Was Wales the birthplace of the builders of Stonehenge?

A grave containing the remains of three children, a teenager and three men discovered last year during road improvements near the Boscombe Down, Wiltshire, UK, revealed that some of the men who built Stonehenge (Fig. 1) were almost certainly born in Wales, according Wessex Archaeology.

Archaeologist Colin Kirby, who is employed by leading science and technology company QinetiQ, made the discovery. “On the second day of the excavations, I noticed human remains in the side of a water pipe trench”, he said. “On investigating the spoil from the trench, beaker fragments and an arrowhead emerged. This was very exciting, as it showed that the burial was probably Early Bronze Age and may be linked to the Amesbury Archer. I immediately informed Wessex Archaeology”.

The skulls of the men and the teenager are so similar that they must be related. Archaeologists are calling the men “The Boscombe Bowmen” because of the flint arrowheads in the grave.

Stonehenge was begun in the late Stone Age, around 3000 BC, as a ditch and a bank enclosing an open space. Around 2300 BC, the world-famous stones were erected. Because the Bowmen lived at the same time, archaeologists believe they may have been involved in the creation of Stonehenge.

Andrew Fitzpatrick of Wessex Archaeology said: “For the first time, we have found the mortal remains of one of the families who were almost certainly involved in this monumental task”.

Chemical tests on the Bowmen’s teeth provided the clue to where they came from. As the enamel forms on children’s teeth, it locks in a chemical fingerprint of where they grew up. Tests by scientists of the British Geological Survey (BGS) on the strontium isotopes in the Bowmen’s teeth showed that they grew up where the rocks were very radioactive – in the Lake District, England, or Wales. The men’s teeth also all have the same pattern, showing that they migrated between the ages of 3 and 13.

“This provides a remarkable picture of prehistoric migration”, said Jane Evans of BGS.

Bluestones used to build Stonehenge were transported 250 km from the Preseli Hills, south-west Wales.

“The Boscombe Bowmen, a band of brothers, must almost certainly be linked with the bringing of the bluestones to Stonehenge”, said Fitzpatrick. “With the discovery that the Amesbury Archer came from central Europe [1], these finds are casting the first light on an extraordinary picture at the dawn of the metal age”.

“Through the mists of time, we can start to see the very people who brought the building blocks of the greatest temple of its age. We can also glimpse the important people who were associated with that Temple to the Gods of the Sun and the Moon. It is an epic story”, he said.

Reference


LC-ESI–MS finds drug residues in meat

Promochem has launched a range of reference standards for the nitrofuran drug metabolites developed by Cambridge Isotope Laboratories to determine drug residues in meat using a new liquid chromatography-electrospray ionization–mass spectrometry (LC-ESI–MS) method.

Figure 1. Stonehenge, the greatest temple of its age.
Nitrofurans are inexpensive and effective veterinary antibiotics that have been widely used for the treatment of gastrointestinal infections in cattle, pigs and poultry. In 1995, following concerns about their potential carcinogenicity, they were banned from use in animals for human consumption that are either produced in, or imported into, the European Union (EU). However, these drugs, which include furazolidone, furaltadone, nitrofurantoine and nitrofurazone are still manufactured and used on food producing animals in many places outside the EU. For example, an investigation early in 2002 found nitrofuran residues in seafood products and poultry, duck and rabbit imported from South-East Asia.

These findings prompted the European Commission (EC) to insist on positive-release testing for residues of nitrofurans and their metabolites in all imported edible animal products from specified countries of origin. As a result, in March 2003, Portugal stopped the sale of all chicken, turkey and quail because of suspicions of nitrofuran contamination. Italy and Greece have also detected some smaller scale instances of nitrofuran contamination.

However, looking for residual parent drugs is not particularly effective in detecting their use, because they are metabolized within hours of administration. However, protein-bound metabolites, such as 3-amino-2-oxazolidinone (AOZ), 5-methylmorpholin-3-amin-2-oxazolidinone (AMOZ), 1-aminohydantoin hydrochloride (AHD) and semicarbazide hydrochloride (SEM), may persist for weeks or even months, and do not degrade significantly during cooking or processing. These residues can be released from proteins under moderately acidic conditions and can be determined as total nitrofurans, or simply as bound residues.

Currently, there are no immunochemical or microbiological screening methods available for detecting all four nitrofuran antibiotics. The only analytical technique that is really effective in meeting the EC’s criteria for sensitivity and confirmation of identity is LC-MS/MS.

**Metabolites determined**

However, a LC-electrospray ionization–mass spectrometry (LC-ESI–MS) method has been developed for simultaneous determination of the metabolites of all four nitrofuran drugs in meat and meat products. In this method, sample clean-up and analyte enrichment are carried out by extraction with ethyl acetate, hydrolysis of the protein-bound drug metabolites, and derivatization with 2-nitrobenzaldehyde (2-NBA). In trials, at least two precursor–product transitions were measured for each metabolite, and each measured decision limit (CCa) was below the EC’s minimum required performance level of 1 mg/kg.

The new standards are available as stable isotope-labeled compounds with deuterium or carbon-13 and nitrogen-15 or unlabelled in methanolic solution.

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**TrAC maintains its position in rankings**

The latest impact factors for analytical chemistry, which cover articles cited in 2003, show that *TrAC* has maintained third place in the rankings for peer-reviewed journals, coming after only *Analytical Chemistry* (published by the American Chemical Society) and *Electrophoresis* (published by Wiley).

Table 1 shows *TrAC*’s record in terms of the number of articles cited and the number of citations, as recorded by Thomson ISI, which maintains the database used to compile the ranking tables for different scientific disciplines.

**Fingerprinting finds fake fine wines**

Together analytical chemistry and chemometrics can prevent the passing off fake substitutes for fine wines, such as French Champagne, Spanish Rioja and Italian Chianti, following work by Professor Ana María Cameán Fernández and colleagues at the University of Seville and the Spanish National Institute of Toxicology.

They showed that French Champagne could be differentiated completely from Spanish Cava using atomic spectrometry and pattern-recognition (PR) techniques [1]. They determined the metal content of the two sparkling wines by:

- inductively coupled plasma atomic emission spectrometry (ICP-AES) – Al, Ba, Ca, Cu, Fe, K, Mg, Mn, Na, P, Sr and Zn;
- graphite furnace atomic absorption spectrometry (GF–AAS) – Cd, Ni and Pb; and,
- hydride generation AAS (HG–AAS) – As.

For the 18 Cava samples, Zn content was found to be half that of the 17 Champagne samples – Cava: 0.208 – 0.575 mg/l, mean 0.31 ± 0.09 mg/l; Champagne: 0.444 – 0.742 mg/l, mean 0.63 ± 0.08 mg/l.

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And, for Cava, Sr content was found to be a quarter that of Champagne – Cava: 0.03 – 0.106 mg/l, mean 0.07 ± 0.03 mg/l; Champagne: 0.22 – 0.37 mg/l, mean 0.0.28 ± 0.003 mg/l.

The researchers found that, with just these two metals, it would be possible to differentiate between samples of Cava and Champagne, but that these two metals alone were not sufficient for authentication, for which a supervised learning PR method was needed. They therefore subjected their data to linear discriminant analysis (LDA) and soft independence modeling of class analogies (SIMCA).

Fig. 2 shows their Cooman’s plot for the square SIMCA distances obtained for the Cava and Champagne classes, showing them to be separated completely.

As SIMCA is a soft modeling procedure, it could detect the numbers of false positives and false positives attributed to each class, which Cameán and colleagues found to be zero in each case.

“These results show that authentication of these different products can be established using some mineral variables as descriptors,” they concluded.

Reference

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Spectra authenticate extra-virgin olive oil

An optical system can detect real extra virgin oil, according to researchers who devised it as part of the European Union-funded OPTIMO project involving Loughborough University (UK), the CNR Institute for Applied Physics (Florence, Italy) and the CNR Trees and Timber Institute (Sesto Fiorento, Italy).

Their motivation was the need to authenticate the high-quality extra-virgin olive oil that is produced in Italy and is renowned worldwide for its distinctive taste and nutritional benefits, as it is low in fat and rich in anti-oxidants. Cheaper, low-grade oils, misleadingly sold as the more expensive oils from Tuscany, have been flooding the market, threatening the livelihoods of genuine producers and duping consumers.

The system was developed in the Department of Electronic and Electrical Engineering, Loughborough University, using optical spectroscopy.

“It’s very difficult to spot significant differences between oils just by looking”, said Peter Smith, Professor of Photonics Engineering, “but this new light-scattering and absorption technique provides a very sensitive indicator. Having illuminated the sample with white light, we can see how much light of each color shines through the sample and how much is scattered. By carefully studying how the absorbed and scattered spectra become brighter or darker at each wavelength, we can determine the grade of the olive oil and its origin”.

“We have been able to produce for the very first time a distinctive ‘optical fingerprint’ that tells us...
exactly where the olive oil is from and
the process by which it has been
made”, said. “Plotting these finger-
prints on a 3D map reveals distinctive
clusters according to oil type. This
allows for reliable, low-cost analysis
that can easily distinguish the frauds
from the genuine extra virgin oils”.

“The technology can be used to
test the authenticity of other high
value food and drink products, such
as wine and beer”, added Prof. Smith.

At present, a vial containing the
oil sample is inserted into a plastic
jig, although an on-line design
would also be possible.

The absorption spectrum of the oil
sample is measured in the visible
spectrum range, and the scattered
spectra are measured at three angles.
In this way, each oil sample is char-
acterized by 148 spectral data points
related to color and turbidity.

Principal Components Analysis
(PCA) is used to create 2D or 3D
maps from the spectral data, each
point on a map unequivocally
identifying an oil sample with
respect to color and turbidity. Oils
with similar characteristics show up
on the map as clusters of points
(Figs. 3 and 4).

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**Toxic traces tracked in plastics**

X-ray instrument supplier PANaly-
tical and rubbers and polymers
analysis center DSM Resolve have
launched the first set of standards for
toxic elements in polyethylene. The
TOXEL set of standards will provide
the plastics industry with an essen-
tial tool for compliance with new
international legislation governing
the manufacture of plastics.

The European Union’s RoHS and
WEEE Directives [1–3] target the use
of toxic stabilizers in the manu-
facture of plastics and polymers with
the ultimate aim of eliminating the
use of substances such as cadmium.

To comply with these Directives,
plastics manufacturers need to
perform precise elemental analysis
down to sub-ppm levels. In order to
achieve this degree of quantitative
analysis, they require certified
standards against which to perform
the analysis.

TOXEL provides the necessary set
of standards by incorporating five
standards (one blank and four multi-
element standards) containing the
regulated elements, such as Cr, Cd,
Hg, Pb, As, Ni, Cu, Zn, Ba and Br.
Plastics manufacturers worldwide
will therefore be able to perform the
crucial analysis of toxic elements in
polyethylene.

TOXEL was developed by DSM
Resolve together with PANalytical
for the use with X-ray fluorescense
(XRF) analysis, which provides a
very effective method of measuring
sub-ppm levels of toxic elements.
PANalytical offers a range of systems for XRF analysis, among which are the Epsilon 5 and Axios spectrometers; both of these are optimized for the sub-ppm analysis of trace elements.

Establishing a calibration with TOXEL standards and Epsilon 5 or Axios spectrometers is simplified by the fact that XRF is essentially a multi-element technique. A single set of TOXEL calibration standards can be used to calibrate all regulated toxic elements, covering the concentrations from trace levels to a few hundreds of ppm.

References


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VOCs identified very quickly in air

An air-fingerprinting technique can detect, in less than a minute, the components of air, including those of an individual’s breath or a perfume, according to researchers in the Department of Chemistry at the University of Leicester, UK.

The technique offers high sensitivity and much greater speed than rival methods of air testing. The research was led by Paul Monks and Andrew Ellis, who combined proton-transfer reaction (PTR) with time-of-flight mass spectrometry (TOF-MS) to fingerprint the characteristic signatures of VOCs in air.

They maintain that their technique revolutionizes the speed and the accuracy by which air composition can be tested and that it has potential applications in the environmental, industrial and medical worlds, and possibly in the forensic field (e.g., decomposing bodies emit a variety of volatile organic compounds (VOCs) that may be a tell-tale sign that a body lies buried beneath a patch of ground). They found that the resolution of their PTR-TOF–MS far exceeded current commercially available quadrupole-based PTR–MS, although the primary advantage of using TOF-MS was multichannel data acquisition that meant that data are captured across the entire mass range simultaneously.

“VOCs are produced naturally in the body and some are expelled in our breath,” said Monks. “The presence of absence of specific VOCs may be a rapid indicator of certain illnesses. A wide range of VOCs are also emitted from man-made sources and these can have a damaging effect on the environment and on human health.

“Chemical plants, oil refineries, gas platforms, vehicle and aircraft emissions, are all major sources of atmospheric VOCs”, he said. “VOCs are also emitted by numerous consumer products such as paints, solvents, glues, newspapers, and cosmetics”.

Warning of danger

“Many of the hundreds of different VOCs emitted by these products are toxic and/or carcinogenic and, although usually present in very small quantities, the constant emission into poorly ventilated buildings means that human safety levels are often exceeded”, warned Monks.

“Increasing concern about the impact of VOCs on human health is feeding a growing demand for devices to detect these compounds”.

The Leicester team became involved in this research to devise a method for urban pollution monitoring, as they have a number of science goals that required fast fingerprinting (e.g., they are looking at ultra-sensitive detection of short-lived VOCs)

Figure 5. PTR-TOF–MS instrument used to identify VOCs in air.
atmospheric species that control photochemical smog formation).

Reference


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UK bids for European Spallation Source

The White Rose University Consortium, comprising the UK Universities of Leeds, Sheffield and York, has received support for its bid to host the €1.5b (£1b) European Spallation Source (ESS), the most powerful neutron-scattering facility in the world.

“The UK has a strong track record and much experience in providing neutron sources. It should look to build on this”, stated the UK House of Commons Select Committee on Science and Technology in a report on the workings of the CCLRC (Council for the Central Laboratory of the Research Councils). “The UK scientific community and the Government should be fully behind any competitive and viable bid to bring a European Spallation Source to the UK”.

In Bonn in 2002, the White Rose team, in partnership with regional development agency Yorkshire Forward, submitted a formal bid to host the ESS and has since been campaigning for the UK Government to support the ESS as the best option for a "next-generation" neutron source for the UK. Yorkshire Forward has purchased a site in Selby, North Yorkshire, and hopes to submit a planning application later this year.

In 2003, a Yorkshire delegation, led by John Grogan, MP for Selby, met with Lord Sainsbury, UK Minister for Science and Technology, to discuss the bid, as a result of which Lord Sainsbury instigated a review of current and future neutron use in the UK to inform the Government’s decision on whether or not to back the ESS bid.

The UK Government should be prepared to provide the political will and the funding necessary for the UK to host large-scale facilities, according to the Select Committee report.

“We believe that there are substantial direct benefits beyond the calculable economic ones from hosting large-scale facilities, for the UK research community and, less tangibly, for the reputation of UK science as a whole”, stated the report. “We recommend that the CCLRC works closely with the White Rose Consortium, European and other UK partners to help develop a viable UK proposal for hosting a European Spallation Source”.

Three bids from green-field sites

There are currently three bids from green-field sites: Yorkshire; Germany; and, a Scandinavian consortium. A further bid from Hungary is expected to be formalized and there is rumored bid to be a bid on its way from Switzerland.

“We are extremely pleased with the report’s recommendations”, said Professor Bob Cywinski of Leeds University, who is technical advisor to the White Rose team. “To have the ESS in the UK would be a major coup and would ensure that Europe maintains its lead in this important area of science. The ESS would be visited each year by thousands of leading scientists to conduct their research. To have the opportunity of potentially hosting it in Yorkshire is incredibly prestigious for the region and would be a wonderful boost to our region’s economy. We are very much looking forward to working with the CCLRC in promoting the UK as the strongest bidder for this prestigious facility”.

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LC columns speed up peptide mapping 20-fold

Liquid chromatography (LC) columns can separate and identify peptides up to 20 times faster than typical methods.

The application, devised by Agilent Technologies, is for high-resolution peptide mapping of anti-
Peptide mapping is a technique for analyzing proteins by separating and detecting the mixture of peptides generated when a protein is broken up with chemicals or enzymes. The analysis can provide information about the sequence of the original protein and detect subtle differences between proteins when coupled with mass spectrometry (MS).

Most peptide mapping methods rely on traditional LC columns containing porous particles. Agilent's application uses LC columns packed with Poroshell, a superficially porous chromatographic medium, that dramatically reduces the time required for peptide mapping of human monoclonal antibodies from 120 to 20.5 min without loss in resolution.

For ultra-high-speed peptide mapping, run times are as low as 5.6 min, a 20-fold increase in speed, with a resolution loss of only 18–19%, which is minimal when using a sensitive, discriminating detector, such as a MS, according to Agilent.

The ZORBAX Poroshell 300SB columns use particles comprising a solid silica core surrounded by a thin outer layer of porous silica, which allows higher throughput and flow rates while maintaining sharp peak shape and resolution. During separation, proteins and peptides rapidly diffuse into and out of the thin porous shell and elute in seconds as easily resolved, narrow bands. These columns are available in a number of internal diameters and bonded phases.

Reference


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Figure 7. Ultra-high-speed peptide maps of human monoclonal, Lys-C digest, using ZORBAX Poroshell 300SB-C18, C8, and C3 columns.
Lawyers challenge quality in forensic laboratories

The quality assurance–quality control (QA/QC) process in forensic crime laboratories has failed across the nation, according to the National Association of Criminal Defense Lawyers (NACDL) in the USA, which has responded by providing guidance [1,2].

The NACDL offered examples of recent forensic failures to create awareness that forensic scientists may be partisan and may make mistakes, before offering a model of discovery of materials that are necessary for a forensic scientist to review to render an opinion as to the validity of a report produced by a fellow forensic scientist.

Apart from individual cases of serious miscarriages of justice involving defects in QA/QC, the NACDL cited a 1997 US Department of Justice investigation into the Federal Bureau of Investigation (FBI) Laboratory that found the following problems: scientifically flawed testimony; inaccurate testimony; testimony beyond the examiner’s expertise; improper preparation of laboratory reports; insufficient documentation of test results; scientifically flawed reports; inadequate record management and retention systems; failures by management to resolve serious and credible allegations of incompetence; and, a flawed staffing structure of a unit.

Again last year, the US Justice Department identified over 3000 cases that could possibly have been affected by poor work product of 13 individuals working for the FBI Lab.

Particularly remarkable last year was the case of William Sybers v. Florida, when the Appeal Court stated: ‘We conclude that the state has failed to carry its burden of establishing by ‘independent and impartial proof’ that the testing procedures used are generally accepted in the relevant scientific community. The only testimony offered by the state to establish the general scientific acceptance of the testing procedures came from Dr. Ballard and LeBeau (the FBI’s chief chemist), each of whom either had a personal stake in the procedure or was prone to potential institutional bias. Such assertions are not, alone, sufficient’.

Most importantly, according to the NACDL, the court noted that: ‘No scientific literature has been cited by the parties, or uncovered by our independent research, which addresses similar testing procedures for succinylmonocholine in embalmed tissues, especially tissues that are many years old’. The FBI simply went forward with an unvalidated testing protocol, unable to show the court that opinions rendered were indeed valid. ‘Why’, asked the NACDL?

Questions to ask in discovery

In response to such problems, the NACDL wants to know what happens to evidence from the time it is collected until the forensic scientist gives an opinion. It therefore proposes to ask for the following items in discovery:

1. Evidence-collection forms or logs (description of evidence, packaging, identification of specimens, identification of individuals collecting samples, and sample-collection procedures).
2. Chain-of-custody records (field-to-lab transfers, and all transfers of evidence and associated analytical samples within the laboratory).
3. Laboratory receiving records (records documenting the date, time and condition of receipt of the evidence in question; laboratory-assigned identifiers; and, storage location).
4. Laboratory procedures for sub-sampling (collection of analytical aliquots) and contamination control.
5. Copies of technical procedures in effect at the time the subject testing was performed (often termed Standard Operating Procedures, SOPs) for each procedure used during sample screening and confirmation, including sample preparation, sample analysis, data reporting, and instrument operation.
6. Copies of the two bracketing controlled substance proficiency results for each analyst and technician responsible for preparation or analysis of subject specimens, including: raw data and reported results, target values and acceptance ranges, performance scores, and all related correspondence.
7. Copies of traceability documentation for standards and reference materials used during analysis, including unique identifiers, origins, dates of preparation and use, composition and concentration of prepared materials, certifications or traceability records from suppliers, assigned shelf lives and storage conditions.
8. Sample-preparation records, including dates and conditions of preparation, responsible analyst, procedural reference, purity, concentration and origins of solvents, reagents, and control materials prepared and used, samples processed concurrently, and extract volume.
9. Copies of bench notes, log books, and any other records pertaining to case samples or instruments; records documenting observations, notations, or measurements regarding case testing.
10. Instrument run log with identification of all standards, reference materials, sample blanks, rinses, and controls analyzed during the day/shift with subject samples (as appropriate, run
sequence, origins, times of analysis and aborted run sequences).
11. Record of instrument operating conditions and criteria for variables, including as appropriate, gas-chromatograph column, instrument-file identification, tuning criteria, instrument-performance check (e.g., ion-abundance criteria), initial calibration, continuing calibration checks, and calibration verification.
12. Record of instrument maintenance status and activities for instruments used in subject testing, documenting routine and as-needed maintenance activities in the weeks surrounding subject testing.
13. Raw data for the complete measurement sequence (opening and closing quality control included) that includes the subject samples. For GC-MS analysis, this would include areas and retention times, injection volumes, dilution factors, chromatograms and mass spectra. As prepared and as determined values for all quality control samples.
14. A description of the library used for spectral matches for the purpose of qualitative identification of controlled substances, including source(s) and number of reference spectra.
15. Copy of records documenting computation of illicit drug laboratory’s theoretical production yield, including the basis for the computation, and the algorithm used, as appropriate.
16. Procedure(s) for operation and calibration checks of analytical balances used to weigh controlled substances.
17. Results of calibration checks and documentation of mass traceability for gravimetric determinations.
18. Results of contamination control surveys for trace-level analytes relevant to test methods at the time of analysis, including sampling design and analytical procedures.
19. Records and results of internal reviews of subject data.
20. Method-validation records documenting the laboratory’s performance characteristics for qualitative identification and quantitative determinations of the controlled substance, to include data documenting specificity, accuracy, precision, linearity, and method detection limits.
21. Copy of the laboratory’s Quality Manual in effect at the time the subject samples were tested as well as the laboratory’s most recent Quality Manual (however named; the document that describes the laboratory’s quality objects and policies).
22. Copy of the laboratory’s ASCLD-LAB (American Society of Crime Lab Directors-Lab) application for accreditation, and most recent Annual Accreditation Review Report, as appropriate.
23. Statement of qualifications of each analyst and/or technician responsible for processing case samples to include all names, locations and jurisdictions of cases in which these personnel testified concerning the same substances found in the present case.
24. Copy of the laboratory’s ASCLD-LAB on-site inspection report, as appropriate, as well as any reports of on-site inspections by any other testing laboratory audit organization.
25. Copy of internal audit reports generated during the period subject samples were tested.
26. List of capital instrumentation in the laboratory at the time subject testing was performed, including manufacturer, model number, and major accessories.
27. Production throughput data for the drug-testing section: numbers of tests performed per month or per year, and the number of full-time equivalent personnel in the drug-testing section of the laboratory.

References

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