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RESULTS OF ASSAYS OF THE NEW ALBANY OIL-SHALE.

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This is the fourth of a series of papers on an investigation of Indiana oil-shales conducted by Indiana University and the Department of Conservation, State of Indiana, in cooperation with the U. S. Bureau of Mines, under the direction of Martin J. Gavin, oil-shale technologist of the Bureau.

Introduction.

The data presented in this paper form the chief part of a general survey of the oil-shale resources of Indiana. Other papers* have already been published which contain data on various aspects of this resource. In combination, these papers present data with reference to the following points; (1) distribution of the New Albany shale; (2) amount of shale available; (3) homogeneity and thickness of the formation; (4) amount and quality of the oil obtained from the shale; (5) nitrogen content of the shale; (6) extractibility of the shale and its amenity to breaking; (7) some economic factors bearing upon the utilization of the shale; and (8) other miscellaneous factors and data.

In the future, technical work on other phases of the general problem is expected to be done with the object of making a complete survey of this resource which is of much potential importance to the state and nation.

Research and field work on the oil shales of Indiana have been conducted by the author since 1920. When the cooperative arrangement for the investigation of the shales was made with the Department of the Interior, Bureau of Mines, it was decided to make a systematic collection of samples from the outcrop district and to study them afterwards in the laboratory. This systematic collection of samples was made during the summer of 1922 by the author, assisted by Mr. Robert L. Kidd.

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*Reeves, John R., Oil-shales of Indiana, Eng. and Min. Jour., Nov. 13, 1920;
The New Albany shale of Indiana, Bureau of Mines, Reports of Investigations, Serial No. 2390, August 1922, 8 pp.;
Preliminary report on the oil-shales of Indiana, Handbook of Indiana Geology, Dept. of Conservation, State of Indiana, 1922, pp. 1059-1130;
A section through the New Albany shale, Bureau of Mines, Serial 2425, December 1922, 5 pp.;
An Economic Study of the New Albany shale, Bureau of Mines, Serial No. 2466, April, 1923.

Methods of Sampling.

Only the fresh unweathered shale was taken in the collection of the samples as it had been found by previous work, that there is a difference in the yield of oil between partly weathered and unweathered shale. The fresh shale is also representative of the true value of the formation.

The selection of each sample was made with reference to the following points; 1- the general areal distribution; because it was desired to have the samples as nearly as possible representative of the outcrop district as a whole; 2- the position of the sample in the vertical section of the formation, as it was desired to sample all parts of the formation; and 3- the ease with which a proper sample could be taken.

For the following reasons the numerous quarries in the formation from which the shale is taken for road building purposes were found to be the best points for collecting samples; (1) the very freshest shale was available; (2) the sample was easily collected, and (3) there was a better opportunity to study the true physical aspects of the rock. More than half the samples listed were taken from quarries.

When the samples were not taken from quarries, the first step in the collection was the clearing away of the face. This was done with a shovel, pick, and oftentimes dynamite. After the face had been cleared off and the fresh shale exposed, a trench was dug into the shale, from which equal portions of the section represented were chipped off. The sample of shale was collected on a burlap blanket, halved, quartered, and then boxed.

Representation of the Samples.

Each sample collected represents a certain vertical portion of the formation. It has been the general practice in the oil-shale region of the mid-eastern States, as probably in the west, to make selective collections of samples; that is, a portion of the richest part of an outcrop would be taken as a sample, the leaner portion of the outcrop not being collected. This practice is misleading as to the value of the formation at the locality at which it is sampled and is as impracticable as using one piece of coal from a six foot vein for a representative sample. The oil yields of several samples listed in this paper are low because of the presence of very lean shale in the section of the formation where the sample was collected.

The sections of the formation represented by the samples collected, vary from 2 to 18 feet. The sum of the sections is about two and one-half times the total thickness of the formation which is approximately 100 feet.

The position of the sample in the formation was determined wherever possible. In many places, however, it was impossible to determine this because of the gentle dip of the rocks, the thickness of the formation, and the lack of recognizable horizons within the formation.

The location of the samples are well distributed over the outcrop district. Each sample represents an area of about 10 square miles.

Experimental Procedure.

The apparatus and methods used in the examination of the oil shale and the shale oil are the same as those used by the Bureau of Mines in its various oil-shale laboratories. The apparatus and methods are described in Bulletin 210 of the Bureau of Mines.*

Distillations of the crude shale oil were conducted at an average barometric pressure of 742 mm.

The following tests were made on each sample; oil yield, water yield, specific gravity of crude oil, distillation of crude oil (atmospheric distillation ending at 275 degrees C.), unsaturation of tops (fraction boiling to 275 degrees C.), carbon residue of residuum from atmospheric distillation, yield of scrubber naptha, and percentage of nitrogen.

Previous experimental work has shown that the quantity and quality of the crude oil obtained from a given shale differs with the different rates of retorting. Figure 1 shows these differences graphically for the New Albany shale. In this work the time of retorting each sample was about one hour and fifteen minutes.

Summary of Conclusions.

The oil yield varies from 4.8 gallons per ton in sample R 14 and R 15 to 15.7 gallons per ton in sample R 40. The average yield for all samples is 10.3 gallons per ton.

The specific gravity of the crude oil varies from 0.924 in sample R 14 to 0.955 in sample R 44. The average for all samples is 0.943. The specific gravity of the crude shale oil changes almost directly with the change in temperature as it does with natural petroleum. The specific gravity of a dozen or more samples was determined as temperatures varying from 15.56 deg. C. to 30 deg. C. These specific gravities were then plotted on a graph and the curve shown in Figure 2 drawn. By the aid of this curve, the specific gravity of a sample of the New Albany shale oil determined at ordinary temperatures may be reduced to standard (15.56 deg. C.) with an error of not more than .001. To use this curve the temperature at which the specific gravity determination is made is found on the left side of the graph. This is followed on the horizontal line to the right until the curve is intersected, at which point the vertical line is followed down to the base line, where the correction to be added to the observed specific gravity is found. For illustration, suppose the specific gravity of a certain sample of oil was found to be 0.931 at 24 deg. C. By following the 24 degree line to the right until the curve is intersected and then down, the correction is found to be 0.007. The specific gravity at 15.56 deg. C., or standard, is then, 0.0931 plus 0.007, or 0.938.

The amount of tops from the crude oil varies from 38.8 to 52.9 per cent and the average of all samples is 44.1 per cent. Apparently the amount of tops varies inversely with the oil yield, that is, the less the oilyield, the greater the percentage of tops. The exact reason for this is not clearly understood but it is believed that in retorting a lean shale, at the same rate as a rich shale, there is less mechanical carrying over of the primary decomposition product of the shale

* Gavin, M. J. Oil-shale, an historical, technical, and economic study. Bureau of Mines, Bull. 210, 1922.

kerogen, and consequently the primary decomposition product is more completely broken up, yielding a crude oil with a higher percentage of tops.*

The tops after standing a few days become intensely red-black color, and if allowed to stand open and evaporate slowly, a small quantity of resinous gum-like material is deposited. This is a characteristic of cracked oils.

When the crude oil is run to coke the changes noted are given in the following tables. (retorting time 1 hr. 15 min.)

Changes in the composition of crude shale oil when run to coke.

Table I

	Crude	Once run to coke	Twice run to coke
Specific gravity	.943	.904	.896
Tops, per cent	46.6	54.3	61.6
Unsaturation tops, per cent	45.8	41.0	39.75

	Once run	Twice run
Oil used, per cent	100.00	100.00
Oil recovered, per cent	91.47	96.05
Coke, per cent	8.38	2.99
Loss, per cent	.15	.96

The unsaturation of the tops varies from 39.0 per cent in sample R 2 to 45.0 per cent in sample R 41, the average of all samples being 42.1 per cent. When the scrubber naptha is added to the tops (end point of scrubber naptha is less than the end point of the tops) the unsaturation of the combination is from 4 to 5 per cent less than that of the tops. The unsaturation of the tops of samples R's 16, 19, 31, 33, 35, 36, 45, 46, 47, 48, 51, and 52 has been calculated with the scrubber naptha included.

The following table shows the unsaturation of the various fractions of the tops. (Retorting time 1 hour and 15 minutes).

Table II

Unsaturation of the various fractions of the tops.

First drop, 56 deg. C.	Per cent fraction.	Unsaturation.
Up to 150 deg. C.	9.8	31.50
" " 200 " "	10.2	39.75
" " 250 " "	14.6	55.50
" " 275 " "	12.0	57.50
Tops	46.6 Average	45.79

*Work cited, p. 181.

SHOWING INFLUENCE OF RATE OF DISTILLATION OF NEW ALBANY SHALE ON PROPERTIES OF OIL PRODUCED.

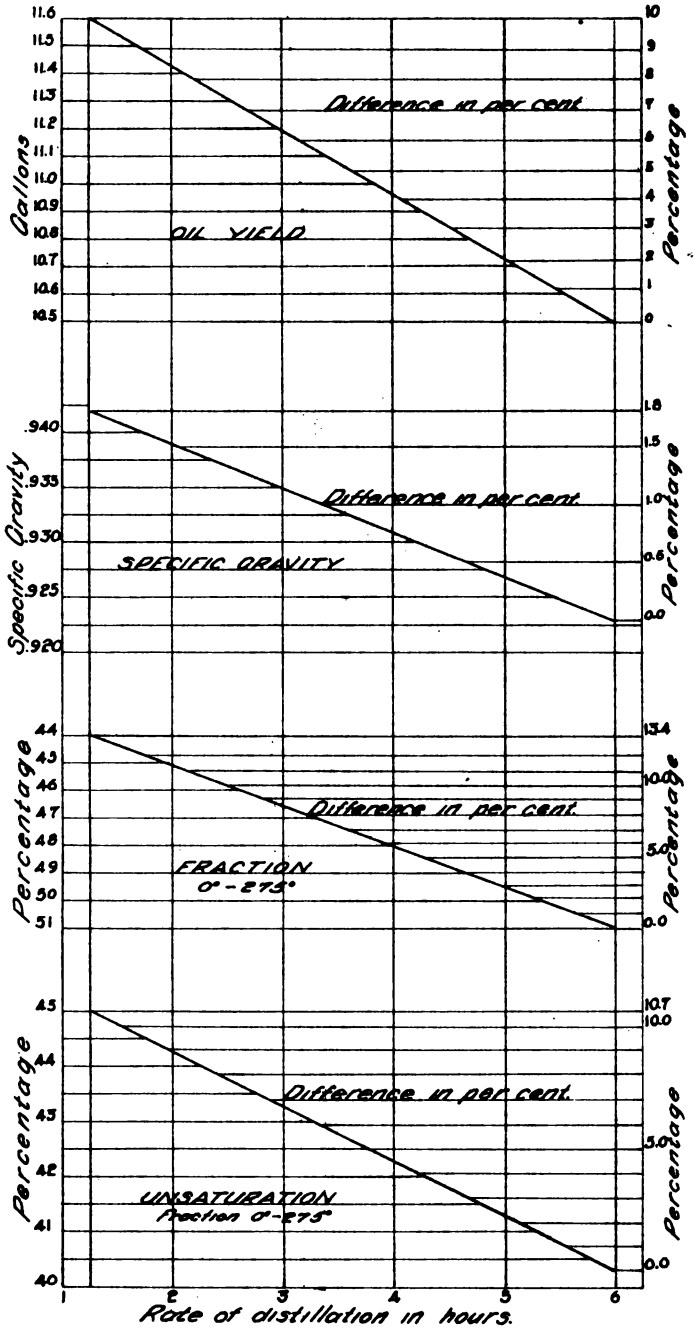

FIGURE 1.

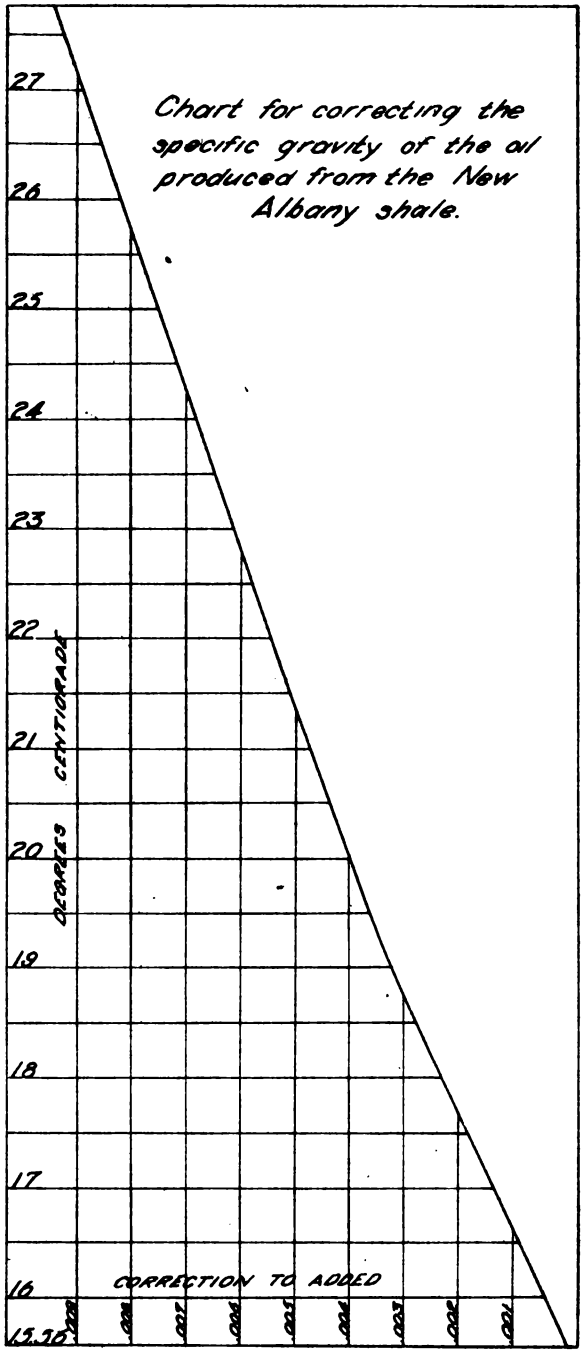

FIGURE 2

The carbon residue of the residuum from the atmospheric distillation (end point 275 deg. C.) varies from 4.95 per cent in sample R 51 to 7.67 per cent in sample R 7, the average of all samples being 6.3 per cent. Apparently the percentage of carbon residue varies inversely with the yield of oil, that is, the lowest yields of oil give the largest percentage of carbon residue and the largest yields of oil give the smallest percentage of carbon residue. The cause for this relation may be the same as that for the large amount of tops occuring in the oil from the lean shales, which was mentioned in the paragraph on that subject.

The amount of scrubber naptha, at the rate of retorting used in this work, varies from 0.31 gallons to 1.83 gallons per ton. Apparently the yield of naptha varies directly with the oil yield. Doubtless the rate of retorting will prove to be an important factor in the production of scrubber naptha. In one sample, R 36, the scrubber naptha recovered is equal to 13.5 per cent of the oil yield.

The following table indicates the boiling range and unsaturation of the scrubber naptha. (Retorting time 1 hour 15 minutes).

Table III

Boiling range and unsaturation of scrubber naptha.

Specific gravity .699
Unsaturation 28.00 per cent

Temperature, deg. C.		Volume distilled, cc.	Per cent distilled.
First drop	35	0	0
	54	5	20
	65	10	40
	81	15	60
	97	20	80
End point	131	24	96
	Loss	1	4
		25	100

As stated before, the addition of the scrubber naptha to the tops reduces the unsaturation of the combination of the two from 4 to 5 per cent. This addition of scrubber naptha also increases the total amount of motor fuel from 6 to 12 per cent. When the scrubber naptha is taken into consideration, the index number of the oil is increased from about 23 to 31. The index numbers of certain samples in the list of data has been calculated after the addition of the scrubber naptha to the tops from the distillation of the crude oil. The increase in the index number is illustrated in the following table.

Table IV

Illustration of the increase of the index number of an oil by the addition of scrubber naptha to the tops.

	Amount of tops per cent.	Unsaturation of tops, per cent.	Index number.
Without scrubber naptha	41.2	42.75	23.58
With scrubber naptha	54.7	39.25	33.23

The total nitrogen of the New Albany shale varies from 0.107 per cent in sample R 43 to 0.777 per cent in sample R 27, the average for 15 samples being 0.381 per cent, equivalent to a theoretical yield of 35.89 pounds of ammonium sulphate per ton. There is a general direct relation between oil yield and nitrogen content of the shale, that is, the greater the oil yield, the larger the percentage of nitrogen. However, this relation does not hold well for several samples.

Location and Description of the Samples given in Table V .

R 1- Grant 44, Floyd County, along the banks of Silver Creek. Fresh shale.

R 2- Weisman quarry, NE $\frac{1}{4}$ Sec. 12, T 4 R 6. $1\frac{3}{4}$ miles northeast of Crothersville. Six foot section. Fresh shale.

R 5-Basement of the First M. E. Church, North Vernon. Three foot section about 40 feet above the base of the formation. Shale slightly weathered.

R 6- Old Verbarg quarry, between North 4th. and 5th. streets, North Vernon. Three foot section taken about 2 feet above the base of the formation. Fresh shale.

R 7- Opposite the Muscatuck Inn between North Vernon and Vernon, along bank of Muscatuck River. Four foot section from 8 to 12 feet above base of the formation. At least one-half of section is grey shale. Fairly fresh.

R 8- North center of Sec. 5, T 5 R 9, about 2 miles northwest of Dupont along state road No. 26. Ten foot section from 5 to 15 feet above the base of the formation. Fairly fresh.

R 9- East center Sec. 33, T 7 R 7, $1\frac{1}{2}$ miles north of Hayden, 200 yards west of the bridge. Three foot section. Fresh.

R 10- One mile east of Hayden where the state road crosses Six Mile Creek. Section, 6 feet of the base of the formation. Fresh shale.

R 11- Northwest corner of Sec. 8, T 5 R 7, locally known as Burning Hill. Section 7 feet, probably near base of the formation. Fresh sample.

R 12- Northwest $\frac{1}{4}$ of Sec. 27, T 6 R 7, southwest of Hayden. Three foot section, probably 25 feet above the base of the formation. From a quarry. Fresh shale.

Table V .

Results of the assays of forty-eight samples of the New Albany Shale.

Sample number.	Oil, gallons per ton.	Water, gallons per ton.	Scrubber naptha, gallons per ton.	Nitrogen in shale, per cent.	Specific gravity of crude oil, 60°F.	First drop, deg. C.	Per cent distilled to 150° C.	Per cent distilled to 200° C.	Per cent distilled to 250° C.	Per cent distilled to 275° C. (Tops).	Tops and scrubber naptha combined, per cent.	Unsaturation of tops, per cent.	Carbon residue of residium, per cent.	Index number of oil without scrubber naptha.	Index number of oil with scrubber naptha.
R 1	8.02				.937						28.7		31.24		
R 2	11.98				.949						39.0	6.02	25.00		
R 5	9.0				.938	58	12.2	23.8		45.2	44.25	6.8	25.20		
R 6	12.2				.933	55	12.0	23.0		44.5	44.5	5.71	24.42		
R 7	5.2			.269	.941	55	11.25	25.0		52.50	51.75	7.67	25.33		
R 8	8.0				.937	59	11.4	22.8		44.4	43.75	5.95	24.95		
R 9	9.0				.931	61	14.0	25.8		47.2	41.38	6.16	27.45		
R 10	10.8			.164	.933	62	11.6	22.4		43.4	43.75	5.07	24.39		
R 11	11.4			.373	.949	61	9.4	21.8		42.0	45.0	6.30	23.10		
R 12	11.6				.951	60	10.6	21.6		44.0	42.75	6.72	25.08		
R 13	10.7				.944	63	8.6	19.6		41.8	43.0	5.80	23.82		
R 14	4.8			.168	.924	54	15.1	28.1		52.9	41.75	5.91	30.86		
R 15	4.8			.128	.931	55	11.1	24.9	39.0	48.9	46.0	7.00	26.40		
R 16	9.2		.46		.929	58	10.0	20.6	33.4	42.0	46.6	40.5	5.97		27.84
R 17	5.2				.934	60	13.5	27.0	41.6	50.18		45.75	7.07	27.22	
R 18	15.2			.209	.932	57	12.2	23.9	34.3	43.2		38.25	4.86	26.65	
R 19	8.6	4.8	.43		.934	58	12.3	23.4	39.7	44.5	49.5	39.5	6.91		30.04
R 20	12.4			.756	.946	57	11.6	22.6		43.6		47.75	6.48	22.75	
R 21	11.6			.155	.943	58	11.0	23.2		45.0		38.25	6.25	27.76	
R 22	12.0			.709	.947	54	12.0	23.0		44.0		44.0		24.64	
R 23	10.8				.952	55	11.0	23.0		44.4		37.5	6.61	27.75	
R 24	10.0			.634	.947	52	12.6	24.4		45.6		42.5	7.52	24.22	
R 25	10.8				.951	58	11.2	22.8		43.3		44.75		23.92	
R 26	12.0		.50	.642	.951	55	9.8	21.2		40.8		44.75	6.29	22.52	

Table V - (continued)

Sample number.	Oil, gallons per ton.	Water, gallons per ton.	Scrubber naptha, gallons per ton.	Nitrogen in shale, per cent.	Specific gravity of crude oil, 60° F.	First drop, deg. C.	Per cent distilled to 150° C.	Per cent distilled to 200° C.	Per cent distilled to 250° C.	Per cent distilled to 275° C. (Tops).	Tops and scrubber naptha combined, per cent.	Unsaturation of tops, per cent.	Carbon residue of residium, per cent.	Index number of oil without scrubber naptha.	Index number of oil with scrubber naptha.
R 27	12.6			777	.949	60	10.0	22.0		44.1		46.75	6.00	23.48	
R 28	12.0	5.0			.948	59	11.4	23.0	35.4	43.8		45.75	6.25	23.75	
R 29	4.8	4.8	.31		.926	55				50.6		42.0	6.9	29.34	
R 30	12.4			.508	.933	58	11.6	23.0		43.0		41.5	5.84	25.15	
R 31	14.0	6.0	1.83		.940	58	8.8	19.0	30.2	38.8	48.0	36.0	4.1		33.08
R 33	11.0	6.0	1.28		.941	57	11.2	22.9	31.4	41.5	66.1	38.5	6.3		38.80
R 34	13.4	10.0			.949	50	10.4	21.6	34.6	42.4		47.5	6.00	22.26	
R 35	11.23	6.0	.85		.941	56	11.4	21.0	34.0	43.0	50.4	38.25	5.8		31.18
R 36	11.2	6.0	1.5		.953	55	10.0	20.4	30.0	41.2	48.7	39.25	5.3		33.23
R 37	9.6	6.0	.66		.951	63	10.4	20.8	33.0	42.0		43.75	6.7	23.62	
R 39	10.0	6.0	1.06		.936	59	11.4	22.4	35.0	44.4		40.5	5.02	26.4	
R 40	15.7			.115	.949	62	9.2	20.6		40.4		44.5	5.41	22.42	
R 41	9.2				.943	58	11.8	21.8	35.2	43.2		45.0	6.85	23.70	
R 42	10.04	5.2	.60		.933	70	12.0	22.8	35.8	44.4		40.0	6.3	26.60	
R 43	5			.107	.934	59	14.5	24.7	39.0	47.75		43.0	7.17	27.19	
R 44	10.8	6.0			.955	83	9.6	20.0	32.0	41.6		43.5	5.61	23.50	
R 45	11.2	6.8	.75		.943	56	10.0	20.9	31.0	42.2	49.3	38.5	5.9		30.12
R 46	11.4	4.0	.77		.929	56	10.4	20.6	33.8	42.4	48.7	37.5			30.43
R 47	10.3	5.2	1.00		.939	58	10.7	20.5	34.6	41.8	51.5	38.2	6.2		31.82
R 48	10.2	5.2	1.04		.954	57	9.8	21.6	33.6	42.8	52.00	39.5	5.72		31.42
R 49	12.02	3.8	1.54		.952	60	10.4	22.0	35.8	44.0		42.25	6.00	25.41	
R 50	11.4	4.8			.940	53	12.6	24.0	37.2	45.6		41.00	5.57	26.89	
R 51	7.2	4.5	.45		.920	67	12.0	24.0	36.6	46.2	52.1	36.00	4.95		33.53
R 52	12.2	4.8	1.25		.936	54	12.0	22.6	35.2	43.4	52.7	31.00	5.46		36.36

R 13- Southwest $\frac{1}{4}$ Sec. 19, T 5 R 8, two miles west of Commiskey. Three foot section from a quarry. About 25 feet above the base of the formation. Fresh shale.

R 14- Southeast $\frac{1}{4}$ Sec. 20, T 5 R 8. In the northwest corner of the town of Commiskey. Three foot section about 20 feet above the base of the formation. Fairly fresh shale from an old quarry.

R 15- Southwest $\frac{1}{4}$ Sec. 9, T 6 R 7, one mile southwest of Hayden along state road number 4. Four foot section.

R 16- Center of Sec. 23, T 6 R 8, on the road between Lovett and Vernon. Three foot section about ten feet above the base of the formation.

R 17- West center Sec. 23, T 5 R 7, one-half mile west of Slate. The following section is representative of this sample.

	6 inches	black shale.
	1 "	sandstone.
	6 "	black shale.
	$\frac{1}{2}$ "	sandstone.
	5 "	black shale.
	1 "	sandstone.
3 feet	0 "	black shale.
2 "	0 "	grey shale.
4 "	0 "	black shale.
10 "	$7\frac{1}{2}$ "	total section.

R 18- Northeast $\frac{1}{4}$ Sec. 2, T 5 R 6, six miles southeast of Seymour where the state road crosses the Muscatuck River. Section 3 feet. Weathered.

R 19- Southwest $\frac{1}{4}$ Sec. 19, T 6 R 9, southeast of Vernon 3 miles, on state road number 26. Section represents the lower six feet of the formation.

R 20- Northwest $\frac{1}{4}$ Sec. 16, T 3 R 7, $1\frac{3}{4}$ miles northeast of Scottsburg, Jim Maggard quarry. Section 6 feet. Fresh shale.

R 21- Center Sec. 17, T 3 R 7, from an old quarry. Section 3 feet. Slightly weathered.

R 22- Northeast $\frac{1}{4}$ Sec. 8, T 3 R 7, Allen Maggard quarry. Six foot section. Fresh shale.

R 23- Northwest $\frac{1}{4}$ Sec. 34, T 4 R 7, quarry. This sample is represented by the following section.

3	feet	00	inches.	soil
		6	"	weathered black shale.
		2	"	weathered brown shale.
		¾	"	black shale.
		3	"	weathered brown shale.
		2	"	weathered brown shale.
		1/8	"	pyrite lense.
		1	"	soft red-brown shale.
		1/8	"	pyrite lense.
1	"	6	"	massive black shale.
		¾	"	sandstone.
11	"	10	"	massive black shale.
		2	"	blue shale.
		3	"	black shale.
		3	"	blue shale.
		3	"	black shale.
		2	"	blue shale.
1	"	1	"	black shale.
16	"	8¾	"	total section.

R 24- Southwest corner Sec. 11, T 3 R 7, 1½ miles southwest of Frankfort. Six foot section. From quarry. Fresh shale.

R 25- East center Sec. 23, T 4 R 7, three foot section of fairly fresh shale.

R 26- Southwest corner Sec. 35, T 4 R 7, from a quarry just northwest of Frankfort. Fresh shale. Section 5 feet.

R 27- East center Sec. 4, T 3 R 7. Five foot section of slightly weathered shale.

R 28- Northeast corner Sec. 35, T 3 R 7, Coleman's quarry. One-half mile southeast of Goshen. Four foot section of fresh shale.

R 29- Center Sec. 7, T 3 R 8, ½ mile southwest of Blocher. Five and one-half foot section of fresh shale from a quarry.

R 30- South center Sec. 34, T 3 R 7, Bridgewater quarry. Nine foot section of fresh shale.

R 31- Northwest ¼ Sec. 11, T 2 R 7, Elliot quarry. Six foot section of fresh shale.

R 32- One-fourth mile west of Lexington along the road. Two foot section of fresh shale.

R 33- Grant 263, ½ mile west of Nabbs, Campbell quarry. Ten foot section of fresh shale.

R 34- Northeast Center Grant 256, Rankin quarry, 2 miles northeast of Henryville. Fairly fresh sample. Five foot section from 5 to 10 feet below the top of the formation.

R 35- Southwest center Grant 271, one mile northeast of Henryville. Fresh seven foot section near top of formation.

R 36- South corner Grant 257, Cummings quarry, 2½ miles east of Henryville. Twelve foot section of fresh shale.

R 37- Center Grant 240, Monk quarry, southeast of Henryville. Ten foot section of fresh shale.

R 38- Center Grant 253. Four foot section of fresh shale.

R 39- Center Grant 246, 1½ miles southwest of Marysville. Eight foot section of fresh shale from a quarry.

R 40- Southwest center Grant 245. Three foot section of slightly weathered shale from an old quarry.

R 41- North corner Grant 186, ½ mile southeast of Memphis. Two and one-half foot section of fresh shale from an old quarry.

R 42- North corner Grant 190. Four foot section of fairly fresh shale.

R 43- Southeast corner Grant 208, 2 miles southwest of Otisco. Five foot section of fairly fresh shale.

R 44- West corner Grant 210, ½ mile southwest of Otisco. Five and one-half foot section of fairly fresh shale.

R 45- One-third mile southeast of Henryville. Six foot section of fairly fresh shale from 15 to 21 feet below the top of the formation.

R 46- Center Grant 136, one mile northwest of Charlestown from a quarry. Four foot section of fresh shale.

R 47- West corner Grant 131, one mile north of Speeds along the interurban track. Two and one-half foot section of fairly fresh shale from 1 to 3½ feet above the base of the formation.

R 48- East corner Grant 239, 1½ miles southeast of Henryville. Four foot section of fresh shale from a quarry.

R 49- Center of Grant 184 between Speeds and Memphis. Three foot section of slightly weathered shale.

R 50- East corner of Grant 86. Three and one-half foot section of fresh shale from a quarry.

R 51- One-half mile south of Sellersburg. Weathered.

R 52- East corner Grant 62. Three foot section of fresh shale.---Department of the Interior, Bureau of Mines, Reports of Investigations.
