Direct Compound Specific Isotopic Analysis (C,H,O,N) of complex samples using analytical pyrolysis (Py-CSIA)

Nicasio T. Jiménez-Morillo, José Maria de la Rosa, Francisco J. González-Vila, José A. González-Pérez*
IRNAS-CSIC, Av. Reina Mercedes, 10, 41012-Sevilla, Spain
*jag@irnase.csic.es

INTRODUCTION

Pyrolysis-compound specific isotopic analysis (Py-CSIA), is a relatively novel technique to measure stable isotope proportions i.e., $\delta^{13}C$, $\delta^{15}N$ and $\delta^2H$ in specific compounds released by pyrolysis. This technique can provide valuable information on natural and synthetic materials that are not soluble and therefore not amenable by conventional GC/MS techniques. The technique provide not only a molecular fingerprinting, but also allowing the traceability of their formation processes and origin. Here we made a brief summary of examples of Py-CSIA applications recently developed in our laboratories in different fields, including the food and synthetic polymer industry, research on fire/heat affected soil organic matter and biomass and paleoclimate studies using fossil plant remains.

METHODOLOGY

Direct pyrolysis compound specific isotopic analysis (Py-CSIA) is conducted using a double-shot pyrolyzer (Frontier Laboratories, model 3030D) attached to a Trace Ultra GC system. At the end of the chromatographic column and in order to locate specific peaks within the chromatogram, the flux is divided and 10% diverted to a flame ionization detector (GC/FID) and 90% to a GC-isotopic System equipped with micro-furnaces for combustion (carbon and nitrogen) and for pyrolysis (hydrogen or oxygen). The system is coupled to a Delta V Advantage IRMS via a ConFlo IV universal interface unit (Py-GC-FID-C/TC-IRMS) (Fig. 1). The identification of specific peaks (structural information) are inferred by comparing the mass spectra from a conventional Py-GC/MS system with the Py-GC/FID and Py-GC/IRMS chromatograms obtained using same chromatographic conditions (Fig 2 and Gonzalez-Perez et al., 2015a).

CONCLUSIONS

Our results indicate that pyrolysis compound specific isotopic analysis (Py-CSIA) is a promising technique that adds the known advantages of conventional pyrolysis analyses to obtain direct compound specific stable isotopic information with no need of any previous sample treatment. The technique is particularly useful for the study of complex matrices (natural or synethetics) that are of low solubility or no soluble at all in conventional solvents.

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REFERENCES


Fig. 1. Main instruments at the MOSS group, stable isotopes laboratory

Fig. 2. Example of compound specific ($\delta^{13}C$ and $\delta^2H$) analysis (Py-GC-FID)-C/TC-IRMS) is soil organic matter

Fig. 3. Biomass increasing heating (burning) experiment. $\delta^{13}C$ and $\delta^2H$ Py-CSIA results for different biogenic families of compounds

Fig. 4. Soil organic matter size fractions (fine and coarse) affected and not affected by a wildfire. $\delta^{13}C$ and $\delta^2H$ Py-CSIA for n-alkanes series. (Jimenez-Morillo et al., 2015)

Fig. 5. Pyrolysis of Holocarolina fossil conifer (c. 90 Million yrs.) $\delta^{13}C$ and $\delta^2H$ (Py-CSIA) for n-alkanes series. CO$_2$ $\delta^{13}C$ and $\delta^2H$ H2O/H2O could be inferred.

Fig. 6. Cane (C4) and beer (C3) sugar and added mixtures $\delta^{13}C$ and $\delta^2H$ Py-CSIA. (Gonzalez-Perez et al., 2015a)

Fig. 7. Very low density polyethylene (VLDP) $\delta^{13}C$, $\delta^2H$ and $\delta^{15}N$ Py-CSIA (Gonzalez-Perez et al., 2015a)