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Authors: Oscar Díaz-Santana, Daura Vega-Moreno, Francisco

Conde-Hardisson

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### ACCEPTED MANUSCRIPT

# GAS CHROMATOGRAPHY-MASS SPECTROMETRY AND HIGH-PERFORMANCE LIQUID CHROMATOGRAPHY-DIODE ARRAY DETECTION FOR DATING OF PAPER INK

#### Oscar Díaz-Santana<sup>a</sup>, Daura Vega-Moreno<sup>b\*</sup>, Francisco Conde-Hardisson<sup>c</sup>

- <sup>a</sup> Laboratorio de Grafística y Documentoscopia del Servicio de Criminalística del Instituto Canario de Análisis Criminologico (ICAC), Las Palmas, Spain. Tel (+34) 928 311 147. www.icac-canarias.com. administración@icac-canarias.com
- b\* Tecnologías, Gestión y Biogeoquímica Ambiental. Departamento de Química. Universidad de Las Palmas de Gran Canaria (ULPGC), Las Palmas, Spain. Tel (+34) 928 454 429. daura.vega@ulpgc.es (\*corresponding author)

#### **Highlights**

- Integrated method GC-MS and HPLC-DAD analysis with a single extraction procedure.
- Paper ink dating through solvents and dyes determination over time.
- Thirty volatile organic compounds and soluble dyes have been measured.
- Multiple linear regression methods for the dating of ballpoint inks on paper.

#### **ABSTRACT**

An extraction and determination method is shown for the analysis of dyes and solvents present in two types of ballpoint pen inks that are deposited onto paper. Ink extracts are analysed using a combination of gas chromatography with mass spectrometry (GC-MS), and high-pressure liquid chromatography with photodiode array detection (HPLC-DAD), within a single sample extraction procedure.

Seventeen solvents and thirteen dyes contained in two Montblanc® inks (black and blue) were monitored for 45 months at monthly intervals, in order to determine variations in the concentrations of the compounds over time. We also studied the relative variations between different compounds and the generation of degradation products such as phenol.

<sup>&</sup>lt;sup>c</sup> Agilent Technologies. Chemical Analysis. Madrid. Spain.

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