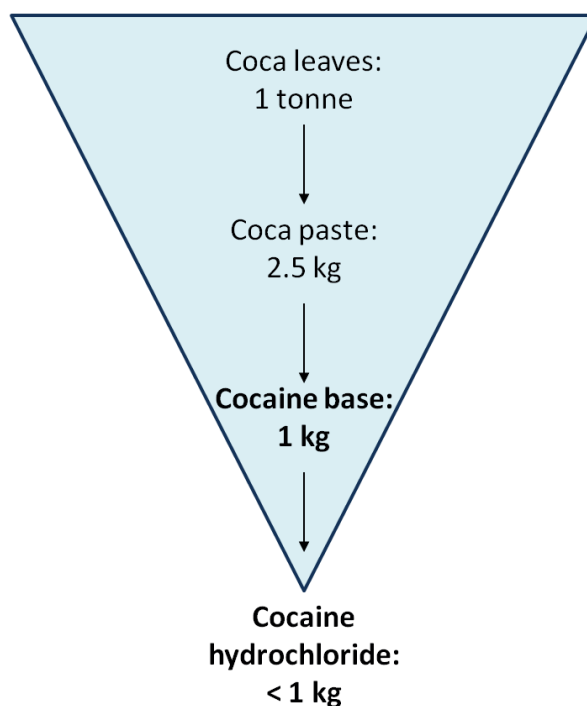


Figure 9: Reduction in yield of cocaine during production⁸.

The resulting pure product of cocaine hydrochloride is thus a mix of natural compounds coming from the coca leaves, also called alkaloids, and synthetic compounds coming from the different steps of synthesis, that is to say the solvents.

Two types of solvents are employed for cocaine synthesis: “wetting” solvents, affecting the outside surfaces of the crystals of formed cocaine, and “occluded” solvents, entering into the crystals, thus directly altering the chemical composition [147]. Solvents entering the crystals (i.e. occluded solvents) are likely to be detected in the final profile as retained by the compound itself.

⁸ Figure inspired from values in Guéniat et al. [141].

Cocaine is usually distributed in its hydrochloride salt form or as cocaine free base (also referred to as crack). Sold products can be divided in three main categories, depending on their way of consumption [148]:

1. Cocaine freebase powder, for inhalation;
2. Cocaine hydrochloride powder, for venous injection;
3. Freebase paste and crack rock, both for smoking.

An increase of cocaine consumption has been observed over the past several decades internationally, expanding world market demand and resulting in the proliferation of criminal trafficking networks [125, 149-151]. Therefore, the fight against drug trafficking has become one of the main goals in South America, but has only led to a continuous shift of cultivations from one area to another in the Andes [151].

The United Nations Office on Drug and Crime (UNODC) report in 2016 stated that cocaine seizures increased worldwide [125], and more specifically in Australia [125]. In February 2017, the largest seizure of cocaine ever made by the Australian Federal Police (more than 1.4 tonnes) occurred in New South Wales [152]. The number of cocaine detections by the Australian Customs Service at Australian borders is increasing each year, as reported by the Australian Drug Trends [153]. The period 2015-2016 recorded the highest value, with 2777 border seizures [154]. Likewise, the Australian Crime Commission stated in their annual 2015-2016 report that the number of national detection (number of seizures and weight) of cocaine was higher compared to the last decade, with a total of 3956 seizures for a total weight of 657.1 kg [154]. Cocaine trafficking in Australia is thus of national concern [155].

2.1.1.2 Heroin

Heroin is part of a family of compounds called opioids, all derived from the poppy plant (*papaver somniferum*), requiring specific climatic conditions to grow, which is the reason why its cultivation is located in the Golden Triangle (Southeast Asia and the Middle East) and Golden Crescent (South West Asia) [110, 156]. Many species exist, but only two are cultivated

for their high morphine content: *papaver somniferum album* and *papaver somniferum glabrum* [90]. The part of interest in the plant is the capsule which contains all alkaloids. An incision is made, allowing the raw opium to get out, which is subsequently collected. Alkaloids in the raw opium differ depending on the cultivation conditions [90].

Different synthetic routes exist, using liquid-liquid extractions, from the raw opium to the resulting heroin hydrochloride [90, 100, 141, 149, 157]. The extraction of morphine from the raw opium is firstly performed, which is then processed into heroin base by chemical process using acetic anhydride [110]. Heroin hydrochloride is finally obtained by dissolving the base form into acetone then precipitating the substrate with hydrochloric acid.

Heroin is available in four different forms (also called ‘grades’):

- Grades 1 and 2 are unprocessed heroin, rarely encountered in Australia [110];
- Grade 3 is heroin that can be smoked;
- Grade 4 is heroin that can be injected (or also heated to inhale its vapours), which is the purest form [110]. Procaine is often added to facilitate the injection as it relieves the pain locally [90].

Heroin is classed as a depressant [110], and an overdose may happen easily even in small amounts [158]. It is considered a high risk to the users health, especially as intravenous use of heroin may expose consumers to viruses including HIV and Hepatitis B and C [110].

In the past twenty years, efforts were invested and succeeded in reducing the cultivation and trade in the Golden triangle region as well as its sale for international demand [156]. Surveys conducted by the UNODC demonstrate that cultivations were reduced until 2006, but a resurgence has been observed in the past few years [159], correlated with a higher consumption demand on an international basis [156], and increasing seizures at international level (more than 13 tonnes in 2014) [125].

Thus, the resurgence of heroin use proves that “*heroin is not a problem unique to the older generation and that it still needs to be prioritized by the international community*” [125].

Two trafficking routes are mainly followed:

- The '**Balkan route**', supplying Western and Central Europe, from Afghanistan through Iran and Turkey;
- The '**Southern route**', through Pakistan and Iran to Africa, South Asia and Oceania by sea, which is increasing [125].

The heroin market in Australia changed considerably after 2000, as it became less accessible following law enforcement strategies [160]. It nevertheless remains an issue, as the number of seizures reported for the 2014-2015 period was the highest in the past decade (1914 seizures) [104]. Malaysia has become a hub for heroin coming from Afghanistan and Cambodia before being shipped to Australia [125].

The Enhanced National Intelligence Picture on Illicit Drugs (ENIPID) project was developed in Australia since 2010 to include state and territory seizures involving heroin, MA, MDMA (and more recently cocaine), in order to centralise the information from seizures occurring in different jurisdictions. According to ENIPID results (2010-2015), seized heroin predominately originates from South-East Asia [104].

2.1.2 Chemical profiles of cocaine and heroin

All compounds can be identified and quantified in the chemical profile of specimens [79]: their relative percentage of presence in the specimen can be established, as well as the overall purity of the specimen [83]. Based on detected compounds in the specimen, it is possible to establish the synthetic route and its geographic origin (area of cultivation).

Geographic origin of specimens can be differentiated, as climatic conditions (humidity, temperature, UV radiation, etc.), specific to each region (and more precisely each producing countries), influence the stable isotope ratios of plants and are retrieved in the final product [161, 162].

When a seizure is performed, a rapid screening test (colour tests are mostly employed [163]) is done directly on site to obtain an indication of the nature of the seized material. However, these tests are not substance-specific, as other compounds containing the same targeted functional group may also react [164], thus further analysis with analytical instruments is needed to confirm the identification and characterise the composition of the specimen.

A sampling procedure is applied on the seizure (see Chapter 1 for a detailed explanation), and, in Australia, specimens are sent for chemical analysis to the NMI to establish their respective chemical profiles. The procedure to analyse cocaine and heroin specimens employed at NMI is identical to the protocol established in the United States of America by the Drug Enforcement Administration's (DEA) Special Testing and Research Laboratory (STRL) [165]. Only parts of the procedure that are exploited in the database and that lead to data provided for this study are described hereafter⁹.

⁹ Due to the confidentiality of the method, the full procedure is not available in the literature. The following information regarding NMI procedure was obtained after discussion with NMI analysts.

Specimens are analysed at NMI with different techniques to obtain four distinct profiles which are:

1. Alkaloids profile:

Alkaloids are naturally present in the raw material (i.e. coca leaves for cocaine and poppy seeds' heads for heroin) and can be retrieved in the final profile.

For cocaine specimens, nine alkaloids plus the cocaine are targeted, using a Gas Chromatograph coupled to a Mass Spectrometer (GC-MS) in Single Ion Monitoring mode (SIM)¹⁰. For heroin specimens, eight alkaloids plus heroin are targeted by Capillary Electrophoresis with Diode Array Detector (CE-DAD).

Resulting values are recorded for each alkaloid with cocaine and heroin in their base form, and their respective equivalent in hydrochloric form is also calculated.

2. a. Truxillines profile in cocaine specimens:

Eleven types of truxillines exist in the coca leaf [165]. They are considered as alkaloids but require a longer preparation procedure than the other ones for analytical detection. Therefore, this analysis is done separately, with a GC-MS in SIM mode [81]. The result is recorded as a percentage value corresponding to the sum of all detected truxillines.

2. b. Acidic and neutral by-products in heroin specimens:

Thirty-three impurities and by-products are targeted and analysed with GC-MS. Resulting values are normalised to the total sum of impurities.

¹⁰ Detailed explanation of each analytical step for the preparation of analysed specimen is provided in [165].

3. Occluded solvents profile:

Solvents are analysed with a Head Space GC coupled to a Flame Ionisation Detector (HS-GC-FID) for cocaine specimens [166], and HS-GC-MS for heroin specimens. Their presence/absence is recorded for heroin specimens. Regarding cocaine specimens, their value is recorded as a relative value to the total of solvents detected in each specimen (Table 2), the total of solvents being therefore 100 % (± 5 %) for each specimen.

Table 2: Solvents values' calculations at NMI

Specimen	Solvent 1 value	...	Solvent 18 value	Total 18 solvents values (100 %)
x	$\frac{v_1}{\sum (v_1 + \dots + v_{18})}$...	$\frac{v_{18}}{\sum (v_1 + \dots + v_{18})}$	$\sum (v_1 + \dots + v_{18})$

4. Diluents and adulterants profile:

Adulterants and diluents are added after the production of pure cocaine and heroin. They might be added at different levels of the distribution network or all at the same level. According to previous studies, adulterants tend to be added at higher levels of the distribution chain [167]. Cutting agents can also be added once imported on the territory where the product is sold to optimise the shipping cost [38].

This analysis is performed with an Evaporative Light Scattering Detector used in conjunction with high-performance liquid chromatography (HPLC-ELSD) for cocaine specimens and CE-DAD for heroin specimens. Each value is independently recorded.

These analyses give the full chemical profile with the targeted impurities for cocaine [84, 85, 101, 168, 169] and heroin [90, 157, 167, 170] specimens as summarised in Tables 3 & 4.

Table 3: Impurities detected in cocaine specimens

Alkaloids	Solvents ¹¹	Adulterants	Diluents
Trans-Cinnamic acid	Acetone ^v	Aspirin	Fructose
Ecgonine	Acetonitrile	Benzocaine	Glucose
Benzoyllecgonine	Benzene ^v	Caffeine	Inositol
N-Formyl cocaine	Chloroform ^v	Dextromorphan	Lactose
Ecgonine methyl ester	Ethyl acetate ^v	Paracetamol	Maltose
Norcocaine	Hexane ^v	Phenacetin	Mannitol
Cis-cinnamoylcocaine	Isobutyl acetate	Phenobarbitol	Sorbitol
Trans-cinnamoylcocaine	Mesitylene	Theophylline	Sucrose
Truxillines	Methyl acetate ^v	Creatine	
Trimetoxycocaine	Methyl ethyl ketone ^v (MEK)	Creatinine	
Tropacocaine	Methyl isobutyl ketone (MIBK)	Diltiazem	
	Methylene chloride ^v	Hydroxyzine	
	N-propyl acetate ^v	Ketamine	
	Toluene	Levamisole	
	MP-Xylene	Dexamisole	

¹¹ Solvents with mention ^v have been identified as highly volatile in cocaine specimens [75].

O-Xylene	Lignocaine
Methylisobutenylketone	Nicotinamide
Metylbenzoate	Procaine
	Quinine
	Strychnine
	Thebaine

Table 4: Impurities detected in heroin specimens

Alkaloids	Solvents	Adulterants	Diluents
Acetylcodeine	Acetone	Caffeine	Fructose
Codeine	Acetonitrile	Dextromorphan	Glucose
Morphine	Benzene	Lignocaine	Inositol
Noscapine	Chloroform	Nicotinamide	Lactose
Papaverine	Cyclohexane	Paracetamol	Mannitol
3-Monoacetylmorphine (3-MAM)	Ethyl acetate	Phenobarbital	Sorbitol
6-Monoacetylmorphine (6-MAM)	Ethyl ether		Sucrose
	Hexane		
	Isobutyl acetate		
	Methyl acetate		
	Methylene chloride		
	MEK		
	MIBK		
	Mesitylene		
	MP-Xylene		
	N-Propylacetate		
	O-Xylene		
	Toluene		

If all compounds are detected and are part of the targeted list (Tables 3 & 4), the sum of values of:

Illicit substance + alkaloids + occluded solvents + adulterants & diluents

Should ideally be 100 %.

This total is relative to the total mass of the specimen, and therefore values of each compound correspond to their percentage in mass compared to the total mass of the specimen, as it is illustrated in Figure 10 which is a typical simplified representation:

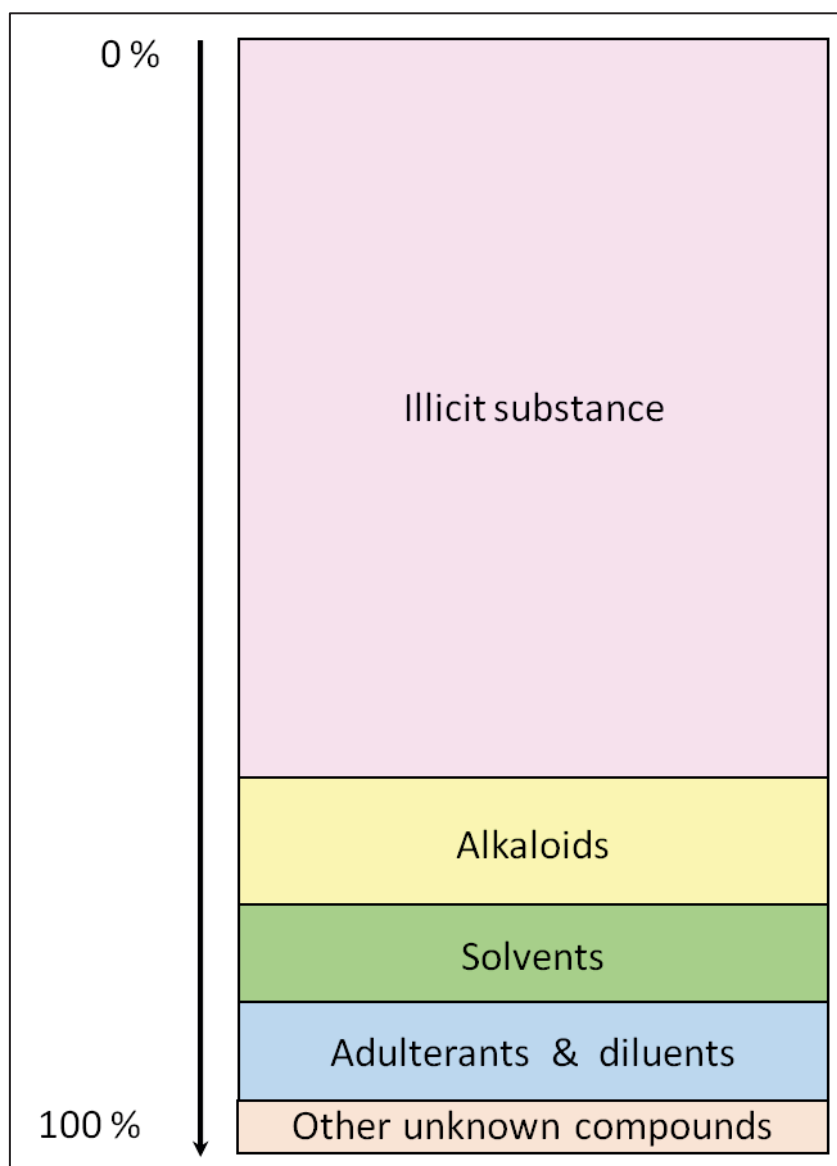


Figure 10: Chemical composition - presence in total mass of the specimen

This illustration is a typical representation of the composition of a specimen, but relative percentages should not be precisely taken into account. Indeed, the presence in mass of alkaloids can vary, as well as solvents. The illicit substance, cocaine or heroin, represents logically the majority of the composition of the specimen, and all other compounds represent only a very small proportion in the total mass of the specimen.

The entire specimen's composition may ideally be identified. However, some compounds might not be part of the targeted list (i.e. Table 3 for cocaine and Table 4 for heroin). They might not be detected, or even if detected they are not taken into account in the final composition of the specimen. Due to uncertainty of the analytical method, a standard error of $\pm 5\%$ has to be taken into account for each individual value.

Therefore, the full chemical profile of one specimen has to be interpreted knowing and taking into account these properties.

2.1.3 Scope of this chapter

Purity of specimens, identified cutting agents and their prevalence in time and space were investigated for both cocaine and heroin Australian border seizures.

The objective was to depict, if any, trends that would bring valuable information to the AFP for operational and strategic purposes.

2.2 Material and method

A subset of the PROMIS database (see Chapter 1), extracted in Excel spreadsheets was provided by AFP. It contained data from cocaine and heroin seizures performed at borders between 2008 and 2013. Three different files were provided for cocaine and heroin:

1. The **Seizures file** contained circumstantial information regarding the seizures, such as the location where the seizure was performed, its total weight, type of camouflage employed, the method of transport and the geographic origin of the seizures. Therefore it was the most 'general' file;
2. The **Subgroups file** contained all information regarding physical characteristics of seizures, such as the shape, size and colour, relative to each subgroup and for each seizure. It also contained the number of items for each subgroup as well as the number of specimens sent to NMI for chemical analysis. As the study was focused on chemical characteristics (see Chapter 1), this file was not fully exploited;
3. The **Specimens file** contained all information regarding chemical analyses performed at NMI for each specimen. This file was the one the most exploited for the study as it contained detailed chemical information of cocaine and heroin specimens (from each subgroup and per seizure).

Data was exploited using the software Microsoft® Excel (Microsoft Corporation, United States) and R® (The R Foundation for Statistical Computing, version 2.14.1, Austria). General trends were produced and interpreted in an intelligence perspective.

2.3 Results

2.3.1 Seizures distribution

First of all, cocaine and heroin seizures (respectively 521 and 470) over the six years period were predominantly 'mono-drug' seizures (i.e. only containing cocaine or heroin for more than 95 % of seizures). This information is important regarding the trafficking routes and distribution markets, because cocaine and heroin seem to be mainly imported into Australia alone rather than combined with other drugs.

Secondly, the distribution of seizures in time and space can be observed in Figure 11 and Figure 12. Locations corresponded to cities from different States where international customs were implemented, at airports or in harbours. Results are given in relative percentage each year for a better visualisation and comparison from one year to another.

The amount of seizures performed each year and in each State fluctuated for both drugs as well as the total weight seized each year.

Sydney had the highest number of seizures as well as the weight seized per year across the six years period. This could be explained by the fact that most flights and cargos arrived in Australia via Sydney, and these were the most frequent methods of transport for seizures performed at customs. Brisbane and Melbourne were also substantial in term of weight for both drugs. Conversely, Adelaide, Canberra (heroin seizures were never performed in Canberra during the six years period), Darwin and Perth were almost non-existent, with almost no seizures and a weight very small compared to others cities.

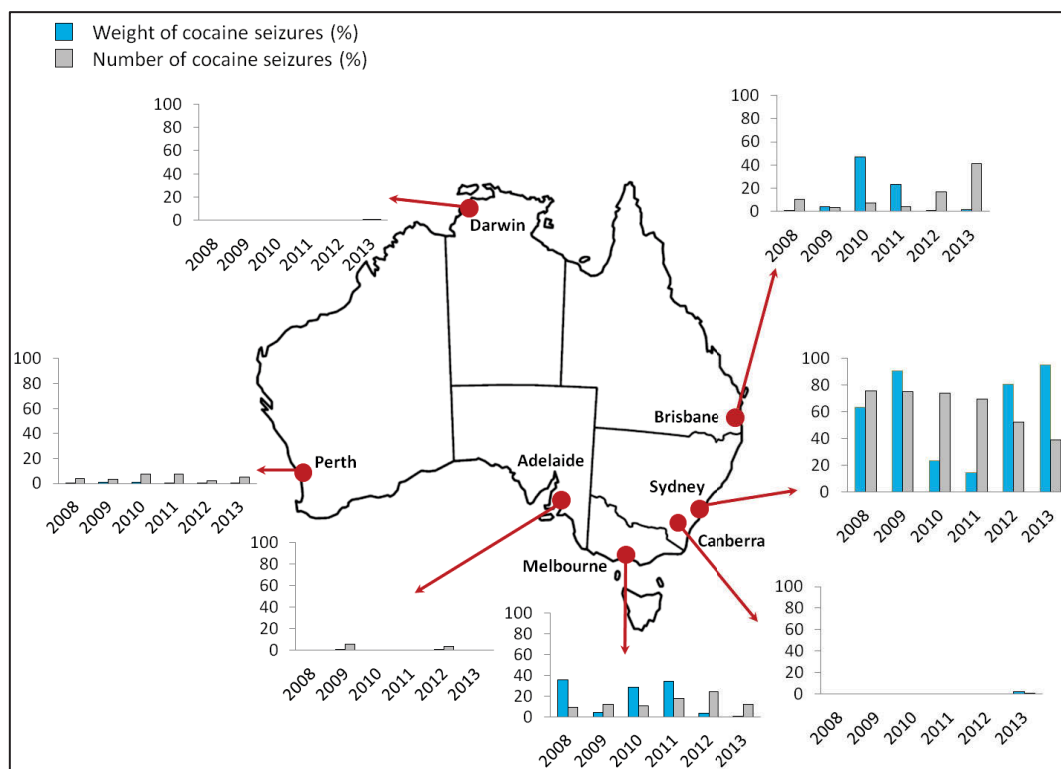


Figure 11: Location of cocaine seizures per year in function of percentages of weights and seizures

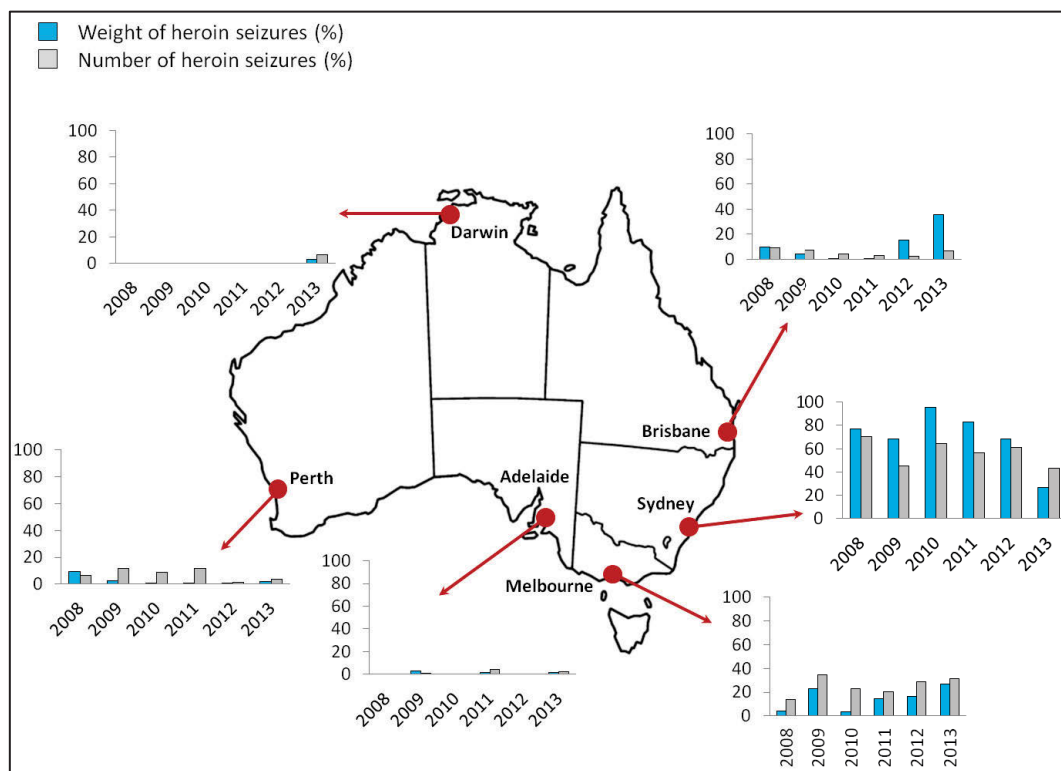


Figure 12: Location of heroin seizures per year in function of percentages of weights and seizures

2.3.2 Purity

Figure 13 is a boxplot representation of the distribution of purity values of cocaine and heroin seizures each year (2008-2013). The more spread the boxplot is, the more variations occurred with purity values.

The distribution of purity was very large each year for both drugs. Variations were noticeable from one year to another, especially when looking at quartiles. Nevertheless, the overall variation each year increased from 2008 to 2013 for both drugs. Indeed, the 2013 values were from close to 0 % to more than 85 %, whereas these variations were lower in previous years, especially when compared to 2008 values. This finding indicates that seizures performed in 2013 were highly variable compared to the ones performed in 2008 (when not taking into account outliers).

Looking more specifically at the median per year, it varied from 68.50 % to 58.65 % between 2008 and 2013 for cocaine, with a relative constant decrease: in less than six years, the purity of cocaine was lowered by almost 10 %.

For heroin, the median value was relatively stable over the six years, around 60 % \pm 2 %. In 2011, a drop was observed with a median purity of 49 %, which corresponded to the first year where heroin seizures were predominantly coming from South West Asia, a region of production characterised with a lower purity of heroin product (communication from AFP).

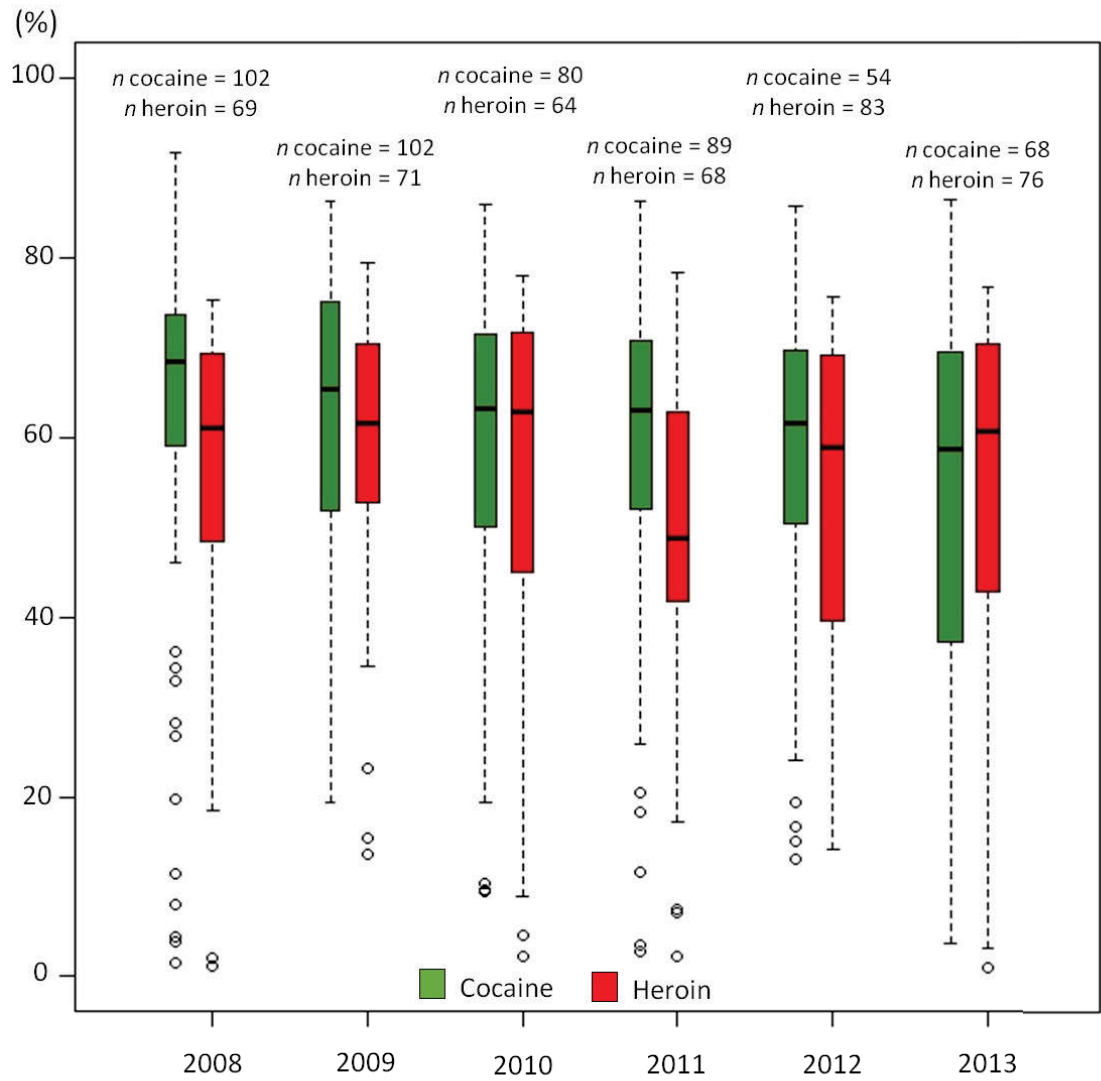


Figure 13: Purity of cocaine and heroin over six years

2.3.3 Solvents

The compounds detected in cocaine and heroin specimens can be found in Appendix 1. The presence of solvents in specimens is directly related to the manufacturing the drug: if a solvent is detected, it suggests its usage in the manufacture the specimens [171].

The relative proportion of each solvent in cocaine specimens was quite stable for the six years period, as illustrated in Figure 14. Nevertheless, a slight increase was observed from 2011 for hexane, methyl acetate, toluene, mp-xylene and o-xylene; methylethyketone was variable from one year to another. Two groups were observed depending on their presence in more than 50 % (i.e. benzene, ethyl acetate, hexane, isobutyl acetate, methyl ethyl ketone, mp-xylene, n-propyl acetate, o-xylene and toluene) or less than 50 % of specimens (i.e. acetone, acetonitrile, chloroform, mesitylene, methyl acetate, methylbenzoate, methylene chloride, methyl isobutyl ketone and methylisobutenylketone). Overall, this relatively stable trend could indicate that same manufacturing routes were employed to produce cocaine using the same solvents in the same proportions during the period 2008-2013.

Fewer solvents were retrieved in heroin specimens compared to cocaine specimens as displayed in Figure 15. One distinct trend was the increase of ethyl acetate and ethyl ether during the six years, contrary to other solvents whose presence remained stable over time. These two solvents are employed during the conversion process to obtain the hydrochloric form from the base form [172]. On the contrary, methyl acetate, which was present in 20 % of specimens until 2010, nearly disappeared in recent years. Toluene remained stable over time, around 20 % of presence in specimens. All other solvents were almost non-existent in the heroin specimens.

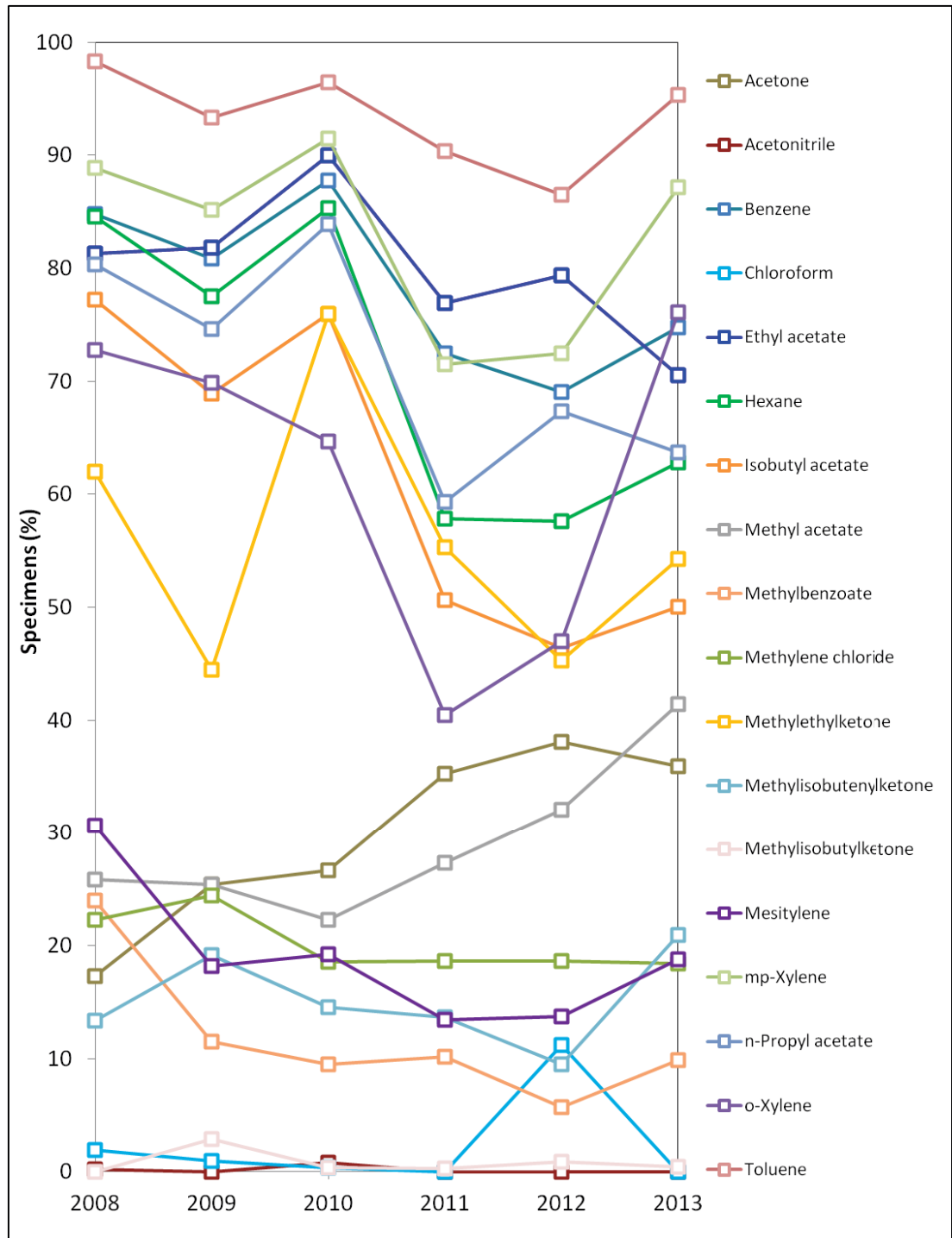


Figure 14: Percentage of solvents present in cocaine specimens over six years

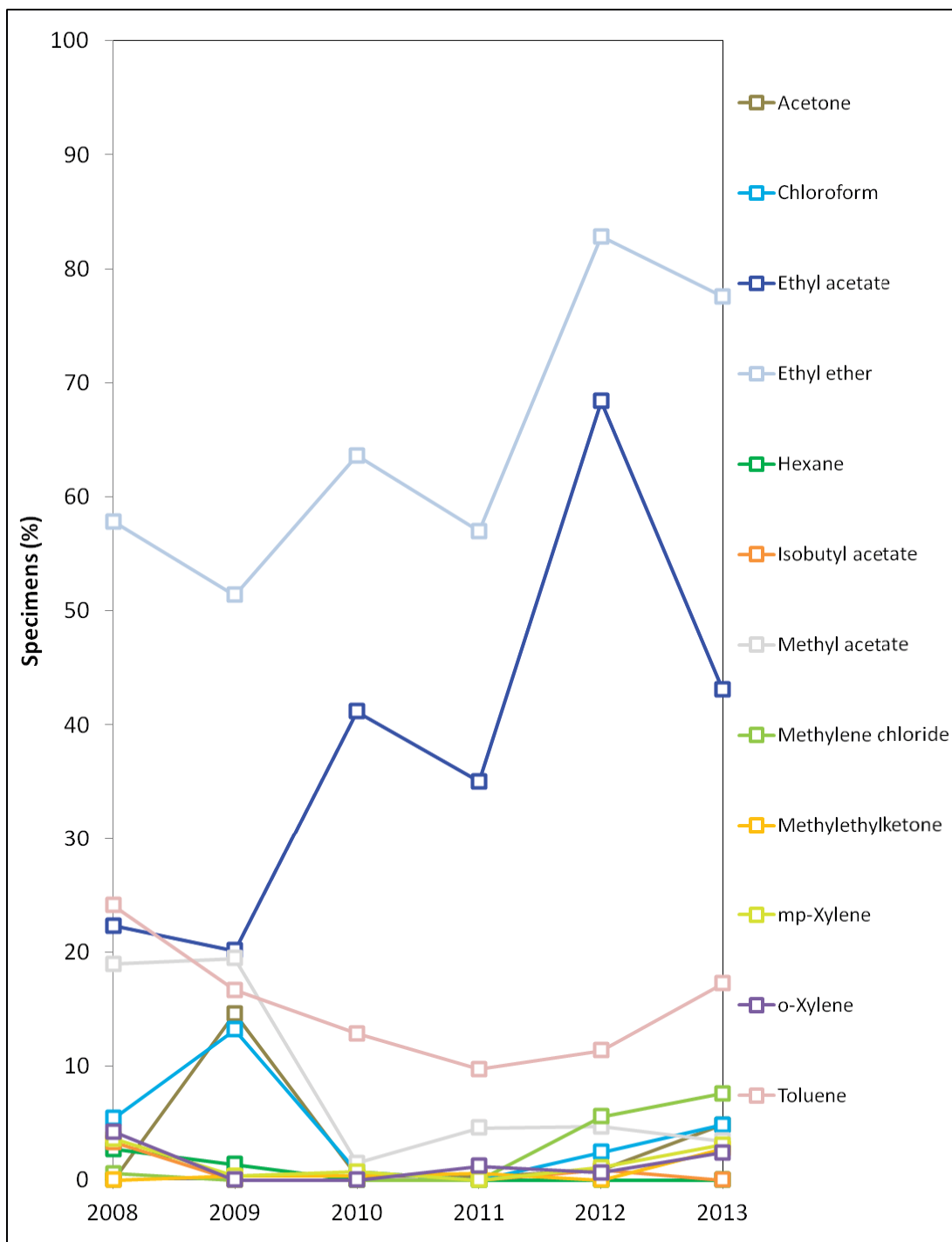


Figure 15: Percentage of solvents present in heroin specimens over six years

2.3.4 Adulterants

The presence of adulterants over time in cocaine specimens is displayed in Figure 16. Levamisole and dexamisole are combined into one variable, as these two enantiomers form the compound Phenyltetrahydroimidazothiazole (PTHIT), commercially used as veterinary medicine (personal AFP communication). PTHIT largely dominated, being present in up to 75 % of specimens in 2010. It nevertheless decreased from 2010, and it could come from the fact that less PTHIT was added to cocaine product in recent years. This assumption cannot be verified because dexamisole was not monitored before 2010 in the AFP database, and analytical procedures have changed: before 2010, levamisole was detected but not separated from its enantiomer. This change may affect values from more recent years where both enantiomers were taken into account in the adulterants' analysis of cocaine specimens.

Fewer adulterants were detected in heroin specimens compared to cocaine specimens, as displayed in Figure 17. Furthermore, only three adulterants were actually detected in more than 10 % of specimens: caffeine (which dominated with 50 % of presence in average), dextromorphan and paracetamol.

For the six years period, looking at the total number of adulterants detected per specimen, half of heroin specimens from the dataset did not contain any adulterant (see Appendix 2), whereas cocaine specimens contained at least one adulterant for at least half of them. Combining this information with Figure 16, this one adulterant would be PTHIT.

The trend changed over time for heroin specimens, as they contained two or more adulterants per specimen in recent years, which may be explained with the increase presence of paracetamol in addition to caffeine, as seen in Figure 17.

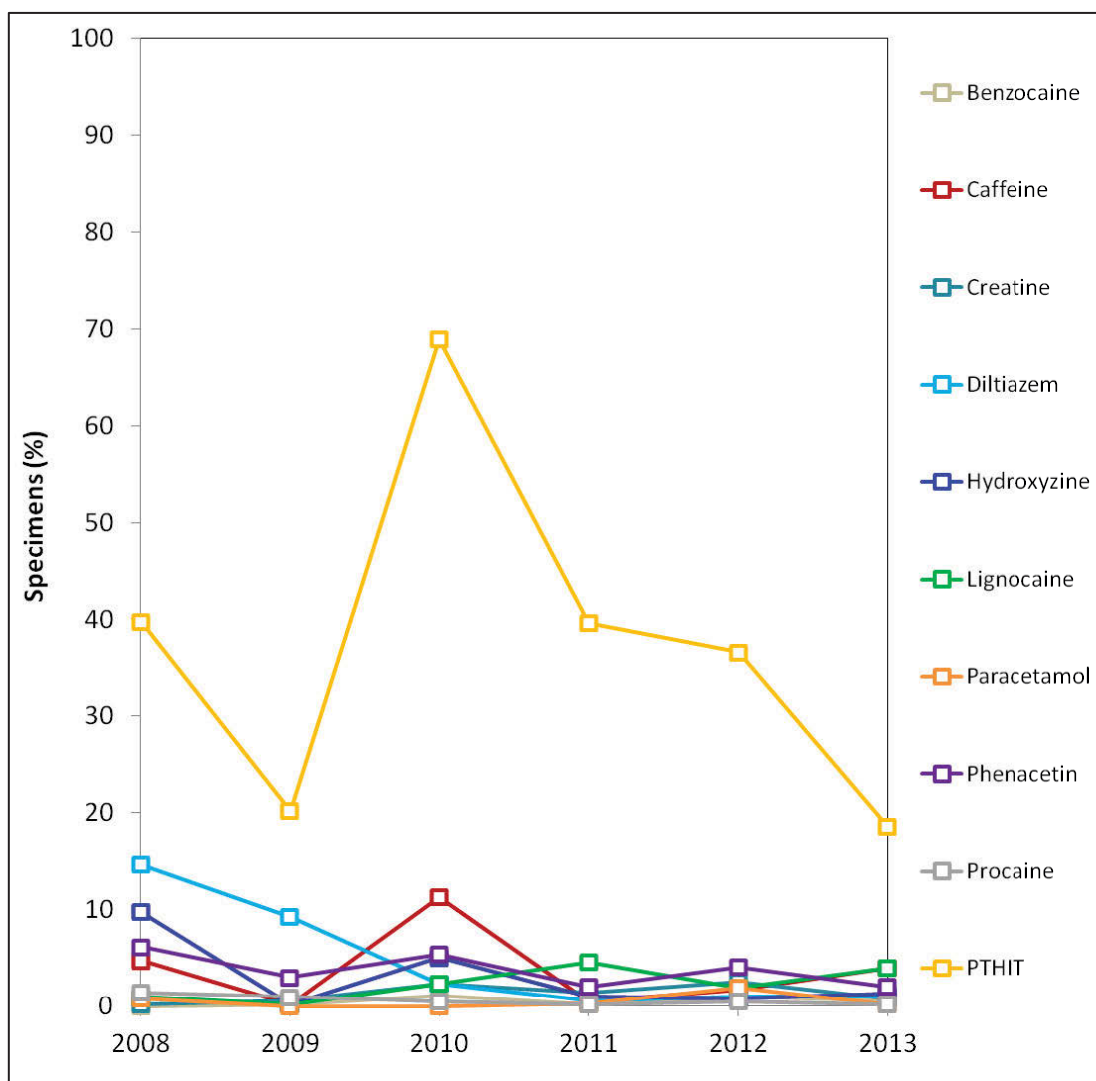


Figure 16: Percentage of adulterants present in cocaine specimens over six years

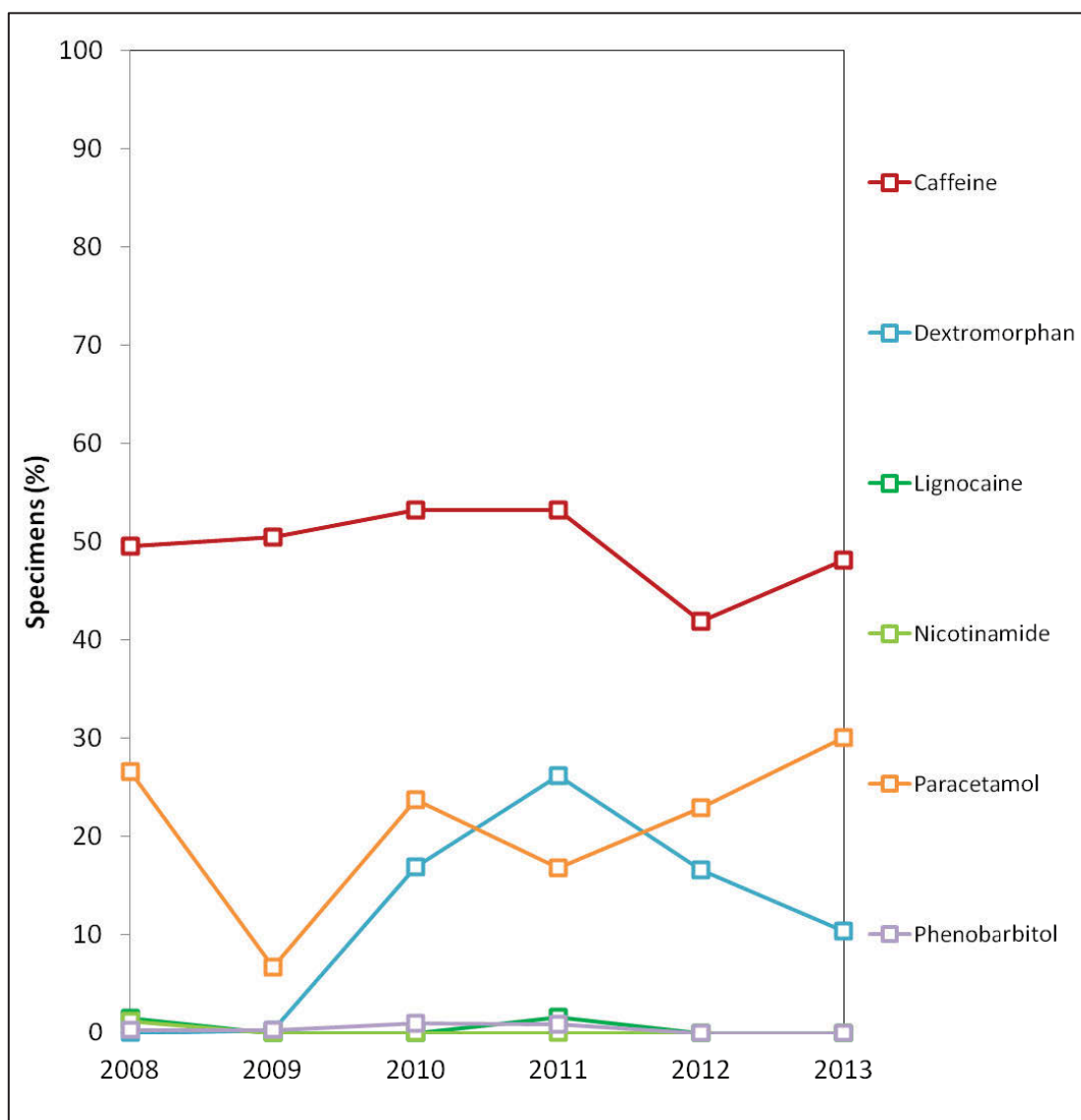


Figure 17: Percentage of adulterants present in heroin specimens over six years

2.3.5 Diluents

The presence of diluents over time (2008-2013) is presented in Figure 18, grouping cocaine and heroin specimens as the same compounds were detected. All diluents fluctuated during the six years and no general trend was observed for both cocaine and heroin specimens. Even if an increase from 3 % in 2012 to 11 % in 2013 was noticeable for mannitol and inositol in cocaine specimens, they were present in less than 10 % of all specimens, so sugars were rarely detected in cocaine or heroin seizures performed at customs.

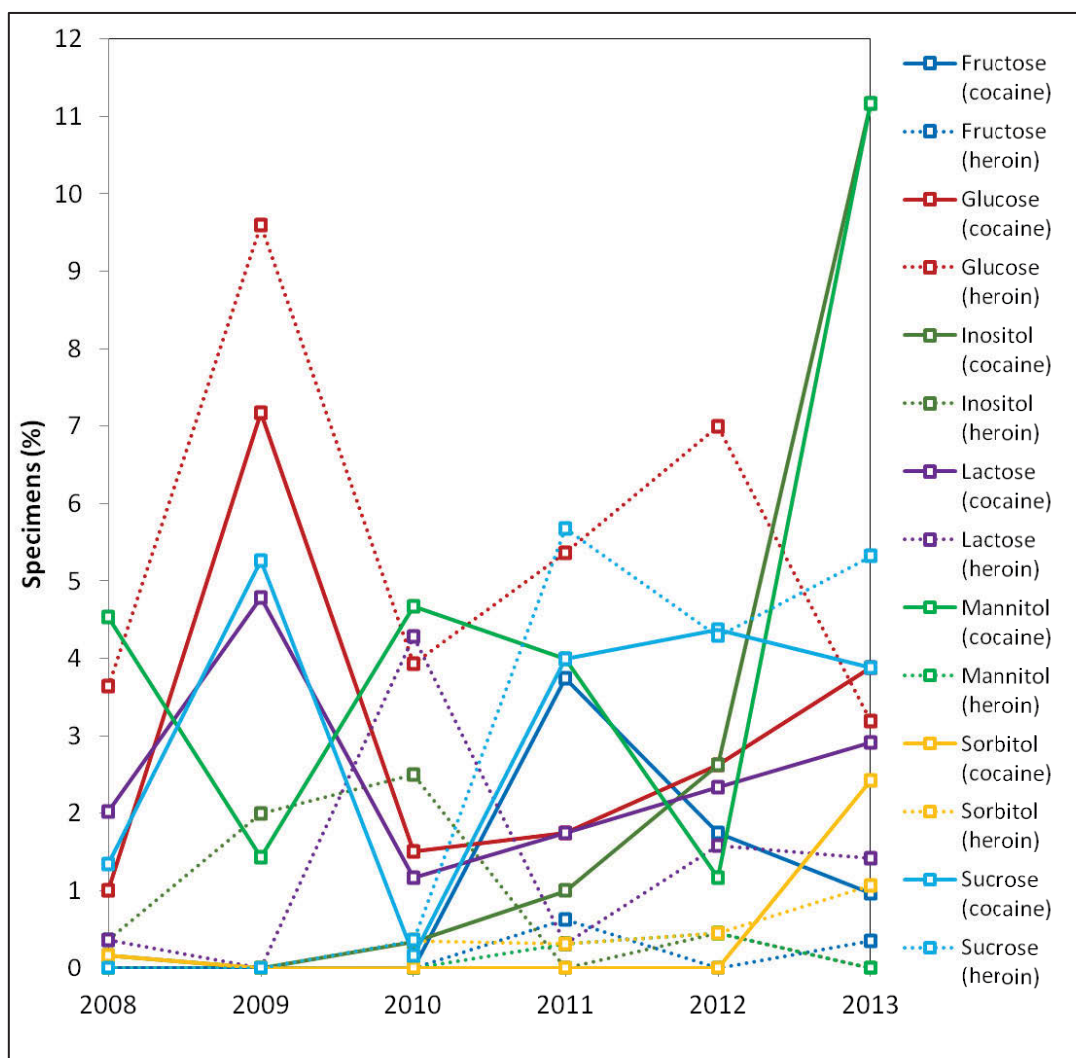


Figure 18: Percentage of diluents present in cocaine and heroin specimens over six years

2.4 Discussion

Cocaine and heroin border seizures' chemical profiles contained valuable information. Trends identified regarding the purity of specimens, cutting agents and their prevalence over six years (2008-2013) can help to get a better understanding of cocaine and heroin trafficking and more specifically provide recommendations to the AFP. The essence and implication of the results presented in the previous section are exposed hereafter.

2.4.1 Purity

As seen in Figure 13, the purity of cocaine and heroin fluctuated over time, and a wide range of purity was observed each year and for each drug. It implies that border seizures (cocaine and heroin) may either be products with high purity (up to 85 %, see Figure 13) or very low purity, thus containing in majority cutting agents.

Studies have investigated the purity of cocaine and heroin, notably in Switzerland regarding the Swiss and European cocaine and heroin markets [86, 88]. The purity of cocaine and heroin specimens seized between 2006 and 2014 in Switzerland was analysed and the variation was very wide each year (from around 0 % to more than 80 % for cocaine and from around 0 % to more than 50 % for heroin) [86]. This finding is similar to what is displayed in Figure 13. Moreover, a small decrease in purity was also observed over nine years for cocaine specimens (from 40% to about 30% between 2007 and 2011), even if the fluctuations (in their study) were smaller than in Figure 13.

One assumption behind the observed decrease is that specimens were more heavily cut in 2013 than in 2008. It was reported that a sudden drop in purity could be associated with a

decrease in the availability of the drug on the market, so traffickers are more inclined to cut the product to maintain a constant volume of sold product [86, 173]. This lower purity may thus be explained by the fact that products may be more heavily cut to preserve stocks.

Regarding the median purity of the dataset, a drop of more than 10 % was observed for cocaine, down to 58.5 % in 2013. This median purity, even if decreasing over time, was still higher than what was reported in other studies around the world. They recorded that the median purity never exceeded 40 % for cocaine, which was almost 20 % less pure than seizures performed in Australia [88]. Lower purity of cocaine seizures was also reported in the European Drug Report 2017 [174] (i.e. median between 36 % and 51 %), although a decrease was not observed over the past six years contrary to Figure 13.

Regarding heroin, the median purity was around 10 % [175], which was considerably low compared to the median around 60 % observed in Figure 13. German data also contained a drop of heroin purity between 2010 and 2011, similarly to what was displayed in Figure 13, from 25 % to 11 % purity [173]. Trafficking networks are surely different to import heroin to Germany and to Australia, so the similarity worth being noted, as it may imply that a shortage of heroin occurred internationally between 2010 and 2011.

However, comparisons have to be considered in context, as the studies mentioned above comprised seizures performed at customs and street seizures, which were already imported and potentially affiliated to a lower level of distribution. One hypothesis for the difference in purities observed could be that cocaine and heroin imported into Switzerland and other European countries where studies were conducted went through additional levels of distribution, and cutting agents would be added before being imported in the countries thus lowering the purity of products, contrary to what was observed in Australia.

Differences in median purity of specimens seized in Australia and Europe could be explained by the different routes and therefore trafficking networks involved in the importation of the drugs to Switzerland and to Australia [125]. Indeed, heroin seized in Europe mainly comes through the Balkan route while heroin seized in Australia mainly comes from production in South-East Asia (Myanmar, Laos) [80, 154]. The importation of the product may involve multiple stops with addition of cutting agents, thus decreasing the purity.

To finish, a study conducted in France observed the purity of cocaine seizures over fourteen years (1995-2009), and distinct results were obtained for custom seizures (average purity 60-80 %) compared to street seizures (average purity 30 %) [176].

A similar study as the one presented in this chapter should be conducted with street seizures. It would then be possible to perform a similar study to what was conducted in France and in Switzerland by comparing purity values obtained at customs with Australian street seizures.

2.4.2 Solvents

Two groups of solvents were obtained with the provided datasets based on their presence in more or less than 50 % in cocaine specimens. Only half of targeted solvents were present in more than 50 % of cocaine specimens: those solvents could help in establishing relations between specimens for strategic purpose as comparing the majority of specimens within the database. Conversely, solvents present in less than 50 % of all specimens cannot be employed for comparison purpose, because more than half of specimens cannot be compared based on those compounds.

In any case, results have to be interpreted with caution as solvents are highly volatile. Their evaporation depends on various parameters, which might affect analytical results: if a specimen is aged, solvents are less likely to be retrieved in the same proportions than a 'new' specimen due to volatile properties of solvents [177]. Moreover, if the storage conditions vary, in term of humidity or temperature, it may also affect specimens' properties, and resulting solvents values may be affected [177]. As a result, the relative abundance of each solvent cannot be used as such when dealing with solvents values for comparison purpose [145].

Some cocaine and heroin specimens from the datasets did not contain solvent values in their chemical profiles. As solvents are required to manufacture drugs, it may seem surprising to retrieve specimens without any solvents residues¹². The study of Morello et al. suggested non polar solvents are less susceptible to be trapped in the crystals of formed cocaine hydrochloride [147], so they are less likely to be detected. This statement may be extended to heroin hydrochloride as employing the same conversion process (based on liquid – liquid extractions and subsequent miscibility between solvent and targeted compound). These non polar solvents correspond to hexane, benzene, toluene, mp-xylene, o-xylene and chloroform

¹² The study focused only on specimens where solvents' analyses were conducted.

in cocaine specimens, and to ethyl ether being present in majority of heroin specimens. As chloroform is part of solvents present in less than 50 % of cocaine specimens, it may be due to its polarity. However, it is not possible to verify this assumption, as none of the parameters of storage of the product before its seizure are known.

Other explanations as to why solvents were not detected in cocaine and heroin specimens can be provided as further interpretation:

- These specimens were stored after being seized for a certain period of time in non-optimal conditions before analysis (high levels of humidity and heat), leading to evaporation of solvents' residues;
- These specimens were coming from an old batch of cocaine or heroin, thus all solvents' residues had already evaporated;
- Analytical method selected at NMI (i.e. GC-FID) did not retrieve any solvent.

Furthermore, the rate of precipitation at production site, or the temperature during the conversion part at manufacturing site and the mixture ratio between organic and inorganic solvents employed during the manufacturing process, can all affect the retrieval of solvents [147]. During the manufacturing process, the organic solvent is usually reused indefinitely, by just adding some more 'fresh solvent' from time to time [140, 166, 178]. The process involves dilution of the base product in solvents considered as **A**, then the miscibility of concentrated hydrochloric acid is obtained by adding solvents considered as **B**, and to finish the hydrochloride drug is isolated with a mixture of solvents A and B. Examples of solvents A include toluene, ethyl acetate, n-propyl acetate, acetone, MEK, hexane, methylene chloride [147, 166]. Examples of solvents B include MEK, MIBK, acetone [147, 166].

Out of the seven examples of solvents cited in the literature as employed as solvent A [166], five of them were part of solvents present in more than 50 % in all cocaine specimens (i.e. toluene, ethyl acetate, n-propyl acetate, MEK and hexane). It could be hypothesised that solvents employed as solvent A during the manufacturing process are more likely to be retrieved in the final composition of cocaine specimens. This can be further illustrated with ethyl acetate being the solvent the most retrieved in heroin specimens. Nevertheless, it should be interpreted with caution, as a solvent can be employed as solvent A or solvent B. In

the targeted solvents detected in cocaine specimens, MEK was in the list of solvent A but could also be employed as solvent B [166].

In any case, few studies investigated the use of solvents from cocaine specimens [147, 166, 177], and with heroin no study was performed. As a result, findings from this study cannot be compared to other countries and for intelligence purpose.

To finish, if purchases of solvents were monitored, a purchase in bulk of solvents determined as being predominantly used for manufacturing cocaine or heroin could potentially lead to clandestine laboratories. However, cocaine and heroin being manufactured outside of Australia, the responsibility goes primarily to law enforcement agencies in countries where semi-synthetic drugs are produced, as well as neighbour countries to control the import / export of such solvents.

2.4.3 Adulterants

The presence of adulterants as presented in Figure 16 and Figure 17 revealed that cocaine and heroin were imported into Australia already cut (in majority PTHIT for cocaine specimens, caffeine and paracetamol for heroin specimens).

2.4.3.1 Cocaine specimens

Since the 1980's, an increase of adulterants in cocaine was observed internationally [85]. In Swiss studies [86, 97], phenacetin was found to be the most present adulterant in Switzerland' specimens (80 %), while it was absent in more than 90 % of specimens in Figure 16. Levamisole was the second highest (65 %) although its presence increased each year. Lidocaine was present in 47 % of specimens, whereas it was totally absent from the data. Caffeine was observed in 39 % of specimens whereas it was not as frequent in Australian border seizures (less than 10 %). To finish, diltiazem was commonly present until 2009 and almost disappeared in recent years, similarly to our data.

Another recent study conducted in Brazil demonstrated that phenacetin was the principal adulterant found in cocaine specimens, followed by benzocaine, lidocaine and caffeine [179]. As lidocaine seems to be frequently detected in cocaine specimens in other countries, it might be of interest to include this compound in the list of targeted adulterants at NMI, as it is not considered at the moment in the analytical procedure. However, seizures in Brazil and Switzerland were performed within the country and not only at customs, so the absence of phenacetin in the data provided could be explained due to different levels of the network. It may be added once imported in the country which is why it was not detected in border seizures. It seems therefore essential to compare these results with results from cocaine seizures at a consumption level, once distributed within the country. A study on Australian street seizures is essential especially for the investigation of adulterants.

Cocaine seized in the United States (US) contained levamisole as the main adulterant and its frequency consistently increased [180]. Several other studies, notably in the US [146], in

Austria [181], and in England [182], established the same outcome: the level of levamisole has increased in the previous years. As a result, monitoring levamisole seems essential. It is not a restricted compound and if a high amount is ordered, it could potentially be used as a cutting agent for cocaine, and tracking down the buyer could lead to seizures of cocaine.

As seen in the different studies, adulterants are different from one country to another, making the cocaine market specific to its region and more difficult to compare at international scale. While Australian cocaine specimens contained in majority one adulterant and a maximum of four in less than 10 % of specimens (see Appendix 1), Swiss specimens contained more adulterants, as the average number of adulterants per specimens in Switzerland was three [86]. Nevertheless this outcome should be interpreted once again with caution as studies were based on border and street seizures contrary to this dataset containing only border seizures.

2.4.3.2 Heroin

Regarding heroin specimens, only three adulterants were detected in the majority of specimens (see Figure 17). The same conclusion was obtained with Swiss seizures, even though it included street seizures [97], similarly to French street seizures [170]. This finding suggests that fewer adulterants are added to heroin compared to cocaine even at consumption level. Moreover, 75 % of Swiss heroin seizures and 90 % of overall European seizures contained caffeine as the main adulterant [167], which was also the most present adulterant observed in Figure 17. Therefore, caffeine seems to be the number one adulterant used at international scale, as also highlighted with Malaysian street seizures' profiles [183]. Monitoring its use, especially for undetermined usage of large quantities may be of interest and may allow the possibility of detecting clandestine laboratories.

However, Swiss specimens contained paracetamol in similar proportions as caffeine, whereas it was less abundant in Australian specimens (less than 30 % presence). The mixture paracetamol / caffeine was very common in heroin seizures in Europe, and it has been suggested that the mixture may already be prepared as such, then added during the manufacture of heroin base or during the distribution process [167, 175]. It may be worth

investigating this possibility in Australia if both adulterants are increasingly present in specimens in more recent years.

A study in Luxembourg investigated adulterants in heroin specimens (border and street seizures) over six years and provided similar results to Figure 17, with an average caffeine presence of 50 % in heroin specimens and paracetamol approximately 30 % of specimens [184]. Other adulterants were almost never retrieved, even in street specimens. Two assumptions can be advanced:

1. Heroin seems to have few distribution levels before being consumed, as custom seizures have similar adulteration profiles than street seizures;
2. The same adulterants might be used at different levels in the distribution chain.

To verify both assumptions, a quantification of adulterants should be performed and a comparison undertaken with the amount present in custom seizures and in street seizures.

As already mentioned with cocaine, an investigation and quantification of chemical composition of street seizures is crucial to get a better overview of the drug market and being able to refine those assumptions.

2.4.4 Diluents

The low presence of diluents in cocaine and heroin specimens as presented in Figure 18 (present in less than 10 % of specimens) was also observed in other countries. Studies found few diluents especially in heroin specimens, notably in Switzerland [88, 97] and USA [185], although they contained border as well as street seizures.

One possible explanation could be that diluents may be added to cocaine and heroin products once imported in the country. This hypothesis would lead to strategic and operational ways of tracking down high amounts of diluents bought in Australia, as it could potentially lead to clandestine laboratories or retailers at different levels of the trafficking network. However, as diluents are not restricted compounds, buying high amounts of sugar can be legitimatised for legal use, especially in food industry similarly to what was exposed with caffeine in the previous section; the implementation of operational surveillance of diluents seems complex.

An increase of inositol and mannitol to more than 10 % in 2013 was observed in cocaine specimens (see Figure 18): a comparison of the results with recent years would be of interest to investigate if this increase continues after 2013.

In essence, analyses of sugars by NMI are not essential for strategic purpose in cocaine and heroin specimen, as the majority of the chemical information is coming from all other compounds but diluents. However, if diluents are being more frequently detected in cocaine and heroin specimens in recent years, they may become compounds of interest. Indeed, changes in diluents 'profiles of cocaine and heroin specimens can be associated with new additional network intermediates, or a change in the distribution chain (more diluents added before entering the country and fewer diluents added once already imported), or less pure product available therefore higher amount of compounds added to increase the mass of the sold product before entering the country [167].

An investigation of street seizures would be essential to confirm or refute those assumptions, similarly to adulterants.

2.4.5 Cutting agents and public health

Public health is of national and international concern. The concentration of diluents in cocaine or heroin specimens is usually small, and therefore these compounds are not toxic for the consumers themselves [85]. However, the constant use of adulterants is itself a concern for public health: they may have serious health implications that have to be carefully looked at [144, 180].

Initiatives have been undertaken, such as in Austria with the 'checkIt!' addiction prevention programme [181]. Consumers can have their product analysed, for instance during music festivals, in a free and anonymous environment, with a mobile laboratory. Information on the composition of the drug is obtained, which may increase awareness of consumers, notably regarding drug use, and possible dangerous added compounds retrieved in products [181].

More initiatives like this should be implemented, as it would allow prevention campaigns against health impacts of drug consumption, and more specifically cutting agents. Moreover, it would allow monitoring street products which are not seized. A comparison of their chemical profile to the ones seized by the police would allow getting a better idea of the consumer market, i.e. if street seizures are representative of the consumer market in term of chemical composition. A comparison of those consumed drugs (not seized) to border seizures would reinforce also the knowledge regarding chemical evolution in time of drugs, a change in composition depending on geographic region, etc.

2.5 Conclusions

Chemical composition of cocaine and heroin border seizures was investigated for a period of six years (2008-2013), and the main findings are:

- The purity over time was examined, and a wide range of purity was observed for both cocaine and heroin (from around 0 % to more than 80 % for the highest specimens). Moreover, a decrease for more than 10 % of the median purity was observed over time for cocaine, whereas the median purity of heroin remained stable.
- Residual solvents were detected in cocaine and heroin specimens and few fluctuations were observed over the six years, suggesting stable manufacturing process in time.
- The presence of cutting agents was investigated: PTHIT was the main adulterants found in cocaine specimens and caffeine and paracetamol were mainly present in heroin specimens. Conversely, less than 10 % of cocaine and heroin specimens contained diluents.

Based on the results presented in this chapter, it is possible to state that cocaine and heroin markets are quite dynamic as the purity and added compounds (mostly adulterants) fluctuate over time. Therefore, if the study is continued in recent years, it may highlight changes in the cocaine and heroin markets or trends over time, as mentioned by Broseus et al. [97].

Many studies were conducted especially in Europe to look at the composition of cocaine and heroin specimens as well as their purity. Those studies included both border and street seizures. No study has yet been conducted in Australia to investigate the chemical trends that could be obtained from street seizures, which is the main limitation of this research. For a proper comparison to border seizures and with European observations, the analysis of street seizures for intelligence purpose appears crucial.

The general statistical analysis as presented in this chapter is essential to get a better understanding of drug markets (in particular cocaine and heroin). This study demonstrated that the analysis of chemical information from drug seizures reinforces knowledge about their purity, manufacturing products as well as cutting agents.

The analysis of chemical profiles of cocaine and heroin specimens is further investigated in Chapter 3, to gain additional information about chemical compositions of cocaine and heroin border seizures for operational and strategic purposes.

Chapter 3:
***Multivariate analysis of chemical
profiles of cocaine and heroin
border seizures***

Chapter 3: Multivariate analysis of chemical profiles of cocaine and heroin border seizures

3.1 Introduction

As seen in Chapter 2, specimens of border seizures in Australia are routinely analysed at NMI following the DEA procedure and chemical results are stored in the PROMIS database. Nevertheless, no statistical model was implemented to establish automatic linkage between seizures (cocaine or heroin) based on their chemical profiles. The investigation of such an approach will be the focus of this chapter.

3.1.1 Development of a training model

Combined information from all chemical profiles stored in a database and investigated simultaneously can be used to identify links between seizures originating from a common source, as described in Chapter 1. The methodology to develop a model to perform automatic comparisons of the entire database is based on statistical methods [91].

Reference values have first to be selected to assess the robustness and validity of this model. Traditionally, statistical approaches in hard science use a training dataset on one side, comprised of reference values allowing to correctly building the model, and a validation dataset in a second stage, comprised of unknown values to be tested and classified accordingly to the built model [186].

However, in this research study, the dataset available is the one provided by AFP (see Chapter 2). No reference values exist, as the dataset is based on seizures, and there is no such thing as a 'reference seizure'. Thus, uncertainty exists regarding the dataset and the values it contains; the model to be created needs to use reference values from this dataset of unknown specimens as illustrated in Figure 19:

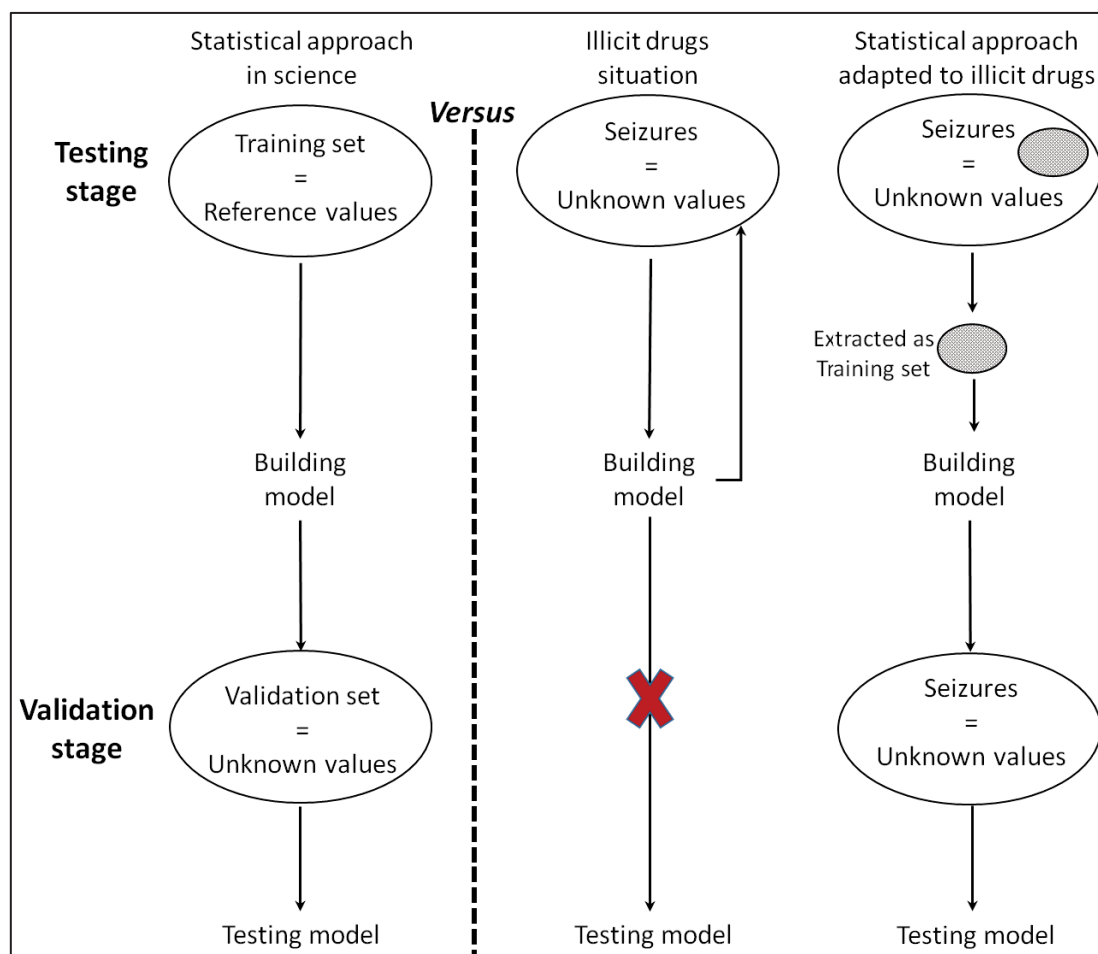


Figure 19: Traditional statistical approach applied to illicit drugs' data

In this context, a 'best scenario' - i.e. the training dataset - needs to be determined, to state if seizures with similar chemical profiles could be classified in the same category, that is to say linked or unlinked populations:

- The **linked population** corresponds to specimens that can be classified as linked due to similar chemical profiles. The variability between all chemical profiles in this population is denominated as the **intravariability** and should be as small as possible. In the same manner, the variability of specimens within one seizure is also defined as the intravariability [101];
- The **unlinked population** corresponds to specimens that cannot be linked based on their chemical profiles (i.e. distinct chemical profiles). This population of unlinked

seizures is denominated as the **intervariability** [101]. The variability within this population is usually larger than the intravariability.

Based on previous studies, it is assumed for this type of work that variations between specimens within one seizure (intravariability) should be smaller than variations between different seizures (intervariability) [187].

The intravariability is evaluated by measuring the similarity value of specimens coming from the same seizure [101]. It is then compared to the intervariability to determine if populations of linked and unlinked specimens could be discriminated [119].

3.1.2 Pre-treatments

The information from chemical profiles of specimens comes from multiple compounds at a time (chemical values obtained from analytical procedure, see Figure 10 in Chapter 2). The analysis of such profiles is called multivariate analysis, where a compound is considered as one variable. When performing a multivariate analysis, pre-treatments are mathematical functions applied to variables. They are employed to enhance the information of each value, reduce instrumental influence and minimise the effect of large differences in concentration of compounds present in the specimens, so that values can be comparable between different specimens [141, 188]. Numerous pre-treatments exist but only a few pre-treatments are tested as already widely described in the literature [90, 189]. **Normalisation** and **Square root** are recommended for cocaine specimens [101, 189], as well as for heroin specimens [188, 190, 191]. Normalisation consists in dividing each original value by the sum of all values of each specimen. Square root consists in applying a square root to each original value independently, in order to reduce the range between values to 10 instead of 100 [101]. The **linear transformation** consists in changing values of one isolated variable instead of applying the same statistic method to all variables at once. This technique has already been employed successfully on heroin profiles [101].

A good pre-treatment is expected to reduce the range of values between each variable, while permitting a wide distribution of values from each variable (i.e. intervariability) [188].

3.1.3 Comparison metrics

For multivariate analyses, different factors have to be taken into account [141]:

- Variables of the profile are comparable for each specimen (i.e. they are present in the majority of specimens);
- Values of variables are comparable between all specimens (which is why pre-treatments are employed);
- Variables are independent.

All these parameters have to be investigated for both intravariability and intervariability.

Ideally, each variable should be independent in a profile. If two variables are correlated to each other, the information they convey is similar, so using both variables does not provide extra information and only one variable is discriminating enough to be employed in a comparison process between different specimens [101].

To investigate correlations between variables, various statistical methods exist based on the normal distribution of the variables. If variables do not follow a normal distribution, a non-parametric test should be used. Two methods are popular for this matter, Spearman and Pearson.

Spearman's correlation coefficient is a non-parametric test not taking into account any priori hypothesis regarding the distribution of values [186]. The 'strength' of the relation between two data is tested, in this case the relation between two variables. The more related these two variables are, the closer to zero the correlation value will be [192].

$$\rho = 1 - \left(\frac{6 \sum d^2}{n(n^2 - 1)} \right)$$

Equation 1: Spearman correlation

Where d is the difference between the two ranks of two values from the two variables
and n the number of values in each variable.

Pearson correlation coefficient is similar to Spearman and gives similar results, with the difference that it calculates the linear relationship between the two variables [186]. This method is preferably applied on normally distributed variables, but it is robust enough to take into account outliers and it can be tested on non-normally distributed variables as a comparison method to Spearman [172].

$$r = \frac{\sum xy}{\sqrt{\sum x^2 \sum y^2}}$$

Equation 2: Pearson's correlation

With x and y corresponding to values of two variables.

Once this step has been completed, the final profile is obtained, with the final number of variables of the profile (ideally all independent variables) to be employed to build the training dataset.

Comparison metrics are subsequently employed to build the model in order to separate linked and unlinked specimens. Previous studies have determined that Pearson and Cosine are the two correlation methods giving the best results for cocaine [101, 189], as well as for heroin [191, 193, 194].

Cosine similarity is close from Pearson, except that no mean is taken into account contrary to Pearson, only raw values are used to calculate the similarity. It calculates the dot product between two vectors obtained from values from two variables to be compared [141].

$$\cos(\theta) = \frac{\sum AB}{\sqrt{\sum A^2} \sqrt{\sum B^2}}$$

Equation 3: cosine similarity

Where $\sum A$ and $\sum B$ are values forming vectors A and B .

Statistical methods also exist for binary values (if variables are composed of binary values), and two popular ones are employed in the literature [195]: Hamming distance and Jaccard similarity coefficient.

Hamming distance gives a score by comparing the two 'strings' forming the profile of the two specimens to be compared [195].

As a simplified example, for one profile comprising four variables, if specimen 1 has a string '1011' (one value per variable) and specimen 2 has a string '1010', the Hamming distance is the score of differences between the two strings. Each bit of the string is compared and if the two values are the same it equals 0; if not, it equals 1.

For this example, the score would be '0001' = 0+0+0+1 = 1.

As a result, the Hamming distance ranks from 0 (complete similarity) to the total number of variables forming the profile (if this value is reached, it corresponds to a complete difference between the two specimens).

Jaccard similarity coefficient is close to the Hamming distance however it cumulates the total number of possible situations between the two specimens as illustrated in Table 5:

Table 5: Illustration of Jaccard similarity coefficient calculations

		Specimen 2	
		0	1
Specimen 1	0	$M_{0,0}$	$M_{0,1}$
	1	$M_{1,0}$	$M_{1,1}$

Where M is the total number of situations for each of the four combinations for the two specimens [196].

As an example, using the previous two strings '1011' and '1010', $M_{0,0}$ is 1, $M_{0,1}$ is 0, $M_{1,0}$ is 1 and $M_{1,1}$ is 2.

Jaccard similarity coefficient is defined as:

$$J = \frac{M_{1,1}}{M_{0,0} + M_{0,1} + M_{1,0}}$$

Equation 4: Jaccard similarity coefficient

Using the previous example, $J = \frac{2}{1+0+1} = 1$

Results with the Jaccard similarity coefficient are comprised between 0 (no similarity) and 1 (complete similarity) [196].

Correlation matrices are obtained, whatever the chosen comparison metric. Calculations in correlation matrices are performed as followed:

- Intervariability: all specimens from one seizure are compared to all specimens from all other seizures (see red values in Figure 20).

		Seizure 1			Seizure 2		
Specimen		1	2	3	4	5	6
Seizure 1	1	1	J	K	A	D	G
	2	J	1	L	B	E	H
	3	K	L	1	C	F	I
Seizure 2	4	A	B	C	1	M	N
	5	D	E	F	M	1	O
	6	G	H	I	N	O	1

Figure 20: Intervariability calculations

Simplified representation in a correlation matrix comprising two seizures with three specimens each

- Intravariability: comparisons of specimens within each seizure are performed without taking into account replicate values (see red values in Figure 21).

		Seizure 1			Seizure 2		
Specimen		1	2	3	4	5	6
Seizure 1	1	1	J	K	A	D	G
	2	J	1	L	B	E	H
	3	K	L	1	C	F	I
Seizure 2	4	A	B	C	1	M	N
	5	D	E	F	M	1	O
	6	G	H	I	N	O	1

Figure 21: Intravariability calculations

Simplified representation in a correlation matrix comprising two seizures with three specimens each

3.1.4 Performance of the statistical method

The performance of the different statistical methods employed to build the model has to be evaluated. Previous studies demonstrated that pre-treatments affect the results [189]. Thus, a Receiver Operating Characteristic (ROC) curve has to be calculated for each combination of pre-treatment and correlation method. This ROC curve calculates [197]:

- **True Positive** rate (TP), which is the number of correct classifications as linked specimens when they are indeed linked;
- **False Positive** rate (FP), which is the number of wrong classifications as linked specimens when they are actually unlinked;
- **True Negative** rate (TN), which is the number of correct classifications as unlinked specimens when they are indeed unlinked;
- **False Negative** rate (FN), which is the number of wrong classifications as unlinked specimens when they are actually linked.

The FP rate (ranging from 0.0 to 1.0) is calculated in function of the TP rate (ranging from 0.0 to 1.0) to build the ROC curve. The ideal scenario is to have a TP rate of 1.0 and a FP rate at 0.0 (i.e. perfectly aligned with y axis). In this case, no overlap happens between the intervariability and intravariability, and the model allows a total separation between linked and unlinked populations [36], as described by Locicero et al. [189] and illustrated in Figure 22:

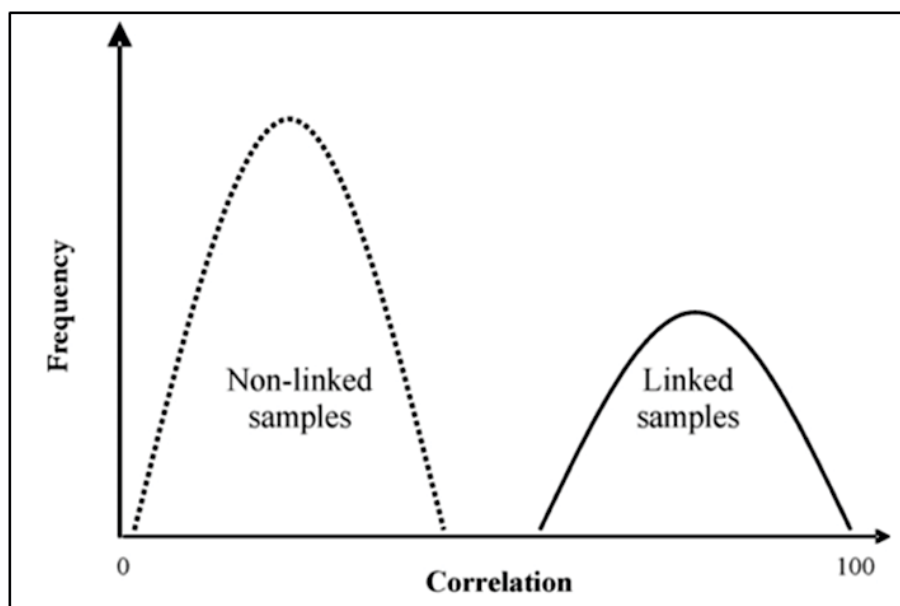


Figure 22: Ideal scenario to discriminate between linked and unlinked population [189]

If there is an overlap, as illustrated in Figure 23 [189], the Area Under the Curve (AUC) is a value allowing the quantification of the overlapping degree, thus giving the probability of classifying a specimen in the correct population. It theoretically ranges from 0.5 (distributions completely overlapped) to 1.0 (distributions entirely separated thus 100 % chance of correct classification of the specimens) [101, 197].

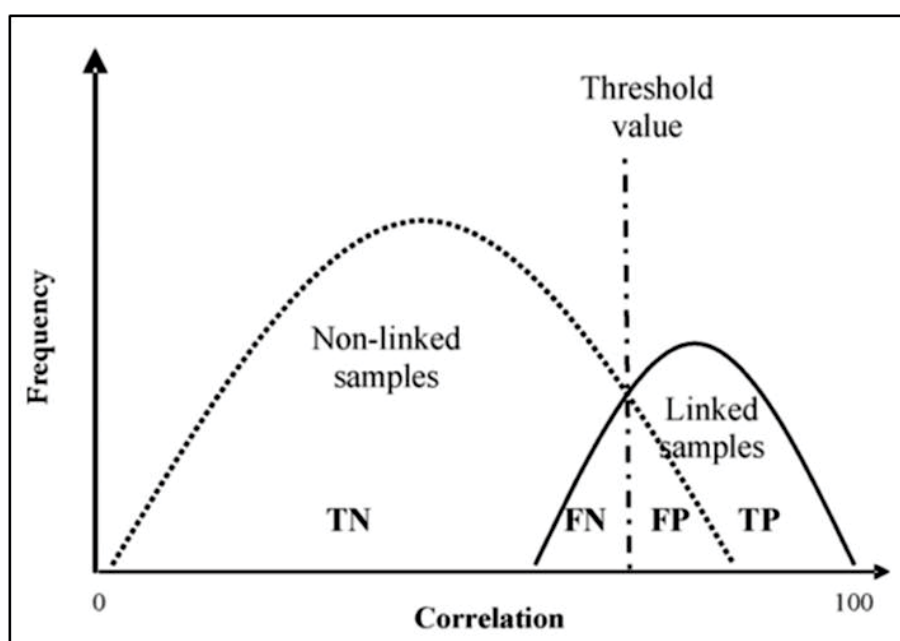


Figure 23: Common scenario to discriminate between linked and unlinked population [189]

The highest AUC determines the best combination of statistical methods to be applied on the dataset to build an efficient model, which permits to discriminate between linked and unlinked specimens with correct classification [10, 101, 186, 189].

To finish, a threshold value conditions the rates of accepted FP and FN. From a forensic intelligence perspective, having two seizures that should be linked but are considered as unlinked (i.e. FN rate) has a major negative effect as it remains not detected, and potential dismantling of trafficking network is hidden [84]. The priority is to minimise this scenario [22].

FP rates are also a concern if two seizures are linked when they should not. Indeed, this would lead to resources deployed to arrest suspects and an entire investigation based on erroneous results, and that could potentially lead to prosecution based on wrong assumption.

As a result, the threshold value has to be determined in order to optimise both FP and FN rates in accordance with accepted error rates by the Police.

3.1.5 Scope of this chapter

The aim of this study was to develop a discriminative model using chemical information from cocaine and heroin border seizures. This was performed by adapting work from previous studies [73, 84, 89, 142, 171, 188, 194]. In those previous studies, a small pool of seizures considered as 'suitable' was selected based on physical and chemical criteria. A sampling procedure was determined and analytical procedures were then optimised using selected specimens. Statistical methods were subsequently applied and optimised to obtain an 'acceptable' discrimination method.

On the contrary, specimens to be profiled in this study could not be selected as they were chemical results from NMI analyses, seizures could not be sampled and analytical techniques could not be optimised for the purpose of comparisons. Therefore, statistical methods had to be employed a posteriori. The overall question was: can the information stored in the database (i.e. chemical profiles of cocaine and heroin specimens) be used to establish a discriminative model and be employed routinely to link seizures supposed to be linked?

The objective of this study was to assist law enforcement and other authorities (the AFP in this case) in getting a better knowledge of useful information contained in the database for intelligence purpose, especially to determine:

- Relevant information to be exploited in the database in a timely fashion;
- Variables of interest allowing the discrimination between specimens based on chemical information, as well as variables to be prioritised for each profile;
- The feasibility for an automated process to be routinely implemented.

A full exploitation of specimens' information (see data provided in Chapter 2) was performed using Microsoft® Excel (Microsoft Corporation, United States) and R® programming (The R Foundation for Statistical Computing, version 2.14.1, Austria).

3.2 Methodology

3.2.1 Determination of suitable seizures

3.2.1.1 *Intravariability*

Selected seizures for the intravariability of the training set comprised either one of the following characteristics:

1. Seizures comprising the highest number of specimens, expected to be the most representative of their chemical composition [10];
2. Seizures with a high weight comprising few specimens (at least three), indicating similar physical properties of all specimens. If a seizure was considered as (visually) homogeneous, then fewer specimens were prepared and sent for analysis;
3. Seizures with a small weight comprising multiple specimens, suggesting a better representativeness of the overall chemical profile of the seizure (reverse assumption from 2. above);

4. One subgroup preferred, suggesting that specimens could not be distinguished based on their physical properties¹³;
5. Specimens preferably in block form, suggesting the integrity of the original batch and reducing the possibility to have different products mixed together at a more advanced level of distribution (whereas it could be the case with powder).

3.2.1.2 Intervariability

Unlinked seizures were determined based on information from the police, using case numbers and operation names from cases (as it was unknown whether or not seizures could be linked based on their chemical profile). Seizures with different PROMIS numbers or case names were considered as unlinked because they had not been previously linked.

All the seizures not linked by the Police were extracted from the dataset. A reduced set of seizures was randomly selected from all unlinked seizures. Twenty-one seizures¹⁴ containing 156 cocaine specimens and 280 heroin specimens for the intervariability were considered as a sufficient representativeness of the whole dataset as it corresponded to a total of 10,251 comparisons performed with cocaine specimens and 35,508 comparisons performed with heroin specimens.

¹³ Specimens sub classified into different subgroups, thus visually differentiable, may imply or not chemical differences. Both physical and chemical information may be complementary [121], but no study has yet established they would provide contrary information.

¹⁴ This number is not arbitrary and is based on the final number of seizures selected for the intravariability, i.e. 7 (see next section). Three times more, i.e. 21, is considered as representative enough from the whole dataset.

3.2.2 Choice of variables

Only variables present in the majority of specimens were considered as discriminative for comparison purposes [141]: variables being absent in more than 90 % of all specimens were not considered for the rest of the study (see Appendix 1): they corresponded to adulterants and diluents. This cut-off value at 10 % was not attributed accordingly to any previous study and was decided in accordance with the AFP.

Solvents present in less than 10 % of cocaine specimens could not be removed due to the methodology employed by NMI (see Chapter 2). Regarding heroin specimens, solvents values were not provided in the dataset. Indeed, their presence or absence was recorded - which allowed to obtain statistics in Chapter 2 - but no numerical value was provided to perform comparison metrics with those variables.

The set of variables was consequently reduced to:

- Alkaloids and solvents' profiles for cocaine specimens;
- Alkaloids and impurities' profiles for heroin specimens.

If combining both alkaloids and solvents that were separately investigated, the chemical information for each specimen may be increased and might lead to better discriminations between both populations [171]. Both correlation matrices of both profiles were combined in one single matrix by calculating the average score of each cell in the matrix [198-200], calculated as follow:

$$Total\ score\ cell\ n_{combined\ matrix} = \frac{score\ cell\ n_{alkaloids\ profile} + score\ cell\ n_{solvents\ profile}}{2}$$

Equation 5: calculations for combined matrix of alkaloids and solvents' profiles

This procedure was not possible for heroin specimens as seizures almost never comprised both alkaloids and impurities profile completed for the same specimens.

3.2.3 Pre-treatments

Pre-treatments were tested and specimens were then compared as follow:

- No pre-treatment (original values of variables);
- Normalisation (N);
- Square root (Sqrt);
- Normalisation followed by square root: a combination of both normalisation and square root was performed (N + Sqrt), by applying first the normalisation, and then applying the square root on the obtained value.

Moreover, the feasibility of including truxillines to the alkaloids' profile of cocaine specimens was assessed. The scales of values was homogenised using a linear transformation (LT) as a factor 10 was noticed between alkaloids' values and truxillines values. Two possibilities were investigated:

1. Lowering values from truxillines to fit the range of values from other alkaloids by dividing truxillines' values by ten (LT/10). The limitation with this methodology is a risk of flattening variations between values as the range is reduced;
2. Increasing alkaloids values to fit the range of values from truxillines by multiplying alkaloids values by ten (LT10). This methodology has drawbacks, because values are obtained from analytical techniques and standard deviations are associated with each value. By increasing these values, there is a risk of increasing these standard deviations and as a result increasing the variations between specimens' profiles.

For heroin specimens, linear transformations were trialled to improve the intervariability's distribution of the alkaloids' profile, as recommended by Esseiva et al. [101]. The values of alkaloids acetylcodeine and 6-MAM were divided by two (LT/2) and three (LT/3).

3.2.4 Comparison metrics

The normal distribution of the selected variables was assessed with Q-Q plots, then Spearman and Pearson correlations were employed to determine what method to apply on the profiles of each drug, i.e. parametric or non-parametric methods.

For cocaine specimens, Pearson and Cosine were tested on the alkaloids' profile. For the solvents' profile, two types of information were used from the specimens file: the values of each analysis, giving the first solvents' profile investigated using Pearson and Cosine correlation; and the second solvents' profile containing binary values, based only on the presence or absence of each solvent, analysed with Hamming distance and Jaccard similarity coefficient.

For heroin specimens, Pearson and Cosine were employed on both the alkaloids and impurities' profiles.

3.2.5 Correlation matrix and classification

Intravariability and intervariability matrices were obtained for each profile of each drug and with the different combinations of pre-treatments and correlation methods tested.

Subsequently, the frequency of each correlation within the matrix was assessed. Values were transformed in absolute values to simplify calculations (i.e. only positive values).

For a better visualisation, values were multiplied by 100 (see Figure 24). Categories ranging from 0 to 100 (i.e. correlation values) were created by attributing the frequency of correlations counted in the matrix.

The total number of correlations was obtained for each category, which was then converted in a percentage value based on the total number of correlations in the correlation matrix (see Figure 24).

The percentage value of each category was finally plotted in a bar chart comprising intervariability and intravariability values.

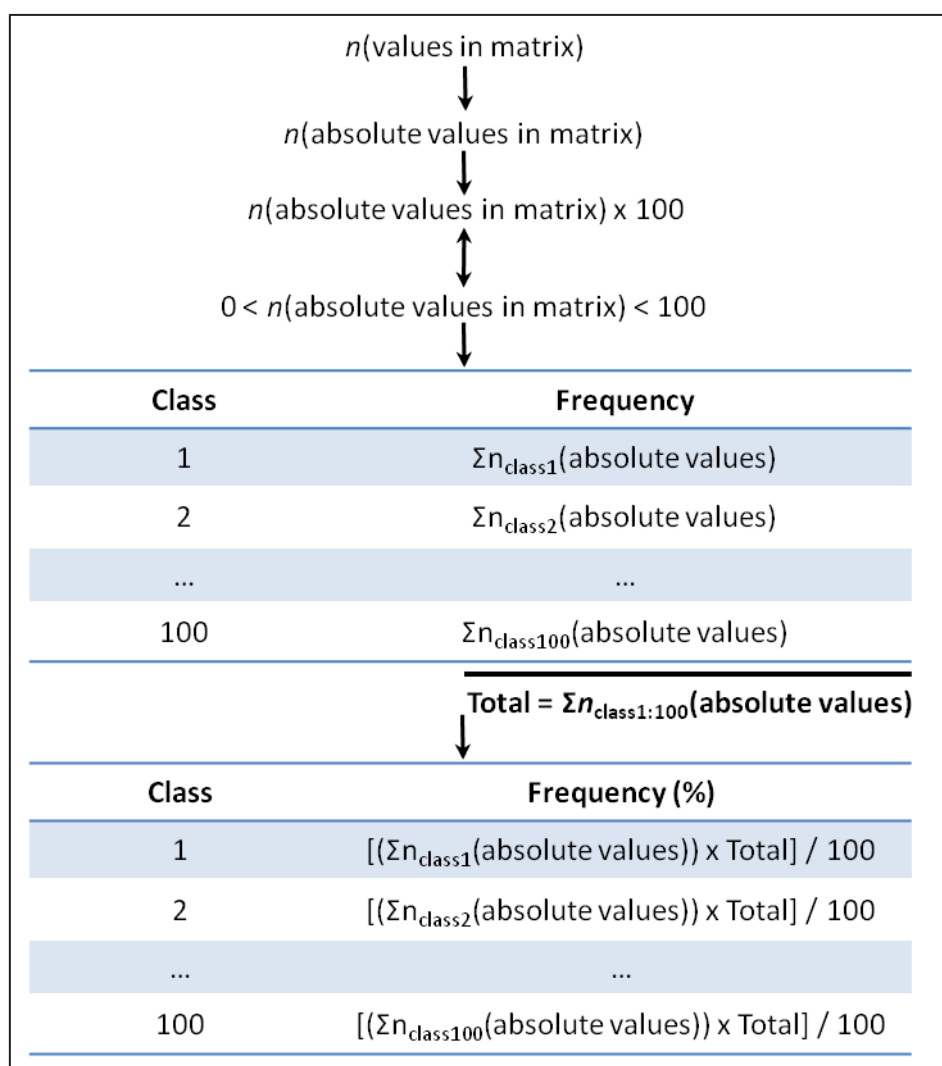


Figure 24: Procedure to obtain categories from correlation matrices

3.2.6 Performance of the statistical method

For each profile, the AUC was obtained for each combination of pre-treatments and correlation methods. The best AUC was selected and a threshold was determined to get the best optimisation between false positives and false negatives.

3.2.7 Seizures linked with circumstantial information

Models obtained for cocaine and heroin data were subsequently tested with specimens coming from seizures that had been previously linked based on circumstantial information. The aim was to investigate if chemical profiles and circumstantial information could be used to link the same seizures (thus conveying similar information), and if potential links that were not previously detected may be highlighted using the model. Seizures linked with a same PROMIS number, thus relative to investigation, were selected.

3.2.8 Summary of the methodology followed in this study

The overall procedure employed for this study is summarised in Figure 25:

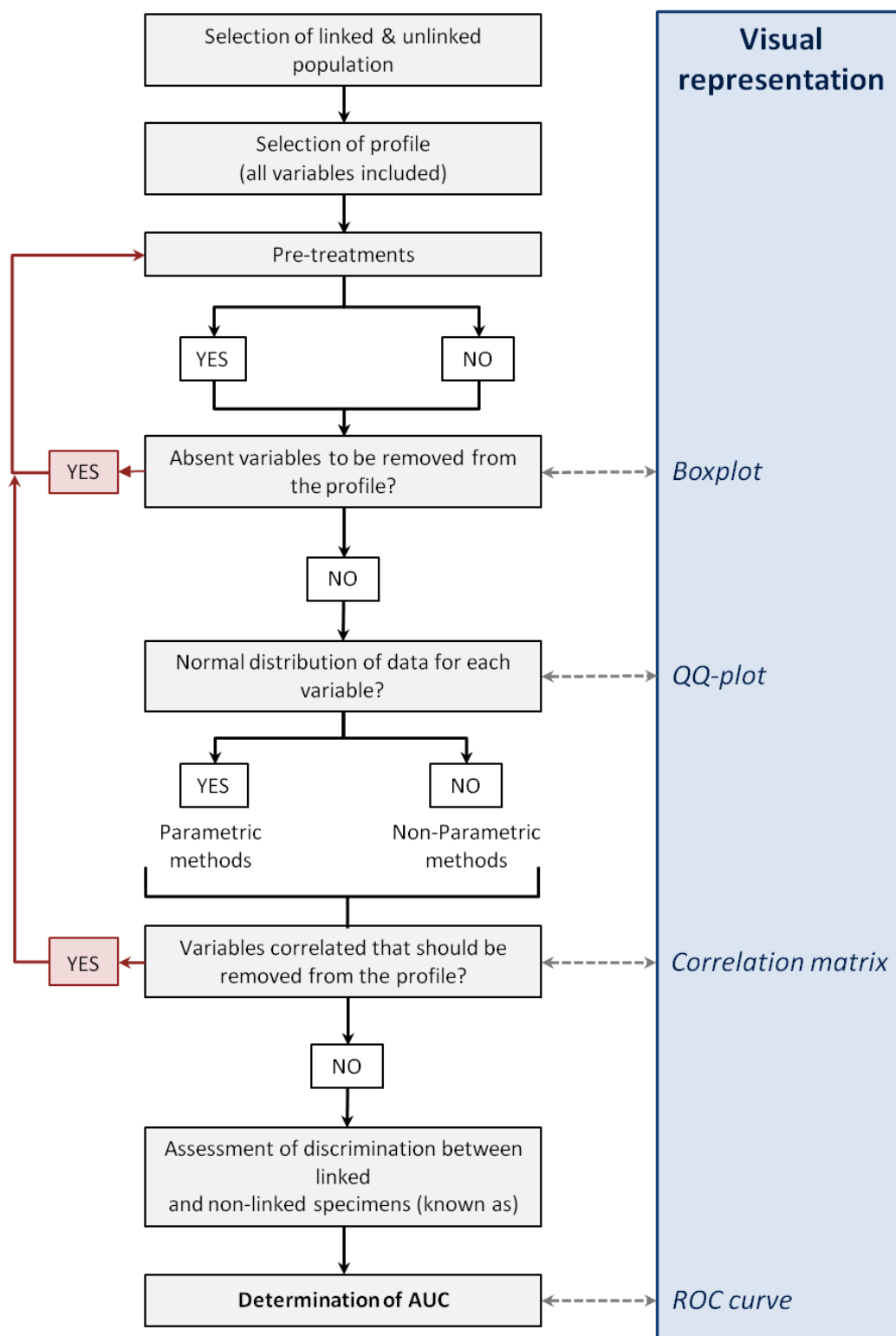


Figure 25: Methodology employed in this study

3.3 Results

3.3.1 Determination of linked and unlinked populations

3.3.1.1 *Alkaloids' profile*

In this profile, values employed were alkaloids values relative to:

- Cocaine in its base form for cocaine specimens;
- Heroin in its base form for heroin specimens.

The intervariability of the alkaloids was analysed: trans-Cinnamic acid values in cocaine specimens, as well as codeine and papaverine in heroin specimens were close to 0 in the majority of specimens. These variables were not discriminative so they were removed from the alkaloids' profile of respectively cocaine and heroin specimens. The resulting alkaloids' profiles were comprised of nine variables for cocaine specimens and five variables for heroin specimens.

The intravariability was subsequently examined. Seizures with the highest number of specimens had a wide variability, which was opposite to the wanted outcome (i.e. small variability) so they were not considered for the training set.

Moreover, block form was considered more homogeneous than powder form due to its physical unity (see section 3.2.1). This assumption was tested by selecting seizures in block form compared to seizures in powder form (each seizure containing one subgroup). The obtained variability was wider with seizures in blocks, which was opposite of what was

expected. Consequently, seizures for the training set were not selected based on this criterion and powder seizures were also considered.

For the remaining seizures considered as good candidates for the training set, the ones with a wider variation, indicating different values between specimens within those seizures, could have been removed for a better result (i.e. smaller variability). However, it would not be consistent with reality (that is to say being representative of variations within the whole dataset) and the model would not be correctly constructed to classify seizures based on their values if only 'ideal' candidates were considered. The seizures corresponding to the characteristics established in section 3.2.1.1 were therefore all kept for the intravariability of cocaine and heroin.

3.3.1.2 Solvents' profile and impurities' profile

In the solvents' profile of cocaine specimens (see Appendix 3), acetonitrile, chloroform, MIBK, values were at zero. The relevance of these three solvents in the profile could thus be questioned as well as the relevance of the search of such compounds in cocaine specimens. Nevertheless, they could not be removed from the profile due to NMI procedure. In addition, mesitylene, methylene chloride, methylisobutenylketone and methylbenzoate only contained outliers meaning that few values were obtained after analysis as boxplots could not be built, making these solvents also questionable. The solvents' profile from heroin specimens could not be used to select linked population because only their presence or absence was recorded.

The impurities' profile of heroin specimens was very similar to the solvents' profile of cocaine specimens (see Appendix 4), because none of the variables could be removed from the profile due to NMI procedure, although eight out of 23 variables had most values close to 0 or were outliers.

Seizures with the highest number of specimens had a wide intravariability, similarly to the alkaloids' profile: they were removed for the rest of the study. Selected seizures for the intravariability of cocaine specimens and heroin specimens had a small variability in their solvents' profiles and impurities' profiles, which was promising for comparison purposes.

3.3.2 Pre-treatments

The comparison of the different pre-treatments performed on alkaloids profiles (see Appendix 5 for cocaine and Appendix 6 for heroin) showed that:

- The distribution of original values (i.e. no pre-treatment applied on values) was wide but not represented by the majority of values comprised between the two quartiles, as there were many outliers for each alkaloid;
- Normalisation applied on original values reduced the range of values as well as the number of outliers, which was a great improvement compared to original values. The intervariability was smaller than with original values, which was not favoured;
- Square root applied to original values had the same effect as normalisation in term of reducing the number of outliers, and in addition it allowed having a wider distribution of the values. This pre-treatment was definitely efficient on this dataset;
- Normalisation followed by square root had similar effect to square root on the distribution of the values. Less outliers were noticed compared to square root, however the intervariability was reduced compared to square root;
- Linear transformation of acetylcodeine and 6-MAM in heroin specimens reduced the difference between the two variables and other alkaloids, especially dividing by three.

Therefore, square root or normalisation followed by square root impacted on resulting values. The number of outliers was reduced while the distribution was improved.

For solvents' profile of cocaine and impurities' profile of heroin, no normalisation or linear transformation could be applied (i.e. dependant variables). Square root was the only pre-treatment that could be employed and it was efficient on both profiles. Nevertheless, in order to verify the performance of the method, the best pre-treatment was determined only after having performed a ROC curve.

3.3.3 Correlation between variables

3.3.3.1 *Alkaloids' profile*

The correlation between variables was assessed. Alkaloids were non-normally distributed for both cocaine and heroin specimens (see Appendix 7), thus non-parametric methods were employed on the alkaloids' profile [172].

Correlation matrices were obtained using both Spearman and Pearson (see Appendix 8 and Appendix 9). For cocaine specimens, the highest correlation between two variables obtained was 0.7 (between two isomers, trans and cis-Cinnamoylcocaine) using Spearman, which was a 'strong' correlation according to Weir [192]. This correlation was expected as isomers have close values due to their chemical and molecular nature, but statistical values have to be reassessed when applied to drug profiling. Indeed, a 'strong' correlation can be interpreted differently when it suggests removing a variable, thus removing part of the chemical information. The right balance has to be found between variables correlated and loss of information if the variable is removed [141]. In this profile, 0.7 was considered as not high enough to remove one variable according to the literature [141]. Moreover, for heroin specimens, weak correlations were observed. As a result, variables from both alkaloids' profiles were kept.

3.3.3.2 *Solvents' profile and impurities' profile*

Both profiles were 'percentage values' profiles: variables were dependant. Thus, there was no value to calculate the correlation between variables from both profiles, as none of them could be removed.

3.3.4 Correlation between specimens

3.3.4.1 Alkaloids' profile

AUC values obtained using different combinations are summarised in Table 6 for cocaine specimens and Table 7 for heroin specimens:

Table 6: AUC values obtained for alkaloids' profile of cocaine data

Pre-treatment	Similarity measurement method	
	Pearson	Cosine
None	0.9369	0.8986
Normalisation	0.9369	0.8986
Square root	0.9702	0.9005
N + Sqrt	0.9702	0.9005

Table 7: AUC values obtained for alkaloids' profile of heroin data

Pre-treatments	Similarity measurement method	
	Pearson	Cosine
None	0.8751	0.8949
Normalisation	0.8760	0.9024
Square Root	0.8814	0.9125
N+Sqrt	0.8814	0.9125
N + LT/2	0.8064	0.8866
Sqrt + LT/2	0.8328	0.8878
N + Sqrt + LT/2	0.8328	0.8878
N + LT/3	0.8116	0.8810
Sqrt + LT/3	0.8382	0.8781
N + Sqrt and LT/3	0.8452	0.8753

First of all, results were better for cocaine specimens using Pearson compared to Cosine, whereas the reverse was observed with heroin specimens.

Secondly, AUC values were identical if no pre-treatment or normalisation was applied to the dataset for cocaine data. Similarly, AUC values were the same if square root or normalisation followed by square root was applied to both cocaine and heroin data. Therefore, normalisation seemed to have no impact on final correlations.

Moreover, linear transformations attempted on heroin data did not improve the AUC, and even lowered its value.

As a result, the correlation between specimens was best using Pearson for cocaine and Cosine for heroin as long as the square root was applied, whether or not data was previously normalised.

The overall distribution of both intervariability and intravariability are presented in Figure 26 for cocaine data and in Figure 27 for heroin data. An overlap occurred between both populations, for cocaine and heroin specimens.

For cocaine data, low Pearson values observed in the intervariability (in blue) suggested specimens with distinct alkaloids' profiles. High correlations were also observed: specimens from different seizures apparently unlinked might have similar alkaloids' profiles. The intravariability (in red) presented high correlations, as it was expected from specimens of a same seizure. Moreover, the highest correlations (superior to 99) were present for almost 50 % of comparisons with cocaine specimens, implying similar alkaloids' profiles. However, lower correlations were also present down to 60; even if being in small frequencies, it could be a problem when willing to classify correctly unknown specimens. Cocaine seizures seemed to contain specimens with highly variable profiles.

For heroin seizures, the intravariability comprised in majority high correlations (more than 80 % of specimens had correlations higher than 99), implying that specimens within one heroin seizure had similar chemical profiles. However, the obtained intervariability contained high correlations, similarly to cocaine seizures, implying that seizures considered as unlinked had similar chemical profiles.

For both cocaine and heroin, both populations could not be clearly identified due to the overlap: a threshold had to be determined.

Regarding cocaine, a TP rate of 90.20 % contained 13.18 % of FP, which was not ideal, as AFP standards of FP rates are willing to be less than 10 % for intelligence purposes (after consultation with AFP). Regarding heroin, the minimal FP and FN rates were higher than 15 %. The establishment of a threshold was thus complicated with alkaloids' profile of cocaine and heroin, as it did not allow obtaining high TP and TN with low risks of wrong classification (i.e. FP and FN).

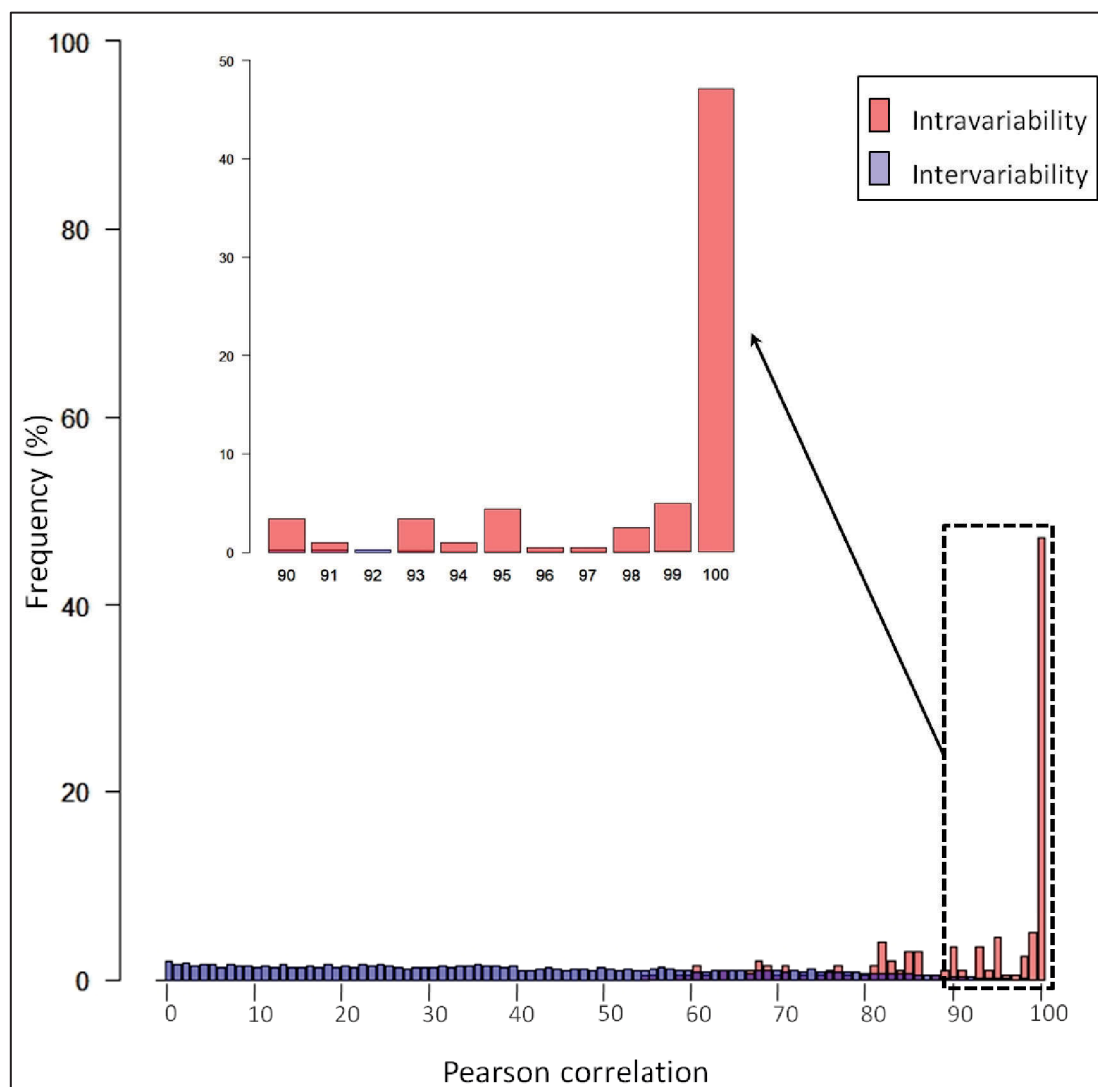


Figure 26: Discriminative model for the alkaloids' profile of cocaine data

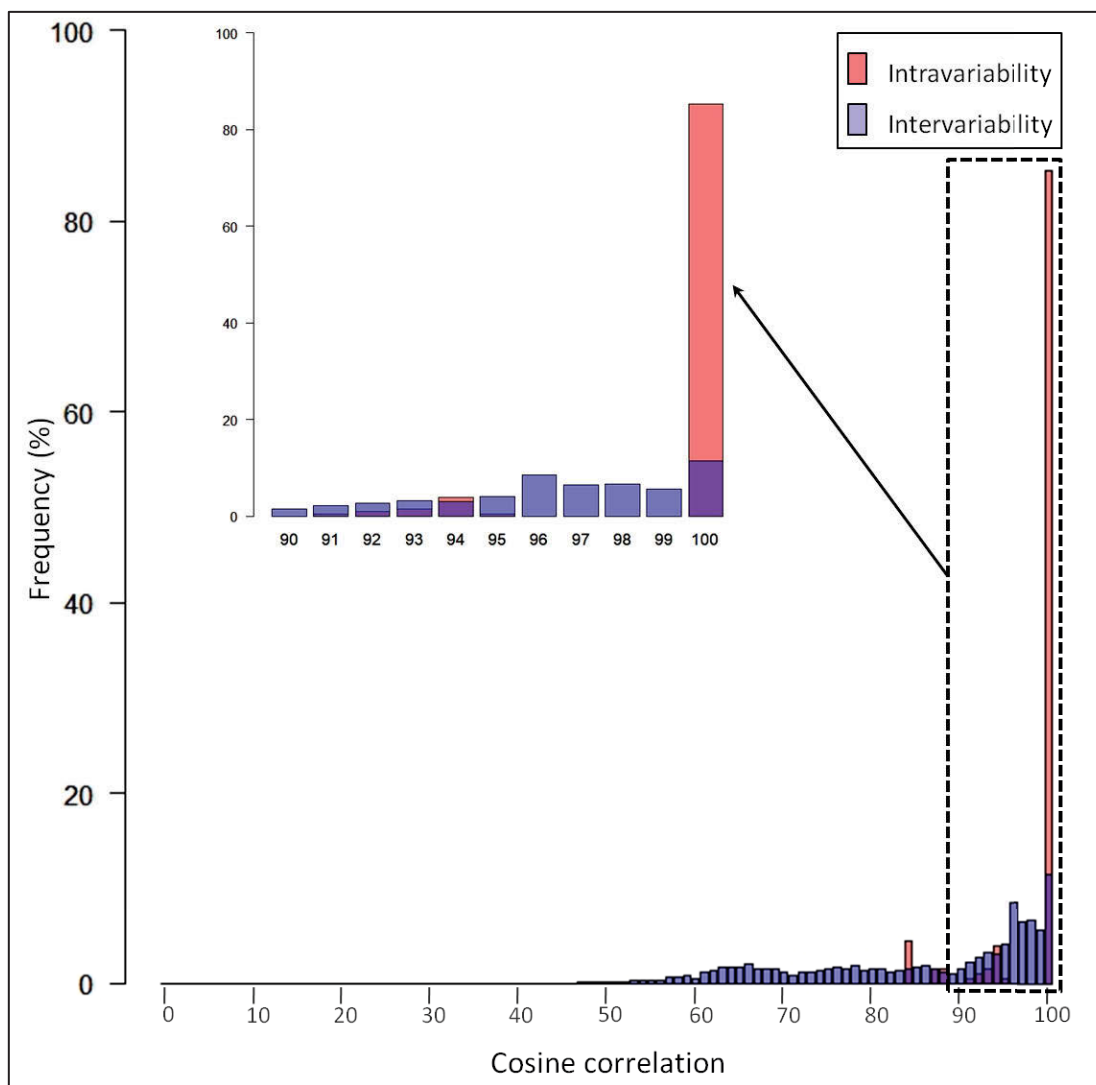


Figure 27: Discriminative model for the alkaloids' profile of heroin data

3.3.4.2 Alkaloids' profile including truxillines of cocaine specimens

Truxillines were subsequently added to the alkaloids' profile of cocaine specimens. The whole process was performed with the addition of a new variable as described in Figure 25.

The normal distribution of truxillines was assessed: similarly to the other alkaloids, the truxillines variable was not normally distributed. Secondly, correlations between variables were analysed: adding truxillines did not change the outcomes obtained with the alkaloids' profile (see section 3.3.1.1), i.e. all variables were not correlated. Thus, all variables were kept (i.e. nine alkaloids previously selected, plus truxillines) to assess the discrimination between linked and unlinked populations.

With the linear transformation from the first possibility (i.e. LT/10 as described in section 3.2.3), rates of FP and FN were high, thus this method did not improve results. However, when employing the second possibility (LT10 as described in section 3.2.3), an AUC of 0.9815 was obtained, which was higher than the AUC previously obtained for the alkaloids' profile without truxillines (i.e. 0.9702). Moreover, similar FP rates (i.e. 9.36 %) were obtained compared to the alkaloids' profile at a higher correlation (correlation of 81 instead of 75) and the number of FN was reduced to 2.45 % at equal correlation, i.e. 81 (instead of 11.76 % of FN at similar FP rate for the alkaloids' profile without truxillines).

Therefore, adding the variable truxillines to the profile provided a better discrimination between linked and unlinked populations while efficiently reducing the FN rates. The threshold was best determined at a correlation of 81, as illustrated in Figure 28, implying that:

- Specimens with correlations lower than 81 could be considered as unlinked (i.e. different chemical profiles), with an associated risk of 2.45 % of not being detected (FN rate);
- Specimens with correlations higher than 81 could be considered as linked (i.e. similar chemical profiles), with a risk of 9.36 % of not being actually linked (FP rate).

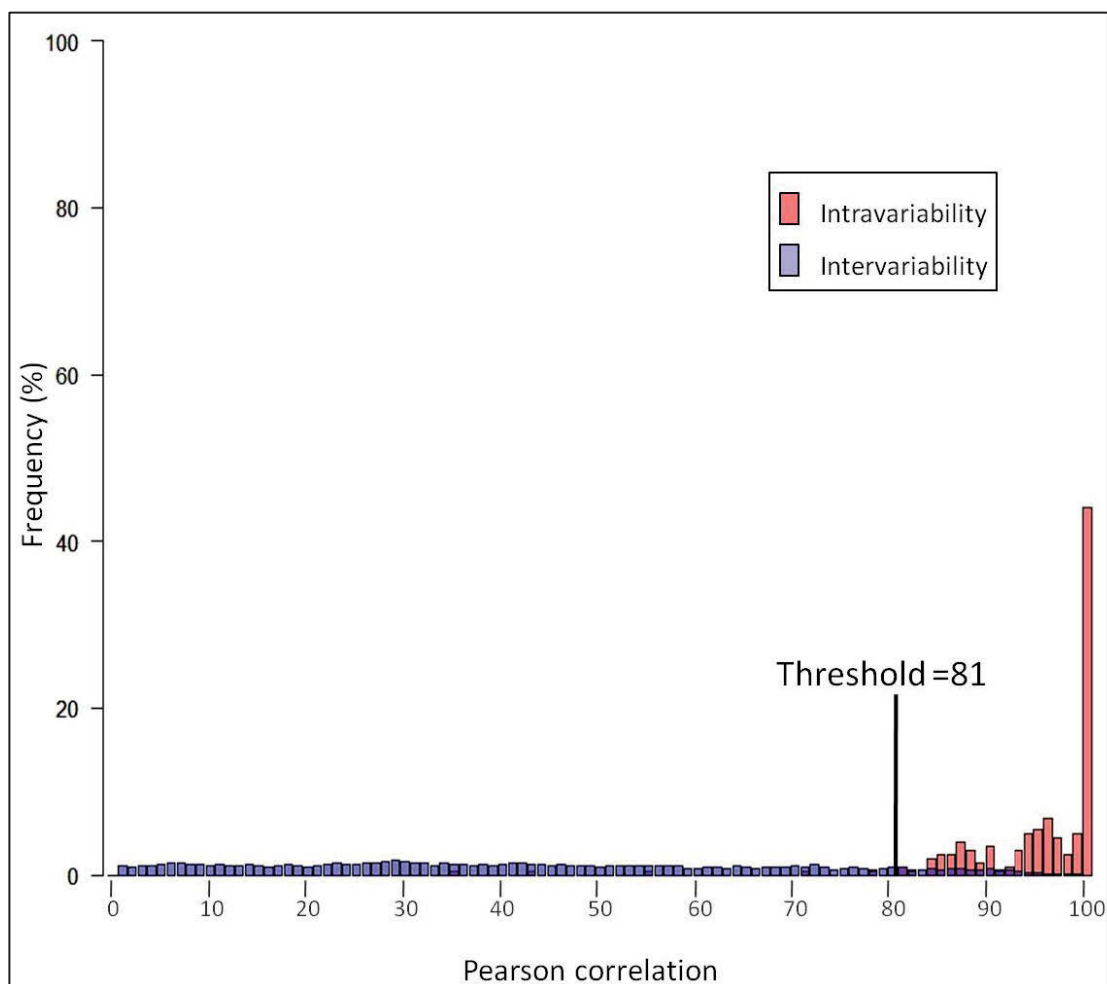


Figure 28: Discriminative model for the alkaloids' profile including truxillines of cocaine data

3.3.4.3 Solvents' profile of cocaine specimens

Obtained AUC values with solvents' profile are summarised in Table 8:

Table 8: AUC values obtained for solvents' profile of cocaine data

Pre-treatment	Similarity measurement method	
	Pearson	Cosine
None	0.7706	0.7924
Square root	0.8487	0.8944

Overall, square root improved results. The best similarity measurement method was Cosine as it gave the highest AUC value. Nevertheless, AUC values were much lower for solvents' profile than for alkaloids' profile.

The overall distribution of both intervariability and intravariability using Square root followed by Cosine produced a complete overlap between both populations (see Appendix 10).

As an alternative to percentage values for the solvents' profile, presence or absence of solvents' data was investigated (binary values). Obtained results were similar to percentage values (see Appendix 11), as a total overlap was observed between both populations with both Hamming distance and Jaccard similarity coefficient.

3.3.4.4 Impurities' profile of heroin specimens

Obtained AUC values with impurities' profile are summarised in Table 9:

Table 9: AUC values obtained for impurities' profile of heroin data

Pre-treatments	Similarity measurement method	
	Pearson	Cosine
Original values	0.9736	0.9778
Square Root	0.9906	0.9942

The best AUC was obtained when square root was applied on the dataset followed by Cosine. The high value obtained, almost close to 1.0 suggests a 'perfect' performance of the method, as presented in Figure 29:

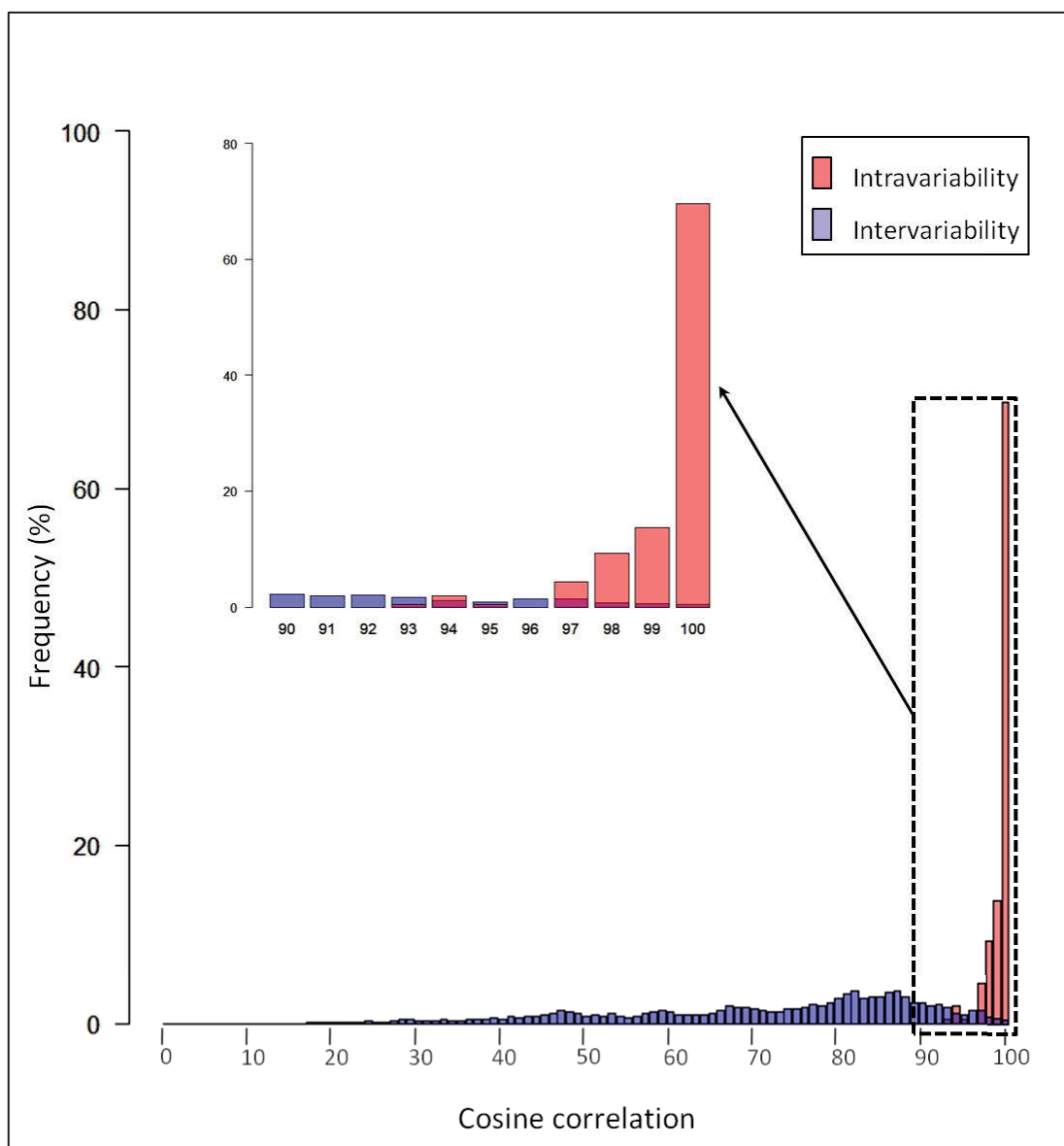


Figure 29: Discriminative model for the impurities' profile of heroin data

FP and FN rates obtained were lower than for cocaine specimens, and it was even possible to establish a threshold at a correlation as high as 96 and obtaining 4.82 % of FP and 2.94 % of FN.

3.3.4.5 *Profile combining alkaloids and solvents for cocaine specimens*

The obtained distribution had a minimised overlap between intervariability and intravariability compared to the alkaloids' profile (see Appendix 12). Truxillines were not included in the profile as the variable did not improve results when combining the solvents' profile.

The obtained FP, TP, FN and TN rates were improved compared to the alkaloids' profile. Indeed, at equal TP rates (see Appendix 13), FP and FN were lower, for instance with a TP rate of 90.20 %, FP were at 5.89 % and FN were at 9.80 %. A FP rate of 10 % would be tolerated in this case, as the FN rate would be reduced to less than 5 % (communication from AFP).

With a threshold at a correlation of 75, compared specimens with a correlation higher than 75 implied that:

- They had 95 % chance of being linked (TP rates);
- There was less than 10 % chance that they were actually not linked (FP rates).

Also, if compared specimens had a correlation inferior to 75:

- They had 90 % chance of not being linked (TN rates);
- There was less than 5 % chance that they were actually linked (FN rates).

3.4 Discussion

3.4.1 Choice of statistical methods

There is a possibility that results could be improved using other statistical methods. Nevertheless, the aim of this study was not to review all possible pre-treatments and similarity measurement methods, but to assess the feasibility of selected statistical methods already identified in the literature to build a discriminative model when applied to the provided dataset [101, 189].

The obtained AUC for the different profiles were directly impacted depending on the pre-treatment and correlation methods employed. Normalisation had no impact on final correlations. A possible explanation could be due to NMI procedure, as values were already normalised to an internal standard, it potentially cancelled out the effect of a second normalisation applied to all variables.

This study demonstrates that the choice of statistical methods can influence the results. Esseiva et al. [101] already demonstrated that pre-treatments have an impact on the results: this is a sensitive step in the process of building the model. Normalisation followed by square root was the pre-treatments' combination advocated by Locicero et al. [189] with their cocaine specimens. Furthermore, linear transformations had an impact on heroin data in the study of Esseiva et al. [101], contrary to the results obtained with this dataset.

Consequently, even following previous studies with similar datasets, different analytical procedures can influence the results and lead to the need to employ different statistical methods when developing a discrimination model. Testing different statistics is thus of importance, but mostly being aware of disparities which may happen at each step of the

methodology, from the pre-treatments, to the selection of variables, to the similarity measurement methods.

Furthermore, uncertainties, or errors rates, were a consequent part of this study: the determination of a threshold was essential. Nevertheless, when establishing definite threshold values, results are interpreted with either black or white answer. For instance, cocaine specimens were considered as not linked if their correlation was below 81. This type of reasoning may engender less detected links as restricting accepted results only above a precise value. For instance, specimens with correlations below – but close to – 81 (i.e. correlation of 78 for instance) could be questioned as being also part of the linked population, because their correlation being so close from the decided threshold. Thus, a ‘grey area’ exists which should be taken into consideration when dealing with false positives and false negatives. Alternatives have been developed to the use of a definite threshold value, especially verbal scales which were proposed in this Chapter to express results with words instead of definite numbers. This approach is currently employed in other fields such as in DNA. This has proven reliability when presenting evidence in court [201, 202]. However, the subjectivity in attributing the value and comparing a binary value (i.e. yes or no) to a verbal probability (i.e. ‘strong conviction’) of specimens to being linked may be questioned. Consequently, pros and cons exist with the different approaches, the most important is to be aware of those grey areas and taking them into account when applying one approach [203].

3.4.2 Selected seizures for the training dataset

Multiple parameters were taken into consideration to select seizures to be included in the training dataset to build the model that could have influenced the results.

First of all, seizures in block form appeared to be non-homogeneous. A possible explanation is that when manufactured, different batches may be combined to form a block, adding more variation in the resulting chemical profile (as it results in a mix of different chemical profiles) [95]. Also, chemical results might be influenced depending on where the specimen is taken on the block (i.e. sampling location), if it does not have a homogeneous composition. This crucial step of the procedure has been investigated by Zamengo et al. [72] and they demonstrated the differences in chemical profiles of specimens depending on the sampling procedure when the material was non-homogeneous. Sampling procedures may have to be changed or at least adapted so that the selected specimens ensure the representativeness of the whole cocaine and heroin seizure.

Secondly, specimens from a same seizure were assumed to have similar chemical profiles and higher correlations than intervariability. In this study, it was found that it is not the case with all seizures, for instance the solvents' profile of cocaine specimens contained high variations within one seizure. Wide variations within seizures impact the model as well as correlations of the intravariability, and it might be a problem when deciding a threshold value based on these variations. Indeed, the more variations in the intravariability, the more difficult it is to set a threshold value.

Thirdly, alkaloids are considered as chemically stable compounds, thus they are assumed to give reproducible results over time, and providing reliable profiles to be compared (assuming similar storage conditions) as established by Nielson et al. [177]. Their study concluded that alkaloids profile was not affected by storage conditions over twelve months. However this assumption could not be verified in this study. The provided seizures might have been stored for more than twelve months and under variable storage conditions (i.e. heat, humidity, and packaging quality) affecting the overall drug profile.

Finally, both physical and chemical characteristics were assumed to convey similar information: seizures divided into several subgroups, thus based on visible differences, were considered as having specimens with more variable chemical profiles than seizures with only one subgroup. However some selected seizures with only one subgroup had wider variability than seizures with multiple subgroups, implying that items considered as visually (and thus physically) non-distinguishable were in fact chemically different.

3.4.3 Chemical profiles of cocaine specimens

Chemical values of alkaloids from cocaine specimens allowed the development of a model able to classify linked and unlinked seizures. However, the associated risk of wrong classification (FP and FN) was greater than to 10 %. The high correlations observed with the intervariability as well as low correlations observed with intravariability (see Figure 26) suggest that cocaine seizures may contain specimens with distinct profiles; this drug seems to be multi-profile. This outcome was already demonstrated with Swiss data [141].

When using alkaloids and solvents information combined together, chemical profiles allowed discriminating between linked and unlinked specimens. Results were greatly improved when adding the variable truxillines to the alkaloids' profile compared to a combination of alkaloids and solvents values, suggesting that:

1. Truxillines were an important variable as they improved the results when added to the alkaloids' profile;
2. When truxillines were added to the alkaloids' profile, higher correlations were obtained compared to all solvents together (i.e. one variable instead of eighteen variables);
3. If truxillines were added to the alkaloids' profile, adding solvents' profile did not improve the model.

This last statement has several consequences. Indeed, it highlights the fact that it is actually possible to optimise a model to separate two populations with only one profile, here being the alkaloids' profile including truxillines. The preparation step for analysing truxillines is longer than for alkaloids; however both profiles are obtained using a GC-MS. Focusing on this profile would save time, resources and money, as only one analytical instrument would obtain the most useful information in the first place.

3.4.4 Chemical profiles of heroin specimens

The best statistical model was obtained with the impurities profile of heroin specimens, and it was possible to set a threshold giving low false positives and false negatives rates (less than 5 %).

It may be explained due to the fact that heroin specimens within each seizure have similar profiles, as seen with the alkaloids' profile. Moreover, with this profile, twenty-three variables were taken into account, and the more variables the more information may be used to discriminate between specimens (even if some variables were close to 0 and should be removed from the profile if this was feasible).

It implies that if implemented routinely by the AFP, this model would classify correctly specimens as linked or unlinked with a confidence of having less than 5 % risk of wrongly linked heroin specimens and less than 3 % risk of not detecting a link.

This outcome demonstrates great potential when using impurities from heroin specimens. Impurities have already proven to be efficient in classifying unknown heroin specimens, using unsupervised methods (i.e. in this case partial least squares discriminate analysis) [204]. Moreover, it is possible to determine the manufacturing process based on the retrieved impurities [205], and going further, profiling of impurities has been used with success in Brazil for the determination of geographic origin of heroin product [147, 185].

Nevertheless, with new specimens entering in the database, the threshold should be re-evaluated. Indeed, with new specimens, new chemical classes may be created, it is thus important to update threshold values to keep the wanted FP and FN rates (also evolving with new specimens entering the database) [206].

3.4.5 Restricted usage of variables

As discussed in Chapter 2, analytical values obtained for solvents have to be employed carefully: this could be a reason why a complete overlap was observed between intervariability and intravariability.

Nevertheless, a study in 2008 conducted by Dujourdy et al. [207] found solvents provided a high discrimination power. Solvents can thus be used as a suitable profile when it is possible to select uncorrelated variables, which was not the case with this dataset as all solvents had to be included in the profile, even if totally absent. The same conclusion can be drawn from impurities' profile.

Moreover, Dujourdy et al. [207] demonstrated that matrix effects occur when cutting agents are added. Indeed, depending on the diluents or adulterants present in specimens, the solvents' composition can vary after analytical analysis. It was for instance demonstrated that if cocaine was cut with more than 75 % of lidocaine, the linking methodology could not be applied on the obtained solvents' profile. In this dataset, specimens were mainly cut with PTHIT (see Chapter 2); it would be interesting to investigate if this adulterant affects the resulting solvents' profile depending on the adulterant's concentration, and if so how this would impact on the correlations between specimens.

Furthermore, a recent study conducted by Malette et al. [166] revealed that artefacts occur depending on the solvents employed during the manufacturing process. Indeed, by-products are often formed during the manufacturing process, indicating the presence of a specific solvent. However they might also hide the presence of other ones. These by-products are currently not targeted in NMI's analyses, and including them in the final profile of cocaine specimens would be valuable information to know the 'real' presence of each solvent and assess if this profile may in the end be of interest to build a statistical model to link seizures. The time of producing results would not be extended as it would on the contrary allow for a first triage of relevant variables to get quick discrimination between specimens.

Variables identified as not being present in cocaine or heroin specimens, such as some solvents and impurities should not be analysed, so the time of analysis would be reduced. Indeed, less targeted compounds potentially allow for:

- Faster detection: analytical instruments require a long time to detect these compounds by HS-GC-FID or GC-MS, for instance the solvent mesitylene which was absent from the profile is detected after 30 minutes (retention time from study Dujourdy et al. [207]);
- Faster interpretation of analytical results, because less values have to be considered;
- Less compounds to be considered to obtain percentage values, thus reducing error deviations from the normalisation step which is done at NMI using the internal standard;
- Interpretation of the profile facilitated when taking into account all values at once (like in this study).

3.4.6 Complementary source of information

Seizures were considered as linked based on circumstantial information (i.e. same PROMIS number), but chemical profiles were different especially with cocaine seizures. Indeed, for cocaine seizures linked with a same PROMIS number, correlations higher than the decided threshold (i.e. 81) were rarely obtained. It implies that those cocaine seizures linked with a same PROMIS number were probably coming from different cocaine products. On the contrary, more than 88 % of heroin seizures from the selected PROMIS numbers were higher than the decided threshold (i.e. 96) and would be considered as linked.

As a result, chemical profiles seem sufficient to convey discriminative information with heroin, but the broad spectrum of correlations obtained with cocaine proves that multiple types of information need to be assessed together for forensic intelligence purpose, as chemical profiles do not depict the whole picture.

Circumstantial information obtained from seizures should be gathered and employed similarly to chemical profiles, as new series might be detected based on transport methods of drugs for instance, or concealment methods, etc.

For each PROMIS (case) number, all seizures occurred within a period from 1 day to 7 months maximum (for cocaine). Therefore, even within less than seven months, linked seizures may contain different products (different chemical profiles), which tends to confirm the dynamic trafficking of cocaine, as already mentioned in the literature [10, 85]. Studies in the United States have established that cocaine is generally consumed the same year it is produced, and in average 24.6 months elapsed between the growth of the coca plant and the cocaine seizure [208], which may explain different chemical profiles through time. Moreover, it has been demonstrated that cocaine or heroin seizures are a mixture of multiple harvests accumulated together [208]. Different steps of production may not be done at the same production sites, thus different batches from the previous production step may be combined at the next production step [95].

3.4.7 Future directions

Physical properties should be investigated (which was not possible as the information was not accessible and fell out of the scope of this study) in order to gain more knowledge about cocaine and heroin markets and origin of seizures. Physical characteristics have proven their usefulness with MDMA tablets [33, 76, 121, 209, 210]. The benefit of recording physical information in a systematic manner with standardised procedure, such as standardised photos, colour, logo and shape, would allow using the information similarly to chemical profiles of seizures. The linking of specimens would be more straightforward as it would be based on binary values (i.e. same colour yes or no). Moreover, it could be used jointly with chemical characteristics and add knowledge about cocaine and heroin trafficking.

A comparison with chemical profiles of cocaine and heroin street seizures would be valuable information, as variables from border seizures considered in this study might not be the ones of interest at a different level of the market if analysing street seizures. It was already suggested in Chapter 2 to investigate purity and cutting agents of street seizures. Analysing their chemical profile with the purpose of building a statistical model similarly to this study would allow a better picture of links between seizures based on their chemical profile. For instance questions such as 'do chemical profiles of street seizure allow linking seizures? If so, is it more discriminative with lower risks of wrong classification than with border seizures?' could be investigated.

Comparing chemical profiles of border and street seizures would determine what information could be retrieved at different levels of the distribution network versus what information is specific only to border seizures or street seizures.

Investigating chemical profiles of street seizures similarly to this study would also bring knowledge to optimise the analytical process (i.e. what variables and what profiles of street seizures should be prioritised), and how analytical methods could be harmonised so that chemical profiles of borders and street seizures would be comparable.

Future work using the same cocaine and heroin datasets could focus on clustering methods such as PCA and HCA, which could be used to obtain chemical classes. Those statistical

methods are popular and have proven to be robust on certain types of datasets [188, 211]. They are not the recommended methodology to follow when data are obtained from different analytical techniques and combined together, as it will be difficult to obtain the similarity between two specimens [35]. However, combining clustering methods to the intra-inter methodology followed in this study could increase the confidence in attributing a specimen to a specific chemical class [35]. Both methods are complementary, and the evolution of obtained chemical classes over time and per geographic location may be obtained using clustering methods, reinforcing the awareness of cocaine and heroin market. In this respect, Broseus et al. [97] investigated temporal and spatial outcomes based on obtained chemical classes, highlighting networks and identifying discrepancies between before and after disruption of some networks. This is precious information for law enforcement agencies that could be obtained if the models built in chapter 3 were further developed and used routinely.

3.5 Conclusions

In this study, statistical models were developed to link specimens from cocaine and heroin border seizures. Combining multiple compounds contain complementary information rather than looked at individually provided better discrimination, as already established twenty years ago by Casale et al. [178]. The systematic use of such models implemented automatically into the database seems realisable with few resources and time constraints. Moreover, few FP and FN were obtained, ensuring the robustness of the method as well as the results. More specifically, the two profiles to be prioritised are alkaloids for cocaine specimens (including truxillines) and impurities for heroin specimens, as summarised in Table 10:

Table 10: Summary of outcomes optimised in this study

	Cocaine	Heroin
Profile	Alkaloids including truxillines	Impurities
Pre-treatment	Sqrt and LT10 for truxillines	Sqrt
Correlation method	Pearson	Cosine
AUC	0.9815	0.9942
Threshold (correlation)	81	96
TP / FP	97.55 % / 9.36 %	97.06 % / 4.82 %
TN / FN	90.64 % / 2.45 %	95.18 % / 2.94 %

Regarding cocaine specimens, both alkaloids and truxillines chemical values were obtained using a GC-MS. Focusing on those variables would save time, resources and money, as only

one instrument would obtain the most useful information. Other profiles are also interesting in order to determine the origin of the drug and manufacturing procedure: the full profile may be obtained later on for tactical intelligence.

Using multiple types of information and combining them to chemical characteristics, such as circumstantial information and physical characteristics, may allow a more holistic overview of cocaine and heroin trafficking.

Chapter 4:
***Forensic intelligence through a
novel approach to detect drugs
via screening of passport's
surfaces***

Chapter 4: Forensic intelligence through a novel approach to detect drugs via screening of passport's surfaces

4.1 Introduction

As seen in Chapter 2 and Chapter 3, valuable information can be obtained from chemical analyses of border seizures to gain a better understanding of illegal drug trafficking. Gathering various points of view may provide a more holistic vision of the illegal drug trafficking phenomenon. This chapter is a proof of concept study examining the feasibility of implementing a rapid screening tool to gain information on substances entering the country at borders from a novel angle.

4.1.1 Field instruments and current uses

Nowadays, increased expectations in productivity and immediate results are driving police and law enforcement agencies to be more proactive and providing results in a timely fashion [5, 18]. Sensitive laboratory techniques¹⁵ continue to be developed to obtain high-quality (with high accuracy) results from a variety of traces [2, 126, 213]. Nevertheless, such laboratory techniques can be time-consuming due to complex sequential laboratory analyses that may sometimes include chemical extraction [214]. They are designed for well-trained and highly specialised laboratory technicians and not always suited to the ability or knowledge of field investigators [215]. Furthermore, the destructive nature of these analyses often prevents any subsequent analysis [216]. To address this challenge, field-testing instruments or rapid techniques are deployed directly at crime scenes, which complement laboratory techniques [117]. Such new technologies are expected to be non-destructive, requiring no specialised knowledge, with little to no specimen preparation, providing discriminative results, with a miniaturised portable instrument directly usable on site to analyse traces inexpensively and in a timely fashion. An alternative to bring laboratories to the scene has been developed when portable instruments cannot be deployed with mobile laboratories directly deployable in the field, such as the Mobilab [217]. These self-contained laboratories, comprising chromatographic instruments and screening tests, triage specimens at the scene and ultimately reduce workload of main laboratories while providing rapid results for the investigation [217].

Law enforcement agencies, notably customs at airports, routinely employ field deployable technologies on various types of traces such as drug residues and other controlled substances

¹⁵ They include - but are not limited to- Gas and Liquid Chromatography Mass Spectrometry (respectively GC-MS and LC-MS), Inductively Coupled Plasma Mass Spectrometry (ICPMS), or Isotopic Ratio Mass Spectrometry (IRMS) [80, 212].

for their detection [53, 218, 219], as well as to obtain chemical information from physical specimens [220]. Each of these techniques has specific advantages and disadvantages, which may influence the choice of the instrument employed.

Table 11 provides a brief review of some of the commonly used instruments by law enforcement agencies for the detection of illicit drugs¹⁶.

¹⁶ Laboratory based equipment is reviewed in this table as a comparison to other instruments used by law enforcement agencies.

Table 11: Summary of techniques employed for illicit drugs detection

Technique	Advantage	Disadvantage	References
Attenuated Total Reflection Fourier Transform Infrared (ATR-FTIR portable)	<ul style="list-style-type: none"> Diamond crystals chemically inert to corrosive solvents and strong acids used to manufacture drugs; Possible to analyse mixtures without physical separation; Non-destructive; Rapid analysis; Small amounts of the sample can be used (mg) 	<ul style="list-style-type: none"> Not designed for non-technical users; Difficult to be deployed in the field because the instrument requires controlled humidity environment; Problem with heterogeneous solid specimens, the sampling area may not allow to analyse the whole sample; Difficult to analyse aqueous solutions due to strong interfering water absorption bands 	[148, 211, 215, 216, 218, 221-223]
Colourimetric tests	<ul style="list-style-type: none"> Rapid (fast analysis and result); Simple (designed for non-technical users, no sample preparation required, straightforward interpretation of results); Easily transportable and usable on site 	<ul style="list-style-type: none"> False positives and negatives frequent due to a lack of specificity and sensitivity to individual compounds; Not possible to detect mixtures; Often employ hazardous substances; Macroscopic amounts of material required (not suitable for residues); Reagents often required on site (possible contamination, use of safety equipment required for manipulation) 	[67, 163, 224-227]

Ion Mobility Spectrometry (IMS)	<ul style="list-style-type: none"> • Low detection limits (ng); • Fast analysis (few seconds); • No sample preparation; • Designed for non-technical users (simple to use and operate); • Less expensive to purchase than other trace detection technologies; • Swabbing of the targeted surface, allowing to work in a safe environment as no direct contact required 	<ul style="list-style-type: none"> • Difficult to analyse mixtures (ions conflicts); • False positives frequent due to matrix effects and background contamination; • Instruments relatively easy to saturate with high amount of sample; • Reduced specificity due to chemicals interfering in samples (ions from different compounds with similar drift times) 	[126, 225, 227-234]
Lab-on-chip	<ul style="list-style-type: none"> • Rapid (providing timely information to investigators); • Low cost; • Portable on-site (miniaturised); • Simple to use (no specific skills needed); • Sensitive to target compounds 	<ul style="list-style-type: none"> • Partial destruction of the sample; • Reagent consumption to be used on site; • Lack of specificity 	[67, 226, 235, 236]
Raman (portable)	<ul style="list-style-type: none"> • Non-destructive analysis; • Field-deployable; • Non-intrusive (compound can be analysed even if contained in packaging or in glass bottles); • Minimal sample preparation; • Possible to analyse aqueous material 	<ul style="list-style-type: none"> • Small sampling area which may not analyse the whole sample, problematic for solid heterogeneous material; • Difficult to analyse complex mixtures; • Not designed for non-technical users 	[148, 214, 218, 219, 237]

Laboratory based technique	<ul style="list-style-type: none"> • Mixtures can be separated into individual components for detection; • High sensitivity and selectivity; • Reliable; • Low detection limits (pg); • All physical forms of material can be analysed (solid, liquid, vapour); • Minimal amount of sample required for analysis (mg) 	<ul style="list-style-type: none"> • Analysis requires partial or complete destruction of the sample; • Extensive sample preparation; • Increased analysis time; • Specific extraction methods required; • Technique limited with the solubility of target compounds; • Samples sent for analysis in laboratories lengthen the investigative process before identification; • Laboratory infrastructure required; • Consume lots of chemicals (not environmental friendly and expensive); • Highly skilled operator required; • Expensive to purchase and maintain 	[126, 163, 215, 225, 228, 236, 238]
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It can be argued that the potential of these field technologies is not fully exploited as their end use is essentially for identification through random screenings (customs) or individual cases [67]. The potential added value and integration with other dimensions of law enforcement remains unclear [6, 50]. It is suggested that using these instruments beyond the traditional case-to-case approach is where the technology could be orientated, notably to gain further knowledge of criminal phenomena.

Previous studies (see next section) involving the detection of drug residues on banknotes have demonstrated the potential of recovering minute amounts from surfaces. By exploring these studies, a suitable methodology for passports can be developed and is presented below as further illustration.

4.1.2 Banknotes studies

The extent of the drug problem can be estimated through different aspects, such as the consumption within a defined area, the number of seizures, etc. Banknotes associated with illegal drug trafficking can be targeted to estimate the prevalence of drug consumption in a population, as the sale of illicit substances is often associated with the exchange of a significant amount of money. As a result, contamination of currency with the substance may occur, for instance due to business usage (drugs are often traded against cash) [239, 240].

Studies have been performed to determine the quantity of illicit substance retrieved on the surfaces of banknotes in order to distinguish the general population of banknotes from the ones contaminated with illicit substances, as this is often a question raised in Court [100, 241-243], due to its *“potential evidentiary value in the prosecution of drug-related crimes”* [244]. Establishing variations in the quantity of illicit substances retrieved on banknotes would provide useful background information to assess such traces to pursue a search warrant, or seizure, or arrest individuals in possession of the contaminated banknotes [245, 246].

These studies used sensitive techniques as well as field instruments for the detection of minute amount of illicit substances. The results revealed that banknotes worldwide are contaminated with minute amounts of illegal substances, mainly cocaine (ranging from 1 ng to 10 µg) [244, 247-249], especially in the United States and in Europe, whether it is associated with money laundering or general circulation [241, 245]. As a result, a significant number of countries have introduced laws that permit the seizure of *“dirty money”* [250].

It has however been demonstrated that powder drugs, such as cocaine, are highly volatile and have the potential to contaminate any type of support: for instance, it has been retrieved on external parts of hair from random individuals [251]. Therefore, these studies indicate that cocaine is ubiquitous and the significance of its detection on banknotes has limited value. Furthermore, it has to be kept in mind that even if the presence of controlled substances is detected on banknotes, it does not automatically imply a direct link with the use or trafficking of that compound [252]. Indeed, it is fair to assume that once a banknote is contaminated, it will remain as such for a long time, possibly for its duration of circulation and it might contaminate other banknotes in the general circulation by direct contact [240, 246, 252].

Consequently, as demonstrated above, using qualitative information obtained from field instruments is not achievable if focusing on banknotes to gain additional knowledge about drug trafficking.

However, the analysis of substrates for illicit drugs for intelligence purposes has potential assuming suitable substrates can be identified. Border security agencies are currently focusing resources to look for drugs based on circumstantial information and random checks. If searches were orientated at customs based on 'first screening knowledge', seizure numbers may increase, and they may improve the understanding of the market. Moreover, trafficking routes' awareness may be improved. In this case, the 'first screening knowledge' that is being proposed is to use the information from screening of passports at borders to detect illicit substances for monitoring illicit drug trafficking, using the approach exploited with banknotes. The concept is presented in the next section.

4.1.3 Screening tools for the detection of illicit drugs on identity documents as a new approach

Unlike banknotes, identity documents such as passports are not in heavy circulation; hence the chances of background contamination are lower. It is fair to assume that the majority of contact expected with an identity document would primarily be coming from its owner.

From analytical and materials standpoints, a parallel can be established between banknotes and passports for the detection of small amounts of illicit drugs. Minute amounts of substances may be transferred onto passports' surface with similar physicochemical properties as with banknotes. If a person, going through customs, is involved in drug trafficking or is a consumer, they will most likely be in contact with the illegal substance. Thus, they may contaminate their passport. This contamination could occur with diverse scenarios, such as:

- Direct contact between contaminated fingers and passport's surface;
- Direct contact between a contaminated support (i.e. tabletop) and passport's surface.

This is why employing identity documents such as passports would be considered as a valuable alternative to banknotes.

This approach could be used to quickly detect patterns in drug importations, without consuming costly resources. From this perspective, portable instruments could be employed routinely for the systematic screening of passports directly on site. Indeed, they would allow the monitoring of illicit substances in a timely fashion, and results would be quickly assessed in a binary manner (i.e. substance X detected or not). The prevalence of drug types and their frequency of detection would inform on consumption trends as well as importation / exportation trends. This procedure would not be intrusive for passport's carriers, as only the external surface would be screened.

This would allow a broader picture to be obtained by focusing on all collected results rather than targeting specific individuals or persons of interest. This is based on the assumption that passports are highly linked to their holder and thus their travel, enabling a possible mapping of the trafficking routes of repeatedly detected substances at customs. As a result, a better perception of the type of illicit substances available in the country (whether consumed, sold or trafficked) would be obtained.

Furthermore, passports (or alternatively accepted identity documents) seem to be a suitable support to be analysed, as they are required to enter and exit a country. Screening instruments are already implemented at airports for the detection of explosives. The screening of passports could be implemented similarly, without recording any personal information from passengers, except their boarding location / country.

One other possibility could be to have an automatic and systematic screening of the passport' surface for the detection of illicit substances (including a cleaning process thus preventing from cross contaminations) while being at automated passport controls (i.e. 'smart gates'), which are increasingly deployed nowadays at customs in airports. As illustration of the proposed approach, a hypothetical scenario is outlined in section 4.1.4.

4.1.4 Hypothetical case study

A person working in a clandestine laboratory is in contact with illegal substance A. This person travels to the airport, thus holding their passport and consequently transferring particles of A onto their passport.

A rapid screening instrument is implemented at customs, and the passport's surface is screened for the detection of illegal substances (i.e. only for identification). No personal information from the passport is retained (thus no individualisation is performed from passports' information, but this could be added if required by the end users).

This process is repeated for each passenger going through customs (arrivals / departures). The fast result (i.e. illegal substances detected or not) along with their boarding location/country is stored in a database, as illustrated in Figure 30:



Figure 30: Database containing results after screening of passports

On each passport analysed, an illegal substance may be detected or not, and different substances may be identified. Links can then be highlighted between passports (automatic comparison process between all results stored in the database), as illustrated in Figure 31:

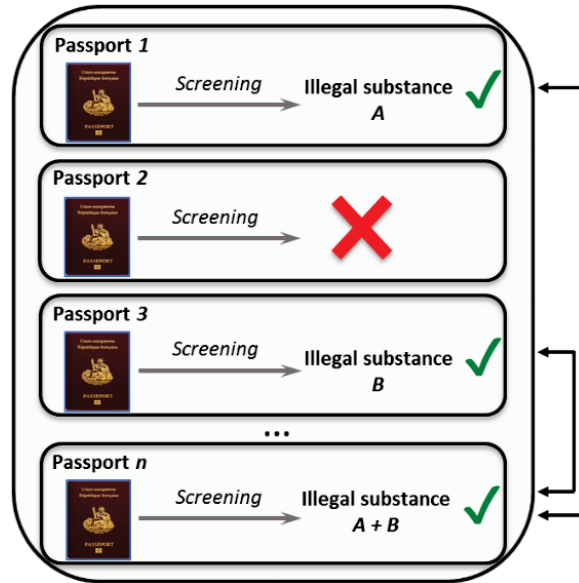


Figure 31: Links established from results in the database

A list of repeatedly detected substances may now be established based on detected links, from the most to the less frequently detected illicit substance, which will provide police with a better knowledge of major and minor illicit substances being trafficked present in the country. More pragmatically, this information may be used to prioritise the work of law enforcement, for instance focusing resource to minimise the importation of substance A into the country.

Furthermore, passengers going through customs are related to an origin or destination country. As a result, it may be possible to connect the illicit substance detected to a specific country, as illustrated in Figure 32:

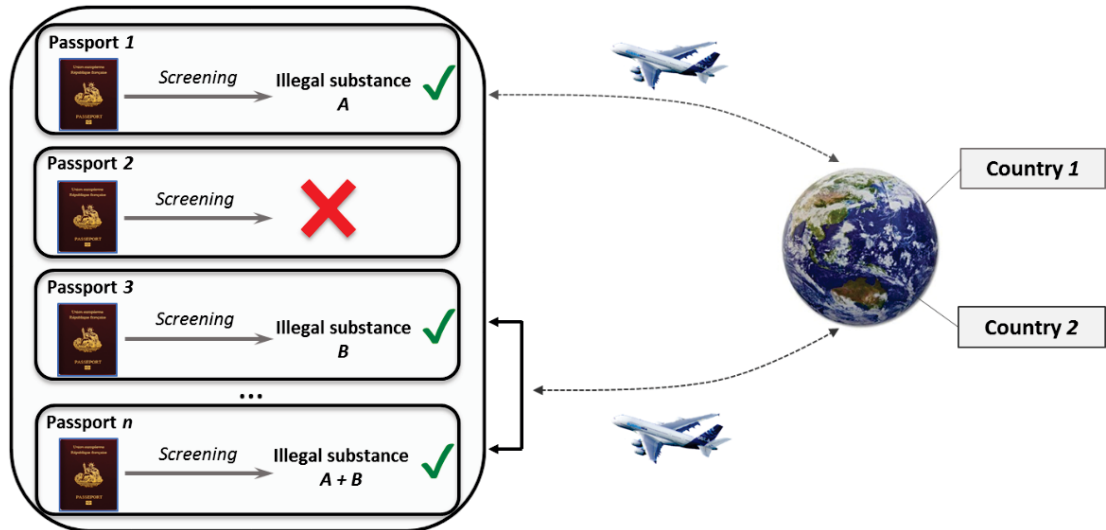


Figure 32: Added knowledge from established links

If *B* is regularly retrieved on the surface of passports from passengers arriving from Country 2, a pattern may be established: *B* may be illegally imported from Country 2. It gives powerful information to customs. Indeed, in addition to random checks, they may now focus on luggage arriving from Country 2 (for instance with detection dogs) based on previous knowledge. This information can also be provided to other authorities to contribute to their overall knowledge of the illicit drug situation, assess and review their policies and procedures.

An obvious limitation to the proposed approach is that not all individuals involved in a drug trafficking network may contaminate their passport, thus giving a negative result. Conversely, chances of environmental contaminations may occur, hence giving false positives. However, these apparent weaknesses are not critical as the approach does not aim at identifying and prosecuting individuals or individual cases. This approach implies screening surfaces of all passports from all individuals arriving or leaving a country during a certain period, which would represent a large dataset. Thus, even if false positives or false negatives may occur (like in any testing procedure), the overall trend (if any) would still be depicted.

4.1.5 Scope of this chapter

The aim of this chapter is to outline a new approach to obtain information for strategic and operational purposes, using rapid instruments already deployed at borders. More specifically, the feasibility to use rapid tools to screen passports for the detection of illicit substances is examined, to illustrate the possibility to monitor the trafficking of illicit drugs into a country.

An experimental design was built in this proof of concept by exploring different parameters that may influence the results. Different scenarios were performed simulating what could happen in real life. Experiments were based on transfer and retention capacities of both donor and surface of passports, including persistence and activity.

Transfer has been defined by Locard [253] and it depends on various parameters, such as the type of contact between the substance and the support, the support to which it is transferred onto, the physical properties of the substance itself, etc. For instance, the contamination of a banknote in direct contact with the substance (through sniffing for example) is an obvious transfer. Different types of transfer exist (primary transfer, secondary transfer, etc.), thus adding some complexity to interactions happening between object and support [1].

In addition, the activity has a broad meaning as it can refer to the activity of the support to which the substance was transferred onto or the activity of the substance itself. As a result, a substantial number of scenarios may be attributed to the activity parameter when it comes to illegal drug trafficking. The action of putting a contaminated banknote in the wallet where it could potentially contaminate other banknotes via secondary transfer highlights the activity concept.

Furthermore, persistence can refer to retention capacity of the support to which the substance was transferred onto, or the adherence capacity of the substance onto the support itself [251]. It is related to the time elapsed since the transfer and may be correlated to the activity. The time that would be required for the substance to be entirely removed from the passport's surface is a persistence scenario.

These three concepts are not solitary and will actually be combinatory resulting in more complex situations and challenges when it comes to the recovery and interpretation of residues of traces.

Different interconnected conditions potentially influencing results were investigated:

- Type of illicit substance;
- Choice of substrate;
- Primary and secondary transfer;
- Activity for a certain period after transfer;
- Persistence after transfer for a certain period of time;
- Inter-individuals variations.

Three different technologies currently used by law enforcement agencies were tested:

1. Portable ATR-FTIR is routinely employed to qualify illicit substances when performing seizures in clandestine laboratories [218], and also to identify major diluents and adulterants in cocaine and heroin specimens [179, 238, 254]. This instrument can be used for gross comparison of illicit drugs in their powder form [216] as it does not require physical separation or preliminary preparation before the analysis [211, 220]. The sampling area of the ATR instrument is quite large as it comprises the surface of the diamond, being 0.10 mm² [218], enabling to analyse a relatively high amount of specimen [218].
2. IMS technology and the concepts of this instrument date from the 1960's [231]. It has been developed for the determination of small quantities of organic compounds [233], and it is nowadays widely implemented for a routine use at customs in international airports [250, 255], for the detection of chemical weapons, explosives [256] and illicit

drugs [230, 255, 257]. It has been successfully used in drug counter operations by the U.S. Drug Enforcement Agency [258], the Canadian Revenue [259], and the U.K. Nottinghamshire Police [260]. In brief, specimens are collected on a Teflon swab, which is then heated to analyse vapours of residues ionised at atmospheric pressure. Resulting ions are accelerated and separated similarly to a time of flight mass spectrometer process. The drift times of separated ions are measured and they are proportional to their masses and sizes (i.e. longer drift time for a bigger ion) [231, 233, 255, 261, 262]. The instrument is set up with a database, comprising reference compounds of interest (i.e. narcotics) [232], no chemical knowledge is therefore needed to interpret the obtained qualitative results (i.e. substance X 'yes' or 'no' detected).

3. Atmospheric Pressure Chemical Ionisation coupled to tandem Ion Trap Mass Spectrometers (APCI-ITMS-MS) for screening analysis is a new technology developed by Hitachi® for the detection of controlled substances at customs. They provided the instrument DS-1100 N™, as illustrated in Figure 33. This instrument is currently deployed and routinely used at borders in Japan and Thailand. The technology is similar to IMS, with one additional MS, making it more selective than IMS. Indeed, the two consecutive MS allow differentiating between compounds based on precursor ion, which is the main ion (identical to the one detected with IMS), specific to a family of compounds; and product ions, coming from the precursor ion and identified with the second MS, which are specific to each compound. Reference substances are integrated in a database, and qualitative results are directly obtained if precursor and product ions are retrieved in the analysed specimen ('yes' or 'no' answer). Similarly to IMS, a threshold value is determined for each compound. A positive result implies that both precursor and product ions are detected above the threshold level.



Figure 33: Instrument DS-1100 N™ from Hitachi®

The method was optimised and the three technologies were compared and discussed to determine:

- The most appropriate instrument to be used for the purpose of this study;
- Essential criteria identified to detect remnants of drugs on the surface of passports;
- The feasibility to develop such a methodology for routine use at Customs.

4.2 Material and Methods

4.2.1 General procedure

The overall study aimed to reproduce realistic conditions through different scenarios of a passport put in contact with an illicit substance. A contact was established between the passport's surface and the substance in powder form, either using fingers or a plane surface (here a benchtop was employed). In order to mimic a realistic scenario, no additional specimen preparation was performed.

Prior to each experiment, surfaces (i.e. passport's and benchtop's surface) were wiped with a tissue (without any solvent to avoid degradation of the surface of the passport and to prevent potential adhesion which may affect the ability of the powder to remain on or be removed from the surface), and a blank swab was collected and analysed to ensure the absence of any background contamination, as summarised in Figure 34:

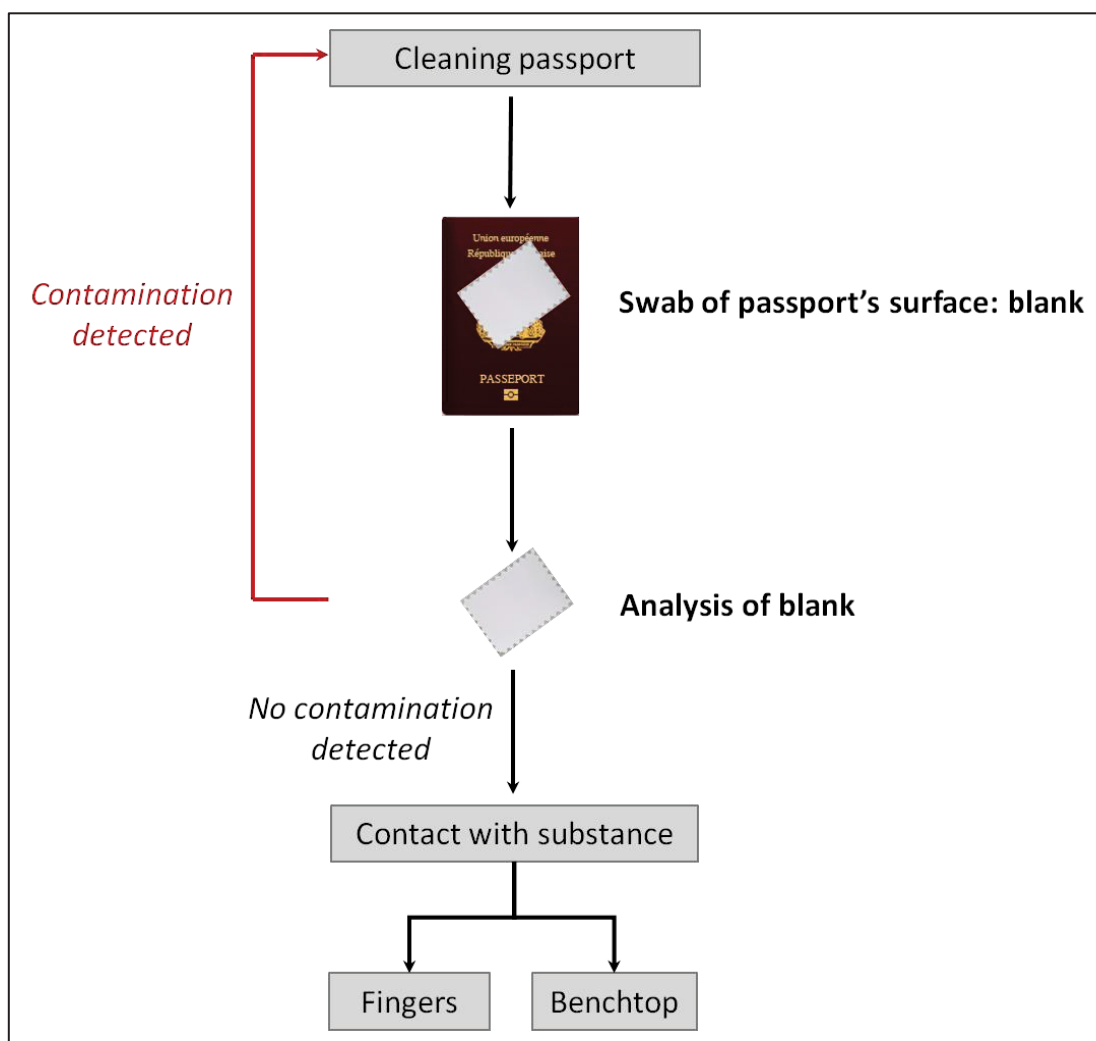


Figure 34: Sample preparation methodology

For a contact with fingers, two fingers were in contact with the compound for a short period of time (less than one second)¹⁷. In an effort to make it realistic, very minute amounts (less than 0.05 mg) were transferred onto the fingers. This amount usually corresponded to an amount that could be detected by instrumentation but not visible to the naked eye. The

¹⁷ Fingers employed were either the thumb, the index, or the middle finger of both hands, as these three fingers are defined as being the most solicited ones on both hands [263].

external surface of each passport was subsequently briefly touched with the two 'contaminated' fingers. The entire surface was then swabbed for analysis.

Different parameters and situations were tested, including activity, transfer and persistence, as summarised in Figure 35 (they are described later on in this section). Each experiment was conducted ten times.

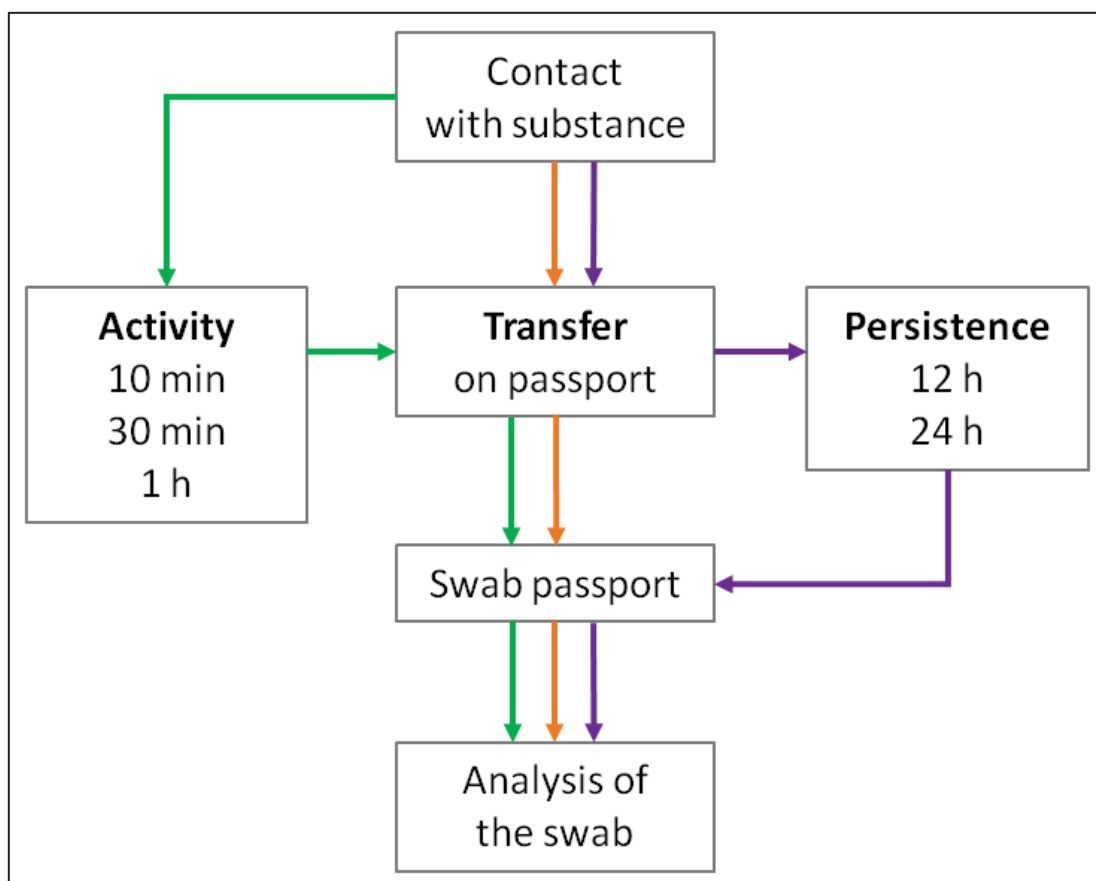


Figure 35: Methodology developed for analysis after transfer, activity and persistence experiments

4.2.2 Material: chemicals, surfaces and swabs

Experiments were performed using standards of pure cocaine HCl (purity 99.8 ± 2.0 %, standard number D757c), heroin base (purity 99.4 ± 2.0 %, standard number D752c), methamphetamine HCl (purity 99.8 ± 1.9 %, standard number D816g), and MDMA HCl (purity 97.0 ± 1.7 %, standard number D792d). The Australian Government National Measurement Institute provided all standards.

The external surface of expired passports from various countries (United States of America, Australia, Canada, France, Ireland, New Zealand, Philippines, Switzerland, and United Kingdom) was used as the deposition surface for the experiments.

The surface of a laminated benchtop was also used as a support. The substance was directly transferred on the benchtop and the passport was left in contact with the benchtop.

Australian banknotes of 5, 10 20 and 50 dollars were employed as surfaces during secondary transfer experiments (see next section).

Different types of swabs were trialled for ATR-FTIR analyses, including Teflon swabs (Nomex AR Mode from Smith Detection®, P/N 6821201-B), alcohol based swabs (Medi-Swab (Isopropyl) from BSN Medical®), sticky-tape swabs (adhesive tape from Officeworks®), nitrile swabs (from nitrile gloves, Bastion®), and cotton-tips (from commonly found store brands, i.e. Coles and Woolworth). All analyses were performed employing Teflon swabs (500-DT AE-ROW swabs, PN 6822254-A from Smith Detection®) for IMS analyses and cotton-based swabs (Bemcot M-1 wipes from Asahikasei®) for APCI-ITMS-MS analyses.

4.2.3 Method

The same methodology was followed with the three instruments employed and each experiment comprised three consecutive analyses (summarised in Figure 34):

1. A clean swab;
2. A blank swab;
3. A swab after contamination of passport's surface.

4.2.3.1 ATR-FTIR analysis

All analyses were carried out in transmittance mode using an ATR-FTIR model 630™ from Agilent® with diamond crystal. The transmission mode is recommended for qualitative analysis by Stuart [264]. Measurements were recorded at a resolution of 8 cm^{-1} from 4000 cm^{-1} to 650 cm^{-1} with 64 scans accumulated. This method was optimised based on the literature and adjusted with the material used for the experiments [148, 211, 218, 265].

Prior to the measurement of the swab containing the targeted substance, a clean swab was employed as the collected background, to be directly subtracted from the final spectrum in order to remove any interference due to the substrate.

The spectra were then treated using the OMNIC™ (Thermofisher Scientific) software. Results were first interpreted manually with visual comparison to the reference spectrum of the pure substance. Then, spectra of drug standards were analysed using an automated search in the library created for this purpose (containing the profiles of pure cocaine, pure heroin, pure methamphetamine, pure MDMA as well as the profile of the swab). The total duration of the analyses, including analysis of one blank followed by one 'contaminated' swab was around two minutes (approximately one minute to prepare each swab on the instrument and proceed to the acquisition of the spectrum).

4.2.3.2 IMS analysis

IMS technology was employed using IonScan 500 DT™ from Smiths Detection® (instrument provided by the AFP).

Prior to each sequence of analysis (once a day), a verification was performed to certify the accurate position of the internal calibrant. Once the verification had been carried out and was positive, analyses were performed. The time required for one analyses was approximately 10 seconds. The parameters used to conduct analyses are summarised in Table 12:

Table 12: Parameters employed for IMS analyses

Desorber temperature (°C)	245
Detection / tube temperature (°C)	260
Drift flow (cm³min⁻¹)	300
Sampling time (s)	8.00

After each positive analysis, a clear down of the instrument was performed by analysing a clean swab. This step was repeated until no substance was detected. If not, a clean cycle was conducted, consisting in cleaning the machine and especially the drift tube where compounds could potentially still be present.

4.2.3.3 APCI-ITMS-MS analysis

Analyses were performed with the DS-1100 N™ instrument from Hitachi® and using optimised parameters from Hitachi® (not communicated for confidential reason). If a positive analysis occurred, a cleaning process of the instrument was automatically started (around five minutes).

4.2.4 Experiments

All experiments described in this section were carried out with the three instruments, otherwise specified.

4.2.4.1 *Limit of detection experiment*

- ATR-FTIR

The quantity for which each of the four substances was detected with an automated search after ATR-FTIR analysis was assessed: a controlled amount of each substance was analysed in powder form, up to 0.20 mg. Samples were weighed with an analytical balance Mettler Toledo™ instrument, model XS105 Dual Range, to ensure the accuracy of the masses, ranging from 0.10 to 0.20 mg \pm 0.05 mg for the four drugs standards.

- DS-1100 N™

Limit of detection values of each standard was unknown with this instrument, contrary to IonScan 500 DT™ (as exposed in the technical manual of IonScan 500 DT™ and confirmed with Dussy's study [255]). Therefore, experiments were conducted, using solutions of standards in methanol (solvent chosen based on previous studies [87, 225, 226, 255]). A controlled amount of substance in solution was applied via pipette on the swab, which was subsequently analysed. The experiment was conducted as long as a positive detection occurred, until a negative result was obtained (thus indicating that signals of precursor and product ions were below the threshold value). The lowest values giving a positive identification for each standard was considered as the limit of detection of this specific compound for the day of the analysis. The experiment was reproduced over ten days in order to obtain inter-day variations as well as mean and standard deviation of each value.

4.2.4.2 *Transfer experiment*

As described in Figure 34 and Figure 35, different transfer experiments were conducted:

- Direct transfer;
- Secondary transfer from passport to passport;
- Secondary transfer from banknote to passport.

To determine the capacity of transfer of the four substances on the surface of the passports, a first set of experiments consisted in generating a contact between the substance and the external surface of the passport. This experiment was split into two sets: the first set was a contact produced with fingers, and the second set was a contact with a benchtop.

For the first set, a non-visible amount (< 0.05 mg) of each substance was applied on a pair of fingers. The two fingers were then briefly put in contact with the passport.

In the same manner, for the second set, a non-visible amount of one compound was placed on the surface of a benchtop, and passports were put in direct contact with the benchtop (no further pressure was applied on the passport once in contact with the benchtop, to simulate a real case scenario).

For both scenarios, a swab was performed directly after the contamination of the surface of the passport, and the analysis of this swab done immediately after swabbing.

Contrary to IonScan 500 DT™, it was possible to analyse multiple compounds at a time with the DS-1100 N™ due to the two consecutive MS identifications. A direct transfer with fingers was thus tested, with cocaine and heroin simultaneously transferred, as well as methamphetamine and MDMA.

A secondary transfer was also simulated by putting in contact a clean passport with another one already contaminated with direct transfer (either with fingers or benchtop). The second passport was subsequently swabbed and the swab analysed.

Moreover, as assumptions of this study were based on results obtained with banknotes (see section 4.1.2), experiments were conducted with DS-1100N™ using Australian banknotes of 5, 10 20 and 50 dollars, by putting a banknote in contact with a clean passport' surface and swabbing the passport for analysis (i.e. secondary transfer).

4.2.4.3 Activity experiment

To determine the capacity of retention of the substance on the surface of passports as well as on fingers, a third series of experiments was performed with each drug standard. A non-visible amount of each drug standard was applied on two fingers (less than 0.05 mg, see section 4.2.1), followed by an activity that lasted from 10 minutes up to 1 hour (such as typing on a keyboard or touching other surfaces, but excluding washing hands). The surface of the passport was then touched with fingers. A swab was performed after the contamination of the surface, and the analysis done immediately after swabbing.

4.2.4.4 Persistence experiment

In order to establish the retrieval of the drug standards after a certain amount of time prior to the analysis, another series of experiments was conducted, using the same procedure as for the transfer experiment. The passports were subsequently left either for 12 hours or 24 hours (two sets of experiments) on a benchtop prior swabbing and analysis.

4.2.4.5 Blind tests

In order to verify obtained results with the different experiments as well as the experimental methodology, blind tests were conducted in which the person analysing the passports had no knowledge of the illicit drug (if any) present on the surface of the passport. Twenty passports were employed for this experiment. On each passport, participants could either not contaminate the passport, or contaminate the passport with one or even multiple drug

standards (non-visible amount of substance applied on fingers). Analyses were subsequently performed and obtained results compared to what had been prepared by the participants. This experiment was only conducted with the DS-1100 N™ instrument.

Inter-individual variations were subsequently verified with three individuals reproducing the direct transfer experiments using fingers with the four drug standards.

4.2.5 Summary of the methodology followed in this study

Figure 36 hereafter summarises the methodology followed using the three instruments for this study:

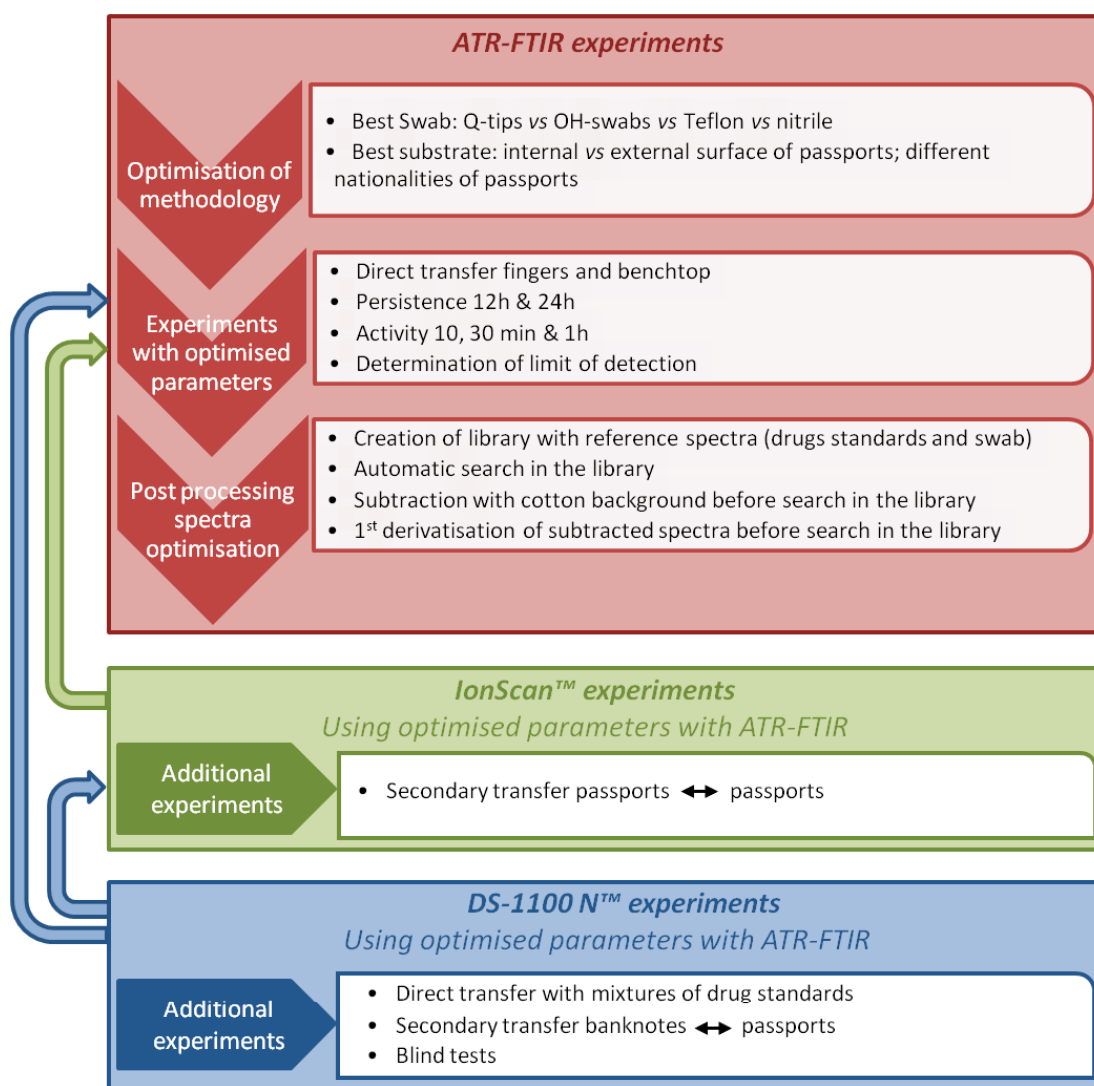


Figure 36: Methodology employed in this study

4.3 Results

4.3.1 ATR-FTIR

Swabs to be used for experiments are vital to the amount that could be detected and characterised, as identified in the literature [266, 267].

Through preliminary experiments, swabs and substrate were determined:

- Cotton-tips were chosen as appropriate swabs with ATR-FTIR due to the lack of interfering peaks with drugs standards' spectra compared to the other tested swabs (i.e. Teflon, alcohol based, sticky tape and nitrile swabs), as well as ease of access and low cost. Moreover, compounds retained by the cotton-tips were more likely to be completely analysed as the diameter of cotton-tips fits perfectly the diamond surface.
- Passports issued by different countries (and expected to have different compositions on their external surface) were considered as the replicate of one another. The profile of each drug standard was retrieved without any surface interference when applied in controlled amount on all passports.

The direct transfer specimens of all four drugs standards returned negative results with automated search in the library (comparison to reference spectra entered in the library). The returned 'match percentage' was very low (less than 30 %, when a good match score is considered if superior to 80 % with ATR-FTIR technology [221]) or the substance was identified as cotton (i.e. swab's material).

Controlled amounts of each substance were subsequently employed for direct transfer to determine the limit of detection of the methodology with automated search. Drug standards were not detected even up to 0.20 mg (which was a visible quantity), the cotton being the only detected profile. A derivatisation of the subtracted spectrum was also trialled with the software to improve results but without any success [264, 268].

4.3.2 IonScan 500 DT™

Obtained results of all experiments with the IMS instrument are presented in Figure 37. Each drug standard is represented for all experiments. As there were ten replicates or more per experiment and per drug standard, the cumulated number of positive detections is displayed as a percentage value each time (error rates will be presented in a separate Figure, see Figure 38).

Results were different for direct transfer experiments if the direct transfer occurred between the surface of passports and fingers or benchtop, notably for cocaine. It was mostly retrieved if a direct contact happened with fingers (90 %), but rarely detected for a direct transfer on the benchtop (20 %). A swab of the benchtop was performed after the direct transfer experiment to ensure it was contaminated with the targeted compound and thus confirming results (i.e. positive detection of the swab confirming a positive detection of direct transfer experiment with benchtop).

Substances were detected after secondary transfer: 20 % for cocaine, 50 % for heroin and 30 % for MDMA. Methamphetamine was never detected after secondary transfer.

Regarding the activity experiments, the recovery decreased with the time elapsed since the contact, as it was expected.

For persistence experiments, less recovery was observed after 12 hours compared to 24 hours.

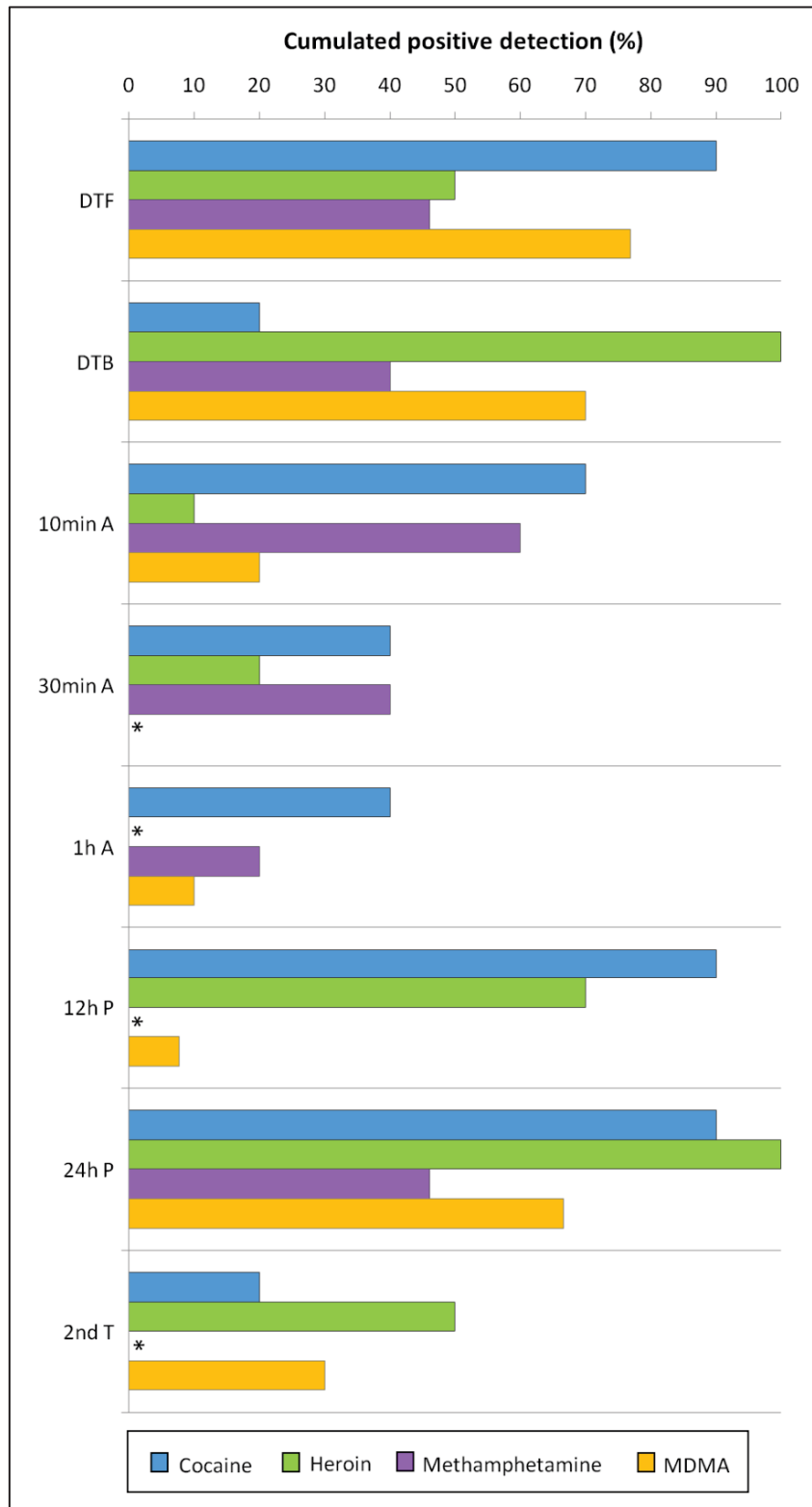


Figure 37: Results obtained with IonScan 500 DT™

*: No detection;

DTF: Direct Transfer with Fingers; DTB: Direct Transfer with Benchtop; 10min A: 10 minutes of Activity prior to transfer; 30min A: 30 minutes of Activity prior to transfer; 1h A: one hour of Activity prior to transfer; 12h P: 12 hours persistence after transfer; 24h P: 24h persistence after transfer; 2nd T: Secondary transfer

However, for many specimens, positive detections were actually false positives (see Figure 38). The percentage of false positives with heroin was even higher than the actual compound's detection. More specifically, tetrahydrocannabinol (THC) was detected in most experiment employing heroin, either in addition to or without the detection of heroin. Also, cocaine was often detected in addition to methamphetamine or MDMA.

False positives were detected in several types of situations:

- One or multiple compounds were detected in addition of a positive analysis of the targeted compound;
- One or multiple substances were detected even if the targeted compound was not detected;
- One or multiple substances were detected with clean swabs.

The comparison of false positives with 'raw results' obtained in Figure 37 demonstrated that results were not reliable enough to draw solid conclusions regarding percentage of recovery of each compound.

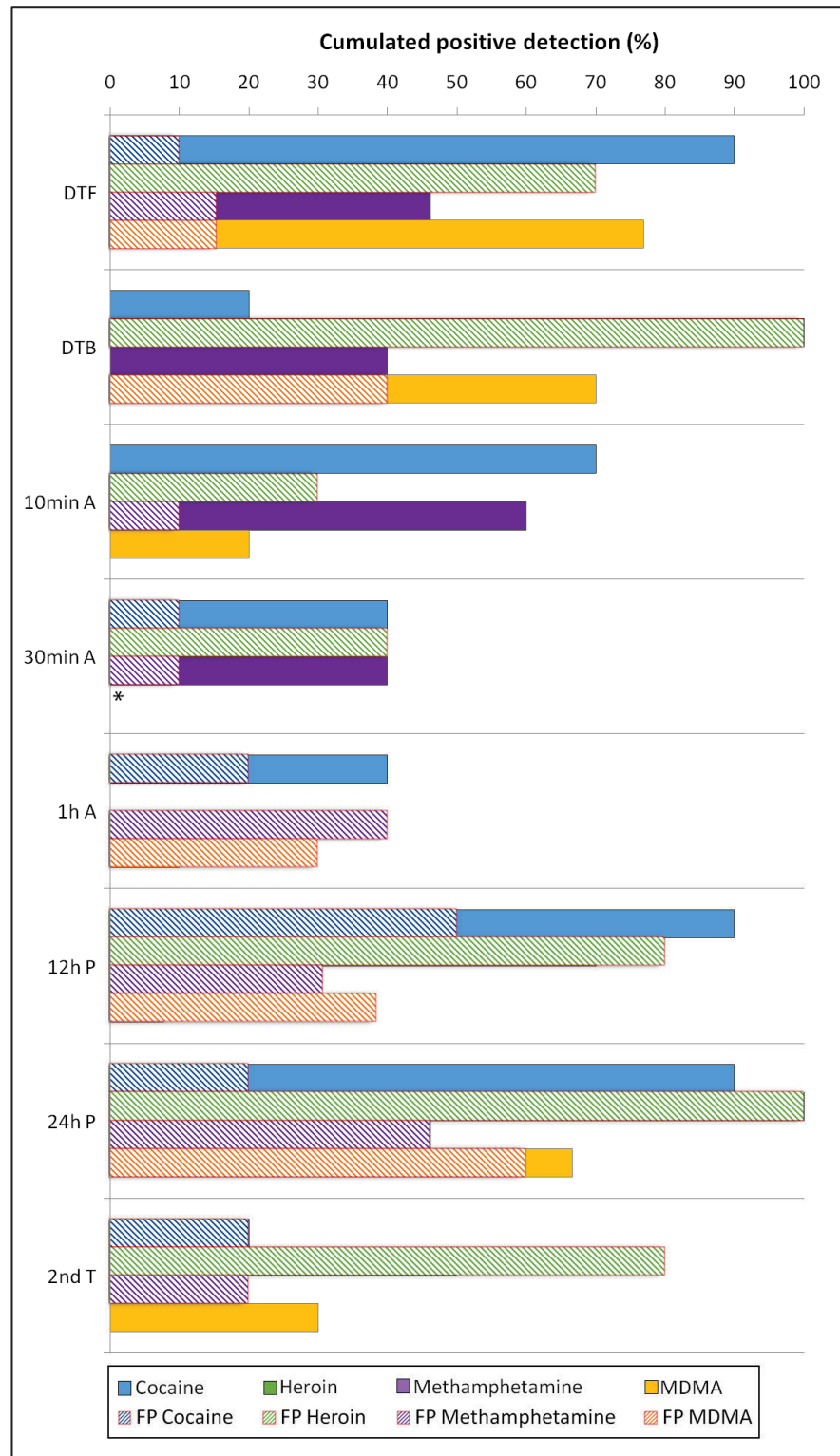


Figure 38: Results obtained with IonScan 500 DT™ including false positives

*: No detection;

FP: False Positives; DTF: Direct Transfer with Fingers; DTB: Direct Transfer with Benchtop; 10min A: 10 minutes of Activity prior to transfer; 30min A: 30 minutes of Activity prior to transfer; 1h A: one hour of Activity prior to transfer; 12h P: 12 hours persistence after transfer; 24h P: 24h persistence after transfer; 2nd T: Secondary transfer

4.3.3 DS-1100 N™

4.3.3.1 *Transfer, persistence and activity experiments*

Results from experiments conducted with the DS-1100 N™ instrument are presented in Figure 39. Contrary to results obtained with IonScan 500 DT™, false positives were not observed.

Positive detections for direct transfer experiments were predominantly observed, either using fingers or using a benchtop, individual compounds' variations aside.

A secondary transfer may allow detecting cocaine (60 %) or heroin (40 %), but methamphetamine and MDMA were rarely detected (10 %): residues were most likely transferred from passport to passport if it was cocaine or heroin than methamphetamine or MDMA.

Moreover, a secondary transfer between a banknote and a passport gave less than 4 % of positive detections (see Appendix 14).

A pattern was noticeable for the activity experiments: the more time elapsed, the less compounds were detected. None were retrieved after one hour of activity, and this could even be generalised to 30 minutes (only 10 % recovery for heroin, other compounds not detected).

Persistence experiments varied from compound to compound and positive detections were different if the passports were left for 12 hours or 24 hours, and no pattern could be deduced, especially as results were higher for heroin after 24 hours (70 %) than after 12 hours (50 %).

Results from multiple compounds analysis are illustrated in Figure 40 and Figure 41. The total of positive detections corresponds to analyses with at least one compound detected.

When cocaine and heroin were both transferred on passports, their detection was reduced (50 %) compared to each of them analysed individually (90 % - 100 % in Figure 39). Moreover, both of them were simultaneously detected in only 20 % of analyses. The reverse was observed with methamphetamine and MDMA as they were both detected in 80 % of all analyses, and the remaining 20 % corresponded to MDMA detected on its own.

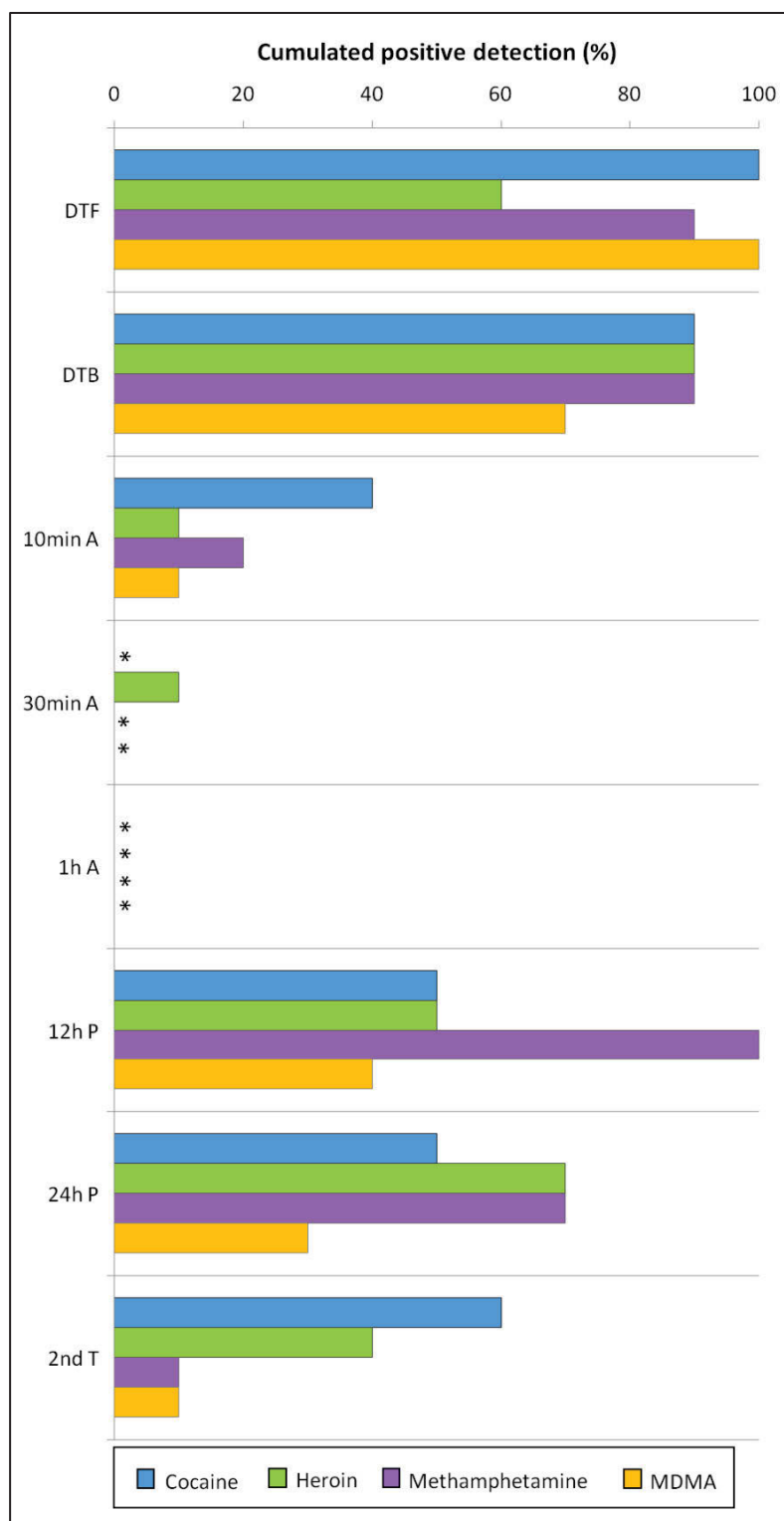


Figure 39: Results obtained with DS-1100 N™

*: No detection;

DTF: Direct Transfer with Fingers; DTB: Direct Transfer with Benchtop; 10min A: 10 minutes of Activity prior to transfer; 30min A: 30 minutes of Activity prior to transfer; 1h A: one hour of Activity prior to transfer; 12h P: 12 hours persistence after transfer; 24h P: 24h persistence after transfer; 2nd T: Secondary transfer

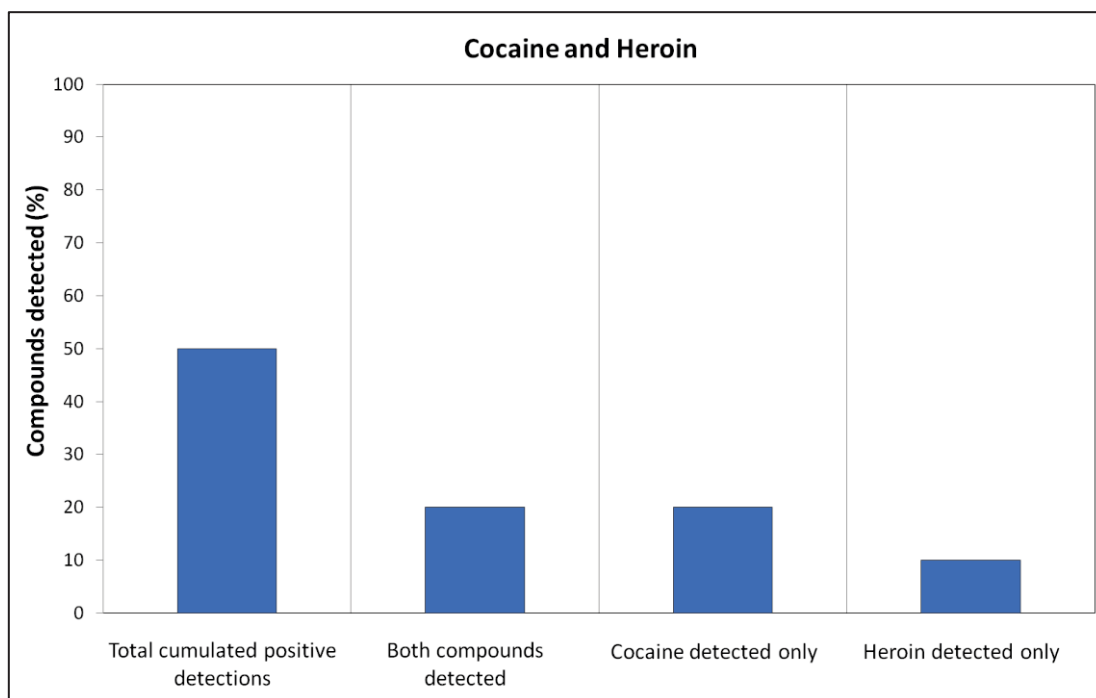


Figure 40: Results obtained for simultaneous transfer of cocaine and heroin with DS-1100 N™

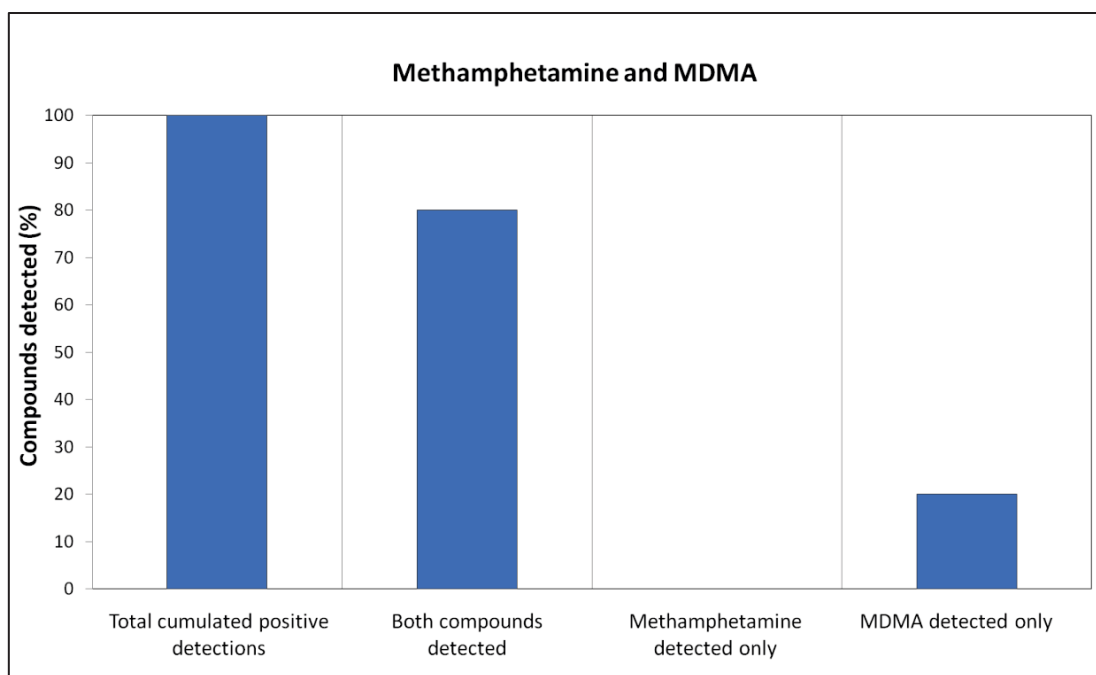


Figure 41: Results obtained for simultaneous transfer of methamphetamine and MDMA with DS-1100 N™

4.3.3.2 Blind tests

The detected compounds were correctly identified for 13 out of 20 analyses (see Table 13). Incorrect results occurred four times out of 20, corresponding to undetected result when a substance was actually deposited on the passport' surface, which was MDMA three times out of four. For three analyses out of 20, only one substance was detected when more than one drug was actually deposited on passports' surfaces. In this respect, when cocaine was present in addition to another substance, it was the only one detected.

Table 13: Results from blind tests

Passport's number	Result	Substance deposited
1	ND ¹⁸	Cocaine
2	MDMA	MDMA
3	ND	none
4	Cocaine	Heroin + cocaine
5	ND	MDMA
6	Heroin	Heroin
7	ND	none
8	Methamphetamine	Methamphetamine
9	ND	none
10	ND	none
11	Cocaine	MDMA + Cocaine
12	ND	MDMA
13	Cocaine ; Heroin	Cocaine + Heroin
14	Cocaine	Cocaine

¹⁸ ND: Not Detected

15	Heroin	Heroin
16	Cocaine	Cocaine
17	ND	MDMA
18	Methamphetamine	MDMA + Methamphetamine
19	ND	none
20	Methamphetamine	Methamphetamine

Subsequently, three individuals reproduced the direct transfer experiment using fingers (three replicates were performed per drug standards, see Appendix 15). Results were different from one individual to another and also depending on the drug standard employed, suggesting variations happening from one person to another when put in contact with remnants of drugs.

4.3.3.3 *Limit of detection*

The limit of detection (LOD) of the four drug standards over ten days is displayed in Figure 42. Variations occurred for all four substances. These variations were very high for heroin as the boxplot is spread from 0.08 µg to 0.36 µg. Conversely, results were the most reproducible with methamphetamine: this substance seemed stable regarding instruments parameters over time, with a tight boxplot representing values comprised between 0.025 µg and 0.057 µg.

Even if taking the highest quantity obtained for each compound, this instrument was sensitive enough to detect:

- More than 0.480 µg of cocaine;
- More than 0.360 µg of heroin;
- More than 0.057 µg of methamphetamine;

- More than 0.170 μg of MDMA.

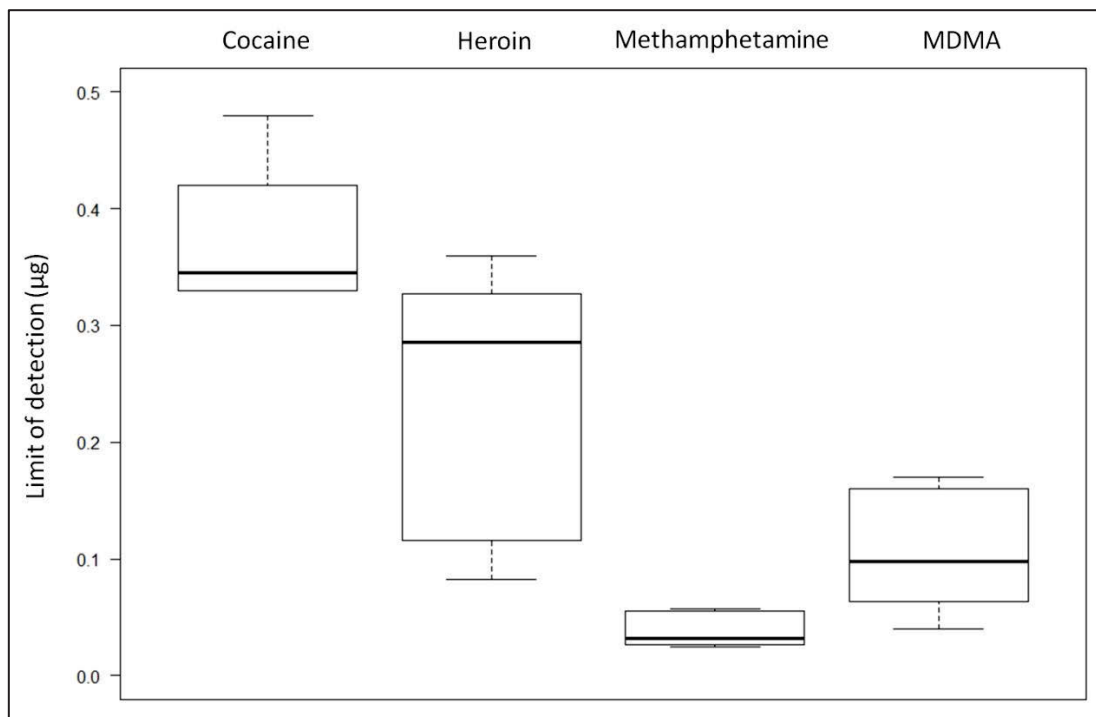


Figure 42: Limits of detection of the four drug standards obtained with DS-1100 N™

4.3.4 Summary of results

Table 14 hereafter summarises results obtained with each instrument employed: each technique presented advantages and disadvantages which will be developed and discussed in section 4.4.

Table 14: Summary of outcomes with comparison of techniques employed in this study

Technique	Advantages	Disadvantages
ATR-FTIR	<ul style="list-style-type: none"> • Rapid analysis (< 10 seconds) 	<ul style="list-style-type: none"> • No detection with automated search in the library; • Not sensitive enough (more than 0.20 mg required); • Not designed for non-technical users (preparation step and interpretation of results require chemical and FTIR knowledge)
IMS	<ul style="list-style-type: none"> • Rapid analysis (< 10 seconds); • Easy to use; • Fast interpretation of results (yes or no answer directly obtained); • Sensitive (detection below 0.05 mg); 	<ul style="list-style-type: none"> • Not specific: false positives observed with the four drug standards; • One compound at a time can be analysed and detected; • Difficult to clean after contamination (carry over of the instrument); • Results from experiments not reliable to draw conclusions

APCI-MS-MS	<ul style="list-style-type: none"> • Rapid analysis (< 10 seconds); • Easy to use; • Fast interpretation of results (yes or no answer directly obtained); • Sensitive: LOD 0.480 µg cocaine, 0.360 µg heroin, 0.057 µg methamphetamine, 0.170 µg MDMA; • Specific (secondary MS to target product ions); • Multiple compounds at a time can be analysed and detected; • Four drug standards detected after direct transfer using fingers and with benchtop; • Four drug standards detected half of the time after persistence experiment 	<ul style="list-style-type: none"> • Undetected samples; • LOD fluctuating: instrumental variations; • Four drug standards rarely detected after activity of 10 min to 30 min; • Four drug standards never detected after activity of 1 h; • Methamphetamine and MDMA rarely detected after secondary transfer
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4.4 Discussion

Three different instruments were selected in this proof of concept, each of them with their advantages and disadvantages which are further discussed hereafter.

4.4.1 ATR-FTIR instrument

ATR-FTIR appeared to be the less sensitive instrument of all three ones, and minute amounts of pure drugs could not be detected. Possible explanations can be advanced:

1. The physicochemical interactions happening when a cotton-tip was in contact with residues of powder drugs deposited on the surface of a passport did not favour the preservation of drugs on the cotton-tip;
2. The physicochemical interferences happening between a powder drugs and the cotton-tip did not allow ATR-FTIR detection of drugs.

Automated search with ATR-FTIR did not allow for any identification even with quantities already 2000 times higher than what is usually retrieved on surface of banknotes (i.e. 0.20 mg tested) [244, 247, 248].

A rapid screening method at customs requires the ease of interpretation of the results by a person that might not be familiar with the instrument itself and the chemical information it provides. An automated identification procedure has to be implemented to avoid any misunderstanding or erroneous interpretation. As this method intends to be developed for routine use by field officers, this specific ATR-FTIR instrument cannot be considered as a feasible instrument for automated detection and identification of compounds.

4.4.2 IonScan 500 DT™ instrument

IMS was found to be a suitable alternative to ATR-FTIR as automatic results were directly obtained. However, many false positives were observed, which may be explained due to:

- Carry over of the instrument. A blank after a positive analysis was considered as negative; however a subsequent blank would produce a positive result of the compound previously detected. The tendency of drugs to stay in the instrument after positive analyses (even if taking into account environmental background as a criterion to reduce false positives, which was observed by Forbes et al. [225]) was causing problems for further analysis, which lead to the systematic use of cleaning cycles after each analysis, increasing the time between each analysis. Bake out cycles were subsequently needed before the instrument could be considered as clean. Cocaine was the most problematic substance after positive analyses;
- Lack of selectivity. In the case of heroin, the drift time of the molecule was very close to the THC's one, explaining why THC was retrieved in addition of heroin in most analyses [255]. Moreover, it has been established that structurally similar substances may interfere with the actual drug, such as caffeine which may produce a positive alarm for cocaine due to a close chemical composition [261].

This instrument has proven good results for traces of explosives, as well as for drug identification [231, 232, 262]. However, in the case of this study and with remnants of drugs in powder forms, the results were not as reliable, the main issue being the lack of selectivity of individual compounds.

Solutions have been investigated in research studies to improve the selectivity of analyses, for instance if the IMS detector is coupled to a GC, thus allowing compounds to migrate and being separated before entering the IMS detector [228]. In this respect, parameters from the IMS detector may influence the recovery of cocaine, especially the temperature for heating the specimen, as demonstrated in the study by Sorribes-Soriano et al. [269]. However this

implies a longer time of analysis, which was not the primary goal of this study as willing to get results in a timely fashion. Parameters of the IonScan 500 DT™ were used as provided and were not optimised to reduce false positives, the overall aim being to find a technology already used routinely by law-enforcement agencies.

4.4.3 DS-1100 N™ instrument

APCI-ITMS-MS was expected to be more specific than IMS due to the presence of a secondary MS. This was confirmed through the different experiments, as false positives were rarely obtained, contrary to IMS experiments. Results from the study can therefore be interpreted with confidence if APCI-ITMS-MS was employed.

As experiments were performed with the aim of mimicking a real case scenario, many uncontrolled parameters may explain the detection or non-detection of substances. Positive results were obtained with the developed methodology employing remnants of drugs: even if particles were not visible on fingers (thus being present in a very small quantity), the instrument was sensitive enough to allow a positive identification.

The fewer positive detections observed after secondary transfer compared to direct transfer suggest that if passports' surfaces were contaminated with minute amounts of illicit substance via direct transfer, the contamination was more likely to be attributed to the passport itself, rather than transfer from a contaminated passport. As an example, if a tour was organised for tourists visiting a country, the guide may collect all passports from all passengers, as it is often the case. In this situation, this experiment demonstrated that if a passport was contaminated with illegal substance, it would have few chances to interfere and contaminate passports from other passengers. This outcome was the opposite of what was established with banknotes, as Keeble demonstrated that banknotes may still be positive after the seventh transfers after contamination between banknotes [252]. Nevertheless, the secondary transfer experiments carried out with banknotes indicated that less than 4 % of positive detections occurred, and the positive detection was always attributed to cocaine. It implies that if for instance a passport was left in a wallet where banknotes were present, there would be 4 % chance that a screening of the passport' surface would produce a positive result with cocaine.

Therefore, the combination of choices of substrate (i.e. passports' surfaces), instrumentation and methodology seemed promising to mitigate most of possible contamination through secondary transfer.

The positive or negative detections observed when performing persistence experiments can be attributed to the time factor. A negative result could be due to physicochemical properties of remnants of drugs: drug standards were in hydrochloric form and thus comprised water molecules. Leaving the passport for at least 12 hours allowed the particles to dry and thus their retention capacity on the swab might have been minimised [148, 205, 250]. Also, even if the substance was transferred onto passport, not enough particles deposited or too few of them retained on the swab might have led to negative results (which were uncontrolled parameters).

Results obtained when dealing with multiple compounds (Figure 40) suggest that cocaine, when present in minute amounts on a passport with other substances, was more likely to be detected and might have 'hidden' other substances, leading to false negatives [229].

Blind tests conveyed similar conclusions to the multiple compounds experiments, as cocaine was always detected when several ones were deposited on the surface of passports (3 times out of 3 experiments), contrary to other compounds. Moreover, MDMA was detected only once (out of six experiments, whether deposited alone or with other compounds).

Conflicting results regarding the detection of MDMA in the different experiments emphasise the need for caution when interpreting results with this compound. Indeed, it was detected in all analyses after direct transfer with fingers. It was thus expected to be detected 4 times out of 4 experiments with the blind tests, but it was detected only once (when alone). It may suggest that the quantity deposited on passports was lower than the LOD established for MDMA, i.e. less than 0.170 µg. Moreover, as different persons were implied in both experiments, it may suggest that variations happen especially with this compound regarding interactions between fingers and particles of MDMA. Many parameters were not controlled in experiments, and it would be expected to observe variations from one individual to another. Particles may not be retained in the same quantities on fingers, they may not be applied similarly on passports' surfaces and thus the amount remaining on passports' surfaces may vary. Those assumption were verified with the three individuals' experiment performed after the blind tests, as different varied from one individual to another and for all drug standards.

The limit of detection experiment (Figure 42) highlights that heroin was not a compound that could be reliably detected at low levels with this instrument. This experiment gave an overall

idea of the quantity required to obtain a positive analysis. Nevertheless, the quantity present on the surface of passports could not be inferred, neither did it gives information regarding the quantity of compound retained on the swab.

Particles were vaporised in the heating process, and the vaporisation of a compound is different if in liquid or in its solid state, adding limitations to perform limit of detection experiments, especially for heroin, as already mentioned by Ebejer et al. [270]. Moreover, this instrument was designed for identification and not for quantification. The technology did not allow for the development of a calibration curve to validate results previously established, as only a yes or no answer was provided. It corresponded to a ratio between the intensity of the signal detected and the established threshold, but this ratio could not be determined.

4.4.4 Future directions

This proof of concept highlighted the range of possibilities that are available nowadays to develop such research, as field deployable instruments are increasingly available to law enforcement and security agencies. In this case, the right balance needed to be found between false positives and false negatives to obtain results reliable enough to detect patterns. Finding an instrument fit for purpose may be possible if conducting further pilot studies, and moreover if thinking out of the box by using screening instrument to gather results which may allow to build patterns and trends, instead of the traditional case to case comparisons.

This last point emphasises the need for future work in a real environment. A methodology has been optimised and promising results have been obtained (especially with the DS-1100 N™ instrument). The next step would be field-testing: collecting results at customs in real time will allow to analyse the drug market, in particular the type of drugs the most as well as the less detected (thus drugs potentially the most and the less exported / imported). Potential patterns may also be observed regarding the period of the day or the year where positive results are the most detected. Geographic trends may also be determined if results are collected at different customs.

Furthermore, replicate experiments from this study using street drugs are required to better reflect the reality. Positive detections at customs may rarely correspond to contaminations from drug standards (i.e. drugs with around 100 % purity). Street seizures including different purity levels should be tested to investigate if various matrices and various concentrations may affect results, leading for example to non-detections or false positives. In this regard, testing different adulterants and diluents in matrices seems essential.

Besides, various scenarios involving persistence and activity parameters should be investigated to optimise the swabbing procedure of passports' surfaces. As an illustration, studying the influence of results if leaving the passport in a wallet for certain duration, or if putting the passport in a pocket will add knowledge about retention capacities of different types of drugs under variable conditions and may provide potential explanations for their detections (or no detections).

Nevertheless, it should be emphasised that this study was investigating a new approach to gain information using tools already deployed at customs, and the optimistic results may also be obtained with another technology. For instance, screening tests like colour tests are used only for identification. They could be used for intelligence purpose if employing the same methodology as the one presented in this chapter, that is to say having all results stored in a database, facilitating the extraction of all information at once, and the ease of interpretation of results as they are yes or no answers.

More generally, the number of data has grown, so the way of dealing with it has to be continuously adapted [5, 271]. Consequently, the dilemma of finding ways of integrating humans into a more and more technologically based society is increasing nowadays. This study is the perfect demonstration. The growing number of data, for instance in drug trafficking, may suggest a few interrogations: does an increase of seizures implies an increase of drug trafficking? Or is it because Police and Borders are more aware of illegal products arriving on the territory to seize them efficiently? Or is it due to prioritisation of work? No research has correctly established the causal relationship yet.

Implementing the proposed method at customs for routine use would answer most questions, as collected results and inferred patterns could be fused with already existing data from seizures (i.e. prevalence of types of drugs, known trafficking routes). Seizures are considered as representative of the criminal activity, nevertheless many uncertainties exist regarding their prevalence and trafficking (i.e. importation / exportation). Data is regularly obtained from a consumer point of view, with surveys about prevalence of drugs intake in certain countries or regions [159], but this type of data is dependent on the willingness of people to answer properly. The information from those surveys may be triangulated with street seizures, but a gap still exists for the 'in between' situation from custom seizures to street seizures (if all considered as representative of the whole criminal phenomenon). What is the actual prevalence of drugs entering and leaving a country and what are potential associated trafficking routes? The proof of concept exposed in this chapter would contribute to provide answers if further investigated.

Chapter 5:
General discussion and
conclusions

Chapter 5: General discussion and conclusions

The aim of this research was to **obtain a better knowledge of illegal drug trafficking** and ultimately suggest possible directions to authorities involved in the fight against illicit drugs. This chapter discusses the findings presented in Chapters 2 - 4 as a whole and their implication in the forensic intelligence field, and suggests possible future work.

5.1 Overview of the research's findings

The findings of Chapters 2-4 contributed to meet the overall aim of this research as they provided knowledge of the illegal drug trafficking phenomenon.

In Chapter 2, the study was exploratory. A general analysis of chemical profiles obtained from AFP border seizures of cocaine and heroin during 2008 and 2013 was conducted. Patterns were detected regarding purity of seizures, cutting agents (adulterants and diluents) and associated variations over time and geography (in Australia).

The median purity was higher than in other countries, even if a decrease of more than 10 % was observed for cocaine seizures over six years. Border seizures of cocaine and heroin appeared to have few added compounds. PTHIT and caffeine were the most detected adulterants in cocaine and heroin specimens, respectively. Only half of specimens comprised adulterants for both drugs. Few diluents were also detected in both drugs. A possible explanation could be that cutting agents may be added to the products once already

imported into the territory. An analysis of geographic location of drug seizures highlighted that NSW and more specifically Sydney was the hub for both drugs.

In Chapter 3, chemical profiles of cocaine and heroin border seizures (same dataset as in Chapter 2) were statistically investigated. The feasibility to build a discriminative model to establish if seizures could be linked based on their chemical profile was examined. Relevant data were extracted from the dataset for operational and strategic intelligence. The accuracy of chemical values from specimens' profiles was essential, especially to determine a threshold and associated error rates. The limitation of this study was to work with assumptions that could not be confirmed, as reference values did not exist (indeed 'reference seizures' do not exist in drug trafficking). Statistical parameters were optimised: the best results were obtained with alkaloids for cocaine and impurities for heroin. With these profiles, a threshold was determined with few associated risks of wrong classification (FP and FN rates). This statistical model provides a basis for law enforcement agencies interested in such an approach.

This study highlighted that most of information allowing a comparison of chemical profiles of border seizures could be obtained using solely GC-MS. It implies that less analytical instruments could be employed at first to gain in efficiency, which would save time and money. This outcome confirms the study from Morelato conducted on MA and MDMA Australian border seizures, in which prioritisation of work in order to be systematic in the analysis of data and being confident when exploiting data was proposed [10].

Chapter 4 explored a new approach to obtain trafficking flows knowledge at customs. The intended purpose was to examine if portable instruments currently deployed for identification and solely employed for case to case comparison could be used in a proactive and systematic manner to gain intelligence about criminal phenomena. An experimental design was developed to screen the surface of passports for the detection of remnants of drugs, testing various parameters such as transfer, activity and persistence. Results varied depending on the instrument employed (ATR-FTIR, IMS or APCI-MS-MS). The timely factor was the essential criterion in this study: a qualitative response (i.e. yes or no substance detected) was expected quickly.

FP and FN had to be considered for the accuracy of the results. The recovery of the drug was influenced by the technology and mostly the threshold established with the specific

instrument, thus impacting on FP and FN rates. Pros and cons were established with each instrument employed, but overall this study demonstrated that the current technologies can be employed to detect remnants of drugs on the surface of passports. The methodology should be further tested on real data obtained at customs.

Different use of chemical information from illegal substances was investigated in this research, which can be combined to get a more holistic view of the phenomenon. Investing time to look at findings from different points of view seems essential in a multi-disciplinary approach that is forensic intelligence. It will ultimately provide a more objective basis for strategic prioritization. Indeed, when examined as a whole, outcomes from the different studies conducted within this research could provide knowledge about:

- Geographical trends regarding drug seizures (see Chapter 2), allowing to be more efficient in the types of drugs to prioritise and the cities or regional locations to intensify controls;
- Manufacturing processes and their evolution in time (see Chapter 2), giving a better insight about trafficking routes and trafficking networks;
- Evolution of chemical profiles of border seizures over time (see Chapter 3), being correlated with manufacturing processes and thus reinforcing knowledge about trafficking networks;
- Chemical trends of border seizures over time (see Chapter 2 and Chapter 3), giving information of drugs' compositions especially regarding their purity and cutting agents as well as their evolution in time. This information is important to get an overview of the evolution of the drug market and specific controlled substances (such as adulterants). Trends over time regarding the increasing presence or absence of specific adulterants may lead to new controlled substances on the territory, knowledge about the networks' distribution, and measures regarding public health;

- Importation routes and packaging methods when drugs arrive in the country (see Chapter 2 and Chapter 4), which allows to refine border controls of luggage in addition to random checks;
- General overview of the identity and prevalence of drugs' residues being detected on passports at borders - but not seized yet - (see Chapter 4), which allows to target the drugs the most detected and intensify their control within the country.

5.2 Perspectives

From the previous section, many findings are valuable information for law enforcement agencies and would justify further research. The knowledge of the illegal drug trafficking phenomenon would benefit from subsequent investigations based on several suggestions and recommendations mentioned hereafter, always following a forensic intelligence approach.

First of all, trends established in Chapter 2 were based on data from 2008 to 2013. The investigation of similar data in more recent years may allow to detect new trends or changes (or not) compared with the already established ones. Findings from those statistics may reorientate police searches for border seizures.

Secondly, using the same approach as Chapter 3, physical data should be explored to gain more knowledge about cocaine and heroin markets and origin of seizures. Results obtained from discriminative models of physical data could be used jointly with results from the study conducted in Chapter 3 as they are a complementary source of information.

Moreover, information from circumstances of cases, such as *modus operandi* (for instance transport methods) could be explored in the same manner as physical data as they may be employed to link different seizures and obtain trends of trafficking methods.

Subsequent studies using the same cocaine and heroin datasets could focus on clustering methods to get the evolution of chemical classes over time and per geographic location. Comparing the findings to the ones obtained in Chapter 3 would broaden the overview of chemical data extracted from cocaine and heroin border seizures and the relevant chemical profiles to be used for operational and strategic intelligence.

Furthermore, additional studies should be performed using chemical data of street seizures, using the same approaches as in Chapter 2 and Chapter 3. Investigating if chemical profiles of street seizures could be employed to build a discriminative model would allow determining if

they are fit for purpose to link seizures. Besides, a more precise overview of the main locations of drug seizures may be obtained within the territory. It could be used to deploy more resource to arrest offenders or intensify controls in specific locations. Locations identified in established trends could be regions, or cities, or even airports or harbours where seizures are the most frequently happening for specific types of drugs.

Going further, investigating simultaneously street and border seizures' data would add knowledge about trafficking into the country versus when seized at borders. Indeed, the comparison between street seizures to border seizures' composition may add knowledge about trafficking commodities, such as added compounds within the territory if retrieved in street seizures, whereas absent from border seizures.

Besides, the comparison of chemical information employed with respectively border and street seizures would permit to identify what information is retrieved at different levels of the distribution network versus what information is specific only to border seizures or street seizures. This would bring awareness regarding harmonised analytical procedures to get comparable chemical profiles of borders and street seizures. Such a study would facilitate the comparison of Australian results with findings in other countries which are using a combination of border and street seizures.

A massive amount of information was provided for Chapter 2 and Chapter 3. In order to extract relevant information from the massive datasets even more efficiently than in its current state, one approach could be to investigate the feasibility to structure and organise the information from seizures in the memory to be employed either for tactical, operational or strategic intelligence purposes. This way, the information not exploited for prosecution, but that could be relevant for intelligence purpose, as it was demonstrated with Chapter 2 and Chapter 3, would be accessible appropriately if necessary. More concretely, chemical data from cocaine and heroin seizures contained in the database could be structured in a waterfall effect from the most employed information (i.e. purity) to the least employed information (i.e. for instance manufacturing processes including solvents information). The prioritization of information should be discussed in accordance with police needs. It would save time in comparing cases as only focusing on the purity of specimens at first, as this question is the most frequent one raised in court. Then if detailed chemical information was to be provided or searched afterwards, it could be accessed without confusion of relevant

data to be extracted. The feasibility of such an approach could be examined, starting from the datasets provided for this research.

Similarly to the previous suggestion, structure the memory to use the information for intelligence purpose from a surface level to a series level should be investigated as it would save time in establishing a preliminary triage of the type of information to be employed. Based on this research, information obtained from Chapter 2, Chapter 3 and Chapter 4 could be combined when needed or looked at separately depending on the wanted result of law enforcement agencies. Nevertheless, data overload may be experienced if storing all information in the database without totally controlling the input. This is one reason why looking at information at a surface level, which requires an easy interpretation of results, may be of interest, such as using a yes or no answer to detect patterns as proposed in Chapter 4.

Furthermore, experiments following the methodology developed in Chapter 4 using street drugs are suggested first, as results may be different from the drugs standards employed in the study. Different purity levels of street seizures should also be tested, as results may vary depending on concentrations. In this regard, testing different adulterants and diluents in matrices seems essential to have a better understanding of positive and negative detection depending on chemical composition of drugs. Moreover, additional scenarios involving persistence and activity parameters should be explored to get a better understanding of retention capacities of drugs on passports' surfaces and therefore optimising the swabbing procedure. Future work in the field at customs using the proposed methodology in Chapter 4 seems essential to get information about the most and the least detected drugs, as well as temporal and geographical patterns.

More broadly, the Intelligence Crime Management Model has been implemented in Australia by the NSW Police Force. This integrated model of policing permits to combine investigation, intelligence, and forensic reasoning [15], aiming at increasing exchange of information's flow to ultimately improve communication. It would be interesting to perform a similar study to this research using the Intelligence Crime Management Model. This way, it would be possible to determine how efficient it is to extract information and comparing findings and obtained trends from the ones obtained in this research (especially in Chapter 2 and Chapter 3).

Finally, to improve the situation regarding collaboration and information sharing, the National Institute of Forensic Science in Australia (NIFS) has recently released a “*forensic intelligence booklet*”, and training has happened in the different States and Territories based on its recommendations [272]. It should raise awareness, thus permitting to implement more efficient approaches to disrupt criminality in a timely fashion. It would be interesting to prospect in a couple of years how trainings affect practitioner’s visions of their tasks for forensic intelligence purpose before and after, and if it had an impact on roles procedures applied within the different law enforcement agencies. In the context of this research, if assuming it would be possible, prospect in a few years how the application of findings from each study affected results and police procedures regarding border seizures would be precious information to get a concrete ‘before / after’ picture of this criminal phenomenon on the Australian territory from an intelligence point of view.

5.3 Final words

This research, especially the study conducted on cocaine and heroin border seizures, builds upon the study conducted by Morelato on the analysis of MA and MDMA border seizures [10]. There are now two consecutive studies approaching forensic intelligence and more specifically forensic drug intelligence from a chemical point of view. Such research was essential to investigate the potential of chemical information and how to integrate it in a forensic intelligence framework. However, new studies in forensic drug intelligence should now focus on other source of information to get a broader view of the drug trafficking problem. Moreover, studies such as this research should be developed, that is to say adopting a multi-approach and trying to use complementary information.

Some questions may remain regarding the role that forensic intelligence has to play in a law-driven system. Indeed, procedures may not be optimised to obtain intelligence in priority. Besides, the appropriate integration of forensic intelligence in the investigative process may be challenging. Consequently, the efficiency of law-enforcement agencies may be negatively influenced for their primary goal, that is to say reducing crime rates. This is why the benefit of following a forensic intelligence approach and exploring ways to understand better the criminality has been one of the central points of this research.

As a closure, Professor Pierre Margot said in October 2015 when he was invited speaker at the UTS and interrogated on the question of what the big step after DNA will be - as it played a major part in the revolution that happened in the past decades in the forensic science world:

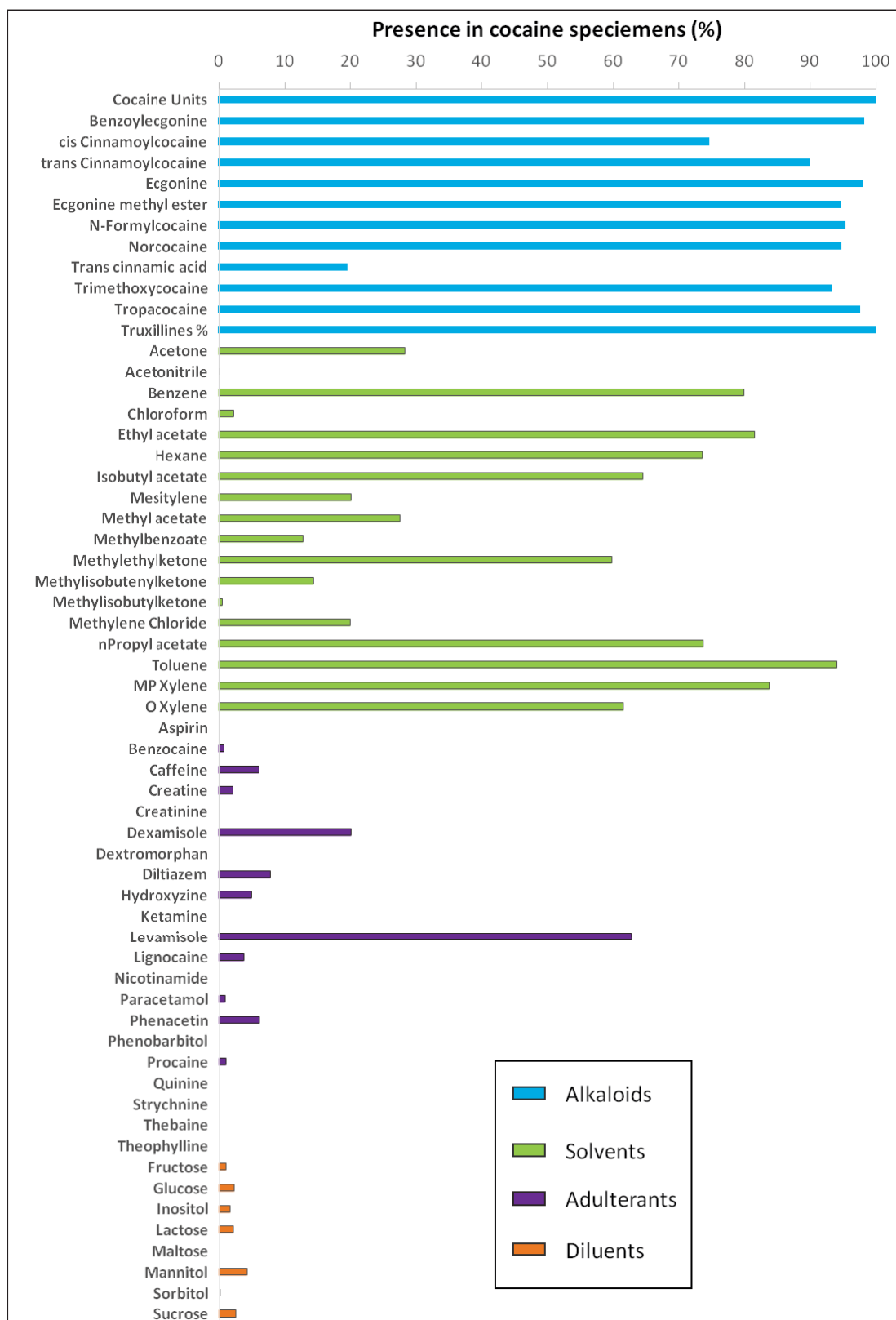
“If we understand what we do, that would already be a big step”.

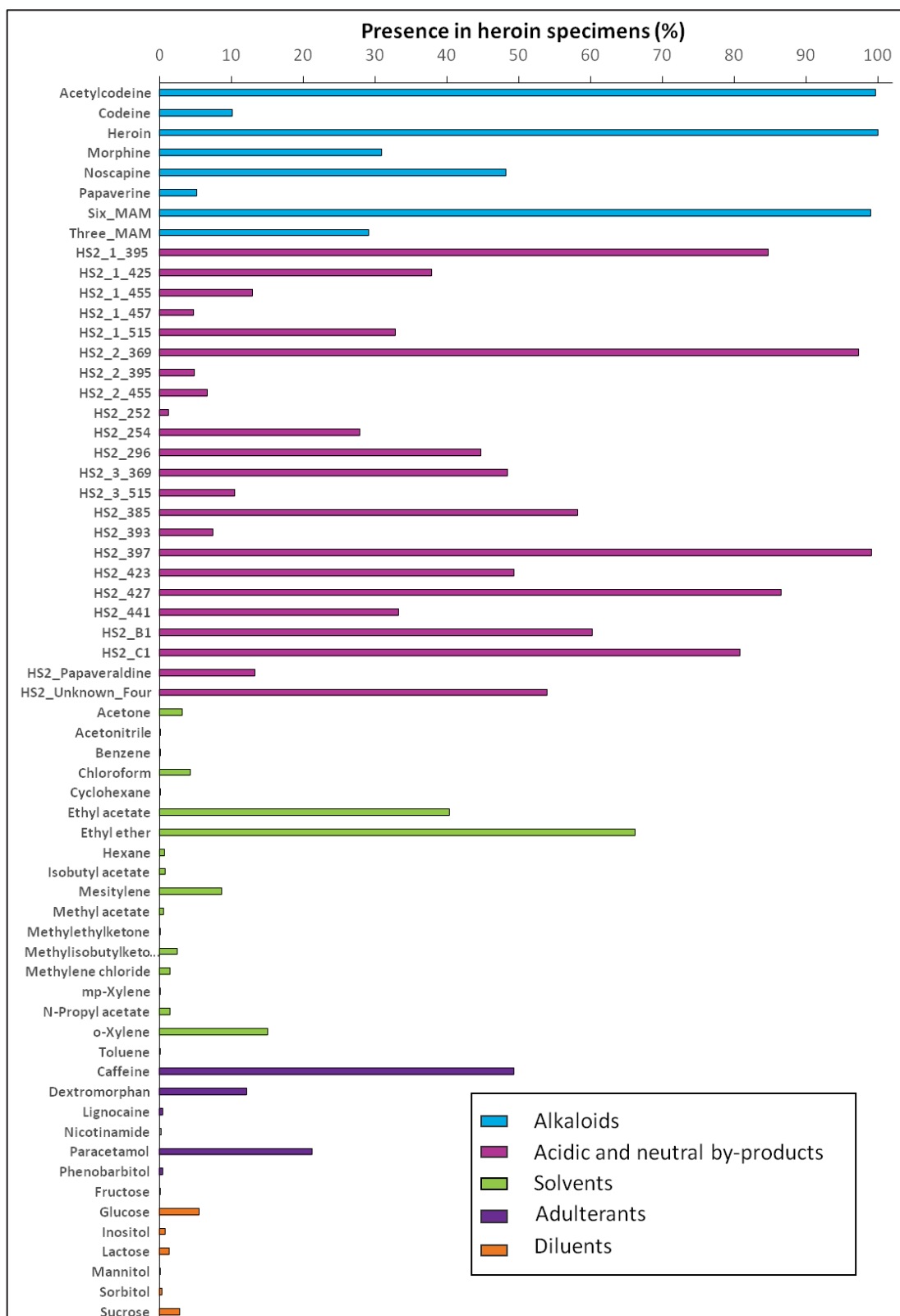
This last statement summarises what should be the fundamental aspiration of forensic intelligence for future research and applications in the field: only a proper understanding of criminal phenomena can lead to their disruption.

It is hoped that this research work contributed to a better understanding of illegal drug trafficking relevant to Australia.

Appendices

Appendix 1: Compounds detected in cocaine and heroin specimens





Appendix 2: Number of adulterants present in cocaine and heroin specimens

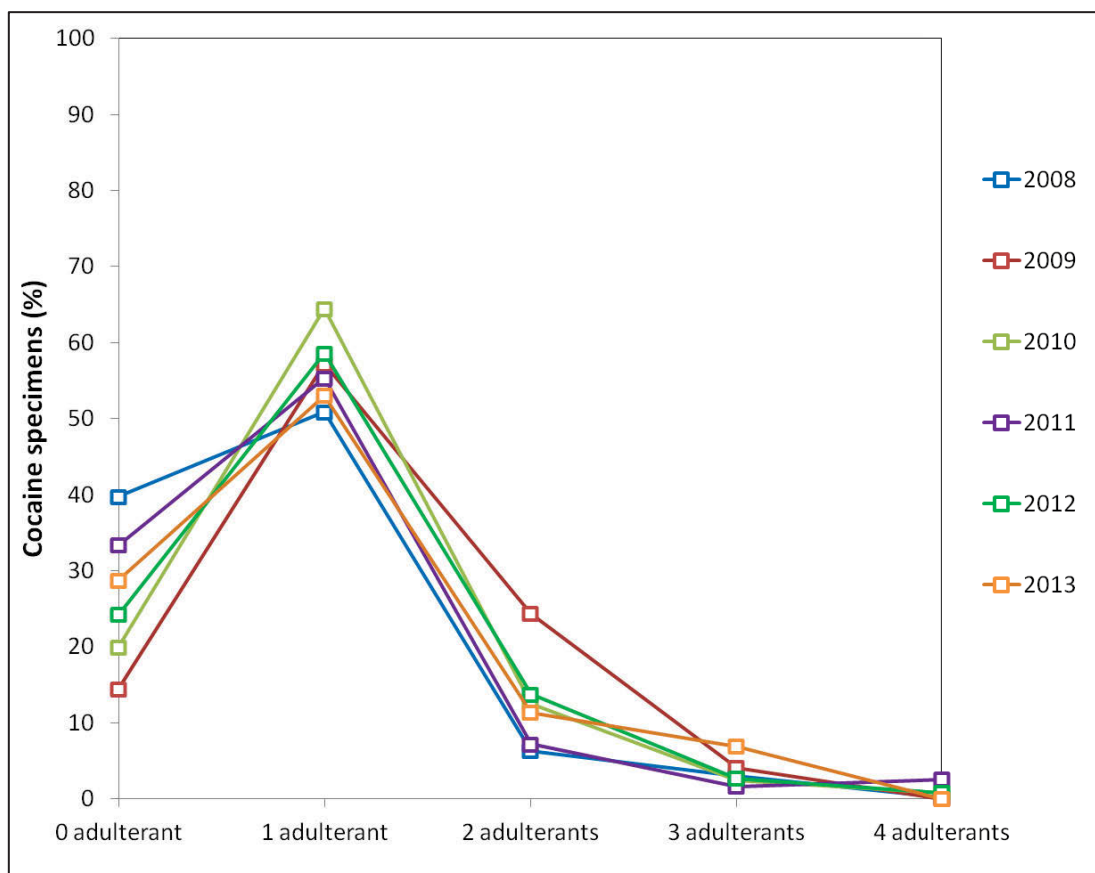
Between 2008 and 2013, a maximum of four different adulterants were present in one specimen for both cocaine and heroin.

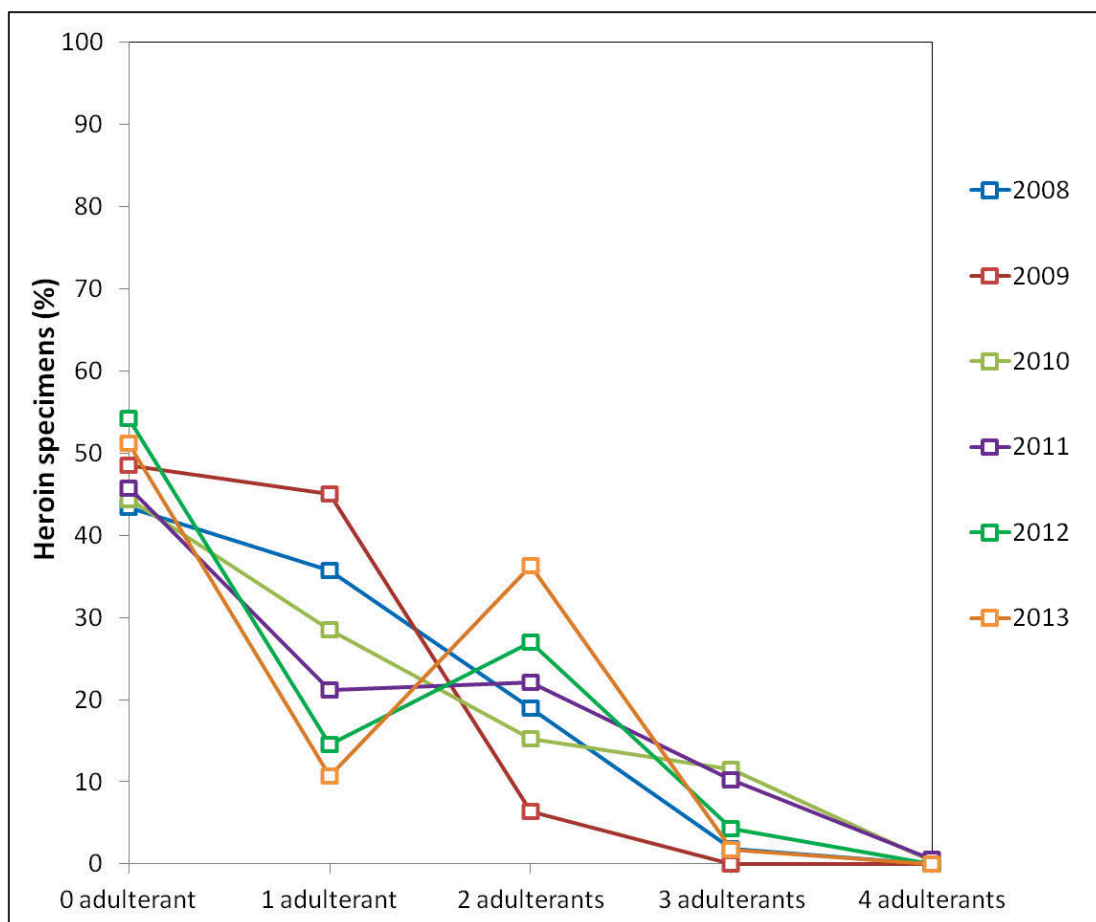
In cocaine specimens, a majority of specimens contained one adulterant, in particular PTHIT.

However, the trend changed between 2008 and 2013. Indeed, the percentage of specimens containing zero adulterant was high in 2008, and it decreased drastically in 2009, to increase again slowly until 2013.

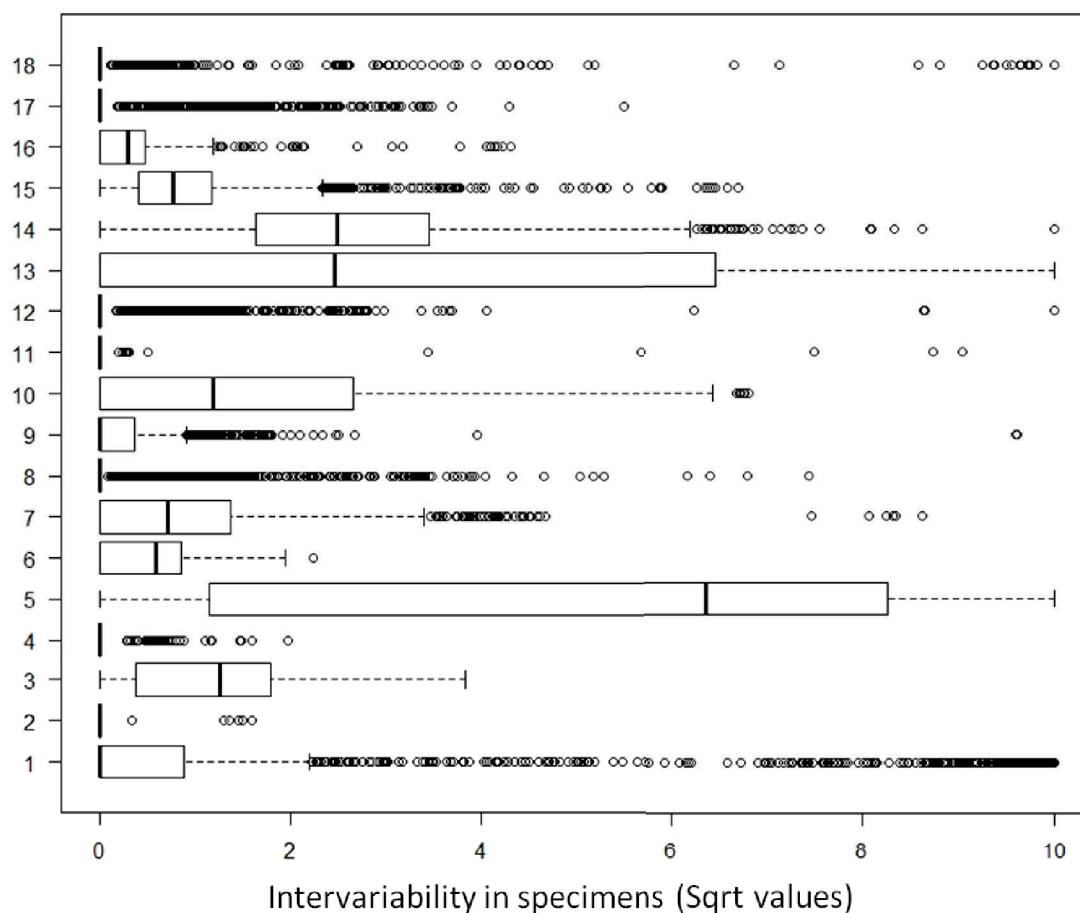
For heroin specimens, the trend was inverted from 2009 to 2013: in 2009, most specimens contained one adulterant (corresponding to caffeine), whereas the majority of specimens in 2013 contained two adulterants, corresponding to the rise of paracetamol over the six years.

Specimens containing three or four adulterants represent a minority of both cocaine and heroin datasets (less than 10 % each year).



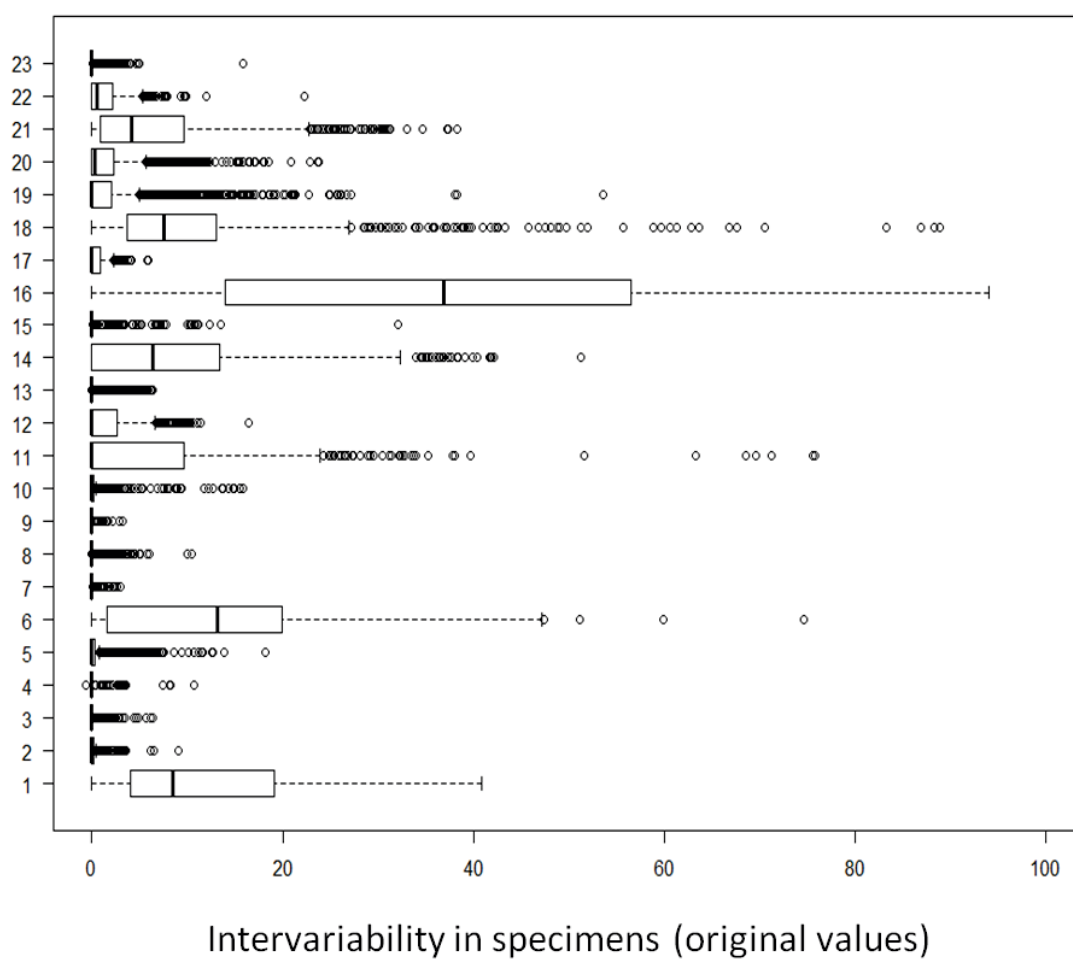


Appendix 3: Solvents' profile of cocaine specimens



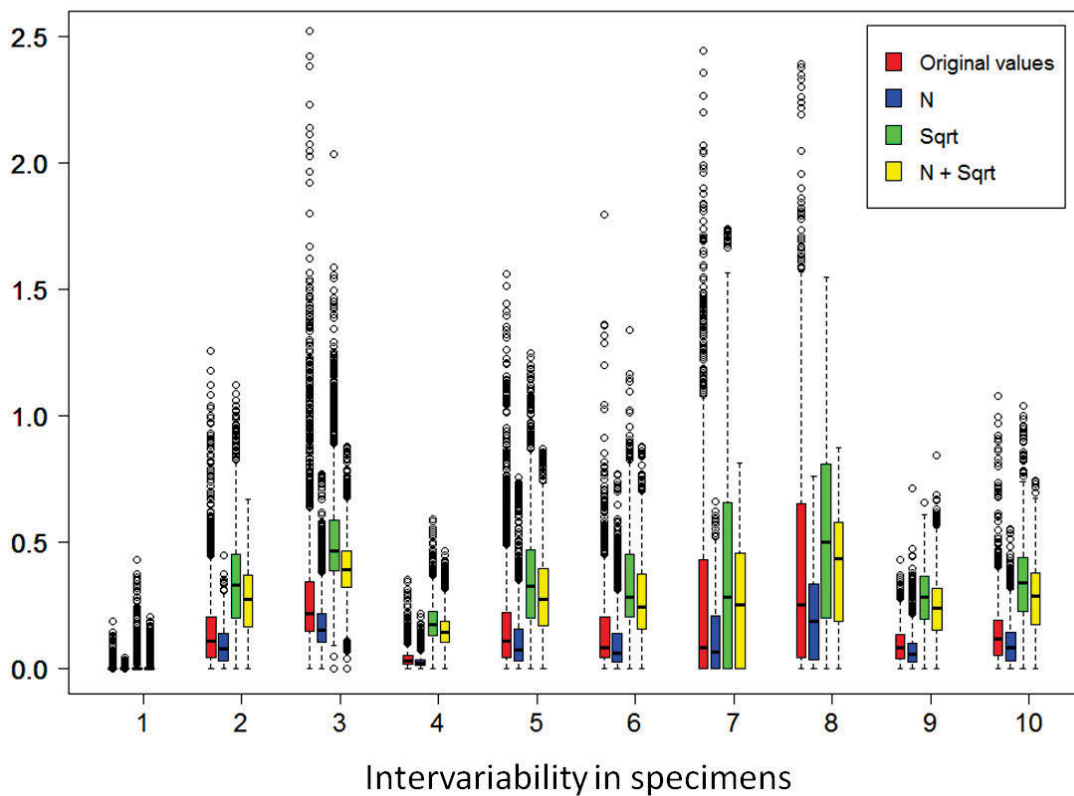
1. Acetone; 2. Acetonitrile; 3. Benzene; 4. Chloroform; 5. Ethyl acetate; 6. Hexane;
 7. Isobutyl acetate; 8. Mesitylene; 9. Methyl acetate; 10. MEK; 11. MIBK;
 12. Methylene Chloride; 13. n-Propyl acetate; 14. Toluene; 15. MP-Xylene; 16. O-Xylene;
 17. Methylisobutenylketone; 18. Methylbenzoate

Appendix 4: Impurities' profile of heroin specimens



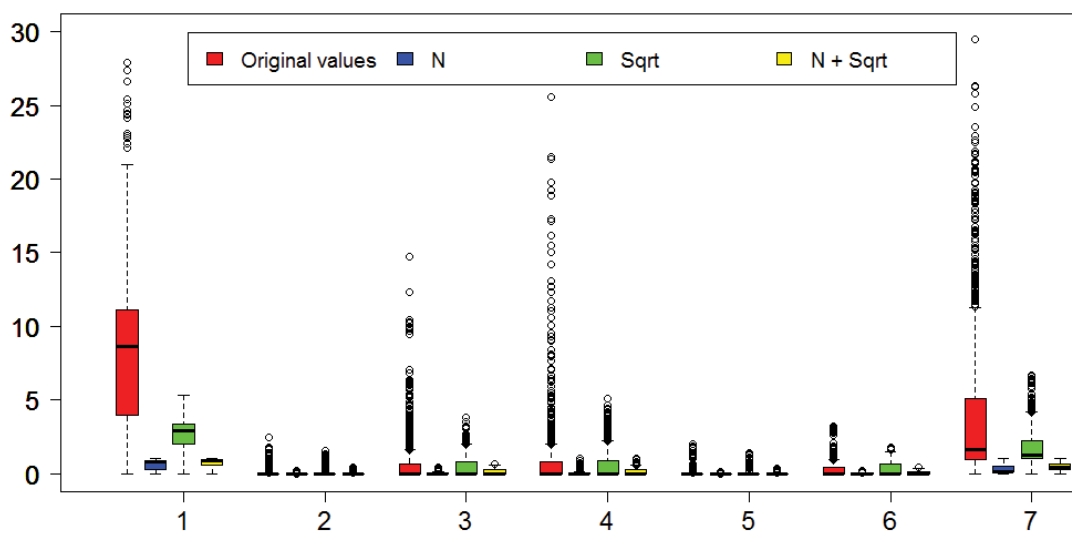
The identity of the 23 variables is unknown.

Appendix 5: Pre-treatments applied to alkaloids' profile of cocaine data



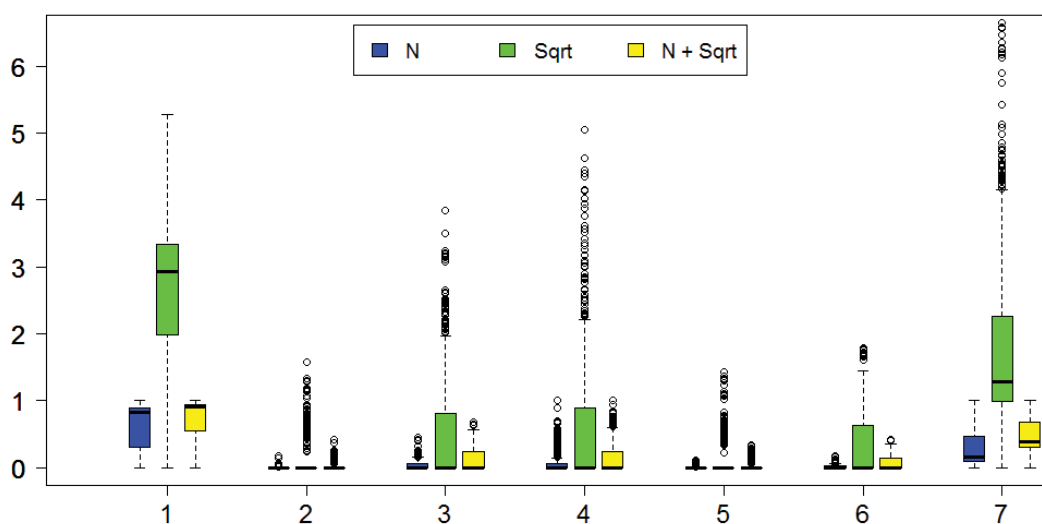
1. *Trans*-Cinnamic acid; 2. Ecgonine; 3. Benzoylecgonine; 4. *N*-Formylcocaine; 5. Ecgonine methyl ester; 6. Norcocaine; 7. *Cis*-Cinnamoylcocaine; 8. *Trans*-Cinnamoylcocaine; 9. Trimethoxycocaine; 10. Tropacocaine

Appendix 6: Pre-treatments applied to alkaloids' profile of heroin data



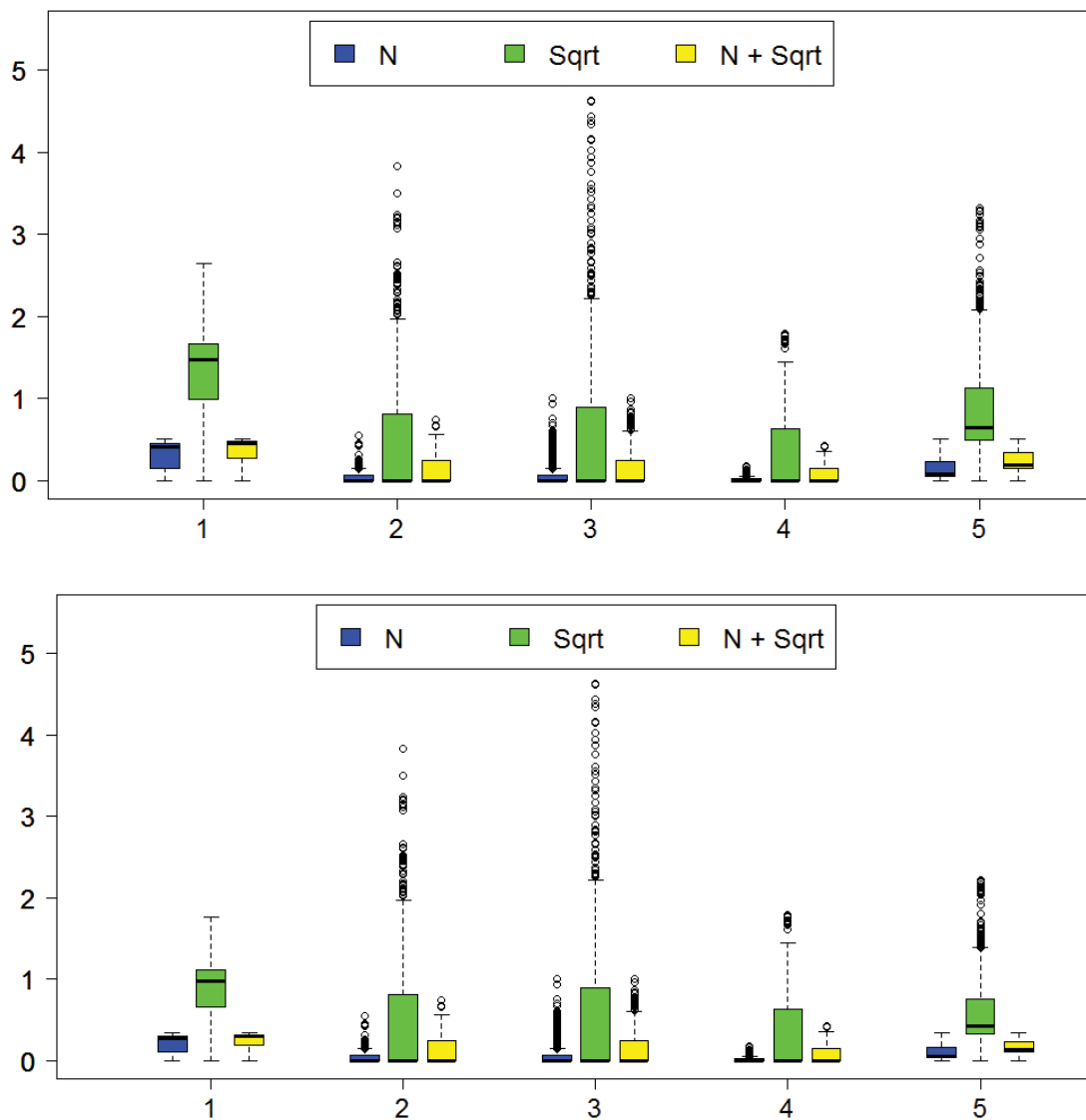
1. Acetylcodeine; 2. Codeine; 3. Morphine; 4. Noscapine; 5. Papaverine; 6. 3-MAM; 7. 6-MAM

Boxplots are displayed without original data for a better representativeness:



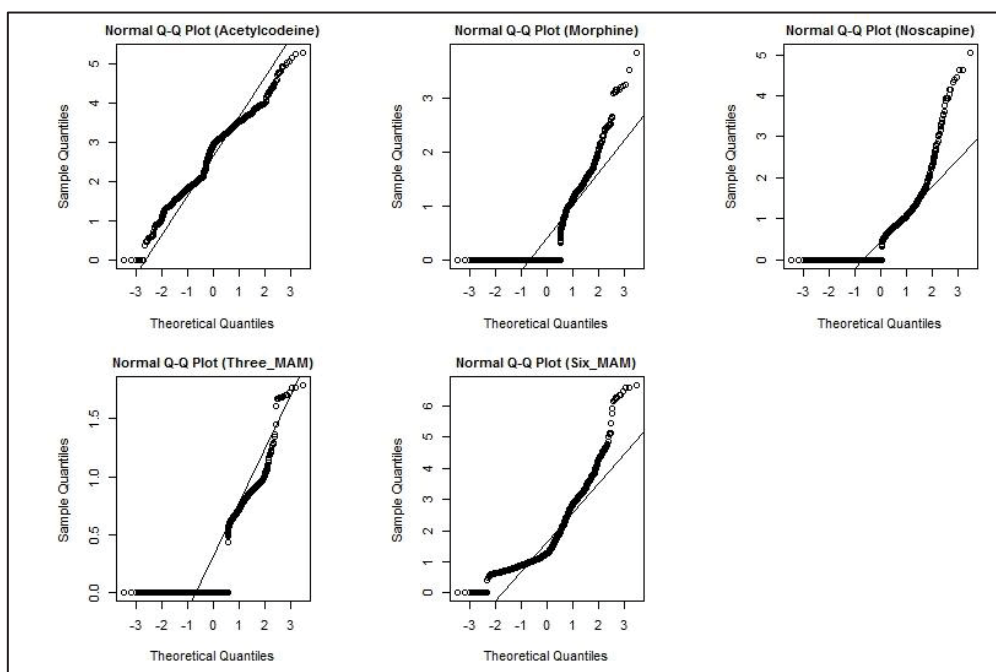
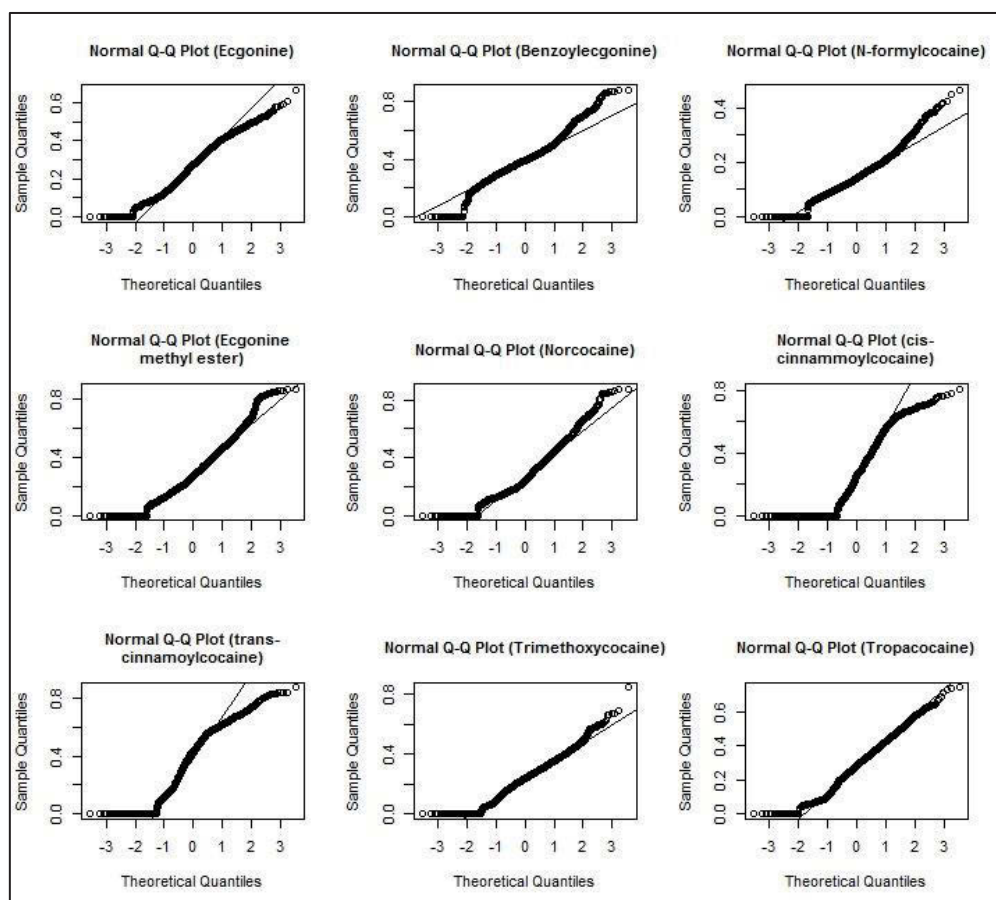
1. Acetylcodeine; 2. Codeine; 3. Morphine; 4. Noscapine; 5. Papaverine; 6. 3-MAM; 7. 6-MAM

Linear transformations are applied to acetylcodeine and 6-MAM (dividing by 2, upper figure, and dividing by 3, down figure) as values are higher than other variables (codeine and papaverine are removed as values are close from 0).



1. Acetylcodeine; 2. Morphine; 3. Noscapine; 4. 3-MAM; 5. 6-MAM

Appendix 7: QQ-plot representation of the distribution of alkaloids in cocaine specimens (up) and heroin specimens (down)



Appendix 8: Correlation between alkaloids of cocaine data using Spearman (up) and Pearson (down)

	Ecgonine	Benzoyllecgonine	Formylcocaine	Ecgonine methyl ester	Norcocaine	cis-Cinnamoylcocaine	trans-Cinnamoylcocaine	Trimethoxycocaine	Tropacocaine
Ecgonine	1								
Benzoyllecgonine	0.48	1							
Formylcocaine	0.09	0.04	1						
Ecgonine methyl ester	0.51	0.08	0.06	1					
Norcocaine	0.01	0.06	0.29	0.18	1				
cis-Cinnamoylcocaine	-0.46	-0.31	-0.35	-0.57	-0.48	1			
trans-Cinnamoylcocaine	-0.56	-0.44	-0.27	-0.58	-0.31	0.7	1		
Trimethoxycocaine	0.1	-0.1	0.34	0.23	0.17	-0.39	-0.27	1	
Tropacocaine	0.33	0.08	0.37	0.42	0.26	-0.57	-0.56	0.59	1

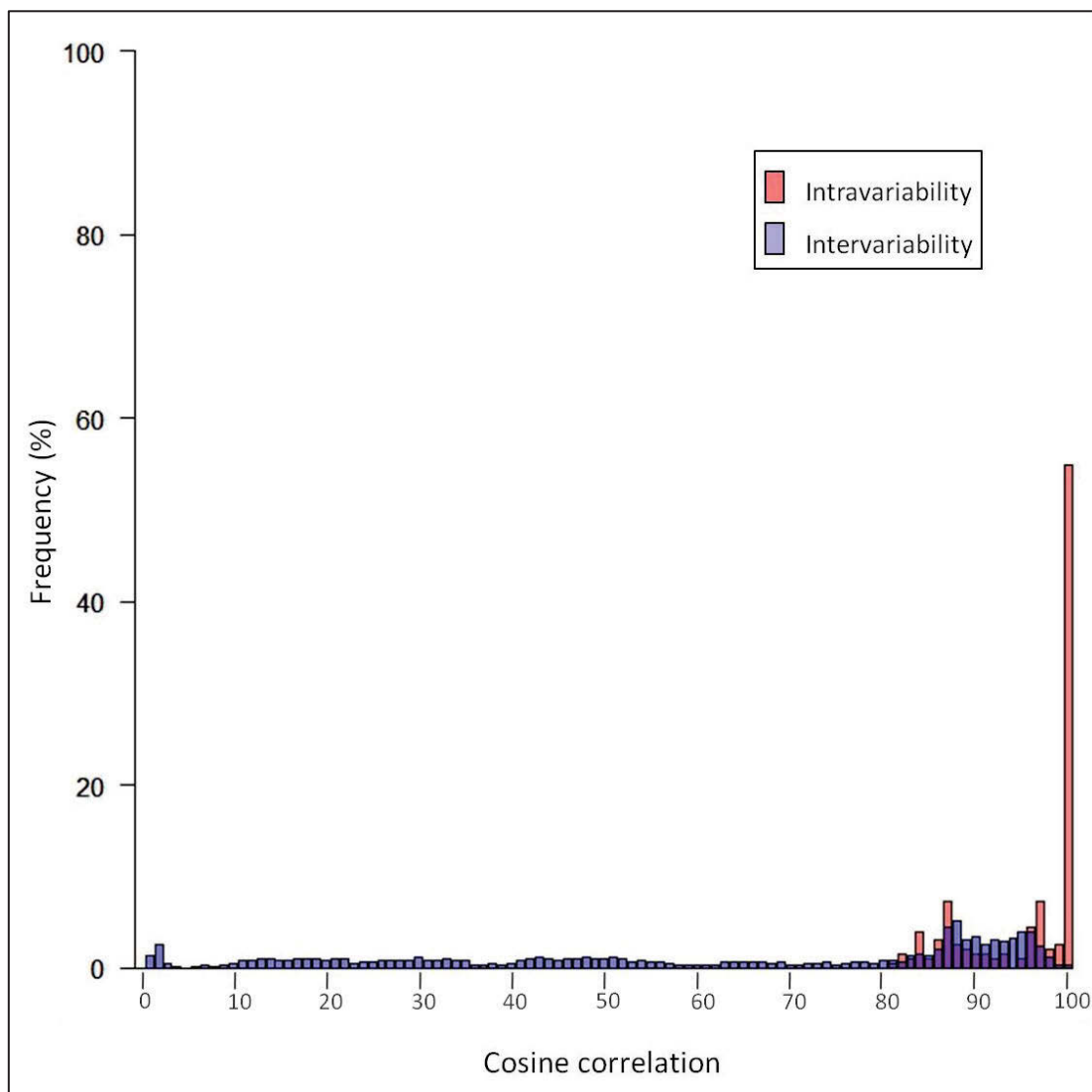
	Ecgonine	Benzoyllecgonine	Formylcocaine	Ecgonine methyl ester	Norcocaine	cis-Cinnamoylcocaine	trans-Cinnamoylcocaine	Trimethoxycocaine	Tropacocaine
Ecgonine	1								
Benzoyllecgonine	0.41	1							
Formylcocaine	0.1	0.04	1						
Ecgonine methyl ester	0.44	-0.03	0.04	1					
Norcocaine	-0.07	-0.03	0.25	0.08	1				
cis-Cinnamoylcocaine	-0.47	-0.29	-0.35	-0.56	-0.46	1			
trans-Cinnamoylcocaine	-0.53	-0.39	-0.27	-0.57	-0.29	0.69	1		
Trimethoxycocaine	0.09	-0.14	0.31	0.18	0.14	-0.43	-0.26	1	
Tropacocaine	0.29	0.02	0.34	0.36	0.21	-0.59	-0.54	0.55	1

Appendix 9: Correlation between alkaloids of heroin data using Spearman (up) and Pearson (down)

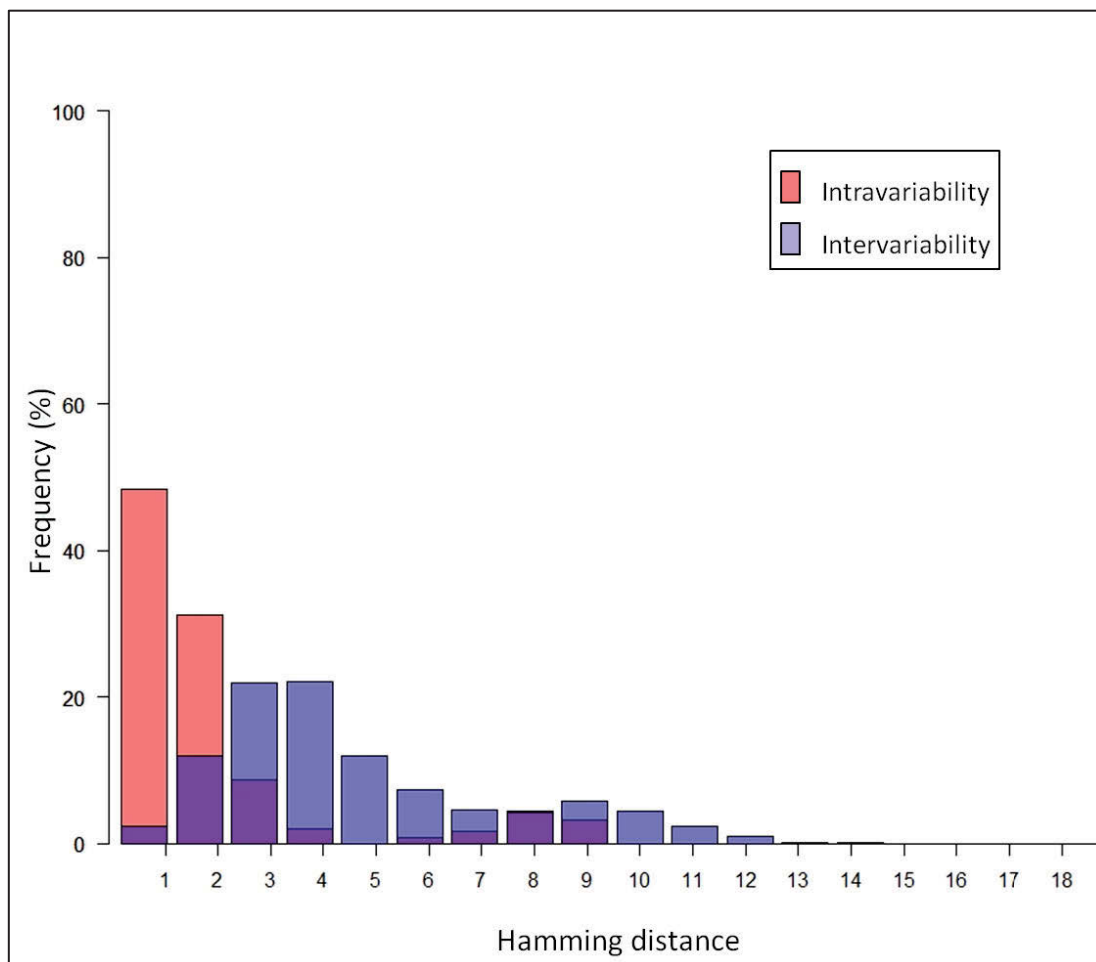
	Acetylcodeine	Morphine	Noscapine	3-MAM	6-MAM
Acetylcodeine	1				
Morphine	-0.61	1			
Noscapine	-0.50	0.62	1		
3-MAM	-0.40	0.67	0.47	1	
6-MAM	-0.43	0.77	0.54	0.69	1

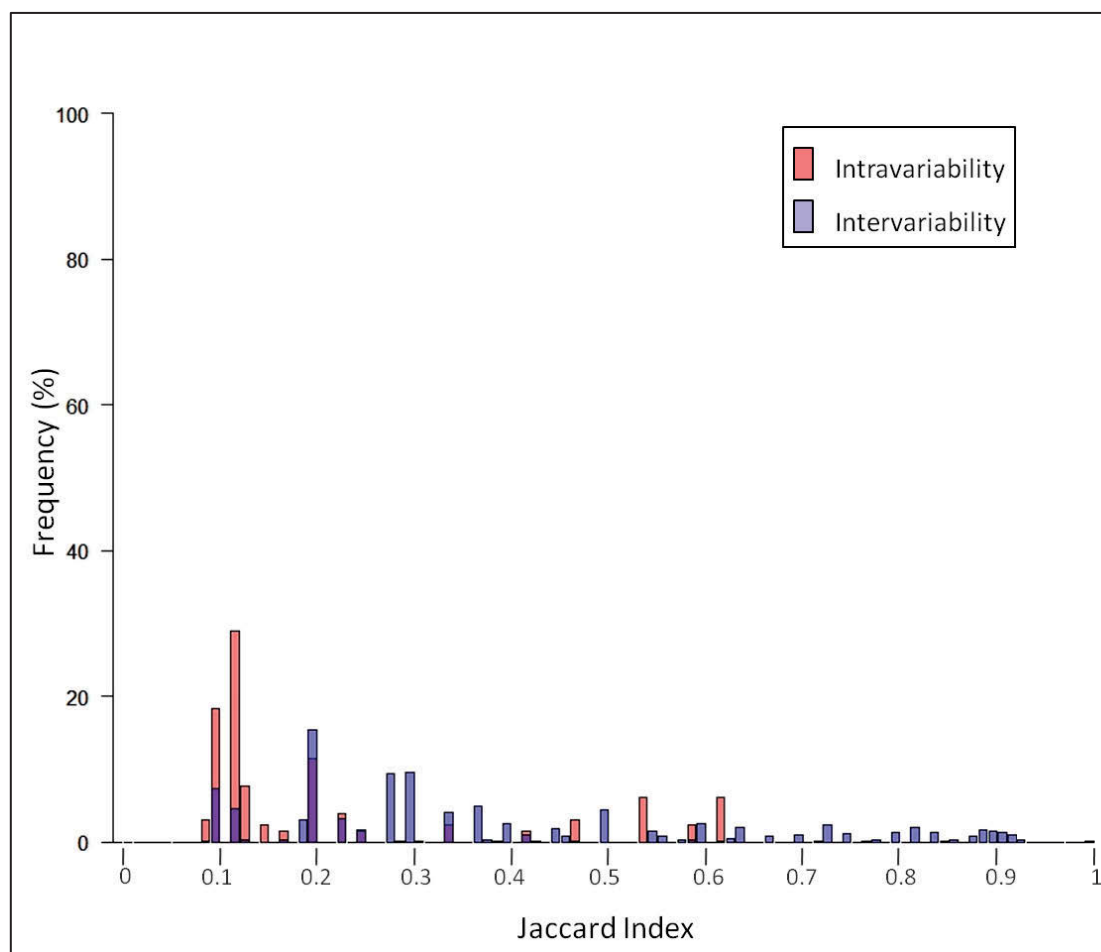
	Acetylcodeine	Morphine	Noscapine	3-MAM	6-MAM
Acetylcodeine	1				
Morphine	-0.58	1			
Noscapine	-0.46	0.57	1		
3-MAM	-0.41	0.61	0.34	1	
6-MAM	-0.50	0.89	0.54	0.68	1

Appendix 10: Cosine correlation for solvents' profile with percentage values of cocaine specimens

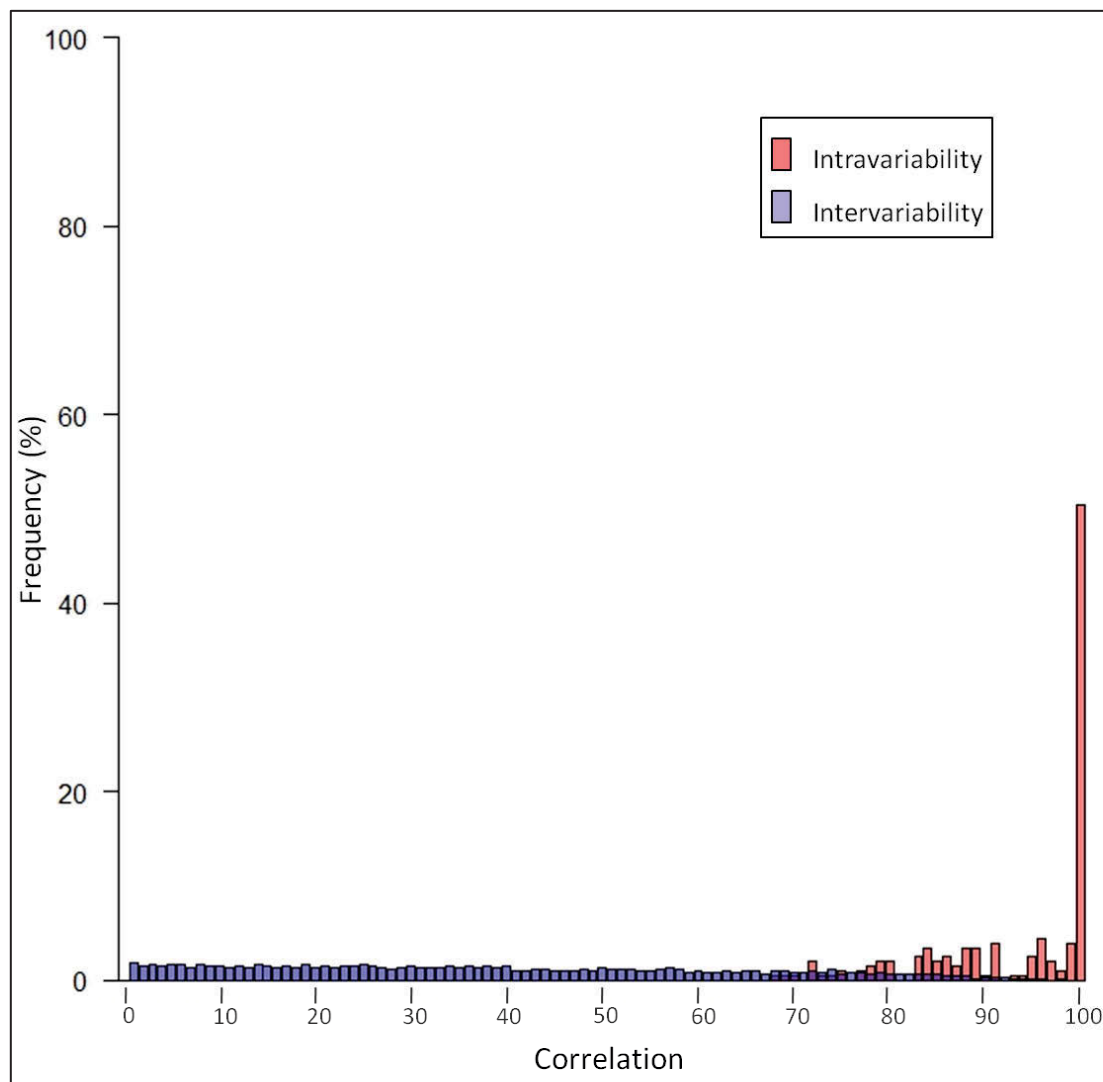


Appendix 11: Hamming Distance and Jaccard Index for solvents' profile with binary values of cocaine specimens





Appendix 12: Correlation for alkaloids and solvents' profiles combined from cocaine specimens



Appendix 13: Comparison of obtained correlations with associated TP, TN, FP and FN for the different profiles investigated with cocaine border seizures

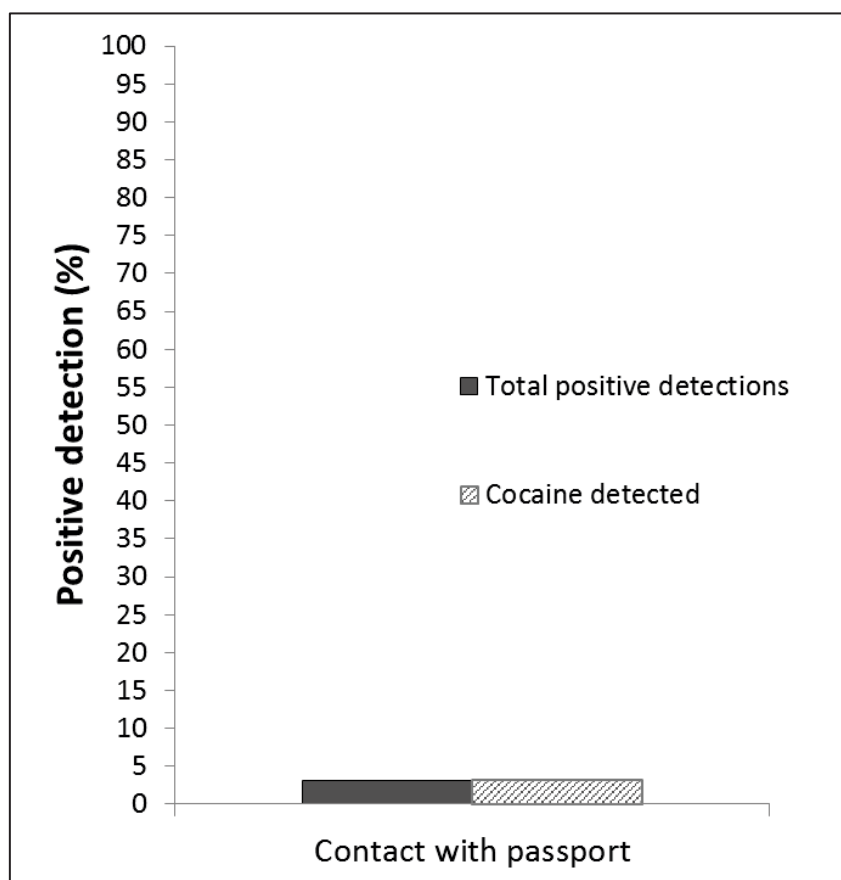
Interesting values used in the study were obtained from correlation 70 (i.e. Corr):

	Alkaloids profile				Alkaloids and truxillines profile				Combined alkaloids and solvents profiles			
Corr [n]	FP	TP	TN	FN	FP	TP	TN	FN	FP	TP	TN	FN
70	14.04	90.69	85.96	9.31	19.12	98.53	80.88	1.47	13.83	99.02	86.17	0.98
71	13.18	90.20	86.82	9.80	17.98	98.53	82.02	1.47	12.98	98.53	87.02	1.47
72	12.36	88.73	87.64	11.27	16.99	98.04	83.01	1.96	12.17	98.53	87.83	1.47
73	11.45	88.73	88.55	11.27	15.72	98.04	84.28	1.96	11.30	96.57	88.70	3.43
74	10.66	88.24	89.34	11.76	14.84	98.04	85.16	1.96	10.53	96.08	89.47	3.92
75	9.57	88.24	90.43	11.76	14.19	98.04	85.81	1.96	9.43	95.59	90.57	4.41
76	8.87	87.75	91.13	12.25	13.45	98.04	86.55	1.96	8.76	94.61	91.24	5.39
77	8.11	86.76	91.89	13.24	12.58	98.04	87.42	1.96	8.01	94.61	91.99	5.39
78	7.40	85.29	92.60	14.71	11.78	98.04	88.22	1.96	7.30	93.63	92.70	6.37
79	6.70	84.80	93.30	15.20	11.11	97.55	88.89	2.45	6.62	92.16	93.38	7.84
80	5.99	84.80	94.01	15.20	10.28	97.55	89.72	2.45	5.89	90.20	94.11	9.80
81	5.40	84.31	94.60	15.69	9.36	97.55	90.64	2.45	5.33	88.24	94.67	11.76
82	4.79	82.84	95.21	17.16	8.45	96.57	91.55	3.43	4.73	88.24	95.27	11.76
83	4.24	78.92	95.76	21.08	7.80	96.08	92.20	3.92	4.20	88.24	95.80	11.76
84	3.67	76.96	96.33	23.04	7.13	96.08	92.87	3.92	3.61	85.78	96.39	14.22
85	3.14	75.98	96.86	24.02	6.30	94.12	93.70	5.88	3.08	82.35	96.92	17.65

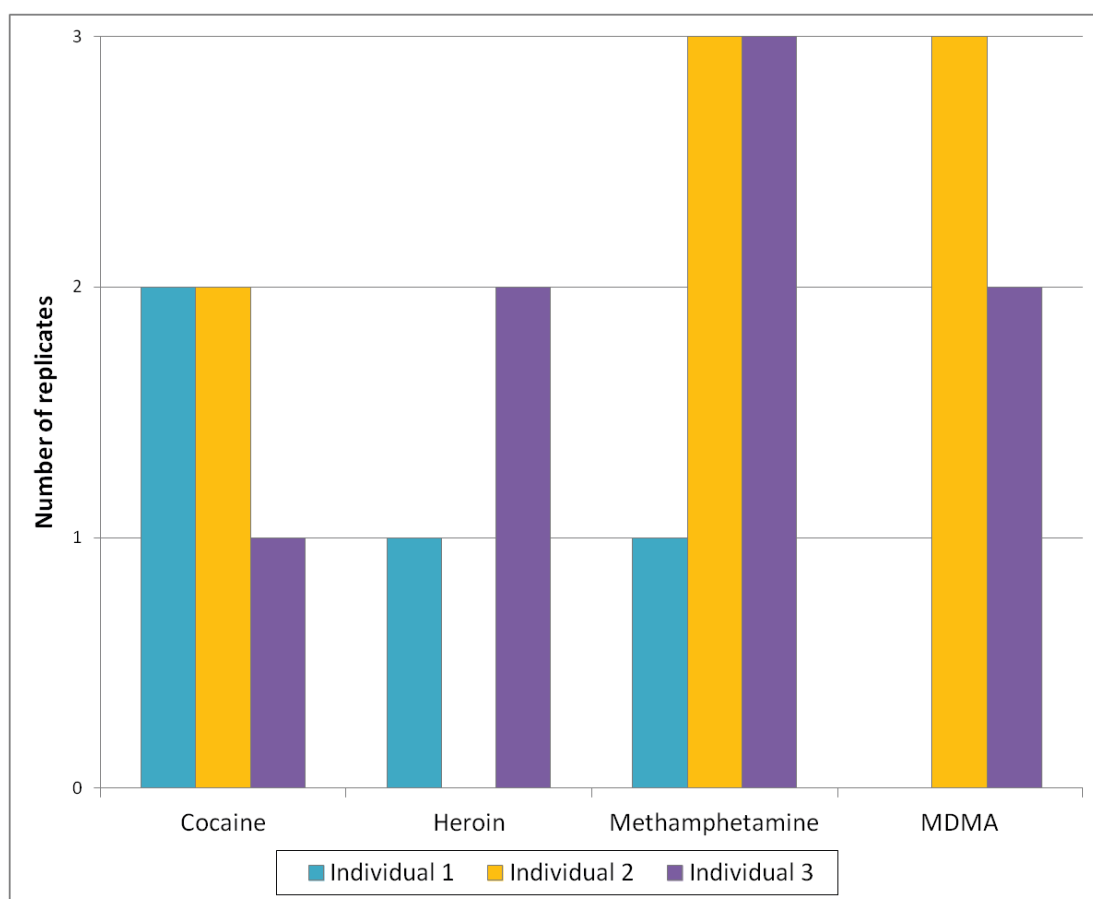
86	2.55	73.04	97.45	26.96	5.65	91.67	94.35	8.33	2.52	80.39	97.48	19.61
87	2.10	70.10	97.90	29.90	4.88	89.22	95.12	10.78	2.07	77.94	97.93	22.06
88	1.67	70.10	98.33	29.90	4.15	85.29	95.85	14.71	1.68	76.47	98.32	23.53
89	1.24	69.61	98.76	30.39	3.48	82.35	96.52	17.65	1.27	73.04	98.73	26.96
90	1.06	68.63	98.94	31.37	2.91	80.88	97.09	19.12	1.10	69.61	98.90	30.39
91	0.84	65.20	99.16	34.80	2.22	77.45	97.78	22.55	0.87	69.12	99.13	30.88
92	0.60	64.22	99.40	35.78	1.67	76.96	98.33	23.04	0.65	65.20	99.35	34.80
93	0.42	64.22	99.58	35.78	1.10	75.98	98.90	24.02	0.45	65.20	99.55	34.80
94	0.30	60.78	99.70	39.22	0.67	73.04	99.33	26.96	0.33	64.71	99.67	35.29
95	0.21	59.80	99.79	40.20	0.41	68.14	99.59	31.86	0.23	64.22	99.77	35.78
96	0.13	55.39	99.87	44.61	0.20	62.75	99.80	37.25	0.14	61.76	99.86	38.24
97	0.10	54.90	99.90	45.10	0.11	55.88	99.89	44.12	0.11	57.35	99.89	42.65
98	0.09	54.41	99.91	45.59	0.07	51.47	99.93	48.53	0.10	55.39	99.90	44.61
99	0.01	51.96	99.99	48.04	0.02	49.02	99.98	50.98	0.01	54.41	99.99	45.59
100	0.00	47.06	100	52.94	0.00	44.12	100	55.88	0.00	50.49	100	49.51

Appendix 14: Secondary transfer with banknotes

Banknotes were tested with the DS-1100 N™ instrument. Results were not differentiated depending on the type of currency (\$5, \$10, \$20 and \$50), as the aim of this experiment was to investigate a potential contamination of passports in contact with banknotes.



Appendix 15: Inter-individual variation for direct transfer with fingers



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