A Method of Charcoal Analysis[1]

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THE CHARCOAL INDUSTRY provides a positive means for the utilization of low-value forest wood and mill residues. Both hardwood and softwood raw material can be converted to charcoal in various types of kilns or by chemical processing, in which there is recovery of both charcoal and associated by products.

Production has expanded markedly during recent years, with by-product facilities still producing more charcoal than do kilns. Kiln production is represented by hundreds of both small and large units operating singly or batterywise. Product diversification is apparent also in the rapid expansion of plants for briquetting charcoal. (See Figure 1.)

Because of the wide range of production methods employed, charcoal quality will not always be uniform nor consistent with the established levels for general market acceptance. For this reason, there is a definite need for unified methods of charcoal analysis for evaluating the products.

Most of the present methods are based on ASTM methods for coal and coke (D-271-48; D–346-35). Various modifications of these and other methods are used. The method described here is intended specifically for charcoal. It employs equipment found in most laboratories, and is adapted to routine analyses of a large number of samples. It is designed to give accurate results for moisture, volatile matter, ash, and fixed carbon.

Since this new method differs from ASTM methods D-271-48 and D–346–35 in several details, data for duplicate determinations on 100 samples were analyzed statistically. The samples were random samples of charcoal obtained from various sources in different parts of the country. The results were obtained by two analysts over a period of several months. Standard deviations of duplicates were: moisture, 0.06; volatile matter, 0.25; and ash, 0.07.

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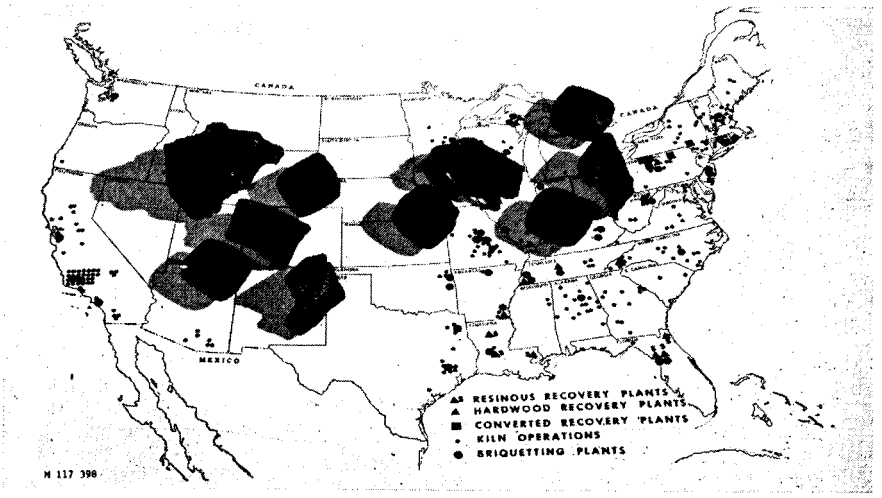

Fig. 1. —Locations of charcoal and briquetting operations.

Because of the wide range of production methods used by the charcoal industry, quality of the finished product does not always meet established levels for general market acceptance. This method of analysis will give accurate results for moisture, volatile matter, ash, and fixed carbon.

Four samples of charcoal were selected for repeated analysis. The selected samples were hardwood charcoals with volatile contents of 12, 15, 20, and 27 percent. Data for the repeated analyses on these four samples are shown in Table 1. Since the stability of the values for volatiles in stored, ground charcoal is unknown, the data were collected over a period of approximately 2 months. The data indicate that, during this time interval, volatility values remained constant for samples stored in sealed containers at room temperature. The data also demonstrate that the accuracy and reproducibility of the method are acceptable for establishing the quality of charcoal and for determining the effects of variables in production.

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[2] Maintained at Madison, Wisconsin, in cooperation with the University of Wisconsn.

The four ground samples (Table.. 1) were submitted to two laboratories of large independent producers of charcoal for analysis by their methods. Results are shown in Table 3. It can be seen that agreement is good.

In view of the data shown in Tables 1, 2, and 3, the following procedure for the analysis of charcoal is outlined. By this procedure, 30 samples in duplicate (60 complete determinations) can be carried out by one analyst in 2 days.

Procedure for the Analysis of Charcoal

Sample Preparation: For the analysis to have value and meaning, a representative sample must be selected. This selection may be carried out according to ASTM methods for sampling of coal (D–271-48; D–346-35). Samples will normally be air-dry charcoal lumps or briquettes. Rain-soaked or wet samples should be spread out to air dry before they are analyzed. For determining the moisture content of the charcoal as received, the sample should be ground to pass a coarse screen such as No. 20 U. S. Standard Sieve, since heat generated by excessive grinding would cause loss of moisture. For the determination of volatile matter, a more finely ground sample is required. All of the selected sample should be ground; no part of the sample is to be rejected.

A Wiley mill, size No. 2 with a 1-millimeter screen, was used to give the screen analysis shown in Table 2. Three hundred grams of charcoal were ground in less than 5 minutes. Longer grinding times resulting from the use

Table 1.—REPEATED CHARCOAL ANALYSES

Date	Moisture Percent	Moisture Average	Volatile Percent	Volatile Average	Ash Percent	Ash Average	Moisture Percent	Moisture Average	Volatile Percent	Volatile Average	Ash Percent	Ash Average
	Sample I						Sample II					
1-16-58	1.83		12.53		4.42		1.85		15.67		2.88	
	1.83		11.45		4.38		1.88		15.35		2.81	
		1.83		11.99		4.40		1.86		15.51		2.84
1-20-58	1.92		11.52		4.36		1.92		15.29		2.79	
	1.87		11.94		4.44		1.91		15.55		2.76	
		1.90		11.73		4.40		1.92		15.42		2.78
1-27-58	1.71		12.72		4.51		1.84		15.62		2.82	
	1.74		11.47		4.49		1.81		15.71		2.86	
		1.73		12.10		4.50		1.82		15.66		2.84
2-3-58	1.84		12.84		4.48		1.95		15.57		2.92	
	1.83		11.41		4.49		1.96		15.66		2.87	
		1.84		12.13		4.49		1.96		15.62		2.90
2-10-58	1.88		11.80		4.47		1.93		15.18		2.97	
	1.91		11.15		4.44		1.92		14.94		2.93	
		1.90		11.48		4.46		1.92		15.06		2.95
2-17-58	1.84		11.36		4.47		1.95		15.30		2.80	
	1.85		11.48		4.39		1.91		15.66		2.81	
		1.85		11.42		4.43		1.93		15.48		2.80
2-24-58	1.59		12.44		4.30		1.66		15.68		2.83	
	1.56		11.69		4.30		1.63		15.56		2.76	
		1.58		12.07		4.30		1.64		15.62		2.80
3-27-58	1.84		12.19		4.41		1.87		15.67		2.86	
	1.82		11.91		4.50		1.88		15.16		3.12	
		1.83		12.05		4.46		1.88		15.42		2.99
Std. Dev.		.107		.288		.063		.099		.191		.077
	Sample III						Sample IV					
1-27-58	----		-----		----		2.77		26.75		2.33	
							2.76		27.03		2.30	
								2.76		26.89		2.32
2-3-58	2.00		19.73		1.88		2.92		27.06		2.29	
	1.98		19.98		1.91		2.88		27.27		2.22	
		1.99		19.86		1.90		2.90		27.16		2.26
2-10-58	1.99		19.83		1.95		3.05		26.49		2.33	
	2.03		19.59		1.89		2.97		26.59		2.31	
		2.01		19.71		1.92		3.01		26.54		2.32
2-17-58	1.98		19.77		1.84		3.04		26.18		2.24	
	1.99		19.89		1.95		3.04		26.07		2.21	
		1.98		19.83		1.90		3.04		26.12		2.22
2-24-58	1.55		20.31		1.85		2.48		27.10		2.11	
	1.54		20.10		1.84		2.46		27.07		2.13	
		1.54		20.20		1.84		2.47		27.08		2.12
3-27-58	1.94		20.31		1.92		2.91		26.76		2.18	
	1.94		20.18		1.86		3.04		27.11		2.22	
		1.94		20.24		1.89		2.98		26.93		2.20
Std. Dev.		.197		.240		.027		.215		.391		.075

of a finer screen or dull knives should be avoided because of the possibility of generating heat, which in turn causes a loss of volatile material. Excessive grinding will also produce a large amount of fine particles (smaller than 100 mesh, U. S. Standard Sieve). These fine particles may be swept out of the crucible during the rapid evolution of gases in the determination of volatile matter, and thus cause errors. Because over-size particles can result in low values for volatiles, particles larger than No. 20 U. S. Standard Sieve should be avoided. The ground samples should be stored in airtight containers such as screw-top bottles. The samples should be well mixed by shaking before they are weighed. "Jack stones" can be used to facilitate mixing.

Steps in Analysis: Analyses are carried out in duplicate, according to the following steps:

1. A muffle furnace that will control temperatures of 750° ± 5° C. and 950° ± 5° C. is required. Heat the muffle furnace to 750° C. and place previously ignited[3]—porcelain crucibles (41 millimeters × 37 millimeters) and lids in furnace for 10 minutes.

2. The crucibles are cooled in desiccator for 1 hour.

3. Weigh crucibles and add an accurately weighed (to 1/10 milligram) sample of approximately 1 gram of charcoal.

4. For moisture content, place samples in oven at 105° C., and dry for 2 hours.

5. Remove dried samples from oven and cool in desiccator for 1 hour and weigh.[4]

6. For the determination of volatiles, heat muffle furnace to 950° C.

7. The crucibles, with lids in place and containing the samples used for the moisture determination, are preheated as follows: With furnace door open, for 2 minutes on the outer ledge of the furnace (300° C.) and then 3 minutes on the edge of the furnace (500° C.).

8. The samples are then moved to the rear of the furnace for 6 minutes with the muffle door closed. The samples are watched through a small peephole in the door of the furnace. If sparking occurs, results will be in error. If the sparking sample does not check the results of its non-sparking duplicate within ± 0.5 percent, the analyses must be repeated.

9. The samples are removed and placed in a desiccator for 1 hour and weighed.

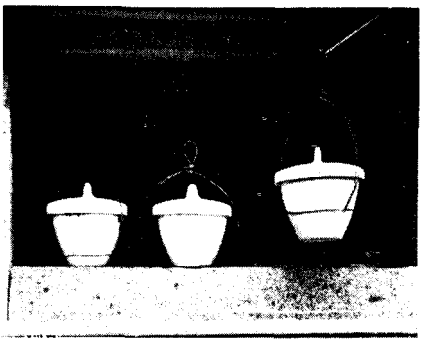

Fig. 2.—Nichrome wire baskets for handling covered crucibles.

[3] In practice, crucibles from previous determinations are used.

[4] The Sample shall be considered oven dry when the decrease in weight of consecutive weighings is 0.0005 gram or less. Successive drying periods shall not be less than 1 hour.

Table 2.- SCREEN ANALYSIS OF CHARCOAL SAMPLES GROUND IN WILEY MILL

Screen size[1]	Sample number I	II	III	IV	V	VI	VII	VIII	IX	X	XI	XII	XIII	XIV	XV	Averags of 15 samplee
	Percent of Sample Retained on Smaller Screen															
Plus 20	0	0	0	0	0	0	0	0	0.8	0	0	0	0	0	0	0.05
20 x 40	18.2	16.9	12.6	12.0	11.4	13.5	13.2	12.6	12.2	11.0	15.4	15.9	16.1	17.1	17.9	14.40
40 x 60	15.8	16.2	22.0	13.6	24.4	16.9	21.4	21.0	21.1	18.7	19.9	17.6	18.6	17.0	16.8	18.73
60 x 80	6.4	6.9	8.6	7.5	8.4	7.2	7.7	8.9	2.9	7.2	6.8	7.1	6.6	6.4	5.8	6.96
80 x 100	2.3	2.1	1.7	2.5	6.7	4.4	5.5	6.1	4.9	1.6	3.3	3.5	1.6	2.6	2.1	3.40
Minus 100	57.3	57.9	55.1	64.4	49.1	58.0	52.2	51.4	58.1	61.5	54.6	55.9	57.1	56.9	57.4	56.46

[1] U. S. Standard Sieve.

10. For ash determination, place the lids and the uncovered crucible containing the sample used for the volatile determination in the muffle furnace at 750° C. for 6 hours.

11. The crucibles with lids in place are cooled in a desiccator for 1 hour and weighed. Burning of the sample is repeated until successive 1 hour periods of heating result in a loss of less than 0.0005 gram.

12. Computations:

A. Moisture percent = $\frac{\text{loss in weight} \times 100}{\text{weight of air-dry sample}}$

Report results to first decimal place. Moisture values for duplicates should agree within 0.1 percent.

B. Volatile percent = $\frac{\text{loss in weight} \times 100}{\text{weight of oven-dry sample}}$

Oven-dry weight = weight of air-dry sample minus moisture. Report results to first decimal place. Volatile values for duplicates should agree within 0.5 percent.

C. Ash percent = $\frac{\text{weight residue} \times 100}{\text{weight of oven-dry sample}}$

Report results to first decimal place. Ash values for duplicates should agree within 0.1 percent. Note: All results except moisture are reported on oven-dry basis.

Table 3. -ANALYSIS OF CHARCOALS BY THREE LABORATORIES

	Forest Products Laboratory	Independent laboratory A, Analyst 1	2	3	1	2	3	Independent laboratory B[1]
	Sample No. I							
Moisture	1.82	1.7	1.7	1.7	----	----	2.4	1.3
Volatile	11.99	11.7	10.9	11.1	11.4	11.2	10.8	10.5
Ash	4.45	----	4.3	----	----	----	----	4.6
Fixed carbon	83.56	----	84.8	----	----	----	----	84.9
	Sample No. II							
Moisture	1.89	1.4	1.5	1.5	----	----	2.3	1.4
Volatile	15.55	15.7	14.9	15.0	15.7	15.7	14.9	15.0
Ash	2.84	----	2.7	----	----	----	----	2.8
Fixed carbon	81.61	----	82.4	----	----	----	----	82.2
	Sample No. III							
Moisture	1.99	1.8	1.8	1.8	----	----	----	1.6
Volatile	19.86	20.1	19.1	20.0	19.8	19.9	----	20.3
Ash	1.90	----	1.8	----	----	----	----	1.8
Fixed carbon	78.24	----	79.1	----	----	----	----	77.9
	Sample No. IV							
Moisture	2.83	2.8	2.7	2.7	----	----	----	2.2
Volatile	27.03	27.1	26.5	26.5	27.2	27.3	----	27.6
Ash	2.29	----	2.2	----	----	----	----	2.5
Fixed carbon	70.68	----	71.3	----	----	----	----	69.9

[1] Data recalculated so that volatile, ash, and fixed carbon are also on the ovendry basis.

Purchased by the Forest Products laboratory, U. S. Department of Agriculture, for official use.