Forensic characterization of the Wilcox: a new multi-disciplinary approach to support stratigraphic correlation sediment dispersal mapping and reservoir zonation

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### Summary

A multidisciplinary workflow (Sandtrak<sup>®</sup>) showcases crossvalidation methodologies and integration of different data streams in the Palaeocene-Eocene Wilcox Group, to achieve a high-resolution provenance analysis and reservoir zonation. The techniques utilized include elemental ICP, Raman heavy mineral analysis, QXRD and zircon geochronology, a suite of techniques which allows understanding of sediment provenance variations in a way that is accurate and allows high-resolution changes to be identified. The analysis results of the three wells included in this study show a clear correlatable subdivision of the Wilcox Group.

## Introduction

Due to the thickness of the Paleocene-Eocene sediments in the Gulf of Mexico and the laterally extensive, but often discontinuous nature of the sandstones, the differentiation and mapping of sandstones within the Wilcox Group is challenging. This challenge affects the forensic characterization of sandstones, whether regional source to sink provenance studies or when attempting to differentiate sand bodies at reservoir scale. Techniques such as XRD, petrography, automated SEM mineralogy techniques, heavy mineral analysis (HMA) and geochronology have been deployed in the past, however this is often in isolation as, due to the thickness and distribution of the Wilcox Group, a full-scale deployment of any technique is time consuming and costly. A new multi-disciplinary, hierarchical workflow based on the integration of chemostratigraphy, targeted QXRD, zircon geochronology and new Raman heavy mineral and textural analyses has been applied to the Wilcox Group in a series of wells as part of this study, to showcase how an integrated approach can help understand sandstone connectivity better.

This paper focuses on the application of the data acquired by Raman heavy mineral and zircon geochronology to determine changes in sediment provenance. Raman spectroscopy provides quantitative heavy mineral data from the silt and sand fractions, as well as providing images of grains for textural analysis, essential for sediment provenance assessment and reservoir scale zonation.

## Analytical Methods

This paper presents the results of an integrated provenance study based on elemental ICP data, combined with Raman heavy mineral, QXRD and zircon geochronology. Samples were analyzed for inorganic geochemical ICP analysis from three study wells "Well A", "Well B" and "Well C" (Pearce et al. 2023a). Of these, 53 cutting samples were chosen for heavy mineral analysis by Raman spectroscopy, where sample selection was based on suitable sandstone lithology (as determined by the ICP data) and constrained using the high resolution biostratigraphic and chemostratigraphic / mineralogical zonation (Cornick et al. 2023; Pearce et al. 2023c).

Sample preparation methodologies for geochemical and biostratigraphic analysis were conducted as discussed by Cornick et al. (2023) and Pearce et al. (2023a).

During preparation for Raman Spectroscopy, heavy minerals are concentrated utilizing ultrasonic cleaning and density separation with a high-density liquid. This drastically increases the statistical representation of the assemblage (typically 200-500 heavy mineral grains) compared to results derived from traditional petrography or automated SEM mineralogy techniques; and removes operator bias from optical heavy mineral analysis. The heavy mineral separates are inserted into a Horiba XploRA PLUS Raman spectrometer and each grain automatically targeted via image analysis. The spectra of each grain are compared to a database using machine learning which automatically identifies heavy mineral phases. Raman analysis also provides garnet end-member compositions based on a reference microprobe-calibrated garnet model as variations in garnet chemistry are reflected by the Raman spectra response. Additionally, an image of each mineral is recorded which is used for an automated morphological grain analysis providing another layer of sediment provenance in, i.e., transport distance, weathering etc.

Zircon U/Pb dating was conducted on 100 randomly selected zircons per heavy mineral sample. Zircon are mounted and polished in resin pucks and then identified using SEM mapping. Isotopic concentrations are acquired using LA-ICP MS. This allows for a unique age fingerprint of each sample to be established revealing subtle variations of zircon ages and resulting sediment mix.

# Forensic characterization of the Wilcox Group

### Results

A series of key element ratios have been selected to best characterize the geochemical variations observed in the geochemical ICP data acquired from the Wilcox Group (Pearce et al. 2023a). The elements used within these key ratios are associated with changes in climate, provenance and geological processes, and element-mineral affinities are corroborated using the QXRD analysis. The Paleocene sandstones typically have higher quartz and lower feldspar contents reflected by higher Si, and lower Na and K contents, compared to Lower Eocene sandstones. The Paleocene sandstones are also characterized by higher amounts of felsic material reflected by increase in Zr, Th and Nb. Plagioclase (albite) content increases sharply in the Lower Eocene sandstones, along with increases in chlorite and meta-mafic and volcanic accessory phases marked by increases in Fe, Mg, Cr and Sc. Although these signals can be correlated from Alaminos Canyon to Keithley Canyon areas, the magnitudes of enrichments also vary indicating lateral proximal to distal variations can be used map sediment dispersal patterns. Notably, partly altered tuffaceous material (marked by andesine feldspars, analcime and zeolites) is recognized at the top of the Wilcox Group 1A associated with siliceous oozes and carbonates. The provenance geochemical signals have been investigated further using Raman heavy mineral analysis, garnet tying and zircon geochronology.

Late Paleocene heavy mineral assemblages of the Wilcox Group are dominated by zircon (up to 70 %) with subordinate amounts of rutile, garnet, tourmaline, apatite, amphibole and chloritoid. Notably, these sandstones are also higher proportion of type A and Bi garnets (likely sourced from high grade granulite / amphibolite rocks (Morton 1985)), than Eocene-age Upper Wilcox Group assemblages. Similar variations in zircon, apatite and amphibole concentrations are observed across the Paleocene sandstones in the study wells and provide useful markers for sand package correlations. Zircon age concentrations are dominated by Paleoproterozoic assemblages, followed by Mesoproterozoic and Mesozoic aged zircons. Variations in Mesozoic and earlier zircon age assemblages are linked to potential source terrain switches derived from the Western Cordillera and Cenozoic volcanic sources found in western USA (Xu et al. 2017).

The Early Eocene Wilcox Group heavy mineral assemblages show an increase in rutile, apatite and chloritoid concentrations across the studied wells with maximum zircon concentrations of ~30 %, compared to up to 70 % of the Late Paleocene sections. In addition, garnet contents are initially low but increase upwards through this section. Chemical variations in garnet composition are dividing the Upper Wilcox Group into three distinct packages, dominated by type Bii, with an influx of type A and Bi and increase of type Ci garnets toward the top of the Upper Wilcox Group. The zircon age populations from the Eocene age sandstones show a distinct increase in the Cenozoic age group and generally greater variations in Mesozoic and Archean zircon than in the Middle Paleocene sections. This reveals subtle sediment provenance changes that can be linked and correlated across wells. Maximum depositional age zircon detects an evolution from 60 to 55-50 Ma zircons through the Wilcox Group succession related to a switch in hinterland sediment source areas.

The uppermost Wilcox Group has relatively depleted HM assemblages although the presence of pyroxene and amphiboles corresponds to the identification of tuffs in calcareous and siliceous rocks based on elemental and XRD data (analcime, zeolites and Ca-plagioclase) and associated by raised Na, Th, Nb contents.

These stratigraphic variations in elemental data, XRD, heavy minerals and garnet types can be recognized in all 3 study wells though the proportions vary, indicating subtle local fluctuations in provenance. Although Well C has a limited amount of sand packages preserved, the overall trend in HM variations can still be recognized and correlated to Wells A and B.

## Conclusions

Many studies focus on a provenance technique and this can lead to uncertain interpretations, as for example several source terranes can produce similar heavy mineral signatures but can significantly differ in crystallization age as revealed by zircon dating. This multidisciplinary workflow (Sandtrak®) provides a degree of cross validation, and highlights the cumulative benefits of integrating ICP elemental, QXRD, along with Raman heavy mineral analysis, garnet compositional analysis and zircon geochronology to provenance a comprehensive sediment provenance investigation. Further work is ongoing to introduce Raman textural analysis, automated SEM mineralogy techniques and Spotfire<sup>®</sup> data integration. This forensic approach provides a robust toolbox to tackle regional correlation, field scale appraisal and reservoir scale production challenges.

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