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Prof Caroline Gauchotte-Lindsay is Professor of Environmental Engineering and Chemistry at the University of Glasgow. She obtained her PhD in Environmental Engineering in 2010 in the School of Planning, Architecture and Civil Engineering at the Queen's University Belfast. She joined the University of Glasgow as a Lecturer in 2012, after carrying out her postdoctoral research in the Department of Civil and Environmental Engineering at the University of Strathclyde. Her research puts analytical chemistry to the service of engineering and environmental engineering in particular. Her group (hiRACE) develops innovative methods using advanced chromatography and mass spectrometry and novel sensitive assays and sensors that can be used with routine lab equipment. Their approaches are designed to help understand and control biological, chemical and process mechanisms. They have a special interest in environmental biotechnologies but have also taken on challenges in combustion engineering, geochemistry and health.

# Thermogravimetric Analyzer coupled with Fourrier Transform Infrared Spectroscopy and Gas Chromatography/ Comprehensive Two-dimensional Gas Chromatography Data Analysis

## Method description in brief.

Thermogravimetric Analyzer coupled with Fourier Transform Infrared Spectroscopy and Gas Chromatography (TGA-FTIR/GC) is employed for simulated distillation of oil samples. Simulated distillation involves heating the sample and measuring the vaporization of its components at different temperatures. TGA and FTIR are coupled to provide information about the sample's weight loss and chemical composition as a function of temperature. FTIR enables the identification of evolved gases and robust quantification of total aromatic and aliphatic. This method allows simulating distillation at higher temperatures (up to 1000°C) compared to SIMDIS with GC. TGA can also be coupled with GC-MS, facilitating compound separation and identification at chosen temperatures compatible with GC.
TGA can be carried out in oxidative conditions, enabling further identification of components. While no specific sample preparation is required, introducing the sample in a way that mimics distillation is essential.

Exhaustive extraction and comprehensive two-dimensional gas chromatography (GCxGC): we have developed method for comprehensive analysis of compounds of coal tar starting with derivatisation using ultrasound-assisted extraction and derivatisation and finishing with a workflow for non-targeted analysis of the chromatograms. We don't have a GCxGC (GCxGC coupled with a mass spectrometer and/or a VUV) so will have to run the sample elsewhere but we can carry out extraction and data analysis. Beyond silylation, other chemical conversions can be used for chemical identification, using machine learning to exhaustively compare the chromatograms of original and converted samples.

## Applicability of method.

TGA-FTIR enables the quantification of aromatics and aliphatics at different temperatures during the distillation/evaporation of a sample. This technique is particularly useful for dealing with high boiling points,

reaching up to 1000°C, and handling high molecular weights. For temperatures up to 450°C, GC-MS can complement the analysis by resolving the compounds present in the evolved gas.
In our GCxGC method, ultrasound-assisted derivatization (silylation) extends the range of compounds compatible with GC analysis, including acidic compounds. Utilizing reverse GCxGC chromatography allows for the separation of compounds in the second dimension based on their functional groups. This method significantly improves the separation of structural isomers compared to one-dimensional GC. Our data workflow is designed to align chromatograms and group compounds based on common characteristics

## Sample preparation required.

TGA-FTIR: no preparation

GCxGC: ultrasound assisted liquid-liquid extraction and derivatisation

## Method strengths.

TGA-FTIR/GC-MS: The strengths are that 1) it can simulate distillation and fraction the samples and quantify aromatic and aliphatic in each fraction, 2) it can be calibrated to determine boiling points precisely, 3) it provides online fractionation for GC-MS analysis.

GCxGC: extension to acidic compounds and non-targeted approach for groupings of compounds through machine learning.

## Estimated time for analysis.

TGA-FTIR/GC-MS: This is a novel approach, some method development would be required, which can be started ahead of the samples arriving. Analysis will take one afternoon for all samples and then two weeks for data processing and interpretation.

GCxGC: Samples can be prepared in our laboratory in one week (including chemical conversion), analysed in another lab (to be determined). Data processing and non-targeted machine learning analysis will take 8 weeks (because of method development requirement).

## Method weaknesses.

TGA-FTIR/GC-MS: this is a speculative approach; while the technology has been used for this type of samples it has not been in this context. The FTIR approach does not enable identification of individual compounds, the GC-MS approach does, but is limited to GC compatible compounds.

GCxGC: Limitation to GC compatible compounds (after derivatisation), *i.e.* boiling points below 450°C (for high temperature GC).

## Result interpretation / visualisation / presentation.

TGA-FTIR/GC-MS: Raw data: TGA spectra, FTIR spectra for given temperatures, GC-MS chromatograms for given temperatures; calculated/ processed data: boiling points for all fractions and quantification of aromatics and aliphatic for all fractions.

GCxGC: Raw data: GCxGC Chromatograms of samples for all conversions; processed data: workflow results, *i.e.* clustering of compounds (through affinity propagation), tentative dentification of compounds in the various clusters using mass spectrometry and GCxGC elution- tables and plots will be presented for both.

## Relevant Papers


1. Afonso, C.[…] **Gauchotte-Lindsay, C**., et al. (2019) Data mining and visualisation: general discussion. Faraday Discussions, 218, pp. 354-371. (doi:10.1039/c9fd90044f) (PMID:31373341)
2. **Gauchotte-Lindsay, C.** , McGregor, L. A., Assal, A., Thomas, R. and Kalin, R. M. (2014) Highlighting the effects of co-eluting interferences on compound specific stable isotope analysis of polycyclic aromatic hydrocarbons using comprehensive two-dimensional gas chromatography. ChemPlusChem, 79(6), pp. 804-812. (doi:10.1002/cplu.201400026)
3. McGregor, L.A., **Gauchotte-Lindsay, C.** , Nic Daéid, N., Thomas, R. and Kalin, R.M. (2012) Multivariate statistical methods for the environmental forensic classification of coal tars from former manufactured gas plants. Environmental Science and Technology, 46(7), pp. 3744-3752. (doi:10.1021/es203708w)
4. **Gauchotte-Lindsay, C.** , Richards, P., McGregor, L.A., Thomas, R. and Kalin, R.M. (2012) A one-step method for priority compounds of concern in tar from former industrial sites: Trimethylsilyl derivatisation with comprehensive two-dimensional gas chromatography. Journal of Chromatography A, 1253, pp. 154-163. (doi:10.1016/j.chroma.2012.06.093)
5. McGregor, L.A., **Gauchotte-Lindsay, C.** , Daéid, N.N., Thomas, R., Daly, P. and Kalin, R.M. (2011) Ultra resolution chemical fingerprinting of dense non-aqueous phase liquids from manufactured gas plants by reversed phase comprehensive two-dimensional gas chromatography. Journal of Chromatography A, 1218(29), pp. 4755-4763. (doi:10.1016/j.chroma.2011.05.045)