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A recommended procedure for establishing the source level relationships between heroin case samples of unknown origins

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Abstract A recent concern of how to reliably establish the source level relationships of heroin case samples is addressed in this paper. Twenty-two trafficking heroin case samples of unknown origins seized from two major regions (Kuala Lumpur and Penang) in Malaysia were studied. A procedure containing six major steps was followed to analyze and classify these samples. Subsequently, with the aid of statistical control samples, reliability of the clustering result was assessed. The final outcome reveals that the samples seized from the two regions in 2013 had highly likely originated from two different sources. Hence, the six-step procedure is sufficient for any chemist who attempts to assess the relative source level relationships of heroin samples.

1. Introduction

Despite the high influx of various synthetic drugs into the black market, heroin remains the most frequently abused sub-stance in Malaysia. This semi-synthetic drug is processed from morphine, one of the active compounds present in the opium latex. The resulting heroin can be diluted/cut with many other substances, such as caffeine and paracetamol, two commonly used diluents in Malaysia. Uncut heroin substances usually contain a high amount of heroin (HR) and quantifiable amounts of codeine (CD), morphine (MP), acetylcodeine (AC) and monoacetylmorphines (MM). It was demonstrated that most heroin samples in Malaysia still contained the aforementioned compounds at detectable levels even though they had been 90% cut.[1] In some cases, papaverine, noscapine and thebaine can also be detected. However, these three compounds can become masked very easily if the heroin substance is diluted with other substances. As a result, it is established that only the five formerly mentioned opium alkaloids can be effectively used as indicators for heroin profiling since they are readily measurable in the local heroin samples.

Opium alkaloids and neutral/acidic manufacturing impurities present in the heroin samples have long been used to establish sample relationships at the origin and source levels. As suggested by Besacier et al.[2], opium alkaloids that are present as major components are the important indicators that one

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Table 1 Recommended procedure for determining the source level relationships between heroin case samples of unknown origins.

No.	Step	Description	Task previously completed or to be undertaken in this study
1.	Method optimization and validation	The analytical method (e.g., gas chromatographic method) must be fit for the profiling purpose. For example, it should meet the acceptance criteria set by the international bodies or the laboratory undertaking the task	A gas chromatography-flame ionization detection method has been developed and proved stable for Malaysian heroin samples in this laboratory.[1,3–10] Note: Previous studies employed concentrations (equivalent to area ratios) for step 1. This study, However, employed peak areas that were corrected by the IS.
2.	Statistical Validation	This must be done by using samples of known origins or known relationships to assess the validity of the preferred clustering technique. For example, it should inform which data pretreatment and distance measure that are able to provide the best clustering results	Hierarchical cluster analysis (HCA) using Ward linkage and Manhattan distance presented promising clustering results for simulated samples of known relationships when GC data heroin were interpreted in 11 normalized parameters ($N_{selected}$), namely AC/HR, AC/MM, AC/(MM + HR), AC/(MP + MM + HR), MM/HR, (CD + MP)/(MM + HR), HR/MM, (CD + AC)/(MP + MM + HR), (CD + MP + MM + HR)/AC, HR/(CD + MP + AC + MM) and (MP + MM + HR)/(CD + AC), which were subsequently standardized (S) to achieve 11 $N_{selected} + S$ variables. Details of this step can be found in the original study[4]
3.	Collection of statistical control sample(s)	Some samples should be collected to serve as statistical controls. The sample should be at least 2-year temporally spaced from the case samples to be profiled	To be undertaken in this study A statistical control sample was collected in 2010 from Kuala Lumpur
4.	Collection of case samples	Case samples are collected for analysis	To be undertaken in this study 22 case samples seized in 2013 from Kuala Lumpur and Penang were collected
5.	Chemical analysis	Statistical control samples and case samples should be analyzed by the same instrumental method determined in step 1	To be undertaken in this study
6.	Statistical analysis	All data obtained should be statistically analyzed using the pretreatment and statistical technique determined in step 2	To be undertaken in this study

should profile because they can reliably be used to correlate with other findings derived from trace manufacturing impurities and occluded solvents. Determination of the major components is very straightforward and it involves less sample preparation. Besides, most of these major components are readily extractable with universal solvents. So, much profiling effort can be eliminated as compared with trace manufacturing impurities as the latter requires a tedious liquid–liquid extraction procedure. Hence, five major components present in the illicit heroin samples were used in this study to explain a recommended procedure for establishing the source level relationships of heroin samples with unknown backgrounds.

2. Recommended procedure

A number of profiling methods for illicit heroin have been established to determine impurities present in the samples. In the current status, some of these studies have focused on method development and validation, statistical validation and some basic steps for classifying the illicit drug samples.[1,3–10] However, there have not been any clear-cut, step-by-step guidelines available to help forensic chemists to determine sample relationships between drug seizures/samples, whether source or street level. In such attempts, one may feel nervous because of three reasons: (1) it is not some kind of routine usually performed in the laboratory; (2) the chemist does not even know how and where to start the profiling work; and (3) the chemist is not sure of the degree of certainty in the result obtained because there lacks some form of quality control (QC) for heroin profiling. Remedially, this paper seeks to put forward a set of practical guidelines to the profiling and determination of the source level relationships between heroin samples of unknown origins using the above-cited five opium alkaloids. One major concern is that HR is easily converted to 6-MM or MP under favorable conditions. Instead of attesting to the stability of these alkaloids, this paper utilizes quotients derived from the five major components to address the issue of stability. With proper pretreatment, the quotients in the form of normalized–standardized variables ($N_{selected} + S$) proved excellent for clustering 216 simulated heroin samples of known relationships in a previous study.[4] Table 1 summarizes the major steps necessary for classifying heroin case samples through unsupervised pattern recognition (where samples of known origins are not available). The subsequent discussion will demonstrate how the procedure should be followed in order to arrive at a reliable conclusion.