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### The Ignition Temperature of North Dakota Lignite

George Anthony Brady

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#### **THE XGNITION TEMPERATURE OF NORTH**

DALOTA LIGITTE

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George Anthony Brady.

& Thesis submitted to the faculty of the College of Engineering at the University of North Dakota in partial fulfillment of the requirements for the degree of Master of Soience in Chemical Magineering.

> University of Nerth Dakota. May 28, 1930.

UNIVERSITY OF NORTH



This Thosis, presented by Coorge Anthony Brady as a partial fulfillment of the requirements for the degree of Master of Seience in the University of North Dakota, is hereby approved by the Committee in charge of his work.

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#### Introduction.

It is very difficult. if not impossible. to accurately define the ignition temperature of a coal. This may be verified by a study of the many attempts appearing in literature to formulate such a definition. If one should apply the generally accepted concept of ignition point to coal it would refer to the temperatupo to which coal must be heated in order that it shall burst into flame. The flame from burning coal is due to combustion of volatile products of distillation from the coal and the nature of these volatile combustibles depends largely on surrounding conditions of heating<sup>2</sup>. Hence for a given coal these varying conditions cause such large fluctuations in the flaming temperature that its determination is valueless. Similarly, to define ignition point of a coal as the temperature at which self-heating begins would be equally unsatisfactory. The accompanying study supports other more extensive experimental evidence<sup>2</sup> that even at atmospheric temperatures some coals will combine with oxygen in a reaction accompanied by the emission of heat. The reaction is not rapid, to be mare, but nevertheless it is very much in evidence. Hence if one defines the ignition point of coal as the temperature at which self-heating bogins, the ignition point of lignite may be placed at a point below atmospheric temperature.

In 1918 Whooler<sup>3</sup> investigated the ignition temperatures of a number of English coals from a standpoint of the oxidizing tendency of the various coals under given conditions. His method consisted of heating a sample of the coal at a constant rate in an electrically heated sand bath. Air was dried by passing through a tube

of calcium chloride and then was admitted to the combustion tube containing the sumple. Simultaneous readings of the coal and sand bath tomperatures were recorded. The results were plotted as two time temperature curres, one of which defined the heating rate of the sand bath, the sther defined the heating rate of the coal. Whooler designated the point there these two curves intersocted as the relative ighition temperature. Since information gained by use of this method seemed to be of both interest and value in connection with problems involved in a study of the oxidizing tendency of bituminous comis, the method was extended to lignite in the present investigation. It was not, however, found to be generally applicable to lignite because of its relatively high moisture content.

Arms<sup>4</sup> made a thorough study of the heating curves of bituminous coals and adopted as the ignition point the temperature at which the coal assumed a uniform glow when heated by an external source. His nothed of heating the coul differed from other methods of the same nature, in that the oven temperature was not increased at a constant rate, but wather the Flow of current in the heating elements of the furnace was kept constant. Arms found the glow point to be rather definite in the heating curve and that it could be checked by use of various types of apparatus. On the strength of these facts he assumed the glow point to be the most logical point to solect as ignition temporature. It appears in the zone of 400-6000 C., depending on the nature of the coal. When coal is heated in air a number of important reactions are known to take place before the temperature of flow point is reached. The

reactions are not concerned in the method of Arms but nevertheless are of importance and can not be neglected.

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In 1925 Parr<sup>5</sup> referred to the temperature at which carbon diouido is ovelved as the sritical exidation temporature for coal in storage and at that time designed an apparatus to indicate the initial appearance of carbon diexide when coal in the presence of oxygon is heated by an external source. The apparatus used was casontially similar to that dosigned by Wheeler with the exception of the additional carbon diomide incleater mentioned above. This unit used a solutica of barium hydrowide as the indicating medium. Gas from the combustion thee was directed against a nonsle from which dripped the barium hydroxide solution. Any CO2 present in the gas expelled from the combastion tube precipitated barium carbonate from the indicating modium.

The temperature of evelution of carbon diexide suggested by Parr as an index to the dritical exidation temperature was the point adapted as ignition temperature in the present investigation of Dakota lignite. This point marks the bosinning of the second stage of exidation of coal as it is explained by the "Carboncomplex" Theory. It may also be regarded as the beginning of the first stage of exidation that occurs above atmospheric temperature because carbon diexide in one of the initial products of conbustion most in evidence. It is not contended that this point on the heating curves of lightte designates the stage of rapid heating irrespective of all other conditions. Neither could it be assumed that the ultimate attainment of the kindling temporature is inevitable when lighits is once houted to the point of evolution of

COg either by an external source or by the heat liberated spontancously from its own oxidation. It seems there has been a genoral tendency to confuse the torms ignition temperature of a coal and its oxidising tendency. Recently Resin<sup>7</sup> pointed out that there is a fundamental difference in the two terms. He defines ignition temperature as "the statistical coordination of a temperature which is dependent upon experimental conditions" and tendency to spentencous combustion as "the property possessed by a substance of heating spontaneously at a rate rapid enough for the ignition temporature to be attained." It is obvious that in the case of spontaneous combustion the ignition temperature is not nearly so inportant as the amount of heat necessary to raise the temperature of the coul to the ignition point. For example, the flame of a match reaches a temperature of approximately 700° C, far above the ignition point of the coal and yet it is generally known that a match alone will produce only enough heat to ignite very small quantition of coal.

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It is possible that ignition temporature assumes its greatest significance in connection with problems involved in the combustion of powdered coul. This practical incontive was one of the fectors which prompted the prosent investigation on lignite. It is generally contended<sup>e</sup> that one of the chief objections to powdered coal in the past has been the large combustion space required to permit complete combustion of the coal without excess sive deterioration of the furnace lining due to the impingement of flame on the brick work. High-volatile coals, therefore, on account of their greater ease of ighition may have an advantage

over the low volatile souls. Aside from the above mentioned possible advantage, lignito has properties which render it well suited for use in the powdered form. For example, specially designed grates are necessary for coonomical combastion of lignite oring to its tondency to slack, followed by subsequent loss due to sifting through the grates. Since it seemed that the ease of ignition is an important factor in the combustion of powdered fuel and the inhorent properties of lignite suggest that it might be used advantageously in this form, it was desired to investigate its ignition temperature. The information was also desired for the puPpose of comparing the ignition tomperature of raw lignite with that of lignite dried by the Fleisener mothod.

The following is a meport of the results obtained from a study of the offect produced on ignition temperature by such physical properties as particle size, moisture content, and provious history of the coul.

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APPARATUS FOR DETERMINING THE IGNITION TEMPERATURE OF LIGNITE

#### Exporincatal

#### Apparatus:

The apparatus used in this investigation is shown diagrammatically in figure 1. The furnace A was constructed purposely for these experiments with the aim in view of having one in which the temperature could be accurately controlled. It consists of a heating chamber 5 inches in length by 2 inches in diameter, surrounded by a S-inch layer of asbestos fiber held in place by a sheet metal sleeve and slate asbestes ond picces. The heating olement was made from 24 fact of No. 31 B. & S. gauge chronol "A" wire wrapped on the refractory which was furnished with grooves built in the form of a spiral on its surface. Temporature was controlled by means of a sliding contact rheostat in series with the furnace circuit. Two SOO-degree aitrogen-filled thermomoters were used to indicate the temperatures of furnace and coal, with the exception of two series of runs in which the thermometers were replaced by platinum-rhodium thermocouples. The accuracy of both thormomoters and thormocouples was checked at five different points by comparison with the boiling points of the following 11quidat



The combustion tube (B) was made of pyrex glass with a capacity of 25 grams of lignito. The outlet of this tube protrudes through an opening in the bottom of the furnace and leads to the cooling coil (C) which is 60 inches in length and surrounded by a water jacket through which tap-water was circulated to cool the gas expelled from the combustion tube.

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The mixing chamber (D) affords a compartment in which gas from the furnace is mixed with the electrolyte.

The system used to indicate the initial appearance of carbon dioxide embodics a number of the features suggested by Taylor and Taylor<sup>0</sup> in their description of an automatic carbon monoxide recorder. The conductivity coll (E) made from a 1 1/4 inch glass tube is fitted with two gold platinized cleetrodes 1.5 cm. in diameter and placed 1.5 cm. apart. These electrodes are connected in series with a milliancter (G) and ten dry colls (H). A double-pole knife switch was placed in the eircuit in such a way that the current could be passed either through the coll or through a standard 500 chm resistance (not shown in diagram). With the resistance coil in series, ten new dry colls produced a current of 32 milliamperes in the circuit. By this method the strength of the colls could be tested regularly and replacement made when necessary. The voltage adjusted in this manner produced a current of approximately 10 milliampores through the cell filled with .1 H ammonium hydromide at 26<sup>0</sup> C. Tomperature of the coll was controlled by means of the constant temperature bath (F). The cleborately designed pump used by Taylor to maintain a conatant ratio between gas and reagent flow was replaced in these

experiments by the capillary (M) and manometer (L) used to indieate gas flow and the constant pressure head (I) to deliver the ammonium hydroxide at a chnatant rath. The bottle (3) served as a reserveir from which solution was drawn to maintain a constant level in (I).

Gas to be passed through the coal was circulated through the train of wash bottics (Kg N. O. F) fälled with the proper solutions to oither dehydrate or wet the gas to the desired moisture content. These bottles are of the standard "Milligan" type, each one of which has the absorbing capacity of a bottle 45 inches high.

#### General Procedure:

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In order to meet desired conditions for different series of experiments a number of modifications (to be described later) were made in both apparatus and method of procedure. The general method adopted, however, was as follows: Coal ground to the desired size was placed in the combustion tube within the furnace which was heated at the rate of 2.5° C. per minute. The desired gas (either onygen or nitrogen) was gassed through the wash bottle (F) which contained a 40% solution of potassium hydroxide to remove all traces of earbon dioxide, and then through the romaining absorbing bottles. to cither dehydrate or wet the gas to the desired moisture content. After leaving the wash bettles the gas was notered and pacced through the coal. From the combustion tube the gas flowed through the cooling coil and then was mixed with a .1 N solution of ammonium hydroxide continually flowing through the coll at a constant rate. Temporaturo of the furnado was recorded at 4-minuto intorvels while temperature of the coal dad milliamperes of current through the

coll were recorded simultaneously at 2- minute intervals. The results were plotted as two time-temperature curves and the initial change in conductivity mecorded as the CO2 point on the curve representing the heating rate of the coal. The mechanism of the COg inlicating system may be stated as follows: Pure oxygen or nitrogen has no effect on the conductivity of ammonium hydroxide but any CO<sub>R</sub> present will be abserbed and the conductivity of the solution increased in a direct proportion to the amount of COA absorbed. The sensitivity of the dog indicator, therefore, is dependent on the relative amounts of gas and solution flowing through the system. After some experimentation it was decided that 70 co. of gas and \$0 co. of ammonium hydroxide constituted the most. desirable ratio. With gas flowing at this rate it was calculated that approximately one minute intervened between the time it left the combustion tube and the time it entered the cell se accordingly the first change in conductivity was marked on the time-temperature curve one minute back of the point at which it was first observed.

#### Results and Discussion.

Comparison of the ignition point of lignites from different mines in North Dakota.

This series of experiments opnaisted of an investigation of ton samples of lignite thken from different mines throughout the State of North Dakota. The purpose was to assertain if there is any material difference in the ignision temperature of lignites from different localition. The samples were all received in sealed containers from the following corporations:

Kaife River Coul Mining Company, Hobron Brick Company, Lohigh Sriquetting Company, Truax-Traer Coal Company, Now Salon Mino. Pittebusch Coal Mining Company, Stevens Bresl Coal Company, Washburn Lignite Company,

Seranton Collieries Company,

Light Bros. Coal Company.

Bismarck, Horth Dakota Hobron, Nerth Dakota Diokinson, North Dakota Columbus, North Dakota Now Salon. North Dakota Diekinsch, North Dakota Garrison, North Dakota Wilton, North Dakota Seranton, North Dakota

Noonan, North Dakota.

Sach sample was air dricd twenty-four hours. The coal was then ground to the proper site so that it passed through a downesh sieve and was retained on a CO-mosh and then sealed in an airtight container until it was to be tested. Temperature moasurements were made with thermomotors. For this series of runs exygen was used that was first passed through a solution of potassium hydroxide to remove COg and then through the remaining wash bottles filled with recontly boiled distilled water to saturate the gas.



An inspection of table 1 reveals no material difference in the ignition temperatures of different lignites. A striking sinilarity may be observed also in the nature of the heating curves, an example of which is shown in figure S. The proken, in this, as in all other graphs in this report, defines the heating rate of the furnace. Other curves in this series are not shown since they would furnish ne additional information because of their general similarit



Comparison of ignition temperature of lignite with those of higher rank coals.

In this series of tosts a comparison was made of five difforent ranks of coals, including lignite, Wyoming bituninous, Illinois bituminous, Focahontas, and anthracite. The coals were air dried one day then ground to 40-60 moch and run in the manner described before using saturated oxygen. Pyromoters were used to indicate the temperatures. Each sample was subjected to two heat treatments; that is, it was treated as described above then allowod to cool to room temperature without removing from the furnace and was reheated under exactly the same conditions as in the first treatment. Duplicate runs were also made on each sample. Results of these tests arelisted in table 2, showing results of first heat treatment and table 3 showing results of second treatmont.



It will be noted that both the COa points and the eressing points of the heating curves of lightte are somewhat lover than these of other lighites, listed in table 1. A possible explanation of this might lie in the fact that the lignite used in this series was taken from the cars and honge had not been subjected to the same conditions as those which were sealed in containers at the mines. It is interesting to observe the decided increase in temperature at which COg was evolved when the scale were heated the second time under the same conditions. This phonomenon is more marked in the younger coals than in those of the higher rank. The erossing tomperature of the curves, however, are lowered in case of the younger conls.

Table & shows the inerease in the two indicated points when the sample was heated the second time.





Apparently the first heat treatment temds to affect materially the reactivity of carbon in the coals of lower fank. No attompt was made to stady the mochanism of this action but it is possible that a number of factors might be evolved such as a physical change in structure of the porce and the action of onygen

on the coul at higher temperatures. A marked difference can also be observed in the shape of the heating curves representing the first and second treatments of the same sample of lignite as is shown in figure 5. It can be observed from tables 2 and 3 that this difference is less pronounced in the higher rank coals; anthracite being the least affected by the first heat treatment. As was expected, there is a marked increase in ignition temperatures of coals ranging in rank from lignite to anthracite.

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Study of the effect of particle size on ignition temporaturo.

Since combustion is a surface phenomenon, and for a given mass of coal the surface exposed is a function of the particle size it was deemed desireble to investigate the relation between ignition temperature and size of the coal particles. The Eincaid lignite used in these tests was air-dried two days and after grinding was graded according to size as follows:

> 80 - 40 mosh 60 - 80 mosh 00 - 100 mosh  $100 - 150$  mosh 150 - 200 mesh Through 200 monh

Samples from each cut were subjected to the treatment desoribed in the general procedure. Oxygen was saturated for use in this series and pyromoters used to indicate temperatures. Results are shown in table 5.

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The results of this series of tests were somewhat disappeinting. As indicated in table 5, the ignition temperature of duplicate runs did not check closely snowgh to warrant the formation of definite conclusions. This was attributed to the practical inpossibility of obtaining a representative sample of each separate cut. For example, if the lignite was ground so that all would pass through a 60-mesh sicve, a relatively large percentage would pass through 200-mosh. The results, hovever, do seem to indicate a higher COg point for the more finely ground portions. This was to be expected from the knowledge that the large amount of surface garlot go exposed by the small particles should increase the absorbing power. In a group of samples sach as those used in this series, where the moisture content may be dssumed to be the same, it appears that the

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Juridic ( crossing points of the meating curves may give some indication as to the relative exidising tendency of the coals, since for the deeresse in particle sine there is an accompanying deeresse in orossing temperature.

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Determination of the effect of moisture content on ignition temperature of lignite.

The effect of moisture was studied by testing a number of samples ranging from 0 to 100% relative vapor pressure. Kineaid lignito "da recoived" from the car was used. The coal was in frozen condition, which lowered its vaper pressure sufficiently to maintain a moisture content of 53.7% as received at Grand Forks. The lignite was ground to 60-60 mesh in the wet condition; both operations of grinding and serecaing being carried out rapid. ly and with small quantities to provent, as far as possible, moisture less by evaporation. Portions of the sample were placed in desiccators over solations of known relative vapor pressuret care being cheerved that each portion contained sufficient moisture to exert a higher relative vaper pressure than that of the solution ever which it was placed in order to assure dehydration of the coal rather than hydration. This precaution was deemed necessary as a result of an investigation on the vapor pressure of lignite by Cauger and Lavine<sup>10</sup> who found that the dehydration of lignite is not reversible. The solutions used and their rolativo vapor pressuro<sup>22</sup> are given in table 6.



The lignite was desiceated pver these solutions for a period of 21 days at which time it was assumed that the vaper presence of the coal had reached equilibrium with that of the solutions. The grams of vater per gram of dry lignite was determined from the known relative vapor pressure by use of data collected by Cauger and Lavino<sup>18</sup>. This set of tests was made in a manner similar to that described before but with a slight modification in the apparatus. Before entering the combustion tube exygen was eiroulated through wash bottles containing the solution over which the sample to be tested had been desigeated. This modification was made to prevent moisture loss from the coal at lover temperature. All

temperature measurements for this series were made by use of thermometers. The results as tabulated in table 7 show a slight tendeney toward decrease in ignition temperature with decreasing moisture content although the rule is not followed rigidly. In this series very little significance can be applied to the crossing points of the heating curves, particularly those representing samples of high moisture content. It seems reasonable to assume there are for spots of local heating in the coal up to the time of evolution of carbon dioxide and honce a thermoneter in any portion of the coal should indicate the approximate temperature of the mass, but it was established that this does not hold during the stages of more rapid heating. In the vicinity of 200° C. there was found to be a variation of approximately 50° C., range ing from top surface to bottom of the coal column. Honce the erossing temperature of the curves can not be used even as a means of comparison when the coals in question are at a different moisture content. There is a material shrinkage in volume with loss of moisture from the coal and since the decrease in volume is a function of the smount of moisture lost it can be seen that it would be inpossible to arrange the thornometer at the proper depth se that it would in every case be the same distance from the top of the coal surface after contraction of the volume had cocurred with loss of moisture in the vicinity of 1000 0. With dry lignito, however, bmly a slight deeresse in volume was observed as a result of heating to 200°. The effect of various moisture contonts on the shape of the heating curves of lignite



is shown graphically in figures 4, 5, and 6.

In connection with this series of tests a comparison was made of the heating curves using vot and dry exygen. Results are tabulated in table 8 below.

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An inspection of bable 8 shows very little difference in the rate at which wet and dry oxygen react with coal and also that oxygen saturated at room temperature does not carry enough moisture to greatly effect the shape of the heating curves.



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Relative comparison of the tendency of coals to exidise at various temporatured.

To investigate the relative tendency of a coal to oxidine at various temperatures a fow experiments were conducted in a mannor vhich differed demovhat from the general procedure adopted in all the previous work of this investigation, in that the temperature of the furnace was kept constant and the use of the coal temperature above that of the furnace was observed, both dry coal and dry oxygon being used. Various temperatures maintained by the furnace were 70°, 80° and 90° C. all of which are above the COg point of dry lignito. The rosalts shown graphically in figure ? indicate that under the special conditions of radiation between coal and furnace, the eritical oxidation temperature of dry lignite lies between 80° and 90° centigrade.

At this point in the investigation the action of air compared with that of emygen on lignite was also studied. Results show no marked difference in the points of evolution of CO, from coal when heated in air or oxygen. As should be expected, the rate of self-heating of the coal was more rapid in exygen than in air. In connection with this sories of tests the effect of heating lignite in an atmosphere of nitrogen was also studied. Using dry nitrogen and dry lignite CO2 was evolved at temperature of approximately 105° C.

Figure 8 indicates that the appearance of COg was accompanied by an endothermic reaction which was in evidence until a temperature of 166º C. was attained. These results are not in

13 accordance with those obtained by Hollings and Oobb who heated bituninous coal in an atmosphere of nitrogen and observed narked excthermic reactions between the temperatures 150° and 250° C. No such reaction can be observed in the heating curves of lighite. Thoro is a slight change in slope of the heating curve shown in figure 8, but this was attributed to the increased temperature gradient between furnace and coal as a result of the endethermic reaction between the temperatures 110° and 165° C.

Effect of ageing on ignition temperature of lignite. Throughout a period of cight weeks a series of tests were made on a sample of lighite to study the effect of ageing on ignition temperature. The sample was collected, ground to 40-60 mesh and sereened in the wet condition. It was first run "as received", then exposed to air in the laboratory and tested at different intervals throughout the following cight weeks by heating in an atmosphere of saturated bzygen as described in the gencral procedure. The remilts shown in table 9 indicate a high COs point for the wet coal. With loss of moisture the COn point is at first correspondingly decreased then tends to be raised again by the subsequent ageing. Wigures 9 and 10 are plotted from data takon in tests on the coal at two stages of the agoing process. The shape of the heating curve was not noticeably effected after the second day of ageing. Figure 10 clearly indicates the presence of an exothermic reaction between the point of evolution of COa and the flat portion of the curve resulting from vaporization of the water. This was characteristic of all the heating curves of

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Lignites in which CO2 appeared before the greater portion of moisture was vaporized and it is, therefore, attributed to ordinary combustion since it was not observed in cases where the COg point was at a relatively high temperature.



#### Conclusions

1. There is no definite point on the heating curves of lignite which marks the transition from slow to rapid stage of solf hoating.

- The point of evolution of earbon dioxide suggests itself as B. the most logical point to choose as ignition temperature since it can be duplicated with a fair degree of accuracy and marks the beginning of the first stage of combustion that cecurs above atmospheric temperature.
- There appears to be no material difference in the ignition S. temperatures of lignite taken from different localities in North Dakota.
- Within the range of 20 to 200 mesh thore is a general ten-4. doney toward raising of the CO<sub>R</sub> point with docrease in particle size.
- There is a gradual increase in ignition temperature of **S.** coals ranging in rank from lignite to anthracite.
- Ignition temperature of lignite is raised with inerease in 6. moisture content.
- Ignition temperature of lignite is decidedly effected by 7. exposure of the coal to air after mining.

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**FURTHER 3TU1^I|£S IN THE CARBONIZATION OF NORTH DAKOTA LIGNITE.** 

- **1 . The Gaseous roduets of Low Temperature** Carbonization of Lignite.
- **8. The Effect of Aluminum Chloride Hydrate I** and Pitch on the High **Temperature** Carbonization of Pre-Carbonized Lignite.
- **3. The Effect of Blending with Bituminous Coals** on the High Temperature Carbonization of Lignite.

**by James Roscoe Taylor** (B. S. in Chemical Engineering, University of **Ncrth Dakota, 1 9 8 9 ).**

A thesis submitted to the faculty of the College of Engineering of the University of North Dakota in partial fulfillment of the requirements of the degree of Master of Science in **Chemical Engineering.**

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University of North Dakota May 29, 1930.