APPROVED:

Hall. Vc ER 6 F.L. Whitier J.M. Lerip. J

THE EAGLE FORD FORMATION OF TRAVIS COUNTY, TEXAS

THE EAGLE FORD FORMATION OF TRAVIS COUNTY, TEXAS

THESIS

Presented to the Faculty of the Graduate School of The University of Texas in Partial Fulfillment of the Requirements

For the Degree of

MASTER OF ARTS

By

Guy Emmett Green, B. A. (Mason, Texas) Austin, Texas June, 1925

PREFACE

The subject of this paper was suggested to the writer by Dr. H. P. Bybee, who at all times has given his constructive criticism and advice. Professor F. L. Whitney has given many valuable suggestions. Mr. L. F. Marek, of the Department of Chemistry of the University of Texas, made this work complete by furnishing the analyses of oil, gas, and bentonite. Acknowledgement is also due Dr. Udden and Dr. Sellards, of the Bureau of Economic Geology and Technology, University of Texas, for valuable suggestions and access to certain literature not available elsewhere. Mr. J. B. Carsey photographed the specimens, and Mr. J. B. Lovejoy assisted in the preparation of the plates. For this aid the writer wishes to express his appreciation.

Guy E. Green.

The University of Texas, May 23, 1925. iii

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THE EAGLE FORD FORMATION OF TRAVIS COUNTY, TEXAS

INTRODUCTION

The Eagle Ford formation is one of the most interesting of the Cretaceous system. It presents a greater variety of changes than any other in that system. Not only does its lithology change, but its thickness in different parts of the state is a problem in itself. The greater part of animal life represented in the Eagle Ford is more or less characteristic of that age. In a number of places, especially in Travis County, asphalt and oil seeps have been found. Inasmuch as there has been no detailed work done on this formation in Travis County it was suggested by Dr. H. P. Bybee that a close study of it be made.

The Eagle Ford was named by Dr. R. T. Hill^{\perp} in 1887 because of its excellent exposure near the town by that name situated on the T. & P. Railroad six miles west of Dallas.

⁺Hill, R. T., The Topography and Geology of the Cross Timbers and Surrounding Regions in Northern Texas: Am. Jour. Sci., vol. 33, p. 298, 1887.

North of the Brazos the outcrops of the Eagle Ford make the extensive black waxy lands of the fertile Black Prairie lying between the White Rock scarp and Eastern Cross Timbers.

The Eagle Ford shale is at the base of the Colorado group. It is believed by Stephenson to be the undetermined part of the Eutaw formation of Alabama and Mississippi. In the western interior area of the United States the Benton² shale is regarded as approximately equivalent to the Eagle Ford.

In the region north of the Brazos River the formation lies conformably upon the Woodbine sands; while south of the Brazos it rests unconformably upon the Buda limestone. It is everywhere conformably overlain by the Austin Chalk.

The formation as a whole consists largely of laminated petroliferous shale which, when first exposed, is dark blue and sometimes with a silky luster, but upon weathering changes to a gray, brown, or light yellow color. It includes, in some places, thin ledges of sandstone, limestone, ledges with concretionary structure, and layers of

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Stanton, T. W., The Colorado formation and its invertebrate fauna: U. S. Geol. Survey Bull. 106, pp. 19, 48, 1893.

bentonite. In the region between the Brazos and Colorado rivers, where the formation is closely associated with the Del Rio clays, and both weather to form a dark brown soil, one experiences some difficulty in recognizing it. There is, however, a marked lithologic difference between them. The Eagle Ford is highly laminated, the laminae often appearing as shaly fragments on the weathered surfaces of the outcrops, and separated slabs. In the Del Rio clay there is no such weathering. This area is one of the most productive cotton regions of the state. South of the Brazos River the beds are more intermittent due to faulting and erosion. They extend from near Waco southward via Moody, Belton, Round Rock, Austin, Buda, and New Braunfels. At this point the exposures take a southwesterly direction and pass north of San Antonio to Hondo, Uvalde and thence to the Rio Grande There they are best exposed in Sycamore Creek and River. its tributaries, south of the S. P. railroad, between Kinney and Val Verde Counties.

The thickness of the formation has a wide range as shown by the following tabulation:³

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Hill, R. T., Geography and Geology of the Black and Grand Prairies, Texas: U. S. Geol. Survey, twenty-first Annual Report, pt. 7, p. 324, 1901.

Section	Thickness
Paris (on Red River)	600 feet
Sherman	600 feet
Dallas	480-500 feet
Midlothian	350 feet
Waco (south of the Bosque)	200 feet
Austin	30 feet
Nueces	200 feet

The thickness of the Austin section was found by the writer to be about 42 feet.

R. T. Hill says this decrease in thickness southward is apparently accomplished by the gradual disappearance of the lower beds of the formation, the upper calcareous layers with laminated flagstones alone persisting and increasing in that direction, until at Austin they are the sole representatives of the beds. He gives the following section:

Section of Eagle Ford shale 2 miles west of Binkley House, at Sherman, Grayson County, Texas. Feet Sandy clay shale with Ostrea lugubris ------10 Thin layers of brown sandstone with conglomerate of small rounded jasper pebbles, Ostrea lugubris, and fish teeth -----5 Blue laminated clay, weathering into limomitic 10 colors -----Massive agglomerate of Ostrea lugubris 2 Sandy clay shale in thin alternations of clay and sand; clay efflorescent and drab on drying; Ostrea lugubris 40 Blue clay with gigantic septaria ------67

The above section does not appear to correlate with the one taken on Bouldin Creek, one mile south of Austin, where the total thickness is exposed in the east bluff of

It is the opinion of the writer that the secthat creek. tions at the two places are so widely different that it would be difficult to say that the formation exposed in the Austin section is the top phase of the formation farther north. It does seem probable, however, that the shales of McLennan County are about the same as found at the type section on Bouldin Creek. Adkins⁴ describes the formation in southern Bosque County in which he divides it into three parts, namely: (a) a basal shale, (b) a middle flag series, and (c) an upper shale. He places the total thickness at about 160 feet, of which the basal shale is about 40 feet, the middle flags about 85 feet, and the upper shale 35 feet. The above divisions are the same as found in the type section at Austin, but here the thickness of each phase is considerably reduced. The sections on the following pages will show the variation in thickness at the two localities.

Just why there is such a variation in the thickness of the formation from north to south is a problem in itself. In order to get at the cause of such changes a knowledge of the geography of Eagle Ford times would be necessary.

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Adkins, W. S., Geology and Mineral Resources of McLennan County: Univ. of Tex., Bull. No. 2340, p. 67, 1923.

South of the Brazos River there is a peculiar relation between the outcrop of the formation and the trace of the Balcones fault, but north of the Brazos there is no probable connection between the two. Whether there is any relation existing between them in any of their parts is altogether hypothetical. It has been suggested by some that the Central Mineral Region upshift might possibly be a factor in causing the formation to thin in the Austin-San Antonio Since this region has been covered by all of the area. Cretaceous⁵ it is probable that the uplift had nothing to do with the thickness. The nature, extent, and elevation of the land mass from which sediments were derived is a probable factor, but to determine this a careful study of the geographic conditions of that time would be necessary. Another probable factor influencing deposition of sediments would be the action of tides. Inasmuch as the Eagle Ford lies unconformably on the Buda limestone in the region around Austin, and conformably on the Woodbine sands north of the Brazos, we recognize a break in deposition which left out the Woodbine sand. This seems to indicate that the area south of

Paige, Sidney, Geologic Atlas, No. 183, U. S. Geol. Survey, 1912.

the Brazos could have been above sea level during the deposition of the Woodbine sands, and also while the greater part of the Eagle Ford shale was being deposited in North Texas. Whether any one of the above factors, alone or acting together, had anything to do with the variation in thickness of the formation cannot be determined until a thorough investigation of sea and land conditions and areas has been made.

Hill⁶ lists the following invertebrate fossils from the Eagle Ford formation:

> Placenticeras syrtalis Nort. var. cumminsi Crag. Ammonites woolgari Mort. Sphenodiscus dumblei Cragin. Buchiceras inequiplicatus Shum. B. swallovi Shum. Tapes hilgardi Shum. Anchura modesta Cragin. Fusus graysonensis Cragin. Natica striatacostata Cragin. Neritopsis brangulatus Shum. Ostrea lugubris Conrad. O. belliplicata Shum. Inoceramus fragilis Hall and Meek. I. labiatus Schlotheim.

Of the above list, Inoceramus sp. is found in great numbers in the middle phase, whereas a few occur in the basal portion of the Austin exposure. A small undetermined Ostrea

Hill, R. T., Geography and Geology of the Black and Grand Prairies, Texas: U. S. Geol. Survey, twenty-first Annual Report, pt. 7, p. 328, 1901.

is found in great abundance in the basal member of the middle phase. This fossiliferous ledge is a very hard, sandy limestone which is very persistent in Travis County and can be used as a good horizon marker. Several small gastropods are found in the basal member but they are not preserved well enough to be identified. In most cases only the mold or cast remains in the shale and upon exposure will crumble away. Some, however, are replaced with pyrite but even these are poorly preserved. Dr. Udden reports a heavy rather blunt-keeled ammonite with outer volution about one foot thick from the basal Eagle Ford blue shale on Bouldin Creek. The following Foraminifera are also found in the section taken on Bouldin Creek:

> Globigerina cretacea D'Orbigny Cristellaria sp. Textularia globulosa Ehrenberg Anomalina ammonoides Reuss

Some very peculiar organic remains were found in the basal phase of the shales from about four feet above the contact with the Buda limestone up to the fossiliferous sandy limestone ledge fifteen feet above the Buda. These remains resemble bones of small vertebrates, but upon close examination are found not to have bone structure. They are microscopic in size and well preserved. This material resembles arm joints of a particular Crinoid, Saccocoma, more than it

does anything else. The following is taken from Zittel, Vol. 1, p. 172:

"The only known genus, Saccocoma occurs profusely in the Lithographic Stone of Eichstadt and Solenhofen, Bavaria. It is a free-swimming form, whose affinities with the monocyclic Plicatocrinidae were first clearly demonstrated by Jaekel in 1892."

Dr. E. H. Sellards found several specimens in a well sample from Bexar County. The well belonged to F. B. Corle, located one-half mile east of the Medina-Bexar county line, and onehalf mile north of the Portranca road. This sample was taken from the well at a depth of 135-140 feet; Eagle Ford shale. (A photograph of this sample is found in plate XII).

Since there has been only one genus of this crinoid described, and that from the Upper Jura of Germany, it does not seem very probable that these remains could be of that genus. Conditions, however, might have been such that the remains were not preserved during Commanchean times. There is a possibility of the material being sponge spicules which are found throughout the Cretaceous; although the remains resemble arm joints of Saccocoma more than they do sponge spicules.

Fish teeth and scales are very abundant throughout the formation, but they are more abundant in the middle phase; some of the sandstone members are especially characterised



A. View showing a small fault in the middle phase of the shales on Walnut Creek one-eighth mile east of Watters Station, ten miles north of Austin.



B. View showing the middle shales, and a portion of the Upper shales on Walnut Creek one-fourth mile east of Watters Station.

by them. The different types of fish scales and teeth are figured on plate XIII.

THE EAGLE FORD FORMATION IN TRAVIS COUNTY

The Eagle Ford formation outcrops in Travis County in a narrow belt, extending from the northern boundary of the county southward to Watters Station on the International and Great Northern Railroad ten miles north of Austin, through the city of Austin to Manchaca and thence to the Travis-Hays county line.

In the northern part of the county there is a narrow belt of the formation from one-half to a mile in width. This locality affords no good section. It is represented on the map as Locality No. 1.

On Walnut Creek, a quarter of a mile east of Watters Station, the formation occurs in the south bank of the creek forming a bluff about thirty nine feet in height. Here the creek has cut down about three feet into the basal shales. Above these the middle phase, which consists of alternating beds of sandy limestone, sandy shale, and bentonite make a total thickness of fourteen feet. The upper shales are more calcareous and weather to a light gray or yellow color; they are about twenty-one feet six inches in thickness. The

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east of Watters Station.

bluff is capped with about ten feet of Austin Chalk. Plate II shows a section taken at this place. About one mile below the foregoing section where the creek makes a sharp turn to the south, the shales are arched into a small anticline and several small faults ranging from throws of one foot to eight or ten feet are noted. Two wells were drilled and a pit was dug at this place several years ago. A small showing of oil was obtained which was supposed to have come from the Eagle Ford. The wells were soon abandoned as there was no commercial production. A well was drilled south of these two wells on the Byrd tract but there seems to be no available record of it. A quarter of a mile below the wells there is another exposure of about fifteen feet of the upper shales in the west bank of the creek overlain with about five feet of gravel of Pleistocene age. The Walnut Creek exposure is designated on the map as No. 2.

On Shoal Creek, about two hundred gards north of the crossing of the Camp Mabry road at Austin, the middle flag member of the formation is exposed in the east bluff of the creek. Here the Eagle Ford is found at the top of the cliff with the Buda underlying. Whether the basal blue shale is

Hill, R. T., The Oil Possibilities of Travis County, Texas: MMS. 1919.

faulted out, or the middle phase is lying unconformably on the Buda limestone has not been determined. The more probable cause is faulting. The beds consist of alternating layers of arenaceous limestone flags, from one to three inches in thickness, sandy shales, and bentonite. The thickness of the outcrop here is about ten feet. This exposure is designated as No. 3 on the map.

No. 4 on the map is the Shoal Creek exposure just north of the Sixth Street bridge. Hill⁸ gives this as a typical exposure resting upon the Buda limestone, but at the present time the banks of the creek are so eroded and covered with debris from the surrounding settlement that it is impossible to measure a section.

Outcrop No. 5 is the bext exposure in the county. Here the full section may be examined along the banks of Bouldin Creek. Plate No. V shows a plat of the type section on Bouldin Creek. The following section was taken at three different places on the creek. At (c) about eight feet of the upper basal shales are exposed. In order to take samples from the remainder of the basal portion, the writer dug a pit down to the contact with the Buda limestone. At point (d)

Hill, R. T., Geologic Atlas, No. 76, U. S. Geol. Survey, 1903.

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the middle phase was accessible, and at (f) the upper shales are all exposed and samples were taken. Plate VI represents a complete section measured along the creek.

The basal fifteen feet is a blue shale, dark on fresh exposures, but upon weathering is dull gray or brown. In places iron stain has colored the shales, and at times one may find the joint planes cemented with iron oxide. As a whole the shales are very homogeneous, but there are a few variations. Four feet below the fossiliferous ledge there is very hard shale, which when struck with a hammer will give a strong petroliferous odor. Just above this ledge is a layer of very soft, thinly laminated shales two and one half feet thick. The following is a detailed section of the basal member:

Inches

9.	Soft, thinly laminated blue-gray shales	
	with Globigerina cretacea, fish teeth and	
	scales, Inoceramus prisms, fossil wood,	
	pyrite, while about 50% of the washed sample	
	was arm joints of crinoids	30
8.	Extremely hard, gray shale; near the top of	-
	the ledge arm joints of crinoids abundant,	
	Inoceramus prisms, shell fragments, fish	
	teeth and bones, fossil wood, about 75% of	
	the washed sample was Globigerina cretacea;	
	at the bottom of the ledge were a few	
	Globigerina cretacea, and a few Inoceramus	
	prisms	24
7.	Hard gray laminated shale carrying arm joints	
	of crincids, while about 75% of the washed	
	sample was Globigerina cretacea	24

Inches

6. Blue laminated shale with small seams of sand; fish teeth and scales, Cristellaria sp., Inoceramus prisms, fossil wood, about 75% of the washed sample was Globigerina cretacea, pyrite -----24 5. Hard shale with fish teeth and scales, few arm joints of crinoids, Cristellaria sp., Inoceramus prisms, Echinoid spines, fossil wood, pyrite, about 50% of the washed sample was Globigerina cretacea ------12 4. Very hard gray shale, compact; arm joints of crinoids, Cristellaria sp. abundant, Inoceramus prisms coated with pyrite, fish 18 abundant, Globigerina cretacea, Anomalina ammonoides, Echinoid spines, fish teeth and scales, phosphate nodules, Inoceramus prisms, shell fragments replaced with pyrite, rounded and angular quartz crystals, about 50% of the washed sample was pyrite-- 12 2. Blue laminated shale with minute layers of sand; Anomaline ammonoides, Globigerina cretacea, fish teeth and scales with distinct crenulations, shell fragments, phosphate nodules, fossil wood, crystalline pyrite, rounded and angular quartz grains ------24 1. Soft, black, fine grained shale; Textularia globulosa, Globigerina cretacea, Cristellaria sp., Echinoid spines, fish teeth and scales, carbonized wood, shell fragments, phosphate nodules, selenite, about 75% of the washed sample was pyrite, some of which showed perfect octohedral crystals, quartz grains, about one half were well rounded while the remainder were angular -----12 Buda limestone ------ર Total thickness of the basal shales 15 feet.

At point (d) in plate V the middle series is well exposed and the entire section may be taken. Here the basal member is a fifteen inch ledge of arenaceous limestone with



Section taken from the type locality on Bouldin Creek one mile south of Austin.

a matrix of Ostrea shells forming the middle part. Above this the section is made up of alternating beds of sandy shale, sandstone, and bentonite. The following section was measured at point (d) in Plate V:

	Inches
27. Bentonite, verv impure, iron stained	1:
Inoceramus prisms. Textularia glob	u-
losa, fish teeth and scales, Globi	
gerina cretacea abundant	1
26. Hard shale layer, very calcareous, o	carry-
ing fish teeth and scales. Inocera	amus
prisms. Globigerina cretacea abuno	iant and
asll preserved	14
25. Bentonite stained slightly with iron	n oxide.
carrying small iron concretions.	selenite.
and quartz crystals. some angular	and
others rounded. The absence of fo	ossil
remains is striking	5
24. Sandstone ledge, forming bed of cree	ek above
the falls, Plate V	10
23. Sandy shale, the top part is soft an	nd
thinly laminated while the bottom	is
harder; fish remains very abundant	t, fossil
wood, Inoceramus prisms, and a few	v Globi-
gerina cretacea	2
22. Bentonite with Inoceramus prisms, f:	ish
șcales, fossil wood, Globigerina (cretacea
(few), iron oxide concretions	
21. Alternating layers of laminated san	dy shale
and sandstone flags	5
20. Sandy shale with fish remains, Inoc	eramus
prisms, and Globigerina cretacea.	The
bulk of the sample was sand	
19. Bentonite with a few fish remains,	fossil
wood, iron concretions, and selen:	lte 1
18. Sandy shale with fish scales, fossi.	
wood, inoceramus prisms, Globiger:	lna
cretacea, much sand	2
17. Calcareous sandstone; thin section s	3hows
iish remains, and Globigerina	3
Lo. Sandy shale with Globigerina cretace	ea not
well preserved, inoceramus prisms	, IOSS11
wood, 11sh remains, 1ron oxide, at	na
much sand	2

15.	Bentonite stained with iron; Inoceramus prisms, fossil wood, iron concretions.	
	Globigerina cretacea $\frac{1}{2}$	
14.	Hard sandstone ledge; thin section shows	
-	fish remains, and Globigerina 3	
13.	Bentonite considerably stained with iron;	
	Inoceramus prisms, Globigerina cretacea	
	(few), iron stained sand grains 2	
12.	Sandy shale, laminated, with Globigerina	
	cretacea well preserved, fish remains,	
	Inoceramus prisms, Iossil wood, much line	
	sana, and iron with iron. fich tooth	
11 ·	Bentonite stained with iron; iish teeth,	
	much iron and a second and the second	
10	Sandy shale thinly laminated forgil wood	
TO •	fich remains Inoceramis nrisms iron	
	Globigerina cretacea make up the bulk of	
	the sample 4	
9.	Bentonite: Globigerina cretacea. Inoceramus	
	prisms, iron 1	
8.	Sandy shale, laminated; Globigerina cretacea	
	not well preserved, yet abundant, fossil	
	wood, fish remains, Inoceramus prisms 11/2	
7.	Bentonite, iron stained; fragments of	
	Globigerina, fish scales, iron, Inoceramus	
1	prisms, and sand 1	
0.	Sandy shale carrying hard concretions 2 to	
	J to 2 feet long: also has a quanter inch	
	laver of hentonite. Incoeramia priama	
	fish remains Echinoid spines fossil	
	wood, selenite, Globigerina cretacea (few)- 5+	
5.	Bentonite with iron concretions, fish scales.	
	selenite, and sand $2\frac{1}{2}$	
4.	Sandy shale, laminated; Globigerina cretacea,	
	fish remains, fossil wood, Inoceramus	
-	prisms, pyrite, and sand 11	
3.	Lowest bentonite layer with selenite, fish	
	scales, iossil wood, iragments of Globi-	
2	gerina, inoceramus prisms, and iron 4	
2.	aretaces (few) fogeil wood Incoeramue	
	prisms, fish remains, purite, and much cond_ 4	
1.	Arenaceous limestone with agglomerate of	
~•	Ostrea about the middle of the ledge: thin	
	section shows fish teeth and scales, plant	
	remains, and many Inoceramus prisms 15	
	Total thickness of middle phase 8 ft.9 in	-
		•

The upper shales are well exposed at point (f) in plate V. Here the shales form a bluff about eighteen feet high with about ten feet of Austin Chalk at the summit. At the base of the cliff the hard blue laminated shales form the bed of the creek, but higher in the section the shales become more calcareous until at the top they are quite chalky. The Austin-Eagle Ford contact is clear cut. The shales are characterized by their numerous joint planes, and in some cases these planes are filled with iron oxide as a cement. Samples were taken continuously from the bottom to the top, and examined under the microscope. The following is a detailed description of the beds exposed just beneath the Austin Chalk on Bouldin Creek one mile south of Austin:

		Inches
12.	Austin Chalk	10 ft
11.	Sandy shale with clay and considerable	
	calcareous material. This layer is just	
	beneath the white chalk scarp, and may	
	be considered as the contact. Sample	
	contained Globigerina cretacea. Textu-	
	laria globulosa. Echinoid spines, sele-	
	nite, fish scales, Inoceramus prisms	
	and phosphate nodules according prisms,	20
10	Hard nodular limestone ledge thin	20
	section shows it to be of very fine	
	grained chalky texture with glauconite	
	grained charky texture with gradeonite	٨
Q	Sondy chale lominated and calconomy	4
7•	stained with increased and calcaleous,	
	(none) where he and les much also	
	(rare), phosphate houses, much clear	0
0	quartz	8
8.	Hard nodular limestone ledge; thin	
	section shows the material to be of	
•	very fine grained, chalky texture,	
	Globigerina	10

-					
T	20	-	5	0	~
1	r 1		TE	-	ы
-		-		-	-

7.	Soft calcareous clay with Globigerina cretacea (few), Textularia globulosa (few), pyrite, and much sand, some of which is stained with iron	48	
6.	Calcareous shale with iron concretions; sample shows Textularia globulosa (abundant), Globigerina cretacea, fish remains, Inoceramus prisms, selenite,	. 0	
5.	and sand grains Hard blue, laminated shale with Globigerina cretacea (abundant), Textularia globulosa (abundant), Inoceramus prisms, fish	48	
4.	remains Hard blue shale, laminated; Globigerina cretacea, Textularia globulosa, Inocera- mus prisms, pyrite, selenite, fish	12	
3.	remains	24	
2.	globulosa, and sand grains Hard blue shale forming bed of creek at the foot of the cliff; Globigerina cretacea,	24	
ı.	Textularia globulosa, fish scales, Inoceramus prisms, and sand grains Hard blue shale one foot above the top bentonite layer; Globigerina cretacea,	12	
	Textularia globulosa, Inoceramus prisms, fossil wood, fish remains, and sand	12	_
	Total thickness of upper a hales	18 ft.	6 in

The fact that the so-called arm joints of crinoids are found only in the basal portion of the section is very interesting. Whether the supposed crinoid lived only in that period or whether the conditions were not favorable for its preservation in the upper members is hard to determine. If these remains could be established as definite horizon markers in the Eagle Ford shales, their identity and place in the formation would readily be recognized as there have

been no such remains described from the other members of the formation, nor from the formations above or below the Eagle Ford.

Exposure No. 6 represents about twenty-five feet of the formation. In a shallow cut, along the Manchaca road just west of the railroad, as shown on the map, a portion of the middle flag member is exposed, but the outcrop is so poor that it was impossible to measure a section. Just how much of the formation and what portion of it is present at this point is difficult to say.

At point 7 on the map the middle phase is found in the bed off the creek between the road and railroad. Here the formation seems to be altered by an intrusion, although the intrusive rock does not make its appearance at the surface. The upper shales form the creek valley walls while the Austin Chalk forms the valley rim above.

Points 8 and 9 represent areas without any good exposures. In both places the basal shales overlie the Buda limestone as a thin mantle only a few feet in thickness.

From the above sections it may be seen that the Eagle Ford thickens northward, which is in keeping with its general tendency to increase toward Red River.

The following thicknesses recorded in well logs were obtained from the Bureau of Economic Geology and Technology



View showing the Middle phase, and a portion of the basal shales on Bouldin Creck one mile south of Austin. through the courtesy of Dr. E. H. Sellards:

Chapin well No. 1, located about three miles east of Austin on the Austin-Manor road --- 40 feet W. W. Childress well No. 1, located on the Antonio Navarro grant, two miles east of Garfield ----- 45 feet Manor well located about sixteen miles east of Austin ----- 25 feet J. J. Davis well No. 1, located four miles north of Manchaca ----- 67 feet The East Fifth Street well, Austin ----- 60 feet G. A. Parkinson's well in South Austin ----- 40 feet This well showed oil and gas from the Eagle Ford.

These logs are more or less inaccurate as it is difficult to distinguish the basal Austin Chalk from the upper Eagle Ford in well cuttings. According to these logs the formation shows a thickening toward the south which is contrary to the facts shown in the exposed sections.

FOSSIL ICE CRYSTALS

Another interesting characteristic of the middle phase of the Eagle Ford formation is the presence of fossil ice crystals.⁹ When walking along the bed of Bouldin Creek one may notice some strange markings on the sandstone flags. The layman often refers to them as bird tracks. They are small straight, shallow grooves from one-half to two inches

⁹Udden, J. A., Fossil Ice Crystals: Univ. of Texas, Bull. No. 1821, 1918.

in length, and from one-sixteenth to one-eighth inch in width. They are joined into patterns in which some spring out from the sides of others and again themselves send out other branches. Some cross each other. There seems to be a rather uniform angle of divergence in the branches, which is about sixty degrees. A photograph of these strange markings is shown in plate VIII.

There is no particular significance attached to these markings other than they may serve as an index to the climate of that period.

CONCRETIONS IN THE EAGLE FORD FORMATION

One of the most striking features of the Eagle Ford formation is the presence of numerous and varied concretions. These forms occur throughout the formation. They assume almost all sizes and shapes; some are microscopic while others weigh a ton. Some resemble dumb-bells, clubs, or bombs, while a very common type is the septarium, which is a globose, or flattened, disk shaped concretion from five to fifteen inches in diameter. Because of the intricate polygonal cracking over the surface, the septarium bears a striking resemblance to a turtle's back and is often so mis-



Fossil ice crystals shown on the lower left hand corner of the sandstone flag. Specimen taken from Bouldin Creek.

taken by the layman.¹⁰ The various types of concretions occur in greater abundance north of the Brazos River, the outcrops in Dallas and Grayson Counties being especially characterized by them. South of the Brazos they are fewer in number and do not take on such a variety of shapes. In the Austin section the typical septarium is absent. In its stead there is a form which usually resembles a flattened log. Plate IX shows one of these concretions before and after it was split open. Some of the specimens are shorter and more spheroidal. Very good examples may be found in the middle phase of the formation on Bouldin Creek, and also on Walnut Creek a quarter of a mile east of Watters Station. When the upper and basa; shales are examined many small iron concretions are found. They range in size from less than one twenty fifth to one half of an inch in diameter. A few of the very small ones present perfect crystalline forms; the marcasite nodes showing the radiating structure on the inside, while the exterior resembles colitic hematite in form.

The larger concretions appear to be the result of the deposition of mineral matter from solution. Calcium carbonate, or iron sulphide, may be deposited around some

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Shuler, E. W., The Geology of Dallas County: Univ. of Texas, Bull. No. 1818, p. 17, 1918.

foreign object as a nucleus, and the concretion grows by the constant deposition on the out side. In larger concretions, as shown in plate IX, the nucleus seems to have been a piece of wood replaced by calcite. In some cases the nucleus is a fossil, while at other times it may be a grain of sand. In many instances when the concretion is broken open no nucleus is found, but instead a hollow space with a dense growth of perfect calcite crystals growing around the interior walls with the crystals pointing inward.

BENTONITE

The middle phase of the Eagle Ford formation is characterized by a series of rather regularly spaced bentonite layers, which are of very wide distribution. Adkins¹¹ describes layers of bentonite in the middle flags of McLennan County. Udden reports them from Brewster County. and Sellards has found them in the formation in Mexico.

Bentonite is a volcanic ash composed chiefly of aluminum silicate associated with several other minerals. The chief, and most important mineral, is leverrierite. Bentonite swells in water and breaks up into a doughy mass.

Adkins, W. S., Geology and Mineral Resources of McLennan County: Univ. of Texas, Bull. No. 2340, p. 71,1923.


A. Concretion obtained from the middle phase of the Eagle Ford formation on Walnut Creek a quarter of a mile cast of Watters Station.



B. The same concretion after it has been broken open.

It is distinctly crystalline and in plates, some of which are a millimeter across. It is soft, sectile, and clay like, but some of the aggregates of the larger plates are more indurated and soften slowly in water. It has an index of refraction, B, of about 1.57, a birefringence of about 0.02, a very small axial angle, and is optically negative in character. The acute bisectrix is sensibly normal to the plates.¹² Under the microscope the ground mass of typical bentonite is an aggregate of crystalline material. The texture varies greatly and in some specimens it is almost submicroscopic, but in others it is so coarse that the rock resembles a sericite schist. Bentonite may have almost any color, and shades of gray, brown, pink, yellow, green, and white are common. The layers found in the Eagle Ford formation do not present such a variety of colors. Here the white bentonite has been stained with iron to such an extent that it is dark yellow on exposure, but when fresh is light yellow or brown.

The source of this bentonite is not known, but since it is of volcanic origin it is thought to have come from the Rocky Mountain region.

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Nelson, W. A., Tenn. Geol. Sur., Bull., No. 25, p. 46, 1920.

The bentonite from Bouldin Creek is considerably stained with iron oxide. There are twelve layers ranging from one-half to five inches in thickness, the bottom and the second from the top layers being the thickest. All layers show sand grains, and all except the second from the top layer show fossils including fish remains. The layer which lies just above the ten inch sandstone ledge contains some glass sand and iron concretions. These facts would seem to indicate that the volcanic ash fell into a very shallow sea.

The correlation of bentonite beds in different parts of the state should be made. If the beds of the Travis County Eagle Ford shales could be correlated with those of McLennan County, then it would be possible to determine what part of the formation thickened, or thinned in different parts of the state.

Bentonite was first described by W. C. Knight under the name of taylorite.¹³ Finding that the latter term was preempted, he proposed as a substitute the name bentonite,¹⁴ from its occurrence in the Benton formation of Wyoming.

> 13 Eng. and Min. Journ., LXIII, p. 600, 1898.
> 14
> <u>Ibid.</u>, LXIII, p. 491, 1898.

It may be seen from the analyses below that bentonite shows a somewhat variable composition, and that its peculiar properties are due no doubt to physical rather than chemical conditions.

Analyses of Bentonite										
relarder	l	2	. 3	4	5	6	7			
SiO ₂ Al ₂ O ₃ Fe ₂ O ₃ FeO CaO MgO Na ₂ O M_2O Loss on Ignition H_2O	54.00 24.48 1.47 0.45 2.08 2.75 1.74 tr 9.68	60.18 26.58 0.23 1.01 1.23	64.00 22.90 3.10 1.00 2.00 7.00	47.56 20.57 8.58 0.24 2.52 0.80 1.28 17.60	63:40 19.54 4.26 0.31 2.66 0.74 9.12	71.48 14.82 3.89 1.97 2.14 0.40 5.19	51.85 27.05 2.75 2.82 0.97 2.62 10.89			
Total	96.65	99.49	100.00	99.15	100.03	99.89	98.95			

1. Bentonite from Bedford County, Tenn. Ordovician in age. Analysis by J. I. D. Hinds.

- 2. Bentonite, Laramie Basin, Wyoming. Analysis by Thomas T. Read. Quoted Darton, N.H. and Siebenthal, C. E.: Geol. and Mineral Resources of the Laramie Basin, Wyo. Bull. U.S.Gecl.Survey, 364, 59, 1909. 3. Bentonite, Laramie Basin, Wyo. Analysis by John Ogden, quoted as above. 4. Leverrierite from Beidel, Colo. Analysis by E.T. Wherry. J. Wash. Acad. Sci. VII, 213, 1917.
- 5. Bentonite from Colorado County, Texas. Tertiary in age. Analysis by J.E. Stullken of the Industrial Chemistry Division of the Bureau of Economic Geology and Technology, University of Texas, 1920. 6. Bentonite from a well drilled at West Columbia, Brazoria
- Co. Texas. Tertiary. Analysis by J. E. Stullken.
- 7. Bentonite from the Eagle Ford formation on Bouldin Creek one mile south of Austin. Analysis by L. F. Marek, Dept. of Chemistry, University of Texas, May, 1925.

Bentonite has been used in the manufacture of soap, as a packing for a special kind of horseshoe, as a diluent for certain powerful drugs sold in the powdered form, and as an adulterant of candy. It has also been employed in the manufacture of antiphlogistine, and makes a good retarder for cement plasters. It is used on a commercial basis for the softening of water, but its chief use so far is for weighting and filling paper. When used on a commercial basis bentonite will bring six to seven dollars per ton f.o.b.¹⁵

The Travis County bentonite is not in sufficient quantities to be worked on a commercial basis, as the thickest bed is only five inches thick. On account of the rather impure condition of the clay and the highly stained material it would be difficult to prepare it for use, even though it were in greater quanties.

15 Ries, H., Clays, their Occurrence, Properties and Uses: John Wiley and Sons, 1914.

PETROLEUM IN THE EAGLE FORD FORMATION

It has been known for some time that the Eagle Ford formation produced oil in small quantities, but just to what extent the formation is a producer has not been deter-Several water wells in Travis County have a small mined. oil production. The wells drilled on Walnut Creek about one mile east of Watters Station have produced a little oil which is thought to have come from the Eagle Ford formation. At this same place on Walnut Creek oil and asphalt seeps have been noted. Here the shales are exposed in the north bank of the creek just beneath the Austin Chalk. In this same locality pits were dug some twenty years ago for the purpose of mining oil, and during a wet season a small amount of oil will accumulate on the surface of the water, but the guantity is not sufficient for commercial purposes. From the strong petroliferous odor given off it was thought that the shales might possibly be a source of oil when sufficiently covered, and protected from weathering. Due to the above facts several laboratory experiments were conducted to determine the approximate amount of oil, fixed gases, and ammonium sulphate contained in the Eagle Ford shales.

The method of retoring and laboratory procedure was similar to that used in the laboratory of the Colorado

School of Mines.

LABORATORY PROCEDURE

The type of retort used in the laboratory investigations is an iron mercury retort with capacity of four pints. The lid of the retort fits tightly on the rim and is rendered air tight by using a thick paste made by mixing "Smooth On" cement (Engineer's "Smooth On", No. 1) with a little water. By using a spatula this paste is quickly and evenly spread on the junction surfaces of the lid and retort. The lid is immediately placed on and the clamp firmly screwed down with the aid of pliers. The retort is then placed in the top of the fire-box made of fire-brick, and a No. four Scimatco burner is placed in the opening of the fire-box at the bottom. An asbestos board eighteen inches square is used at the top to keep heat from passing out around the top of the retort. The lid is equipped with a half-inch pipe three inches long extending down into the shale. This pipe is sealed at the lower end and holds the thermometer. The lid also has a quarter inch pipe six inches long extending three inches into the shale for the purpose of admitting

> 16 Alderson, Victor C., The Oil Shale Industry, 1920.

steam from the boiler. This steam pipe is perforated so that the steam may be distributed through the shale. The delivery tube is made of three-quarter inch pipe, and comes out at the center of the lid. Heat is supplied by means of one, two, or three Scimatco burners as needed to increase the temperature.

The condenser used is divided into two parts; the first section, connected to the delivery tube, is two feet long. The inner tube is made of quarter inch brass pipe and screws into the delivery tube. The outer cylinder is one and a half inches in diameter, and is composed of brass. The second section is three feet long, with an inner tube of onequarter inch glass tubing and an outer brass tube two inches in diameter. The inner tubes are connected by means of rubber tubing, and the outer tubes are also joined by the same method. The condenser is attached to the hydrant by means of rubber tubing. Circulation of the water is started before the gasses begin coming over.

DESCRIPTION OF THE SHALE USED

The shale used in the retorting tests came from the section on Bouldin Creek, one mile south of Austin. The shale was taken from the bottom phase of the section and is typical of the shales of the bottom member of Travis County.

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Apparatus used in the retorting of the Eagle Ford shales.

1. Steam generator 2. Thermometer

- 3. Fire box
- 4. Retort
- 5. Scimatco burners
- 6. Bunsen burner
- 7. Delivery tube 8. Condensers

 - 9. Outlet hose for condenser
 - 10. Intake hose for condenser
 - 11. Steam intake pipe
 - 12. Separatory funnel
 - 13. Distilled water scrubber for taking out emulsified oil
- 14. Meyer tube
- 15. Carboy 16. Water jar for catching over flow from carboy
- 17. Jar to catch gas for analysis
- 13. Water bottle for over flow from gas jar
- 19. Pinchcocks
- 20. Shale crusher

The weathered surfaces are of a bluish gray to brownish black color and in some instances have a silky luster. Freshly broken surfaces are black to gray. The shale is laminated and tends to fracture readily along the planes of lamination as is characteristic of most shales, the fractures being conchoidal. Small concretions of pyrite are found throughout the bottom phase of the shale, and upon retorting leave a thin coating of altered pyrite on the bottom of the retort. The samples used in the experiments were taken all along the section from top to bottom and mixed well so that a representative sample might be obtained.

PREPARATION OF SHALE FOR RETORTING

A small jaw crusher was used in crushing the shale to the desired size. Two sizes were prepared, the larger composed of fragments passing through a half inch mesh but not through a quarter-inch mesh sieve. This was termed "large size" shale and recorded as such in the data. The smaller size was crushed so that it passed through a quarter inch mesh, a considerable portion of the sample being powdered. This was recorded as "small size" shale in the data.

AMOUNT OF CHARGE AND TEMPERATURE

In all tests, except sample No. 3, one and a half kilograms of crushed shale were used. In the case of sample No. 3 the charge was the spent shale from sample No. 2 crushed so that it would pass through a quarter inch mesh sieve. In the other six samples the charge seemed to be too great for the method of retorting. When the spent shale was removed from the retort there was a small portion of the fragments in the bottom and on the sides of the retort showing complete distillation as they were white or grayish white in color while the material near the center of the charge was coal black. This, however, could be due to carbon formed in the deposition of carbon on the fragments of spent shale as there is considerable carbon formed in the distillation process. In many cases when digging the shale, pure carbon was found.

The temperature was carried as high as 750 degrees Fahr. using two Scimatco burners. The temperature was increased rapidly at first and gradually decreased toward the end of distillation. From the data shown in table No. 1 it can be seen that the rate of increase of temperature from hour to hour had nothing to do with the final results of the experiment.

				and a second s		
Rate	of	Increase	in	Temperature	per	Minute

TABLE I

Sample	lst.Hour	2nd .Hour	3rd.Hour	4th.Hour		Sp.Gr.B.	Gas per ton			
No. 1	60	3.33°	10	•3°	22 cc		1330 cu. ft.			
No. 2	7.33°	2.53°	1.3°		17 cc		2141 " "			
No. 4	4.63°	4.14°	1.110	• 80.	21.5 "	18.2	2141 " "			
No. 5	4.7°	2.50	•37°		24 "	17.09				
No. 6	4.80	2.5°	1.66°	.88°	28 "	17.8				
No. 7	6.76°	3.00	•72°	.25°	27 "	17.2	2352 " "			
Average	5.83°	3.00	1.26°		23.2 "	Concestion of the				
No. 3	8.54°	2.53°	1.3°	•3°	trace		363 " "			

From a study of the data tabulated in table No. 2 it may be seen that there is a difference in the amount of oil obtained from the "large size" shale and the "small size" shale. In each case there was a greater yield from the smaller size. Whether this difference is due to the size of the fragments or the method of retorting, that is, by the steam or dry method, is not easy to determine. It is the opinion of the writer, however, that the size of the shale in this particular case is the determining factor. The "small size" gave a greater yield than the "large size" when distilled under similar conditions, with one exception when the "small size" was heated for thirty minutes longer. This should not make any material difference, however, as most of the oil comes off below 600 degrees Fahr. These results do not agree with those obtained by Stewart and Trenchard of the University of California in their experiments on oil shales from Elko, Nevada. In their experiments the larger fragments gave a greater recovery of oil. The

following is taken from their discussion of results:¹⁷

"The yield increases with the size of the shale under similar conditions of heating because of the fact that the vapors are cracked in the retort. Vapors at the center of the lumps are

¹Stewart and Trenchard, The Destructive Distillation of Oil Shale: Quarterly of the Colorado School of Mines, July, 1923.

retarded in emerging to the surface on account of the distance to be traversed by the vapors in the thicker pieces of shale and thus the resistance offered by the shale itself. In the case of the large size shale the temperature on the outside of the pieces of shale may be 400 degrees Fahr. while on the inside would be only about 250 degrees Fahr. Now by the time distillation has been completed on the surface of the lump and the temperature has risen to 700 degrees Fahr., for instance, the temperature on the inside of the lump may just be sufficient to cause distillation. Therefore these vapors which are formed on the inside of the lumps will, on reaching the surface of the shale, be subjected to a temperature of 700 degrees Fahr, which causes them to crack forming vapors composed of lighter molecular structure. In the case of the smaller size shale, resistance is offered by the shale and the difference in temperature would not be so great and hence cracking would not occur to as large an extent."

From the data on the experiments carried on, it seems that all of the hydrocarbons were not driven off when the large size shale was used. Consider sample No. 2, which was of the "large size" and note the results as compared with those obtained on sample No. 1. Sample No. 3 was the spent shale from sample No. 2. In this case a few drops of oil were given off with a little water, and hydrocarbons in the form of gases which about make up for the difference between the recovery of the different sizes of This is not conclusive evidence, however, that the shale. smaller particles will give up more oil than the larger Furthermore, the results obtained by Stewart particles. and Trenchard can hardly be compared with those obtained in

TABLE II	3

Sample	Size	Oil cc.	Gr.B.	% Unsat.	Gal. per ton	(NH4)2SO4 per ton	Method	H20 c.c.	Time	Temp.	Loss in di	of wt. st.
No. 1	Small	22			3.52		Dry		3:55	686 F.	-	
No. 2	Large	17	Caracteria - Caracteria da Caracteria - Cara		2.72	2.57 lb.	Dry	170	3:25	750 F.	300	gms.
No. 4	Large	21.5	18.2	an a	3.44	3.24 "	Dry	158	3:20	640 F.	340	#
No. 5	Large	24	17.09		3.84	Constanting on Constanting of the	Steam	annguagaa	3:30	565 F.	242	11
No. 6	Small	28	17.8	I. No. 83.59 Br.No. 52.62	4.48	2.99	Dry	70	3:45	640 F.	210	11
No. 7	Small	27	17.2	Statistican direction	4.32	3.09	Steam	57	4:15	650 F.	210	11
No. 3	Small	tr.			Cite Grand providers		Dry	tr.	2:45	667 F.	32	Ħ

Sample No. 3 consisted of the spent shale from sample No. 2 which had been crushed to the smaller size.

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this laboratory because the shales of this locality and those of Nevada are very dissimilar, and besides the methods of retorting were different, and it has been proved several times in the laboratory of the Colorado School of Mines that the method of retorting will change the results of distillation.

Since the results obtained by either the dry or steam method of retorting were not always consistent, no definite conclusion can be drawn. In the case of the "large size" shale there was a greater recovery when steam was used, but in the case of the "small size" one sample distilled dry gave more oil than when another sample under similar conditions was distilled using steam. It seems probable that the steam was more effective in the "large size" because it tends to equalize the temperature there by preventing the formation of places that are too hot or too cool. Moist heat is more penetrating than dry heat. Therefore, when using the steam method the larger particles will give up their hydrocarbons at a lower temperature than when retorted dry. It is also thought by Dr. Victor C. Alderson and others that the use of steam in retorting diminishes the production of unsaturates in the oil. Only one sample from this series was tested for unsaturates so no comparison can be made between the percentage of unsaturates when the

dry method is used and that obtained when steam is injected. Sample No. 6 was distilled dry, and tested for unsaturates which were found to be rather high according to table No. 2.

The percentage of ammonium sulphate obtained is comparatively low when compared with other American shales, Some of the Utah shales producing as much as thirty pounds per ton of shale. It could not be determined whether the steam increased the amount of ammonium sulphate in the samples run by the writer. In one case, however, where small shale was used there was a greater yield when steam was injected. According to Alderson of the Colorado School of Mines injected steam should increase the amount of ammonium sulphate per unit of shale. Dr. Alderson says:

> "Nitrogen, in combination, exists in oil shale in appreciable quantity, perhaps as complex ammonia compounds or otherwise. During the distillation the ammonia compounds may give off ammonia, either free or in combination. Free nitrogen and hydrogen, at the moment of their production, may unite to form ammonia. One recent investigator claims his experiments show that practically all the available ammonia may be obtained without the use of steam if proper temperature conditions are observed. According to his tests the most favorable temperature for obtaining the maximum yield of ammonia is about 735 degrees Fahr. If the ammonia once formed is subjected to a further increase of temperature, it is liable to be more or less decomposed into its constituents, nitrogen and hydrogen. It will begin to decompose at a little below 940 degrees Fahr., and at about 1440 degrees Fahr. the decomposition is complete. Where the temperature is not otherwise controlled, it is thus possible that if steam is used it again acts as an equalizer. By preventing excessive cracking of the

oil vapors it may also prevent the occurrence of the conditions under which the undesirable nitrogen compounds are formed, at the same time preventing the decomposition of any ammonia once formed. It is difficult to see how steam can aid in the formation of ammonia during the distillation of the oil by any chemical interaction." (Oil Shale Industry, 1920).

The condensable portion of the distillate is caught in the separatory funnel, while permanent gases pass through distilled water which acts as a scrubber, taking out a part of the emulsified oil. The gases then bubble through a solution of approximately one-normal sulphuric acid which removes the ammonia and then collects over water in the carboy as shown in plate X.

After distillation is complete, steam is sent through the retort, condenser, separatory funnel, and Meyer tube so that all parts are thoroughly rinsed until free of ammonium compounds and the rinsings, with the water condensed in the separatory funnel, added to the solution of sulphuric acid. This solution is then neutralized with sodium hydroxide and the ammonium sulphate in the neutral solution changed to sulphuric acid and hexamethylenetetramine by boiling one minute with neutral forty per cent formaldehyde. The solution is cooled to about 60 degrees C., phenolphthalein added and the sulphuric acid formed titrated with the standard sodium hydroxide. Each

cubic centimeter of fifth-normal sodium hydroxide is equivalent to 0.017613 pound of ammonium sulphate per ton of shale when 1500 grams are used for the distillation.

The liquid in the separatory funnel is carefully separated and measured. Each cubic centimeter represents 0.16 gallon of oil or water per ton of shale when 1500 grams are used for the distillation. The volume of water flowing from the carboy is equal to the volume of permanent gases. This volume is cubic centimeters times 0.0213 is equal to the yield of permanent gases in cubic feet per ton of shale when 1500 grams are used for the distillation.

The oil from the several distillations was collected in a flask and distilled dry, using the Standard Engler End Point Apparatus. The following table shows the results of this distillation.

Fractions	Temperature	Gr. Baume	Per cent.
Water			1.0%
Gasoline	410° F.	34.2	20.0%
Kerosene	410 572° F.	24.4	36.5 %
Fuel oil	572 680° F.	16.5	33.2 %

Above 680 degrees Fahr., or after all fuel oil came off, the residue began to decompose with copious noncondensible fumes. The residue was a hard, black semi-

solid, and was not soluble in benzol, but was soluble in pyridine. The liquid part was soluble in benzol.

The gasoline and kerosene fractions from the above distillation were taken and distilled together. The fractions were collected in four parts as shown in the following table:

Temperature	Amount c.c.	per cent	Description of fraction
210° C.	15.8	27.96	Light yellow, chang- ing to deep red color on standing
210 300° C.	25.1	44.42	Orange or medium red color deepened on standing
300 320°C.	5.7	10.08	Red, did not change color
Small amount of heavy lubricati	residue remand ng oil, deep	ained in the red color.	e flask, resembling

The work done in the laboratory on the Eagle Ford shales was thorough enough to draw definite conclusions. The method of retorting was of the simplest type. The kind of retort used in any distillation is the most important factor. Let us consider what might be expected to happen if oil shale is distilled in a retort such as was used in this laboratory. Let the retort be almost full of crushed shale and a strong heat be applied at the bottom and all around the lower outside. The shale next to the walls distills while the interior portions and top are still comparatively cool. The outside shale will become carbonized and spent, shrinking somewhat from the walls, while the interior portions continue distilling. The vapors from the interior portions will seek the easiest channels of escape, which are through the hot spent shale and up against the hot walls through the shrinkage space. The conditions are thus seen to be unfavorable for producing the maximum yield of oil. Unsaturates and gas will be formed in undue amount, at the expense of good oil.

It was not the purpose in the beginning to determine the exact amount of oil, gas, and ammonium sulphate contained in the shales, but to determine whether or not they could be called "oil shales" and mined and retorted as such on a commercial basis. It is the opinion of the writer that the Eagle Ford shales do not contain sufficient oil to be worked on a commercial basis.

From the results obtained in the experiments it seems that the amount and quality of gas might justify further investigation. The gas represents the uncondensed portions of the vapors. Its composition varies with the nature of the material retorted, temperature of distillation, and the efficiency and nature of condensers. The following gas analysis of an oil shale is given in the "Jour. Soc. Ch. Ind.," 1897, p. 983:

Carbon dioxide	22.08%
Oxygen	1.18%
Heavy hydrocarbons	1.38%
Garbon monoxide	9.77%
Methane	3.70%
Hydrogen	55.56%
Nitrogen	6.33%

It will be seen that the above analysis does not differ widely from those obtained from the gas of the Eagle Ford shales.

The following discussion is given by Mr. L. F. Marek, of the Department of Chemistry, of the University of Texas, by whom the analyses were made:

This shale oil gas offers several contrasting points when compared with other commercial gases. First it is unusually high in hydrogen having an amount that is approximated only by water gas such as produced in the Loomis-Pettibone producer. It is low in methane as are most artificially produced gases. In contrast to its methane content we may take natural gas which usually has as much as 90 per cent of this constituent. The gas runs much higher in carbon dioxide than the ordinary gases indicating probably that incipient combustion of some of the hydrocarbons had taken place during the distillation. Ordinary producer gas is characterized by a high nitrogen content, having as much as 50 to 60 per cent of this constituent, whereas shale oil gas will average a nitrogen content of 14 TABLE III

Gas Analysis

Sample	Cu. ft. per ton	Temperature Gas collected	c02	Ole- fins	02	H2	CO	^C 2 ^H 6	CH4
No. 1	1330	212 686	27.1	3.2	1.7	29.00	3.4	1.7	3.4
No. 2	2141	212 750	29.0	2.5	.6	37.2	7.8	3.4	5.5
No. 4	2141	212 640	30.8	2.3	2.6	28.00	11.1	4.6	4.1
No. 5	No	data		3			10 - 10 - 10 - 10 - 10 - 10 - 10 - 10 -	10 - 10 A	10 - 10 - 10 - 10 - 10 - 10 - 10 - 10 -
No. 6a		500 600	40.0	2.7	.1	32.5	13.8	1.6	4.6
No. 6b		640 640	34.9	.5	.7	43.9	10.8	1.6	1.5
No. 3	363	212 667	31.2	.2	2.6	55.9	1.5		
No. 6a No. 6b No. 3	363	500 600 640 640 212 667	40.0 34.9 31.2	2.7 .5 .2	.1 .7 2.6	32.5 43.9 55.9	13.8 10.8 1.5	1.6	4.6

per cent. This nitrogen and carbon dioxide serve as dilutents and add nothing to the heat value of a gas. They are, however, constituents sometimes desirable in power work due to their flame retarding capacity. The illuminants, that is the unsaturated gases known as olefins, are two or three times as high in the shale oil gas as in ordinary producer gas and might cause trouble due to their tendency to produce a luminous, smoky flame. The oxygen content is low as it should be and is probably due to the air in the retort and in the shale itself.

The characteristic and extremely disagreeable odor of the gas is probably due to the products resulting from cracking in the distillation process. The temperature during the distillation runs fairly high and cracking of the petroleum is almost sure to occur especially if the distillation is carried out dry. The unsaturated constituents in the gas give an odor. The high sulphur content of the oil, over three per cent, would possibly give a high sulphur content in the gas. Sulphur compounds usually possess a disagreeable odor when found in oils and such compounds would also add to the odor of the gas. The gas obtained from the Eagle Ford possesses the following special characteristics:

For use as power:

1. Too snappy (high in hydrogen) for gas engine work.

2. Should not produce excessive carbon deposits.

3. Could be mixed with natural gas which is rich in methane and makes an ideal power gas, being rich, pure, and rather slow burning.

4. Coming from a source rich in sulphur, the gas is rich in sulphur which on burning goes to sulphur dioxide which will do no harm while the engine is running, but when the engine is stopped reacts with the water formed from the burning of the hydrogen to form sulphurous acid which corrodes the cylinders.

As an illuminating gas:

1. Heat value too high (rich in hydrogen).

2. Flame would be luminous but a great deal of heat would be wasted.

3. Small amount of unsaturates would not be likely to give a smoky flame.

4. Low in methane, which is a very good illuminating gas. For heat production:

1. Relative low heat value as compared with the standard value of 575 B. T. U. per cubic foot as adapted by seventeen states in 1917.

2. Relatively little smoke producing constituents.

3. Should burn with a clean flame.

4. Large hydrogen content lowers the heating value somewhat, due to the formation of much water vapor which is not condensed where the heat is utilized.

Average gas analysis on 100 cu. ft. of gas for combustibles.

Methane	3.6	х	1010 eq	uals	3635					
Ethane	2.4	х	1764 -	11	4240					
CO	8.1	х	323	11	2609					
Hydrogen	37.8	х	326	Ð	12340					
Olefins	1.9	х	1590	11	3020					
					25844	В.	T.	U.	per	100
cu ft	ase st	60) degrees	C	and	20	ina	of		~^11 ~ v

cu. ft. gas at 60 degrees C., and 30 ins. of mercury pressure.

This compares with other gas values as follows:

Natural gas (ave.)	equals 1000 B.T.U. per cu.ft	
Blast furnace gas	" 90	
Producer gas	" 145	
Water gas	" 300	
Ether	" 3264	
Shale oil gas	" 258.5	
at 60 degrees C. and 30 i	ins. of mercury pressure.	

CONCLUSIONS

(a) The "small size" shale produced more oil than the larger particles.

(b) The use of steam with the "large size" shale gave a greater recovery than when distilled dry.

(c) The specific gravity was higher when distilled dry.

(d) The rate of increase of temperature apparently had nothing to do with the results of distillation.

(e) The greater bulk of oil can be extracted below 600° F.

(f) The use of steam increases the amount of ammonium sulphate.

(g) The amount of permanent gases per ton of shale is just a little less than the average amount for the Colorado shales.

BIBLIOGRAPHY

Adkins, W. S.: Geology and Mineral Resources of McLennan County; University of Texas, Bull. No. 2340, 1923.

Alderson, Victor C.: The Oil Shale Industry, 1920.

- Bailey, T. L.: The Geology and Natural Resources of Colorado County; University of Texas, Bull. No. 2333, 1923.
- Christner, D. D. and Wheeler, O. C.: The Geology of Terrell County; University of Texas, Bull. No. 1819, 1918.
- Hill, R. T.: The Topography and Geology of the Cross Timbers and Surrounding Region in Northern Texas; Am. Jour. Sci., Vol. 33, p. 298, 1887.
- Hill, R. T.: Geography and Geology of the Black and Grand Prairies, Texas; U. S. Geol. Survey, twenty-first Ann. Rept., pt. 7, pp. 323-328, 1901.
- Hill, R. T.: The Oil Possibilities of Travis County, Texas; MMS. 1919.
- Hill, R. T. and Vaughn, Wayland T.: The Austin Folio, No. 76, U. S. Geol. Survey, 1902.
- Knight, W. C.: Description of Bentonite; Eng. and Min. Jour., LXIII, 1898.
- Liddle, R. A.: The Geology and Mineral Resources of Medina County; University of Texas, Bull. No. 1860, 1918.

Nelson, W. A.: Tenn. Geol. Survey, Bull. No. 25, 1920.

Paige, Sidney: Llano-Burnet Folio, No. 183, U. S. Geol. Survey, 1912. PLATE XI



PLATE XII



PLATE XIII

PLATE XIII

Figures 1, 2, 3. Fish teeth, natural size Figures 4, 5, 6. Fish teeth, x35. Figures 7, 8, 9. Fish scales, x35.


PLATE XIV

PLATE XIV

- Figures 1, 2. Textularia globulosa Ehrenberg, x35. different individuals.
- Figure 3. Cristellaria (?) sp., x35.
- Figure 4. Globigerina cretacea D'Orbigny, x35. a. ventral view; b. dorsal view.



