

siderably by the presence of 1 per cent. or more of sucrose; only slightly by the presence of 5 per cent. of raffinose.

Sucrose also accelerates the osazone precipitation in dilute solutions of fructose, but as these react much more rapidly than the corresponding glucose solutions, the effect of the sucrose is scarcely noticeable.

Maltose retards the precipitation of glucosazone, interfering much more seriously in the case of glucose than in the case of fructose.

Lactose interferes with the glucosazone test in a similar manner and to a greater degree than maltose.

The bearing of these results upon the analytical application of the osazone reaction is too evident to require detailed discussion.

We plan to continue this work by studying the influence of maltose, lactose, and sucrose upon the yield and physical properties of glucosazone and later to extend the investigation to other substances and to the corresponding reaction with derivatives of phenylhydrazine.

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## THE SAMPLING OF COAL AND CLASSIFICATION OF ANALYTICAL DATA.

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IF A history of the sample of coal were given with the results of the analysis, it would be of great service, and if this becomes standard practice, one would be able to arrive at a more definite conclusion as to what an analysis represented. It is very often the case that the chemist analyzes a sample stated to be representative of a certain mine, seam, locality or kind of coal, and yet knows nothing regarding its selection; the result is, that he may present a report that is erroneous, because the sample may have been a picked one, better than is possible with the average product. Or, on the other hand, it may be an inferior one, with the result that in each case the chemist's work does not appear to the best advantage. A comparison of various published analyses of what is presumably the same coal, will show a most confusing series of results that cannot be explained by any assumption that the analytical methods differ. Many of the composi-

tions given, particularly in engineering text-books, are from analyses of coal used in boiler trials, and as it is quite often the case that selected fuel is used, it results in a value unduly high being assigned to the coal by those who do not know the source of the sample.

Often samples are taken from a seam in mines, and presented as representing the coal shipped to the consumer, when the more exact method would be to sample the product loaded for shipment. Or again, a seam may be sampled without it being stated whether the object was to determine its entire composition, or that portion supposed to be taken for shipment. It is the author's opinion that the sampling is as important as the analysis, and that the person receiving results of analysis should insist on a statement giving the history and description of the sample, and that the chemist would advance his interests by insisting on a full statement when practicable. At all events, if those who receive reports of analyses would use discretion in accepting results in the absence of such history and description of the sample, it would tend to the avoidance of confusion, and compel better practice.

Not only the interests of the consumer and purchaser demands better sampling methods, but the matter is of even greater importance to the dealer or producer. Assuming the heating power of semi-bituminous coal at 14,000 B. T. U. as a maximum value for coal, and screenings or "slack" at 9,000 B. T. U. as a minimum value, and that an unscrupulous dealer should substitute the latter for the former fuel, the purchaser would be the loser by 36 per cent. This would represent the maximum opportunity for the dealer to defraud the purchaser, because the coal producer would not cause the miners to add dirt to the fuel for the purpose of increasing the quantity, for the reason that he would have to pay more for mining rock and clay than for coal. Thus the producer, dealing with large quantities, cannot afford to purposely adulterate his goods. The unscrupulous purchaser, on the other hand, in the preparation of a sample—a very small quantity—is not limited by reason of cost in the quantity of foreign matter taken, even to the total exclusion of combustible, but assuming a composition of 90 per cent. foreign matter, the seller may be the loser by 89 per cent. Thus by carrying these considerations to their maximum ranges, there appears the possibility of greater variation in the quality of the sample, than in the fuel supply.

The sampling is fully as important, should be conducted under as reliable auspices, and safeguarded as much as the analysis.

#### DETERMINATION OF VOLATILE MATTER.

The author considers it most desirable and necessary that a more reliable method of volatilization be adopted. The chemist finds volatile matter in coke which has been some days at a considerable temperature in an oven. From this it may be concluded that an important interval of time and a high temperature are essential to the operation, but with present practice, if heating is continued beyond a certain unknown interval, the combustion of fixed carbon becomes greater in amount than that of the unexpelled volatile matter. For this reason it appears necessary to effect volatilization in an inert atmosphere that distillation may be more effective and without combustion of fixed carbon. The most that has been expected from the best established method is that it gives "concordant" results. It is the author's conviction, however, that such results are neither concordant nor correct, and he is now disposed to advocate the use of hydrogen as an inert atmosphere rather than nitrogen, because of its greater simplicity of production and purification.

#### FORMS OF ANALYSIS AND RELATIVE COMPOSITION.

The engineer requires the relative composition of the moist and dry coal, and what is often erroneously called the combustible, to be stated, while it is more often the practice of the chemist to present only the constituents and values of the moist coal. For reasons given later, it will appear that the engineer's requirements are justified, therefore such demands are recognized and considered in the following treatment.

From the standpoint of the proximate analysis, it is useful to consider coal in four divisions, and the relation is illustrated by the following three equations:

1.  $\left. \begin{array}{l} \text{Fixed carbon} \\ \text{Hydrocarbons} \\ \text{Sulphur} \end{array} \right\} + \left\{ \begin{array}{l} \text{Water of composition} \\ \text{Nitrogen} \end{array} \right\} = \left\{ \text{Pure coal.} \right.$
2. Pure coal + ash = Dry coal.
3. Dry coal + moisture = Moist coal.

The combination designated "water of composition" is proposed by Parr.<sup>1</sup> Following this scheme the author has adopted the arrangement presented in Tables I and II, wherein the moist,

<sup>1</sup> This Journal, 26, 294; Bull. Univ. of Ill., July 15, 1904.

dry, pure coal and combustible compositions are given. As shown, three divisions or analyses are presented, namely, usual proximate, what the author designates as an extended proximate, and an ultimate analysis of the pure coal, the elements being also presented in the other columns at their respective values. The author is indebted to C. H. McClure for the classification in the three latter divisions. It is not the author's intention to propose methods for analysis, but to offer a system of classification which will display relative values of different coal compositions, as shown in Tables I and II, and while these tables are sufficient in themselves to illustrate the proposition, it is well to present the source of the data used in compiling them, which represents two coal seams in Illinois.

The analytical work consisted of the usual proximate analysis, and the determination of total carbon according to Parr. Sulphur and nitrogen were separately obtained. Available hydrogen is calculated by  $\frac{\text{Total Carbon} - \text{Fixed Carbon}}{5}$ , the value for fixed carbon being obtained by the usual proximate analysis. This hydrogen ratio was proposed for Illinois coals by Parr, and is considered to be a constant, and its determination required analyses wherein carbon and hydrogen were obtained by combustion, but it may also be determined by  $H - \frac{O}{8}$ .<sup>1</sup> It is not the author's object to propose the acceptance of Parr's method of determining available hydrogen, but to insist upon recognition and presentation of the inert matter in the pure coal, for which there is as much reason as with that other inert matter, namely, the ash. In fact, as the determination of available hydrogen is dependent on his ratio, which is of itself dependent on an ultimate analysis, and as it is considered as a constant, it might be best to establish such factors directly by ultimate analysis.

Thus the source of the data for the compositions in the three divisions of Tables I and II is as follows:

Total carbon, method of Parr.

Fixed carbon, by usual proximate analysis.

Available hydrogen, calculated according to hydrogen ratio.

Water of composition, by difference.

<sup>1</sup> In some cases, at least, this formula does not give correct values for available hydrogen.—EDITOR.

Sulphur and nitrogen, by separate determinations.

Ash and moisture, by proximate analysis.

Heating power, by calorimeter.

The presentation of values in the four columns shows how and wherein the two seams differ, and illustrates their true relation. For example, the fixed carbon in the moist coal of Table I appears higher than in that of Table II, but an inspection of the pure coal columns which give the true relative values, shows that it is just the reverse, because the ash and moisture of the moist coal differ in amount; thus a proper comparison between the two coals cannot be made unless the values are presented on a common basis. Likewise, the values for available hydrogen show why the heating power of the combustible of No. I is higher than with No. II, and an explanation of the difference in heating power of the moist and dry coal is principally afforded by the ash and moisture of these compositions. Following the consideration of relative composition further, Table III is presented, wherein the essential features of the two coals are shown in parallel columns, from which the relation can be directly noted.

Inasmuch as Parr proposes a constant, that of the hydrogen ratio, the possibility of the more extended use of constants is presented, and the author urges the feasibility of considering the pure coal compositions as constants for a coal seam, or particular locality of such seam. This possibility has been suggested, principally by the fact that the heating power of the pure coal from a general locality does not vary over greater limits than that of the calorimetric method, and he has been able to employ it as a constant in calculating the heating power of dry and moist coal, having determined only moisture and ash, and obtained results that check with calorimetric determinations made on the same samples. The author, however, does not claim originality in this observation, but does insist that the use of such constants is of advantage.<sup>1</sup>

<sup>1</sup> A better method of calculating the pure coal would seem to be that suggested by the Committee on Coal Analysis, in this Journal, 21, 1131, where 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." This recommendation was based on the fact that the sulphur in coal is usually largely in the form of pyrites, and when in this form it is replaced in the ash by three-eighths of its weight of oxygen. If

It is a fact that coal from a general locality has been analyzed over and over again, with always a more or less different result when presented in only the moist coal composition, so that after all of the multiplicity of work, a final conclusion or full understanding is still unattained. It appears to the author, that instead of so much superficial work, it would be better that a limited amount of more thorough investigation be made, with the object of determining values of the combustible, and particularly the pure coal. There would then be more opportunity to consider the two great variables, namely, ash and moisture, and a number of such values could be collected, thus establishing standards. This would also be of great assistance in coal inspection service, because locality and coal seam being known, it would be essential to determine only ash and moisture, and specifications governing these constituents only need be drawn.

This view concedes that coal from a certain locality or seam does not vary in quality, but that the variation is due to the presence of ash and moisture, which are impurities associated with coal.

As illustrating the matter of treating the constituents and values of the pure coal as constants, the composition in Tables I and II are made up from values taken from different sources. Ash, moisture and heating power are the average of a number of samples taken from the full height of the two seams, and the determinations were made under the author's direction. Nitrogen was determined on the author's samples by another chemist. All other values are from work done by S. W. Parr on different samples from the same seams.

#### THE TERM PURE COAL.

The word combustible, used more particularly by engineers to designate fuel free from ash and moisture, is erroneous and confusing, therefore the author has adopted the term pure coal; the expression "coal free from ash and moisture" is more definite the combustible matter, or "pure coal," of the coal is calculated by this formula, it will be found that for a given seam of coal the heat of combustion of the combustible matter is very nearly constant. Thus for Coal 1 in the analysis given below the heat of combustion of the combustible matter is 14,747 B. T. U., while that for No. 2 is 14,457 B. T. U., and it would probably be found that the heat of combustion of the combustible matter of other samples of coal from these respective seams would agree very closely with these values.—EDITOR.

and self-explanatory, but it is composed of many words and is cumbersome. Objection has been made because it contains sulphur, which was regarded an impurity, but all coals contain it, and as the volatile portion must be considered as in chemical combination, it is necessarily always a constituent, and cannot be separated without destroying the combination. Thus the term pure coal is justifiable, and the ultimate conception of coal is this pure coal, having associated with it, but not in combination, the two impurities, moisture and ash, the fixed sulphur being considered one of the constituents of the ash.

TABLE I.

|                                 | Moist coal. | Dry coal. | Pure coal. | Combustible. |
|---------------------------------|-------------|-----------|------------|--------------|
| 1. Usual proximate analysis:    |             |           |            |              |
| Moisture.....                   | 6.29        | ....      | ....       | ....         |
| Ash.....                        | 8.74        | 9.33      | ....       | ....         |
| Volatile matter.....            | 34.91       | 37.25     | 41.08      | ....         |
| Fixed carbon.....               | 50.06       | 58.92     | 58.92      | ....         |
| 2. Extended proximate analysis: |             |           |            |              |
| Moisture.....                   | 6.29        | ....      | ....       | ....         |
| Ash.....                        | 8.74        | 9.33      | ....       | ....         |
| Carbon, total.....              | 68.06       | 72.63     | 80.11      | 93.90        |
| Carbon, fixed.....              | 50.06       | 53.42     | 58.92      | 69.06        |
| Volatile combustible....        | 22.41       | 23.92     | 26.38      | 30.94        |
| Water of composition... ..      | 11.09       | 11.82     | 13.04      | ....         |
| Hydrogen (available) ...        | 3.60        | 3.84      | 4.24       | 4.97         |
| Sulphur, volatile.....          | 0.82        | 0.87      | 0.96       | 1.13         |
| Sulphur, fixed.....             | 0.76        | 0.82      | ....       | ....         |
| Sulphur, total.....             | 1.58        | 1.69      | 0.96       | 1.13         |
| Nitrogen.....                   | 1.41        | 1.51      | 1.66       | ....         |
| Total non-combustible ..        | 27.53       | 22.66     | 14.70      | ....         |
| Total combustible.....          | 72.47       | 77.34     | 85.30      | 100.00       |
| B. T. U. per pound.....         | 12,416      | 13,250    | 14,613     | 17,131       |
| 3. Ultimate analysis:           |             |           |            |              |
| Carbon.....                     | 68.06       | 72.63     | 80.11      | 93.90        |
| Hydrogen.....                   | 4.99        | 5.32      | 5.87       | 4.97         |
| Oxygen.....                     | 9.70        | 10.34     | 12.41      | ....         |
| Nitrogen.....                   | 1.41        | 1.51      | 1.66       | ....         |
| Sulphur.....                    | 1.58        | 1.69      | 0.96       | 1.13         |

TABLE II.

|                              | Moist coal. | Dry coal. | Pure coal. | Combustible. |
|------------------------------|-------------|-----------|------------|--------------|
| 1. Usual proximate analysis: |             |           |            |              |
| Moisture.....                | 9.91        | ....      | ....       | ....         |
| Ash.....                     | 11.51       | 12.78     | ....       | ....         |
| Volatile matter.....         | 30.16       | 33.47     | 38.38      | ....         |
| Fixed carbon.....            | 48.42       | 53.75     | 61.62      | ....         |

TABLE II (Continued).

|                                 | Moist coal. | Dry coal. | Pure coal. | Combustible. |
|---------------------------------|-------------|-----------|------------|--------------|
| 2. Extended proximate analysis: |             |           |            |              |
| Moisture.....                   | 9.91        | ....      | ....       | ....         |
| Ash.....                        | 11.51       | 12.78     | ....       | ....         |
| Carbon, total.....              | 63.55       | 70.54     | 80.87      | 94.73        |
| Carbon, fixed.....              | 48.42       | 53.75     | 61.62      | 72.20        |
| Volatile combustible....        | 18.65       | 20.70     | 23.73      | 27.80        |
| Water of composition...         | 10.28       | 11.40     | 13.08      | ....         |
| Hydrogen (available)...         | 3.03        | 3.36      | 3.85       | 4.51         |
| Sulphur, volatile.....          | 0.50        | 0.57      | 0.65       | 0.76         |
| Sulphur, fixed.....             | 0.70        | 0.78      | ....       | ....         |
| Sulphur, total.....             | 1.20        | 1.35      | 0.65       | 0.76         |
| Nitrogen.....                   | 1.23        | 1.37      | 1.57       | ....         |
| Total non-combustible...        | 32.93       | 25.55     | 14.65      | ....         |
| Total combustible.....          | 67.07       | 74.45     | 85.35      | 100.00       |
| B. T. U. per pound.....         | 11,348      | 12,596    | 14,442     | 16,921       |
| 3. Ultimate analysis:           |             |           |            |              |
| Carbon.....                     | 63.55       | 70.54     | 80.87      | 94.73        |
| Hydrogen.....                   | 4.31        | 4.78      | 5.48       | 4.51         |
| Oxygen.....                     | 9.00        | 9.97      | 11.43      | ....         |
| Nitrogen.....                   | 1.23        | 1.37      | 1.57       | ....         |
| Sulphur.....                    | 1.20        | 1.35      | 0.65       | 0.76         |

TABLE III.—RELATIVE CONSTITUENTS OF COALS NOS. 1 AND 2.

|                            | Coals. |        |
|----------------------------|--------|--------|
|                            | No. 1. | No. 2. |
| Combustible:               |        |        |
| Carbon, total.....         | 93.90  | 94.73  |
| Carbon, fixed.....         | 69.06  | 72.20  |
| Volatile combustible.....  | 30.94  | 27.80  |
| Hydrogen (available).....  | 4.97   | 4.51   |
| Sulphur, volatile.....     | 1.13   | 0.76   |
| B. T. U.....               | 17,131 | 16,921 |
| Pure coal:                 |        |        |
| Water of composition.....  | 13.04  | 13.08  |
| Nitrogen.....              | 1.66   | 1.57   |
| Total non-combustible..... | 14.70  | 14.65  |
| B. T. U.....               | 14,613 | 14,442 |
| Dry coal:                  |        |        |
| Ash.....                   | 9.33   | 12.78  |
| Fixed sulphur.....         | 0.82   | 0.78   |
| B. T. U.....               | 13,250 | 12,596 |
| Moist coal:                |        |        |
| Moisture.....              | 6.29   | 9.91   |
| B. T. U.....               | 12,416 | 11,348 |