NEBEL

Sampling of Coal an Investigation of the Effect of Size of Impurities on the Size of the Sample Required

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SAMPLING OF COAL
AN INVESTIGATION OF THE EFFECT OF SIZE OF IMPURITIES ON THE SIZE OF THE SAMPLE REQUIRED

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MERLE LOUIS NEBEL

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S A M P L I N G O F C O A L

AN INVESTIGATION OF THE EFFECT OF SIZE OF IMPURITIES ON THE SIZE OF THE SAMPLE REQUIRED

I. PURPOSE AND SCOPE OF WORK

The subject of coal sampling is one about which a great deal has been written, but very little accomplished. Considerable attention has been paid to the refinement of laboratory methods of analysis of samples, but very little has been done in the study of the sampling itself. The method of taking samples has, as a matter of course, developed from the earliest crude process to the more or less accurate method employed by the most up-to-date engineers at the present time, but the accuracy of even the most careful sampling depends upon conditions and laws so little understood that it seems probable that the actual value of many samples is greatly over-estimated.

Many unsolved problems are presented in the study of coal sampling; such as the size of sample which should be taken in any particular case, the effect of size of particles of impurities upon this size, the effect of variable proportions of impurities upon this size, and the fineness to which any given sample should be broken or crushed before further reducing it in size. All men who are in a position to know, state, that a large sample should be taken, and that it should be broken or crushed fine before being furthur reduced in size, but how large, and how fine, have been left to the judgement of the individual who happens to be taking the sample.

It is the object of the present experiments to add a

little

little to the understanding of the subject. The problems are by no means simple, and the amount of work required to be done in order that accurate conclusions can be drawn is so great, that for the purposes of this paper only one phase of the subject could be considered; that is, the effect of size of particles of impurities upon the size of sample required.

The ordinary condition met with in the sampling of coal is that in which the coal is present in all sizes ranging from that of the maximum diameter down to zero, and the free impurities, of which shale is the chief one, are also present in the same range of sizes. Another important condition often met with is that of sized coal. In Illinois and many other states, coal is sold on a sized basis, being sized at the mines between fairly close limits. A considerable amount of coal is also washed and sized, so that a very important part of the coal produced contains impurities which are practically the same size as the particles of coal. It is, therefore, important from a commercial as well as a theoretical standpoint to understand the difference in action between sized and unsized material during the process of sampling.

To make results really valuable and accurate, a large number of samples should be taken and a large number of determinations made, but the time available for this work was so limited that only a small amount could be done. Consequently the work can be regarded as only preliminary, and the results are only indicative and not final in nature.

II. T H E O R Y

The subject of probability and chance has been covered very thoroughly in the literature of the past twenty-five years. A considerable amount of involved higher mathematics has been adapted to the solution of problems which have to do with chance, and certain definite laws, variously called "Laws of Chance","Laws of Probability", "Laws of Sampling", etc. have been deduced. The earliest use of which we have any record to which these laws were put was in connection with games of chance, and we owe their development chiefly to men who were trying to work out some system for consistently winning at these games. Later they were applied to life-insurance, and still later to statistics of all kinds. Although the laws are often called "Laws of Sampling", they were not applied to the solution of sampling problems such as are met with in the sampling of coal or ore until very recently. The only work of this kind on which published data is available is a series of experiments carried on in 1908 and 1909 by Mr. E. G. Bailey, Engineer of the Fuel Testing Co. of Boston, Mass., who studied the application of the above laws to coal sampling. It is surprising that a subject so important should be neglected for so long after the development of the "Laws of Sampling".

Although no works of reference are mentioned in the course of the thesis, the following works have been consulted freely and used in the discussion of the theory of sampling:

"An Introduction to the Theory of Statistics", by G. Udny Yule.

Appendix to: "Type and Variability of Corn", by Eugene Davenport. Appendix by Henry L. Rietz.

The impurities in coal may be divided into two classes: (a) free impurities, such as shale, pyrite, calcite, gypsum, etc.; (b) disseminated impurities inherent in the coal itself, due to the mineral matter in the vegetable mass from which the coal was formed. This latter impurity has a nearly constant value for the same coal bed over an extended area, and if the free impurities are removed, the remaining substance has a fairly constant value for its ash content.

Clean bituminous coal has a specific gravity ranging from 1.15 to 1.35, ~~never~~ rarely greater than the latter. By thoroughly stirring the crushed coal in a solution having a specific gravity of 1.35, the clean coal can be separated from the free impurities, which sink to the bottom of the container and can be removed. The clean coal, which will hereinafter be designated as "float coal", remains floating in the solution, and it contains only a small proportion of disseminated impurities together with some few small particles of pyrite, calcite, etc., which may have been attached to lumps of coal. By adding to this float coal particles of shale of known composition, a mixture can be obtained in which both constituents are fairly homogeneous, and sampling of such a mixture should follow the laws of simple sampling. That is, the sampling of such a mixture is comparable to taking a sample from a mixture of black balls and white balls, instead of coal and shale.

The simplest case of simple sampling is the tossing of a coin. It is to be expected that in any long series of throws the coin will fall with either face uppermost an approximately equal number of times. If a number of coins are tossed similar results

might

may be expected, and this case is comparable to that of drawing a sample from a mixture of equal proportions of black and white balls. If the proportions of black and white balls are not the same the case is slightly different from that of coin tossing, but is similar to that of sampling such a mixture of coal and shale as mentioned above. If the mixture is divided by means of a riffle sampler, it is probable that one portion or the other will have an excess of shale particles, but as the number of samples divided increases the average will show a more and more even division.

If the total number of particles is represented by "n", then the theoretical probable error involved is $E_p = 0.6745\sqrt{n}$*, when the proportions of coal and shale or of black and white balls are the same. Where the proportions vary the theoretical probable error is $E_p = 0.6745\sqrt{npq}$*, where "p" is the proportion of one constituent and "q" is the proportion of the other. If the division is made experimentally a large number of times and the number of particles of shale in each portion counted each time, the experimental probable error may be obtained from the expression, $E_p = 0.6745\sqrt{\frac{\Sigma D^2}{N}}$*, in which "N" is a large number of trials, "D" is the deviation of the number of particles discovered in any given trial from the mean number, and "ΣD^2" is the sum of the squares of all of these deviations. A comparison of the experimental probable error with the theoretical probable error will indicate whether or not the method of sampling comes under the laws of simple sampling. If the experimental "E_p" is not widely different from the theoretical "E_p" then this relation is true.

The term "probable error" has no reference to errors in computations

*The ordinary mathematical formula for probable error.

computations or to experimental errors, nor does it set the limits within which errors must lie, for such limits cannot be set. It simply means that the true value lies within the range set by $\pm E_p$; that is, if the determination shows a value of say 6.00, with a probable error of $\pm$ 0.05, then the chances are even that the true value is not less than 5.95 (6.00-0.05), nor greater than 6.05 (6.00+0.05). The chances are also even that the true value may lies outside this range, but these chances rapidly decrease as the range is increased. Thus the chances against the true value lying outside of twice the probable error are as 4.5 to 1. The following table shows the rapid increase in the chances that the true value lies within the range set by $\pm E_p$, $\pm 2E_p$, etc.:

$\pm E_p$,	the chances are ------------------	even.
$\pm 2E_p$,	" " " --------------	4.5 to 1.
$\pm 3E_p$,	" " " ------------	21 to 1.
$\pm 4E_p$,	" " " -----------	142 to 1.
$\pm 5E_p$,	" " " ----------	1310 to 1.
$\pm 6E_p$,	" " " --------	19,200 to 1.
$\pm 7E_p$,	" " " -------	420,000 to 1.
$\pm 8E_p$,	" " " -----	17,000,000 to 1.
$\pm 9E_p$,	" " "	about a billion to 1.

By the time an allowance of four times the probable error has been made the chance amounts to practical certainty, and even the 21 to 1 chance obtained by using three times E_p involves far less chance than is involved in most business transactions.

In studying the sampling of coal according to the above laws, two distinct conditions were investigated by the writer:

(1) That in which the particles of shale range in size from that of the maximum of the coal particles to zero.

(2) That in which the particles of shale are the <u>same</u> size as that of the maximum of the coal particles.

In crushing any material the number of particles increases as the size is diminished. This amount of increase is inversely proportional to the cubes of the diameters of the respective particles, where the broken down particles retain the same shape as the original. For instance, decreasing the diameter one-half increases the number of particles by eight. This is evident from a consideration of the cube. If a two-inch cube is broken into one-inch cubes, eight of the smaller ones will be formed. The shale used in this work was found to vary practically according to this law. By counting of 1000 particles, the weight per particle of $\frac{1}{4}$-inch shale(sized thru 0.251-in. and on 0.206-in.) was found to be 0.000527 lb.. Similarly that of 1/8-in. shale (sized thru 0.126-in. and on 0.104-in.) was found to be 0.000067 lb, which is practically one-eighth that of the larger particles, which have twice the diameter and consequently eight times the volume.

When the shale is of all sizes a better mixture can be obtained than when it is all of a maximum size, due to the larger number of particles in the sample. Since the mixture is better, the error involved will be less, or what amounts to the same thing, a smaller sample can be used without exceeding the error

from

from the other type of sample. Indeed the actual relative sizes of samples required for the two types of mixtures can be obtained by sampling a large number of mixtures of both types and finding the probable error for each. Then by comparing these probable errors the ratio of sizes of samples required can be found.

It is difficult to say just how many samples should be taken in order to obtain results that can be depended upon, but for the most accurate work it would be advisable to take, let us say, a thousand samples. It was impossible in the present case to carry on the work on such a large scale, so a uniform series of sixteen samples was selected for each of the cases taken up. It has been found that such a series gives fairly good results, although of course the dependence cannot be placed upon it that could be placed upon a larger series.

The accuracy of the work done can be illustrated graphically by means of the probability curve. This is a curve plotted with positive and negative deviations from the mean value as abscissae, and numbers of cases in which each particular deviation occurs as ordinates. Such curves show the distribution of the errors involved for any set of analyses. Probability curves were plotted for each of the sample series discussed, and they are shown on page 27. The curves obtained from the experimental data are shown in black, while the ideal curves as they would probably appear for a large sample series are super-imposed in red. A comparison of the two curves in each case readily shows how necessary it is to carry on an extensive series of experiments in order to obtain consistent results.

III. E X P E R I M E N T A L W O R K .

A. METHOD OF PROCEDURE.

The original plan as outlined before starting to work included the following cases:

(1) A comparison of the action of coal and shale when crushed by the same machine, and between the same limits of size.

(2) The effect on accuracy of sampling of mixing a known amount of float coal (defined above), under 1/4-inch in size and with a specific gravity less than 1.35, with one-tenth of that amount of shale of all sizes, ranging from a maximum diameter of 1/4-inch to zero.

(3) The effect of substituting for the above shale the same weight of similar shale, the particles of which were all the same size as that of the largest particles of coal; e.g. 1/4-in.

(4) The effect of substituting for the above shale the same weight of similar shale, the particles of which were all of the same size as the mean diameters of the coal particles; e.g.1/8in.

(5) It was also decided to further investigate any point of interest which might be brought out by the earlier experiments; and if time permitted to study the effect of particles of shale which were of the same <u>weight</u> as the mean <u>weight</u> of the particles of coal, instead of the same <u>size</u> as in case No.4.

Owing to lack of time, cases No.4 & 5 both had to be left for investigation at some future time.

The coal used in the experiments was a high grade bituminous coal from the Williamson Co. field of southern Illinois. About 150 lb of the coal were crushed in a small roll-jaw sample crusher

crusher to pass through 0.251 in. sieve openings. A solution of zinc chloride was prepared with a specific gravity of 1.35, and the unsized, crushed coal was thoroughly stirred into it. The "sink" or refuse, which dropped to the bottom was discarded, and the float coal was carefully washed and dried. Practically all of the free impurities such as pyrite, free shale, calcite, gypsum, etc., were removed in the refuse, the float coal containing only such impurities as the finely divided shaly material disseminated throughout the coal, and such small scales of pyrite and gypsum as may have remained attached to a few of the larger particles of coal.

This float coal, after being air-dried, was sized on a complete set of laboratory screens with openings varying from 0.251 in. to 0.00286 in. in size; the size of each screen varying from that of the next nearly by the ratio of the fourth root of two ($\sqrt[4]{2}$). From this sizing test a cumulative percentage curve was plotted, showing graphically the effect of crushing upon the size of coal particles. This curve is shown on page 17 .

After sizing the coal, the next step was to divide it by means of a riffle sampler so that four 16-pound representative samples were obtained. The riffle sampler, or simply "riffle"as it is usually called, consisted of ten chutes with openings 3/4-in. across, alternate chutes sloping in opposite directions and delivering approximately equal portions of any material poured through them to opposite sides of the sampler. One of the four samples was saved for an investigation of case No.2, as explained above; a second for case No.3; a third for case No.4; and the fourth for case No.5. A fifth sample was taken and reduced to about 1.5 lb by passing it

through

through the riffle. It was then ground to 80-mesh in a disc pulverizer, reduced to laboratory sample size (about enough to fill a 4-oz bottle),and analyzed in triplicate for ash and moisture. The ash content as determined in this manner was used in the calculation of ash in the samples of mixed coal and shale as explained below. The results of the analysis are shown on page 14.

In all of the analyses a moisture determination was made as well as an ash, and the values of the ash were recalculated to the dry basis. It is obviously of no value to compare the ash content of a number of samples when calculated upon any basis but the dry one, for two apparently similar samples might have considerably different proportions of moisture, even after careful air-drying. No other constituents of the coal were determined, as it is the ash content which is most greatly affected by errors in sampling.

The shale used in the investigation was a tough, light-gray, clay shale from near Danville, Illinois. From its appearance it was believed that it would stand up well without excessive breakage under the usage given it in ordinary sampling. This quality was afterward doubted and was tested by passing exactly 1000 counted particles 1/4-in. in diameter through the riffle a number of times, and giving them approximately the same treatment that the samples were given. After this treatment the total weight of shale remained the same, but on counting them carefully 1006 particles were found. This would indicate that a number of particles had broken up so as to form 6 additional particles. This conclusion was borne out by the fact that several particles smaller in size than the ordinary were noticed. This action of the shale

will be referred to later.

About 50 lb of the selected shale were crushed in the same sample crusher used for the coal, to pass through the same screen, and then were sized on the same set of sample screens. A cumulative percentage curve was drawn on the same scale as that used for the coal curve. (See page 17). The whole sample was then passed through the riffle described above, and a 1-pound sample saved. This was ground to 80-mesh in the disc pulverizer, reduced to a laboratory sample, and analyzed in triplicate for ash and moisture. Another representative sample was obtained weighing 1.60-lb,and containing all sizes of shale from 0.251 in. down to zero. From the remainder of the shale about 3-1/3 lb of 1/4-in. size were obtained, being sized between 0.251 in. and 0.206 in.. Likewise about 3-1/2 lb of 1/8-in. size were obtained, being sized between 0.126 in. and 0.104 in.. One-thousand particles of the larger size and two-thousand of the smaller size were counted and weighed, and the weight per particle thus obtained. (See page 24.)

Then with a representative sample of the float coal, weighing 16.00 lb, was mixed the all-size sample of shale, weighing 1.60 lb. This mixture was divided by means of the riffle into 16 samples, and all were weighed, then ground to 80-mesh in the disc pulverizer. They were next reduced to laboratory samples, and analyzed in duplicate for ash and moisture. The results of these analyses are shown on page 14 , and the division on page 18.

With another sample of the float coal weighing 16.00 lb was mixed 1.60 lb of the 1/4-in. shale. This mixture was also divided by means of the riffle into 16 samples, and each sample was weighed.(See page 19.) Next the shale particles were carefully

removed from each sample by hand-picking, counted, weighed, and returned to their respective samples. Eight of the samples were ground to 80-mesh, reduced to laboratory samples, and analyzed in duplicate for ash and moisture. (See page 20.)

Knowing the proportion of ash in the float coal and in the shale, calculated on the dry basis from previous analyses, it was possible to calculate the ash content of the mixture, since the number of particles of shale, and the weight per particle are known. From a consideration of conditions, it was believed that the proportion of ash as calculated in this manner would check with the ash as shown by analysis. Such being the case, it would be unnecessary to make analyses of all samples, the counting sufficing. Therefore the ash was calculated for all 16 samples from the counted particles, but only 8 were analyzed as a check. The results of this investigation are tabulated on page 23.

Having obtained the data from the above laboratory work, the various probable errors were calculated for each set of analyses, and their relation to each other studied so as to determine the effect of the various conditions imposed.

III. EXPERIMENTAL WORK.

B. TABULATION OF DATA OBTAINED.

ASH IN FLOAT COAL AND SHALE

	Ash %	Moisture %	Dry Ash %
FLOAT COAL	5.85	6.44	6.25
SHALE	91.94	0.87	92.75

ASH DETERMINATIONS FOR CASE NO. 2.

Sample No.	Ash %	Moisture %	Dry Ash %
101	13.72	4.40	14.35
102	13.81	5.14	14.57
103	13.94	3.80	14.49
104	13.89	4.97	14.61
105	12.38	5.01	13.03
106	13.32	4.78	13.99
107	14.04	3.83	14.60
108	13.88	5.02	14.61
109	13.64	4.39	14.27
110	13.79	3.47	14.29
111	13.61	4.88	14.31
112	13.13	4.40	13.73
113	13.74	4.52	14.39
114	12.91	4.76	13.55
115	14.00	4.14	14.60
116	13.49	4.26	14.09

SIZING TEST OF FLOAT COAL

Sizes of screen openings thru	on	Weight in lbs	Cumulative weights	Per cent of total	Cumulative percents
.251	.206	1.70	1.70	8.38	8.38
.206	.178	1.36	3.06	6.70	15.08
.178	.150	2.23	5.29	10.98	26.06
.150	.126	3.45	8.74	16.99	43.05
.126	.104	3.38	12.12	16.65	59.70
.104	.089	1.52	13.64	7.49	67.19
.089	.075	1.03	14.67	5.07	72.26
.075	.063	0.68	15.35	3.35	75.61
.063	.053	0.99	16.34	4.87	80.58
.053	.0445	0.84	17.18	4.14	84.62
.0445	.0376	0.54	17.72	2.66	87.38
.0376	.0317	0.45	18.17	2.22	89.50
.0317	.0265	0.24	18.41	1.18	90.78
.0265	.0222	0.36	18.77	1.77	92.45
.0222	.0186	0.30	19.07	1.48	93.93
.0186	.0155	0.42	19.49	2.07	96.00
.0155	.0132	0.00	19.49	0.00	96.00
.0132	.0110	0.12	19.61	0.59	96.69
.0110	.0092	0.20	19.81	0.99	97.68
.0092	.0077	0.02	19.83	0.10	97.78
.0077	.0068	0.10	19.93	0.49	98.17
.0068	.0055	0.11	20.04	0.54	98.71
.0055	.00463	0.07	20.11	0.35	99.06
.00463	.00394	0.02	20.13	0.10	99.16
.00394	.00328	0.02	20.15	0.10	99.26
.00328	.00286	0.04	20.19	0.20	99.46
.00286	------	0.13	20.32	0.64	100.00

SIZING TEST OF SHALE

Sizes of screen openings thru	on	Weight in lbs	Cumulative weights	Per Cent of total	Cumulative percents
.251	.206	6.00	6.00	16.03	16.03
.206	.178	2.25	8.25	6.01	22.04
.178	.150	2.10	10.35	5.61	27.65
.150	.126	3.29	13.64	8.80	36.45
.126	.104	4.25	17.89	11.37	47.82
.104	.089	1.39	19.28	3.72	51.54
.089	.075	1.43	20.71	3.82	55.36
.075	.063	1.25	21.96	3.34	58.70
.063	.053	1.86	23.82	4.96	63.66
.053	.0445	1.60	25.42	4.28	67.94
.0445	.0376	1.35	26.77	3.61	71.55
.0376	.0317	1.18	27.95	3.15	74.70
.0317	.0265	0.70	28.65	1.87	76.57
.0265	.0222	1.02	29.67	2.73	79.20
.0222	.0186	1.09	30.76	2.92	82.22
.0186	.0155	0.73	31.49	1.95	84.17
.0155	.0132	0.67	32.16	1.79	85.96
.0132	.0110	0.37	32.53	0.99	86.95
.0110	.0092	0.76	33.29	2.03	88.98
.0092	.0077	0.06	33.35	0.16	89.14
.0077	.0068	0.31	33.66	0.83	89.97
.0068	.0055	0.27	33.93	0.72	90.69
.0055	.00463	0.21	34.14	0.56	91.25
.00463	.00394	0.25	34.39	0.67	91.92
.00394	.00328	0.19	34.58	0.51	92.43
.00328	.00286	0.35	34.93	0.94	93.37
.00286	------	2.48	37.41	6.63	100.00

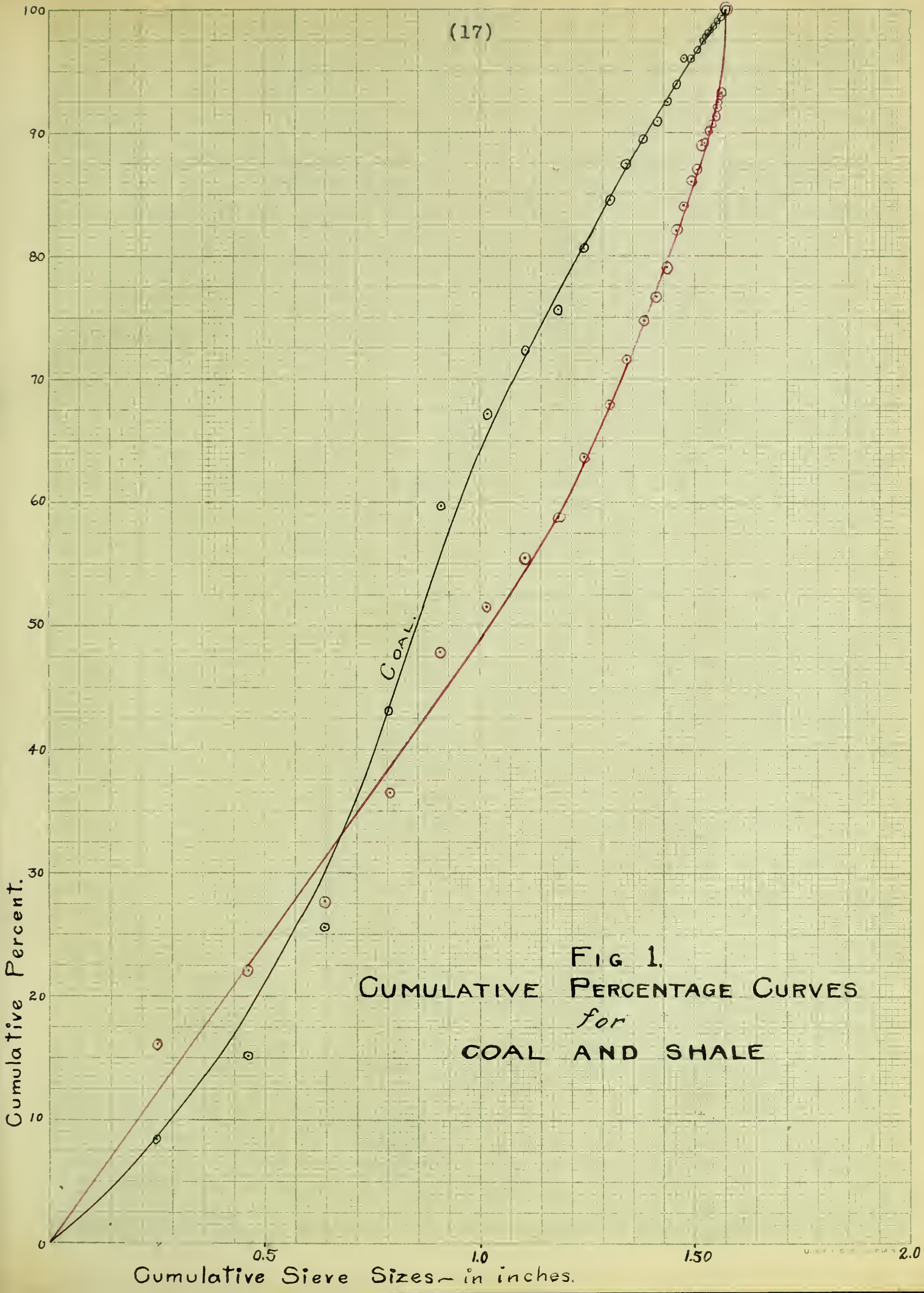


Fig 1.
Cumulative Percentage Curves
for
Coal and Shale

DIVISION OF SAMPLE IN CASE NO.2.

Sample No.	Weight of sample-lbs.
101	1.08
102	1.08
103	1.08
104	1.16
105	1.06
106	1.02
107	1.08
108	1.15
109	1.09
110	1.10
111	1.13
112	1.16
113	1.10
114	1.08
115	1.12
116	1.12
Mean	1.10

Float Coal- 16.00 lb

Shale ($\frac{1}{4}$"-0")-1.60 "

Total ----- 17.60 "

DIVISION OF SAMPLE IN CASE NO. 3.

Sample No.	Weight of Sample in lbs.
201	0.98
202	0.95
203	0.87
204	0.92
205	1.14
206	1.01
207	1.06
208	1.09
209	1.12
210	1.19
211	1.15
212	1.19
213	1.16
214	1.20
215	1.22
216	1.35

Float Coal-$\frac{1}{4}$"--- 16.00 lb

Shale --$\frac{1}{4}$" ---- 1.60 "

Total -------- 17.60 "

ASH DETERMINATIONS FOR CASE NO. 3.

Sample No.	Ash %	Moisture %	Dry Ash %
209	13.10	4.74	13.75
210	15.15	4.77	15.90
211	13.82	4.92	14.54
212	13.85	5.30	14.62
213	13.88	4.96	14.60
214	14.52	4.61	15.22
215	14.62	3.67	15.19
216	14.36	4.30	15.00

PROBABLE ERROR IN SAMPLING 80 MESH MATERIAL.

Sample No.	Ash by Analysis A	Ash by Analysis B	D	D^2
209	13.12	13.07	0.05	.0025
210	15.13	15.16	0.03	.0009
211	13.79	13.84	0.05	.0025
212	13.80	13.90	0.10	.0100
213	13.84	13.91	0.07	.0049
214	14.47	14.57	0.10	.0100
215	14.68	14.55	0.13	.0169
216	14.28	14.43	0.15	.0225
101	13.71	13.72	0.01	.0001
102	13.79	13.83	0.04	.0016
103	13.98	13.90	0.08	.0064
104	13.84	13.93	0.09	.0081
105	12.36	12.40	0.04	.0016
106	13.34	13.29	0.05	.0025
107	14.06	14.01	0.05	.0025
108	13.88	13.87	0.01	.0001
109	13.65	13.63	0.02	.0004
110	13.79	13.78	0.01	.0001
111	13.62	13.60	0.02	.0004
112	13.12	13.14	0.02	.0004
113	13.73	13.74	0.01	.0001
114	12.96	12.86	0.10	.0100
115	14.00	14.00	0.00	.0000
116	13.46	13.51	0.05	.0025

$\Sigma D^2 = .1070$

$N = 24$

$$E_p = 0.6745 \sqrt{\frac{\Sigma D^2}{N}}.$$

$$= \underline{0.0452.}$$

EXPERIMENTAL PROBABLE ERROR FOR CASE NO. 2.

Sample No.	Dry Ash	D	D^2
101	14.35	+0.13	0.0169
102	14.57	+0.35	0.1225
103	14.49	+0.27	0.0729
104	14.61	+0.39	0.1521
105	13.03	-1.19*	1.4161
106	13.99	-0.23	0.0529
107	14.60	+0.38	0.1444
108	14.61	+0.39	0.1521
109	14.27	+0.05	0.0025
110	14.29	+0.07	0.0049
111	14.31	+0.09	0.0081
112	13.73	-0.49	0.2401
113	14.39	+0.17	0.0289
114	13.55	-0.67	0.4489
115	14.60	+0.38	0.1444
116	14.09	-0.13	0.0169
Mean	14.22		$\Sigma D^2 = 3.0246$

* See page 30.

$$E_p = 0.6745 \sqrt{\frac{\Sigma D^2}{N}}$$

$$= 0.6745 \sqrt{\frac{3.0246}{16}}$$

$$= \underline{0.2933}.$$

A C O M P A R I S O N

of

ASH VALUES BY ANALYSIS, AND BY COUNTING PARTICLES OF SHALE.

Sample No.	Dry Ash	Calculated Ash	Variation
209	13.75	14.13	+0.38
210	15.90	14.93	-0.97
211	14.54	14.30	-0.24
212	14.62	14.61	-0.01
213	14.60	15.18	+0.58
214	15.22	15.21	-0.01
215	15.19	14.60	-0.59
216	15.00	14.90	-0.10

Maximum positive variation +0.58

Maximum negative variation -0.97

THEORETICAL PROBABLE ERROR FROM COUNTING PARTICLES. CASE NO.3.

Material.	Proportion.	Weight per Particle. lbs.	Total weight. lbs.	Number of particles.
Shale ----	.091	.000527	1.617	3068
Coal ----	.909	.000288	15.983	55497
Total ----	1.000		17.600	58,565

$$n = \frac{58,565}{16}$$

CALCULATED ERROR.

$$E_p = 0.6745\sqrt{npq}$$

$$= 0.6745\sqrt{\frac{58,565}{16}(.091)(.909)}$$

$$= \frac{45.94.}{4}$$

$$= \underline{\underline{11.48}}.$$

WEIGHT OF COAL AND SHALE PARTICLES

Weight of 1000 particles of 1/4-in. shale --- 0.527 lb

Weight per particle ---------------------- 0.000527 "

Weight of 1390 particles of 1/4-in. coal --- 0.400 "

Weight per particle ---------------------- 0.000288 "

Weight of 2000 particles of 1/8-in. shale --- 0.135 "

Weight per particle ---------------------- 0.000067 "

EXPERIMENTAL PROBABLE ERROR FROM COUNTING PARTICLES

Sample No.	Shale Particles	D	D^2
201	167	-25	625
202	179	-13	169
203	128	-64	4096
204	151	-41	1681
205	228	+36	1296
206	172	-20	400
207	170	-22	484
208	183	- 9	81
209	184	- 8	64
210	215	+23	529
211	188	- 4	16
212	207	+15	225
213	216	+24	576
214	226	+34	1156
215	212	+20	400
216	242	+50	2500
Mean No. =	192	408	14,298 = ΣD^2

$$E_p = 0.6745 \sqrt{\frac{\Sigma D^2}{N}}$$

$$= 0.6745 \sqrt{\frac{14,298}{16}}$$

$$= 20.14.$$

EXPERIMENTAL PROBABLE ERROR FOR CASE NO.3.

Sample No.	Dry Ash %	D	D^2
209	13.75	-1.10	1.2100
210	15.90	+1.05	1.1025
211	14.54	-0.31	0.0961
212	14.62	-0.23	0.0529
213	14.60	-0.25	0.0625
214	15.22	+0.37	0.1369
215	15.19	+0.34	0.1156
216	15.00	+0.15	0.0225
	Mean = 14.85		ΣD^2 = 2.7990

$$E_p = 0.6745 \sqrt{\frac{\Sigma D^2}{N}}$$

$$= 0.6745 \sqrt{\frac{2.7990}{8}}$$

$$= \underline{0.3986.}$$

PROBABILITY CURVES.

Fig. 2.

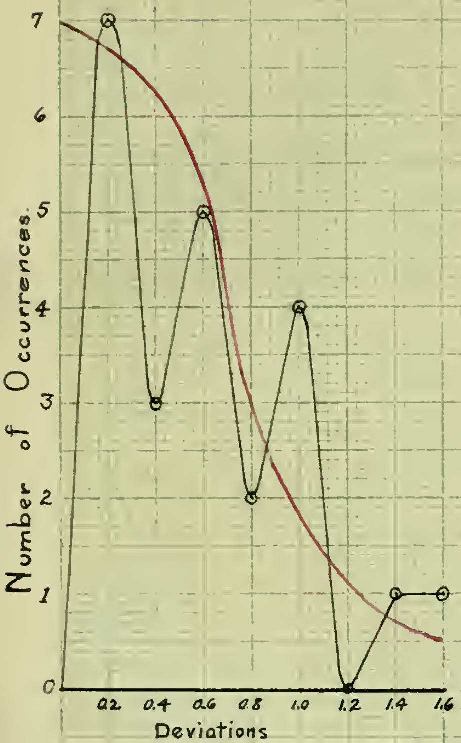


Fig. 3.

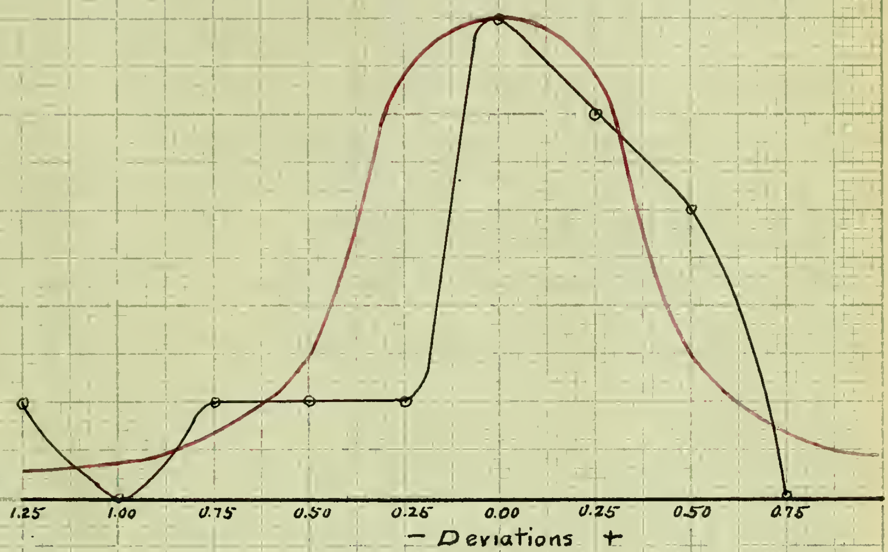


Fig. 4.

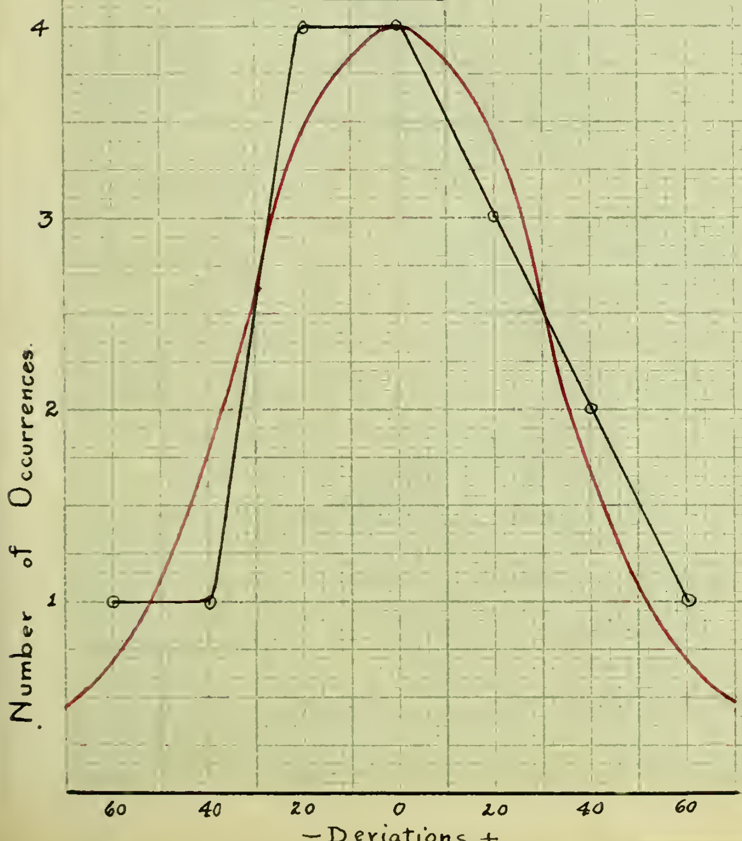
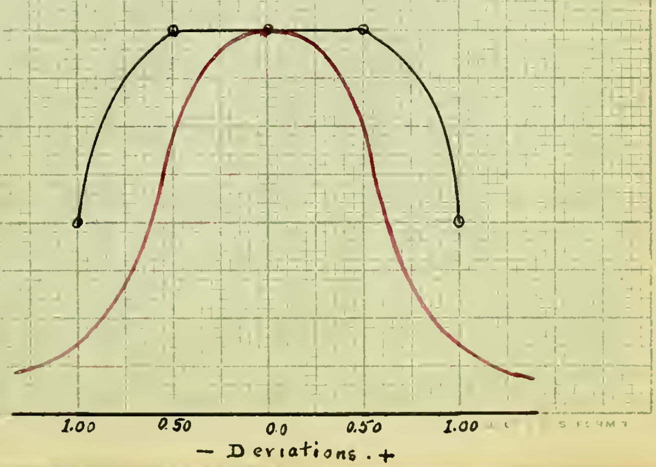


Fig. 5.



IV. DISCUSSION OF DATA AND INTERPRETATION OF RESULTS.

The first thing to be considered is the action of the coal and shale when crushed, as explained under case No. 1. The data obtained in sizing them after crushing is tabulated on pages 15 and 16, and the curves on page 17 were plotted from this data. Cumulative sizes of sieve openings were used as abscissae, and cumulative percentages of total weight as ordinates. The curves for the two materials were plotted to the same scale and from the same origin. From a study of these curves and the data from which they were obtained it is evident that the two materials, although differing widely in composition and physical characteristics, act practically the same when broken down under identical conditions. The coal curve, being a trifle steeper, indicates a more brittle material, but the difference is so slight that it can safely be neglected for the purposes of this investigation. Therefore in sampling a mixture of these two materials, no appreciable error should be introduced by the production of a greater number of particles of the one than of the other under identical conditions of crushing.

In reducing a 1.76-pound sample of mixed coal and shale from a maximum size of 0.251-in. to a 1-gram sample with a maximum size of 0.0068 in.(80-mesh), it is evident that a certain error is involved; due mainly to the difference in number of particles present in the two cases. It is essential to know how great this error is, for an excessive error at any one point in the process may make the final result worthless. This error can be determined by considering the differences between the values of ash for each of a number of duplicate analyses on the 80-mesh material.

The results obtained from a series of 24 such analyses are tabulated on page 21 of this report. The probable error, found by the expression $E_p = 0.6745 \sqrt{\frac{D^2}{N}}$ as explained above, is shown to be 0.0452. A 1-gram sample was taken for analysis. To obtain the same error in sampling 1/4-in. material a much larger amount would have to be used than was taken for the present samples. Since the size of sample required varies directly as the cube of the diameters of the particles in the sample,

then:-

$$x : (.251)^3 :: 1 : (.0068)^3.$$

or $$x = 50{,}653 \text{ gm.}$$

$$= \underline{112 \text{ lb}}.$$

Therefore a sample of 112 lb of the 0.251-in. material would have to be taken in order to reduce the probable error to the same value as that involved in sampling the 80-mesh material. The probability curve for this series of experiments is shown in Fig. 2, page 27.

In the present case a sample of 1.76 lb was used, and sampled as explained above under case No. 2, so a much larger error was involved. Since the probable error involved in simple sampling varies directly as the square root of the size of sample taken,

then:-

$$x : \sqrt{112} :: .0452 : \sqrt{1.76}$$

or $$x = \sqrt{\frac{112}{1.76}} . (.0452)$$

$$= 0.3616.$$

This is the probable error which might be expected for the sample of 0.251-in. material, using the probable error of the 80-mesh material as a basis.

The error in sampling the 0.251-in. material may be deter-

mined experimentally by making a series of analyses, finding the deviation of each from the mean value, and substituting in the expression $E_p = 0.6745 \sqrt{\frac{\Sigma D^2}{N}}$. The results of such a series in which the coal and shale were both of all sizes from 0.251 to 0.00, are tabulated on page 22. They show a probable error of 0.2933, which is but slightly less than the calculated error of o.3616 as obtained above. This indicates that the conditions surrounding the present work are similar to those required for simple sampling, and that the laws of simple sampling are correctly applicable.

Since only once in 142 times may any value be expected to deviate from the mean by as much as <u>four</u> times the probable error, it is reasonably certain that if a probable error of 1/4 is arbitrarily selected, only once in 142 times will any experimental error of as much as 1 % be reached. (See page 6.) Selecting, then, 1 % as the maximum allowable error, and using 0.25 as the probable error, it is evident that the value of 0.2933 obtained above is too high. In other words, a sample larger than 1.76 lb should be used for a sample containing the size of material used is this work. Since the amount of material required varies directly as the square of the probable errors involved,

then;-

$$x : (.2933)^2 :: 1.76 : (.25)^2.$$

or

$$x = 2.41 \text{ lb.}$$

Therefore to keep the experimental error within 1-%, a sample of unsized 1/4-in. material should contain at least 2.41 lb.

From the data on page 22, it will be seen that one sample of the series, No.105, shows a variation greater than the allowable four times the probable error. Since only one of these

occurs, and it has been shown that the laws of simple sampling do apply to the sampling of the mixture of coal and shale used, it is safe to assume that this one result was the one which might be expected once in 142 times. Therefore the series of data may be accepted as approximately correct. The probability curve for this series is shown in Fig. 3, page 27.

The worst possible case to be met with, in which the shale is the same size as that of the largest particles of coal, was considered next. As stated above, it was believed that by using the number of particles of shale in each sample as determined in each case by careful counting, and the weight per particle, as determined above, the ash content could be calculated with sufficient accuracy to obviate the necessity of making a large number of analyses. However, when it came to checking these calculated values with values determined by analysis for a number of the samples, it was found that the variations were so extremely great in several cases that it would be impossible to use the calculated values. A comparison of these values is shown on page 23.

In some cases the calculated values were higher than the others and in some were lower. In only two cases were they higher, and in each of these the variation was considerable, so it seems probable that they were due to some external factor. The most reasonable explanation is that during the process of sampling, several particles of shale became broken, so that in counting, these smaller broken particles were given the same weight as the normal-sized particles, thus causing the number of particles

used in calculating the ash to be excessive. This in turn would cause an excessively large ash value. The large negative variations might be accounted for by an increase in the moisture content of the shale during sampling. An analysis of the shale shows less than one per cent of moisture, which is very low, so it is not unreasonable to believe that more moisture was taken up from the air. It is also possible that in the two cases observed the shale particles happened to be larger or heavier than the average. This would tend to cause a negative variation.

As explained in the theory, it is possible to determine the theoretical probable error in sampling by the use of the expression,

$$E_p = 0.6745 \sqrt{npq},$$

in which "n" is the number of particles in the sample, "p" is the proportion of one constituent, and "q" is the proportion of the other. In the samples for case No.3, "p" and "q" are known, and "n" can be calculated if one assumption is made. This is, that float coal of all sizes ranging from 1/4-in. to zero will act approximately the same in sampling as if it were all of one size,— in this case 1/4-in. in diameter. If this is the case, the number of particles of coal can be found by dividing the weight of coal by the average weight of a particle of 1/4-in. coal. This number, added to the number of particles of shale as determined by counting, gives the total number of particles in the sample series. The average number "n" for any one sample is 1/16 of this. From the data tabulated on page 24 the probable error is found to be 11.48.

The probable error involved, as shown by the counting of the shale particles, was calculated in the usual manner from the

data tabulated on page 25. This error was found to be 20.14, which is considerably greater than the theoretical error of 11.48. That it is greater is probably due, at least in part, to the breakage of shale, whereby some particles were counted twice, and others were probably broken so fine that they were not counted at all. This would tend to give a larger probable error than the theoretical one. The fact that not enough samples were taken might alone be sufficient to explain this difference. The probability curves illustrating this are shown for the series of calculated ash from counting particles,in Fig. 4, page 27. The curve for series of analyzed ash is shown in Fig. 5, on the same page.

The experimental probable error for case No.3, was determined from the data tabulated on page 26, and it was found to be 0.3986. For the normal case of sampling, in which the impurities range in size from that of the largest sized particles of coal to zero, the probable error was found to be 0.2933. In the discussion above it was pointed out that this error was too high, and a standard error of 0.25 was selected. Using this error as the basis of calculation, it was shown that the original sample should have been 2.41 lb. Since the error involved in case No.3 is still greater, it is evident that an even larger sample should be taken. This actual size may be determined as before:

$$x : (.3986)^2 :: 1.76 : (.25)^2.$$

or $x = 4.51$ lb.

Then, having the same probable error, the ratio of sizes of sample for the two different types of mixtures would be $\frac{4.51}{2.41}$, which is equal to 1.74. That is, a sample of sized coal, one in

which the free impurities are all nearly the same size as the maximum size of coal, should be about 1.7 times as large as one in which the impurities are of all sizes. This ratio is merely a tentative one for the type of mixture used, and would by no means hold for all cases. It is, however, at least indicative of the true state of affairs, and it gives something more definite for consideration than the mere statement that a sample should be larger or smaller, as the case may be.

Using these values for weight of samples as a basis, it is possible to compute the size of sample required for coal of different sizes. It is evident that the accuracy of sampling depends upon the number of particles present in the sample. The number of particles varies inversely as the cube of their respective diameters, so

$$w_2 : (s_2)^3 :: w_1 : (s_1)^3$$

or $$w_2 = w_1\left(\frac{s_2}{s_1}\right)^3.$$

where

w_1 weight of sample of smaller size of coal.

w_2 " " " " larger " " " .

s_1 size of largest particles in w_1.

s_2 " " " " " w_2.

If a sample of 1/2-in. coal is required,

then

$$w_2 = 2.4\left(\frac{0.5}{0.25}\right)^3 = \underline{19.2 \text{ lb.}}$$ (See page 36.)

--------------:--------------

V. CONCLUSIONS.

(1) In order to be reasonably certain that results, secured in sampling coal which contains about 9 % of free impurities, will not differ from the true values by more than 1 %,

(a) about 2.4 lb should be taken for a sample of unsized coal with a maximum size of 1/4-in; and

(b) about 4.5 lb should be taken for a sample of sized coal with a maximum size of 1/4-in.

(2) A definite ratio exists between the sizes of sample required for sized and unsized coal with the same maximum size of particles. For a coal with about 9 % of free impurities, this ratio is about 1.74.

(3) Figuring upon a basis of about 9 % of free impurities, the sizes of sample required for the various sizes of sized and unsized coal prepared in Illinois, as well as a few intermediate sizes, have been computed, and are tabulated herewith. (See page 36). These are about the sizes of samples that should be taken if the experimental error is to be kept within 1 %. It is seldom that samples of coal taken from the car or at the mine are large enough. A mere glance at the accompanying table will show the reason for the extreme variations that often occur between analyses of even duplicate samples of this sort.

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SUGGESTED SIZE OF SAMPLES.

Trade Name	Size of Coal Inches	Size of Sample in Pounds.	
		Unsized Coal.	Sized Coal.
Egg	6	33,180	57,750
No.1	3	4,150	7,350
	2	1,230	2,180
No.2	1-3/4	830	1,540
No.3	1-1/4	300	560
	1	155	270
No.4	3/4	65	120
	1/2	20	34
No.5	1/4	2.4	4.5

--------0--------