Institution: Queen's University, Belfast

Unit of Assessment: 8

Title of case study: Practical Raman chemical analysis for forensic applications

1. Summary of the impact (indicative maximum 100 words)

Techniques that can produce detailed chemical information rapidly and non-destructively for many forensic applications have been developed by Queen's University Belfast based on Raman analysis. The techniques have been adopted by the Forensic Science laboratory in Northern Ireland (FSNI) to trace the source of seized drugs, identify novel psychoactive substances ("legal highs") and study paint evidence. More than 2000 cases of supply/possession of ecstasy drugs, 947 paint casework samples and 100 'legal highs' have been analysed. Other law enforcement agencies are now adopting the methods developed at Queen's.

2. Underpinning research (indicative maximum 500 words)

Raman spectroscopy is a powerful method for characterising materials; however, due to the cost and lack of background information it has not been used for routine analysis of forensic samples. In 1999, the Queen's team led by Bell recognised the potential of Raman as a method to analyse for drugs of abuse, specifically ecstasy. In collaboration with the Forensic Science Agency in Northern Ireland, Bell's group demonstrated, for the first time, the use of Raman spectroscopy for the identification of ecstasy and related drugs.[see reference 1 *in section 3*] In addition, the method was sensitive enough to identify the cutting agents as well as the drug present in a wide range of seized samples thus providing critical information on the particular blend in order to discriminate between different batches of tablets that had been seized in raids. The ability to link a single ecstasy tablet to the dealer in less than a minute had great potential for drugs intelligence work since it allowed distribution pathways to be tracked and a large scale pilot study on seized samples was performed. [see reference 2 *in section 3*] This led to work which established the method for moving Raman from a specialist university laboratory method to routine tool for high throughput drugs analysis.[reference 3 *in section 3*]

This work was extended to the identification of cathinones, a family of once 'legal highs' which was banned in 2010, by Bell and Fletcher in Queen's. Here the Raman methods were supplemented by NMR and mass spectroscopy techniques in order to identify which of the many potential drug compounds from this family were actually being distributed. Queen's synthesised reference samples of important cathinones to provide standard samples which were used to populate a materials library and to allow the relationship between structure and spectra to be determined.[see reference 4 *in section 3*].

Work on paint analysis followed a similar pattern, with initial studies in 2005 on what information the spectra could provide followed by a demonstration of how those data could be used to provide discrimination between real world samples.[references 5,6 *in section 3*] In particular, analysis of white paints was important since they are common in forensic casework but difficult to discriminate using standard methods.

3. References to the research (indicative maximum of six references) * signify the references which best indicate the quality of the underpinning research

*1 "Rapid analysis of ecstasy and related phenethylamines in seized tablets by Raman spectroscopy", Bell, S.E.J., D.T. Burns, A.C. Dennis, and J.S. Speers, Analyst, 125, 541-544, 2000. DOI:10.1039/a908091k

*2 "Tracking the distribution of "ecstasy" tablets by Raman composition profiling: A large scale feasibility study", Bell, S.E.J., L.J. Barrett, D.T. Burns, A.C. Dennis, and S.J. Speers, Analyst, 128, 1331-1335, 2003. DOI: 10.1039/b308312h Note resulting news coverage: Test to link drug to dealers (BBC) http://news.bbc.co.uk/1/hi/northern_ireland/3222061.stm Fast way to find out what's in a tab of E (New Scientist) http://www.newscientist.com/article/mg18024162.700-fast-way-to-find-out-whats-in-a-tab-of-e.html

3 Development of sampling methods for Raman analysis of solid dosage forms of therapeutic and illicit drugs", Bell, S.E.J., J.R. Beattie, J.J. McGarvey, K.L. Peters, N.M.S. Sirimuthu, and S.J. Speers, Journal of Raman Spectroscopy, 35, 409-417, 2004. DOI 10.1002/jrs.1160

4 “Raman Spectroscopy for Forensic Examination of β-Ketophenethylamine “Legal Highs”: Reference and Seized Samples of Cathinone Derivatives”, Samantha P. Stewart; Steven E. J. Bell; Nicholas C. Fletcher; Samira Bouazzaoui; Yen Cheng Ho; S. James Speers and K. Laota Peters. Front cover “Feature Article” Analytica Chimica Acta, 711, 1-6, 2012. DOI 10.1016/j.aca.2011.10.018.

5 "Rapid forensic analysis and identification of "lilac" architectural finishes using Raman spectroscopy", Bell, S.E.J., L.A. Fido, S.J. Speers, and W.J. Armstrong, Applied Spectroscopy, 59, 100-108, 2005. DOI 10.1366/0003702052940404

6 "Forensic analysis of architectural finishes using Fourier transform infrared and Raman spectroscopy, Part II: White paint", Bell, S.E.J., L.A. Fido, S.J. Speers, W.J. Armstrong, and S. Spratt, Applied Spectroscopy, 59, 1340-1346, 2005. DOI 10.1366/000370205774783232

4. Details of the impact (indicative maximum 750 words)

Research by the Queen’s team led by Bell and Fletcher has changed the working practice of the Forensic Science Agency in Northern Ireland, FSNI, in their analysis and case evidence accumulation in serious crime investigations. The new methods adopted have led to reduced analysis time and an increased ability to track distribution and supply of drugs. Following the research undertaken at Queen’s, FSNI invested in a £50K in a Raman spectrometer suitable for drugs analysis, which was funded by the Government's Assets Recovery Agency.

Figure 1 High throughput drug analysis-tablets being analysed in a Raman spectrometer as used in FSNI

In 2004 they began profiling ecstasy tablets for drugs intelligence purposes i.e. to detect common sources and patterns of distribution and supply. In 2008 FSNI adopted Raman spectroscopy as the technique of choice for the initial characterisation of *all* bulk white powders and tablets submitted to the Alcohol, Drugs and Toxicology section (Figure 1). At that time these types of samples constituted around 40% of the drugs

casework at FSNI, therefore, in approximately 500 cases per annum the techniques based on the QUB methods supported prosecutions for supply/possession.(reference 1 *in section 5*)

Similarly, the research on forensic analysis of household paint by QUB led directly to the Physical Evidence section of FSNI purchasing and validating dedicated Raman and IR microscopes for paint evidence examination in 2008. (reference 2 *in section 5*) Paint evidence is usually only examined in "serious cases" that include murder, rape and assaults, numbering up to 200 each year. This allowed the laboratory to stop investing in pyrolysis GC-MS as their confirmatory technique, leading to substantial savings in equipment estimated as £75K. A newspaper report of the crucial part paint evidence played in the first murder investigation using the method is given as reference 5 *in section 5*. As part of the long term rollout of the Raman method, since 2010 every paint casework sample which is submitted to the laboratory is subjected to Raman analysis (reference 2 *in section 5*). Again, as for the ecstasy work, the rationale is that Raman analysis increases the throughput and decreases the cost of hundreds of examinations per annum. The total number of paint examinations to date is 947.

The most recent impact of this Queen's research has been on β-ketophenethylamine "Legal Highs" (also known as "cathinones"). This new generation of synthetic psychotropic drugs was widely available through the internet until 2010 when legislation made supply and possession an offence. Due to their relative novelty and the potential to make hundreds of variants of cathinones, FSNI had problems characterising many of these until work carried out by Fletcher and Bell in 2010 on identifying the novel variants in seized samples. This had the immediate effect of allowing criminal prosecutions which had been stalled until then due to lack of evidence to proceed (reference 3 *in section 5*). As a result of the work at Queen's, the Director of Public Prosecutions has been able to allow the first criminal trials and convictions have been obtained. This work is now being extended through a FSNI-based trial on the use of the vibrational analysis method developed by QUB for high throughput screening of suspected cathinones seized by the postal authorities.

The work on cathinones has allowed the FSNI laboratory to establish a strategy for reacting quickly to the appearance of new classes of synthetic drugs and the corresponding legislation which follows. This is important because the frequency with which new classes of abused substances appear is increasing, as is the speed at which they are adopted to become global problems. (reference 3 *in section 5*).

The impact of this work is being extended beyond FSNI. Firstly, the cathinone trial has been extended to the "Celtic Fringe" group of government forensic laboratories which include Scottish and Eire labs (reference 4 *in section 5*). In addition, the Palestinian National Authority are in the process of purchasing a Raman system for routine drug analysis for their new forensic laboratories due to be completed 2013 (reference 1 *in section 5*). The FBI in the United States has been investigating the use of Raman spectroscopy for paint examination using a sample library supplied by QUB (reference 6 *in section 5*) and in a recent publication described the QUB series of paint papers as "important to the forensic community because they provide data to support the highly discriminating nature of comparative paint examination" (reference 7 *in section 5*). All the above clearly shows the global impact of the research undertaken given its widespread adoption.

Whilst the immediate beneficiaries of the research are the forensic science agencies, the work also has significant societal benefit in providing more robust analysis methods which can be performed rapidly and routinely. This combination allows a wider range of samples to be examined and the

creation of patterns of behaviour which will lead to prosecution and convictions.

5. Sources to corroborate the impact (indicative maximum of 10 references)

Letters from the appropriate users are available as follows:

1. Ecstasy analysis at FSNI by Operations Director and Head of Alcohol, Drugs and Toxicology (ADT) at FSNI during the time period when the method was adopted.
2. Paint analysis at FSNI by Principal Scientific Officer and Lead Scientist at FSNI, in charge of Physical Methods section during the time period.
3. Cathinone characterisation and screening by Senior Scientist and Department Head ADT (Alcohol Drugs and Toxicology) in FSNI.
4. Cathinone screening The Forensic Science Laboratory, Dublin by Director of Operations, Forensic Science Laboratory, Garda HQ, Dublin.

Other sources

5. http://www.belfasttelegraph.co.uk/news/local-national/shirley-finlay-murder-trial-paint-flakes-link-accused-to-body-scene-court-told-28477121.html
6. Copy of E-mail from FBI Laboratory, Quantico confirming supply of samples.
7. Analysis and Discrimination of Single-Layer White Architectural Paint Samples", D. M. Wright, M. J. Bradley and A. Hobbs Mehltretter, *Journal of Forensic Sciences*, Volume 58, p 358–364, March 2013.