228. *Studies in the Composition of Coal. A Method of Estimating the Decomposition Points of Bituminous Coals.*

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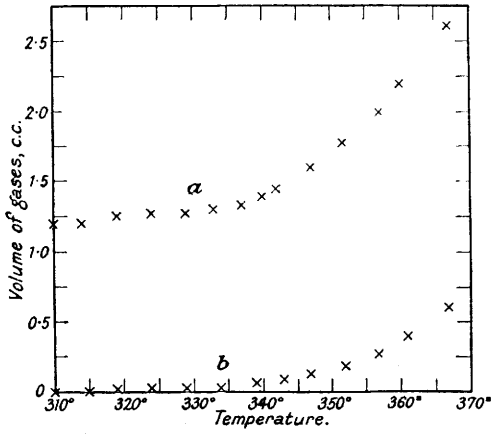
In a study of the composition and properties of a bituminous coal, measurement of its decomposition point, *i.e.*, the temperature at which breakdown of the structure of the ulmins begins, is of considerable value, for that temperature is characteristic of the " rank " of the coal.

Holroyd and Wheeler (J., 1928, 3197) determined the decomposition points of a number of bituminous coals by long-continued distillations under vacuum with gradual increase in temperature. The decomposition point is then marked by an abrupt change in both the quantity and the character of the gases and oils distilled. This method, which gives accurate values within $\pm 2°$, is laborious and may take several weeks to complete. A shorter method was therefore sought.



The method evolved has been based solely on the sudden change that occurs in the quantity of gases yielded by a coal when its decomposition point is reached. During preliminary experiments it became evident that the rate of rise of temperature of the coal was the most important factor in determining the decomposition point in this manner. With a given coal, the more rapid the rise the higher the temperature at which the change in the rate of evolution of gases became pronounced; in fact, it was necessary to approach the exceedingly slow rate of heating adopted by Holroyd and Wheeler (about 25° per week) to obtain absolute values. Relative values are satisfactory, however, provided that, for a series of coals of different carbon contents, the differences from the true values are constant.

The method, which is described in detail later, involves raising the temperature of 1-g. charges of the finely divided coal at the rate of 0·5° per minute, and measuring periodically the volumes of gases withdrawn continuously by a vacuum pump. From the graph obtained on plotting the yield of gases against temperature, as in *a* in the fig., the decomposition point under the conditions of test can readily be assessed. We have thus determined the relative decomposition points of a number of bituminous coals, of carbon content ranging between 77 and 90%, including most of those used by Holroyd and Wheeler.

Where comparison can be made, the temperatures have been found to be 10—15° higher than the absolute values, which can therefore be estimated with sufficient accuracy by subtracting 12° from the temperatures indicated by this rapid method. The results recorded in Table I illustrate this.

TABLE I.

The Decomposition Points of Bituminous Coals.

Description of coal.	Carbon content, %*.	Decomposition points. Absolute.†	Relative.	Difference.
Seven Foot (Kingsbury)	77·0	295—300°	312°	15°
Barnsley (Brodsworth), vitrain	81·2	305—310	320	13
Barnsley (Brodsworth), durain	82·7	310	324	14
Wigan Six Foot (Maypole)	82·8	315—320	333	15
Parkgate (Cadeby)	83·4	325—330	337	10
Silkstone (Rockingham)	83·7	325—330	337	10
Parkgate (Rotherham Main)	85·0	—	346	—
Virtuewell (South Blair)	86·3	—	350	—
Six-Quarter (Haig)	86·6	320—325	335	13
Busty (Tudhoe)	87·4	335—340	352	15
Busty (Beamish Mary)	88·4	340—345	355	13
Pensford (No. 2 Pensford)	88·4	—	360	—
Two Foot Nine (Ferndale No. 7)	89·8	360—365	374	12

* Calculated on the "pure coal" basis
† See Table III, J., 1928, 3197.

It will be noted that, apart from the Six-Quarter (Haig) coal (which is abnormal in other respects), the relative decomposition points increase with the carbon contents of the coals.

EXPERIMENTAL.

In order that the sample of coal should be heated uniformly, it was necessary that it should be finely divided and distributed as a thin layer over the heating surface. The size of particles found most suitable was through 100 and on 200 mesh (I.M.M. standard). The retort ultimately adopted was made of hard glass and provided a flat heating chamber, measuring 5 × 2·5 × 1 cm., in which 1 g. of the powdered coal formed a layer 3 mm. thick. An outlet tube, 20 cm. long, made connexion through a ground-glass joint with an automatic mercury Sprengel pump, a side tube in the pump-connexion enabling a thermocouple to be passed within the retort so that its hot junction was embedded in the charge of coal. The ground-glass joint was held together by spiral springs, and cooled by a water-jacket. The retort was heated in an electric resistance tube-furnace, the temperature of which could be raised at a regular rate by suitable adjustment of an external resistance.

When making a determination of the decomposition point of a bituminous coal, the empty furnace was heated to a constant temperature of 250—300° (dependent on the carbon content of the coal to be tested); the retort containing 1 g. of the powdered coal (which had been evacuated) was then pushed into the furnace and allowed to attain its temperature. During this preliminary heating period, occluded gases were continuously removed by the pump. The temperature of the furnace was now raised at the rate of 0·5° per minute and the gases evolved collected at the delivery tube of the pump, which maintained a vacuum, within 3 mm., throughout the heating. For the collection of the gases, a graduated glass tube, 0·5 cm. in diameter, was used, and their volume could be measured accurately within 0·02 c.c. at regular intervals, usually of 4 minutes. On plotting the yield of gas against temperature, as in the fig., the point at which a rapid evolution of gas occurred could readily be determined.

A check on the decomposition point as thus determined could be obtained, if the heating of the sample was not continued too long, by allowing the residue to cool in a vacuum and reheating it; for the decomposition of the ulmin is gradual. Such a check could, in fact, be more accurate than the original determination, for the decomposition of the ulmins in the residue was not masked by the evolution of occluded gases or gases arising from the decomposition of minor constituents of the coal. For example, the decomposition point of the Silkstone coal as determined directly (*a* in fig.) was 337°. Heating was continued until the temperature had reached 367°, *i.e.*, during 1 hour. The residue was allowed to cool to 300°, a vacuum being maintained, and was then reheated at the same rate as before. Hardly any gas was evolved until a temperature of 337° was reached (see *b* in fig.), but thereafter the volumes were but

little less than during the original heating. It may be noted that with the Six-Quarter (Haig) coal, which has an abnormally low decomposition point for its carbon content (see Table I), reheating indicated a second decomposition point at a higher temperature.

The Effect of the Rate of Heating.—It will be understood that a too rapid rise in the temperature of the sample of coal would cause the sudden evolution of gas, indicative of the decomposition point, to be registered at too high a temperature, even if the decomposition of the ulmins were not gradual. The records in Table II show the effect of varying the rate of heating between 0·3° and 1° per minute for two coals.

TABLE II.

The Effect of Rate of Heating on the Decomposition Point of Bituminous Coals.

Rate of heating, per minute.	"Decomposition point."	
	Pensford.	Busty (Tudhoe).
1·0°	372°	365°
0·8	366	359
0·5	360	352
0·3	355	346
"nil"	—	337

If the values for the "decomposition points" of the Busty coal are plotted against the rates of heating, it will be found that they lie on a straight line which cuts the axis for a rate of heating "nil" at 337°, *i.e.*, the value found for the same coal by Holroyd and Wheeler. A convenient rate of heating, which has been adopted as standard, is 0·5° per minute. With this rate of heating the values obtained for the decomposition points of bituminous coals can be regarded as uniformly 12° higher than the true values.

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