FIELD APPARATUS FOR DETERMINING ASH IN COAL.

By C. E. LESHER.

INTRODUCTION.

Many engineers, mine superintendents, and purchasing agents have doubtless, like the writer, felt the need of a portable apparatus for determining the percentage of ash in coal without having to wait days or perhaps weeks for the shipment of a sample to the laboratory, for the performance of the laboratory work, and for a report of its results. Such an apparatus is described in this paper—an apparatus light, compact, and portable, which can readily be assembled by anyone and carried on a handcar, a wagon, or a pack horse. The paper also gives instructions for the use of the apparatus in such terms that it is believed anyone, even one without technical training, can in a short time make tests with a degree of accuracy that will meet all but the most exacting demands of commercial work.

The apparatus was designed to meet the needs of field parties of the United States Geological Survey, and has been in successful use for three years. It is simple to operate and if used with a moderate amount of care will determine ash with a possible error of less than 2 per cent.

APPARATUS.

The complete outfit is shown in Plate I. It weighs, including the case for shipping, about 34 pounds. It is all contained in a box measuring 11 by 13¾ by 14 inches, which is strongly made with brass-bound corners and a handle for carrying. The lid is fitted with a lock and key for ordinary use but can be screwed on securely when the box is packed for shipment. The arrangement of the apparatus when packed in the case is shown in Plate II, *B*.

The following is a list of the articles included in the outfit. The first list includes such apparatus as may be obtained directly from any of the laboratory supply houses [1] by means of the descriptions given, which are taken directly from the catalogues, and the second list includes material that must be obtained elsewhere, as indicated.

[1] For the information of those not familiar with laboratory supply houses the following list of some of the better known is given: Eimer & Amend, New York City; E. H. Sargent & Co., Chicago, Ill.; Scientific Materials Co., Philadelphia; A. H. Thomas Co., Philadelphia; Braun, Knecht & Braun, San Francisco; Denver Fire Clay Co., Denver, Colo.

Parts that may be obtained from a laboratory supply house.

Two burners, Barthel's blast lamp. (See description below.) Any gasoline torch used by plumbers and obtained from or through hardware stores may be substituted for this.

Two iron rings with fastening screws; outside diameter, 4 inches. If base for burner as shown in figure 2 is not made, then ring stand with iron base should be ordered to hold these rings. These are described in the catalogues as "Supports, iron, rectangular base and steel rod; size of base, 6 by 4 inches; length of rod, 18 inches."

One air bath or drying oven, single wall, of heavy polished copper, with openings for thermometer and ventilation, with shelf, but without iron leg support, 6 by 8 inches.

One mortar and pestle, porcelain, with lip; shallow form; diameter, 6 centimeters (2½ inches).

One brass-wire screen, 10 mesh; 5 inches in diameter, with bottom.

One thermometer, 105° C.

One balance (pocket scales; see description below).

One pair of tweezers for handling weights; furnished with balance.

One set of weights; German silver, in box with sliding cover, each weight in separate compartment; 1 milligram to 500 milligrams.

Twelve glazed crucibles with covers; Royal Berlin porcelain, size 000; or Royal Meissen porcelain, size 8.

One pair black iron crucible tongs, 9 inches long.

Three camel's-hair brushes, small.

Six iron pipe-stem triangles, pipe stem covered; length inside, 2 inches.

One iron spatula; wood handle; length of blade, 4 inches.

One dozen round tin boxes; 2½ inches in diameter, 1⅛ inches deep.

Lead foil, 3 ounces.

Ordinary Bunsen burners may be substituted for the gasoline burners when a supply of illuminating gas is available. In ordering, these may be specified on the list in place of the blast lamps: Two Bunsen burners, ordinary type, height about 5½ inches; 12 feet tubing, white, heavy wall, inside diameter one-fourth of an inch.

List of parts to be obtained from various sources.

Two iron bases for burners, as shown in Plate I, with rod support for ring. Can be made by blacksmith or machinist.

One galvanized-iron gasoline can, capacity about 1 gallon; can be made in any tin shop.

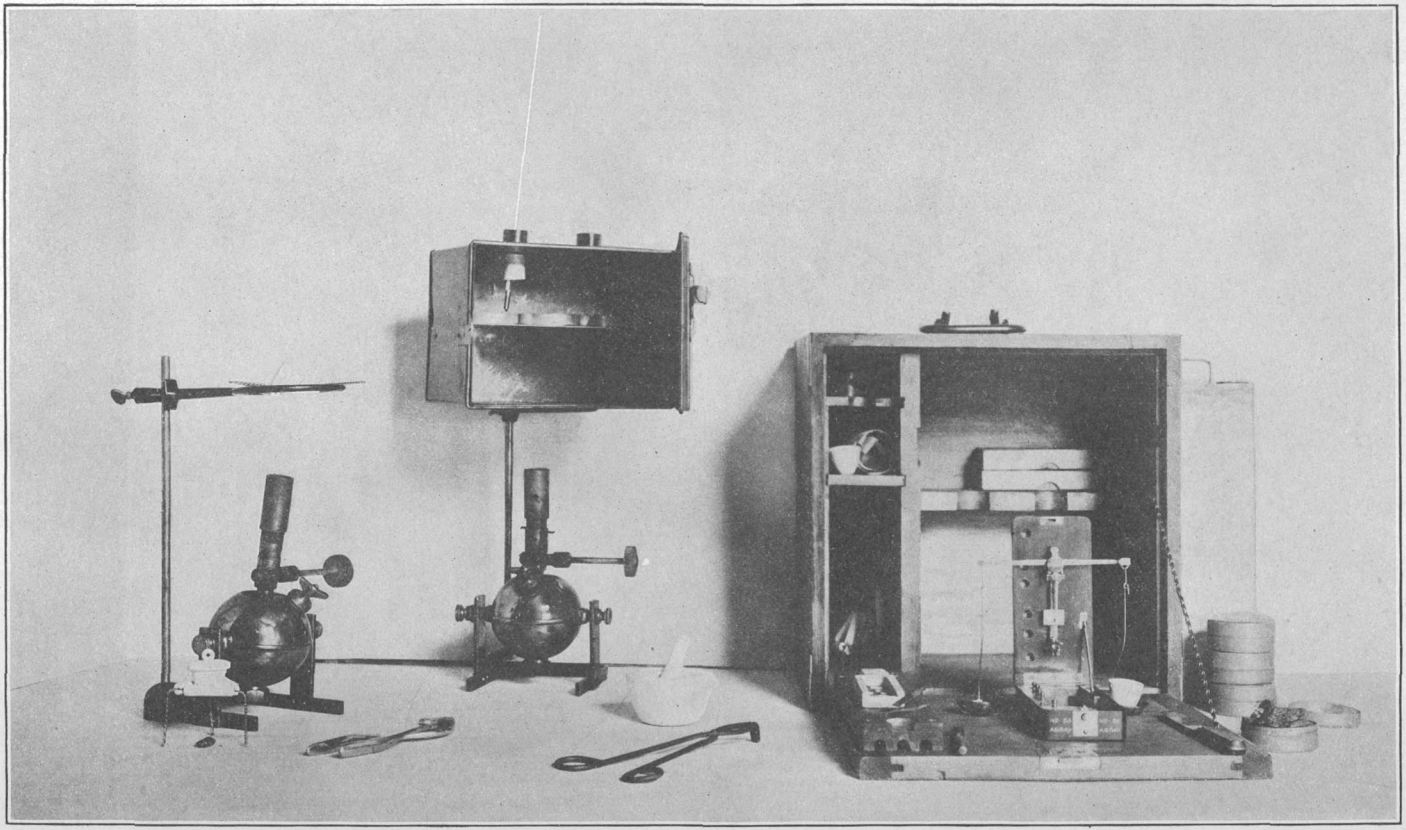
One pair nickeled combination pliers, 6½ inches long. Sold by hardware stores.

One flat file, 4 inches long.

One box with compartments for holding parts of outfit. To be made by carpenter. Necessary only for portable outfit.

Six inches of fine iron wire.

Regarding most of the articles named, no comment is necessary The legs usually provided with the air bath are not necessary, as the bath can be heated on a camp or heating stove or by one of the gasoline burners (Pl. I). The sample tins are ordinary druggist's "salve boxes," the lids of which fit tightly enough to prevent loss of moisture, and being smooth and rounded inside, furnish a convenient receptacle for the sample while it is being air-dried. Corresponding numbers are stamped on the lids and bottoms to prevent confusion in handling. The crucibles are about 1 inch in diameter



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at the top and 1 inch deep and are small enough to allow the full flame from the burner to envelop them. The crucible is provided with a lid, which should be in place at the beginning of the operation of burning and also while the crucible is cooling. The pliers are necessary in handling the gasoline burners.

The two articles calling for the most care in selection and of which there are a variety of types available are the balance and burner. The balance, selected after several others, large and small, were considered, is described in the trade catalogue as a "Pocket scale, capacity 10 grams, and sensitive to 1 milligram." This balance is of the "take down" variety and when closed measures 6 by $2\frac{1}{2}$ by $1\frac{1}{8}$ inches. It is easily sensitive to 5 milligrams and by careful manipulation can be read to 1 milligram. As furnished it has one serious disadvantage, namely, that the pans are attached to the beam by cords and can not readily be removed. To overcome this defect supports may be made consisting of a German silver wire hooked to the cross beam above and attached below to flat folding cross pieces bent to fit the pan, on which the pan may be placed and from which it can be removed at will. One of these supports is shown in figure 1. They nest into the case with the pans and contribute largely to the successful use of the balance. A set of milligram weights accompanies the balance, but they are all in one small compartment and are not readily accessible. It is best to keep these as a reserve and to add to the outfit an additional set of German silver weights (500 milligrams to 1 milligram) in a small box with sliding cover and with a compartment for each weight. A slide in the back of the balance case holds the tweezers for handling the weights.

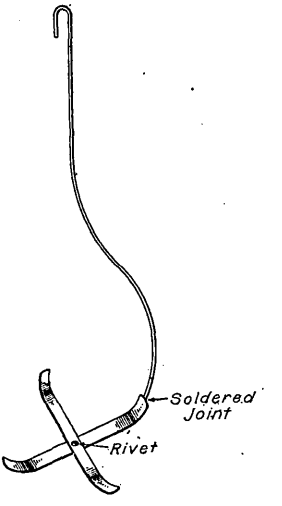


FIGURE 1.—Pan support for balance.

Gasoline was selected as a fuel because it is cleaner than kerosene and gives a hotter flame than alcohol and can be used in any type of gasoline burner. The burner adopted for the field outfit is a special type known as "Barthel's automatic blast lamp" (fig. 2), which has several decided advantages over the ordinary horizontal flame plumber's torch. It uses gasoline for fuel; it has the Meker burner, which gives better combustion and a hotter flame than other burners; it is automatic in that it requires no pumping to keep up the air pressure; and in its normal position it gives a vertical flame. This burner can be purchased without the cast-iron base and a light skeleton base (fig. 2) can be made to support both the burner and

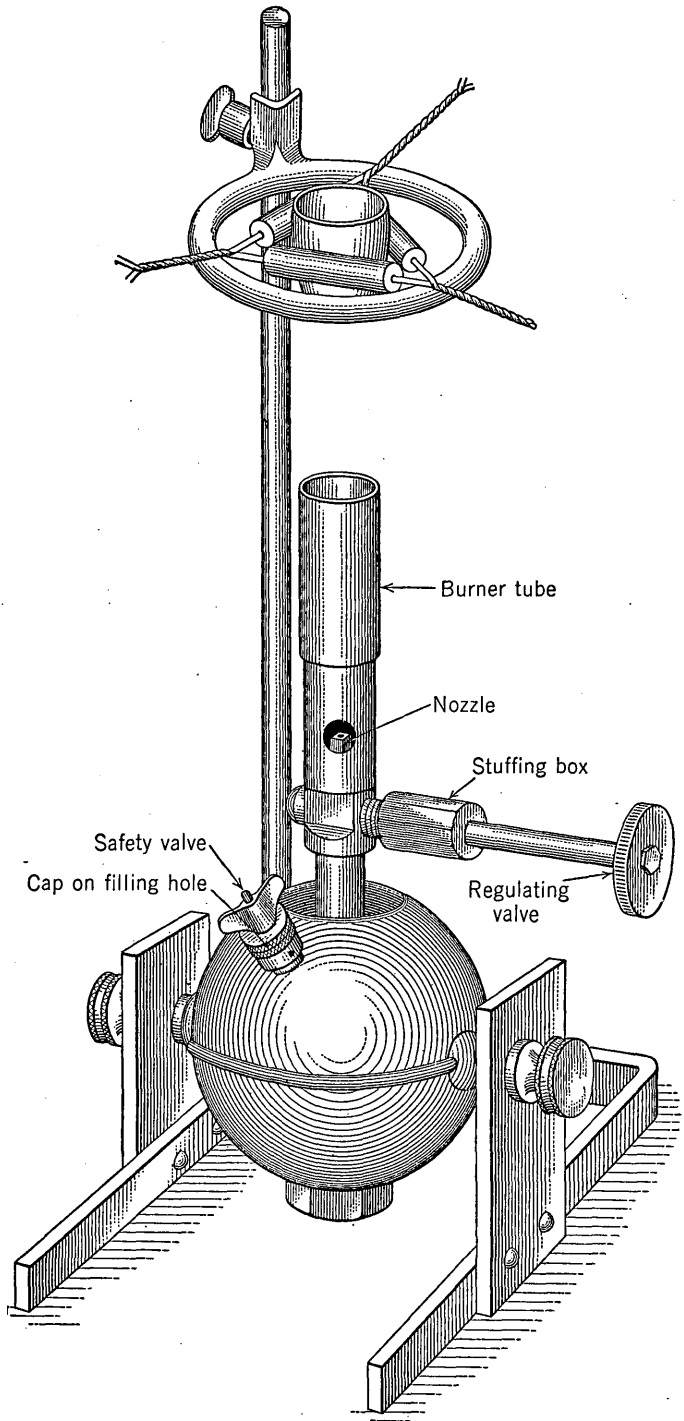


FIGURE 2.—Barthel's gasoline blast lamp.

the ring stand on which the crucible is set. This base can be easily made by any blacksmith or machinist and is inexpensive. The lamp has two burners, so that one operator can almost double the number of determinations in a given time.

OPERATION OF THE BURNER.

The following instructions for the use of the Barthel blast lamp should be carefully followed: In filling close the regulating valve (fig. 2), remove the cap to the filling hole, and fill the reservoir with gasoline to within about an inch of the top and screw the cap on tightly with the pliers.

In preparing to light set the burner upright and fill the hollow around the base of the burner tube with gasoline. A convenient way of doing this is to pour the gasoline into the top of the burner tube, through which it will flow into the hollow. Light the gasoline in the hollow and protect the flame from drafts. If an excess of gasoline is poured, overflowing the hollow, and the burner be enveloped in flame, no harm may be expected to result, for if the reservoir is overheated, the pressure, if excessive, will open the safety valve in the cap on the filling hole. When the valve is thus opened gasoline or gasoline vapor is forced out of the small hole in the side of the screw cap and, of course, will take fire. By opening wide the regulating valve and closing the safety valve by tapping on the point in the end of the cap proper conditions will be restored. Some who have used this burner advise leaving the regulating valve slightly open while the burner is heating to avoid too high a pressure. When the gasoline in the hollow is nearly all burned out open the regulating valve and light the escaping vapors at the upper end of the burner. The flame when burning properly is blue to almost colorless and can be adjusted as to height by opening or shutting the valve. To extinguish the flame close the valve tightly by turning it to the right. If when extinguished a small yellow flame appears in the lower part of the burner, it should be blown out to prevent carbon from depositing in the valve opening, and the handle should then be turned to the left about a quarter of a turn, so that when cool the needle valve will not be jammed tight. If relighted without refilling, the valve should first be opened to allow the air to enter the reservoir, then closed and heated as described above.

Should the burner not be heated sufficiently at the beginning, it will burn improperly, giving an irregular, sputtering, yellow flame. To remedy this trouble shut off the burner and heat again with gasoline in the hollow cup without waiting for the burner to cool. Irregular action may be caused by the clogging of the valve opening. To remedy this trouble unscrew the burner tube and clean the hole with

the cleaning wire that is provided with the outfit. If this does not help, unscrew the nozzle of the valve (fig. 2) and replace the fine wire gauze. After it has been long used the wick in the reservoir must be replaced by unscrewing the cap on the bottom of the reservoir. If leakage around the cap over the filling hole can not be stopped by tightening the cap, a new washer should be put in. This washer should have a hole in the center to allow the safety valve to work. Likewise, if there is leakage of vapor around the spindle of the regulating valve, the stuffing box should be tightened or repacked. The reservoir may be refilled while hot without danger.

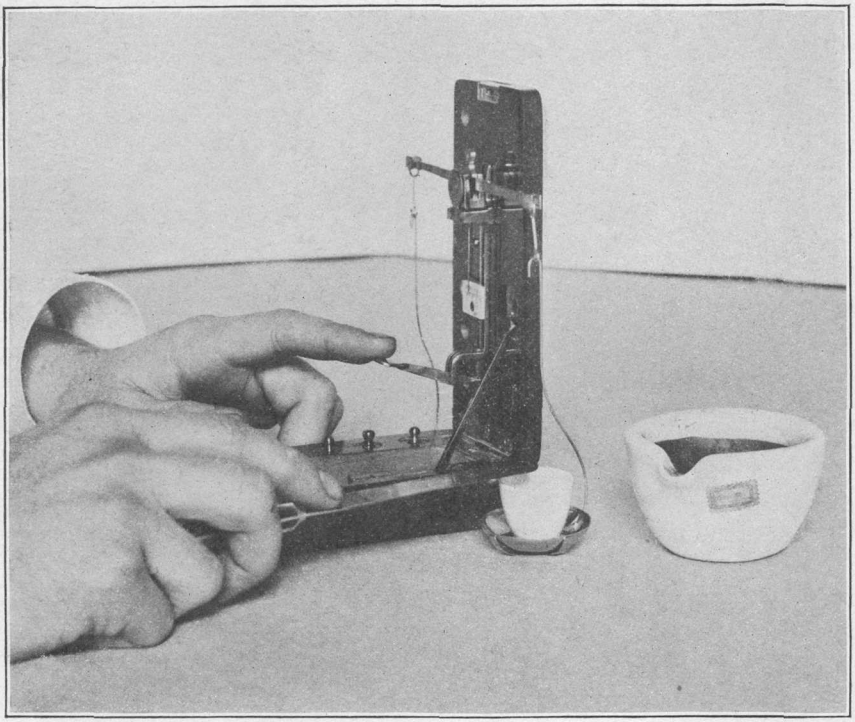
OPERATION OF THE BALANCE.

In setting up the balance for the first time try different combinations of hangers and pans to find which pans are in balance; then mark, by scratching with a pin or a knife blade, those on the right with "R" and those on the left with "L."

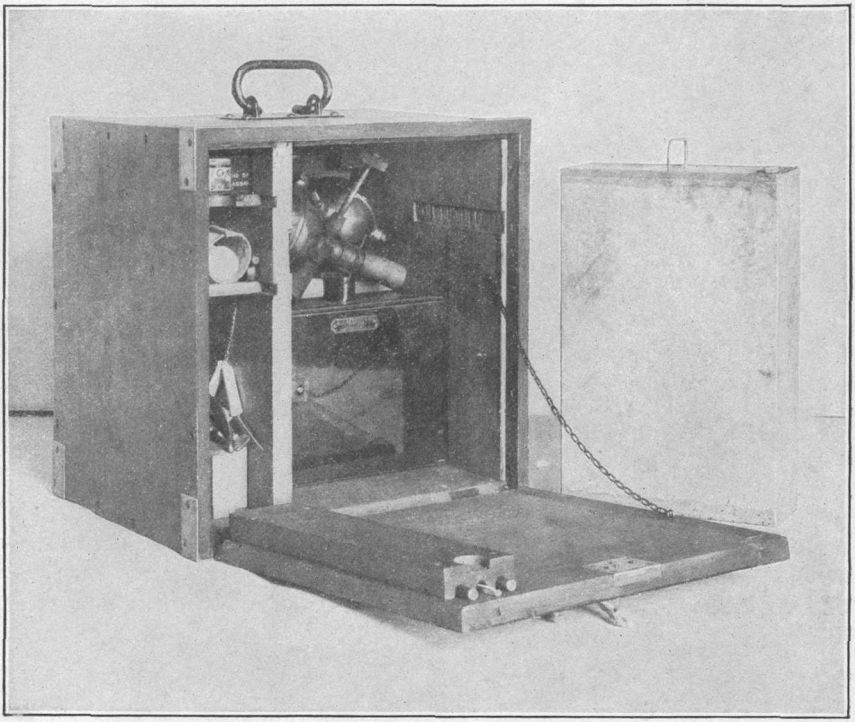
The easiest and quickest way of determining when the pans are in balance is to press (Pl. II, *A*), not too quickly, the lever which raises the beam and observe the action of the pointer. If the end of the pointer holds its position over the center of the scale, the pans are of equal weight. If it moves to one side or the other, the side to which it moves is light. If it moves quickly the difference in weight is large; if it moves very slowly, the difference is slight. As soon as the way the pointer tends to move is observed the lever should be released, weight added or removed, and the operation repeated. This method is better and quicker than that of raising the beam and allowing the pointer to oscillate back and forth till it comes to rest. It is generally more convenient to place the material to be weighed in the right pan and the weights in the left pan. When a sample of coal is to be weighed put the brass weight, together with the corresponding lead counterpoise (pp. 8–9), in the left pan and the crucible in the right pan. Add coal to the crucible in decreasing quantities as the amount approaches a gram, holding the spatula with powdered coal on its tip in the right hand and tapping it with the index finger so that small quantities of the powder may fall into the crucible, using the forefinger of the left hand for raising and lowering the balance pans (Pl. II, *A*). After each addition of coal to the crucible raise the beam and observe the action of the pointer. In weighing the ash put the crucible in the right pan and add weights to the left pan with the counterpoise to balance.

METHOD OF ANALYSIS.

The sample should first be crushed to half-inch size and then reduced by successive quartering and crushing, so that finally not more than a quarter of a pound remains, all of which will pass through the 10-mesh screen. The sample can be mixed and quartered to



A. WEIGHING A SAMPLE OF COAL FOR DETERMINATION OF ASH.



B. APPARATUS PACKED IN CARRYING CASE, WITH GASOLINE CAN REMOVED TO SHOW INTERIOR ARRANGEMENT.

advantage on a piece of heavy wrapping paper, or on oilcloth or rubber cloth. Three grams of the sample are obtained by taking small quantities on the point of the spatula from different places in the bottom pan of the screen. This sample is weighed directly on the scale pan and from there brushed with a camel's-hair brush into the lid of the sample tin and placed in the air bath for drying.

Before the coal is burned to ash it should generally be dried, for all coal contains moisture, partly as water adhering to and wetting the particles and partly as water in the coal itself. The amount of water differs in different coals, Pennsylvania anthracite containing as low as 0.6 per cent and some North Dakota lignite containing more than 40 per cent. The moisture content of specimens of coals that contain more than a small percentage of water and that have been exposed to the atmosphere varies more or less with changes in the humidity of the air and with the length of exposure, because a part of the moisture is loosely held in the coal and, if the coal is exposed in a dry atmosphere, will evaporate.

The sample should be submitted to a preliminary drying, because evaporation may take place rapidly, as, for instance, in a lignite or low-grade subbituminous coal, where loss of water occurs so quickly that the surface of a sample may be seen to crack.

The effect of varying amounts of moisture in the samples analyzed may be illustrated simply as follows: Assume a 100-pound sample of lignite to contain 30 pounds of moisture and 10 pounds of ash. Analysis will show, therefore, 10 per cent of ash. If, however, the sample is exposed to the air, water will evaporate. If 10 pounds of water evaporates, the sample will weigh 90 pounds, of which 10 pounds are ash. Analysis will now show 11.1 per cent of ash, an increase of 1.1 per cent. If the sample is analyzed after it has lost 20 pounds of moisture, it will be found to contain 12.5 per cent of ash, an increase of 2.5 per cent, and if the loss of moisture is 30 pounds, the percentage of ash will be 14.2 per cent, an increase of 4.2 per cent. In other words, two samples of the same coal will, if one is wetter than the other, show on analysis different percentages of ash. If, however, the loosely held water in both is driven off under like conditions of temperature, the percentage of ash should be the same.

The obvious method of removing such a source of apparent discrepancy is, at the beginning, to dry the coal at room temperature until all the moisture that it will give off at that temperature is removed. A sample so treated will undergo the minimum amount of change in moisture content thereafter when exposed to the air. For this reason the usual laboratory practice is to dry all samples of coal under certain fixed conditions before they are analyzed. The standard practice at the Pittsburgh laboratory of the United States Bureau of Mines is to pass a current of air at a temperature of 35° C. (95° F.)

over the coal until it shows no further appreciable loss of moisture. This process is called air-drying, and the difference in weight before and after drying is called the "air-drying loss."

In using the field apparatus it is best to dry the samples at 60° C. (140° F.) with the sample on the middle shelf, the bulb of the thermometer half an inch above the coal, and the door of the oven open about an inch. It is of course impossible by a treatment lasting but an hour to duplicate results on air-drying loss that are obtained in the laboratory under conditions carefully chosen and controlled and by drying for long periods.[1] Other things being equal, the error in percentage of air-drying loss will vary with the amount of moisture, being greatest with the highest moisture coals. It will not by this method, however, be over 3 or 4 per cent in a sample of lignite showing an air-drying loss of 30 or more per cent and as a difference of 3 per cent in a sample having 30 per cent air-drying loss makes a difference in the percentage of ash of but 0.03 per cent if the ash is 5 per cent, or 0.06 per cent if the ash is 10 per cent, it is evident that the method is sufficiently accurate for the purpose intended. After drying for 1 hour the coal is brushed into the balance pan and weighed, the loss in weight being the air-drying loss. An example showing the calculation of this in percentage will be given later.

The method ordinarily employed in laboratories for determining ash in coal is to heat a small weighed portion of the powdered sample to a red heat and to allow fresh air to come in contact with the heated coal. All water is driven off and the solid carbon and tarry compounds are burned, passing off as gases and leaving behind the mineral matter or ash, which is composed of silica, clay, iron oxides, and sulphates in various proportions. The procedure recommended with the field outfit is as follows:

The air-dry sample is quickly pulverized very fine with the mortar and pestle and 1 gram is weighed out into a crucible. It may not always be necessary or even advisable to use 1 gram of coal for burning to ash. If the ash is more than 15 per cent the time required to burn off completely 1 gram is considerably increased, and for this reason one-half gram may be used. It should be borne in mind, however, that the smaller the sample used the greater is the liability of error in the final result, and when the smaller amount is used extra care should be taken in the weighing of the sample both before and after burning. For each crucible in use a counterweight

[1] Mine samples of coal crushed to pass a ¼-inch screen, and spread out to a depth not exceeding 1 inch, in circulating air at a temperature of 10° C. higher than room temperature will become approximately air dry in the following periods of time: Appalachian coals, 8 to 24 hours; Illinois and similar coals, 12 to 48 hours; subbituminous coals and lignites, 48 to 72 hours (Fieldner, A. C., The sampling and analysis of coal: U. S. Bureau of Mines Tech. Paper 76 p. 52, 1914).

of lead foil should first be made, so that by having it on the opposite pan of the balance when the coal and the ash are weighed the actual weighing of the crucible is obviated. The counterpoise should be within 1 or 2 milligrams of the weight of the crucible, and since the latter changes weight with use it is necessary after several days' use to correct the weight of the counterpoise by addition or subtraction of lead. After the sample is weighed set the covered crucible upright in a pipestem triangle on the ring stand, adjusting the opening in the triangle by bending the wires so that about two-thirds of the crucible projects below. Adjust the regulating valve of the burner until the flame of the blast lamp is about 3 inches long, and arrange it so that the top of the flame just touches the bottom of the crucible on one side. Increase the heat gradually by turning the regulating valve to the left so that after 10 minutes the bottom of the crucible is red hot. Ordinarily by this time all moisture and tar are driven off, a portion of the tar having condensed on the upper part of the crucible and on the under side of the lid, and no fumes or smoke can be observed coming off. Lignite and subbituminous coal (black lignite), being high in moisture, are apt to sputter when first heated. By heating slowly at first, as described above, with the flame directed well up against the side of the crucible this tendency is reduced and the cover prevents loss if the sputtering is slight. At the first evidence of this tendency the heat should be reduced by turning down the flame of the burner. If, however, the sputtering takes place with sufficient violence to bounce the lid and throw out powdered coal, which will be shown by sparks appearing in the flame outside the crucible, the test is probably spoiled and should be repeated.

As soon as all moisture and tar are driven off, remove the cover, tilt the crucible to about 45° on the triangle (Pl. I), and apply the full blast, arranging it so that little or no flame is in front of the crucible to interfere with the free access of air to the coal. As burning proceeds the coal will glow and at times a small flame may be seen playing round inside the crucible. A cover of ash soon forms on top of the mass and checks the speed of burning, so that by stirring every few moments with a fine iron wire, taking care each time to tap the wire before removing it to dislodge adhering ash and coal, the operation is expedited. The crucible should be turned around as the burning proceeds until all the black coating of tar on the inside, formed in the earlier part of the operation, is burned off and the heating and stirring should be continued until the residue no longer glows but appears "dead" as contrasted with the hot pulverized coal, which when stirred appears to flow like water or quicksilver.

The burning of a 1-gram sample to ash should be completed in about 30 minutes, after which the crucible should be removed from the flame, covered, and allowed to cool. A convenient holder for the hot crucible is made by bending down the wires of a pipestem triangle (Pl. I) to form legs. When cool enough to handle, the ash may be examined to see whether it contains any unburned black particles. If completely burned it will show no such particles, and its color will be either grayish white or, if it contains much iron, pink or red. The crucible containing the ash is placed on one pan of the balance and the lead counterpoise for that crucible on the other, with sufficient weights to balance the ash. Calculations of the result in percentage will be given later.

Until the operator has had considerable experience in making tests there is always the possibility of mixing the crucibles, their counterpoises, and the sample tins, so that in the end there may be uncertainty as to what sample is represented by the test, or whether the proper counterpoise has been used. To avoid this it is advisable to mark the crucibles and counterpoises with numbers corresponding to those on the sample tins. A number may be marked on the lead with a hard pencil or written in ink, and may be written on the bottom of the crucible with a piece of red ocher (hematite); or notches may be filed on the upper edge of the crucible with the corner of a file. These notches should of course be made before the lead counterpoise is made. With experience it will be found that if the same order and arrangement is always followed, numbering will cease to be necessary and there will be no danger of confusing the samples and crucibles.

EXPLANATION OF WEIGHTS AND CALCULATION OF RESULTS.

The brass weights in the case range from 1 gram to 10 grams, and are stamped with figures representing the number of grams. The German silver weights range from 0.001 to 0.5 gram and are stamped with the number of milligrams. The equivalents of milligrams in fractions of a gram are shown below.

Number of milligrams.	Fraction of a gram.
500	0.5
200	.2
100	.1
50	.05
20	.02
10	.01
5	.005
2	.002
1	.001

That is, 1,000 milligrams equal 1 gram and 10 milligrams equal 0.01 (one one-hundredth) gram. Therefore, if the ash from 1 gram of coal weighs 10 milligrams the coal is one one-hundredth (0.01) or 1 per cent ash.

Example: Three grams of coal are taken, which after drying require 2 grams and 310 milligrams (2.31 grams) to balance. What is the air-drying loss?

	Grams.
Original weight	3.00
Weight after drying	2.31
Loss of weight	.69

As 0.69 represents the loss for 3 grams, the loss for one gram is 0.69 divided by 3; that is, 0.23 grams = 230 milligrams. Each 10 milligrams corresponds to 1 per cent; $230 \div 10 = 23$; the air-drying loss is 23 per cent. The same result is obtained by the familiar method of calculating percentages. Three grams of sample contains 0.69 grams of moisture.

$$\frac{\text{Per cent of moisture}}{100} = \frac{0.69}{3} \qquad \text{Per cent of moisture} = \frac{100 \times 0.69}{3} = 23$$

If 1 gram of this dried coal, after burning, leaves 120 milligrams (0.120 grams) of ash, the percentage of ash is $(120 \div 10)$ 12, as each 10 milligrams of ash represents 1 per cent of the original gram. Calculated by method of percentage the result is:

$$\text{Per cent of ash} = \frac{100 \times .120}{1.00} = \frac{12.00}{1.00} = 12$$

Should one-half gram of coal be taken, the ash of the coal last considered will weigh 60 milligrams (0.060 grams) and as with one-half gram sample each 5 milligrams of ash corresponds to 1 per cent, the result is $60 \div 5$, or 12 per cent ash. Calculated by percentage, the result is:

$$\text{Per cent of ash} = \frac{100 \times .060}{.5} = \frac{6.0}{.5} = 12$$

In order to give the reader an idea of the relative accuracy of an analysis made with the field outfit as compared with that made in the laboratory the results of tests made by the writer and others on duplicate samples analyzed by the Pittsburgh Laboratory of the Bureau of Mines are given in the following table:

Comparison of results of analyses of samples of coals by laboratory of Bureau of Mines and by Geological Survey's field apparatus.

	Kind of coal.	Bureau of Mines laboratory No.	Percentage of ash in air-dry coal.	
			Bureau of Mines.	Field outfit.
1	Lignite	[a] 14729	12	13
2	Bituminous	[b] 17707	16.2	16
3	.....do	[b] 17705	25.6	25
4	.....do	20168	17.95	17.8
5	.....do	20170	20.9	21

[a] U. S. Bureau of Mines Bull. 85, p. 60, 1914.
[b] U. S. Geol. Survey Bull. 541, p. 412, 1914.

Field analyses 1, 2, and 3 were made by the writer in camp at 30 to 40 miles from a railroad, analysis 1 in North Dakota, under adverse conditions because of high winds. Tests of the apparatus have been made by many members of the Geological Survey on coal of all grades, from Rhode Island anthracite to brown lignite.