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Portable X-ray fluorescence identification of the Cretaceous–Paleogene boundary: Application to the Agost and Caravaca sections, SE Spain

Jordi Ibáñez-Insa [a], Jordi Pérez-Cano [b, c, *], Víctor Fondevilla [b, d], Oriol Oms [b], Marta Rejas [a], José L. Fernández-Turiel [a], Pere Anadón [a]

[a] Institute of Earth Sciences Jaume Almera, ICTJA-CSIC, Lluís Solé i Sabarís s/n, E-08028 Barcelona, Catalonia, Spain
[b] Unitat d'Estratigrafia, Departament de Geologia, Universitat Autònoma de Barcelona, Edifici C, E-08193 Bellaterra (Barcelona), Catalonia, Spain
[c] Departament de Dinàmica de la Terra i de l'Oceà, Facultat de Ciències de la Terra, Universitat de Barcelona, Martí i Franquès s/n, 08028 Barcelona, Catalonia, Spain
[d] Institut Català de Paleontologia Miquel Crusafont, Carrer de l'Escola Industrial, 23, E-08201 Sabadell, Catalonia, Spain

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ABSTRACT

It is shown that portable X-ray fluorescence (pXRF) is a powerful tool for the identification and geochemical characterization of prospective Cretaceous–Paleogene (K–Pg) boundary sites. Field measurements in two well-known K–Pg boundary sequences, located at Agost and Caravaca, SE Spain, have been performed. A sizable enrichment around the K–Pg horizon of several elements such as K, Ti, Fe, Ni, Cr, Cu, Zn, As or Pb, together with a strong reduction in the Ca content, is found with the pXRF instrument. These observations represent a primary geochemical signature of the K–Pg boundary in distal marine sections such as those of Agost and Caravaca. Also, the intensities of the pXRF peaks correlate well with elemental composition data obtained by inductively coupled plasma-mass spectrometry (ICP-MS) on collected samples. Hence, the pXRF field measurements are shown to provide fast and useful quantitative information about K–Pg boundary sequences.

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1. Introduction

The Cretaceous–Paleogene (K–Pg) boundary (or Cretaceous–Tertiary (K–T) boundary) around 66 Ma ago marks one of the 'Big Five' mass extinctions in Earth's history (Raup and Sepkoski, 1982; Gradstein et al., 2012; Renne et al., 2013). Although different hypothesis such as massive flood basalt volcanism in the Deccan Plateau, India, have been proposed to explain the K–Pg event (Keller, 2014; Schoene et al., 2015), it is widely accepted that it was triggered by the impact of a large meteorite (~10 km in diameter) on the Yucatan Peninsula, Mexico, creating the Chicxulub crater (Swisher et al., 1992; Schulte et al., 2010; Renne et al., 2013).

The characteristic micropaleontological, geochemical and mineralogical fingerprints of the K–Pg boundary have enabled its identification in marine sections worldwide (Schulte et al. (2010) and references therein). The K–Pg boundary can be identified from a combination of several of the following observations: (i) turnover of numerous nannofossils and microfossils like those of planktonic foraminifera (Luterbacher and Premoli Silva, 1964; Smit, 1982; Culver, 2003; Alegret, 2007); (ii) a change in lithology, with an abrupt reduction of biogenic calcareous content and the presence of a 2–3-mm, often reddish, iron-rich layer known as impact layer or ejecta layer, which is attributed to impact ejecta that were dispersed globally and deposited in a very short period of time (Smit, 1997); (iii) the presence of the so-called boundary clay, i.e., a thin layer (5–10 cm) of clay-rich sediments just above the ejecta layer that were probably deposited during 5–20 kyr after the impact event (Christensen et al., 1973; Smit, 1999); (iv) enrichment in iridium and other platinum group elements (PGE) of presumably meteoritic origin in the impact layer (Alvarez et al., 1980; Smit and Hertogen, 1980); (v) enrichment of other elements such as K, Ti, As, Sb, Zn, Fe, Cr, Ni, V, Co, Sr, and Ba, as reported for the case of the Agost and Caravaca sections (Martínez-Ruiz et al., 1992; Smit, 2004); (vi) observation of microtektite glass spherules (Sigurdsson and Hondt, 1991), microkrystites and/or shocked

* Corresponding author.
E-mail address: jordi_perez-cano@ub.edu (J. Pérez-Cano).

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minerals that are attributed to products generated during and after the impact (Smit and Klaver, 1981; Bohor, 1984; Montanari et al., 1983; Schulte et al., 2009; Belza et al., 2015). Also, the previous observations are usually accompanied by a strong negative anomaly in the $\delta^{13}C$ isotopic signature (Hsu, 1980; Romein and Smit, 1981), which is indicative of a decrease in primary productivity.

Most of the literature published on the identification and geochemical characterization of the K–Pg boundary at numerous sites around the world made use of different laboratory techniques such as neutron activation analysis (NAA), inductively-coupled mass spectrometry (ICP-MS), electron probe microanalysis (EPMA), X-ray fluorescence (XRF), X-ray diffraction (XRD), or stable isotope analysis (see Schulte et al. (2010) and references therein). In order to identify new K–Pg boundary sites and also to further characterize those already known, the availability of portable analytical tools for in-situ measurements could be highly advantageous. In spite of the clear interest of field methods for this type of studies, the use of field analyses to recognize the K–Pg boundary is very limited. For instance, a magnetic susceptibility (MS) field method was successfully employed (Ellwood et al., 2003) and later confirmed by inductively-coupled plasma mass spectrometry (ICP-MS) and particle-induced X-ray emission (PIXE) measurements to identify the K–Pg horizon in Oman.

In the past decade, portable XRF (pXRF) devices were widely used to obtain fast and non-destructive in-situ elemental information in many different research and industrial areas (see for instance Potts and West, 2008) such as: environmental research and soil pollution assessments; workplace monitoring; archaeology and cultural heritage; metal and alloy sorting; mineral prospecting and ore-grade evaluations; etc. With regard to geochemistry studies, pXRF has already been employed in different settings and scenarios (Gazley et al., 2011; Marsala et al., 2011; Hall et al., 2014; Bourke and Ross, 2015). Recently, the pXRF technique has been shown to be particularly useful for lithogeochemical explorations (Piercey and Devine, 2014; Quye-Sawyer et al., 2015; de Winter et al., 2017).

In the present work, the usefulness of pXRF for the identification and characterization of K–Pg boundary sites is explored. For this purpose, pXRF field measurements have been performed in two well-known marine K–Pg boundary locations in the SE Iberian Peninsula: the Agost and the Caravaca sections. Several previous works have reported comprehensive geochemical information on these two sections (Smit, 1990, 2004; Smit and Hertogen, 1980; Martínez-Ruiz et al., 1992, 1997), showing that they contain a complete sequence of events across the K–Pg horizon. Thus, both sections are useful case-study locations to assess the suitability of the pXRF technique for this type of investigation. It is shown that pXRF provides valuable geochemical data on a number of major and trace elements related to the specific lithology of the K–Pg transition in these two sections, suggesting that this technique may become a useful tool for the characterization of potential new K–Pg boundary sites. The pXRF technique could be even useful in sections where palaeontological or geochemical information is fragmentary or where there is no evident lithological contrast marking the boundary (Ibáñez-Insa et al., 2015).

2. Geological setting

The sections of Agost (Alicante) and Caravaca (Murcia) are located in the external zones of the Betic Cordillera (Southeast of the Iberian Peninsula, see Fig. 1), within the Jorquera Formation. The materials from the Agost section studied in this work were situated next to the Agost-Castalla road and could be easily found. In contrast, the materials from the Caravaca section were placed on top of Barranco del Gredero creek, in an area with small and weathered outcrops located next to a small dump, which makes its precise identification and localization less easy, even with GPS coordinates. Although better, less weathered outcrops can be found

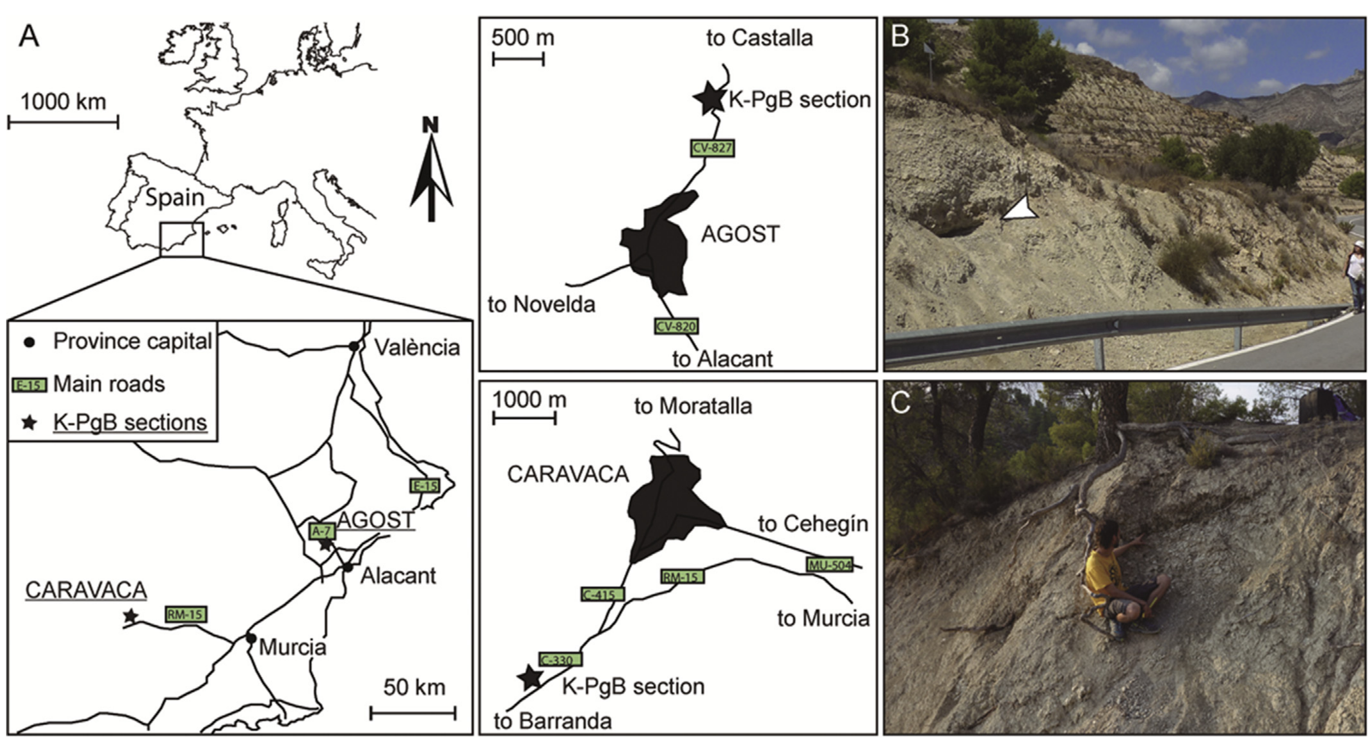

Fig. 1. Location of the Cretaceous–Paleogene (K–Pg) boundary at Agost and Caravaca sections in the SE of Iberian Peninsula (A). The right panels show an overall view of the K–Pg boundary at Agost (B) and Caravaca (C) sections.

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