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U–Pb calcite age of the Late Permian Castile Formation, Delaware Basin: a constraint on the age of the Permian–Triassic boundary (?)

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Abstract

Organic-rich calcite laminated with gypsum in the evaporitic, non-fossiliferous Castile Formation of the Delaware Basin, southwest USA, yields a Total Pb/U isochron age of 251.5±2.8 Ma (MSWD=1.3). The Castile Formation is almost certainly Late Permian based on its $^{87}Sr/^{86}Sr$ value of 0.706923, identical to the distinct minimum in the late Permian Sr curve. In this paper we explore the potential for using U–Pb calcite ages to date traditionally undatable sections and show how this southern-midcontinent USA (far west Texas and southeast New Mexico) deposit can be correlated to the type section in China. We accept that diagenetic alteration can bias U–Pb results, but the data set we present shows no evidence for such alteration. Clearly with alteration the age we present would represent a minimum age for the Castile Formation. If the age actually dates the Castile Formation then it requires the Permian–Triassic boundary to be younger than 251.5±2.8 Ma.
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1. Introduction

U–Pb has been successfully applied to dating of organic-rich paleosol calcite [1,2] and organic-rich marine cements [3,4]. Organic-rich calcite is common in the rock record, and these studies indicate that it can be enriched in uranium, and that the presence of organic matter may help to preserve the U–Pb system even through later diagenesis. We present results from U–Pb dating of organic-rich calcite of the Castile Formation, a laminated evaporite deposit where calcite layers occur interlayered with anhydrite/gypsum on a mm scale.

The purpose of this study is to place constraints on the age of the Permian–Triassic boundary in North America by dating calcite in the evaporites of the Ochoan Castile Formation. While there are no fossils to constrain the Castile Formation directly biostratigraphically, Ochoan fauna from the nearby overlying Rustler Hills Formation, Delaware Basin [5] (Fig. 1) allows global correlation and the diagnostic marine Sr minima places the Castile Formation below the Permian–Triassic boundary [6–8]. Thus, a depositional age will be

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a maximum age for the Permian–Triassic boundary.

There are several models for the major extinction at the Permian–Triassic boundary when about 90% of marine species were eliminated [9]. Testing of the models is hampered by the incompleteness of the stratigraphic record, because many of the marine deposits from this time are evaporites that are non-fossiliferous. As sea level fell to a Phanerozoic minimum during the Permian, evaporite deposition was reaching a maximum. Permian–Triassic evaporites account for nearly 35% of the world's evaporite resources [10]. While the biostratigraphy of normal marine sedimentary sequences across the Permian–Triassic boundary may be well established, it is not clear how these sequences correlate to those from terrestrial and non-fossiliferous marine sections.

1.1. Regional geology

The Castile Formation was deposited in the Delaware Basin of far-west Texas and southeast New Mexico, USA (Fig. 1). The Delaware Basin and Midland Basin are sub-basins of the Permian Basin. The Permian Basin is thought to represent the most complete Permian record of the southern midcontinent [11]. During the Permian–Triassic transition, the Delaware Basin was located near 10–15° north of the paleoequator [12] and near the western coast of Pangea, resulting in hot desert conditions with high rates of evaporation [13,14].

The Castile Formation is bracketed by the overlying (and partly age equivalent) Ochoan Salado Formation and the underlying Lamar member of the Guadalupian Bell Canyon Formation. The Castile Formation consists of up to 600 m of evaporites with mm scale laminations of gypsum (or anhydrite) and calcite that is stained brown by about 1.5% organic matter (Fig. 2A,B). There are also meter scale cycles that are dominated by anhydrite or halite. Laminations of individual couplets of mm scale are correlated basin-wide and the Castile Formation is thought to be the most complete and continuously laminated evaporite deposit in the world [15,16]. The couplets are interpreted as phytoplankton blooms, which resulted in $CaCO_3$ precipitation that occurred almost instantaneously across the entire basin, followed by deposition of gypsum as salinity increased with a rise in temperature [17]. Assuming the couplets are annual events, the Castile Formation was deposited in 200 000 yr [16]. The lamination couplets have been counted and assembled into a paleoclimate record that is interpreted to contain evidence of Milankovitch orbital forcing [15,16,18].

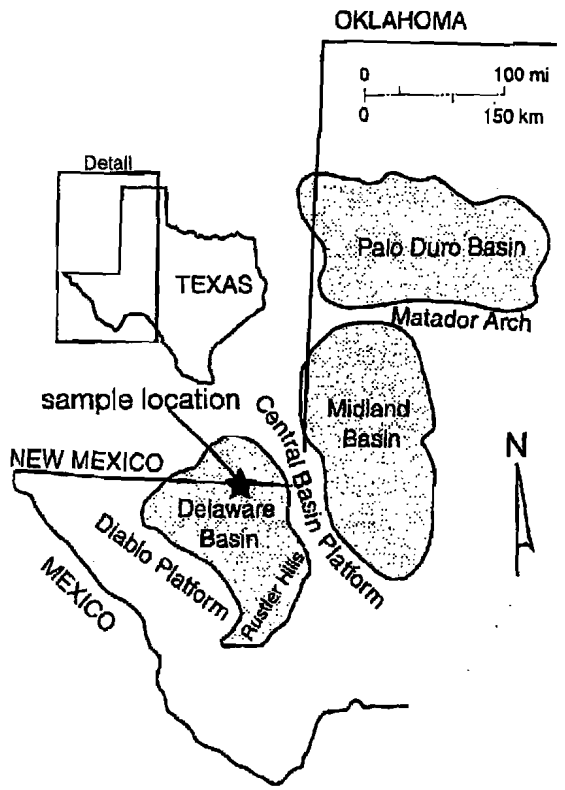

Fig. 1. (A) Generalized map of far-west Texas and southeast New Mexico, USA, with the geographical positions of the Palo Duro and Permian (Midland and Delaware) basins. The Rustler Hills Formation is located in the southeast part of the Delaware Basin. The sample locality for the Castile Formation is the State Line Outcrop, within the Delaware Basin.

1.2. Biostratigraphic relationships

There are no fossils to place direct biostratigraphic constraints on the Castile Formation [5].

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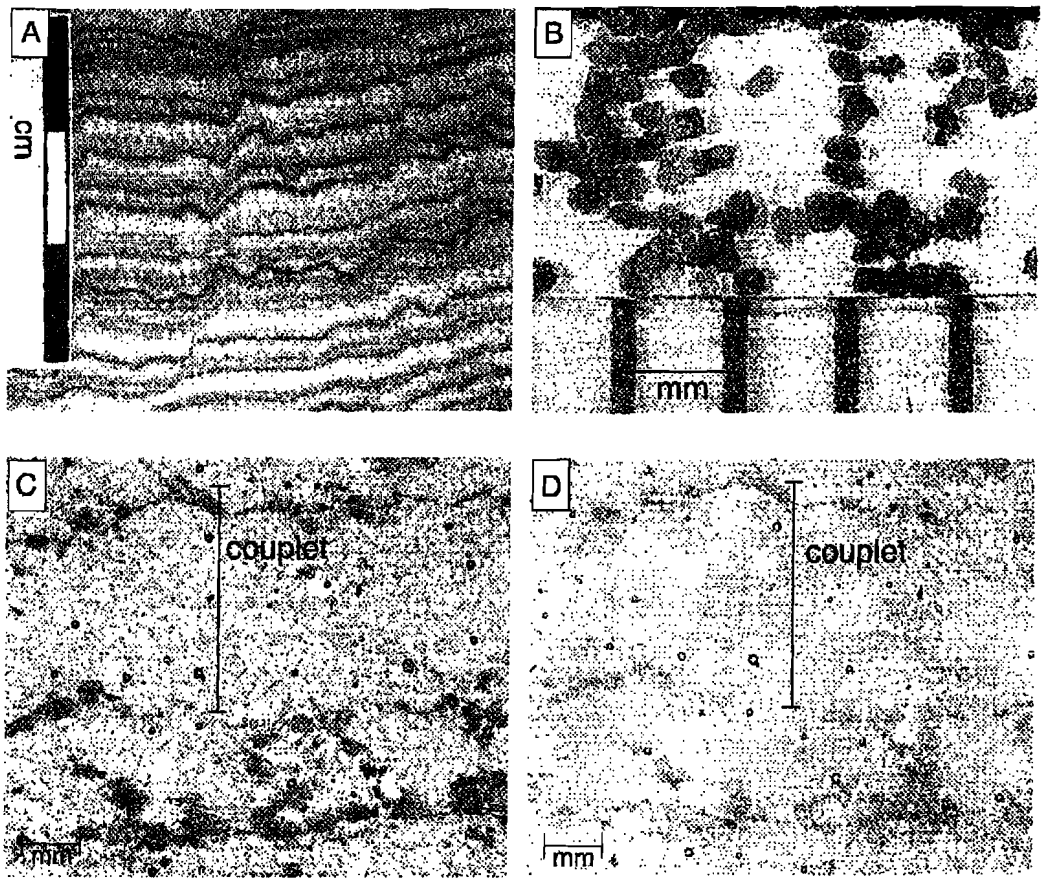

Fig. 2. (A) Laminations of calcite (dark) and gypsum (white) from a State Line Outcrop sample. The continuity of the laminations suggests $CaCO_3$ precipitation occurred instantaneously across what is now the Delaware Basin. The scale bar represents 1 cm. (B) Hand picked clean calcite sample, separated from the gypsum, used for U–Pb analysis. The scale bar represents 1 mm. (C) Plain light photomicrograph of the Castile Formation from the State Line Outcrop. A couplet is composed of one layer of calcite and gypsum. Scale bar is 1 mm. (D) The same photomicrograph, but with a white card [33], which shows the presence of organic material within the calcite. Scale bar is 1 mm.

However, the Ochoan Rustler Formation of the Rustler Hills (Fig. 1), southeast of the State Line Outcrop, clearly overlies the Castile Formation and contains a well defined Permian marine fauna [5,19]. On the basis of these faunas, the Ochoan has been correlated with the Late Permian Tatarian Series of the Russian Ural Platform [19].

1.3. Chemostratigraphy

Based on $^{87}Sr/^{86}Sr$ chemostratigraphy the Castile Formation is Late Permian in age. This relationship has been used to correlate the Castile Formation with evaporites of the Zechstein Basin and marine Tethyan sections [20,21]. Detailed $^{87}Sr/^{86}Sr$ studies of the laminations throughout the thickness of the Castile Formation exhibit characteristics of Late Permian seawater with $^{87}Sr/^{86}Sr$ of 0.706923 [7]. In fact, the Castile Formation records some of the lowest $^{87}Sr/^{86}Sr$ values known in the Phanerozoic, providing a unique signature for comparison to other sections [6,8] (Fig. 3A,B). In the type Chinese section the $^{87}Sr/^{86}Sr$ ratio rises from a value slightly higher than the Castile Formation to a value of 0.70717, just below the 'boundary ash' [6].

Calcite-replaced marine cements from the Permian Reef Complex retain primary aragonite Sr

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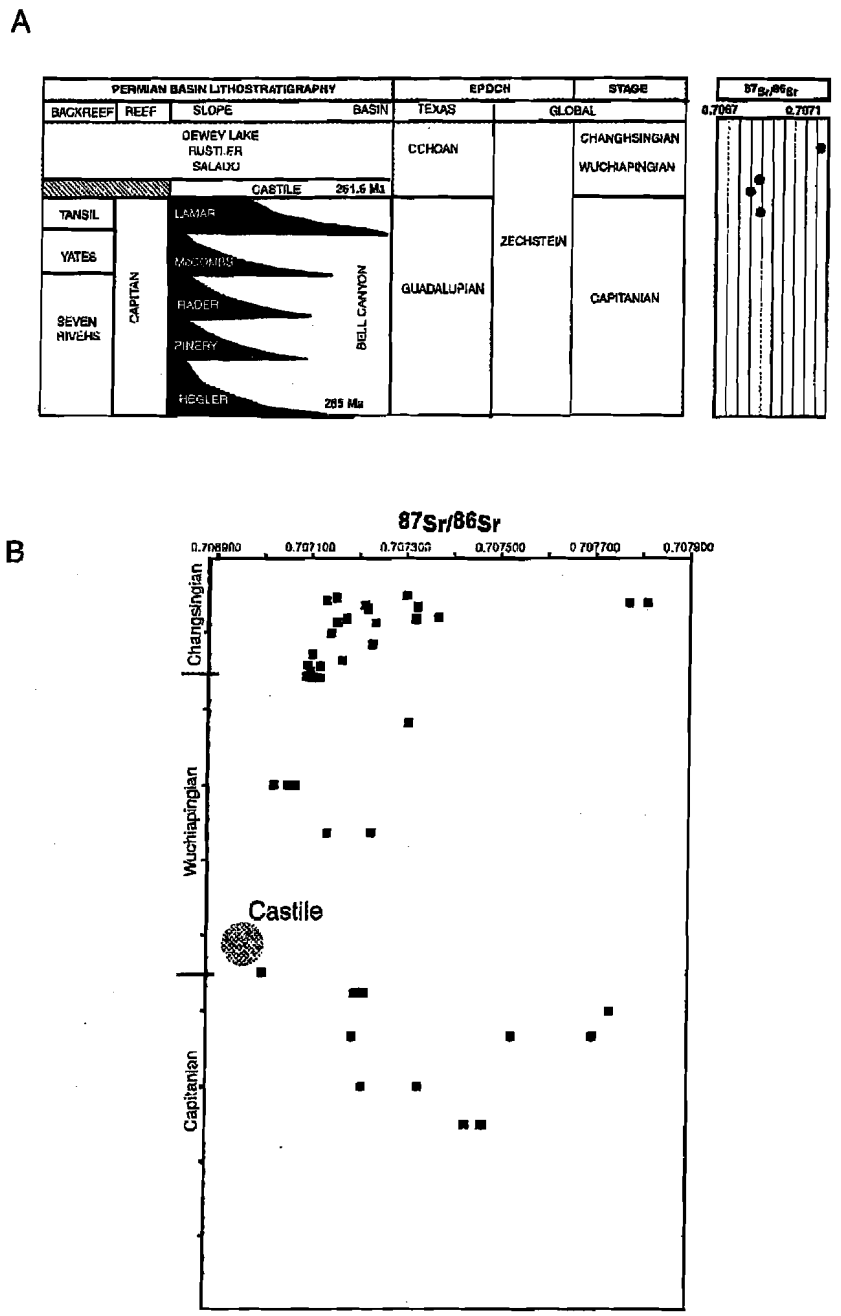

Fig. 3. (A) Detailed stratigraphy of the Permian Basin. $^{87}Sr/^{86}Sr$ values [6] from far-west Texas is plotted with respect to stratigraphic position and no age is implied. The age of 265 Ma for the base of the Capitan is from Bowring et al. [25]. (B) $^{87}Sr/^{86}Sr$ values [8] for the Chinese section up to the Triassic boundary. This shows the profound minimum in the $^{87}Sr/^{86}Sr$ curve. The $^{87}Sr/^{86}Sr$ from the Castile Formation is plotted with respect to its stage correlation. The stage names are taken directly from the data set of [8].

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Table 1
Data from the Castile organic-rich calcite from the State Line Outcrop, Delaware Basin, southeast New Mexico and far-west Texas

	Weight (mg)	Pb (ppm)	U (ppm)	$^{238}U/^{204}Pb$	2σ (%)	$^{235}U/^{204}Pb$	2σ (%)	$^{206}Pb/^{204}Pb$	2σ (%)	$^{207}Pb/^{204}Pb$	2σ (%)	$^{208}Pb/^{204}Pb$	2σ (%)	Error correlations, Total Pb/U isochron ρ 238–206	ρ 235–207	ρ xy	ρ xz (10^{-2})	ρ yz (10^{-3})
C-1	70.34	0.71	4.50	515.18	0.33	3.736	0.33	38.636	0.15	16.653	0.12	38.178	0.17	0.78	0.34	0.16	2.49	−1.13
C-2	73.59	0.74	4.50	486.22	0.34	3.526	0.34	37.524	0.14	16.599	0.12	38.127	0.16	0.70	0.31	0.16	2.50	−1.39
C-3	73.62	0.74	4.58	500.09	0.85	3.627	0.85	38.078	0.14	16.637	0.12	38.216	0.16	0.28	0.13	0.06	0.89	−1.30
C-4	74.58	0.62	4.65	652.85	1.02	4.735	1.02	44.071	0.20	16.926	0.14	38.458	0.18	0.30	0.15	0.04	0.53	−0.55
C-5	91.33	0.60	4.57	670.49	0.39	4.863	0.39	44.910	0.17	16.982	0.12	38.418	0.16	0.67	0.31	0.12	1.77	−0.74
C-7	69.17	0.83	4.61	433.91	0.34	3.147	0.34	35.482	0.13	16.469	0.12	38.105	0.16	0.64	0.29	0.15	2.56	−1.74
C-8	68.58	0.77	4.46	462.32	1.07	3.353	1.07	36.392	0.23	16.589	0.21	38.422	0.24	0.28	0.20	0.03	0.47	−0.55

All ratios are blank and fractionation corrected.

isotope geochemistry. This calcite yields the same $^{87}Sr/^{86}Sr$ ratios of 0.7069 as the Castile Formation. Based on this evidence we interpret these as late marine cements deposited while the basin was filled with evaporitic marine brines during the time of deposition of the Castile Formation. This is consistent with the model of Kirkland et al. [7], who hypothesize that the Sr isotopic signature of the Castile Formation is derived from marine seawater which recharged the basin from the direction of the Permian Reef Complex.

1.4. Age of the Permian–Triassic boundary

Testing of the models for this major extinction event depends on precisely dating the boundary, i.e. with an uncertainty of one million years or less [9]. The age obtained for the Permian–Triassic boundary based on U–Pb dating of zircon from ash layers above and below the boundary within the South China marine stratotype at Meishan are under debate because of the range of concordant U–Pb ages for zircon, up to 15 million years, found within individual ash layers [9]. Decisions as to which zircon ages to accept were based on evaluating the importance of inheritance or lead loss. The assumption in these studies is that igneous zircons found in a given ash crystallized only shortly prior to the development of the ash because the lifetime of most magma bodies is similar to or less than the analytical uncertainties. A bentonite bed immediately below the Permian–Triassic boundary (18 cm below) in conjunction with ages in ash beds above and below the boundary has been the locus of attempts to precisely date the boundary. The weighted mean $^{206}Pb/^{238}U$ SHRIMP age for individual zircons from this bed is 251.2 ± 3.4 Ma [22]. Sanidine gives a mean $^{40}Ar/^{39}Ar$ age of 249.9 ± 0.3 Ma [23], which adjusts to an U/Pb age of 255 Ma if corrected by 2% [24]. A weighted mean $^{206}Pb/^{238}U$ age of selected concordant zircon U–Pb ages using TIMS on single and multiple zircons yields 251.1 ± 0.3 Ma [25]. Single zircon analyses by TIMS in [9] and the single zircon analyses in [25] have a range in concordant U–Pb ages from 241 to 257 Ma [9]. However, based on an age 9 cm above the boundary of 252.5 ± 0.3 Ma, Mundil et al. [9] proposed

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that the age of the boundary is older than 253 Ma.

It is quite clear that there are complexities in the U–Pb ages for zircon that are not yet well understood. These complexities may be due to lead loss, extended periods of zircon crystallization prior to eruption, or inheritance that lead to age discrepancies that are much larger than the analytical uncertainty [9].

2. Analytical procedures

The samples for this study are calcite interlayered with gypsum on a mm scale (Fig. 2) from the State Line Outcrop (Fig. 1), located within the Anhydrite III unit [13]. However, only the calcite was used in evaluation of U–Pb. A hand sample was crushed with a mortar and pestle, to medium sand-size grains. The crushed sample was then washed with water five times in a sonicator to remove any dust and rinsed with acetone to dry. Seven 70–90 mg aliquots of brown calcite (Fig. 2B) were separated by hand at 10× magnification on a binocular microscope and every effort was made to avoid sampling gypsum. The aliquots were spiked with a mixed ^{205}Pb–^{236}U tracer and dissolved with 1 N HNO_3. Pb and U were co-precipitated in a Fe hydroxide–ammonia step. Pb was separated using standard HBr anion exchange chemistry. U was separated using HNO_3 anion exchange chemistry. Pb samples were loaded with silica gel and phosphoric acid for fractionation control on single Re filaments and run on a Finnigan Mat 262 at 1250°C in static mode. Pb was corrected for a fractionation of 0.11 (0.01)% per atomic mass unit based on 15 runs of SRM 982; average values and 2σ standard deviations for 15 runs are $^{206}Pb/^{204}Pb = 36.6830 \pm 0.0075$, $^{207}Pb/^{206}Pb = 0.466599 \pm 0.00004$, and $^{208}Pb/^{206}Pb = 0.99809 \pm 0.00034$. The total procedural Pb blank was 150 ± 50 pg. U was loaded with graphite and H_3PO_4 on a single Re filament and run on a Finnigan MAT 262 at 1800°C in static mode. Multiple runs of the U standard U930 showed no need for fractionation correction for U. All of the data were reduced using the PBDAT program of Ludwig [26].

3. Results

The analytical data for seven aliquots of brown calcite are given in Table 1. All reported uncertainties are 2σ. The data were evaluated using Isoexcel [27]. A $^{238}U/^{204}Pb$–$^{206}Pb/^{204}Pb$ isochron yields an age of 252 ± 5 Ma (MSWD = 2.5) and a $^{235}U/^{204}Pb$–$^{207}Pb/^{204}Pb$ isochron provides an age of 252 ± 25 Ma (MSWD = 2.5) (Fig. 4). The initial $^{206}Pb/^{204}Pb$ and $^{207}Pb/^{204}Pb$ ratios are

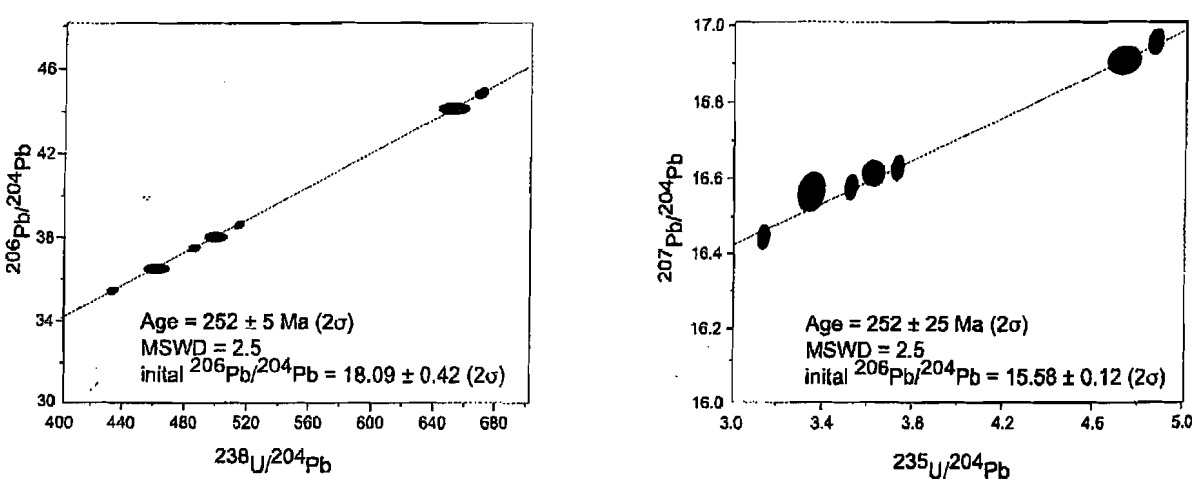

Fig. 4. (A,B) ^{238}U–^{206}Pb and ^{235}U–^{207}Pb isochron plots, showing isochron ages and initial isotopic compositions derived from the isochron.

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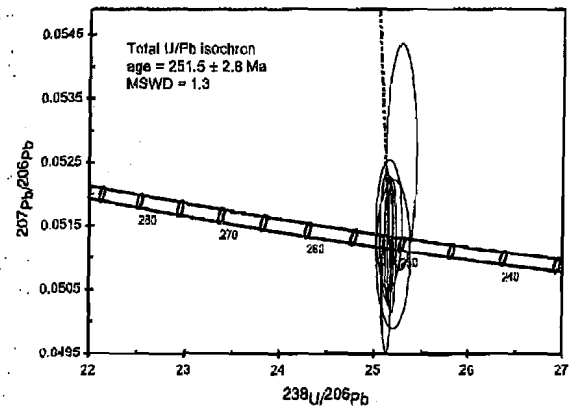

Fig. 5. A total Pb/U isochron plot with axes of $^{207}Pb/^{206}Pb$–$^{238}Pb/^{206}Pb$–$^{204}Pb/^{206}Pb$. The 3-D linear regression falls on the concordia curve, strongly suggesting that these samples have an undisturbed U–Pb system and share an identical common Pb component.

18.09 ± 0.42 and 15.58 ± 0.12, respectively (Fig. 4). For a system that has remained closed since its formation, a Total Pb/U isochron provides the optimum solution, taking into account all U/Pb and Pb/Pb ratios [27–29]. The Castile Formation yields a Total Pb/U isochron age of 251.5 ± 2.8 Ma with a MSWD of 1.3 (Fig. 5), and an initial $^{206}Pb/^{204}Pb = 18.17 \pm 0.23$ and $^{207}Pb/^{204}Pb = 15.58 \pm 0.02$. The $^{206}Pb/^{204}Pb$ and $^{207}Pb/^{204}Pb$ initial values are quite reasonable for rocks of this age and region. Jones et al. [4] report initial isotopic compositions of $^{206}Pb/^{204}Pb = 17.20 \pm 1.74$ and $^{207}Pb/^{204}Pb = 15.595 \pm 0.140$ for carbonates of the Permian Reef complex, within the uncertainty of our more precise values.

4. Discussion

The U–Pb data for the Castile Formation yield a Total U/Pb isochron age of 251.5 ± 2.8 Ma (2σ) (MSWD = 1.3) with reasonable initial Pb isotopic compositions. These provide sound evidence that this age should reflect the time of deposition. The $CaCO_3$ in the Castile Formation has been interpreted to have formed annually. At the salinity reflected by the precipitation of gypsum, aragonite would be the favored phase. There is little constraint on when the aragonite was converted to calcite, but the Sr isotopic composition of the calcite is identical to the gypsum and anhydrite ($^{87}Sr/^{86}Sr = 0.706923$) [7] and the Sr and U concentrations are fairly high, >2000 ppm [6,7] and 4.5 ppm, respectively, suggesting that these elements were not lost during conversion. We have to recognize that the calcite age may give the time of diagenetic alteration. However, the coherence of the U–Pb data set and the retention of marine Sr signatures argue that any diagenesis happened quite early in the history of the Castile Formation calcite. Possible initial heterogeneity in the Pb isotopes due to the short residence time of Pb in oceans [30] as well as a clear history of dehydration of the gypsum to anhydrite [31] and then, in the case of the State Line Outcrop, back to gypsum [13] certainly may explain the slight scatter. Therefore our preferred interpretation is that the U–Pb age of 251.5 ± 2.8 Ma dates the deposition of the Castile Formation within the stated uncertainties and further that this provides a maximum age constraint for the Permian–Triassic boundary. The existing radiometric ages for Ochoan units of the Delaware and Palo Duro basins (Fig. 6) are consistent with the Permian–Triassic boundary occurring somewhere in this sequence of largely intercalated terrestrial and evaporite deposits. This hypothesis remains to be tested by dating other sections that are well constrained with re-

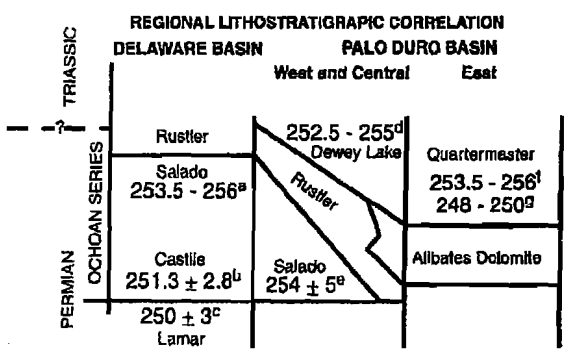

Fig. 6. Stratigraphical correlation of the Delaware and Palo Duro basins (see Fig. 1). This figure also shows the current assignment of Late Permian radiometric ages for Ochoan units, including the age of the Castile Formation from this study. The Ar/Ar ages have been adjusted (1–2%) to reflect the U–Pb age. [a] Ar/Ar (langbeinite), [34]; [b] U–Pb (calcite), this report; [c] U–Pb (carbonate), [4]; [d] Ar/Ar (biotite), [34]; [e] Rb–Sr (clay), [35]; [f] Ar/Ar (sanidines), [36]; [g] U–Pb SHRIMP (zircons), [36].

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spect to the diagnostic Sr curve. The age of the Castile Formation may be consistent with the U–Pb zircon age of 251.1 ± 0.3 Ma [25] for the Permian–Triassic boundary and is not even inconsistent with an age greater than 253 Ma proposed by Mundil et al. [9] for the boundary. The time represented by the $^{87}Sr/^{86}Sr$ minimum is poorly constrained [32] and thus the age of the Permian–Triassic boundary is allowed to be much younger than the Castile Formation. Clearly the complications in the zircon data from the Permian–Triassic boundary of South China [9] suggest that U–Pb ages for zircons sometimes make it difficult to evaluate the time of deposition of a volcanic ash. To test our conclusion that the U–Pb age of the Castile Formation places a maximum constraint on the Permian–Triassic boundary, more ages from carbonates that constrain the Sr curve from marine sections on other continents are needed.

Acknowledgements

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