New Enhanced Analytical Monitoring Installation at Swansea University

Dr Ruth Godfrey, University of Swansea

We are pleased to announce the arrival of a new and unique UK analytical capability that offers a 'step change' in measurement, with the potential to detect a much broader range of organic substances (including isomers) within solids and liquids. By utilising Omni International's (a PerkinElmer company) bead beating homogenisation technology, followed by a multi-modal sample introduction system (GL Sciences, supported by SepSolve Analytical) combined with separation/detection via Gas Chromatography-Vacuum Ultraviolet spectroscopy (VUV Analytics, supported by Scientific Support Services UK), this technology installation will enable an enhanced picture of sample materials to be established. Collaborative partners at SepSolve Analytical will support the development of new sample introduction methods to expand this analysis across a wide range of complex matrices. We will apply this to design new, safer, cleaner and more accurate treatments, processes and technologies for recycling/re-use and manufacturing for more effective healthcare, and a resilient and sustainable Circular Economy (CE).

Swansea University's Open Innovation environment houses world-leading experts and sector-led research in life science, engineering, analytical science, environmental and public health protection. The co-location of the environmental regulator (NRW) at Swansea enables industry, academia and government to act collectively, to better understand the content and, environmental and health impacts of manufactured products and green(er) replacements. By interfacing homogenisation and a multi-modal sample introduction to release 'hidden' chemicals across material types, and the applicability of Gas Chromatography-Vacuum Ultraviolet spectroscopy (GC-VUV) to a broader range of volatile and semi-volatile chemistries, Swansea's new installation offers enhanced analytical monitoring for substances routinely measured using gas chromatography-mass spectrometry (GC-MS). Thus, this installation will inform the development of improved technologies, processes, products and policies that bridge the life science, health, manufacturing and environmental sectors, whilst enabling the UK to establish a more resilient and sustainable CE to protect environmental and public health.

What are the capabilities and how do they work?

Homogenisation

Omni's technology can mechanically disrupt, extract and/or homogenise virtually any sample through rapid agitation with a homogenisation (grinding) matrix. These matrices consist of beads that differ in material, size, shape and composition, and are selected according to sample type, mass/volume of sample, the target particle size, and the stability of the target molecule. Therefore, the bead type can be used to control homogenisation efficiency given surface area, density, hardness, durability, and chemical resistance all influence performance (see *Figure 1*). For example, smaller beads can be used to generate smaller sample particle sizes, and in some cases, multiple bead sizes are used to achieve the desired pulverisation. Whilst, to control the extent and type of disruption the bead shape is key; spherical beads use impaction and hammering to isolate organelles or unstable molecules (e.g. RNA and specific proteins), and angular beads chop and cut samples by applying mechanical shear force to release stable molecules such as DNA and small molecules from challenging samples (e.g. cell walls and fibrous tissue).

Multi-modal sample introduction

The OPTIC-4 by GL Sciences offers a range of sample introduction options for interrogating intact solids and liquids. With a programmable inlet capable of rapidly reaching temperatures of 600°C the system can perform thermal desorption, on-column injection, and large volume injections (LVI) for enhanced sensitivity, in-liner derivatisation and pyrolysis for these sample types. This offers significant flexibility for applications that span the breadth of applications for volatile and non-volatile species.

Gas Chromatography-Vacuum Ultraviolet spectroscopy

GC-VUV uses the power of high resolution GC separations with the enhanced analytical scope and selectivity of the non-destructive VUV detector, for reliable qualitative and quantitative analysis. VUV offers significant potential for measuring substances across a broad range of applications given almost all chemical species strongly absorb at VUV wavelengths due to the excitation of a broad range of high energy electronic transitions (see

Figure 1: Diagram detailing the application need and potential matrices used to homogenise example materials (image published with permission from Omni International).

Figure 2). As a result, VUV spectra are directly related to the molecular electronic structure and are therefore, highly specific. GC-VUV may be considered as the GC equivalent of High Performance Liquid Chromatography-Diode Array Detection (HPLC-DAD).

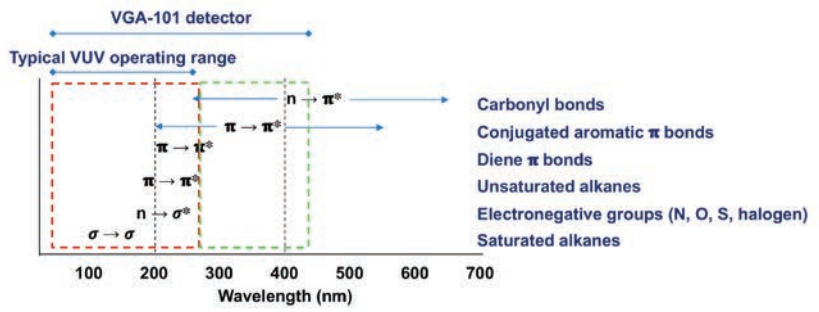

Figure 2: Wavelength range of relevant molecular bonding and the operating range of the VUV VGA-101 detector.

Following sample introduction onto the GC column, analytes are transferred through the column to the detector by the mobile phase (carrier gas), and separated according to their interaction with the column stationary phase and volatility. Analytes elute off the column and flow through the transfer line into the flow cell, where photons of light from a deuterium lamp are absorbed by the analytes and their spectra (125 - 430 nm) are recorded by a charged coupled device (CCD) detector (see *Figure 3*). Given nearly all molecules have a unique electronic structure (and absorption cross-section), this can provide a highly specific spectral 'fingerprint', enabling the identification of analytes through comparison with reference spectra.

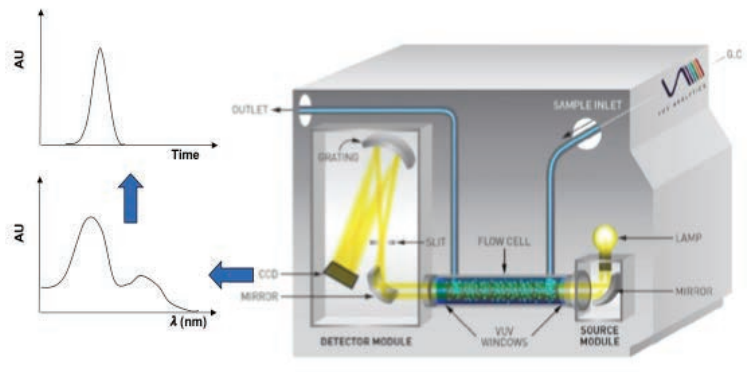

Figure 3: Cross-sectional diagram of the VUV detector showing the application of light to the flow cell, with the acquisition of subsequent spectra and chromatogram (image published with permission from VUV Analytics).

Key 'take aways' and benefits of the technologies

Bead Beating Homogenisation

- Wide range of accessories and homogenising matrices facilitate processing sample sizes of <0.5-50 mL.
- Is extremely quick compared to rotor stator homogenisation, operates with tubes or well-plates for higher sample throughput.
- Cryo-cooling for thermally labile analytes to ensure analyte preservation.

Multi-modal sample introduction

- Variety of sample introduction modes for different sample matrices - improved laboratory versatility (TD/Pyrolysis/LVI).
- Low thermal mass allows fast and uniform heating for applicability up to C100.
- Inert surfaces minimise degradation of labile components.

VUV

- 'Universal' detector capable of discriminating highly similar analytes when in the gas phase (including structural isomers).
- Highly repeatable and additive spectra - enables molecular identification (including those that co-elute) by searching against reference spectra, and operation of faster, higher-throughput chromatographic methods without fully separating analytes.
- Quick and robust analyte classification using post-acquisition spectral filters, algorithms, regression statistics for spectral matching and a retention index.
- Fast scanning capability (data acquired up to 77 Hz) - enables good sampling statistics per GC peak, offering reliable and accurate quantitation using a simple linear relationship (Beer-Lambert Law) to pg sensitivities.
- Complementary detection to MS - can measure volatile analytes not amenable to MS and distinguish some isomers and isobaric compounds.

Applications

Despite the primary usage of VUV detection for petrochemicals, and the initial remit of this installation in addressing environmental-sector challenges, its applicability to a broader range of sample materials, volatile and semi-volatile chemistries, can provide enhanced analysis of those measured across many sectors. For example, monitoring approaches within environmental, biotech, pharma, fine chemical, materials manufacturing, forensic and clinical sectors can involve laborious protocols to displace analytes from sample materials for detection, and often fail to discriminate similar (isomeric) substances. Given substance activity (and toxicity) is dependent on molecular shape (e.g. pharmaceuticals, pesticides, polyaromatic hydrocarbons, polychlorinated biphenyl (PCB) pollutants), these limitations pose a significant environmental and public health risk. Therefore, by better releasing substances sorbed to materials, and distinguishing structural isomers and co-eluting species, this installation offers significant benefit for a diverse set of application areas. Specifically, this can enhance monitoring for the design of safer, cleaner and more accurate treatments, technologies, recycling/re-use and manufacturing processes, for more effective healthcare, resilient and sustainable CE [1-6].

To deliver this agenda, the installation will:

1) actively seek industry, government and academic collaboration to explore new application areas for a CE and beyond, to fully exploit capacity, capability and impact,

2) support users continuous professional development (CPD) in the technologies, and

3) assist in the expansion of the VUV spectral database for ease of future substance detection.

For further information on the technology installation and potential collaborative projects, please contact the capability lead, Dr Ruth Godfrey (a.r.godfrey@swansea.ac.uk).

Acknowledgements

The authors gratefully acknowledge the support from industry collaborators, and funding from Welsh Government in helping to establish this capability.

Mewn Partneriaeth â Llywodraeth Cymru
In Partnership with Welsh Government

1. *WG Beyond Recycling: a strategy to make the circular economy in Wales a reality.*
2. *Well-being of Future Generations Act (WFGA).*
3. *Environment (Wales) Act, Natural Resources Policy.*
4. *WG Prosperity for All: the national strategy.*
5. *UKRI The Business of the Environment.*
6. *HMG UK Industrial Strategy*