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The Age and Geochemistry of Volcanic Ash in the Catahoula Formation of Louisiana, Mississippi, and Texas, USA

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ABSTRACT

New radiometric dating of volcanic ash in Oligocene-Miocene nonmarine deposits in Louisiana, Mississippi, and Texas reveals regional distribution of early Oligocene volcanic ash across the northern part of the Gulf of Mexico from Texas to Mississippi. Fallout volcanic ash in the Chalk Hills Member of the lower Catahoula Formation type section is dated with cooccurring sanidine and zircon, yielding a sanidine $^{40}\text{Ar}/^{39}\text{Ar}$ date of 30.149 ± 0.011 Ma and a zircon U/Pb date of 29.98 ± 0.05 Ma; bentonite in quarry exposure of the Glendon Formation at Brandon, Mississippi, yields a zircon U/Pb date of 32.59 ± 0.13 Ma. These dates are close to the age of 30.65 ± 0.06 Ma volcanic ash in the lower Catahoula Formation of east Texas. The geochemistry of the Louisiana Chalk Hills Member volcanic ash matches that of the lower part of the Catahoula Formation in Texas. Trace-element and rare earth element (REE) compositions of single glass shards of east Texas volcanic ash reveal a dual-source composition. Trace-element and REE compositions indicate a normal mid-ocean ridge basalt composition and a subduction-melt source. This correlates with the west North American ignimbrite flare-up of Mexico. The problematic palynological late Miocene (~ 9 Ma) dating of upper Catahoula Formation strata in Catahoula Parish, Louisiana, is reconciled by proposing the presence of a fault in the Catahoula Formation type section. Better dating of the Catahoula Formation is needed to improve reconstructions of Oligocene-Miocene continental climates, drainage patterns, and paleohydrology in the region.

Introduction

Oligocene-Miocene strata exposed in the outcrop belt of north-central and northwestern parts of the Gulf of Mexico basin are mostly nonmarine deposits that lack detailed dating, apart from current radiometric dating of volcanic ash (Heintz et al. 2015; Yancey et al. 2018) and the work presented in this report (fig. 1). Studies of paleoclimate, paleodrainage, and sediment sources have relied on sequence unit correlations from marine deposits of the Gulf of Mexico basin (Galloway 1989; Brown and Loucks 2009; Galloway et al. 2011; Xu et al. 2017) and on inferences of placement of depositional environments within fluvial and/or coastal plain depositional models applied to thick intervals of strata (Galloway et al. 1977,

1986). These are the basis for site-specific geological studies (e.g., Albertson et al. 1986) that are constrained by age and correlation uncertainties. Age-diagnostic fossils deposited in the strata have been destroyed by diagenetic dissolution, making correlation across facies boundaries impractical. The Catahoula Formation contains most of the Oligocene-Miocene deposits in Louisiana and adjacent Mississippi, while coeval deposits in Texas are placed in the Catahoula and Oakville Formations (fig. 2), which are 100+ m thick (Renick 1936; Godfrey et al. 2018). They contain a long-duration record of nonmarine conditions in the northern Gulf of Mexico, but this record is not supported with reliable age control. Vertebrate fossils of the Fleming Formation in Louisiana and eastern Texas indicate that the upper boundary of the Catahoula Formation lies within the middle Miocene (Williams and Schiebout 2003; Schiebout et al. 2006).

Manuscript received July 5, 2018; accepted November 12, 2018; electronically published January 28, 2019.

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[The Journal of Geology, 2019 volume 127, p. 000–000] © 2019 by The University of Chicago.
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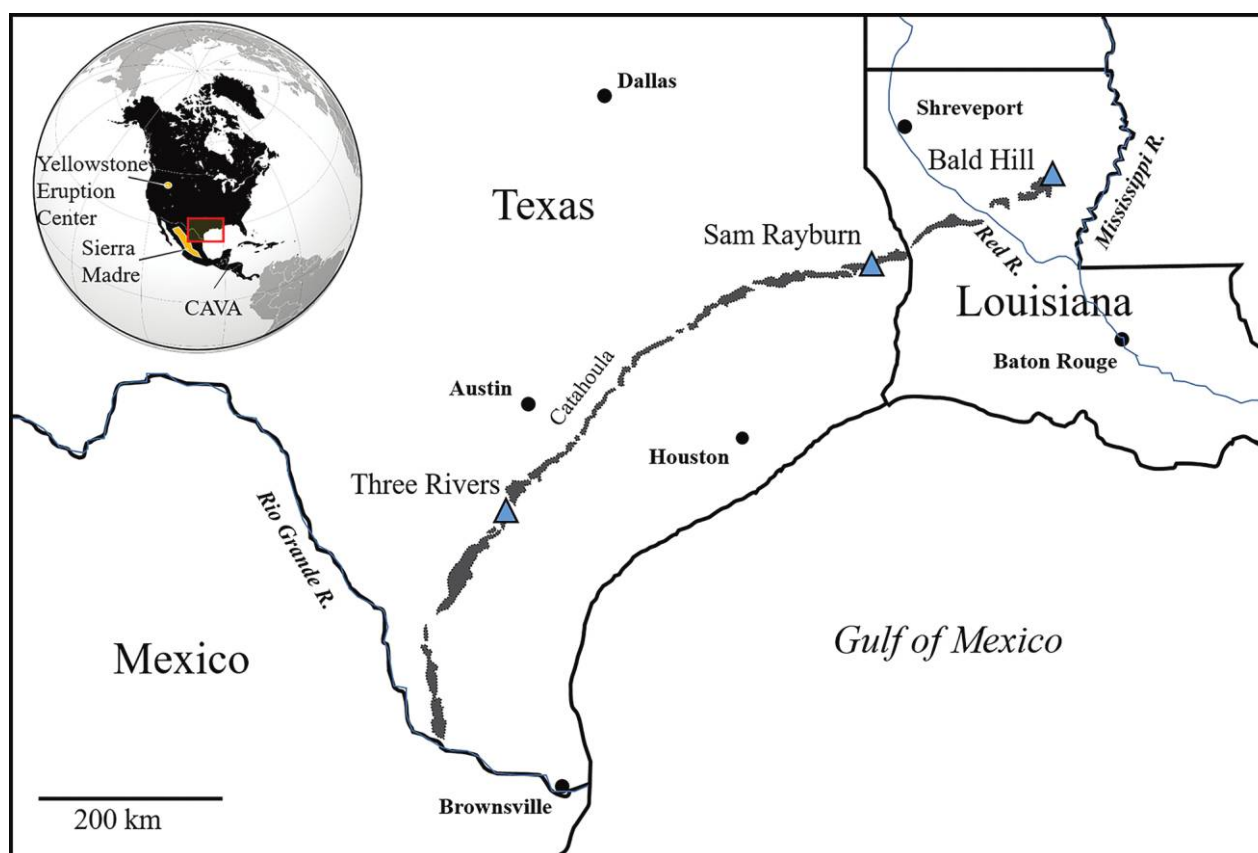


Figure 1. Outcrops of the Catahoula Formation across Texas and Louisiana (gray); gaps in the belt are areas covered by younger deposits. Triangles represent sample collection sites. Inset map shows locations of the Sierra Madre, the Yellowstone eruptive center, and the Central American Volcanic Arc (CAVA) relative to the Catahoula Formation outcrops.

The Oligocene–early Miocene was a time of great climatic change, of continental cooling and drying and the appearance of large glaciers on Antarctica (Zanazzi et al. 2007; Liu et al. 2009; Miller et al. 2009). The potentially valuable record of these changes in continental deposits of south-central North America remains inaccessible without reliable dating. The Catahoula Formation and its equivalent Catahoula plus Oakville Formations in Texas remain poorly dated along its entire exposure trend from Texas to Mississippi (Brown and Loucks 2009; Dockery and Thompson 2016). The Catahoula Formation is best known for containing beds of volcanic ash in non-marine strata, but this is evident only in the lower parts of the formation. Consequently, the formation is identified by varying lithologic criteria along its outcrop trend and, as currently defined, has formation boundaries that are time transgressive to the east.

Palynological dating of lignite in the upper Catahoula Formation in Catahoula Parish (Wrenn et al. 2004) brought the subject of dating Catahoula For-

mation strata to a critical concern. Wrenn et al. (2004) reported an early late Miocene age (8.68 Ma) for the upper part of the formation in northern Louisiana. This is disconcerting because it suggests a 20-My age difference between the lower and upper parts of the Catahoula Formation in the type area of the formation. Inasmuch as this region is characterized by high rates of sediment accumulation (Hardin 1962), such a long time span for deposition of a formation is unrealistic. There is no clear way to resolve this question when age control for nonmarine strata is lacking and lithostratigraphic tracing of formation units lacks enough precision to provide reliable age control.

This presence of volcanic ash in the Catahoula Formation provides a means of determining age and reducing uncertainty about correlations of the formation. The lower part of the formation in Louisiana contains beds of volcanic ash (Chawner 1936; Fisk 1938, 1940), and volcanic glass is reported to occur in sandstones of the formation across Louisiana (Chawner 1936; Fisk 1938; Paine and Meyerhoff 1968).

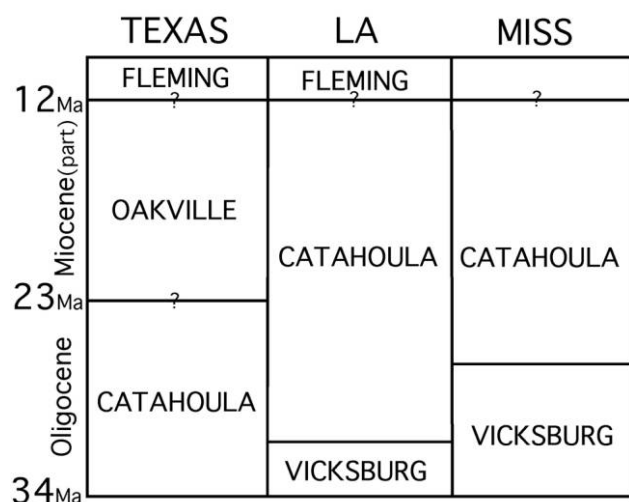


Figure 2. Oligocene to middle Miocene formations of Texas, Louisiana, and Mississippi, placed on the geological timescale of the International Commission on Stratigraphy, 2012 version, with ages of stage boundaries rounded to whole numbers. Ages of formation boundaries are approximations, reflecting past and present convention. Formation boundaries with question marks are those with greatest uncertainty of age.

Dating and geochemical characterization are documented here for early Oligocene volcanic ash in Louisiana, Mississippi, and Texas.

Geochemical characterization is also needed to determine the source of volcanic ash. A Miocene date for what has been regarded as the upper Catahoula Formation in Louisiana presents the possibility that volcanic ash derived from Yellowstone hotspot volcanism could be present in the formation, in addition to volcanic ash from the Oligocene ignimbrite flare-up event in western North America (Heintz et al. 2015; Yancey et al. 2018). Volcanic ash attributed to a Yellowstone hotspot source is reported in marine subsurface strata in Louisiana (Hintze 1967; Paine et al. 1968). However, geochemical composition data on volcanic ash can distinguish between the contrasting sources of subduction-zone origin and continental

crust-derived silicic volcanism, providing a means of distinguishing between the two sources of silicic volcanic ash.

Three samples provide geochemical data on volcanic ash in the lower Catahoula Formation (table 1): (1) a 1.5-kg sample of volcanic ash from the Chalk Hills Member of the formation in the headwaters of Sugar Creek, at a location called Bald Hill, Catahoula Parish, Louisiana, in the center of the southern half of section 18, Township 10N, Range 5E (northwestern Aimwell 7.5-minute quadrangle), 5 km (3 miles) south of the town of Rosefield; (2) a 1.5-kg sample of volcanic ash from near the dam site of Sam Rayburn Reservoir in Jasper County, far eastern Texas (Ledger 1988), supplied by E. Ledger of Steven F. Austin State University, Nacogdoches, Texas; and (3) a 5-kg sample of volcanic ash from the lower Gueydan Formation (Catahoula) collected from a site 16 km (10 miles) north of Three Rivers, Live Oak County, Texas, at measured section 1 (location L-10) of McBride et al. (1968).

Catahoula Formation

The Catahoula Formation in northern Louisiana is an interval of mudstone, sandstone, tuffaceous sandstone, and volcanic ash (Chawner 1936). The type area is located in bluffs along the Ouachita River and in the Chalk Hills of Catahoula Parish (fig. 1). In the Chalk Hills area, the formation is up to 110 m thick and is divided into a basal Cassel Hill Member, an overlying tuffaceous Chalk Hills Member, and a thicker top unit not divided into members (fig. 3). Chawner (1936) noted that the 15-m Chalk Hills Member contains a 2.4-m white volcanic ash layer in the Bald Hill area of western Catahoula Parish and that volcanic ash occurs in other layers. The Bald Hill volcanic ash of the Chalk Hills Member consists mostly of small (0.01-mm diameter; fig. 4) glass shards and contains few nonglass particles. Some sandstone beds in the Catahoula are completely silica cemented and occur as sedimentary quartzite. At Catahoula Shoals bluff on the Ouachita River, the formation is ~30 m thick, and a similar thickness

Table 1. Location Data and Dates for the Bald Hill, Sam Rayburn, and Three Rivers Samples of the Catahoula Formation of Louisiana and Texas

Sample	Decimal latitude	Decimal longitude	Age (Ma)
Brandon	32.262055	-89.969123	32.59 ± .13
Bald Hill ⁴⁰ Ar/ ³⁹ Ar	31.859121	-92.002006	30.13 ± .01
Bald Hill U/Pb	31.859121	-92.002006	29.98 ± .05
Bald Hill (whole rock)	31.859121	-92.002006	24.1 ± 1.1
Sam Rayburn	31.062366	-94.108534	30.65 ± .06
Three Rivers	28.589270	-98.212530	32.72 ± .03

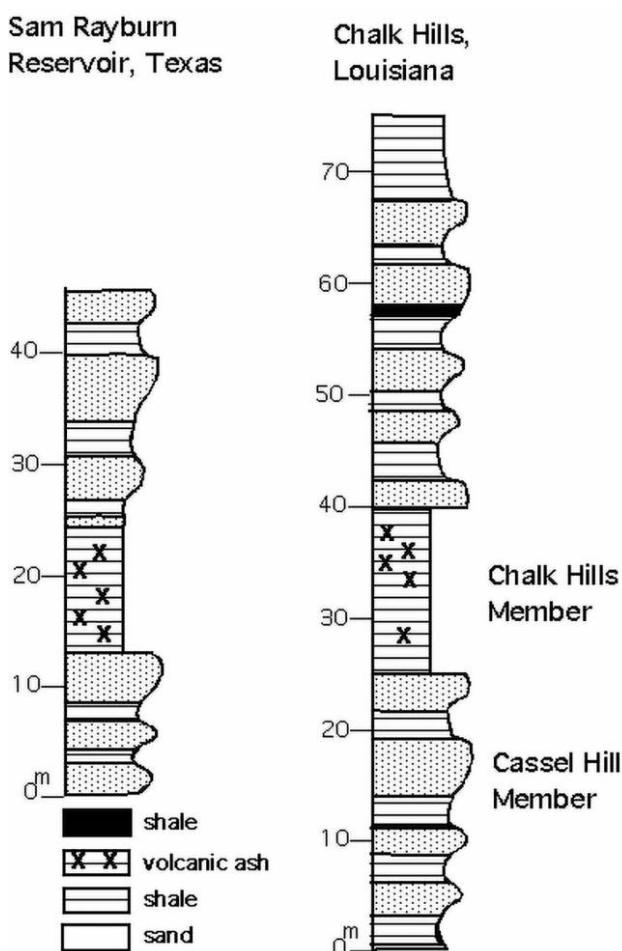


Figure 3. Stratigraphic sections of Catahoula Formation collection sites providing samples in east Texas (Ledger 1988) and north Louisiana (Chawner 1936).

(26 m) overlies the bluff strata in nearby Big Creek on Sicily Island (Chawner 1936).

Fisk (1938) reports a twofold subdivision of the Catahoula Formation in Grant and LaSalle Parishes, west of Catahoula Parish, with a 17-m-thick lower member of siltstone and volcanic ash and an upper sandstone and mudstone member of undetermined thickness. Silica-cemented sandstone is prominent in the middle of the formation, mostly in the top part of sandstone beds. The lower member of Fisk (1938) is similar to the combined Cassel Hill Member and Chalk Hills Member of Chawner (1936). The occurrence of resistant silica-cemented sandstone is characteristic of the middle of the Catahoula Formation in Louisiana. This interval of resistant silica-cemented sandstone can be traced into the adjoining states of Texas and Mississippi.

In central Mississippi, the Catahoula Formation is ~100–150 m thick and composed of mudstones

and sandstones, with many beds of silica-cemented sandstone in the lower part of the formation (Dockery and Thompson 2016). In east Texas, the Catahoula Formation is limited to an interval with volcanic ash, mudstone, paleosols with pedogenic carbonates, and sandstones that occur below an interval dominated by silica-cemented sandstones that is placed in the Oakville Formation (Renick 1936; Brown and Loucks 2009). The Oakville Formation name is limited to Texas, but stratigraphic units dominated by silica-cemented sandstones occur above volcanic ash-bearing strata eastward into Louisiana and Mississippi and are included in the Catahoula Formation of those areas. This implies that the Catahoula Formation of Texas could be much older than the Catahoula Formation of Mississippi (fig. 2).

Variable lithologic criteria for identifying the limits of the Catahoula Formation make inferred age determinations within the formation unreliable. The base of the formation is placed at the appearance of volcanic ash within nonmarine strata in Texas and Louisiana, but in Mississippi, volcanic ash (as bentonite) occurs within marine strata below the Catahoula Formation. The upper boundary of the Catahoula Formation is set at the base of the Oakville Formation in the outcrop belt of Texas, where it is assumed to approximate the Oligocene-Miocene boundary (Galloway 1989; Brown and Loucks 2009), whereas in Louisiana it is set at the base of the Fleming Formation. The age of this boundary is uncertain in most places, varying from the older than middle Miocene of the Castor Creek Member, Fleming Formation, in southwestern Louisiana (Williams and Schiebout 2003) to the older than late Miocene–early Pliocene in southeastern Louisiana (Schiebout et al. 2006). The early late Miocene date of Wrenn et al. (2004) in northern Louisiana is 20 My younger than the age of volcanic ash in the lower Catahoula Formation (Heintz et al. 2015; this study).

Analytical Methods

Elemental composition of a whole-rock split of the Bald Hill sample was determined by laser ablation inductively coupled mass spectrometry (LA-ICPMS) at Michigan State University, where Sr and Zr, determined separately by X-ray fluorescence, were used as internal standards. Elemental composition of a whole-rock split for the Sam Rayburn sample was determined by LA-ICPMS on a powdered whole-rock sample at Activation Laboratories, Ancaster, Ontario, Canada. Whole-rock elemental analysis was not obtained for the Three Rivers sample.

Elemental analyses of glass shards in the Bald Hill and Three Rivers samples were done at the LA-ICPMS

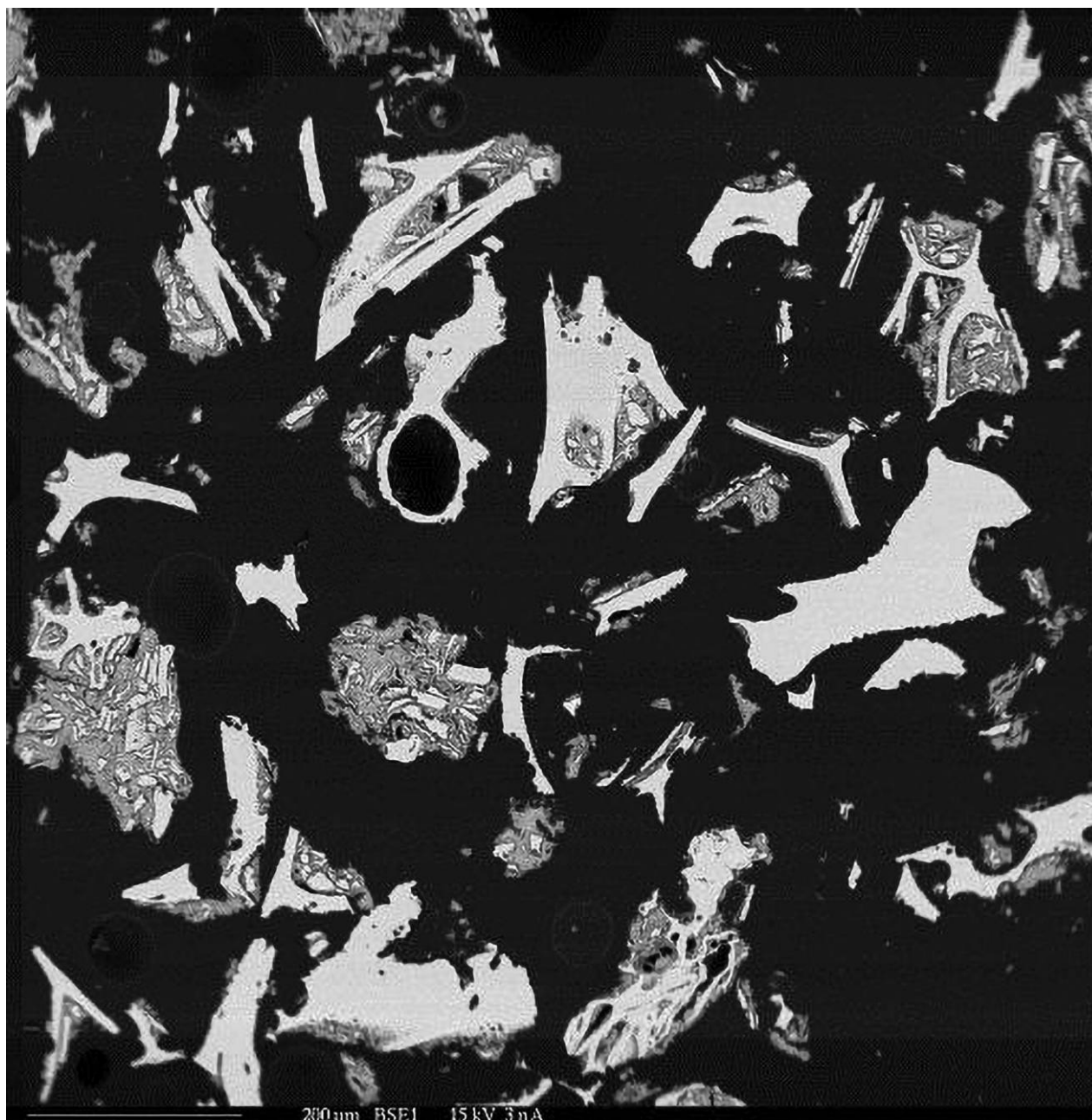


Figure 4. Photomicrograph of volcanic glass in the Bald Hill sample, Chalk Hills Member of the Catahoula Formation. Glass shards and clay matrix dominate the composition of this sample.

lab at Columbia University's Lamont-Doherty Earth Observatory. Elemental composition of glass shards of the Sam Rayburn sample was determined by electron microprobe at Texas A&M University, but a second analysis on six individual glass shards was also done at the LA-ICPMS lab at the Lamont-Doherty Earth Observatory.

A whole-rock K-Ar date for the Bald Hill sample was determined at the Earth Science and Resource Engineering Group at the Commonwealth Scientific

and Industrial Research Organisation (CSIRO), Australia. Potassium content was determined in duplicate by atomic absorption (Varian Spectra AA 50) using cesium at a 1000-ppm concentration for ionization suppression. Two approximately 50-mg sample aliquots were dissolved with HF and HNO₃. The samples, once in solution, were diluted to within a range of 0.3–1.5 ppm K for the atomic absorption analysis. The pooled error of duplicate K determination of all samples and standards is better than 2%. The

K blank was measured at 0.34 ppm (=0.000034%) K. Ar isotopic determinations were performed with a procedure similar to that described by Bonhomme et al. (1975).

The sanidine $^{40}\text{Ar}/^{39}\text{Ar}$ dates for all three samples were obtained at the Geochronology Laboratory of the New Mexico Bureau of Geology and Mineral Resources (NMGR), Socorro, using phenocrysts in the sand size fraction. Sanidine $^{40}\text{Ar}/^{39}\text{Ar}$ dating for all samples was calculated as the weighted mean age from single-crystal analyses. As part of the analysis, the prepared samples were irradiated in the NM-293 package for 8 h at the US Geological Survey TRIGA reactor in Denver, Colorado, along with the standard Fish Canyon tuff sanidine as a neutron flux monitor. The sanidine was then analyzed by the laser fusion method, with a CO_2 laser to fuse the sample. The $^{40}\text{Ar}/^{39}\text{Ar}$ dates from NMGR were obtained by dating individual sanidine phenocrysts, then combining the grain ages to produce a sample date based on calculating a mean for the dominant grain population in the sample. Culling of outlier grain ages continued until the mean square of weighted deviates was between 1 and 3 (see Heintz et al. 2015 for a discussion of this method). Additional details of procedures used for $^{40}\text{Ar}/^{39}\text{Ar}$ dating and the analysis of results are available from the Geochronology Laboratory in Socorro, New Mexico.

Zircon U/Pb analyses were conducted by thermal ionization mass spectrometry isotopic dilution (ID-TIMS) in the R. Ken Williams ^{45} Radiogenic Isotope Geosciences Laboratory at Texas A&M University. All Pb and U isotope ratio analyses were conducted at Texas A&M University on a Triton thermal ionization mass spectrometer. The U/Pb date was also obtained by combining the grain ages to produce a sample date based on calculating a mean for the dominant population in the sample. "YourLab" algorithms (Schoene et al. 2006; Schmitz and Schoene 2007) were used for data reduction, and IsoPlot 3.7 (Ludwig 1991) was used for plotting concordia diagrams. Additional details of procedures used for U/Pb dating and the analysis of results follow those described in Yancey et al. (2018).

Radiometric Dating of the Catahoula Formation in Louisiana

As a means of resolving uncertainty about the age of the Catahoula Formation in its type area, revealed by late Miocene palynology dating of the upper part of the formation (Wrenn et al. 2004), volcanic ash in the Chalk Hills Member was sampled for radiometric dating and a geochemical characterization (McGuire et al. 2010). Preliminary dating was done with

the K/Ar method on a whole-rock sample of volcanic ash with abundant glass shards and associated clays and a small amount of silicate grains. This analysis yielded a date of 24.1 ± 1.1 Ma, a much younger age for the Catahoula Formation than in areas to the west (Heintz et al. 2015), raising doubts about the usefulness of the K/Ar date as an indication of the age of deposition of the Catahoula Formation in Louisiana.

The young date from the preliminary work is an expected result of using K/Ar dating on a whole-rock sample composed mostly of glass shards. Glass alters rapidly by hydration, followed by alteration and loss or migration of mobile elements. Both potassium and argon are prone to migration and loss in glass as it alters. The amount of alteration was not estimated for the first sampling, but examination of the resample showed that the glass shards have an appearance similar to east Texas glass shards shown to be hydrated but having little alteration, as determined by microprobe analysis (Yancey and Guillemette 1998).

Louisiana volcanic ash was resampled in 2016 by Paul Heinrich at Bald Hill, a site within the Sugar Creek watershed where Chawner (1936) described the lower members of the Catahoula Formation in Louisiana. The resample $^{40}\text{Ar}/^{39}\text{Ar}$ date presented here is compatible with high-precision sanidine dates for Texas samples (Heintz et al. 2015), and the resample date is considered to be a reliable age of deposition for the Chalk Hills Member. Upon processing to remove fines and adhering clay, the sample was split into low-density and high-density fractions, and the low-density fraction was sent for $^{40}\text{Ar}/^{39}\text{Ar}$ single-crystal sanidine dating at New Mexico Tech and the high-density fraction retained for single-crystal zircon ID-TIMS dating at Texas A&M University. The cooccurrence dates yielded a high-precision $^{40}\text{Ar}/^{39}\text{Ar}$ sanidine date of 30.13 ± 0.01 Ma and a U/Pb zircon date of 29.98 ± 0.05 Ma (table 1). The Chalk Hills Member volcanic ash is of early Oligocene age (Cohen et al. 2013). These two cooccurring sample dates are within the margin of error of each other and confirm a high-precision date for the Bald Hill sample of volcanic ash in northern Louisiana.

A new ID-TIMS zircon date is available for a bentonite at the base of the Glendon Formation in quarry exposures at Brandon, Mississippi (Dockery et al. 2017; LaFleur et al. 2018). Prismatic, nondetrital grains in the zircon assemblage yield a U/Pb zircon date of 32.59 ± 0.13 Ma. The new Louisiana dates are close to the radiometric dating of volcanic ash in the lower Catahoula Group in east Texas at 30.65 ± 0.06 Ma (Heintz et al. 2015), while the Mississippi date is close to the dating of the lower Gueydan Formation (lower Catahoula Group) in south Texas (Yancey et al. 2018).

Sanidine $^{40}\text{Ar}/^{39}\text{Ar}$ dating for the Louisiana sample is based on both single-crystal dates and multigrain-aliquot dates. Single grains released small amounts of argon but yielded a weighted mean age of 30.08 ± 0.07 Ma. Multigrain aliquots composed of 10–20 grains released higher levels of argon that provided higher precision and yielded a weighted mean age of 30.149 ± 0.011 Ma. The very small sanidine crystal size (0.006–0.01 mm) in this volcanic ash is close to the size limit for single-crystal dating of sanidine of this age. Given its fine-grained characteristics, the north Louisiana sample is most likely part of a far-distal deposit of the source ash cloud.

Geochemical Characterization of Catahoula Formation Glass Shards

Major-element composition of individual glass shards, determined by electron microprobe analysis for all three samples, and LA-ICPMS-determined whole-rock compositions for the Bald Hill and Sam Rayburn samples are presented in table 2. Trace-element and rare earth element (REE) data of both samples, plus the Three Rivers sample of south Texas, are shown in table 3 and plotted in figures 5 and 6. The three samples have very similar compositions, with the Sam Rayburn sample having lower concentrations for all elements and minor variation in the negative Eu anomaly. The small differences seen in the Sam Rayburn data are probably artifacts related to not being analyzed in the same lab as the other two samples. Overall, the three ash samples are very close in composition. The Bald Hill, Sam Rayburn, and Three Rivers samples have heavy-REE values very close to the REE values of normal mid-ocean ridge basalt (N-MORB) composition magma (Wilson 1989; Rollinson 1993) and have strong negative Eu and Sr anomalies, along with a positive Th anomaly.

There is some variation within the Sam Rayburn glass shard compositional data, which contains glass shards that cluster into two groups (fig. 7). It is noteworthy that two of the Sam Rayburn grains are pumiceous—Sam Rayburn 12 and Sam Rayburn 13—and that the pumice particles separate into the two compositional groups. This indicates that the difference in glass composition is real and not an artifact of analytical error. This difference in glass composition within the same ash sample suggests the likelihood of magma mixing during the eruption, which is not an uncommon event. Magma mixing before or during an eruption is well documented (among many examples, see Anderson 1976; Bacon and Druitt 1988; Mandeville et al. 1996). Examples of magma mixing have been determined within Sierra Madre ignimbrites by isotopic composition (Verma 1984; Verma and Verma 1986; Cameron et al. 1987) as well as in Miocene Central American Volcanic Arc ignimbrite ash (Jordan et al. 2006). This variation in composition could indicate a new injection of magma at the time of eruption, a compositionally mixed magma chamber resulting from fractional crystallization or assimilation of country rock, or due to the presence of a chemically stratified magma. Given the geochemically evolved nature of the ash, it is more likely the latter process rather than the former ones.

The values of major and trace elements are well within acceptable limits of precision for chemical analyses of these elements, except for a larger-than-expected variation in Na_2O , Sr, and Ba in the Three Rivers sample. Because of this, the mean of the Na_2O values was not used, and selected single-spot analyses from the cleanest run were used to represent the Na_2O values for all samples. Sr and Ba values from Three Rivers were still used, but it must be noted that they may contain microlites of a composition differ-

Table 2. Major-Element Data (wt%) for Whole Rock and Glass Shards of the Bald Hill, Sam Rayburn, and Three Rivers Samples of the Catahoula Formation

	Bald Hill (glass)	Sam Rayburn (glass)	Three Rivers (glass)	Bald Hill (whole rock)	Sam Rayburn (whole rock)
SiO_2	73.90	73.49		66.38	70.12
TiO_2	.10	.10	.08	.20	.114
Al_2O_3	11.13	11.78	11.13	19.52	13.7
FeO	1.69	.54		1.62	.4
MnO	.03	.05		.02	.052
MgO	.00	.06		1.38	.34
CaO	.24	.36	.44	.88	.46
Na_2O	2.56	1.47	2.90	.57	1.25
K_2O	4.08	4.31		1.59	3.53
P_2O_5	.01	.02		.01	.02
Total	93.74	92.18		92.17	89.986
Loss on ignition				5.7	8.46

Table 3. ICPMS Trace-Element and Rare Earth Element Data (ppm) for Glass Shards and Whole-Rock Fractions of the Bald Hill, Sam Rayburn, and Three Rivers Samples

	Bald Hill		Three Rivers	Sam Rayburn								
	Glass	Whole rock	Glass	6 (glass)	8 (glass)	9 (glass)	10 (glass)	11 (glass)	12 (glass)	13 (glass)	Glass avg.	Whole rock
La	32.65	108.39	32.03	21.713	6.714	8.707	7.263	18.709	19.961	8.262	21.67	33
Ce	73.47	227.33	70.27	43.383	18.829	19.657	17.089	40.502	35.167	16.088	43.13	64.8
Pr	7.58	28.5	7.71	4.537	2.175	2.382	2.188	4.179	3.897	1.892	4.64	7.65
Nd	30.93	106.58	31.66	18.719	9.165	10.534	9.268	16.271	16.150	7.646	18.79	27.7
Sm	6.45	23.48	7.26	3.631	2.884	3.210	2.727	4.179	3.514	2.612	4.07	6.06
Eu	.88	.97	.68	.598	.108	.212	.124	.510	.423	.185	.57	.67
Gd	5.68	22.67	6.02	3.164	3.436	3.483	3.358	2.692	3.116	2.820	3.26	5.97
Tb	.9	3.67	1.01	.602	.654	.783	.714	.494	.552	.542	.6	1.06
Dy	5.47	20.66	6.38	3.407	4.838	4.898	3.151	3.048	3.302	3.928	3.45	6.73
Ho	1.15	4.06	1.28	.577	1.039	.983	.897	.690	.676	.882	.68	1.4
Er	3.29	10.5	3.68	2.111	2.835	2.778	2.620	2.145	1.998	2.658	2.14	4.22
Yb	3.46	9.83	3.83	1.876	3.549	3.464	3.626	1.943	2.288	3.309	2.05	4.47
Lu		1.37		.317	.566	.500	.465	.279	.327	.446	.33	.73
Cs	4.91		5.3	1.850	6.284	6.089	5.786	2.242	2.051	4.263	3.82	5.2
Rb	152.5	141	164.09	87.666	188.649	194.533	177.225	98.068	136.838	150.688	142.25	212
Ba	849.99	68.27	594.11	810.466	11.521	97.703	17.823	447.492	476.874	286.058	365.42	897
Th	11.41	71.77	12.56	10.841	11.401	10.419	10.529	10.706	9.560	8.335	10.49	16.1
U	4.03	16.99	4.21	2.346	7.455	5.857	6.554	2.672	2.058	3.933	4.19	6.27
Nb	13.46	53.54	13.35	6.530	13.709	10.709	11.739	6.792	5.412	8.920	8.83	12.2
Ta	.91	8.96	.96	.384	1.318	.971	.939	.407	.524	1.167	.77	1.06
Pb	17.5	80.98	18.59	12.662	14.779	12.537	12.972	13.906	11.749	19.690	13.98	23
Sr	29.82	31	23.72	14.170	.933	15.854	3.816	8.643	36.911	38.275	17.16	54
Hf	4.1	27.53	3.57	2.602	2.766	3.042	2.759	2.807	2.778	2.310	2.82	3.6
Zr	122.92	864	107.06	80.415	48.521	57.324	48.656	72.739	67.093	48.576	63.27	82
Y	34.16	110.12	37.7	18.940	30.217	32.146	30.506	17.893	20.703	27.461	25.06	39.1

Note. Data for the Sam Rayburn whole-rock sample are from Heintz et al. (2015).

ent from that of the glass. However, since most of the geochemical correlation is based on an REE comparison, these outliers do not influence our interpretations. Finally, concentrations for Pb were based on two different isotopes (^{206}Pb and ^{208}Pb). The concentration value of the isotope with the lowest error was chosen as the representative value.

These major-element, trace-element, and REE analyses of glass shards are comparable to major-element, trace-element, and REE analyses of whole-rock volcanic ash samples for 14 late Eocene and early Oligocene volcanic ash beds in Texas reported by Heintz et al. (2015). Individual glass shard analyses for the Sam Rayburn sample were done on a split of the same sample used by Heintz et al. (2015) for the whole-rock analysis of the volcanic ash.

Miocene Volcanic Ash

Volcanic ash of Miocene age has also been reported in Louisiana from subsurface sites in southern Louisiana and the adjacent continental shelf. Both Hanan et al. (1998) and Jones (2008) argued that the source of these volcanic ash beds was Snake River Plain eruptions of the Yellowstone hotspot.

If a young age is accepted for the upper Catahoula Formation, then Yellowstone hotspot volcanic ash beds might also be present in the upper part of the formation.

Dating by biostratigraphy on ash beds in two boreholes reported by Hanan et al. (1998) ranges between 10.8 and 14.8 Ma, with the ash beds in four boreholes dated to approximately 7.75 Ma on the Styzen (1996) biozone correlation chart. Hintze (1967) reported lithified volcanic ash beds that show as prominent markers on resistivity logs, known as the "Operc" lime or "Regional Operc" markers, in the Rousseau Field of Lafourche Parish, Louisiana. According to Styzen (1996), the "Operc" interval lies within the late part of the early Miocene (~17 Ma). Paine et al. (1968) reported that indurated middle Miocene Krumbhaar sands of the Cristellaria "I" shale within the Hollywood Field, Terrebonne Parish, Louisiana, contain abundant volcanic ash and bentonite. Gutierrez (2016) reported the occurrence of 11 correlative beds of volcanic ash in middle and late Miocene sandstones and mudstones of the offshore Green Canyon and Walker Ridge outer Continental Shelf areas. The average thickness of subsurface ash beds reported by Hanan

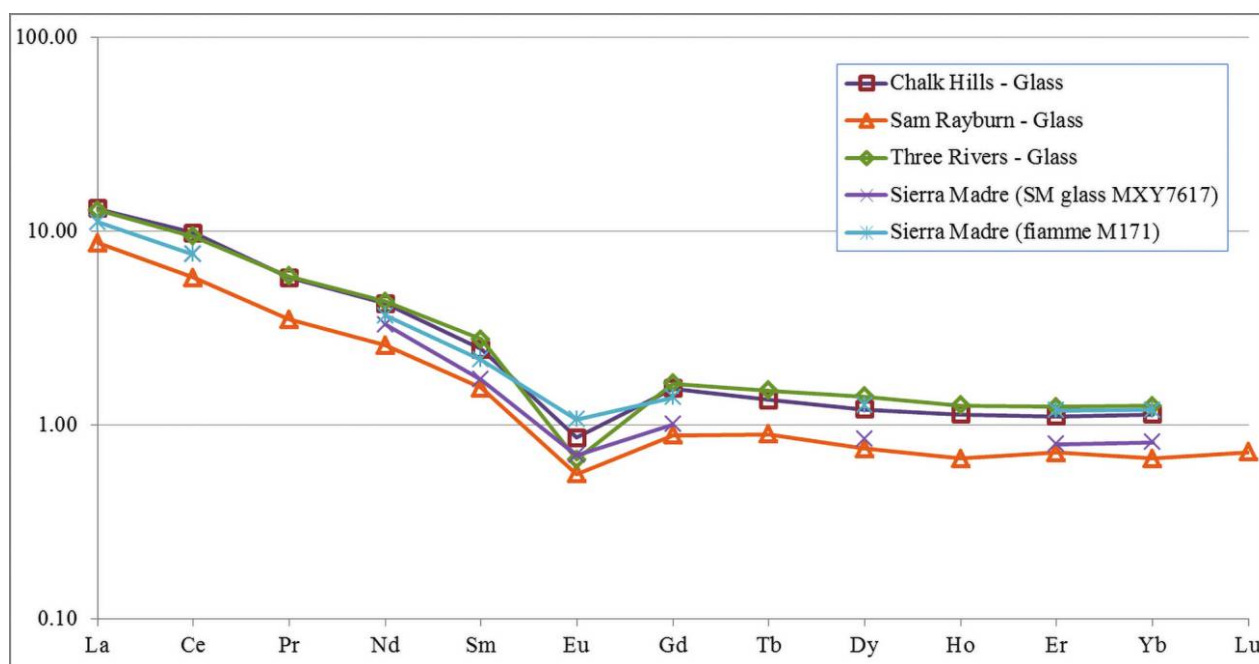


Figure 5. Trace-element diagram of Bald Hill (Chalk Hills), Sam Rayburn, and Three Rivers samples of the Catahoula Formation of Texas and Louisiana, normalized to normal mid-ocean ridge basalt.

et al. (1998) and Gutierrez (2016) is over 10 m, and both report the presence of much thicker beds of volcanic ash.

The authors of these reports regarded the ash to be too thick and too silicic to have come from Cen-

tral American volcanoes and too young to have come from west Texas and west Mexican Paleogene sources. Hanan et al. (1998) and Jones (2008) presented trace-element and isotopic signatures that are consistent with Yellowstone hotspot eruptions as sources

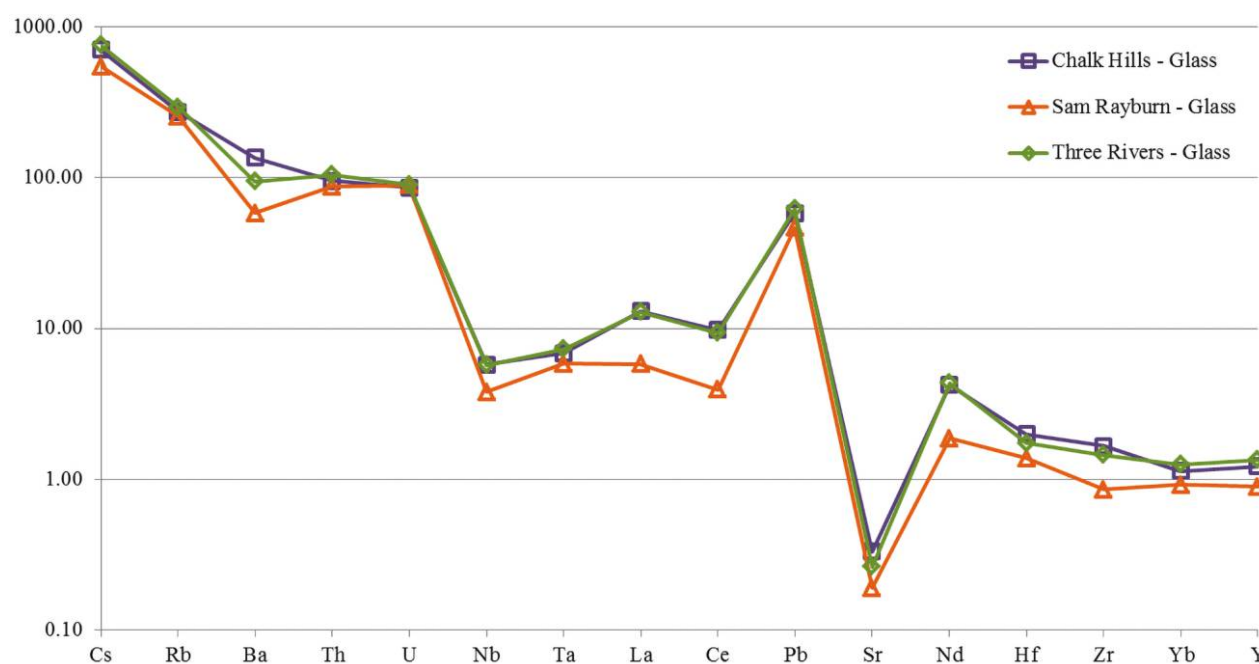


Figure 6. Rare earth element plot of Bald Hill (Chalk Hills), Sam Rayburn, and Three Rivers samples of the Catahoula Formation of Texas and Louisiana, normalized to normal mid-ocean ridge basalt.

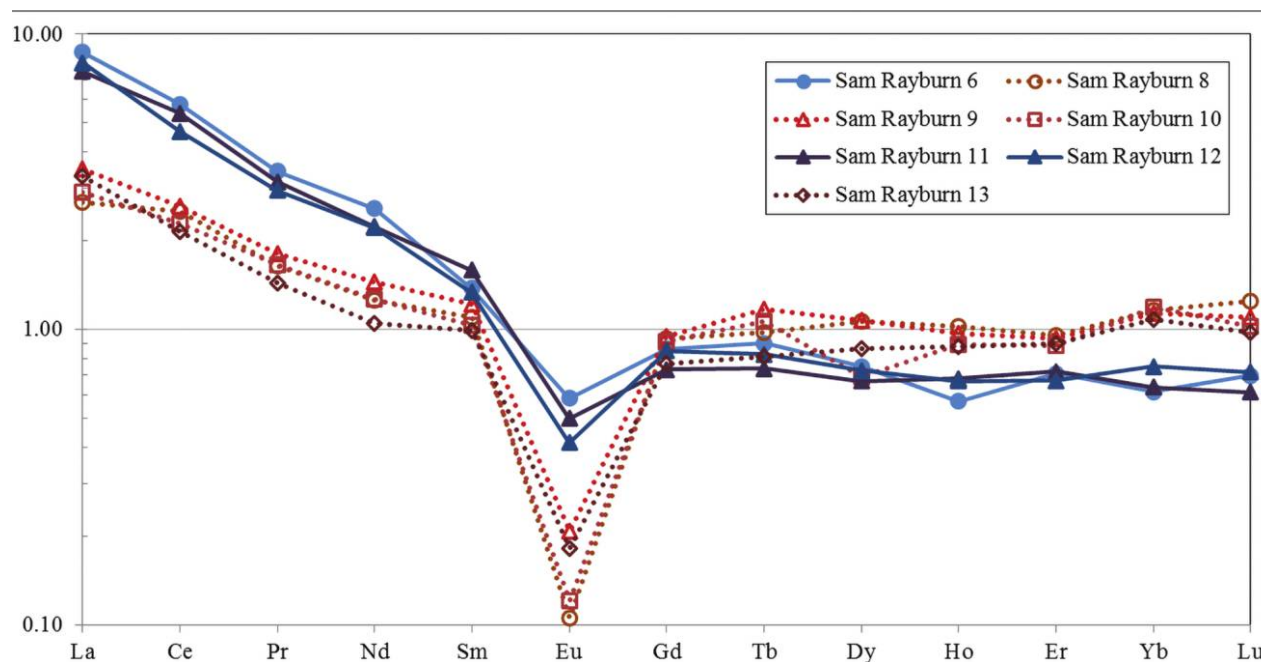


Figure 7. Rare earth element composition for individual glass shards in the Sam Rayburn sample, illustrating two distinct compositional groups of glass shards.

for these ash beds. Element composition of Louisiana Miocene glass samples are compared with our dated early Oligocene ignimbrite flare-up samples in figure 9, showing distinct differences in several trace and REE elements, with a strong difference in Ba and REE content. Yellowstone eruptive centers associated with supereruptions occurred in continental crust with Precambrian basement rock (Nash and Perkins 2012), and all silicic magmas from those centers are expected to have a composition different from the N-MORB-related composition typical of western Mexico Oligocene magma.

Discussion

The great similarity of Bald Hill and Three Rivers trace-element data and the close fit of Sam Rayburn trace elements to the other two samples indicates a similar, if not identical, source for all three dated volcanic ash deposits (fig. 5). The three samples all date to the early Oligocene mid-Rupelian stage (table 1). The Bald Hill sample now has high-precision dates of sanidine $^{40}\text{Ar}/^{39}\text{Ar}$ at 30.13 ± 0.01 Ma and zircon U/Pb at 29.98 ± 0.05 Ma. The geochemical similarity of the Bald Hill ash, as well as the similarity of its radiometric age, to the 30.65 ± 0.06 Ma Sam Rayburn volcanic ash in east Texas is important, because the Catahoula Formation in east Texas is very similar to the volcanic ash-bearing Catahoula For-

mation in northern Louisiana. In both places, volcanic ash deposits occur low in the section but not at the base of the formation (Chawner 1936; Fisk 1938; Ledger 1988). A slightly older early Oligocene age (32.72 ± 0.03 Ma) is available for the base of the Gueydan Formation (Catahoula) in south Texas, where volcanic ash also is most abundant in the lower part of the formation (Yancey et al. 2018). This suggests that the volcanic ash deposits of the Catahoula Formation across Texas and Louisiana are from related eruptive events that occurred during the early Oligocene. The lower Catahoula Formation volcanic ash reported here matches the composition of late Eocene and Oligocene volcanic ash deposits in the coastal plains of Texas that are also considered to be from volcanism of the Sierra Madre of western Mexico (McBride et al. 1968; Heintz et al. 2015).

The presence of thick layers of volcanic ash is a distinctive character of the lower Catahoula Formation across both states. Volcanic ash also occurs in Mississippi, as bentonite in marine deposits of the early Oligocene Glendon Formation (Calhoun et al. 2014; Dockery and Thompson 2016; LaFleur et al. 2018).

All three lower Catahoula volcanic ash samples have Nb and Ta anomalies (fig. 6), indicating a subduction-zone source (Wilson 1989; Rogers and Hawkesworth 2000). Heavy REEs are the least mobile of elements, supporting the idea of an N-MORB-

type melt source producing the silicic melt for these volcanic ashes. The elevated light REEs indicate some fractionation during silicic-melt formation. There also are strong negative Eu and Sr anomalies and a positive Th anomaly that are most likely the result of plagioclase/sanidine fractionation (Philpotts 1968; Rollinson 1993; Dunn 1994). Given the geochemistry and age of the volcanic ash, eruptive centers of the ignimbrite flare-up event in the Sierra Madre of Mexico have a high probability of being the source area for the volcanic ash. The east Texas–north Louisiana Oligocene volcanic ash deposits fit within the peak interval of silicic volcanic activity in the northern portion of the Sierra Madre Occidental (Ferrari et al. 2018).

The compositional similarity of these volcanic ash deposits provides evidence that invalidates the concept of a local volcanic eruptive center source for Catahoula Group sediments in south-central Texas proposed by Morony et al. (2011). Glass composition data for the volcanic deposits discussed by Morony et al. (2011) are inconsistent with volcanic activity present at an intraplate location (Nash and Perkins

2012), and the radiometric dating and glass composition data are fully consistent with a Sierra Madre Occidental source (Heintz et al. 2015; this study).

Plots of glass shard composition and whole-rock composition for the same samples on a total-alkali-versus-silica (TAS) diagram show that whole-rock values plot within the dacite field, whereas glass shard values plot within the rhyolite field (fig. 8). Glass shard analysis was done with spot analysis in the interior of glass shards, avoiding compositional change associated with margin alteration and adhering secondary clays. Composition for the Three Rivers sample was not plotted because whole-rock analysis was not determined for the sample. The TAS diagram plots indicate that the magma composition (glass shards) was rhyolite and that diagenesis and inclusion of clay with the glass shards have altered whole-rock samples to a lower silica composition, a composition typical of bentonite deposits with or without glass shards present (Michaelides 2011). The same alteration character is shown for Eocene-Oligocene silicic volcanic ash deposits in Texas (Heintz et al. 2015). This offset is characteristic of whole-rock analyses of volcanic

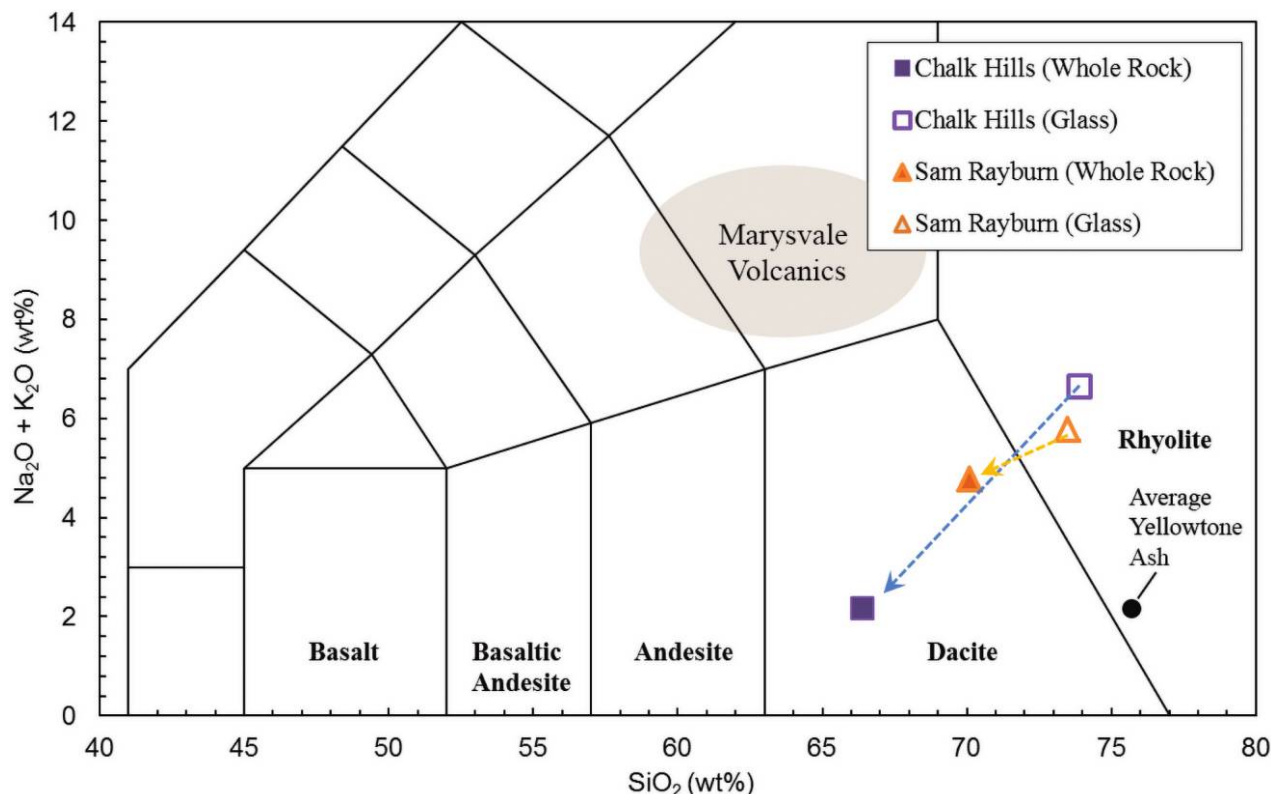


Figure 8. Compositional difference of glass shard and whole-rock composition of the Bald Hill (Chalk Hills) and Sam Rayburn samples when plotted on a total-alkali-versus-silica diagram of chemical classification of igneous rocks. Arrows show the direction of compositional change during alteration.

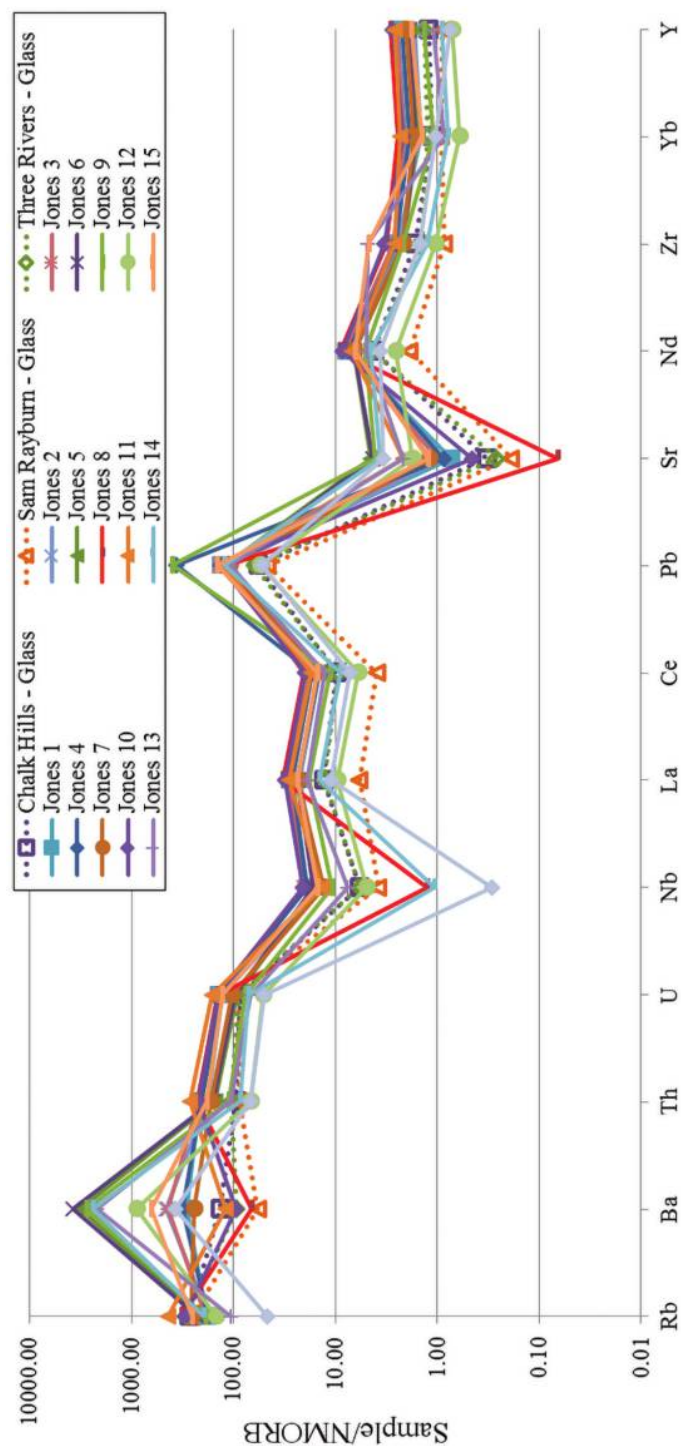


Figure 9. Trace-element and rare earth element diagram of Catahoula Formation Bald Hill (Chalk Hills), Sam Rayburn, and Three Rivers samples compared to data of Jones (2008) on Miocene subsurface and offshore volcanic ash in Louisiana, normalized to normal mid-ocean ridge basalt (NMORB).

ash beds and bentonites in the Paleogene of the Gulf Coast.

The upper part of the Catahoula Formation in northern Louisiana and Mississippi is poorly known, lacking detailed stratigraphic description and good age control. The occurrence of an early late Miocene palynology age (8.68 Ma; Wrenn et al. 2004) for strata in the Big Creek area of Sicily Island, overlying silica-cemented sandstones in the type section along the Ouachita River, aggravates the problem. It points to a 20-My difference in age for strata within a short interval of section, making it probable that there is an unrecognized fault or an unconformity in the type section. There is no evidence for a structural uplift in the area to account for lack of deposition or erosion, so the presence of a fault is a more realistic interpretation of this feature. Chawner (1936) records a high-angle fault in the Chalk Hills area, and Levent (1959) mentions that down-to-the-Gulf normal faults are common along the Catahoula trend. This suggests the presence of a fault between the resistant sandstones at Catahoula Shoals and the late Miocene lignite on Sicily Island.

Several publications report large amounts of volcanic ash of Miocene age in the subsurface of Louisiana, but none has been found in outcrop exposures. Any occurrence of volcanic ash found in the currently defined upper Catahoula Formation or Miocene formations will have to be analyzed to determine origin in the context of possible sources from the ignimbrite flare-up or the Yellowstone hotspot. Older eruptions along the Yellowstone hotspot pathway can be distinguished from ignimbrite flare-up volcanic ash by differences in trace-element and REE composition (fig. 9). Yellowstone hotspot magmas move through continental crust and can be expected to have much higher concentrations of REE elements and lack a Nb-Ta anomaly, in comparison to subduction-generated magmas of the ignimbrite flare-up. Along the Yellowstone eruptive track there is a compositional difference between western eruptive centers in Nevada, erupted through crust with embedded oceanic terranes (Brueseke et al. 2008; Nash and Perkins 2012), and eastern eruptive sources in Idaho, erupted through Precambrian continental crust (Doe et al. 1982; Hanan et al. 1998; Perkins and Nash 2002; Nash et al. 2006). This is an expected result of North America's move westward over the top of the Yellowstone mantle plume at 15.2 Ma, when the movement carried the $^{87}\text{Sr}/^{86}\text{Sr}$ 0.706 line over the hotspot (Nash and Perkins 2012). This occurred before deposition of Miocene volcanic ash known in Louisiana. An age date or a geochemical characterization of a Miocene volcanic ash bed should be enough to determine the source.

Conclusions

The Chalk Hills Member of the Catahoula Formation in Louisiana has been dated as early Oligocene (mid-Rupelian stage), and new ID-TIMS zircon dates are reported for Louisiana and Mississippi. Geochemical composition and age show a correlation with the lower part of the Catahoula Formation in east Texas. Volcanic ash was derived from subduction-related magmas with an N-MORB-type melt composition. The geographic span of these volcanic ashes indicates wide distribution of volcanic ash from very large silicic volcanic eruptions in western North America during the early Oligocene. The source of this volcanic ash is constrained to be located in the Sierra Madre Occidental of western Mexico. Supereruptions from this area are believed to have spread fallout volcanic ash layers over the western and northern parts of the Gulf of Mexico. The easternmost occurrence currently attributed to this source occurs in central Mississippi (Dockery et al. 2017; LaFleur et al. 2018).

Volcanic ash of Miocene age is present in several subsurface fields in Louisiana but not known to occur in the Catahoula Formation. Miocene volcanic ash from the Yellowstone hotspot is compositionally distinct from early Oligocene volcanic ash. There appears to be no overlap in age of Louisiana occurrences of fallout ash from these two sources, but dating and geochemical analysis are needed to determine the source of any volcanic ashes reported.

The presence of late Miocene deposits within strata previously placed in the upper part of the type section of the Catahoula Formation points to the presence of a fault or an unconformity in the section within northern Louisiana. In the absence of evidence for local structural uplift and presence within a known trend of down-to-the-Gulf faulting, a fault offset is indicated. The type section should be reexamined.

ACKNOWLEDGMENTS

Thanks are given to the late Glenn Embree of the Brigham Young University-Idaho Department of Geology for his help analyzing the Bald Hill sample; T. Vogel at Michigan State University and T. Plank at Columbia University for the ICPMS data; M. Heizler of the New Mexico Bureau of Economic Geology for $^{40}\text{Ar}/^{39}\text{Ar}$ dating of the Texas and Bald Hill samples; H. Zwingmann at CSIRO for providing the whole-rock Bald Hill age data; R. McCulloh, of the Louisiana Geological Survey, and the late John H. Wrenn for collecting the samples; D. Dockery, Mississippi State Geologist, for providing reports on the Catahoula Formation in central Mississippi; S. Warny Bart, of the Center for Excellence in Palynology, for providing the

sample for our research; the Louisiana State University Department of Geology and Geophysics; the Louisiana Geological Survey; the Brigham Young

University–Idaho Department of Geology; and the Brigham Young University–Hawaii Department of Natural Sciences.

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