direct proportion to the amount of furfural present, so that calculations can be made directly from the readings.

The temperature greatly affects the reaction and it should be carefully regulated.

Hydrochloric acid was used instead of acetic acid on account of the fact that the acetic acid available gave a very decided color reaction, which interfered with the test. Another advantage of hydrochloric acid is that it is more uniform in quality than acetic acid and in these color tests it is necessary that the conditions shall remain constant in order to obtain comparable results. The occurrence of furfural in acetic acid or some substance that gives a similar color reaction with this reagent seems to be quite common, as it has been noted by a number of observers. The substance that gives the test can be destroyed by heating the mixed reagent, but this interferes with its sensitiveness.

In order to settle the question whether all the furfural will be found in the distillate and also whether there is any formation of furfural in distillation, the determination was made on a number of samples of slightly colored and colorless spirits, in the original and in the distillate.

The results on 120 samples gave an average of 23.4 mg. per liter on the original and of 23.2 mg. per liter on the distillate. This shows that there is no loss or gain of furfural by distillation; it also shows that the method is reliable, as the same conditions held in these determinations as in the work on aldehydes.

MOISTURE IN COAL.[1]

By E. E. Somermeier.

Received September 17, 1906.

The determination of moisture in a sample of coal is apparently simple and a result is easily obtained. The proper relation that this result bears to the original sample is, however, not so easily determined. Variations in this relation directly affect the application of all analytical work done upon the sample and the possibility of unaccounted-for moisture losses during the taking, shipping and preparing of the sample for chemical analysis too often receives little or no attention.

The method of determining moisture recommended by the

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[1] Published by permission of the Director of the U. S. Geological Survey.

Committee on Coal Analysis appointed by the American Chemical Society[1] is as follows: "Dry 1 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, cool in a desiccator and weigh covered." The report of the committee also states that "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."

The results in moisture obtained by following these directions do not, as is well-known, represent the total but only the loosely held moisture present and it represents this only approximately, as the loss in weight represents not the amount of moisture expelled but the moisture expelled plus or minus any changes due to oxidation, or any changes in weight due to the expulsion of gases from the coal. In most coals the oxidation changes are small and the loss in weight can be considered as representing moisture loss without appreciable error. The process does require, however, close attention to certain details, if uniform and accurate results are to be obtained and the precautions given by the committee to prevent moisture loss in the sample are too often neglected. The experience of the writer is that these precautions cannot be too strongly emphasized.

The precautions and details which experience has shown require careful attention may be considered under the following heads:

(1) It is absolutely necessary that the greatest of care be exercised to prevent moisture changes in the coarse sample while being taken and during transit to the laboratory.

(2) The fine sample for chemical analysis should be in nearly an air-dry condition so as to lessen danger from large moisture changes while in the laboratory.

(3) The air of the oven in which the sample is heated to drive out the moisture should be of a uniformly dry condition, if uniformity in results is to be obtained.

(4) The sample after removal from the oven should be cooled in a desiccator over sulphuric acid.

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[1] This Journal, 21, 1119.

Care in Preparation and Shipment of Sample.—As a rule precautions to prevent moisture losses during the taking of the sample are not given sufficient attention and the too common practice of shipping the coarse sample by mail or express in canvas bags cannot be too strongly condemned where the moisture result is expected to represent the moisture in the coal as sampled. A sample of coal in a canvas or muslin sack may and usually does lose a large portion of its loosely held moisture while in transit in warm mail or express cars, and the moisture results on such a sample can be only the moisture present in that particular sample in a more or less nearly air-dry condition. This is a point which has been too much overlooked in the past and as a consequence many hundreds of published analyses of coal supposedly representative of the coal as mined really lack as much as several per cent. of the moisture actually present in the sample as mined or shipped.

Failure to guard against moisture losses is not necessarily of serious consequence on samples which have been selected more or less at random and sent to the chemical laboratory for analysis. On such samples the sender understands that the particular results obtained can represent the seam of coal only in a general way. The moisture result on these samples, while low and to that extent misleading, is a minor determination, as the ash and sulphur are usually the determinations particularly desired. When samples from actual steaming tests are handled in this way the unaccounted-for moisture losses will, of course, directly affect the accuracy of the test and the heat balance in such a case is necessarily misleading, as the coal usually analyzes better than the coal as actually fired under the boilers. This makes the unaccounted-for losses larger by perhaps several per cent. than they should be and some coals with an originally high moisture content and consequent high moisture loss may be made to appear superior to really better coals with an originally low moisture content and a consequent lower moisture loss in transit to the laboratory. If correct moisture results are desired, the only safe way is to have the sample shipped from the mine or testing plant in air-tight cans, as was recommended by the committee.[1]

Handling of Sample in the Laboratory.—The experience of the writer while connected with Professor N. W. Lord as coal analyst

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[1] Loc. cit. p. 1117.

for the Ohio Geological Survey, and especially since his connection with the U. S. Geological Survey Coal Testing Laboratory, is that the only satisfactory method of handling all kinds of coal samples is to subject all or a portion of the coarse sample (5 to 10 pounds in weight) to a rather thorough air-drying before pulverization, weighing the sample from time to time until the loss becomes small. This air-dried sample is then ground in a closed ball mill and when pulverized the portion for analysis is at once put into bottles and securely stoppered.[1]

The drying of the coarse sample to a nearly constant weight by simple exposure to the atmosphere of the sampling room is a slow process, often requiring several days or a week. When a large number of samples are to be handled or where there is any hurry for results the air-drying by simple exposure to the air is too slow and too troublesome. In the U. S. Coal Testing Laboratory to facilitate this drying the samples are spread upon large trays and put into a large drying-oven designed for the purpose. The air of this oven is kept 10° to 20° warmer than the air of the sampling room and is continually renewed by means of an electric fan attached to the upper portion of the oven. By this method the material is usually dry enough to sample in from six to eight hours and can be sampled the day after being received or in special cases on the same day.

The sample as it comes from the oven after the treatment above outlined is described as "air-dried" and the analysis of the laboratory sample prepared from it is designated as the analysis of the air-dried sample. Experience in the laboratory has shown that the percentage of moisture remaining in the coal in this condition is affected to a considerable extent by the temperature and humidity of the air, the nature of the coal, the weight and coarseness of the sample, as well as its age and previous treatment. For this reason, it is not to be considered as a determination of any definitely fixed quality of the coal and it was originally adopted merely as a stage in the sampling in order to make it possible to further handle and pulverize the coal to the condition required for analysis in the laboratory without incurring more than trifling alterations in moisture. Experience has shown, however, that where the method is conducted systematically and

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[1] See Preliminary Report of the operations of the Fuel Testing Plant of the U. S. Geological Survey, 1905, Bulletin No. 290.

the drying continued, as is the case at the Testing Laboratory, until the loss between two successive weighings[1] made at least two hours apart is less than 1 per cent. of the weight of the sample, the air-drying loss with rare exceptions lies within a sufficiently definite range to give it some importance as showing the effect of standing and exposure on the percentage of moisture in the coal. This is a matter of considerable commercial importance, as so far as the moisture content is concerned coals having a large air-drying loss are obviously much more affected than coals having a small air-drying loss. It further has appeared that the amount of residual moisture in the air-dried sample prepared under the described conditions usually lies within a range which is somewhat characteristic of different kinds of coal, as from 12–16 per cent. for lignites, 3 to 6 per cent. for Illinois coals and in the neighborhood of 1 per cent. for many of the West Virginia coals.

Understood in this way, the air-drying loss has been deemed of sufficient importance to be worthy of publication in connection with the analytical results. The amount of air-drying loss has also a special significance in the case of wet samples, and the high results for moisture upon samples of washed coal or wet slack are more easily explained and understood when accompanied either by the analysis of the air-dried coal or by the amount of air-drying loss on the coarse sample.

Moisture Determinations in the Fine and Coarse Sample.—Our experience is that the preliminary air-drying of the coarse sample and the subsequent determination of the moisture in the fine air-dried sample taken together give much more satisfactory and uniform results than determinations made upon independent coffee-mill samples ($^1/_8$–$^1/_{10}$ inches in size) of the undried coal, as while in most coals the results are comparable, with some coals more or less lignitic in character the moisture determinations made on the coarse samples require several hours' heating before giving anywhere near constant weight, as much as several per cent., which will be given off by continuing the heating for one or two hours longer, often remaining in the sample at the end of an hour's heating.

Moisture in Air of Oven.—The loss of weight of the sample by

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[1] Where the sample is dried by simply exposing it to the air of the laboratory, the time between weighings is at least eight, and, more often, from sixteen to twenty-four hours.

heating for one hour at 105° in an ordinary drying-oven represents the moisture in the coal when the sample is dried in laboratory air heated to that temperature. On different days the humidity of the laboratory air varies greatly with the result that the moisture results fluctuate correspondingly, being higher in dry weather when the humidity is low and lower in damp weather when the humidity is high. These variations may be as great as 0.4 or 0.5 per cent. In the U. S. Coal Testing Laboratory to eliminate this error use is made of a special moisture oven designed by Professor N. W. Lord. The oven is constructed with double walls and the space between the walls partially filled with a solution of calcium chloride of such strength that the boiling-point of the solution is high enough to raise the temperature of the oven to 105°. Concentration of the solution is prevented by means of a reflux condenser fitted into the top of the oven. Air is admitted into the drying-chamber through a coil of block-tin tubing which passes through the calcium chloride solution. The inner end of the tubing is soldered into the rear wall of the drying-chamber and the outer end is connected to a flask containing concentrated sulphuric acid. During a determination air under pressure is bubbled through this sulphuric acid, passes through the block-tin coil into the drying-chamber and escapes through a small orifice in the door of the oven. The air is passed through at such a rate that a volume equal to the capacity of the oven passes through every six or eight minutes. Most of the moisture of the sample is driven off during the first fifteen or twenty minutes so that by the end of the hour the air of the oven is practically air-dried over sulphuric acid. By working in this way variations in the humidity of the laboratory air do not affect the determinations and duplicates run at different times agree much more closely than where an ordinary air oven is used.

Use of Concentrated Sulphuric Acid in Desiccators.—Formerly dry calcium chloride was used in the desiccators in which the samples were allowed to cool after being taken from the oven. From the results of a number of tests it is shown that coal dried at 105° has a much greater affinity for moisture than ordinary calcium chloride and the samples, if left in the desiccators for any length of time, always gave low moisture results. Tests with concentrated sulphuric acid as the drying agent in the desiccator show that the sample can remain in the desiccator for several hours before weighing without affecting the moisture result. On this account all moisture determinations are now cooled in desiccators over concentrated sulphuric acid.

Chemical Records.—The air-drying of the coarse sample and the analytical determinations on the air-dried sample necessitate the recalculating of results to obtain the analyses of the "sample as received." The regular analytical records of a sample as kept in the chemical laboratory at the Coal Testing Plant are shown by the following record of the results obtained on a sample of West Virginia coal:

Laboratory sample number	1561
	Per cent.
Loss of moisture in air-drying of coarse sample	3.10

Analysis of air-dried sample:

Proximate :	
Moisture	1.01
Volatile matter	29.53
Fixed carbon	62.67
Ash	6.79
	100.00
Ultimate :	
Hydrogen	5.04
Carbon	79.35
Nitrogen	1.63
Oxygen	6.39
Sulphur	0.80
Ash	6.79
	100.00

	Calories.	B. T. U.
Calorific value determined	7984	14371
Calorific value calculated from ultimate analysis	7890	14202

The analysis of this sample as received is obtained from the results on the air-dried sample by multiplying each result by $\frac{100 - 3.10}{100}$ and adding to the moisture result so obtained the 3.10 per cent. loss on the coarse sample and to the hydrogen and oxygen results so obtained this 3.10 per cent. moisture loss in the proportion in which the two elements unite to form water or 1/9 of the moisture loss to the hydrogen and 8/9 of the loss to the oxygen.

Performing these operations the analysis on the sample as received is as follows:

Proximate:	
Moisture	4.08
Volatile matter	28.61
Fixed carbon	60.73
Ash	6.58
	100.00
Ultimate:	
Hydrogen	5.23
Carbon	76.89
Nitrogen	1.58
Oxygen	8.95
Sulphur	0.77
Ash	6.58
	100.00
Calorific value determined—Calories	7736
B. T. U.	13925

The result on the air-dried sample must not be confounded with the "dry coal" of the mechanical engineer. which may be obtained from either of the above ultimate analyses by subtracting from the hydrogen and oxygen shown in the analysis the amount of hydrogen and oxygen present in the moisture of the proximate analysis corresponding to the ultimate, then dividing each of these remainders and each of the other percentages of the ultimate analysis by 100 minus the moisture present in the proximate analysis.

Performing these operations the ultimate analysis for the "dry coal" on this sample is as follows:

Hydrogen	4.98
Carbon	80.15
Nitrogen	1.65
Oxygen	5.55
Sulphur	0.81
Ash	6.86
	100.00

This seems to be a multiplication of results but all appear to be necessary. The "as received" results certainly cannot be dispensed with, as they represent the actual sample. The results on the air-dried sample are the actual results obtained in the laboratory and are of interest as showing the analysis of the coal when in an approximately air-dried condition. The chemist has no use for the "dry coal" results but it is necessary to the mechanical engineer in calculating the heat balance by the code prescribed by the American Society of Mechanical Engineers. In the results given under the work of the chemical laboratory in the final report of the operations of the Coal Testing Plant at the World's Fair, St. Louis, Mo., these analyses are all plainly marked "analysis of air-dried sample" and "sample as received." The analyses published in the same report under the report of the Boiler Test Division also have the ultimate analyses plainly designated as "dry coal." In the preliminary report of the Testing Plant for 1905 the different analyses are not so specifically designated but the same general arrangement holds and in all chemical reports any analysis unless otherwise marked refers to the "sample as received." On the other hand, the mechanical engineer using the code adopted by the American Society of Mechanical Engineers uses only ultimate analyses calculated to a "dry coal" basis.

The above explanation of the different methods of reporting chemical analyses may appear unnecessary but may possibly be of use to the many engineers who are not chemists and to the many chemists who are not engineers, and especially to the many who are neither chemists nor engineers but who are interested in coal analysis.

Further data relating to the experiments mentioned in this paper and a more complete description of the apparatus and methods used in the U. S. Coal Testing Laboratory will appear in the final report of the Coal Testing Plant for the year ending July, 1906, which report is now in course of preparation. This report will not, however, be available for some months and the importance of the moisture determinations appears to warrant calling attention to and emphasizing at this time some of the precautions necessary to be observed in order to secure accuracy of results.

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