

## REPORT OF THE COMMITTEE ON COAL ANALYSIS.

*To the President and Members of the American Chemical Society:*

SOME time ago a preliminary report of this committee was presented to the society.<sup>1</sup> The committee desire, now, to present the following as their final report. In doing this, they are well aware that more experimental work on some points in the report is very desirable. But no member of the committee is able, at present, to give much further time to the matter and it is felt that nothing can be gained by further delaying the report.

In presenting the report the committee wish to express their thanks to those who have so kindly aided the committee by their criticisms and otherwise; especially to Mr. W. H. Clingerman, of the Frick Coke Co., who furnished samples of the Pennsylvania coking coals; to Mr. R. C. Hills, of the Colorado Fuel and Iron Co., of Denver; to Mr. William Kent, of the *Engineering News*; to Mr. William Glenn, of the Baltimore Chrome Works; to Professor N. W. Lord, of Ohio State University, Columbus; to Mr. G. L. Heath, of the Calumet and Hecla Smelting Works, South Lake Linden, Mich; to Mr. Geo. H. Eldridge, of the U. S. Geological Survey; and to Messrs. R. W. Atkinson and A. J. Atkinson, of Cardiff, Wales.

### METHODS OF ANALYSIS.

1. *Sampling*.—In sampling from cars proceed as follows: Beginning at one corner of the car, drive a scoop-shovel vertically down as deep as it will reach. Bring it out with all the coal it will hold and throw into a cart or wheelbarrow. Repeat, taking six scoopfuls along one side of the car, at equal intervals, six through the center and six along the other side. Place the coal taken on a close, tight floor. Break all lumps larger than an orange. Mix by shoveling it over on itself, back and forth. Quarter, and reject opposite quarters. Break finer, as may be necessary, and continue to quarter down till a sample is obtained small enough to go into a quart fruit jar and having no pieces larger than one-fourth inch cube. The sample may, with advantage, be run rapidly through a mill which will break it to

<sup>1</sup> This Journal, 20, 281.

the size mentioned. Transfer to the jar and make sure the latter is sealed air-tight before it is set aside. All of these operations should be conducted as rapidly as possible to guard against any change in the moisture content of the coal.

Modifications of this method of sampling will, of course, suggest themselves, and in some cases will be necessary. When possible, a more representative sample may be secured by taking shovelfuls of the coal at regular intervals during the loading or unloading of the car. In any method of sampling, two conditions must be insisted on; the original sample should be of considerable size and thoroughly representative, and the quartering down to an amount which can be put in a sealed jar should be carried out as quickly as possible after the sample is taken. Unless the coal contains less than two per cent. of moisture, the shipment of large samples in wooden boxes should be avoided.

In boiler tests, shovelfuls of coal should be taken at regular intervals and put in a *tight, covered* barrel, or some air-tight covered receptacle, and the latter should be placed where it is protected from the heat of the furnace.

In sampling from a mine, the map of the mine should be carefully examined and points for sampling located in such a manner as to fairly represent the body of the coal. These points should be placed close up to the advancing crescent-shaped front of the workings. Before sampling, make a fresh cut of the face from top to bottom to a depth which will insure the absence of possible changes or of sulphur and smoke from the blasting-powders. Clean the floor and spread a piece of canvas to catch the cuttings. Then, with a chisel, make a cutting from floor to roof, say three inches wide and about one inch deep. Do not chisel out the shale or other impurities that it is the practice at that mine to reject. Measure the length of the cutting made but do not include the impurities in this measurement. With a piece of flat iron and hammer break all pieces to quarter inch cube or less, without removing from the cloth. Quarter down and transfer to a sealed bottle or jar. For the "run-of-mine" sample, samples taken at several points in this manner should be mixed and quartered down. If the vein varies in thickness at different points, the samples taken at each point should correspond in amount to the thickness of the vein. For instance, a small

measure may be filled as many times with the coal of the sample as the vein is feet in thickness. Should there appear differences in the nature of the coal, it will be more satisfactory to take, in addition to the general sample, samples of such portions of the vein as may display these differences.

For analysis quarter down further to about 100 grams. Run this portion through a mill which admits of quick grinding with little exposure to the air. A coffee-mill set to grind as finely as possible will answer. The grinding of 100 grams is recommended because less water will be lost than if a smaller sample is ground.

When an accurate determination of moisture is required and especially with coals high in moisture a portion of this coarsely ground sample must be transferred at once to a tightly stoppered tube for use in determining moisture.

Grind twelve to fifteen grams of the remainder moderately fine in a porcelain or iron mortar and transfer to a tightly corked tube for use in other determinations than that of moisture.

The committee wish to urge very strongly upon chemists and engineers the importance of careful sampling and of *careful treatment of the samples* in order to secure reliable results, especially in the determination of moisture.

That other factors must also be taken into consideration, in some cases, is illustrated by the following occurrence, cited by Mr. R. C. Hills.

"A railway manager had a car of coal from the South Platte field sampled and got me to analyze it. The sample was taken in the summer time and although a lignite, containing, usually, twelve per cent. of moisture, it only afforded ten per cent. Later on he concluded to try a car from the Franceville field, also lignite, affording in the summer time about fifteen per cent. of moisture. The sampling was done in the latter part of November and I obtained about twenty-one per cent. out of it. In both cases determinations were made elsewhere. The result was great injustice to the Franceville operators and to the best interests of the railway. Evidently there was accordance between the chemists on each sample but had the conditions under which the samples were taken been reversed each would have varied several units from the values found."

A car-load of many western coals may lose several hundred

pounds of moisture daily while standing on the track and the same coal may lose several per cent. of moisture by standing for a few days or weeks in a loosely stoppered bottle.

2. *Moisture*.—Dry one gram of the coal in an open porcelain or platinum crucible at 104°–107° for one hour, best in a double-walled bath containing pure toluene.<sup>1</sup> Cool in a desiccator and weigh covered.

With coals high in moisture, and in all cases where accuracy is desired, determinations must be made both with the coarsely ground and with the powdered coal. When, as will usually be the case, more moisture is found in the coarsely ground than in the powdered coal, a correction must be applied to all determinations made with the latter. Thus, if one per cent. more of moisture is found in the coarsely ground sample a total of one per cent. must be subtracted from the quantities of the other constituents as determined with the powdered sample. Or, in the form of a rule: Divide the difference in moisture by the per cent. of other constituents than moisture as found in the powdered coal. Multiply the per cent. of each constituent as found in the powdered coal by the quotient and subtract the resulting product from the amount of the given constituent.

Thus, suppose the results of an analysis give:

	Coarsely ground coal.	Powdered coal.
Moisture	12.07	10.39
Volatile combustible matter	....	34.25

then the correction factor will be

$$\frac{12.07 - 10.39}{100 - 10.39} = \frac{1.68}{89.61} = 0.0187$$

and the true per cent. of volatile combustible matter will be

$$34.25 - (34.25 \times 0.0187) = 33.61.$$

It is possible that volatile combustible matter and ash may be determined with the coarsely ground coal without serious error but we have not enough data at our command to warrant such a recommendation.

The toluene bath is recommended for convenience, but any other bath at the proper temperature will answer equally well. In all cases recorded below, the coals gained in weight, probably

<sup>1</sup> Victor Meyer: *Ber. d. chem. Ges.*, 17, 2999.

from oxidation, after one hour's heating, so that longer heating is not only unnecessary but undesirable. A higher temperature appears also to be undesirable (see below).

The results of Dr. Hillebrand as to the loss of moisture *in vacuo* over sulphuric acid, recorded in our first report, have been confirmed by the following results obtained by Mr. N. M. Austin at the Rose Polytechnic Institute. The results are in most cases, averages of from two to six determinations.

No.	Toluene. First hour. 107° C.	Toluene. Second hour. 107° C.	Xylene. First hour. 138° C.	Xylene. Second hour. 138° C.	In vacuo over sulphuric acid. Seven hours.	In vacuo over sulphuric acid. Twenty-four hours.	In vacuo over sulphuric acid. Sixty-four hours.
1	0.97	0.92	1.03	0.98	1.17	....	1.18
2	4.55	4.61	4.60	4.56	4.69	....	5.05
3	1.39	1.27	1.27	1.15	1.46	1.45	1.56
4	1.31	1.29	1.27	1.20	1.36	1.38	1.39
5	1.00	0.97	0.97	0.96	1.10	1.14	....
6	1.44	1.31	1.22	1.15	1.46	1.49	1.56
7	14.53	14.23	14.85	14.18	14.91	15.47	15.25
8	8.43	8.21	8.31	8.12	8.91	8.90	8.95
9	1.34	1.32	1.23	.	....	....	....
10	10.50	....	....	.	11.04	11.55	....

While it is evident that more water, and doubtless, more nearly the amount of water actually present, can be obtained by drying for twenty-four hours *in vacuo* over sulphuric acid than by drying for one hour at 104°-107°, the committee are not prepared to recommend so radical a departure from common usage for general adoption. They believe that drying *in vacuo* should be used when great accuracy is desired and in all cases where the determination is to be combined with an ultimate analysis, but that so long as the conditions of sampling are not more carefully controlled than at present, the difference between the two methods (average 0.37 per cent.) is not great enough to justify the general adoption of the new method.

The small difference found between the determinations at 107° and those at 138° (0.03 per cent. more at the lower temperature on the average) indicates that a variation of several degrees in the temperature of the bath will have no appreciable effect on the determination, provided the temperature is above the boiling-point of water.

A series of comparative determinations made in porcelain and platinum crucibles gave slightly higher values, on the average 0.12 per cent., in the porcelain. This was probably due to the greater depth of the platinum crucibles, causing the moisture to diffuse away more slowly.

For coals containing less than two per cent. of moisture, satisfactory results can be obtained by drying for twenty-four hours in a watch-glass over sulphuric acid, at atmospheric pressure (C. B. Dudley). For lignites, and for coals containing a high per cent. of moisture, the method is not satisfactory.

The following determinations made by the chairman of the committee demonstrate the impossibility, under some laboratory conditions, of grinding coal in a mortar without serious loss of moisture. In these determinations about 100 grams of the coal were ground in a coffee-mill and then ten to twenty grams of this coarsely ground coal were ground as quickly as possible in an iron mortar and transferred at once to a stoppered tube.

No.	Temperature of laboratory.	Ground once in a coffee-mill.		Ground twice in a coffee-mill.		Ground in an iron mortar.	
		16 to 20 grams, one hour at 135°.	1 gram, one hour at 107°.	16 to 20 grams, one hour at 135°.	1 gram, one hour at 107°.	1 gram, one hour at 107°.	1 gram, one hour at 107°.
1	88° F	{ 12.07 12.14	11.70	....	....	{ 10.39 11.09	
1	76° F	....	....	11.96	11.92	11.43	
2	88° F	12.32	..	....	....	11.05	
3	77° F	13.61	13.85	13.38	13.67	12.83	
3	66° F	13.92	14.06	13.56	13.72	13.24	
4	78° F	1.33	1.22	1.33	1.16	1.03	

Coal No. 3 gave *in vacuo* over sulphuric acid for twenty-four hours:

	Per cent.
Ground once .....	14.03
Ground twice .....	13.88
Ground in mortar .....	13.62

Coal No. 4 gave on further drying *in vacuo* for twenty-four hours after heating for one hour at 107°:

	Per cent.
Ground once .....	1.26
Ground twice .....	1.24
Ground in mortar .....	1.07

A large amount of additional work on the subject of moisture, with coals of a greater variety, seems desirable. Enough has been done, however, to demonstrate that a close approximation to the truth can be obtained only by the most careful treatment of the samples. Every coal analyst, who has not given careful attention to the question, should convince himself by comparative determinations that the methods which he uses do not cause serious loss of water before the determination is made.

*Volatile Combustible Matter.*—Place one gram of fresh, undried, powdered coal in a platinum crucible, weighing twenty or thirty grams and having a tightly fitting cover. Heat over the full flame of a Bunsen burner for seven minutes. The crucible should be supported on a platinum triangle with the bottom six to eight cm. above the top of the burner. The flame should be fully twenty cm. high when burning free, and the determination should be made in a place free from draughts. The upper surface of the cover should burn clear but the under surface should remain covered with carbon. To find "Volatile Combustible Matter" subtract the per cent. of moisture from the loss found here.

The method recommended in our first report, and given here without change, has been the occasion of considerable discussion within the committee, and also by others. Very little experimental evidence has been submitted to us, however, and that obtained by Mr. Austin at the Rose Polytechnic Institute appears to be decidedly in favor of the method given (see below).

The method differs from that of Muck<sup>1</sup> in placing the crucible six to eight cm. above the burner instead of three cm. and in continuing the heating for a definite length of time. The flame is much hotter at six to eight cm. above the burner than at three cm., and, if the full flame is used, as directed, the crucible will be sufficiently well covered by it. The time limit corresponds more nearly to present practice in America and is likely to give more concordant results than Muck's direction to stop heating with the disappearance of flame at the edge of the lid of the crucible. The time limit is that proposed by Hinrichs, but the use of a blast is omitted.

In discussing the subject, it should be distinctly understood,

<sup>1</sup> *Chemie der Steinkohle*, p. 10.

at the outset, that the term "Volatile Combustible Matter" does not represent any definite compound or class of compounds which exist in the coal before heating. Mr. Austin's results demonstrate that some of the carbon which escapes in the form of volatile compounds on rapid heating, is separated in the free state and remains as "Fixed Carbon" when the first heat is applied slowly. It is well known, too, that many of the compounds contained in the gas and tar formed by heating bituminous coals are decomposed by heat with the formation of free carbon. The "Fixed Carbon" retains a considerable amount of hydrogen which is progressively expelled by heating to higher and higher temperatures, until, at temperatures approaching that of the electric arc, it all escapes and the carbon remaining crystallizes in the form of graphite. It follows, therefore, that, no matter what method of heating, or what temperatures are used, the determination must be an arbitrary one.

The two considerations which should have greatest weight in the selection of a method appear to be that the method chosen should, if possible, give results which approximate closely to those obtained in the commercial manufacture of coke from the same coals, and that the method should be one which can be accurately described so that different operators will secure concordant results with samples of the same coal. No experimental evidence on either point has been furnished the committee. Some evidence upon the first point is furnished by Mr. Austin's work below, and the results are certainly favorable to the method which we propose.

The most serious objection which has been raised against the method is the claim that certain non-coking coals suffer mechanical loss from the rapid heating. No evidence as to the extent of such loss has been given. The following determinations of ash in the original coal, and in the coke obtained by the method recommended, prove that in the case of these coals, at least, any mechanical loss which occurred must have been insignificant. Coals 5, 6, 7, and 8 were coking coals; 10 was a non-coking coal from Colorado.



	Ash from coal.	Ash from coke.
5	{ 4.74 { 4.73	4.75
6	{ 5.43 { 5.35 { 5.39	5.43
7	{ 2.32 { 2.37	2.32
8	13.52	13.52
10	10.62	10.60

Further determinations of this kind with non-coking coals are very desirable, but, even if a small amount of mechanical loss is found to occur in such cases, it does not follow that a different method should be used for them. Since the determination is, in any case, an empirical one, uniformity in the method for all cases is extremely desirable.

One chemist has furnished us with data which show that he has obtained, with the use of a Rose crucible in a muffle, results which agree closely with those obtained by the method proposed by the committee. Further evidence upon this point is very much desired, as the exigencies of many laboratories demand that the determinations should be made in a muffle.

The following determinations of coke by different methods were made by Mr. N. M. Austin. The coals used were:

3. Coking coal, Leith Mine, Pa.
4. Coking coal, Mammoth Mine, Pa.
5. Coking coal, McClure Mine, Lemont, N. Y.
6. Coking coal, Valley Mines, Pa.
7. Lancaster semiblock coal, Owen Co., Indiana.
8. Coking coal used at the Terre Haute Water Works.
9. Coking coal from Hantana, Tennessee.
10. Non-coking coal from Colorado.

Coals 3, 4, 5, and 6 were furnished by Mr. Clingerman of the Frick Coal Co., with the statement that these coals run very evenly for the production of coke, and give a yield of sixty-eight to seventy per cent.

No.	Seven minutes, full flame. Wet coal.	Seven minutes full flame. Dry coal.	Five minutes, inch high flame. Five minutes, full flame.	Three and a half minutes, full flame. Three and a half minutes, over blast.
3	{ 67.33 67.36	{ 67.03 67.50 67.62 67.96	71.55	66.52
4	{ 68.96 69.02	68.92	73.41	67.92
5	{ 68.55 68.31	67.78	72.88	67.71
6	{ 68.97 68.90	68.99	{ 73.60 72.92	67.41
7	{ 48.82 49.27	{ 50.30 50.46	54.32	46.91
8	{ 53.70 53.65	53.57	59.00	51.58
9	{ 65.68 65.94	65.75	70.30	65.35
10	41.48	{ 41.55 37.40	45.43	39.31

The moisture in these coals is given on p. 1120. The agreement with the commercial coking value for the Pennsylvania coals must be considered as satisfactory. The effect of slow heating in increasing the amount of coke is very evident. The amount of coke, or of fixed carbon, by slow heating was, on the average, 4.58 per cent. higher than by the rapid heating. That this is not due to mechanical loss is proved by the ash determinations above.

In using the blast, the flame of the blast was applied before the flame of the Bunsen burner was removed. The results are, on the average, 1.38 per cent. lower than with the Bunsen alone. It is believed that this is not due entirely to the expulsion of volatile matter, but, in part, to the fact that the flame of the blast is more strongly oxidizing and carbon dioxide and water-vapor enter the crucible from it in larger amount. The fact that the western coals, 7, 8, and 10, with their more porous, and, in the last case, pulverulent coke, show much greater differences than the others, supports this view. The results with the Bunsen alone approach more nearly to the commercial coking values, and it is believed, too, that they are much more likely to give concordant results in the hands of different operators.

Little difference is shown in the average between the results with the original wet coal and with the dry coal. The use of the wet coal is recommended because the drying causes changes in the coal otherwise than by expelling the moisture, and because undried coal is always used in the manufacture of coke. No time is lost by such a procedure, since the ash can be determined much more quickly with the coal used for the moisture determination than with the coke. It took four hours to burn the coke to ash in a platinum crucible, while the dry coal may be burned in a porcelain crucible in two hours or less.

The results of Messrs. Meade and Attix published in this Journal (21, 1137) seem to show very clearly that the heating of coke or anthracite coal in a crucible with the blast, causes a very considerable loss by oxidation. It is probable that heating with the blast in an atmosphere of nitrogen or hydrogen is the most satisfactory method for materials of this class, but further comparisons of the results obtained in that way with the results obtained by the method recommended by the committee are very desirable.

*Ash.*—Burn the portion of powdered coal used for the determination of moisture, at first over a very low flame, with the crucible open and inclined, till free from carbon. If properly treated, this sample can be burned much more quickly than the dense carbon left from the determination of volatile matter. It is advisable to examine the ash for unburned carbon by moistening it with alcohol.

When the sulphur in the coal is in the form of pyrites, that compound is converted almost entirely into ferric oxide in the determination of ash, and, since three atoms of oxygen replace four atoms of sulphur, the weight of the ash is less than the weight of the mineral matter in the coal by five-eighths of the weight of the sulphur. While the error from this source is sometimes considerable, the committee does not recommend such a correction for "proximate" analyses. When analyses are to be used as a basis for calculating the heating effect of the coal a correction should be made (see below).

5. *Fixed Carbon.*—This is found by subtracting the per cent. of ash from the per cent. of coke as found in paragraph 3. Sulphur, which passes partly into the "Volatile Combustible

Matter" and partly into the coke, is not considered in the calculation.

6. *Sulphur*.—Eschka's method is recommended for general use. The following directions, which are given for the convenience of those using this report, are those of G. L. Heath<sup>1</sup> with slight modifications.

Mix thoroughly, one gram of the finely powdered coal<sup>2</sup> with one gram of magnesium oxide and one-half gram of dry sodium carbonate, in a thin platinum dish having a capacity of 75 to 100 cc. A crucible may be used but a dish is preferred. The magnesium oxide should be light and porous, not a compact, heavy variety.

The dish is heated on a triangle over an alcohol lamp, held in the hand at first. *Gas must not be used*, because of the sulphur it contains. The mixture is frequently stirred with a platinum wire and the heat raised very slowly, especially with soft coals. The flame is kept in motion and barely touching the dish, at first, till strong glowing has ceased, and is then increased gradually till, in fifteen minutes, the bottom of the dish is at a low, red heat. When the carbon is burned, transfer the mass to a beaker and rinse the dish, using about fifty cc. of water. Add fifteen cc. of saturated bromine water and boil for five minutes. Allow to settle, decant through a filter, boil a second and third time with thirty cc. of water, and wash till the filtrate gives only a slight opalescence with silver nitrate and nitric acid. The volume of the filtrate should be about 200 cc. Add one and a half cc. of concentrated hydrochloric acid, or a corresponding amount of dilute acid (eight cc. of an acid of eight per cent) Boil till the bromine is expelled and add to the hot solution, drop by drop, especially at first, and with constant stirring, ten cc. of a ten per cent. solution of barium chloride. Digest on the water-bath, or over a low flame, with occasional stirring till the precipitate settles clear quickly. Filter and wash, using either a Gooch crucible or a paper filter. The latter may be ignited moist in a platinum crucible, using a low flame till the carbon is burned.

In the case of coals containing much pyrites or calcium

<sup>1</sup> This Journal, 20, 630.

<sup>2</sup> With coals high in moisture a correction may be necessary on account of the loss of water in powdering the coal. (See above under moisture.)

sulphate, the residue of magnesium oxide should be dissolved in hydrochloric acid and the solution tested for sulphuric acid.

If desirable, the burning of the coal with Eschka's mixture may be carried out in a muffle, from twenty to thirty minutes being required.<sup>1</sup>

The following determinations made by Geo. Steiger, under the direction of Dr. Hillebrand, demonstrate the necessity of examining the residue of magnesium oxide and ash for sulphur. In these determinations both ammonium nitrate and bromine water were used.

	Sulphur extracted by water.	Additional sulphur ex- tracted by hydrochloric acid.	Total sulphur.
1	4.447	0.114	4.561
2	0.744	0.057	0.801
3	0.613	0.037	0.650
4	1.888	0.111	1.999
5	2.652	0.067	2.719
6	1.392	0.082	1.472
7	1.879	0.083	1.962
8	0.957	0.041	0.998
9	0.593	0.032	0.625

A mixture of five grams of the sodium carbonate and ten grams of the magnesium oxide should be boiled several times with water, the solution filtered and acidified, the amount of sulphuric acid present, if any, determined, and the appropriate correction applied. The operator should, of course, satisfy himself that the bromine water and hydrochloric acid used are free from sulphur.

The following method of R. W. and A. J. Atkinson deserves consideration from those who find it more convenient to work in a muffle. The method has also been applied to iron ores.<sup>2</sup>

One gram of the finely ground coal or coke is mixed thoroughly with five grams of dry sodium carbonate, spread evenly over the bottom of a flat or shallow platinum dish, and the latter placed on a rectangular rest made of clay-pipe stems inside of a muffle which, though hot, is still black. The temperature of the muffle should be raised gradually during half an hour to clear cherry-redness and then kept at the latter temperature for ten or fifteen minutes. The sodium carbonate should not sinter or fuse.

<sup>1</sup> Rothe: *Stahl und Eisen*, XII, 31 (1894).

<sup>2</sup> *J. Soc. Chem. Ind.*, March 29, 1886; *J. Iron and Steel Inst.*, No. 2 (1896).

The mass should not be stirred. When the carbon is burned, usually in about forty-five minutes in all, cool, digest with 100-120 cc. of warm water, allow to settle, decant through a filter and wash twice by decantation and then on the filter, adding a few drops of a solution of pure sodium chloride, if the residue tends to pass through the filter. The filtrate is acidified with twelve cc. of concentrated hydrochloric acid and precipitated with barium chloride as above. No oxidizing agent is required.

R. W. Atkinson has demonstrated that the evaporation with hydrochloric acid to remove silica, which some recommend, is unnecessary. His statement is confirmed by the experience of one of the members of the committee in similar cases.

A few determinations made by this method under the direction of one of the members of the committee have given good results, but no careful examination of the method has been made.

7. *Ultimate Analysis.*—It seems to be unnecessary to give directions for the determination of carbon, hydrogen, and nitrogen here. In determining carbon and hydrogen, lead chromate or some other means for retaining sulphur must, of course, be used. The amount of nitrogen is so small that the use of a copper spiral is not necessary.

The method to be used in calculating the oxygen of the coal presents, perhaps, the question of greatest difficulty. If we could be sure that all of the sulphur is present in the form of pyrites, and that this is converted into ferric oxide in the ash, the oxygen should be found by subtracting from 100, the sum of carbon, hydrogen, nitrogen, ash and *five-eighths* of the sulphur. This is probably the safest rule which can be given for general use, and especially for coals high in sulphur. The operator should, however, satisfy himself as to whether the ash is practically free from sulphates, and, if possible, whether the sulphur is mainly in the form of pyrites. If necessary, the rule should be modified, in particular cases, accordingly.

8. *Heating Effect.*—In the preliminary report the recommendation was made that the heating effect be given on the basis of the coal burned to vapor of water at 100° C. After some criticism from others and further consideration, we have concluded to recommend that results be given for the coal burned

to liquid water at the ordinary temperature. The reasons for this recommendation are that this appears to be the common practice in this country, and because coals are burned to liquid water in the bomb calorimeter, which undoubtedly furnishes the best determinations of heating effect at present available. Engineers and others will, of course, understand that the heating effect, when stated in this manner, includes three and a half to four per cent. of heat which can never be secured under the conditions of practical use.

The most reliable formula for the calculation of the heating effect of a coal burned to liquid water is that of Dulong, which gives the calorific power in calories per kilogram.

$$\text{Calorific power} = 8080C + 34,460 (H - \frac{1}{8}O) + 2250 S.$$

For the calculation of the oxygen, see the paragraph on ultimate analysis.

The calorific power in British Thermal Units per pound may be found by multiplying that in calories per kilogram by nine-fifths.

The theoretical evaporative effect is to be calculated by dividing the number of calories per kilogram by 536, or the number of British Thermal Units per pound by 965, and subtracting from the result one-seventh more than the amount of water formed by burning one kilogram of the coal. The addition of one-seventh is given because the liquid water, on the basis of which the heating effect is given, must be considered as changed from water at ordinary temperature to steam at 100° C. The amount to be subtracted may be taken as 0.55 for most bituminous coals. The result gives the theoretical number of kilograms, or pounds, of water converted into steam from, and at, 100° C by one kilogram, or pound, of coal.

The rule given, tentatively, in our preliminary report for the calculation of heating effect, from the amount of combustible matter present in bituminous coals, has been found to be of limited application.

The following averages for the heating effect of the combustible matter of a series of bituminous coals, and for the composition of the combustible matter which they contain, have been calculated from the results obtained by Professor N. W.

Lord and Mr. F. Haas<sup>1</sup> with a bomb calorimeter. The combustible matter for the calculations included carbon, hydrogen, nitrogen, and oxygen. The oxygen was calculated on the supposition that the sulphur was present in the form of pyrites. (See paragraph 7.) The calorimeter result was corrected by subtracting the heating effect of the sulphur. The sulphur averages are given, though it was not included in the combustible matter. The results are for the coals burned to liquid water.

No of samples.	S.	C.	H.	O.	N.	Calories per kilo-gram by bomb calorimeter.	Calories per kilo-gram, calculated.
3	1.59	80.63	5.40	12.26	1.71	7860	7847
2	3.01	82.15	5.36	10.97	1.53	8148	8012
9	2.20	83.66	5.51	9.33	1.51	8255	8255
15	2.29	83.97	5.60	8.79	1.66	8372	8335
2	1.28	85.93	5.47	7.06	1.54	8508	8523
3	0.59	91.28	4.52	3.29	0.91	8783	8792

The agreement between the calorimeter results and those calculated by the formula indicates that the latter are sufficiently accurate for most purposes, when calorimeters are not available.

Professor Lord concludes from his work<sup>2</sup> that the heating effect of the combustible matter of coal from the same seam over wide areas, is nearly identical. If the heating effect has been determined for several samples of the coal a factor similar to those in the last two columns above may be calculated and this may be used with a good degree of accuracy for the calculation of the heating effect of other samples from the same seam. For the purpose of such calculations it is recommended that the combustible matter of the coal be calculated by subtracting from 100 the per cents. of ash and moisture and one half of the sulphur. If the sulphur were all in the form of pyrites five-eighths of the sulphur should be subtracted but the rule given is considered sufficiently accurate for the purpose in question.

In a similar manner, if the percentage composition of the combustible matter in a seam of coal has been determined, this

<sup>1</sup> Trans. A. I. M. E., 27, 259.

<sup>2</sup> *Loc. cit.*



may be made the basis for the calculation of a factor which can be applied for the calculation of the heating effect of other samples from the same seam.

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## LEMON FLAVORING EXTRACT AND ITS SUBSTITUTES.

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AS many valuable papers have lately appeared upon the subject of vanilla flavoring extract, while little has been done upon lemon extract and its substitutes, I have ventured to here outline the results of the investigation of these goods as found upon the market, and made in the course of work for the Wisconsin Dairy and Food Commission.

Lemon extracts, following the U. S. P. formula for "Spiritus Limonis" should contain at least five per cent of oil of lemon dissolved in deodorized alcohol and should be colored only with the coloring from lemon peel.

A preliminary examination of the extracts supplied by grocers showed them to contain alcohol in amounts varying from four-teen to ninety-four per cent. by weight and oil of lemon from none to eight per cent.

The extracts low in alcohol had in many cases a fine aroma derived from agitation with new oil of lemon in some cases but more frequently produced by citral and the so-called "soluble oil of lemon" or from lemon-grass or citronella aldehyde and frequently with the addition of tincture of nutmeg, mace, or capsicum. The cheaper grades contained so little oil in solution that the addition of water frequently failed to produce turbidity.

As no standard methods were in use for the examination of lemon flavoring extracts, it was thought desirable to make an examination of the oils used. Optical methods proved most satisfactory for this purpose. The following table gives the behavior of pure oil of lemon and of the various oils and substances entering into the composition of adulterated and sophisticated lemon extracts.