

*Geological Society, London, Special Publications*

Analysis of soil following a police led open area search and the recovery of a cold case homicide grave

L. J. Donnelly, J. Cassella, D. Pirrie, L. Dawson, G. Blom, A. Davidson, P. Arnold, M. Harrison & C. Hope

DOI: <https://doi.org/10.1144/SP492-2017-337>

Received 10 December 2017

Revised 30 May 2018

Accepted 24 September 2018

© 2019 The Author(s). Published by The Geological Society of London. All rights reserved. For permissions: <http://www.geolsoc.org.uk/permissions>. Publishing disclaimer: [www.geolsoc.org.uk/pub\\_ethics](http://www.geolsoc.org.uk/pub_ethics)

To cite this article, please follow the guidance at <https://www.geolsoc.org.uk/onlinefirst#how-to-cite>

**Manuscript version: Accepted Manuscript**

This is a PDF of an unedited manuscript that has been accepted for publication. The manuscript will undergo copyediting, typesetting and correction before it is published in its final form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the book series pertain.

Although reasonable efforts have been made to obtain all necessary permissions from third parties to include their copyrighted content within this article, their full citation and copyright line may not be present in this Accepted Manuscript version. Before using any content from this article, please refer to the Version of Record once published for full citation and copyright details, as permissions may be required.

## **Analysis of Soil Following a Police Led Open Area Search and the Recovery of a Cold Case Homicide Grave**

Donnelly, L.J.<sup>1</sup>, Cassella, J.<sup>2</sup>, Pirrie, D.<sup>3</sup>, Dawson, L.<sup>4</sup>, Blom, G.<sup>2</sup>, Davidson, A.<sup>2</sup>, Arnold, P.<sup>5</sup>, Harrison, M.<sup>6</sup> and Hope, C.<sup>7</sup>

<sup>1</sup>Chair, IUGS-Initiative on Forensic Geology, geologist@hotmail.co.uk; <sup>2</sup>Staffordshire University, Stoke-on-Trent; UK, <sup>3</sup>Helford Geoscience LLP, Cornwall, UK; <sup>4</sup>James Hutton Institute, Aberdeen, UK; <sup>5</sup>Head of Operations, Regional Scientific Support Services, Yorkshire and Humber, UK; <sup>6</sup>Australian Federal Police & University of Canberra, Australia; University of Keele, UK (formerly UK National Crime Agency, National Search Adviser, retired)

**Abstract:** Police in the UK received information that a person had been reported as missing. Despite a diligent search and investigation, the person was not found. Several years later police received intelligence giving the location of a grave believed to contain the remains of the person previously reported as missing and now believed to be a victim of homicide. This new information suggested the missing person had been murdered and their remains were buried in shallow, unmarked grave. Following a systematic search, the murder victim's body was found at a shallow depth, less than 1 m. Following the forensic recovery of the body soil samples were collected at, beneath the floor of the grave, along strike of the grave, downslope and upslope. Analysis of the soil samples showed elevated levels of putrescine, at nearly 150 ppb in the soils beneath, downslope and for several meters upslope from the body at localities where detector dogs had showed an 'interest' before the grave was discovered. The mineralogical analysis, using integrated automated mineralogy and petrology detected the presence of diagenetic calcite in the soil profile beneath the grave. Additionally, the organic analysis detected the presence of elevated stanols at the grave and down slope.

### **Missing Person: Case Overview**

Police in the United Kingdom received a missing person report. A police investigation was initiated into the circumstances of this disappearance and despite extensive enquiries that included multiple searches, no trace of the missing person was found. The case remained open with enquiries ongoing. All aspects of the investigation were subject to regular and ongoing review. Where any new or different information came to light this was investigated with regular assessment and challenge. This process continued until the known circumstances of the disappearance changed when a person subsequently confessed to police that they had murdered the missing person. The offender disclosed how the grave has been concealed. A detailed description was provided of the location where the victim had been buried together with the circumstances of the burial and the concealment.

This admission and the information provided by the offender led to the identification of a search location that was situated within a large, rural and isolated area of moorland. The site was known to the offender and victim as they had visited many times in the past. Whilst acknowledging the validity and accuracy of the information provided in the admission, it became apparent that the nature of the location, in terms of its appearance and in particular the size and areal coverage of the vegetation had changed considerably in the period that had elapsed since the burial took place. These changes had altered the appearance of the location to such an extent that the exact position of the grave could not initially be identified. Access routes and footpaths across the site, that were available in the past, had also been altered or in some cases removed completely, making orientation on the basis of the description given (from recollections of the site as it appeared in the past), extremely difficult.

## Geology of the Search Area

The search area was located in an area of outstanding natural beauty, within a range of hills that comprise elevated moorland with incised stream valleys. The natural vegetation consisted of deciduous woodland, with low lying, dense ferns up to 2.5 m high, which provided heavy ground cover. The bedrock was Namurian strata, consisting of Kinderscout Grit (known also as the Millstone Grit). This consists of massive, strong, well-jointed, cross-bedded sandstone that forms a prominent escarpment of differentially weathered rock tors overlying shale, mudstone or siltstone. Beneath this escarpment extends a boulder strewn field with individual boulders at least 10-20 m<sup>3</sup>. These moved down-slopes probably during periglacial conditions approximately 10,000 to 13,000 years before present (Donnelly, 2008). These boulders were probably used by the offender to navigate across the landscape and to provide cover during the digging of the grave and burial of the missing person. Peat and periglacial solifluction deposits up to c0.5-1.0m thick are present on the upper and middle valley slopes.

Peat, covering the bedrock, is a biogenic deposit, which represents partially decomposed and disintegrated plant remains. These are preserved under conditions of incomplete aeration and high water content. Peat accumulates where there is high rainfall and the ground is poorly drained. Peat is acidic, with a pH in the range of 3.0 to 5.0, which may have contributed to the preservation of human remains over a period of many years. Peat can also facilitate the relative rapid digging of a grave.

## Search Strategy

### *Geoforensic advances in search*

A search definition is defined as, '*the capability to locate specified targets, using intelligence assessment, systematic procedures, and appropriate detection techniques*' (College of Policing, 2012). In the past decade geological methods, strategies and techniques commonly used in mineral exploration and ground investigations have been applied to law enforcement ground searches for burials. This commenced in 1994 during the search for an unmarked grave in a remote part of the Pennines, a range of hills in northern England (Donnelly, 2003). These new approaches are based on: the development of a Conceptual Geological Model (CGM) for the suspected grave (including the topography, geomorphological processes, properties of the superficial deposits and bedrock and the hydrogeological conditions); an assessment of the conditions of the burial and their detectability (e.g. the degree of preservation or decomposition of a body and associated items); an evaluation of the diggability of the ground; the choice of suitable suite of search assets (e.g. ground and air based observations, detector dogs, geochemical surveys, geophysics and auguring), a specific search methodology, utilisation of Red-Amber-Green (RAG) prioritisation maps and a dedicated forensic recovery and recording team. These are conveniently managed, designed and implemented in pre-search, search and post-search phases. Further information on recent advances in ground searches may be found in Donnelly 2008, 2013a, 2013b, 2013c; Donnelly & Harrison 2010, 2013, 2015, 2017; Harrison & Donnelly 2007, 2008, 2009; Pringle et al., 2012; Ruffell 2012; Hope 2013; Peffers 2013; Ferguson 2013; Cook and Tattersall 2016.

### *Preliminary search*

As noted above, an original search conducted at the time the person was reported missing did not locate the victim. Several years later a preliminary search operation was initiated when the police received intelligence that a victim had been buried in a remote location. This involving a forensic excavation led by a forensic archaeologist. Unfortunately, despite an appropriately planned and resourced operation, the victim's remains was not located.

### *Advance search*

It became apparent to the police that the size of the search area, complexity of the vegetation and changes that had been made to the landscape (e.g. road access and footpaths) had led to confusion and disorientation on the part of the offender. It therefore became necessary for a search planning team to be established. A Police Search Advisor (PolSA) was appointed to lead the planning and management of the search. Throughout the early planning stages, a forensic geologist (co-author) and the National Crime Agency (NCA) National Search Advisor (co-author) were also consulted.

It was necessary for the police to develop a proportionate search strategy. The police were aware that a new search strategy had been developed associated with the search of another missing person who went missing about 50 years earlier (Donnelly, 2003). This search was also being conducted in a similar geographical and geological setting. As such, an open area, scenario based and feature focused search strategy was designed and deployed to locate the victim's grave. This commenced with a site visit and inspection and walk-over survey of the search area. As a result, a planning structure was subsequently agreed, which resulted in the development of a cost-effective, proportionate and pragmatic search strategy and a resulting Standard Operating Procedure (SOP) for searching the location in question.

The initial stages of the planning process engaged are provided below:

- Reconnaissance site inspection and walk-over survey with all interested stakeholders including; the police, Home Office (CAST), NCA, a forensic geologist and forensic archaeologists. Significant landmarks (such as huge boulders) were identified and marked, and logical challenges (such as dense vegetation) were noted.
- An assessment of the vegetation at the site was conducted by a forensic ecologist, and a forensic geologist provided an evaluation of the geology, geomorphology and hydrogeology. It was thought that this might identify changes to the natural balance of the flora that could be linked to the presence of the burial. However, primarily as a result of the period of time that had elapsed, together with the managed works (e.g. new footpaths) that had been carried out at the site, no such changes were identified.
- A desk study review was conducted of all available data, information and intelligence relating to the case. This included comparisons of past edition and most recent ordnance survey maps that were available from the years relating to the date the person was reported as missing.
- The collation of the various archives containing historical aerial imagery was conducted. A comparative analysis of aerial imagery of the location was carried out involving the use of historical images together with images obtained from a new capture. These two sets were analysed and common topographical features were identified. Where it was clear that changes had occurred (for example with footpaths), these changes were highlighted. This analysis was able to confirm the position of some of the key features identified by the offender and therefore helped orientate the search by providing an opportunity to use these key features as reference points for planning.

With the permission of the landowners, the site was then cleared of much of the low-lying vegetation, particularly the Common Fern (*Dryopteris filix-mas*). By clearing the site in this way, other low lying, rocky outcrops were revealed that in some cases appeared to be consistent with landmarks identified in the information provided by the offender.

A topographical site survey was also completed and detailed site maps were then prepared. As a result of this process, it had been possible to formally identify the outer boundaries of the search area and to decide upon the size, shape and specific location of the search sectors within it.

### *Search team*

The search involved the contribution of a number of relevant experts from different scientific disciplines. This included a Senior Investigating Officer (SIO), Police Search Advisor (PolSA), NCA National Search Advisor, Crime Scene Manager, Crime Scene Investigation, Victim Recovery Dog's and their handlers, a forensic geologist, a team of forensic archeologists, forensic geophysicists from

the Home Office (CAST), forensic ecologist and a surveyor. An effective PolSA is considered critical to the success of an operation that is likely to be complex and that will require the integration of skills and subject matter experts from a variety of backgrounds.

This search operation conducted therefore utilised trained and equipped police resources, a clear management structure led by the PolSA, proportionate but very detailed planning, the selection of appropriate resources and the identification of deployment methodologies. This search also benefited from the support and expertise available from scientists engaged from a range of disciplines noted above and who are experienced in working with the police. Policy and credible research is now available from which decision making can be supported and an effective SOP developed. In this way, the search operation was delivered to a high level of assurance.

#### *Standard Operating Procedure (SOP)*

By the PolSA ensuring that each expert, and therefore each discipline, was engaged and contributed, the process meant that the search area was fully assessed from all perspectives. A comprehensive SOP was an absolute pre-requisite for the successful delivery of the search. Any search for a clandestine burial that is conducted within the criminal context must now be carefully planned and managed and therefore requires an effective SOP (Peffer, 2013; Hunter et al., 2013; Donnelly 2013a, 2013b). This described in detail all aspects of the search and should be established before any search activity can begin. This particular SOP included the determination of the size and boundaries of and within the search area; the resources required during the search and in what order they will be deployed. The SOP identified additional resources that will be required (such as vegetation management). The SOP also took into account all forensic considerations and requirements.

#### *Search strategy*

The SOP was based upon the identification of Points of Interest (POI), together with the establishment of a prioritisation order in which the POI would be searched. Resources were then deployed at each POI, in the manner described below. The search strategy was as an exemplar of the 'blended approach' to operations of this type and was delivered in a way that was and is consistent with existing best practice. The structure and strategy employed here should always be considered by specifically law enforcement agencies in the United Kingdom and more broadly elsewhere (Donnelly & Harrison 2015, 2017).

The search required an understanding of the geological characteristics and ground conditions. A conceptual geological model (CGM) for the grave was developed by the forensic geologist. This assisted to determine the geophysical search asset requirements, comprising magnetic, ground conductivity and ground penetrating radar (GPR) methods. The likely conditions of the human remains and associated target items were also evaluated to determine their detectability. The search included a critical evaluation of the offender's account of the burial, analysis of the victim's last movements, and an understating of the dynamics of the burial site. This ensured the search was intelligence informed and based on a hypothesis that was realistic and credible, therefore avoiding speculative searches being conducted. An evaluation of the physical characteristics of the superficial deposits (soils) and groundwater regime enabled diggability to become assessed. This was enhanced through the preparation of a Red-Amber-Green (RAG) site assessment and a diggability survey, which further supported the identification of likely locations for the burial (Donnelly & Harrison 2013).

Victim Recovery Dogs (VRD) were deployed, which are trained to identify gases and volatile organic compounds (VOC) that are released as a result of the process of human decomposition. VRD have been shown to be effective at grave sites that are over fifty years old (personal experience of a case from the UK). In addition, other specialist dog handlers report positive instances that involve periods of time since burial of 20 years, 35 years and in some cases VRD are reported as having the ability to detect remains in 17th century cemeteries. The VRD did not indicate at the grave site. This was thought to be the result of the dense wrapping materials subsequently found at the scene. In addition, the nature of the free draining soil at the site together with the length of time that had passed since the burial is thought to have led to traces of VOC being washed away from the primary deposition site. The VRD handlers engaged in this operation were able to assess the ground conditions present at the search site and then to provide advice with regards to the optimal conditions needed for the most effective deployment of their dogs. Involving specialist officers of this type, and at an early stage in the planning, is important as it allows for the early consideration of all necessary logistical and welfare issues (for example, how to transport the dogs to the site and how long they can effectively work in the prevailing conditions).

A forensic archaeologist and a forensic geologist supported the identification and definition of the external and internal search boundaries. The archaeologist then produced a schedule to illustrate how the search (and excavation) of each POI would be recorded. In visiting each POI, an assessment of the geology was also conducted. In the right circumstances, ground disturbances may sometimes be identified (such as soil colour or textural differences or the settlement of backfilled soil) that could indicate the presence of a grave, although this is not always the case. Unfortunately, no recognisable topographic features of this type were evident in this case. However, by following accepted best practice the process and methodology adopted enabled the development of a robust search methodology at each POI. In addition, the forensic archaeologist provided a court compliant methodology for the logging and recording of activities conducted during the excavation (Cheetham & Hanson 2009).

In addition to the search being scenario-based, the search was also feature-focused. During the reconnaissance visit to the search area this enabled the forensic geologist to identify geological, geomorphological and physical features of the landscape that potentially assisted the offender's modus operandi. A parking lay-by, footpath and several erratic boulders were considered to be relevant reference points in enabling the offender to navigate across the ground to the chosen burial site. The general search philosophy was to progress from the non-invasive to the invasive and from the macro to the micro. This was envisaged to preserve forensic evidence that may be contained on items recovered. The search strategy developed was proportionate, achievable within the permitted time frames, cost-effective, measureable and defensible. This followed the pre-search, search and post-search stages advocated by Donnelly & Harrison 2010, 2015, 2017 (Table 1). Following the deployment of this search strategy the victim's body was found in a shallow and unmarked grave (Figure 1).

**Table 1.** Initial search strategy (modified after Donnelly & Harrison 2010, 2015, 2017).

Phase	Stage	Activity
Pre search	Briefing and set up and desk study	<p>Meeting at police station, provision of case background and case intelligence.</p> <p>Review of case information and intelligence.</p> <p>Collation and analysis of geological data and information, including past and recent topographic maps.</p> <p>Preliminary reconnaissance visit to the search area and walk-over survey.</p> <p>Delineate search area outer boundary, identification of search constraints, and development of a search strategy.</p> <p>Evaluation of diggability, detectability, hydrogeology and the production of a conceptual geological model.</p> <p>Analysis of past and current comparison of air photographs. Identification of landscape changes that had occurred in the time elapsed since the alleged burial took place.</p> <p>Confirm the position of some of the key features identified by the offender and therefore helped orientate the search by providing an opportunity to use these features as reference points for planning.</p> <p>Assessment of vegetation by a forensic ecologist. It was thought that this might identify changes to the flora that could be linked to the presence of the burial. However, primarily as a result of the period of time that had elapsed, together with the managed works (e.g. new footpaths) that had been carried out at the site, no such changes were identified.</p> <p>Topographical site survey completed and detailed plan prepared. This facilitated the identification of the boundaries of the search area and helped to decide upon the size, shape and specific location of the search sectors within it.</p> <p>Obtain access permissions from land owner.</p> <p>Clearance of vegetation in the designated search zone.</p> <p>Identification of Points of Interest (POI)</p>
Search	First deployment of detector dogs	Two detector dogs independently deployed.
	Search area delineation	Define search area and cordon.
	Geophysics I	Deployment of ground penetrating radar.
	Geophysics II	Deployment of fluxgate magnetometer.

	Geophysics III	Deployment of ground conductivity survey.
	Auguring	Auguring at no less than 0.2 m intervals by the Tactical Aid Unit.
	Second deployment of detector dogs	Two detector dogs deployed to detect any volatile organic compounds/scent/odour, facilitated by auguring.
Post search	Recovery	Forensic invasive investigation and recovery of anomalies associated with the detector dog, geophysics or auguring.
	Recording	Recording and cataloging of finds.  Provision of a court compliant methodology for the logging and recording of activities conducted during the excavation.
	Exist strategy	De-brief and site rehabilitation.

### Body Recovery

Support from the UK Centre for Applied Science and Technology (CAST) at the Home Office was sought for this operation. A range of geophysical search equipment was deployed. This included a single channel radar antenna ground penetrating radar (GPR) system, a passive flux gate magnetometer and ground conductivity. It was a feature of the planning on the part of the PoISA that the most appropriate type of search instrument, given the ground conditions and available intelligence, was expected to be the magnetometer. However, the use of a range of instruments provided an opportunity for the team to compare the results generated from each type of equipment. It is also worth noting that personnel from CAST apply a degree of rigor to the deployment of geophysics at a search that is not always a feature of search activity of this type (Ferguson 2013). A grid, in this case using a size of 1 metre x 1 metre, was placed as an overlay at each POI. In ensuring that boundary overlaps were included when planning the area coverage for each survey, a high degree of certainty was achieved that the whole of each sector was and had been surveyed.

A magnetic anomaly was discovered at the grave site following deployment of the magnetometer. This was consistent with the available intelligence. A subsequent desk-based review of the ground conductivity data showed a high conductivity anomaly that was also consistent with magnetometer data. However, the GPR data did not identify a coherent anomaly. The magnetic anomaly identified led to the excavation of the POI site. This was led by a forensic archaeologist and was conducted in partnership with the Crime Scene Investigators. This subsequently led to the recovery of the victim.

### Soil Sampling



In 2008 to 2013 soil (peat) samples were collected and analysed in an area of similar geology as part of an ongoing open area search for another grave. The results indicated the presence of volatile organic compounds (VOC) and possibly leachate from human remains in the ground surrounding a suspected shallow, unmarked, homicide grave (unpublished). The results reduced substantially the search area, such that a focused, manageable and systematic SOP could be deployed. This gave the idea for a similar research based investigation but in this case to investigate for the presence of leachate. The presence of detectable geochemical signatures as a result of human decomposition is thought to be dependent on several variable factors including for example, the context of the burial, age of the grave, cause of death, decomposition/preservations rate, associated items, time elapsed since burial, geology and hydrogeology.

Following the discovery of this grave and the recovery of the victim's body, this presented an opportunity to test the soils to see if there were any mineralogical or chemical signatures that could be detected in the soil.

### *Objective*

Three replicate sets of soil samples were collected from; (a) the top and (b) the base of the soil profile at 12 sampling locations giving a total of 72 soil samples. These were taken from the grave and its immediate vicinity to see if it was possible to detect the presence of leachate or mineralogical changes to the soil associated with human decomposition.

### *Method*

As noted above, the soil sampling methodology was originally developed in connection with another search for a missing person and it was subsequently adopted for this search (Donnelly 2003, Donnelly & Harrison 2017, Vass 2015). Following the forensic recovery of the body from the grave, soil samples were taken at and beneath the floor of the grave, along strike (slope) of the grave, up to 100 m downslope and 200 m upslope (Figure 2). Two soil samples were taken from each auger at the top (upper) and base (lower) of the soil profile. At each locality three sets were taken, A, B and C (Table 2).

At each soil sample location personal protective equipment was checked. The sample collector did not wear deodorant, aftershave, perfume, hand cream or other cosmetics or toiletries that may potentially cause cross contamination of the soil samples. A hat, two pairs of protective gloves, protective glasses and a face mask were worn. If it was not possible to take a soil sample, for example due to the presence of a boulder or the soil was too thin, an alternative site was chosen within a 1m radius, close-by. The information recorded for each locality included; (a) photograph of a pre-prepared, white, laminated, A4 sheet containing the sample number; (b) photographs of the site and surrounding area; (c) date; (d) start and finish time; (e) weather conditions; (f) GPS coordinates; (g) geology (including topography, geomorphology, stratigraphy, lithologies, soil types, structural geology, hydrogeology (groundwater, surface water, water course, seeps, springs, gullies, streams); (h) anthropogenic features (walls, wells, fence lines, field boundaries); (i) vegetation type; and (j) land use.

The steel screw auger and soil extractor was cleaned with deionized water and dried with clean paper towel that was disposed subsequently of. No other solvents were used. The 1.2 m long,

300mm diameter auger was inserted vertically into the ground with sufficient pressure and rotation, until 'refusal' at the bedrock interface (or a pebble, cobble or boulders). The auger was extracted vertically without rotation. The soil profile was observed and inspected in the window of the auger. A description of the soil was recorded including the colour, grain size, mineralogy, texture, fabric, structure and type. Samples were collected from the top and base of the soil profile. Using a pre-cleaned steel blade the soil was transferred into 40ml glass vials that each had a screw cap and polypropylene septa. The depths of the soil samples were recorded. Each vial was subsequently wrapped in bubble wrap and suitably secured for transport with seals inhibiting tampering.

**Fig. 2.** Soil sample locations (blue arrows show general direction of surface and groundwater flow. Dip in degrees show general direction of topography).

**Table 2.** Soil sample depth and locations.

Sample Number	Depth (mmbgl)	Location	Soil Type
1A upper	0-80	Centre of grave	Organic and granular grave backfill
1A lower	220-310		
1B upper	10-80	Grave (feet)	
1B lower	330-390		
1C upper	10-160	Grave (head)	
1C lower	330-400		
2A upper	10-110	West of grave	Brown fibrous peat
2A lower	240-310		Black fibrous peat, sand
2B upper	10-170		Black fibrous peat
2B lower	250-360		Brown sandy peat
2C upper	10-90		Black fibrous peat
2C lower	160-270		Brown sandy peat, sand
3A upper	30-170	East of grave	Brown fibrous peat
3A lower	400-510		Black fibrous peat, sand
3B upper	10-100		Black sandy peat
3B lower	430-530		Brown sand clay
3C upper	20-100		Black sandy peat
3C lower	370-500		Brown sand clay

4A upper	10-120	North of grave	Brown fibrous peat
4A lower	240-320		Black peat, sand
4B upper	10-120		Brown fibrous peat
4B lower	260-350		Black peat, sand
4C upper	10-110		Black sandy peat
4C lower	280-390		Organic clay
5A upper	10-80	North of grave	Brown fibrous peat
5A lower	260-350		Orange sand, organic clay
5B upper	10-120		Brown fibrous peat
5B lower	260-340		Orange sand, organic clay
5C upper	10-80		Brown fibrous peat
5C lower	18-300		Orange sand, organic clay
6A upper	10-160	100m north of grave	Black peat
6A lower	290-350		White-grey clay
6B upper	10-160		Black peat
6B lower	290-350		White-grey clay
6C upper	10-160		Black peat
6C lower	290-350		White-grey clay
7A upper	10-120	South of grave	Black peat
7A lower	460-540		Orange sand and peat
7B upper	10-90		Black peat
7B lower	290-320		Orange sand and peat
7C upper	10-110		Black peat
7C lower	300-350		Orange sand and peat
8A upper	10-90	South of grave	Black fibrous peat
8A lower	280-320		Orange sand and peat
8B upper	10-110		Black fibrous peat
8B lower	250-300		Orange sand and peat

8C upper	10-130		Black fibrous peat
8C lower	210-300		Orange sand and peat
9A upper	10-90	South of grave	Black fibrous peat
9A lower	690-760		Orange sand and peat
9B upper	10-120		Black fibrous peat
9B lower	700-840		Orange sand and peat
9C upper	10-100		Black fibrous peat
9C lower	500-610		Orange sand and peat
10A upper	10-90	South of grave	Peaty sand
10A lower	290-360		White-grey clay and sand
10B upper	10-90		Peaty sand
10B lower	210-300		White-grey clay and sand
10C upper	0-80		Peaty sand
10C lower	250-280		White-grey clay and sand
11A upper	10-90	South of grave	Black fibrous peat
11A lower	480-520		Brown sand
11B upper	10-110		Black fibrous peat
11B lower	490-540		Brown sand
11C upper	10-110		Black fibrous peat
11C lower	390-460		Brown sand
12A upper	10-70	200m south of grave (control)	Black peat, sand, clay
12A lower	280-350		Sandy clay
12B upper	10-130		Black peat, sand, clay
12B lower	310-380		Sandy clay
12C upper	10-90		Black peat, sand, clay
12C lower	310-390		Sandy clay

## Soil Mineralogy

### *Sample analysis*

The mineralogy of 24 soil samples collected from the grave site and surrounding area was determined using automated mineralogy. A small subsample was removed from each vial using a single using sterile spatula and placed into a 30 mm diameter plastic mould. The samples were then gently dried at 50°C before being gently disaggregated within the mould. The samples were then mixed with epofix resin and left to degas in a pressure vessel for 24 hr. Each sample was then back-filled with araldite resin and left to cure in a laboratory oven at 50°C for 2 hrs. The samples were then polished and carbon coated prior to mineral analysis. Automated mineral analysis was carried out using QEMSCAN technology (Pirrie *et al.*, 2009, 2014). The samples were measured using the particle mineral analysis (PMA) measurement mode, with a beam stepping interval of 6 µm with only particles between 20 and 400 µm being measured. During the automated measurement the sample area is divided into a series of fields; the measurement was set to stop collecting data once a threshold of 4000 mineral particles had been achieved, although the software continues collecting data until the area of the last field has been measured. Consequently, between 4001 and 4078 individual mineral grains were measured (see Table 3); two samples (2CU and 8CU) contained <4000 mineral grains with 981 and 2159 mineral grains respectively. The mineralogical dataset is based on the acquisition of between 117941 and 930359 individual energy dispersive X-Ray spectra per sample. Carbon-based particles such as organic grains are not measured during automated analysis.

Once a sample is measured it is compared with a database and the raw data are processed; the modal mineralogical data are reported for defined mineral groupings; those used in this study are shown in Table 4. Data outputs include; (a) modal mineralogy, (b) particle size, (c) mineral association data and (d) false colour particle images. The prepared polished blocks for representative samples were also examined using a manual scanning electron microscope and imaged.



	Calcite	0.82	0.00	0.00	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Monazite	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.01	0.00	0.00	0.02
	Fe sulphides	0.02	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.04	0.00	0.00	0.01
	Barite	0.17	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Others	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.01	0.00	0.00

**Table 3a.** Modal mineralogy data based on automated SEM-EDS analysis (cont.)

Measurement	Sample Code	7CL	7CU	8CL	8CU	9CL	9CU	10CL	10CU	11CL	11CU	12CL	12CU
	No. Particles	4033	4027	4003	2159	4067	4010	4057	4066	4021	4023	4069	4078
	No. Analysis Points	794609	869183	835082	588049	330544	613526	727177	333641	813821	794542	530220	535309
<b>Mineral (%)</b>	Quartz	80.99	84.18	78.14	82.60	51.54	69.23	70.06	58.41	73.23	68.52	85.77	88.38
	Plagioclase feldspar	3.83	3.00	4.12	2.86	15.01	9.03	8.17	11.76	6.65	8.14	4.24	3.20
	K feldspar	13.01	10.96	13.79	12.56	19.97	14.84	12.56	17.96	15.66	18.77	6.56	5.71
	Muscovite	1.40	1.19	2.24	1.35	4.10	2.92	3.09	4.18	2.87	2.69	1.20	0.99
	Biotite	0.06	0.06	0.14	0.11	2.13	0.83	1.34	1.39	0.29	0.49	0.09	0.03
	Kaolinite	0.28	0.21	0.80	0.21	1.49	1.22	1.93	2.84	0.71	0.50	1.45	1.36
	Fe Al silicates	0.04	0.09	0.25	0.09	4.40	1.35	2.53	2.97	0.36	0.52	0.25	0.11
	Ca Fe Al silicates	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Ca Mg Fe silicates	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.01	0.00	0.00	0.01	0.00
	Zircon	0.07	0.06	0.07	0.06	0.18	0.02	0.02	0.04	0.05	0.05	0.08	0.03
	Rutile	0.29	0.25	0.33	0.14	0.57	0.29	0.14	0.21	0.13	0.17	0.28	0.18
	Ilmenite	0.01	0.00	0.11	0.00	0.09	0.10	0.04	0.04	0.01	0.02	0.01	0.01
	Fe-Ox/CO <sub>3</sub>	0.00	0.00	0.01	0.01	0.25	0.15	0.09	0.16	0.02	0.11	0.05	0.00
	Chromite	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.01	0.00	0.01



	Mn phases	0.00	0.00	0.00	0.00	0.24	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Calcite	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00
	Monazite	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Fe sulphides	0.00	0.00	0.00	0.00	0.02	0.01	0.01	0.01	0.00	0.00	0.00	0.00
	Barite	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
	Others	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00

**Table 4.** Mineral categories used to process the soil mineralogy data.

Mineral Category	Description
Quartz	Quartz and other SiO minerals
Plagioclase feldspar	Plagioclase feldspars (Na,Al,Si,O to Ca,Al,Si,O)
K feldspar	K-feldspars (K,Al,Si,O)
Muscovite	Muscovite mica
Biotite	Biotite and phlogopite
Kaolinite	Kaolinite/halloysite/dickite
Fe Al silicates	Any phase with Fe,Al,Si,Mg,O or Fe,Al Si,O such as chlorite
Ca Fe Al silicates	Any Ca Al Fe Silicates such as epidote and zoisite
Ca Mg Fe silicates	Any phase with Ca,Mg,Fe,Si, (with or without Fe & Al) such as amphiboles and some pyroxenes
Zircon	Any phase with Zr, Si and O
Rutile	Any phase with Ti,O
Ilmenite	Any phase with Fe,Ti,O, may have low Mn
Fe-Ox/CO <sub>3</sub>	Fe oxides and carbonates such as siderite, hematite, magnetite, goethite, Ti-magnetite and any other Fe-oxide/carbonate
Chromite	Chrome spinel
Mn phases	Any phase with Mn,O, Mn silicates and Fe-Mn oxides
Calcite	Calcite
Monazite	Any phase with Ce,La,P with or without Th
Fe sulphides	Pyrite/pyrrhotite and alteration phases such as jarosite
Barite	Any phase with Ba and S
Others	Any other mineral not included above

## Results

### Detrital mineralogy and spatial variability

The modal mineralogy for the 24 samples analysed is provided in Table 4 and shown graphically in Figure 3; major phases form >10% of the sample, minor 1-10% and trace minerals <1%.

Representative particle images are provided in Figure 4. The mineralogy of all of the samples is dominated by major quartz along with major/minor K-feldspar and plagioclase feldspar. Muscovite occurs as a minor phase in all of the samples along with minor/trace biotite, kaolinite and FeAl silicates.

Trace minerals present in most of the samples are: rutile, ilmenite, FeOx/CO<sub>3</sub> and zircon; CaFeAl silicates, CaMgFe silicates, chromite, Mn phases, calcite, monazite, Fe sulphides and barite occur rarely in some of the samples analysed. The overall modal mineralogy is entirely consistent with the underlying Carboniferous sandstone bedrock (Hallsworth & Chisholm 2008; Tyrell et al., 2006). None of the soil mineralogy is indicative of sediment supply to the soil profiles as a result of glacial processes during the Quaternary.

Despite the relatively limited range of minerals present within the soil samples, when the modal data are plotted relative to the sampling locations, clear spatial variations in the soil modal mineralogy are observed (Figure 5).

**Fig 3.** Modal mineralogy of the analysed soil samples. Note that the data are normalised to % but are based on the analysis of >4000 mineral grains per sample.

**Fig 4.** Representative QEMSCAN mineral particle images arranged by area for soil sample 1CL.

**Fig 5.** Spatial variation in soil mineralogy around the clandestine grave site.

Based on the modal mineralogy, four “groups” of soils can be described which relate to different sun-environments around the grave site. The four soil samples collected in a localised area around the grave form a discrete group, with typically 64-77% quartz, 14-19% K feldspar, 3-12.6% plagioclase feldspar, 2-4% muscovite, 0.1-1.7% biotite and 0.3-2.0% kaolinite. Samples 1CL and 4CU are slightly different to this overall mineralogical group. Sample 1CL is less quartz-rich and more K-feldspar-rich than the other samples within this group. It also contains more muscovite, biotite and FeAl silicates than the other soil samples. Trace abundances of CaFeAl silicates, CaMgFe silicates and BaSO<sub>4</sub> (barite) occur in this sample but not the others analysed within this cluster. In addition, calcite has abundance in Sample 1CL of 0.82%; this phase is absent from the other samples in this group, other than in Sample 2CU in which it has an abundance of 0.05%. Sample 4CU also differs from the other samples in this group, having more abundant plagioclase feldspar, kaolinite and FeAl silicates and less abundant K feldspar (Table 2, Figure 1).

Soil samples 5 and 6 collected from a woodland area to the north of the grave site, are both much less quartz-rich than the other samples analysed but are also very variable to each other. Quartz abundance ranges between 32 and 67.9%, with 7-17% K feldspar and 7.9-35% plagioclase (sample 6 is very enriched in plagioclase and kaolinite when compared with the other samples analysed). It should be noted that the clear correspondence between plagioclase and kaolinite abundance is in part a function of the partial alteration of plagioclase feldspar to kaolinite within the samples as a result of geological processes. The four samples contain between 2.5 and 4.0% muscovite and 0.1-6.9% biotite. Sample 5CL is significantly enriched in FeAl silicates (18.5%) when compared with the other samples from this area (0.7-5.1%) (Table 3, Figure 3).

In contrast, soil samples 7 and 8 collected from locations immediately to the SE of the grave are distinctive in being much more quartz-rich than all of the other samples analysed with 78-84% quartz, 10.9-13.8% K-feldspar, 2.8-4.1% plagioclase and 1.1-2.2% muscovite (Table3, Figure 3).

Soil samples 9-12 were collected again in a south east direction moving away from the grave site (Figure 2 and 5). This group is somewhat variable in modal mineralogy, with 51-88% quartz, 5.7-19.9% K feldspar, 3-15% plagioclase, 1-4.1% muscovite and 0.5-2.8% kaolinite, but they can be distinguished from the other soils based on an overall increased abundance of FeAl silicates with between 0.1 and 4.4%.

The overall soil modal mineralogy data shown in Figures 3 and 5, demonstrate that although the area has the same underlying bedrock geology, there are systematic variations in the soil mineralogy. Thus if soils had been recovered from, for example, items of clothing, or a digging tool from a potential offender, then the spatial soil variations observed would allow the potential for that soil to have come from the immediate area of the grave to have been tested forensically.

#### *Soil sample 1CL*

When the 24 soil samples collected from the area are compared, it is clear that there are several distinct attributes to soil sample 1CL, the soil sample recovered from the base of the clandestine grave. Barite occurs as a trace mineral in sample 1CL but is not present within the other samples analysed. In Sample 1CL the barite occurs as two discrete angular barite grains. However, most noticeably, a mineral reporting to the category calcite is much more abundant within soil sample 1CL than in the other soil samples analysed. Soil sample 1CL contains 0.82% calcite. In the other samples analysed, calcite is only observed in samples 2CU (0.05%) and 9CU (0.01%). Calcite can occur within Upper Carboniferous sedimentary rocks as either a diagenetic phase or potentially as fossil bioclasts and as such could be present in associated soil samples. However, previously published work suggests that calcite is rare or absent within the Kinderscout Grit (e.g. Tyrell et al., 2006) and given the strongly acidic nature of the prevailing groundwaters in this area, any calcite released from weathering of the bedrock lithologies may have a limited residence time before being dissolved. Texturally the observed grains of calcite are either made up of numerous small crystals with pore spaces between the crystals or have a skeletal form (Figure 6).

There are two potential causes for the mineralogical variation seen in the sample from the base of the clandestine grave. (1) It is possible that mineral particles were introduced into the base of the grave, either during the initial clandestine burial, or subsequently during the body recovery phase. However, as can be seen in Figures 3 and 5, the overall mineralogy of sample 1CL is consistent with the other soil samples collected in this area; the only observed differences being the grains of barite and the increased abundance of calcite. (2) The texture of the calcite grains, in particular the distinctive skeletal form (Figure 5b) is consistent with the calcite having been precipitated *in-situ*. Calcite is a common mineral in an archaeological context and is commonly considered to be either; (a) derived from the associated geological units, (b) derived from biogenic sources such as invertebrate shells or (c) from anthropogenic sources such as lime plaster / mortar - the primary lime (CaO) can convert to calcite, along with wood ash. When wood is heated to around 500°C calcium oxalate crystals in the wood alter to form calcite (e.g. Regev et al., 2010). Calcite also commonly occurs as a diagenetic product during the alteration of bone (e.g. Trueman et al., 2004). The textural appearance of the calcite, along with the restricted occurrence of the calcite to the soil

sample collected from the base of the grave, supports an interpretation that the calcite is diagenetic in origin, and relates to the decay processes. Further work is required to constrain the exact, potentially microbial, processes operating.

**Fig. 6.** Calcite grains from soil sample 1CL from the base of the clandestine grave. (A) QEMSCAN particle images arranged by calcite (dark blue) and area. (B-E) Scanning electron microscope images of fragmental (B, D) and skeletal (C, E) calcite grains. The calcite is interpreted to be diagenetic in origin.

## Leachate

Decomposition products putrescine and cadaverine have regularly been reported as markers of decomposition and have also been suggested to be one of the key chemicals in locating human remains by Victim Remains Detection (VRD) dogs. (Vass et al. 2002; Dent et al. 2004; Statheropoulos et al. 2007; Stadler et al. 2012; Tipple et al. 2014). Putrescine and cadaverine are biogenic amines that were first identified in 1885 through isolation from decomposing animals (Brieger, 1885) and are nitrogen-containing compounds present in vegetables, microbial and animal cells, which are mainly formed through decarboxylation of amino acids but also through transamination of ketones and aldehydes (Karovicova & Kohajdov, 2005; Innocente *et al.* 2006).

Biogenic amines are acknowledged indicators of decomposition in the food industry and the detection and quantification of putrescine and cadaverine from various complex matrices using gas chromatography (GC) has been widely published in food, wine, environmental and physiological studies (e.g. Fernandes & Ferreira 2000; Ngim et al. 2000; Kumudavally et al. 2001; Ali Awan et al. 2008; Cunha et al. 2011). The paucity of reporting in the forensic literature of the detection of putrescine and cadaverine is likely due to their physical-chemical properties as these compounds require derivitisation for optimal response on GC systems (Vass et al. 1992; Dekeirsschieter et al. 2009; Swann et al. 2010a; Swann et al. 2010b; Paczkowski & Schüt, 2011). A robust and reliable method to detect these biogenic amines in a forensic context has however, been applied to the identification of a mock-clandestine grave and has demonstrated the use of these markers in locating a grave and furthermore, that the compounds are detectable over 15 years' post-burial (Blom et al. 2015). This technique has therefore been applied to the cold case grave in the current study. With regard to the use of ionic chemicals as indicators of decomposition, conductivity has long been used as a geophysical technique (Pringle et al. 2010, 2015) who showed that elevated levels of soil conductivity persist over a period of many years, which indicates the potential for ionic analysis in such a context. A number of studies have investigated ionic species as markers of decomposition with Vass (1992) and Aitkenhead-Peterson (2011) utilising ion chromatography while others employed other methods (e.g. Hopkins 2000; Benninger 2008). As ion-chromatography has the advantages of a simple sample preparation, to detect multiple ions with a high degree of selectivity, this technique was also applied to the samples from this case.

## Method

For the analysis by ion-chromatography, a portion of each soil sample was weighed into 20 ml labelled glass vials (unlidded) and dried in the oven at 60°C for 15 hours. After drying the soil was re-weighed (to determine the moisture content) then ground and sieved and a 3.0 g portion of each prepared soil weighed into a centrifuge tube and 15 mL of deionised water added. The samples were

then sonicated for 15 minutes and centrifuged for 20 minutes after which the aqueous layer was collected and filtered. The extracted samples were analysed using a Dionex ICS 900 Ion Chromatograph with an IonPac® AS22 Column (2 x 250 mm), 2N Sulphuric Acid regenerant (Thermo), 4.5 mM Na<sub>2</sub>CO<sub>3</sub> / 1.4 mM NaHCO<sub>3</sub> eluent (Thermo) and using electrical conductivity detection.

Five samples were analysed by gas chromatography mass spectrometry (GC-MS): those taken from the upper layer of soil at the grave site (site 1); the site immediately north and in a downslope direction beyond a large boulder of sandstone (site 4); a site approximately 100 m further to the north (site 6); a site upslope from the grave at or near dog indications (site 8) and a control site upslope and about 200 m from the grave (site 12). Prior to extraction, 1.0 g soil was dried with 1.0 g anhydrous sodium sulphate to prevent the amines from volatilising; the amines were then extracted following the same procedure as for the ion-chromatography. For the derivatisation of the putrescine and cadaverine, 1 mL of each aqueous soil extract was transferred into a 4 mL glass vial and the pH adjusted to pH 11. A 500 µL portion of 10% pentafluorobenzaldehyde (PFB) in acetonitrile was added to each sample, the vial covered with aluminum foil placed in an oven at 60°C. After one hour of heating the vials were transferred to an ice bath and 1 mL of 0.1M sodium hydroxide was added to each vial along with 1 mL of hexane which had been spiked with the internal standard, undecane, at 0.5%. The vials were resealed and vortexed for 15 seconds and the organic layer of the solution was then transferred into 2 mL autosampler vials. This process was performed in duplicate to test the reproducibility of the derivatisation process. Samples were also prepared of a negative control (deionized water), and positive control / calibration standards containing putrescine and cadaverine at 1 mMol L<sup>-1</sup>.

Analysis of the samples and standards was performed using a Perkin Elmer Clarus 500 GC-MS using a Supelco SLB-5MS 30 m x 0.32 mm 0.25 µm column. Quantification for the biogenic amines was conducted using m/z fragments 181, 222 and 263 (cadaverine) and 181, 208 and 249 (putrescine) with the limit of quantification for putrescine determined as 86 ppb, and for cadaverine 92 ppb.

## Results

Analysis by GC-MS demonstrated that, in the upper layer of soil taken from the grave site putrescine was detected at a concentration of approximately 150 ppb, a level far exceeding that of the other samples analysed. Traces of putrescine were also detected in the soil downhill from the grave (sample 6) and uphill from the grave at or near a dog indication (sample 8) although levels at these sites were below the limit of quantification. Putrescine was not detected at the remaining sites (site 4 immediately downslope, or the control site) and cadaverine was not detected in any of the samples (Figure 7). For the ion-chromatography, the gravesite generally showed lower levels of soluble anions compared to the immediate surrounding area, possibly due to improved drainage of the soil following excavation. However, there were slightly elevated levels of nitrate in the lower levels of the grave soil compared to samples in the immediate area and elevated levels of phosphate were found in the sample taken from the upper layer of soil immediately west of the grave (sample 2). In addition, elevated levels of phosphate were detected in sample 8 from at or near a dog indication position upslope from the grave.

An analysis of soil samples from a recently excavated clandestine burial demonstrated that the proposed decomposition marker chemical putrescine was present in soil where a body had been present for more several years. These findings are consistent with earlier studies indicating the

potential of biogenic amines as markers of decomposition. The findings also suggest that putrescine migrated through the soil matrix as it was detected downslope and for several meters upslope at localities where detector dogs had showed an interest before the grave was discovered, thereby also corroborating that indication. Phosphate levels were also found to be higher at the site of this dog indication but otherwise data from analysis by ion chromatography was less conclusive. However, nitrate was found deep within the grave cut. Cadaverine was not identified in any of the samples analysed.

**Fig 7.** GC-MS chromatograms for analysis of putrescine and cadaverine

### **Soil Chemical Markers**

#### *Sterol Analysis Method*

The organic analysis was conducted according to the method of von der L  he et al. 2013. The elemental analysis method, after acid digestion, was using inductively coupled plasma - optical emission spectroscopy (ICP-OES) or inductively coupled plasma - mass spectroscopy (ICP-MS) to determine their concentration.

#### *Results*

Organic markers have been shown to be useful in the identification of body decomposition (Dawson 2017; von der L  he et al. 2013, 2017). Several main stanols in the samples examined were detected, this were: coprostanol, epicoprostanol, cholesterol, 24-ethylcoprostanol, 24-ethylepicoprostanol, campesterol, B-sitostanol/stigmastanol.

Further focus was on coprostanol and epicoprostanol as they have been shown by our research (data unpublished) to be the most consistent biomarker for humans. Both coprostanol and epicoprostanol were shown to be present in; the grave samples (upper and lower layers (1BU and 1BL)), one sample near to the grave (sample 2, west (2BU and 2BL)) and one sample further away from the grave (sample 5, lower, north, 100m (5BK)). These two compounds are absent from all the other samples analysed including the control soil sample.

#### *Elemental analysis by ICP-MS/ICP-OES, C and N isotope analysis*

The elemental concentrations were assessed on two samples, 1BU taken from the grave and 12BU taken from the control, however there were no obvious differences (Table 5). In addition, carbon and nitrogen isotope analysis was carried out using GC IR MS; however, there was no obvious effect on the carbon and nitrogen isotopes.

**Table 5.** Elemental concentrations for samples 1BU and 12 BU

Sample	Ag mg/kg	As mg/kg	Ba mg/kg	Cd mg/kg	Co mg/kg	Cr mg/kg	Cu mg/kg	Hg mg/kg	Mo mg/kg
1BU, grave, 1-15cm depth	0.12	2.13	12.83	0.06	0.28	18.01	3.54	<0.045	0.66
12BU, control, upslope 220m from grave	0.10	1.51	17.50	0.18	0.13	56.21	2.90	<0.045	0.68

Sample	Ni mg/kg	Pb mg/kg	Pt mg/kg	Se mg/kg	Zn mg/kg	Al mg/kg	B mg/kg	Ca mg/kg	Fe mg/kg
1BU, grave, 1-15cm depth	2.20	34.36	0.05	<0.05	7.82	1943	12.77	193.5	2815
12BU, control, upslope 220m from grave	2.54	37.81	0.04	0.44	6.26	1936	11.23	416.6	1341

Sample	K mg/kg	Mg mg/kg	Mn mg/kg	P mg/kg	S mg/kg	Ti mg/kg
1BU, grave, 1-15cm depth	294.7	109.1	18.87	183.9	313.9	23.97
12BU, control, upslope 220m from grave	355.7	139.8	31.33	219.8	530.3	30.68

## Conclusions



In this case, the full range of available police assets were used including police search teams and subject matter experts specialised in search, including forensic geology. The search was open area, scenario based, feature focused and it was designed and deployed for a person who went missing several years earlier. The victim had been murdered and their remains were found in a shallow, unmarked grave, in a remote location. Preparatory work by a forensic geologist, supporting a PolSA and NCA National Search Adviser, produced a search strategy and developed a conceptual geological model (CGM) for the grave. This supported the selection of appropriate geophysical search equipment. Using police intelligence, these were subsequently deployed by CAST (UK Home Office) and recovery was assisted by a forensic archaeologist.

Resources and search assets were deployed both independently during the planning phase and then sequentially during the main search, when the highest level of assurance was required. The approach taken in planning and then in delivering this search operation was successful. It allowed those involved to overcome a number of difficulties presented by the challenging rural location. In particular, by the offender's confusion and misinterpretation that arose as a result of natural and man-made changes to the landscape since the victim had been buried. A thorough and comprehensive planning process involved the development of an SOP that facilitated the 'blending' of expertise from a variety of sources. This enabled the effective delivery of a successful search. Involvement of relevant scientific disciplines prior to the ground search activity being conducted ensured the most suitable assets were used in a proportionate and appropriate way. This integrated search strategy is a good example of the application of what is now acknowledged best practice, and reaffirms the view that a phased approach, involving desk-based planning, is vital as a prelude to any physical search activity. This continues to be used with considerable success by law enforcement agencies in the UK and in some cases, internationally

Following the discovery of the victim, soil samples were taken after the body had been forensically recovered from below and in the vicinity of the grave. The mineralogy, leachate. Elemental composition and organics were analysed and results compared to the equivalent control sample. The mineralogical analysis detected the presence of calcite (at an abundance of less than 1%) in the soil profile beneath the grave. No calcite was detected using automated analysis in any of the other samples analysed. The texture of the calcite as imaged using scanning electron microscopy indicates that it is likely to be diagenetic in origin, precipitated within the soil profile rather than being detrital in origin. Experimental techniques were developed to extract anions and bio-amines from the soil samples. The data showed elevated levels of putrescine, at nearly 150 ppb at the grave, downslope and for several meters upslope at localities where detector dogs had showed an 'interest' before the grave was discovered. Furthermore, organic analysis detected the presence of elevated stanols at the grave site and downslope from the grave.

These analytical techniques support the theory of the changes in soil mineralogy, movement of compounds associated with body decomposition and the generation of leachate from clandestine graves. It is postulated the leachate subsequently flows from the grave as a plume controlled by the geology, topography and hydrogeology.

This blended approach to search and the development of a pragmatic, practicable and cost effective method to detect leachate, organic compounds and mineralogical changes in the soil profile has been advocated as a method that can assist with open area ground searches for homicide graves

(Donnelly & Harrison 2017) and subsequent use as potential evidence. It is recommended that a multi proxy approach is used: where a combination of complementary methods are adopted to allow the best possible chance of a positive grave location identification. Observations and results published in this paper and other case work are supportive of this hypothesis. This search method is still in its infancy and further development. More research is recommended at human decomposition research facilities and through historic unsolved investigations and when permitted in case work. This approach has potential to assist police-led, open area, ground searches for the detection of human criminal burials.

## Acknowledgments

The authors would like to acknowledge: the National Crime Agency and Police National Search Centre; Yorkshire and Humberside Regional Scientific Support Services; North Yorkshire Police (Det. Supt. Dai Malyn); West Yorkshire Police (Det. Ch. Insp. Adrian Taylor, Det. Supt. Mark Ridley; Sgt. David Gillan, Sgt. Andrew Shaw, Mr Andrew Morrow); Home Office Centre for Applied Science and Technology; University of Cranfield and Aleto Forensics (Dr Tim Fletcher and Dr Karl Harrison), Mr Max Krens, Ms Stephanie van Rens, Mr Vincent Voorwerk and Mrs Jasmine Ross.

## References

- Aitkenhead-Peterson, J.A., Owings, C.G., Alexander, M.B., Larison, N. & Bytheway, J.A. 2012. Mapping the lateral extent of human cadaver decomposition with soil chemistry. *Forensic Science International*, 216(1–3), pp.127–134.
- Ali Awan, M., Fleet, I. & Paul Thomas, C.L. 2008. Determination of biogenic diamines with a vaporisation derivatisation approach using solid-phase microextraction gas chromatography–mass spectrometry. *Food Chemistry*, 111(2), pp.462–468.
- Blom, G., Davidson, A., Cassella, J.P. & Pringle, J.K. 2015. Incorporating chemical methods to aid in locating clandestine human burials, paper presented to the European Academy of Forensic Science, 6–11 September.
- Bommarito, C.R., Sturdevant, A.B. & Szymanski, D.W., 2007. Analysis of Forensic Soil Samples Via High-Performance Liquid Chromatography and Ion Chromatography. *Journal of Forensic Sciences*, 52(1), pp.24–30.
- Brieger, L. 1885. Weitere Untersuchungen über Ptomaine, A. Hirschwald.
- Cheetham, P. & Hanson, I. (2009). Excavation and recovery in forensic archaeological investigations. In *Handbook of Forensic Anthropology and Archaeology*, Blau and Uberlaker, D (Eds). 2nd Ed, 2016. Pp 141 to 149. Routledge. London.
- College of Policing 2012. Manual of Guidance for Police Search Advisers (PoISA), Vol One. College of Policing Ltd.
- Cook, T & Tattersall, A. 2016. Blackstone's Senior Investigating Officer's Handbook. 4th Ed. Oxford University Press. Oxford, UK.

Cunha, S.C., Faria, M.A. & Fernandes, J.O. 2011. Gas Chromatography–Mass Spectrometry Assessment of Amines in Port Wine and Grape Juice after Fast Chloroformate Extraction/Derivatization. *Journal of Agricultural and Food Chemistry*, 59(16), pp.8742–8753.

Donnelly L. J. 2013a. Use of Geology in Forensic Science: Search to Locate Burials. In: Elias S.A. (ed.) *The Encyclopedia of Quaternary Science*, Vol. 4 pp. 521-534. Amsterdam: Elsevier.

Donnelly, L. J., 2013b, The Applications of Forensic Geology and Geophysics for Police and Law Enforcement Ground Searches: Second International Conference on Engineering Geophysics, United Arab Emirates University, Al Ain City Municipality, 24-27 November 2013, pp. 82-86, Keynote Presentation

Donnelly, L. J., 2013c, The Design and Implementation of a High Assurance Forensic Geology and Police Search to Verify Total Gold Recovery Following the Discovery of the Staffordshire (Anglo Saxon) Gold Hoard. In: Pirrie, D., Ruffell, A. R. & Dawson, L. (2013). *Environmental and Criminal Geoforensics*. Geological Society of London, Special Publication, v.384, pp.195-208.

Donnelly, L. J., 2008. Communication in geology: A personal perspective and lessons from volcanic, mining, exploration, geotechnical, police and geoforensic investigations. In: Livermann, D.G.E., Pereira, C.P & marker, B. (eds) *Communicating Environmental Geoscience*. Geological Society, London, Special Publication, v.305, pp.107-121.

Donnelly, L. J. 2003. The applications of forensic geology to help the police solve crimes. *European Geologist*. *Journal of the European Federation of Geologists*, December 2003, 16, 8 - 12.

Donnelly, L. J. & Harrison, M. 2017. Ground Searches for Graves and Buried Targets Related to Homicide, Terrorism and Organised Crime. In; Donnelly, L. J. (Guest Editor) 2017. *Forensic Geology Themed Issued. Episodes*. *Journal of the International Union of Geological Sciences*, June 2017, 106-117.

Donnelly, L.J., & Harrison, M., 2015, A collaborative methodology for ground searches by a forensic geologist and law enforcement (police) officer: detecting evidence related to homicide, terrorism and organized crime: *Proceedings of the 3rd International Conference on Engineering Geophysics, Session on Forensic Geosciences, Al Ain, UAE, FG02*, pp.260–268.

Donnelly, L.J., & Harrison, M., 2013, Geomorphological and geoforensic interpretation of maps, aerial imagery, conditions of diggability and the colour coded RAG prioritisation system in searches for criminal burials. in Pirrie, D., Ruffell, A.R., and Dawson, L. (eds.), *Environmental and criminal geoforensics: Geological Society of London, Special Publication*, v. 384, pp. 173–194.

Donnelly, L.J. & Harrison, M., 2010, Development of geoforensic strategy & methodology to search the ground for an unmarked burial or concealed object: *Emergency Global Barclay media Limited*, July 2010, pp. 30–35.

Dawson, L.A. & Hillier, S. 2010. Measurement of soil characteristics for forensic applications. *Surface and Interface Analysis*. 42, 363-377.

Dawson, L.A. & Mayes, R.W. 2014. *Criminal and Environmental Soil Forensics*, In: B Murphy & R Morrison (eds), *Introduction to Environmental Forensics*, 3rd Edition. Academic Press.

Degreeff, L.E. & Furton, K.G., 2011. Collection and identification of human remains volatiles by non-contact, dynamic airflow sampling and SPME-GC/MS using various sorbent materials. *Analytical and Bioanalytical Chemistry*, 401(4), pp.1295–1307.

Dekeirsschieter, J., Verheggen, F.J., Gohy, M., Hubrecht, F., Bourguignn, L., Lognay, G. & Haubruge, E. 2009. Cadaveric volatile organic compounds released by decaying pig carcasses (*Sus domesticus* L.) in different biotopes. *For Sci Int*, 189(1-3), pp.46–53.

Dent, B.B., Forbes, S.L. & Stuart, B.H. 2004. Review of human decomposition processes in soil. *Environmental Geology*, 45(4), pp.576–585.

Ferguson, M. 2013. The UK Home Office (Centre for Applied Science and Technology) support to the police search community. Specialist ground and marine search technology. Abstract - Paper presented at the Second International Conference on Engineering Geophysics, Al Ain, Abu Dhabi, 2013.

Fernandes, J.O. & Ferreira, M.A, 2000. Combined ion-pair extraction and gas chromatography, mass spectrometry for the simultaneous determination of diamines, polyamines and aromatic amines in Port wine and grape juice. *Journal of Chromatography A*, 886(1–2), pp.183–195.

Hallsworth, C.R. & Chisholm, J.J. 2008. Provenance of late Carboniferous sandstones in the Pennine Basin (UK) from combined heavy mineral, garnet geochemistry and palaeocurrent studies. *Sedimentary Geology*, 203(3), 196–212.

Harrison, M. & Donnelly, L.J. 2009. Locating concealed homicide victims: developing the role of geoforensics. in Ritz, K., Dawson, L., and Miller, D. (eds.), *Criminal and environmental soil forensics: Soil Forensics*, Springer, pp. 197–219.

Harrison, M. & Donnelly, L.J. 2008. Buried homicide victims: Applied geoforensics in search to locate strategies: *The Journal of Homicide and Major Incident Investigations*. Produced on behalf of the Association of Chief Police Officers (ACPO) Homicide Working Group, by the National Policing Improvement Agency (NPIA).

Harrison, M. & Donnelly, L.J. 2007. The coordinated approach of multi-disciplinary teams to locate concealed victims of homicide, developing the role of forensic landscape investigation: *Soil Forensic International*, October 30–November 1, The Macaulay Institute, Herriot Watt University, Edinburgh.

Hope, C. 2013. The UK Police Service Approach to Specialist Search, Structure, Training and Capability of Search Assets. Abstract - Paper presented at the Second International Conference on Engineering Geophysics, Al Ain, Abu Dhabi, 2013.

Hopkins, D.W., Wiltshire, P.E.J. & Turner, B.D. 2000. Microbial characteristics of soils from graves: an investigation at the interface of soil microbiology and forensic science, *Applied Soil Ecology* 14, 283–288

Hunter, J. Simpson, B & Sturdy Colls, C. 2013. *Forensic Approaches to Buried Remains*. Wiley Blackwell. Chichester. UK.

Innocente, N., Biasutti, M., Padovese, M. & Moret, S. 2006. Determination of biogenic amines in cheese using HPLC technique and direct derivatization of acid extract. *Food Chemistry*, 101(3), pp.1285–1289.

Karovicova, J. & Kohajdova, Z. 2005. Biogenic Amines in Food. *ChemInform*, 36(34), pp.479–484.

Kumudavally, K. V., Shobha, A., Vasundhara, T.S. & Radhakrishna, K. 2001. Chromatographic analysis of cadaverine to detect incipient spoilage in mutton. *Meat Science*, 59(4), pp.411–415.

Mayes, RW, Macdonald, LM, Ross, J. & Dawson, L.A. 2009. Discrimination of domestic garden soils using plant wax compounds as markers. In: Ritz, K., Dawson, L.A. & Miller, D.R. (eds.). *Criminal and Environmental Soil Forensics*. Springer, Chapter 29, 463-476.

Ngim, K.K., Ebeler, S.E., Lew, M.E., Crosby, D.G. & Wong, J.W. 2000. Optimized Procedures for Analyzing Primary Alkylamines in Wines by Pentafluorobenzaldehyde Derivatization and GC–MS. *Journal of Agricultural and Food Chemistry*, 48(8), pp.3311–3316.

Paczkowski, S. & Schütz, S. 2011. Post-mortem volatiles of vertebrate tissue. *Applied Microbiology and Biotechnology*, 91(4), pp.917–935.

Pirrie, D., Power, M.R., Rollinson, G.K., Wiltshire, P.E.J., Newberry, J. & Campbell, H.E. 2009. Automated SEM-EDS (QEMSCAN®) mineral analysis in forensic soil investigations; testing instrumental variability. In: Ritz, K., Dawson, L. & Miller, D. (eds) *Criminal and Environmental Soil Forensics*. Springer, 411-430.

Pirrie, D., Rollinson, G.K., Andersen, J.C., Wootton, D. & Moorhead, S. 2014. Soil forensics as a tool to test reported artefact find sites. *Journal of Archaeological Science*, 41, 461-473.

Pringle, J.K., Ruffell, A., Jervis, J.R., Donnelly, L., McKinley, J., Hansen, J., Morgan, R., Pirrie, D., & Harrison, M. 2012. The use of geoscience methods for terrestrial forensic searches. *Earth-Sci review*. 114, 108-123.

Peffer, G. 2013. A Case Study: The Application of a Blend of UK Police Service Assets in a Specialist Search. Abstract - Paper presented at the Second International Conference on Engineering Geophysics, Al Ain, Abu Dhabi, 2013.

Pringle, J.K., Cassella J.P. & Jervis J.R. 2010. Preliminary soilwater conductivity analysis to date clandestine burials of homicide victims. *Forensic Sci Int*, 198(1-3), 126-133

Pringle, J.K., Cassella, J.P., Jervis, J.R., Williams, A., Cross, P. & Cassidy, N.J. 2015. Soilwater Conductivity Analysis to Date and Locate Clandestine Graves of Homicide Victims. *J Forensic Sci*, vol. 60(4), 1052-1060.

Regev, L., Poduska, K.M., Addadi, L., Weiner, S. & Boaretto, E. 2010. Distinguishing between calcites formed by different mechanisms using infrared spectrometry: archaeological applications. *Journal of Archaeological Science*, 37, 3022-3029.

Ruffell, A., 2005. Searching for the IRA “disappeared”: Ground Penetrating radar investigation of a churchyard burial site. *Journal of Forensic Sciences*, v.50, pp.1430-1435.

Stadler, S., Stefanuto, P.-H., Byer, J.D., Brokl, M., Forbes, S.L. & Focant, J.F. 2012. Analysis of synthetic canine training aids by comprehensive two-dimensional gas chromatography-time of flight mass spectrometry. *Journal of Chromatography A*, 1255, pp.202–206.

Statheropoulos, M., Agapiou, A., Spiliopoulou, C., Pallis, G.C. & Sianos, E. 2007. Environmental aspects of VOCs evolved in the early stages of human decomposition. *Science of the Total Environment*, 385(1–3), pp.221–227.

Swann, L., Chidlow, G.E., Forbes, S., Lewis, S. W. 2010. Preliminary studies into the characterization of chemical markers of decomposition for geoforensics. *Journal of Forensic Science*, 55(2), pp.308–314

Swann, L., Forbes, S. & Lewis, S.W. 2010. Observations of the temporal variation in chemical content of decomposition fluid: A preliminary study using pigs as a model system. *Australian Journal of Forensic Sciences*, 42(3), pp.199–210.

Tipple, C.A., Caldwell, P.T., Kile, B.M., Beussman, D.J., Rushing, B., Mitchell, N.J., Whitchurch, C.J., Grime, M., Stockham, R. & Eckenrode, B.A. 2014. Comprehensive characterization of commercially available canine training aids. *Forensic Science International*, 242, pp.242–254.

Tolliver, S.S. 2005. Identification of *Canis familiaris* signature odor chemicals in human remains using derivatization solid-phase microextraction/gas chromatography/mass spectrometry. Master's thesis, Florida International University, Department of Chemistry and Biochemistry 11.

Truemana, C.N.G., Behrensmeyer, A.K., Tuross, N. & Weinerd, S. 2004. Mineralogical and compositional changes in bones exposed on soil surfaces in Amboseli National Park, Kenya: diagenetic mechanisms and the role of sediment pore fluids. *Journal of Archaeological Science*, 31, 721–739.

Tyrrell, S., Haughton, P.D.W., Daly, J.S., Kokfelt, T.F. & Gagnevin, D. 2006. The use of the common Pb isotope composition of detrital K-feldspar grains as a provenance tool and its applications to Upper Carboniferous paleodrainage, Northern England. *Journal of Sedimentary Research*, 76, 324–345.

Vass, A.A., 2012, Odor mortis: *Forensic Science International*, v. 222, no. 1–3, pp. 234–241.

Vass, A.A., Bass, W.M., Wolt, J.D., Foss, J.E. & Ammons, J.T. 1992. Time since death determinations of human cadavers using soil solution. *Journal of forensic sciences*, 37(5), pp.1236–1253.

Vass, A.A., Barshick, S.A., Sega, G., Caton, J., Skeen, J.T., Love, J.C. & Synstelien, J.A. 2002. Decomposition chemistry of human remains: a new methodology for determining the postmortem interval. *Journal of forensic sciences*, 47(3), pp.542–553.

von der Lühe, B., Fiedler, S., Mayes, R.W. & Dawson, L. 2017 Temporal fatty acid profiles of human decomposition fluid in soil, *Organic Geochemistry* 111, 26–33

von der Lühe, B., Dawson, L.A., Mayes, R.W., Forbes, S. & Fiedler, S. 2013 Investigation of sterols as potential biomarkers for the detection of pig (*S. domesticus*) fluid in soils, *Forensic Science International* 230, 68–73

Zychowski, J. 2012. Impact of cemeteries on groundwater chemistry: A review. *Catena*, 93, pp.29–37.















