

Distribution of rare earth and other critical elements in lignites from the Eocene Jackson Group, Texas

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ABSTRACT

Coal is increasingly evaluated as a source of rare earth elements (REEs) in the United States to address the overreliance on imported REEs. The objective of this study was to assess the distribution of REEs in lignites from selected mining areas in the Texas Gulf Coastal Plain region. Thirty-one archived lignite and rock samples previously collected by the U.S. Geological Survey were analyzed for their rare earth element and critical mineral content. These include samples from one core (5400 and 5500 lignite horizons) and two opencast lignite mines (Gibbons Creek 3500 and 4500 horizons, and San Miguel horizons A to D) in the Eocene Jackson Group of the Texas Gulf of Mexico Coastal Plain. Some lithologies in the Gibbons Creek 3500 and 4500 lignite-bearing sections have high total rare earth, yttrium (Y), and scandium (Sc) (REYSc) values, up to 7800 ppm (ash basis) REYSc. The lignite lithologies show an enrichment in rare earths, [samarium (Sm) through gadolinium (Gd)]. The basal Gibbons Creek 3500 lignite bench shows a heavy rare earth element enrichment pattern resembling that often seen in peats through high volatile A bituminous coals. The 5500 lignite sequence, overlying the latter lignite sections, shows a light rare earth enrichment. The San Miguel lignite benches have heavy rare earth enrichments with a negative europium (Eu) anomaly.

1. Introduction

1.1. Basic importance of the study of rare earth elements and critical elements in coal and coal byproducts

Rare earth elements (REEs) are essential in the manufacture of electronics, magnets, catalysts, optics, ceramics, and numerous other products necessary for the functioning of modern society (Greene, 2012; Hatch, 2012; Dobransky, 2013; Basu, 2017; Watson, 2018; Home, 2020; U.S. Geological Survey, 2022). Based on a combination of their importance in utilization and their relative abundance or scarcity, some REEs are more “critical,” “uncritical,” or “excessive” (Seredin and Dai, 2012). Coal combustion products, including fly ash, are among the resources considered to be potential sources of lanthanides, Sc, and Y (Clarke and Sloss, 1992; Ratafia-Brown, 1994; U.S. Department of Energy, 2017). The potential economic value of REEs extracted from coal and coal

byproducts has been discussed by the U.S. Department of Energy (2017), Das et al. (2018), and Bagdonas et al. (2022).

The fundamental importance of critical elements in current and future industrial applications has been emphasized in the implementation of several research initiatives, including the ongoing U.S. Department of Energy Carbon Ore, Rare Earth and Critical Minerals (CORE-CM) initiative which seeks to identify the most promising sources of critical minerals in U.S. coal basins (Mullen, 2022). Research on the extraction of critical minerals, as defined by the U.S. Geological Survey (2022), including REEs, from coal and coal combustion products has been conducted over the past decade (Blissett et al., 2014; Kose-Mutlu et al., 2022; Lo et al., 2014; Mutlu et al., 2018; Park et al., 2017; Park et al., 2020; King et al., 2018; Lin et al., 2018; Taggart et al., 2016, 2018; Honaker et al., 2019; Smith et al., 2019; Hower et al., 2020b, 2022a, 2022b; Middleton et al., 2020; Zhang et al., 2020; Fu et al., 2022; among others).

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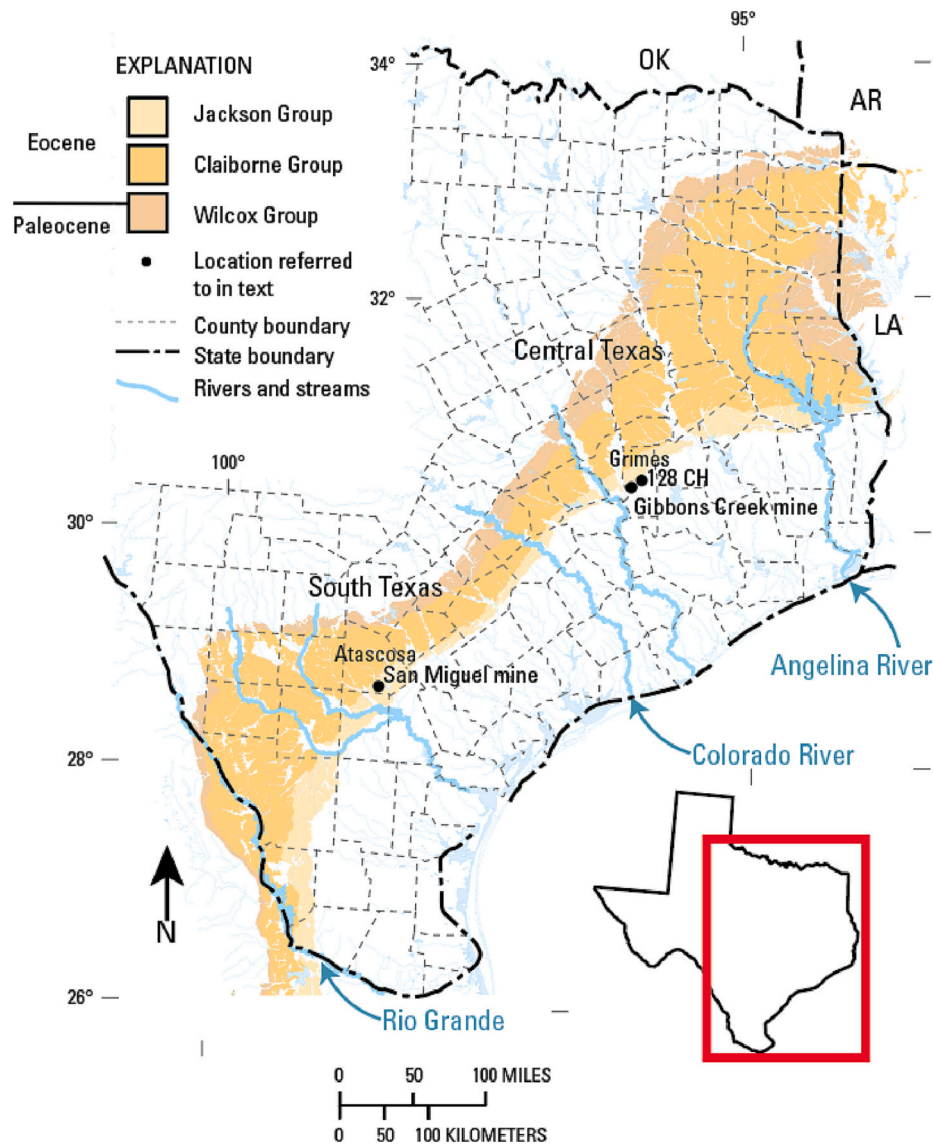


Fig. 1. Outcrop distribution of Paleocene to Eocene lignite-bearing rocks in Texas. Counties referred to in Table 1 are labelled. Central Texas is the region between the Angelina and Colorado Rivers. South Texas is the region between the Rio Grande and the Colorado River. Index map shows area of Texas considered in figure. Modified from Warwick et al. (1997) and Hook et al. (2011).

1.2. Previous geologic, geochemical, and petrographic investigations

Lignite at the San Miguel and Gibbons Creek opencast mines in Texas occur in the Eocene Jackson Group (Fig. 1). Outcrops of the Jackson Group follow the trend of the modern-day Gulf Coast of Texas. Except in areas influenced by salt structures and faulting, rocks of the Jackson Group dip gently toward the Gulf of Mexico at four degrees or less (Hook et al., 2011). In central Texas (Fig. 1), the Jackson Group consists of four formations: in ascending order, the Caddell Formation, Wellborn Sandstone, Manning Formation, and Whitsett Formation (Hook et al., 2011) (Fig. 2). In several studies, the Wellborn Sandstone and Whitsett Formation are included in the Manning Formation (Swaldi and Cezeaux, 1986; Warwick et al., 1997). In most of south Texas (Fig. 1), the Jackson Group is undivided (Barnes, 1992). Lignite production from the lower part of the Jackson Group began at the San Miguel mine in 1990, and the mine is still in operation in 2023. The Gibbons Creek mine produced lignite from the Manning Formation from 1982 through 1996 (Hook et al., 2011).

Depositional systems of the Jackson Group were described on a regional scale (Fisher et al., 1970; Kaiser et al., 1978, 1980; Ayers Jr. and Kaiser, 1986; Hook et al., 2011). In central Texas, the Jackson Group contains a coarsening-upward, deltaic to fluvial-deltaic sequence beginning with the mudstone-dominated Caddell Formation, which was deposited in prodeltaic environments (Hook et al., 2011) (Fig. 2). The overlying Wellborn Sandstone, represented by fine- to very fine-grained, slightly glauconitic quartz sands, was deposited in a delta-front environment (Hook et al., 2011). The Manning and Whitsett Formations were deposited in delta plain and fluvial environments and contain fine- to medium-grained quartzose and tuffaceous sands. Clay beds are sandy and locally bentonitic, and silicified wood is common in both formations (Hook et al., 2011). In central Texas, Jackson Group lignite beds have an average thickness of approximately 1.5 m (Kaiser et al., 1980; Hook et al., 2011).

In south Texas, the lower part of the Jackson Group coarsens upward to fine-grained, well-sorted sands that accumulated as strand plain or barrier-bar deposits, and back-barrier muds and clays dominate the

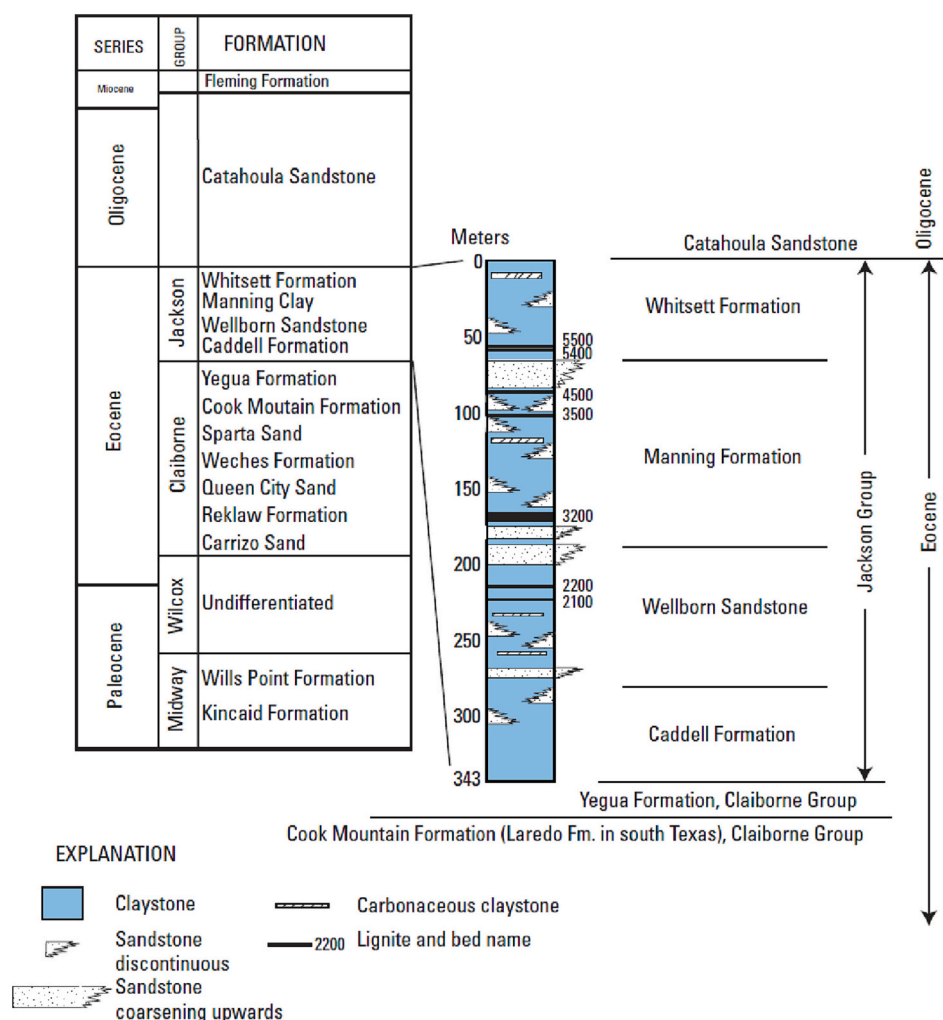


Fig. 2. Generalized regional stratigraphy of the lignite-bearing units of the Texas Gulf of Mexico Coastal Plain showing the detailed stratigraphic section of the lignite-bearing Jackson Group (modified from Warwick and Crowley, 1995; Hook et al., 2011). Formation names are for lithologies present in central Texas (from Hosman, 1996; Hook et al., 2011). Fm. = Formation.

upper part of the group (Fisher et al., 1970; Hook et al., 2011). Fossils of nearshore marine invertebrates are common (Plummer, 1932). The peats that later formed into lignites were deposited in lagoonal mires landward (west) of a sandstone-dominated barrier island complex (Ayers Jr., 1986, 1989) that shows an upward transition from brackish to freshwater conditions (Gennett et al., 1986; Mukhopadhyay, 1989; Hook et al., 2011). In south Texas, average Jackson Group lignite bed thickness is approximately 2.1 m (Kaiser et al., 1980; Hook et al., 2011).

Geochemical and petrographic studies of the Jackson Group lignite include those by Tewalt (1986), Oman and Meissner Jr. (1987), Mukhopadhyay (1989), Warwick et al. (1996, 1997), Palmer et al. (2015), Zygarlicke et al. (2019), and others. In central Texas, lignite ash yield and sulfur content average 36% and 2.2%, respectively, and average gross calorific value is 8047 Btu/lb. (18.72 MJ/kg; dry-basis) (Tewalt, 1986; Hook et al., 2011). Warwick et al. (1997) reported that lignite samples from the Gibbons Creek mine were dominated by the maceral euglenite (Sýkorová et al., 2005) which, along with the occurrence of siliceous sponge spicules in the lower benches of the 3500 bed (Fig. 2), suggests the original peat in this part of the bed was deposited in standing, fresh water. Lignite partings at the Gibbons Creek mine contain volcanic ash material (Ruppert et al., 1994; Warwick et al., 1997). In south Texas, lignite ash yield and sulfur content average 40 and 2.5%, respectively, and average calorific value is 7234 Btu/lb. (16.83 MJ/kg; dry basis) (Tewalt, 1986; Hook et al., 2011). Similar to

the Gibbons Creek lignite, petrographic study of lignite samples from the south Texas San Miguel mine by Warwick et al. (1996) found a dominance of euglenite and that the partings contain reworked volcanic ash material (Ayers Jr., 1986, 1989; Senkayi et al., 1987; Warwick et al., 1996). The deposition of volcanic ash may contribute trace elements of interest, as reviewed by Dai et al. (2016b).

Several studies (Senkayi et al., 1987; Ruppert et al., 1994; Warwick et al., 1996, 1997; Zygarlicke et al., 2019) have shown that some lignite beds within the Jackson Group contain elevated levels of some trace elements. Zygarlicke et al. (2019) collected lignite, coal, and rock parting samples from several mines in Texas and analyzed them for REE concentrations. They found that lignite and rock samples from the San Miguel mine ranged from 78 to 216 ppm (dry basis, total REYSc).

1.3. This study

In this study, we investigate the distributions of critical minerals including REEs in 31 lignite and rock samples from one core and two Jackson Group mines in Texas (Table 1). It is emphasized that belonging to the same geologic group does not necessarily mean that the lignite-bearing intervals are correlative. The distance between the two mines is >300 km and the geochemical characteristics of the intervals sampled can differ markedly.

Table 1

Laboratory number; field number; sample locations by north latitude, west longitude, and county; and thickness in feet and meters. Data sources: ⁺USGS Energy Resources Program (2014); ^{*}this study.

Lab Number ^x	Field Number ⁺	Lat. (N) ⁺	Long. (W) ⁺	County ⁺	Thickness ⁺	Thickness ⁺
					(feet)	(m)
202202141-004	M128 CH1	30.6044	-96.0144	Grimes	2	0.61
202202141-005	M128 CH2	30.6044	-96.0144	Grimes	2	0.61
202202141-006	M128 CH3	30.6044	-96.0144	Grimes	0.95	0.29
202202141-007	M128 CH4	30.6044	-96.0144	Grimes	1.1	0.34
202202141-001	GC 4500 Roof	30.5675	-96.0669	Grimes	Grab sample	
202201316-018	GC-4500.1	30.5675	-96.0669	Grimes	0.46	0.14
202201316-021	GC-4500 - P1 Channel	30.5675	-96.0669	Grimes	0.67	0.2
202201316-019	GC-4500.2	30.5675	-96.0669	Grimes	0.95	0.29
202201316-020	GC-4500.3	30.5675	-96.0669	Grimes	0.95	0.29
202201316-022	GC-3500.1	30.5675	-96.0669	Grimes	1.25	0.38
202201316-025	GC-3500-P1	30.5675	-96.0669	Grimes	0.06	0.02
202202141-002	GC 3500.2	30.5675	-96.0669	Grimes	0.07	0.02
	GC-3500-P2	30.5675	-96.0669	Grimes	0.26	0.08
202201316-023	GC-3500.3	30.5675	-96.0669	Grimes	1.18	0.36
202202141-003	GC 3500.4	30.5675	-96.0669	Grimes	0.26	0.08
202201316-024	GC-3500.5	30.5675	-96.0669	Grimes	1.11	0.34
202201316-030	SM-2-A1	28.6672	-98.4875	Atascosa	0.72	0.22
202201316-031	SM-2-A2	28.6672	-98.4875	Atascosa	0.26	0.08
202201316-032	SM-2-B1	28.6672	-98.4875	Atascosa	0.56	0.17
202201316-033	SM-2-B2	28.6672	-98.4875	Atascosa	1.05	0.32
202201316-034	SM-2-C1	28.6672	-98.4875	Atascosa	1.02	0.31
202201316-035	SM-2-C2	28.6672	-98.4875	Atascosa	1.15	0.35
202201316-036	SM-2-D1	28.6672	-98.4875	Atascosa	1.18	0.36
202201316-037	SM-2-D2	28.6672	-98.4875	Atascosa	1.08	0.33
202202141-008	SM1 P2	28.6672	-98.4944	Atascosa	0.75	0.23
202202141-009	SM1 C.1	28.6672	-98.4944	Atascosa	1.02	0.31
202202141-010	SM1 C.2	28.6672	-98.4944	Atascosa	1.15	0.35
202202141-011	SM1 P3.1	28.6672	-98.4944	Atascosa	0.3	0.09
202202141-012	SM1 P3.2	28.6672	-98.4944	Atascosa	0.3	0.09
202202141-013	SM1 P3.3	28.6672	-98.4944	Atascosa	0.3	0.09
202202141-014	SM1 D.1	28.6672	-98.4944	Atascosa	1.18	0.36

Table 2

Laboratory number; field number; and available as received moisture, dry ash, sulfur, and British thermal units (BTU) for samples analyzed in this study. (Calorific value, CV; no data, -.) Data sources: ^{*}this study; ⁺USGS Energy Resources Program (2014); SM samples, Warwick et al. (1996); GC samples, Warwick et al. (1997).

Lab Number ^x	Field Number ⁺	As Received	Dry	Sulfur	CV	CV
		Moisture	Ash			
		%	%	%	Btu/lb	MJ/kg
202202141-004	M128 CH1	-	-	-	-	-
202202141-005	M128 CH2	-	-	-	-	-
202202141-006	M128 CH3	-	-	-	-	-
202202141-007	M128 CH4	-	-	-	-	-
202202141-001	GC 4500 Roof	-	-	-	-	-
202201316-018	GC-4500.1	42.49	74.61	0.57	2126	4.95
202201316-021	GC-4500 - P1 Channel	-	-	-	-	-
202201316-019	GC-4500.2	59.42	41	1.37	5188	12.07
202201316-020	GC-4500.3	61.63	47.65	0.94	4492	10.45
202201316-022	GC-3500.1	45.23	16.7	2.96	9786	22.76
202201316-025	GC-3500-P1	-	-	-	-	-
202202141-002	GC 3500.2	42.67	16.07	2.8S	10,292	23.94
	GC-3500-P2	-	-	-	-	-
202201316-023	GC-3500.3	46.71	10.21	3.81	10,619	24.70
202202141-003	GC 3500.4	45.62	13.62	5.42	9964	23.18
202201316-024	GC-3500.5	35.55	59.33	2.05	4256	9.90
202201316-030	SM-2-A1	29.49	37.34	2.29	7888	18.35
202201316-031	SM-2-A2	30.15	34.6	2.81	8201	19.08
202201316-032	SM-2-B1	32.93	27.45	3.06	9055	21.06
202201316-033	SM-2-B2	34.17	18.96	2.71	10,336	24.04
202201316-034	SM-2-C1	36.53	17.38	2.76	10,273	23.90
202201316-035	SM-2-C2	32.3	18.77	2.75	10,270	23.89
202201316-036	SM-2-D1	37.44	15.77	2.46	10,521	24.47
202201316-037	SM-2-D2	34.11	27.3	2.6	9019	20.98
202202141-008	SM1 P2	-	-	-	-	-
202202141-009	SM1 C.1	31.22	23.82	2.81	91,173	212.07
202202141-010	SM1 C.2	30.64	25.97	2.54	9032	21.01
202202141-011	SM1 P3.1	-	-	-	-	-
202202141-012	SM1 P3.2	-	-	-	-	-
202202141-013	SM1 P3.3	-	-	-	-	-
202202141-014	SM1 D.1	29.86	25.66	2.65	8922	20.75

Table 3Major oxides (%; dry, ash basis). (dl, below detection limit; –, no data.) Data sources: ⁺USGS Energy Resources Program (2014).

Field Number ⁺	USGS	%; dry	%; ash basis ⁺									
	Lab No. ⁺	Ash ⁺	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	Fe ₂ O ₃
M128 CH1												
M128 CH2	D537512	17.8	0.64	2.1	19	45	0.03	16.4	0.62	10	1.4	2.5
M128 CH3	D537513	57	0.49	1.1	20	56	0.04	3.45	1.5	1.7	0.63	8.9
M128 CH4	D537514	24.7	0.31	1.3	15	45	0.02	7.48	0.51	3.6	0.84	21
GC 4500 Roof	–	–	–	–	–	–	–	–	–	–	–	–
GC-4500.1	W258404	71.88	0.46	0.82	19	72	0.05	1.05	1.3	0.13	0.94	4.2
GC-4500 - P1 Channel	–	–	–	–	–	–	–	–	–	–	–	–
GC-4500.2	W258405	38.96	0.13	0.58	26	56	0.03	2.6	0.37	0.15	0.82	4
GC-4500.3	W258406	44.96	0.11	0.33	21	57	0.05	1.72	0.33	0.12	0.94	11
GC-3500.1	W258396	15.72	0.36	0.45	25	56	0.03	2.41	0.63	0.67	0.96	4.1
GC-3500-P1	D549831	68.6	0.76	0.24	–	54	0.05	–	0.72	0.26	0.31	0.46
GC 3500.2	W258397	13.44	0	0.74	31.99	58.04	0	3.05	0	1.49	1.49	2.23
GC-3500-P2	D549832	74.3	0.3	1	–	56	0.03	–	54	0.22	0.93	3.8
GC-3500.3	W258398	9.32	0.41	0.72	29	49	0.02	4.58	0.42	1.8	0.94	8.4
GC 3500.4	W258399	13.36	0.34	0.58	27	49	0.02	3.54	0.34	1.3	0.62	15
GC-3500.5	W258400	57.48	0.9	0.48	22	71	0.03	0.69	1.1	0.46	0.48	2.1
SM-2-A1	W258407	36.6	4.4	0.39	13	58	dl	8.28	1.9	4.3	0.49	1.3
SM-2-A2	W258408	33	4.5	0.47	12	57	dl	10.2	1.5	5.2	0.71	2.8
SM-2-B1	W258409	26.76	4.9	0.48	14	51	0.02	11.6	1.4	5.8	0.63	5.2
SM-2-B2	W258410	21.28	6.2	0.6	13	40	0.02	18.3	1.2	8.8	0.78	4.3
SM-2-C1	W258411	17.16	7	0.63	12	34	0.04	21.1	1.1	9.6	0.64	5.4
SM-2-C2	W258412	18.52	6.2	0.63	14	40	0.04	18.5	0.94	9.1	0.91	6.2
SM-2-D1	W258413	15	8	0.74	13	34	0.02	22.2	0.99	10	0.73	6
SM-2-D2	W258414	26.2	4.6	1	16	49	0.02	12.1	1.7	5.8	0.92	4.2
SM1 P2	–	–	–	–	–	–	–	–	–	–	–	–
SM1 C.1	D532638	24.11	7	0.63	12	34	0.04	21.1	1.1	9.6	0.64	5.4
SM1 C.2	D532639	26.13	6.2	0.63	14	40	0.04	18.5	0.94	9.1	0.91	6.2
SM1 P3.1	–	–	–	–	–	–	–	–	–	–	–	–
SM1 P3.2	–	–	–	–	–	–	–	–	–	–	–	–
SM1 P3.3	–	–	–	–	–	–	–	–	–	–	–	–
SM1 D.1	D532640	25.44	8	0.74	13	34	0.02	22.2	0.99	10	0.73	6

2. Methods

2.1. Sample collection

Thirty samples used in this study were from splits of air dried, ground (0.25 to 4.7 mm particle diameter) archived samples of lignite and rock collected by the U.S. Geological Survey (USGS) in the 1990s as part of a regional study of the Gulf of Mexico Coastal Plain coal resources (Table 1). Because no archived sample material remained for sample GC 3500-P2 (Table 1), analytical data from Warwick et al. (1997) and USGS Energy Resources Program (2014) were used in this study. Mine sampling methods and sample intervals are described by Warwick et al. (1996, 1997). At the Gibbons Creek mine (Fig. 1), incremental channel samples of lignite and associated rock were collected from the 3500 and 4500 lignite beds exposed in a highwall at the active opencast pit and are described by Warwick et al. (1997). In addition, and not described in Warwick et al. (1997), a coal core (128 CH, Fig. 1, Table 1) of the 5400 and 5500 beds was also collected by the USGS. The 128 CH core was located approximately 7.5-km northeast of the Gibbons Creek mine sample location in an interval that is approximately 23-m above the 4500 lignite bed (Figs. 1 and 2). At the San Miguel mine (Fig. 1) incremental channel samples (Table 1) of lignite and associated rock were collected from lignite beds exposed in highwalls of active opencast pits and are described by Warwick et al. (1996).¹

In 2022, the USGS archive samples used in this study were run through a riffle splitter to obtain about 50 g of each sample for critical mineral and REE analyses. The sample splits were donated to The University of Texas at Austin, Bureau of Economic Geology for REE analyses.

¹ Note that the location for the SM-1 samples reported by Warwick et al. (1996) was in error and the correct location for these samples is included in Table 1 of this study.

2.2. USGS/geochemical analyses

Basic analytical data including moisture, ash, sulfur, British thermal units (BTU), oxide, and elemental data from most lignite samples used in this study (Tables 2 and 3) were previously reported by Warwick et al. (1996, 1997) and USGS Energy Resources Program (2014). Standard moisture, ash, sulfur, and BTU analyses were performed by a USGS contract laboratory (Geochemical Testing, Somerset, PA) on lignite samples in accordance with ASTM test methods (ASTM, 1993) and the results are reproduced in Table 2. The USGS oxide analyses in Table 3 were performed by the USGS Energy Inorganic Laboratory in Denver, CO, and followed the USGS analytical methods of Golightly and Simon (1989). The oxide values were determined using inductively coupled plasma atomic emission spectroscopy (Na₂O, SiO₂, Al₂O₃, CaO, MgO, K₂O, Fe₂O₃, TiO₂, P₂O₅). The elemental analytical methods for sample GC 3500-P2 (Table 1) are described in Warwick et al. (1997) and USGS Energy Resources Program (2014).

2.3. Standard Laboratories, Inc./geochemical analyses

Standard Laboratories, Inc. in Freeburg, Illinois ground and prepared the samples and performed the REE analysis on the samples shown in Tables 4 and 5. Remnant moisture analysis was determined prior to the REE analysis. The REE analyses were performed following ASTM (2021) D6357 using Method B for Inductively Coupled Plasma Mass Spectrometry (ICP-MS) determination. As part of that method, the samples are ramped to a temperature of 500 °C in a muffle and remained at that temperature for at least 2 h, the method stipulates that the samples can remain at that temperature longer to remove any visible carbonaceous material if necessary. The ash was transferred to a perfluoroalkoxy (PFA)/Teflon digestion vessel. Nitric, hydrochloric, and hydrofluoric were added and the solution was digested to near dryness. Then the vessel sides were washed down with a small amount of ultrapure water and evaporated to near dryness again. Finally, a small portion of nitric acids was added along with a quantity of ultrapure water to

Table 4

Remnant moisture, ICP-MS major and minor elements plus World brown coal average and 5× average values from Ketris and Yudovich (2009) on a dry coal (a) and ash (b) basis (the “5×” values are highlighted in blue, bold, italic font; ppm; dry coal and ash basis; dl, below detection limit; –, no data). Data sources: ⁺USGS Energy Resources Program (2014); ^{*}this study, unless noted otherwise.

(a)	%;	%; dry	μg/g; dry basis [*]											
Field Number ⁺	Moisture [*]	Ash [*]	Li	Be	Mg	Al	Ti	100*Ti/Al	V	Cr	Mn	Co	Ni	Zn
M128 CH1	9.61	11.85	10.2	2.5	508	8960	706	7.88	15	10	508	0.6	5	5
M128 CH2	10.08	16.77	19.8	1.8	427	17600	1410	8.01	30	15	427	1.3	6	12
M128 CH3	7.98	55.09	45.3	2.4	281	59600	2130	3.57	52	30	281	13.3	24	153
M128 CH4	7.74	25.82	11.1	9.7	258	19900	1210	6.08	36	135	258	6.3	79	42
GC 4500 Roof	3.34	96.79	12	1	79	50000	1960	3.92	34	99	79	3.6	50	44
GC-4500.1	21.22	72.94	31.4	2.3	61	74400	3900	5.24	84	35	61	5.4	12	40
GC-4500 - P1 Channel	14.79	87.69	41.2	1.4	79	125000	4130	3.3	99	29	79	4.8	9	59
GC-4500.2	35.49	37.02	15.2	4.6	26	52200	1920	3.68	44	18	26	21.3	27	22
GC-4500.3	32.18	46.27	21.6	2.4	27	57700	2440	4.23	42	22	27	8.3	11	14
GC-3500.1	10.15	18.75	20.5	7.4	44	25800	1000	3.88	23	8	44	7.1	18	46
GC-3500-P1	4.89	57.78	126	4.5	62	104000	1680	1.62	10	4	62	4.2	11	34
GC 3500.2	8.64	15.24	34.9	7.7	46	25500	1150	4.51	24	10	46	3.6	14	40
GC-3500-P2 ⁺	-	74.3 ⁺	43.8 ⁺	2.2 ⁺	-	-	-	-	74 ⁺	31 ⁺	89 ⁺	5.2 ⁺	8 ⁺	89 ⁺
GC-3500.3	10.54	10	7.3	4	56	15000	522	3.48	18	4	56	5.4	8	19
GC 3500.4	20.16	8.81	7.1	4.3	56	14500	550	3.79	18	8	56	8.1	10	23
GC-3500.5	8.38	58.2	26.8	3.7	98	68400	1500	2.19	21	12	98	4.6	9	50
SM-2-A1	8.4	37.14	19.2	8.8	104	28200	1140	4.04	33	11	104	2.1	4	37
SM-2-A2	7.98	33.67	23	6.2	131	22800	1430	6.27	43	17	131	1.5	5	16
SM-2-B1	9.01	25.23	14	2.5	94	20500	1040	5.07	26	10	94	2.8	4	18
SM-2-B2	9.97	17.91	11.8	3	108	14100	957	6.79	32	9	108	1.7	4	25
SM-2-C1	10.05	18	10.7	3.4	134	13900	717	5.16	28	8	134	2.4	5	19
SM-2-C2	9.35	18.47	8.4	3.9	117	15000	1030	6.87	21	8	117	1.2	4	19
SM-2-D1	10.76	15.32	5.5	2.7	110	11300	678	6	15	6	110	1.6	3	17
SM-2-D2	9.69	25.21	15.6	3.1	130	23700	1450	6.12	31	12	130	1.5	6	11
SM1 P2	4.4	84.61	105	0.8	51	110000	4710	4.28	38	18	51	1.2	9	44
SM1 C.1	9.08	23.02	8.7	3	124	16000	962	6.01	41	16	124	2.3	7	34
SM1 C.2	7.69	24.08	7.2	3.1	129	18000	1070	5.94	21	9	129	0.9	5	11
SM1 P3.1	6.22	84.82	33.8	1.1	69	96600	3370	3.49	20	13	69	0.7	6	57
SM1 P3.2	6.95	84.46	30	0.8	121	118000	2320	1.97	22	12	121	3	7	196
SM1 P3.3	5.48	82.04	42.9	1.4	63	81900	3380	4.13	33	20	63	2.1	9	27
SM1 D.1	8.71	25.42	4.1	2.4	144	14900	748	5.02	18	12	144	1.2	6	21
World brown coal ash			10	1.2	-	-	-	-	22	15	100	4.2	9	18
5x World brown coal ash			50	6	-	-	-	-	110	75	500	21	45	90

Field Number ⁺	%;	%; dry	μg/g; dry basis ⁺											
	Moisture ^x	Ash ^x	Ga	Ge	As	Zr	Nb	Sn	Sb	Ba	Hf	Ta	W	U
M128 CH1	9.61	11.85	3.2	2	2.3	33	4.4	1.6	0.19	24	0.9	0.22	1.1	0.9
M128 CH2	10.08	16.77	8.1	4	3.5	51.3	7.9	1.2	0.42	42	1.4	0.59	1.6	1.7
M128 CH3	7.98	55.09	17	3	9.1	122	13.4	2.6	0.81	164	3.5	0.99	1	4.1
M128 CH4	7.74	25.82	9.7	6	7.2	59.6	6.6	3.5	0.5	31	1.6	0.39	2.9	2.1
GC 4500 Roof	3.34	96.79	12.9	2	5.4	79.6	12.1	4.2	0.67	638	2.6	0.87	1.2	2
GC-4500.1	21.22	72.94	29.8	8	12.2	231	24.6	4.2	1.39	894	6.6	1.76	2.5	17.6
GC-4500 - P1 Channel	14.79	87.69	40.7	4	3.2	145	34.1	3.8	1.04	110	5.8	3.23	3.9	2.8
GC-4500.2	35.49	37.02	52.6	28	9.7	258	32.9	8.2	1.06	316	8.3	1.5	7.9	35.2
GC-4500.3	32.18	46.27	37	28	17.3	201	19.8	6.3	1.2	801	6.2	1.57	11.7	7.8
GC-3500.1	10.15	18.75	15	9	7.4	78.2	8.2	2.4	0.55	91	2.4	0.6	2.8	2.6
GC-3500-P1	4.89	57.78	33.6	4	3.6	73.6	4.3	1.1	0.86	210	3	1.47	1	2
GC 3500.2	8.64	15.24	17.3	5	5.5	75.7	9	2.6	0.48	46	2.6	0.8	1.7	2.9
GC-3500-P2 ⁺	-	74.3 ⁺	63.9 ⁺	4 ⁺	5.2 ⁺	-	62.4 ⁺	7.4 ⁺	2.23 ⁺	200 ⁺	4.5 ⁺	2.23 ⁺	3.7 ⁺	5.7 ⁺
GC-3500.3	10.54	10	8	13	7.2	31.8	3.5	2.4	0.47	64	1	0.23	4.7	2.1
GC 3500.4	20.16	8.81	8.6	14	7.3	30.7	3.9	2.3	0.48	33	1	0.24	4.9	2.1
GC-3500.5	8.38	58.2	19.9	2	7.1	137	16	2.8	0.79	182	5.2	2.18	1.2	11.6
SM-2-A1	8.4	37.14	10.5	25	8.1	230	20.8	3	1.64	691	4.8	0.62	5.3	4.4
SM-2-A2	7.98	33.67	13.1	4	7.5	202	9.2	2.9	1.78	507	5.6	0.61	3.6	6.3
SM-2-B1	9.01	25.23	7	2	6.1	110	5	1.2	1.16	462	3	0.41	3.5	2.9
SM-2-B2	9.97	17.91	6.1	2	7.9	229	5	1.2	1.06	288	4.9	0.35	2.6	3.3
SM-2-C1	10.05	18	6.5	2	18.7	171	14	1.1	0.8	269	4.5	1.1	1.8	4.7
SM-2-C2	9.35	18.47	7	3	6.6	154	11.3	1.1	0.47	267	3.8	0.65	1.4	2.8
SM-2-D1	10.76	15.32	3.6	2	4.8	85.2	4.1	0.7	0.45	237	2.3	0.33	2.2	1.7
SM-2-D2	9.69	25.21	7.6	2	13.3	94.9	6.6	1.3	0.56	226	2.6	0.59	1	2
SM1 P2	4.4	84.61	32.3	2	5.7	126	23.9	3.8	1.2	519	4.5	2	2.5	3.2
SM1 C.1	9.08	23.02	8.8	2	7.4	230	24.8	2.8	1.11	195	5.9	2.19	1.8	5.3
SM1 C.2	7.69	24.08	8.1	2	7.7	154	14.4	1.7	0.82	79	4.1	0.77	1.4	3.6
SM1 P3.1	6.22	84.82	31.7	1	12.9	177	27.2	3.7	1.23	338	6.7	2.03	2	3.1
SM1 P3.2	6.95	84.46	38.5	1	25.1	376	20	5.2	2.42	558	12	2.32	1.4	3.3
SM1 P3.3	5.48	82.04	26.8	1	4.2	195	26.6	5	0.68	737	5.6	2.1	1.8	4.2
SM1 D.1	8.71	25.42	4.4	2	14.2	76.7	5.5	1	0.53	227	2.1	0.35	2.1	1.6
World brown coal			5.5	2	7.6	35	3.3	0.79	0.84	150	1.2	0.26	1.2	2.9
5x World brown coal			27.5	10	38	175	16.5	3.95	4.2	750	6	1.3	6	14.5

(b)	%; dry	$\mu\text{g/g}$; ash basis*											
Field Number ⁺	Ash*	Li	Be	Mg	Al	Ti	100*Ti/Al	V	Cr	Mn	Co	Ni	Zn
M128 CH1	11.85	86	21	18903	75612	5958	7.3	127	84	4287	5	42	42
M128 CH2	16.77	118	11	13894	104949	8408	7.42	179	89	2546	8	36	72
M128 CH3	55.09	82	4	7279	108187	3866	3.45	94	54	510	24	44	278
M128 CH4	25.82	43	38	7823	77072	4686	5.73	139	523	999	24	306	163
GC 4500 Roof	96.79	12	1	2490	51658	2025	3.77	35	102	82	4	52	45
GC-4500.1	72.94	43	3	4277	102002	5347	4.98	115	48	84	7	16	55
GC-4500 - P1 Channel	87.69	47	2	5736	142548	4710	3.2	113	33	90	5	10	67
GC-4500.2	37.02	41	12	2863	141005	5186	3.55	119	49	70	58	73	59
GC-4500.3	46.27	47	5	1826	124703	5273	4.06	91	48	58	18	24	30
GC-3500.1	18.75	109	39	2768	137600	5333	3.73	123	43	235	38	96	245
GC-3500-P1	57.78	218	8	1577	179993	2908	1.59	17	7	107	7	19	59
GC 3500.2	15.24	229	51	2368	167301	7545	4.32	157	66	302	24	92	262
GC-3500-P2	74.3	59	3	-	-	-	-	100	42	120	7	12	120
GC-3500.3	10	73	40	3800	150000	5220	3.36	180	40	560	54	80	190
GC 3500.4	8.81	81	49	4030	164586	6243	3.65	204	91	636	92	114	261
GC-3500.5	58.2	46	6	2595	117526	2577	2.15	36	21	168	8	15	86
SM-2-A1	37.14	52	24	2302	75929	3069	3.89	89	30	280	6	11	100
SM-2-A2	33.67	68	18	2667	67716	4247	5.9	128	50	389	4	15	48
SM-2-B1	25.23	55	10	2925	81252	4122	4.83	103	40	373	11	16	71
SM-2-B2	17.91	66	17	3791	78727	5343	6.36	179	50	603	9	22	140
SM-2-C1	18	59	19	3700	77222	3983	4.91	156	44	744	13	28	106
SM-2-C2	18.47	45	21	3687	81213	5577	6.43	114	43	633	6	22	103
SM-2-D1	15.32	36	18	4191	73760	4426	5.66	98	39	718	10	20	111
SM-2-D2	25.21	62	12	5791	94010	5752	5.77	123	48	516	6	24	44
SM1 P2	84.61	124	1	2139	130008	5567	4.11	45	21	60	1	11	52
SM1 C.1	23.02	38	13	3245	69505	4179	5.67	178	70	539	10	30	148
SM1 C.2	24.08	30	13	3289	74751	4444	5.61	87	37	536	4	21	46
SM1 P3.1	84.82	40	1	3407	113888	3973	3.37	24	15	81	1	7	67
SM1 P3.2	84.46	36	1	5873	139711	2747	1.93	26	14	143	4	8	232
SM1 P3.3	82.04	52	2	2474	99829	4120	3.96	40	24	77	3	11	33
SM1 D.1	25.42	16	9	3084	58615	2943	4.78	71	47	566	5	24	83
World brown coal ash		49	6.7	-	-	-	-	140	82	550	26	52	110
5x World brown coal ash		245	34	-	-	-	-	700	410	2750	26	260	550

Field Number [†]	%; dry	µg/g; ash basis [‡]											
	Ash [*]	Ga	Ge	As	Zr	Nb	Sn	Sb	Ba	Hf	Ta	W	U
M128 CH1	11.85	27	17	19	278	37	13.5	1.6	203	7.6	1.9	9.3	7.6
M128 CH2	16.77	48	24	21	306	47	7.2	2.5	250	8.3	3.5	9.5	10.1
M128 CH3	55.09	31	5	17	221	24	4.7	1.5	298	6.4	1.8	1.8	7.4
M128 CH4	25.82	38	23	28	231	26	13.6	1.9	120	6.2	1.5	11.2	8.1
GC-4500 Roof	96.79	13	2	6	82	13	4.3	0.7	659	2.7	0.9	1.2	2.1
GC-4500.1	72.94	41	11	17	317	34	5.8	1.9	1226	9	2.4	3.4	24.1
GC-4500 - P1 Channel	87.69	46	5	4	165	39	4.3	1.2	125	6.6	3.7	4.4	3.2
GC-4500.2	37.02	142	76	26	697	89	22.2	2.9	854	22.4	4.1	21.3	95.7
GC-4500.3	46.27	80	61	37	434	43	13.6	2.6	1731	13.4	3.4	25.3	16.9
GC-3500.1	18.75	80	48	39	417	44	12.8	2.9	485	12.8	3.2	14.9	13.9
GC-3500-P1	57.78	58	7	6	127	7	1.9	1.5	363	5.2	2.5	1.7	3.5
GC 3500.2	15.24	114	33	36	497	59	17.1	3.1	302	17.1	5.2	11.2	19
GC-3500-P2	74.3	86	5	7	-	84	10	3	270	6	3	5	7.7
GC-3500.3	10	80	130	72	318	35	24	4.7	640	10	2.3	47	21
GC 3500.4	8.81	98	159	83	348	44	26.1	5.4	375	11.4	2.7	55.6	23.8
GC-3500.5	58.2	34	3	12	235	27	4.8	1.4	313	8.9	3.7	2.1	19.9
SM-2-A1	37.14	28	67	22	619	56	8.1	4.4	1861	12.9	1.7	14.3	11.8
SM-2-A2	33.67	39	12	22	600	27	8.6	5.3	1506	16.6	1.8	10.7	18.7
SM-2-B1	25.23	28	8	24	436	20	4.8	4.6	1831	11.9	1.6	13.9	11.5
SM-2-B2	17.91	34	11	44	1279	28	6.7	5.9	1608	27.4	2	14.5	18.4
SM-2-C1	18	36	11	104	950	78	6.1	4.4	1494	25	6.1	10	26.1
SM-2-C2	18.47	38	16	36	834	61	6	2.5	1446	20.6	3.5	7.6	15.2
SM-2-D1	15.32	23	13	31	556	27	4.6	2.9	1547	15	2.2	14.4	11.1
SM-2-D2	25.21	30	8	53	376	26	5.2	2.2	896	10.3	2.3	4	7.9
SM1 P2	84.61	38	2	7	149	28	4.5	1.4	613	5.3	2.4	3	3.8
SM1 C.1	23.02	38	9	32	999	108	12.2	4.8	847	25.6	9.5	7.8	23
SM1 C.2	24.08	34	8	32	640	60	7.1	3.4	328	17	3.2	5.8	15
SM1 P3.1	84.82	37	1	15	209	32	4.4	1.5	398	7.9	2.4	2.4	3.7
SM1 P3.2	84.46	46	1	30	445	24	6.2	2.9	661	14.2	2.7	1.7	3.9
SM1 P3.3	82.04	33	1	5	238	32	6.1	0.8	898	6.8	2.6	2.2	5.1
SM1 D.1	25.42	17	8	56	302	22	3.9	2.1	893	8.3	1.4	8.3	6.3
World brown coal ash		29	11	48	190	18	4.7	5	900	7.5	1.4	6	16
5x World brown coal ash		145	55	240	950	90	24	25	4500	38	7	30	80

Table 5

Remnant moisture, Sc, Y, and the lanthanides plus World brown coal average and 5× average values from Ketris and Yudovich (2009) on a dry coal (a) and ash (b) basis (the “5×” values are highlighted in blue, bold, italic font; ppm; dry coal and ash basis; dl, below detection limit; —, no data; Ch., Channel). Data sources: ⁺USGS Energy Resources Program (2014); *this study, unless noted otherwise.

(a)		%;	%; dry	μg/g; dry basis ^x														
Field Number ⁺	Moisture ^x	Ash ^x	Sc	Y	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
M128 CH1	9.61	11.85	3	14.2	6.5	12	1.4	5.3	1.1	0.19	1.3	0.22	1.5	0.36	1.19	0.18	1.16	0.19
M128 CH2	10.08	16.77	3.8	13.8	9.4	15.7	1.7	6.4	1.3	0.24	1.5	0.25	1.7	0.38	1.19	0.17	1.13	0.17
M128 CH3	7.98	55.09	7.7	31.3	44.5	95.8	10.5	39	7.5	1.28	6.9	1.06	6	1.14	3.33	0.48	3.1	0.45
M128 CH4	7.74	25.82	4.9	45.5	<i>56.2</i>	109	10.9	39.1	6.7	1.31	7.2	1.06	6.1	1.27	3.72	0.51	3.18	0.48
GC 4500 Roof	3.34	96.79	5.3	12.9	20.7	43.2	5	18.7	3.7	0.74	3	0.45	2.6	0.51	1.54	0.24	1.62	0.24
GC-4500.1	21.22	72.94	16.5	<i>78.3</i>	<i>95.8</i>	283	<i>36.6</i>	147	<i>33.6</i>	5.58	<i>25.7</i>	<i>3.95</i>	<i>21.6</i>	<i>3.99</i>	11.7	<i>1.72</i>	12.1	1.73
GC-4500 - P1 Ch.	14.79	87.69	8.3	24.2	33.6	68.3	7.8	29.6	5.6	0.96	5.1	0.77	4.5	0.92	2.79	0.43	2.9	0.45
GC-4500.2	35.49	37.02	<i>23.5</i>	<i>296</i>	<i>321</i>	<i>1060</i>	<i>141</i>	<i>567</i>	<i>131</i>	<i>21.7</i>	<i>102</i>	<i>15.8</i>	<i>86.6</i>	<i>15.7</i>	<i>45.7</i>	<i>6.58</i>	<i>45.2</i>	<i>6.59</i>
GC-4500.3	32.18	46.27	12.4	<i>166</i>	<i>208</i>	<i>662</i>	<i>86.3</i>	<i>351</i>	<i>80.1</i>	<i>13</i>	<i>59.3</i>	<i>8.93</i>	<i>48.3</i>	<i>8.8</i>	<i>25.4</i>	<i>3.65</i>	<i>24.7</i>	<i>3.52</i>
GC-3500.1	10.15	18.75	4.4	<i>110</i>	<i>75.3</i>	<i>230</i>	<i>30.3</i>	<i>127</i>	<i>31.8</i>	<i>5.57</i>	<i>27.2</i>	<i>4.53</i>	<i>26.6</i>	<i>5.16</i>	<i>15.6</i>	<i>2.37</i>	<i>16.1</i>	<i>2.37</i>
GC-3500-P1	4.89	57.78	3.3	<i>67.2</i>	<i>59.8</i>	<i>154</i>	<i>19.5</i>	<i>80</i>	<i>18</i>	<i>3.13</i>	<i>15.9</i>	<i>2.52</i>	<i>14.4</i>	<i>2.8</i>	<i>8.27</i>	1.22	7.79	1.15
GC 3500.2	8.64	15.24	5	<i>122</i>	<i>84.9</i>	<i>232</i>	<i>28.3</i>	<i>112</i>	<i>23.5</i>	<i>4.03</i>	<i>22.8</i>	<i>3.6</i>	<i>20.5</i>	<i>4.07</i>	<i>11.5</i>	<i>1.53</i>	<i>8.67</i>	<i>1.25</i>
GC-3500-P2 ⁺	-	74.3 ⁺	7.43 ⁺	32.69 ⁺	<i>52.01⁺</i>	81.73 ⁺	8.17 ⁺	29.72 ⁺	4.46 ⁺	dl ⁺	5.2 ⁺	dl ⁺	4.46 ⁺	dl ⁺	2.23 ⁺	dl ⁺	1.49 ⁺	-
GC-3500.3	10.54	10	2.8	<i>68.8</i>	<i>53.4</i>	<i>129</i>	<i>13.9</i>	51.1	8.5	<i>1.29</i>	<i>10.1</i>	<i>1.48</i>	<i>8.3</i>	<i>1.73</i>	<i>4.9</i>	0.62	<i>3.39</i>	0.51
GC 3500.4	20.16	8.81	3	<i>70.2</i>	<i>47.8</i>	<i>125</i>	<i>13.7</i>	<i>49.5</i>	8.3	<i>1.28</i>	<i>9.7</i>	<i>1.44</i>	<i>8.2</i>	<i>1.71</i>	<i>4.81</i>	0.61	<i>3.37</i>	0.5
GC-3500.5	8.38	58.2	5.2	<i>44.7</i>	33.4	74.4	8.3	30.5	6.4	0.88	6.5	1.1	6.9	1.45	<i>4.53</i>	0.7	4.78	0.73
SM-2-A1	8.4	37.14	8.5	37.4	15.4	33	4.1	16.1	3.9	0.69	4.3	0.81	5.5	1.21	3.85	0.58	3.85	0.59
SM-2-A2	7.98	33.67	8.7	28.7	11.5	24.3	3	12.5	3.3	0.58	3.7	0.7	4.6	0.97	2.93	0.43	2.77	0.41
SM-2-B1	9.01	25.23	6.2	17.5	20.5	42.1	4.9	18.6	4	0.67	3.7	0.6	3.4	0.65	1.86	0.26	1.65	0.24
SM-2-B2	9.97	17.91	7.8	19.4	11.8	24.4	3.1	12.6	3.2	0.58	3.3	0.59	3.7	0.73	2.14	0.31	2.03	0.3
SM-2-C1	10.05	18	8	27	24.6	<i>51.5</i>	6.1	24.3	5.5	0.93	5.2	0.87	5.2	1.02	2.96	0.42	2.73	0.4
SM-2-C2	9.35	18.47	6.3	30	25.4	52.5	6.3	24.8	5.5	0.85	5.4	0.88	5.4	1.09	3.27	0.48	3.15	0.47
SM-2-D1	10.76	15.32	3.9	22.2	15.7	34	4	15.8	3.5	0.54	3.5	0.59	3.5	0.72	2.16	0.31	1.99	0.3
SM-2-D2	9.69	25.21	5.2	24.3	15.6	29.7	3.5	13.9	3.1	0.57	3.3	0.53	3.4	0.71	2.14	0.3	1.91	0.29
SM1 P2	4.4	84.61	6.5	8.9	24.9	46.3	5	17.9	3.3	0.6	2.6	0.39	2	0.35	0.99	0.13	0.86	0.12
SM1 C.1	9.08	23.02	8.5	22.8	20.3	42.1	4.9	19.6	4.4	0.75	4.2	0.7	4.2	0.84	2.48	0.36	2.3	0.34
SM1 C.2	7.69	24.08	6.3	23.8	19.1	40.5	4.9	19.5	4.4	0.67	4.3	0.72	4.4	0.9	2.67	0.39	2.56	0.38
SM1 P3.1	6.22	84.82	6.5	11.9	19.8	46.7	5.4	20.4	4.3	0.57	3.5	0.55	2.9	0.54	1.52	0.22	1.45	0.21
SM1 P3.2	6.95	84.46	7.2	14.3	23.6	50.9	6.1	22.9	4.9	0.62	4	0.62	3.4	0.62	1.8	0.25	1.65	0.24
SM1 P3.3	5.48	82.04	6.2	16.1	24	51.7	5.9	22.2	4.6	0.7	4	0.62	3.5	0.68	2.03	0.3	1.99	0.29
SM1 D.1	8.71	25.42	3.7	18.4	9.7	20.3	2.5	10.2	2.4	0.42	2.5	0.42	2.6	0.53	1.57	0.22	1.37	0.21
World brown coal			4.1	8.6	10	22	3.5	11	1.9	0.5	2.6	0.32	2	0.5	0.85	0.31	1	0.19
5x World brown coal			<i>20.5</i>	<i>43</i>	<i>50</i>	<i>110</i>	<i>17.5</i>	<i>55</i>	<i>9.5</i>	<i>2.5</i>	<i>13</i>	<i>1.6</i>	<i>10</i>	<i>2.5</i>	<i>4.25</i>	<i>1.55</i>	<i>5</i>	<i>0.95</i>

(b) Field Number ⁺	%; dry	μg/g; ash basis ^x															
	Ash ^x	Sc	Y	La	Ce	Pr	Nd	Sm	Eu	Gd	Tb	Dy	Ho	Er	Tm	Yb	Lu
M128 CH1	11.85	25.3	119.8	54.9	101.3	11.8	44.7	9.3	1.6	11.0	1.9	12.7	3.0	10.0	1.5	9.8	1.6
M128 CH2	16.77	22.7	82.3	56.1	93.6	10.1	38.2	7.8	1.4	8.9	1.5	10.1	2.3	7.1	1.0	6.7	1.0
M128 CH3	55.09	14.0	56.8	80.8	173.9	19.1	70.8	13.6	2.3	12.5	1.9	10.9	2.1	6.0	0.9	5.6	0.8
M128 CH4	25.82	19.0	176.2	217.7	422.2	42.2	151.4	26.0	5.1	27.9	4.1	23.6	4.9	14.4	2.0	12.3	1.9
GC 4500 Roof	96.79	5.5	13.3	21.4	44.6	5.2	19.3	3.8	0.8	3.1	0.5	2.7	0.5	1.6	0.3	1.7	0.3
GC-4500.1	72.94	22.6	107.4	131.3	388.0	50.2	201.5	46.1	7.7	35.2	5.4	29.6	5.5	16.0	2.4	16.6	2.4
GC-4500 - P1 Ch.	87.69	9.5	27.6	38.3	77.9	8.9	33.8	6.4	1.1	5.8	0.9	5.1	1.1	3.2	0.5	3.3	0.5
GC-4500.2	37.02	63.5	799.6	867.1	2863.3	380.9	1531.6	353.9	58.6	275.5	42.7	233.9	42.4	123.5	17.8	122.1	17.8
GC-4500.3	46.27	26.8	358.8	449.5	1430.7	186.5	758.6	173.1	28.1	128.2	19.3	104.4	19.0	54.9	7.9	53.4	7.6
GC-3500.1	18.75	23.5	586.7	401.6	1226.7	161.6	677.3	169.6	29.7	145.1	24.2	141.9	27.5	83.2	12.6	85.9	12.6
GC-3500-P1	57.78	5.7	116.3	103.5	266.5	33.8	138.5	31.2	5.4	27.5	4.4	24.9	4.9	14.3	2.1	13.5	2.0
GC 3500.2	15.24	32.8	800.5	557.1	1522.3	185.7	734.9	154.2	26.4	149.6	23.6	134.5	26.7	75.5	10.0	56.9	8.2
GC-3500-P2 ⁺	74.3 ⁺	10 ⁺	44 ⁺	70 ⁺	110 ⁺	11 ⁺	40 ⁺	6 ⁺	dl ⁺	7 ⁺	dl ⁺	6 ⁺	dl ⁺	3 ⁺	dl ⁺	2 ⁺	
GC-3500.3	10	28.0	688.0	534.0	1290.0	139.0	511.0	85.0	12.9	101.0	14.8	83.0	17.3	49.0	6.2	33.9	5.1
GC 3500.4	8.81	34.1	796.8	542.6	1418.8	155.5	561.9	94.2	14.5	110.1	16.4	93.1	19.4	54.6	6.9	38.3	5.7
GC-3500.5	58.2	8.9	76.8	57.4	127.8	14.3	52.4	11.0	1.5	11.2	1.9	11.9	2.5	7.8	1.2	8.2	1.3
SM-2-A1	37.14	22.9	100.7	41.5	88.9	11.0	43.4	10.5	1.9	11.6	2.2	14.8	3.3	10.4	1.6	10.4	1.6
SM-2-A2	33.67	25.8	85.2	34.2	72.2	8.9	37.1	9.8	1.7	11.0	2.1	13.7	2.9	8.7	1.3	8.2	1.2
SM-2-B1	25.23	24.6	69.4	81.3	166.9	19.4	73.7	15.9	2.7	14.7	2.4	13.5	2.6	7.4	1.0	6.5	1.0
SM-2-B2	17.91	43.6	108.3	65.9	136.2	17.3	70.4	17.9	3.2	18.4	3.3	20.7	4.1	12.0	1.7	11.3	1.7
SM-2-C1	18	44.4	150.0	136.7	286.1	33.9	135.0	30.6	5.2	28.9	4.8	28.9	5.7	16.4	2.3	15.2	2.2
SM-2-C2	18.47	34.1	162.4	137.5	284.2	34.1	134.3	29.8	4.6	29.2	4.8	29.2	5.9	17.7	2.6	17.1	2.5
SM-2-D1	15.32	25.5	144.9	102.5	221.9	26.1	103.1	22.9	3.5	22.9	3.9	22.9	4.7	14.1	2.0	13.0	2.0
SM-2-D2	25.21	20.6	96.4	61.9	117.8	13.9	55.1	12.3	2.3	13.1	2.1	13.5	2.8	8.5	1.2	7.6	1.2
SM1 P2	84.61	7.7	10.5	29.4	54.7	5.9	21.2	3.9	0.7	3.1	0.5	2.4	0.4	1.2	0.2	1.0	0.1
SM1 C.1	23.02	36.9	99.0	88.2	182.9	21.3	85.1	19.1	3.3	18.3	3.0	18.3	3.7	10.8	1.6	10.0	1.5
SM1 C.2	24.08	26.2	98.8	79.3	168.2	20.4	81.0	18.3	2.8	17.9	3.0	18.3	3.7	11.1	1.6	10.6	1.6
SM1 P3.1	84.82	7.7	14.0	23.3	55.1	6.4	24.1	5.1	0.7	4.1	0.7	3.4	0.6	1.8	0.3	1.7	0.3
SM1 P3.2	84.46	8.5	16.9	27.9	60.3	7.2	27.1	5.8	0.7	4.7	0.7	4.0	0.7	2.1	0.3	2.0	0.3
SM1 P3.3	82.04	7.6	19.6	29.3	63.0	7.2	27.1	5.6	0.9	4.9	0.8	4.3	0.8	2.5	0.4	2.4	0.4
SM1 D.1	25.42	14.6	72.4	38.2	79.9	9.8	40.1	9.4	1.7	9.8	1.7	10.2	2.1	6.2	0.9	5.4	0.8
World brown coal ash		23	44	61	120	13	58	11	2.3	16	2	12	3.1	4.6	1.8	5.5	1.1
5x World brown coal ash		115	220	305	600	65	290	55	12	80	10	60	16	23	9	28	6

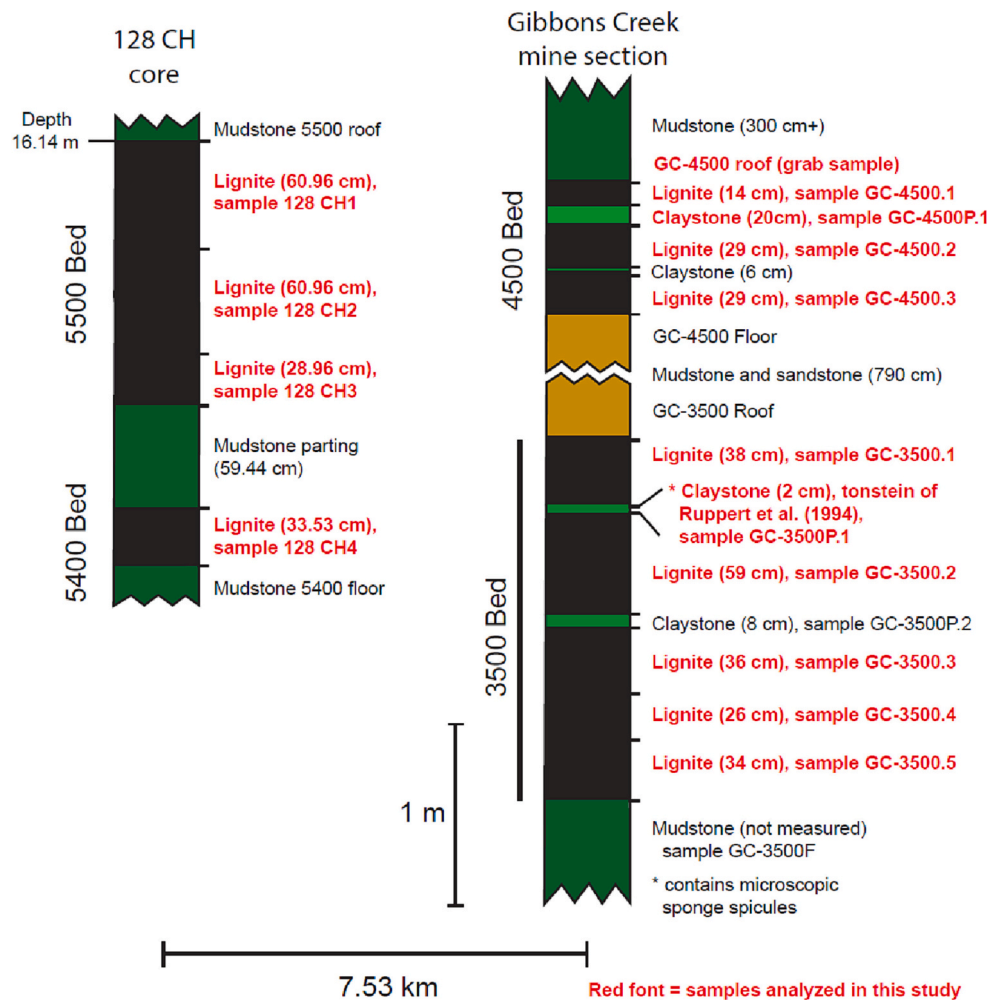


Fig. 3. Stratigraphic profile of the Gibbons Creek (GC) mine site and core CH 128 showing the relative location of the lignite beds and the samples analyzed in this study. After Warwick et al. (1997).

dissolve the remaining salts into solution. The sample was then transferred and diluted to final volume and analyzed. The samples were analyzed using a Perkin Elmer NexION 300× Inductively Coupled Plasma Mass Spectrometer (ICP-MS). Eleven different standard reference materials (SRMs) were analyzed in order to verify the analytical technique; five National Institute of Standards and Technology (NIST) SRMs (1632D, 1633b, 1633c, 2702, and 2711), two South African Reference Material (SARM) standards (18 and 19), and four USGS standards (AGV-2, BHVO-1, MAG-1, and SGR-1) (written communication, 2023, Tim Hutchison, Laboratory Manager Standard Laboratories, Inc.; also see Murphy et al., 2018). Concentrations are reported on both a dry coal and ash basis for Sc, Y, and the REEs (Tables 4 and 5).

2.4. Notes on the expression of REE results

While the International Union of Pure and Applied Chemistry includes lanthanide elements, Y, and Sc as REEs (Connelly et al., 2005), in the present study we use REEs to describe only the lanthanides, with REY designated for REE + Y, and REYSc for REE + Y + Sc. Light REEs (LREEs) are designated as La through Sm and Heavy REEs (HREEs) are defined as Eu through Lu after Seredin (1996) and Hower et al. (1999). In this classification, elements that would alternately be considered MREEs (Sm, Eu) are included with the LREEs, and others (Gd, Tb) are included with the HREEs. The Upper Continental Crust (UCC) normalization of REE abundances (indicated by the suffix “N”) (after Taylor and

McLennan, 1985) is used to delineate L-type (light type; $La_N/Lu_N > 1$), M-type (medium or middle type; $La_N/Sm_N < 1$, $Gd_N/Lu_N > 1$), and H-type (heavy type; $La_N/Lu_N < 1$) enrichment patterns (Seredin and Dai, 2012) as well as the decoupled Ce, Eu, and Gd distributions used to indicate anomalous enrichment or depletion of these elements (Eqs. (1), (2), and (3)) (Bau and Dulski, 1996; Dai et al., 2016b, 2017a, 2017b):

$$Eu_N/Eu_N^* = Eu_N / (0.67Sm_N + 0.33Tb_N) \quad (1)$$

$$Ce_N/Ce_N^* = Ce_N / (0.5La_N + 0.5Pr_N) \quad (2)$$

$$Gd_N/Gd_N^* = Gd_N / (0.33Sm_N + 0.67Tb_N) \quad (3)$$

All modes of Seredin and Dai's (2012) REE enrichment in coals (tuffaceous, detrital, infiltrational/leaching, and hydrothermal) are possible in the Jackson Group lignites. Along with the latter modes of enrichment, peat and low-rank coals can have organic associations, with HREEs having a greater affinity for organics than the LREEs leading to a relatively low LREE/HREE (Eskenazy, 1978, 1987a, 1987b, 1987c, 1999, 2015; Given, 1984; Eskenazy et al., 1986; Hatcher and Clifford, 1996; Pédrot et al., 2010; Davranche et al., 2011; Aide and Aide, 2012). If organic-bound rare earths are removed from the coal structure they could be leached from the coals, bound to clays (Eskenazy, 1995, 1999; Seredin, 1996), or incorporated into carbonates and phosphates. Further redistribution of light and heavy REEs could be accomplished via leaching of LREEs from adjacent and in-seam rocks (Dai et al., 2006,

Table 6

Calculated rare earth parameters (Dist., Distribution; H, Heavy; L, Light; –, no data). Data sources: ⁺USGS Energy Resources Program (2014); ^{*}this study.

Field Number ⁺	$\mu\text{g/g}$; ash basis [*]			LREE/ HREE	Eu _N /Eu _N *	Ce _N /Ce _N *	Gd _N /Gd _N *	Dist
	REE	REY	REYSc					
M128 CH1	275	395	420	4.18	0.76	0.91	1.05	H
M128 CH2	246	328	351	5.13	0.82	0.89	1.05	H
M128 CH3	401	458	472	8.31	0.86	1.01	1.05	H
M128 CH4	956	1132	1151	8.93	0.94	1.00	1.13	L
GC 4500 Roof	106	119	124	8.35	1.05	0.97	1.03	H
GC-4500.1	938	1045	1068	6.77	0.88	1.06	0.98	H
GC-4500 - P1 Channel	187	214	224	7.70	0.86	0.96	1.06	H
GC-4500.2	6931	7731	7794	6.42	0.87	1.08	0.99	H
GC-4500.3	3421	3780	3807	7.09	0.88	1.08	0.98	H
GC-3500.1	3199	3786	3810	4.69	0.88	1.06	0.97	H
GC-3500-P1	672	789	794	5.79	0.88	1.02	1.01	H
GC 3500.2	3666	4466	4499	6.17	0.84	1.06	1.04	H
GC-3500-P2 ⁺	255 ⁺	299 ⁺	309 ⁺	-	-	0.89 ⁺	-	-
GC-3500.3	2882	3570	3598	7.92	0.7	1.08	1.17	L
GC 3500.4	3132	3929	3963	7.73	0.72	1.11	1.15	H
GC-3500.5	310	387	396	5.55	0.64	1.02	1.01	H
SM-2-A1	253	353	376	3.39	0.77	0.95	0.95	H
SM-2-A2	213	298	324	3.19	0.75	0.94	0.95	H
SM-2-B1	409	478	503	6.91	0.82	0.96	1.01	H
SM-2-B2	384	492	536	4.03	0.82	0.92	0.97	H
SM-2-C1	732	882	926	5.68	0.82	0.96	1	H
SM-2-C2	734	896	930	5.46	0.74	0.95	1.03	H
SM-2-D1	565	710	736	5.36	0.72	0.98	1.01	H
SM-2-D2	313	410	430	5.00	0.86	0.92	1.06	H
SM1 P2	125	135	143	12.13	0.97	0.94	1.01	L
SM1 C.1	467	566	603	5.65	0.82	0.96	1	H
SM1 C.2	438	537	563	5.20	0.72	0.95	1	H
SM1 P3.1	127	141	149	8.42	0.68	1.03	0.99	H
SM1 P3.2	144	161	169	8.22	0.66	0.97	1.01	H
SM1 P3.3	149	169	177	7.68	0.77	0.99	1.02	H
SM1 D.1	216	289	303	4.58	0.81	0.94	1.02	H
World brown coal ash	311	355	378					
5x World brown coal ash	1557	1777	1892					

2008), by the hydrothermal mobilization of HREE-depleted phosphates and HREE- and Y-enriched organic compounds (Dai et al., 2013a, 2013b, 2016a), and by enrichment in LREEs by hydrothermal fluids (Williams-Jones et al., 2012; Hower et al., 2020a).

3. Results and discussion

3.1. Gibbons Creek section

Among samples investigated, certain lignite lithologies within the 3500 and 4500 beds at the GC site (Fig. 3) show strikingly high ash-basis REE, REY, and REYSc values, approaching 7800-ppm (ash basis) REYSc for sample 4500.2 (Table 6). The plots of the Upper Continental Crust normalized REE values (after Taylor and McLennan, 1985) emphasize the magnitude of the seven highest values (lignite samples 4500.1, 4500.2, 4500.3, 3500.1, 3500.2, 3500.3, and 3500.4; Fig. 4). With the exception of 4500.1, all of the latter samples have REE, REY, and REYSc

concentrations of 5× the World average for brown coals (averages after Ketris and Yudovich, 2009). Lignite 4500.1 is just under 3× the World average. Aside from the greater magnitude of 4500.2, the 4500.1, and 3500.1 lignites, the latter occurring above the 3500P.1 tonstein, basically have parallel trends showing an enrichment in Sm through Dy. Only when the high-REE samples are removed from consideration, do the patterns for the other lignites and rocks emerge (Fig. 5). Among the latter samples, 3500.5, the lowermost lignite bench, shows a HREE-enriched pattern resembling those seen in peat and lower- to moderate-rank coals elsewhere, for example, in Maine peats (Cameron and Wright, 1977) through high volatile C bituminous coal (Eagle Pass, Texas; Dai et al., 2022) through high volatile A bituminous coals (various eastern Kentucky coals; Hower et al., 2015, 2022a; Johnston et al., 2015).

For the analyzed 3500 bed samples, 3500.1, 3500.2, and 3500.3 (the only sample among the 3500 and 4500 samples with an L-type enrichment), coals immediately below the roof, 3500P.1 parting (a claystone

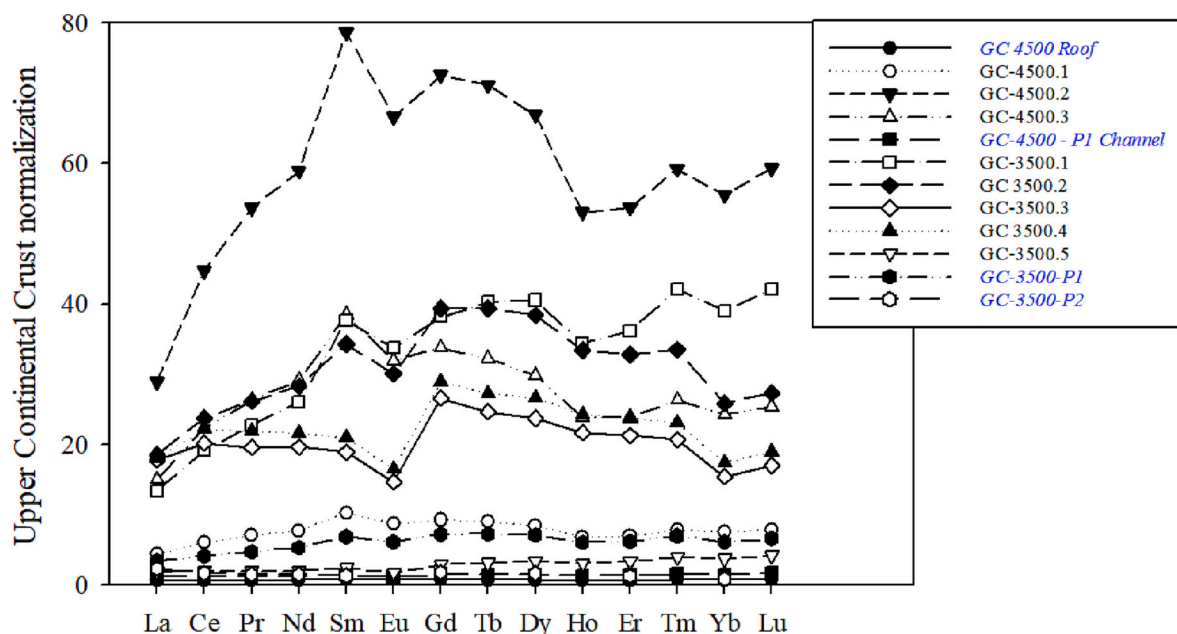


Fig. 4. Upper Continental Crust normalized REE values for all of the GC samples (after Taylor and McLennan, 1985). The rock (roof and parting) samples are designated by the blue italic font in the legend. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

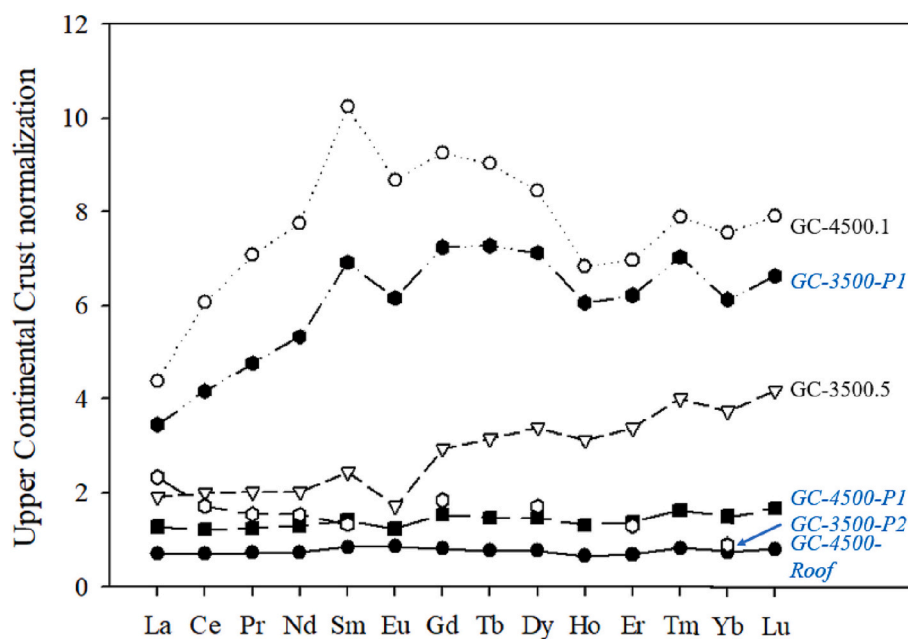


Fig. 5. Upper Continental Crust normalized REE values for the lowest-REE GC samples (after Taylor and McLennan, 1985). The roof and parting rocks are labelled in blue italic font. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

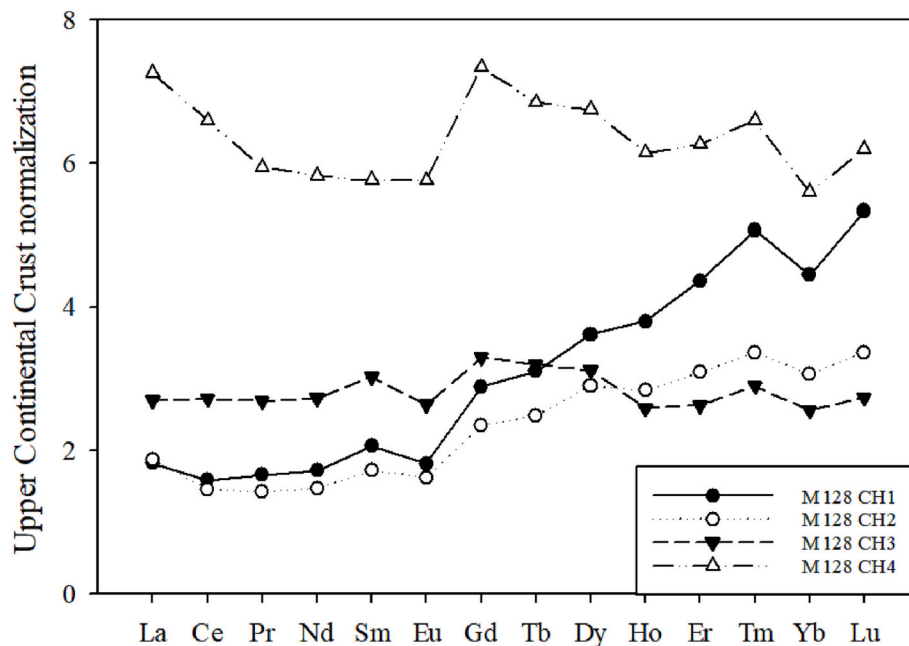


Fig. 6. Linear plot of the Upper Continental Crust normalized REE values for the 5500 coal samples (after Taylor and McLennan, 1985).

tonstein described by Ruppert et al. (1994)), and the 3500.P2 parting, respectively, show enriched REE concentrations. Lithium and Be are enriched in coals 3500.1 through 3500.4, with the Be concentration being 5× the World brown coal average on an ash basis and Li being up to 4× the average (Ketris and Yudovich, 2009). The parting 3500.P1, with 218-ppm Li (ash basis), underlies coal 3500.1 and overlies coal 3500.2, both with high Li concentrations. Coals 3500.3, immediately below the 3500.P2 claystone parting, and 3500.4 have Ge concentrations 5× the World average for brown coals. Along with 3500.1, below the roof, and 3500.2, overlain by the 3500.P1 tonstein and above the 3500.P2 parting, the enrichment in Ge, Li, and REEs in coals overlain by the roof or a parting suggests that leaching may have been a factor in the element enrichment in these lignites (after discussions of Ge enrichment by Yudovich, 2003). In any case, the discernment of trends is impacted by the thickness of the samples. Sampling, both here and in general, was not done with the resolution of minor element trends in mind.

The 5500 lignite overlies the 4500 lignite (Fig. 2). Four samples of the 5500 lignite show high Mn levels in CH1, CH2, and CH4; above average Li in CH1 through CH3; and high Be, Cr, and Ni in CH4 (Table 4). The UCC-normalized REE patterns are shown on Fig. 6. Lignites CH3 and CH4, above a mudstone parting and the mudstone floor, respectively, have high higher rare earth values than CH1 and CH2. The CH3 and CH4 light rare earths are relatively enriched compared the heavy rare earths, as is common for crustal materials in general, including coals (Dai et al., 2006, 2008, 2013a, 2013b, 2016a, 2017a, 2017b; Hower et al., 1999, 2015, 2020a, 2022a). This enrichment is particularly notable in the LREE/HREE average of 8.62 for CH3 and CH4 versus 4.65 for CH1 and CH2 (Table 6). Overall, the L-type distribution suggests that monazite or a similar LREE-rich mineral dominates the assemblage of REE minerals. The L-type distribution contrasts with the H-type distributions seen in the 4500 and 3500 coals. Further details of the differences in the concentrations and distributions of the REEs in the 5500 coals versus the other coals is discussed below.

3.2. San Miguel section

Within the San Miguel lignite section (Table 1; Fig. 7), the Upper

Continental Crust normalized REE values (after Taylor and McLennan, 1985) (Fig. 8) show that all of the benches have H-type REE distributions with negative Eu anomalies, relative to UCC. Sample A1, overlain by a carbonaceous shale roof, is the only sample with a high Ge content (81 ppm; ash basis).

3.3. Relative distribution of REEs and other elements

The distributions of the decoupled Eu_N/Eu_N^* vs. Ce_N/Ce_N^* , Eu_N/Eu_N^* vs. Gd_N/Gd_N^* , and Gd_N/Gd_N^* vs. Ce_N/Ce_N^* values are shown on Fig. 9–11, respectively. The Eu_N/Eu_N^* vs. Ce_N/Ce_N^* and Gd_N/Gd_N^* vs. Ce_N/Ce_N^* plots (Fig. 9 and 11), in particular, show the isolation of the high REYSc samples from the other lignites. In all three plots (Fig. 9–11), lignite 3500.4, a sample with an H-type distribution, shows a greater affinity to the L-type-distribution lignite 3500.3 than it does to the other high-REYSc lignites. The LREE/HREE of 3500.4 is 7.73, closer to the 3500.3 LREE/HREE (7.92) than to the average LREE/HREE of the other four high-REYSc/H-type-distribution lignites (6.09).

With minor overlap, the $100^*(Ti)/(Al + Ti)$ vs. LREE/HREE shows separation between the two sites, with the SM coals tending to have a higher $100^*(Ti)/(Al + Ti)$ and, to a lesser extent, lower LREE/HREE than the GC 3500 and 4500 lignites (Fig. 12). The GC-5500 coals fall into distinct regions with the lowest REYSc (<60 ppm on the ash basis) lignites being on the high $Ti/(Ti + Al)$ side of the SM cluster and CH3 and CH4 being on the high- LREE/HREE side of the GC cluster. The geochemistry of the CH1 and CH2 lignites suggests that the mineral input may have had more heavy minerals than CH3 and CH4 (and, indeed, perhaps the rest of the GC and SM lignites). While we do not have direct evidence for the mineralogy, the average Ti, suggesting a TiO_2 -bearing mineral input, for CH1 and CH2 is 7183 ppm (ash basis) compared to 4276 ppm for CH3 and CH4, 5341 ppm for the 3500 and 4500 lignites, and 4565 ppm for the SM-2 lignites. The Zr concentration, suggesting a zircon component, is less pronounced with only 292 ppm and 226 ppm averages for CH1 + CH2 and CH3 + CH4, respectively. The latter Zr averages are considerably less than the averages for the GC-3500 and -4500 lignites and the SM lignites, 408 ppm and 706 ppm, respectively.

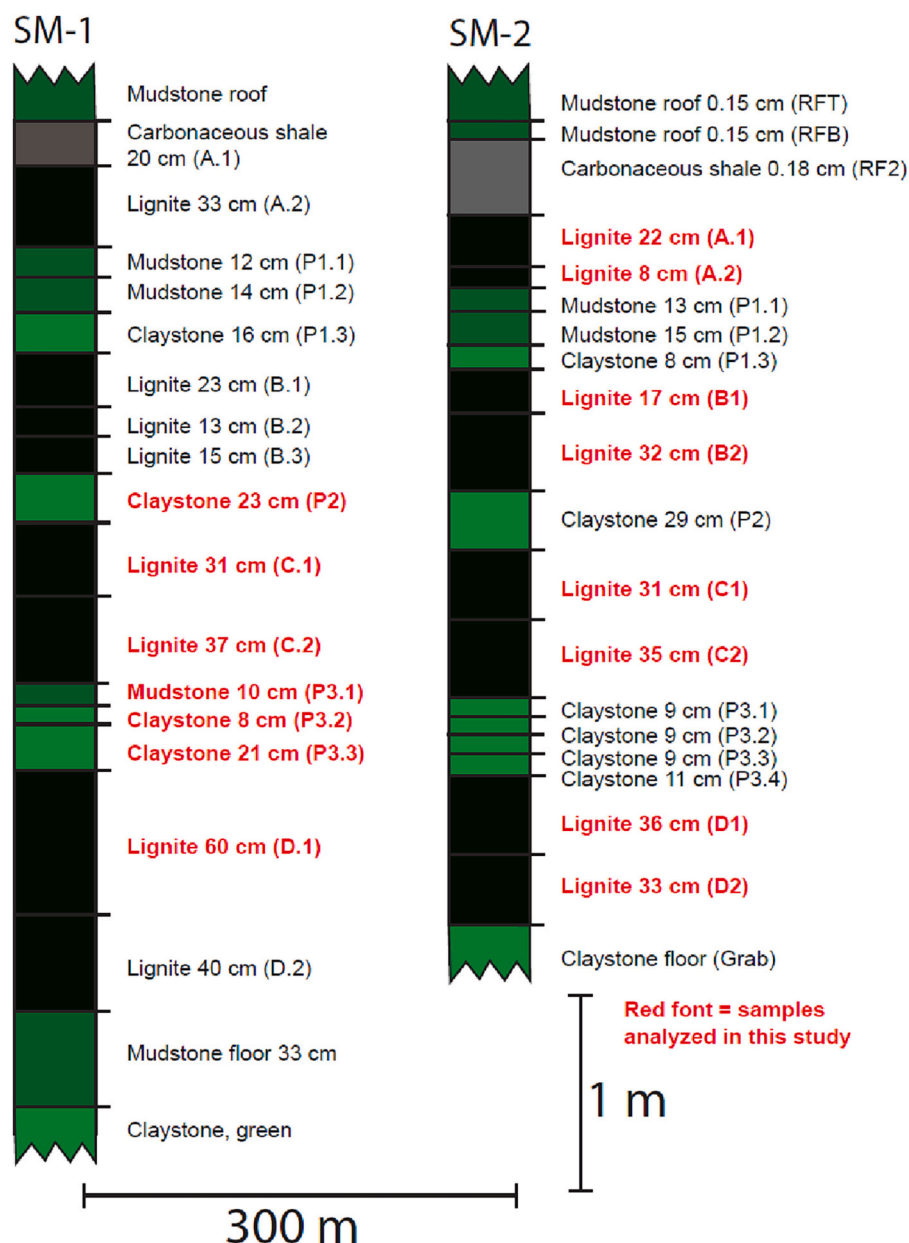


Fig. 7. Stratigraphic profile of the San Miguel (SM) mine site showing the relative location of the lignite beds and the samples analyzed in this study. After Warwick et al. (1996).

4. Conclusions

In central Texas, the Jackson Group lignite beds found in the Gibbons Creek mine section and the 128 CH core formed in delta plain and fluvial environments and are associated with fine- to medium-grained sands, siltstone and clay beds, and local bentonitic and tuffaceous volcanic ash beds. The San Miguel lignite beds in south Texas formed in lagoonal mires along with associated fine-grained sands, siltstone and clay beds, and reworked volcanic ash material. Lignite benches enriched in REEs in both intervals are associated with known volcanic ash beds. Infiltration/leaching (Seredin and Dai, 2012) of the volcanic ash material may have been an important REE enrichment mechanism in the lignite benches.

Lignite lithologies within the 3500 and 4500 beds at the GC site show high ash-basis REE, REY, and REYSc values, approaching 7800-ppm (ash basis) REYSc for sample 4500.2. The latter lignite, along with the 4500.1

and 3500.1 lignites, have parallel UCC_N REE patterns showing an M-type enrichment in Sm through Dy.

Lignite 3500.5, the lowermost lignite bench, shows an H-type (enrichment in the HREEs) pattern resembling those seen in Maine peats (Cameron and Wright, 1977), the Cretaceous high volatile C bituminous Eagle Pass, Texas coal (Dai et al., 2022), and various Pennsylvanian eastern Kentucky high volatile A bituminous coals (Hower et al., 2015, 2022a; Johnston et al., 2015). This suggests that there is a broader relationship between the basal peat and the underlying soil than just the pattern seen at our sites. The presence of the H-type enrichment is not necessarily dependent upon the mire vegetation and, at least up to a high volatile A bituminous rank, is independent of the subsequent coal rank.

Lithium and Be are enriched in coals 3500.1 through 3500.4. The parting 3500-P1, with 218-ppm Li (ash basis), underlies coals 3500.1 and overlies coal 3500.2, both with high Li concentrations. Coals

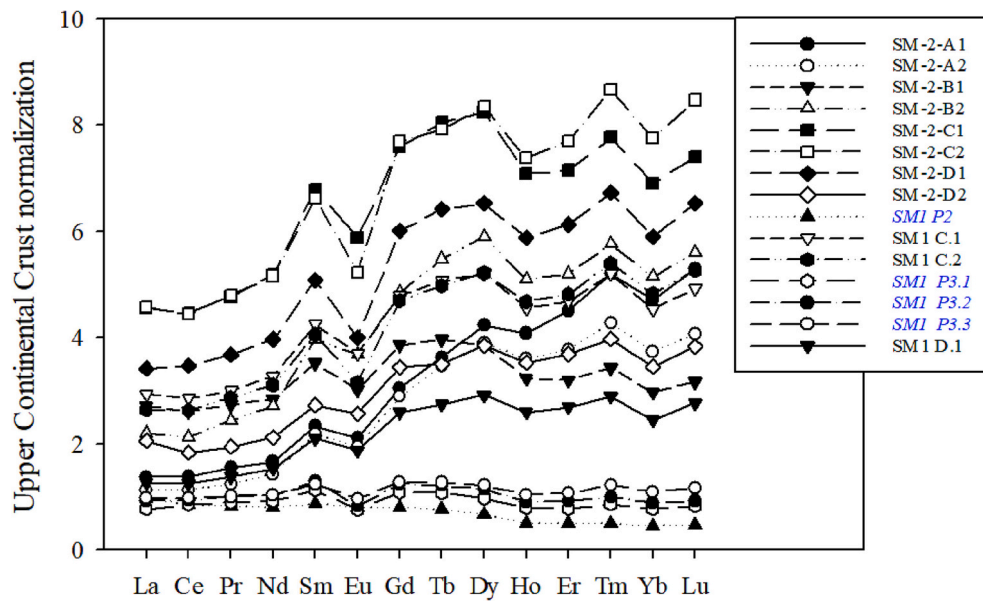


Fig. 8. Upper Continental Crust normalized REE values for all of the SM samples (after Taylor and McLennan, 1985). Sample numbers refer to abbreviated sample designation shown in Fig. 7. The rock partings are labelled in blue italic font. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

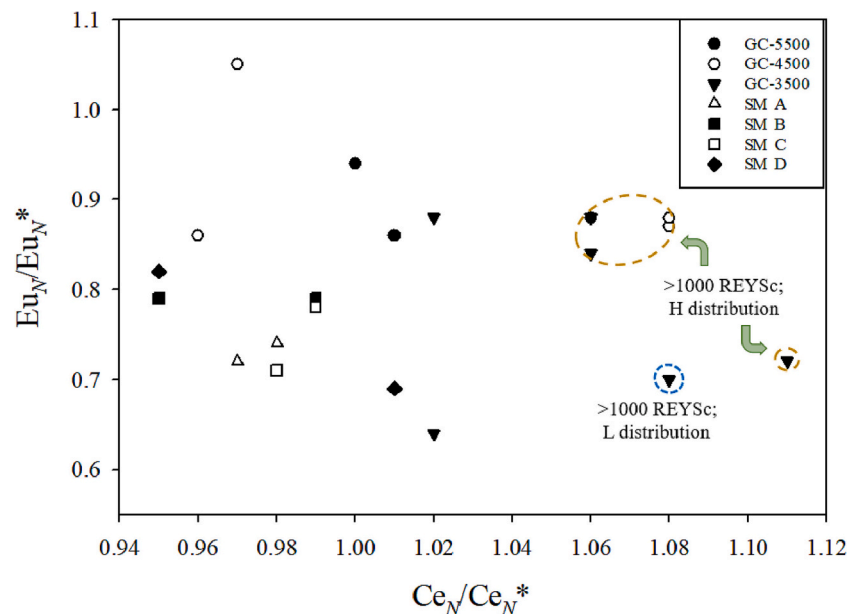


Fig. 9. Decoupled Eu_N/Eu_N^* vs. Ce_N/Ce_N^* (after Taylor and McLennan, 1985).

3500.3, immediately below the 3500.P2 claystone parting, and 3500.4 have Ge concentrations $5\times$ the World average for brown coals. Along with 3500.1, below the roof, and 3500.2, overlain by the 3500.P1 tonstein, the enrichment in Ge and REEs in coals overlain by the roof or a parting suggests that leaching may have been a factor in the element enrichment in these lignites, a phenomenon noted in other coals by Yudovich (2003) among others.

Among the 5500-series lignites, CH3 and CH4, above a mudstone parting and the mudstone floor, respectively, show a significant LREE enrichment compared to CH1 and CH2.

The LREE/HREE vs. $100*(Ti)/(Al + Ti)$ plots emphasize the separation of the two sites, with the SM coals tending to have a higher $100*(Ti)/(Al + Ti)$ and a lower LREE/HREE than the GC 3500 and 4500 lignites (Fig. 11). The GC-5500 coals are distinctly different from the other GC or the SM lignites, with the geochemistry of the CH1 and CH2 lignites suggesting that the mineral input may have had more heavy minerals (such as zircon, Ti-oxides, monazite) than in the underlying CH3 and CH4 lignites and in the rest of the GC and SM lignites.

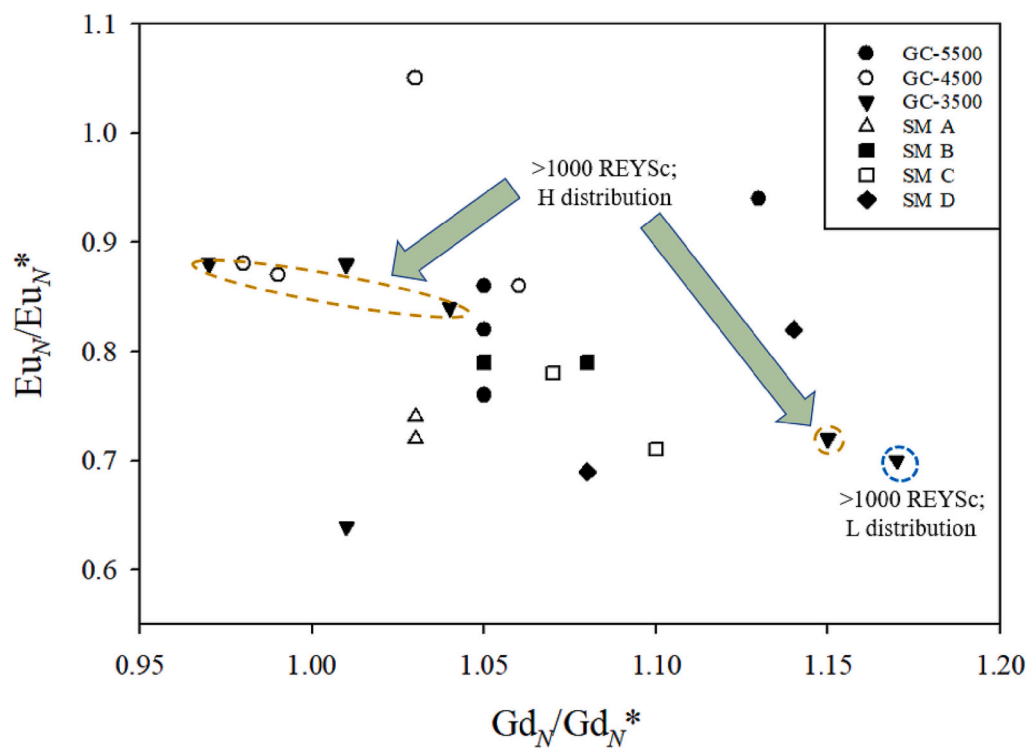


Fig. 10. Decoupled $\text{Eu}_N/\text{Eu}_N^*$ vs. $\text{Gd}_N/\text{Gd}_N^*$ (after Taylor and McLennan, 1985).

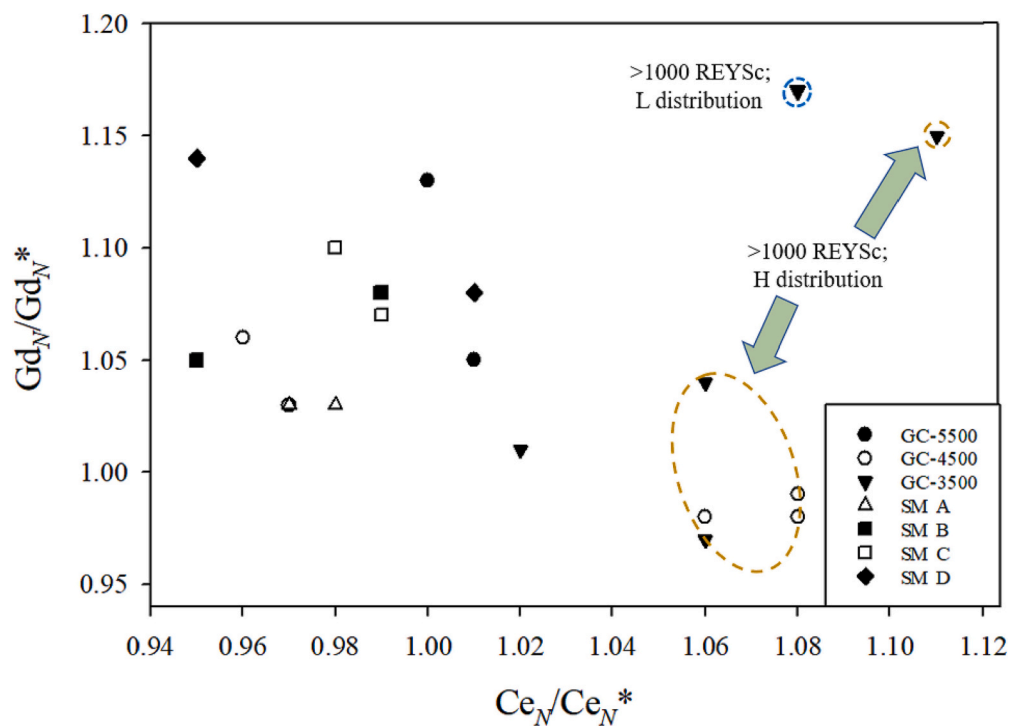


Fig. 11. Decoupled $\text{Gd}_N/\text{Gd}_N^*$ vs. $\text{Ce}_N/\text{Ce}_N^*$ (after Taylor and McLennan, 1985).

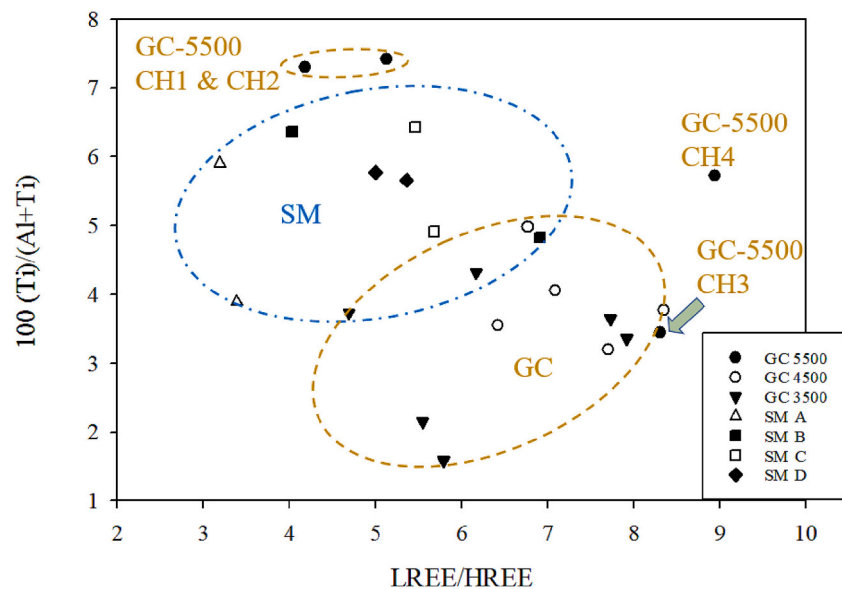


Fig. 12. $100 \cdot \text{Ti}/(\text{Al} + \text{Ti})$ vs. LREE/HREE for all samples. The areas for the SM and GC samples are indicated on the plot.

Declaration of Competing Interest

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the U.S. Department of Energy nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the U.S. Department of Energy.

The authors declare no conflicts of interest. All authors contributed to the writing of the manuscript.

Data availability

All data used in this study are included in the manuscript.

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