THESIS
ON
COMPOSITION OF WASTE CHIMNEY GASES
FOR THE DEGREE OF
B. S.
IN THE
SCHOOL OF CHEMISTRY
BY
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1890
1. Chemical Composition of Coal

Coal is a dark amorphous substance of vegetable origin underlying about 35,000 sq. mi. of this state. Its color varies from a brown to jet black. The general divisions of coal are Bituminous or Soft and Anthracite or Hard coals. The former has a very irregular fracture, dull or shiny luster, and a brownish color when powdered; while the latter has a somewhat conical fracture, bright, lustrous surface and color jet black when powdered.

Coal consists mainly of carbon and hydrogen; it also contains in smaller quantities, nitrogen N., oxygen O., and sulphur S. The residue of incombustible matter called ash contains principally, $\text{SiO}_2$; $\text{Al}_2\text{O}_3$; $\text{Fe}_2\text{O}_3$; $\text{Fe}_3\text{O}_4$; $\text{S}_0$; $\text{P}_2\text{O}_5$; $\text{CaO}$; $\text{MgO}$; $\text{K}_2\text{O}$; $\text{Na}_2\text{O}$. The composition of ordinary bituminous coal...
Commonly used as fuel, is, carbon taken as 100 %.
C - 81.12 %;  H - 6.10 %;  N + O - 11.25 %;  ash 2.53 %.
Sp. gr. of coal 1.25-3.
Anthracite in comparison with bituminous contains a higher % of C and ash, but a smaller % of H, N + O.
This difference is due to the more perfect carbonization of the vegetable matter from which coal is formed. The average composition of anthracite, C = 90 %.
C - 90.40 %;  H - 2.48 %;  N + O - 2.48 %;  ash 4.67 %.
Sp. gr. of coal 1.462.

**Combustion of Coal and Its Products.**

Combustion is the application of heat to a combustible, causing chemical union of the supporter with the combustible, with the production of new chemical compounds, either gaseous...
liquid, or solid, and causing an increase of temperature due to this union.

When heat is applied to coal there takes place two distinct chemical reactions. The first takes place at a low temperature, by the absorption of the sensible heat by the coal and driving off the more volatile products. This process does not need the presence of air or supporter and results in what is called "destructive distillation." The products of this distillation are, when heat is first applied, the decomposition of the coal and the rearrangement of the elements to form -

1st Hydrogen, H. - 2nd Marsh gas, CH. 4 - 3rd Carbonic oxide, CO. - 4th Aliphatic gas, C 2 H 2 - 5th Benzene, C 6 H 6

and other hydrocarbons of this group. 6th Water vapor.

The hydrogen and marsh gas are contained in the coal in their natural state as they are simply driven off. The carbonic oxide CO. is formed from the
carbonic acid \( \text{CO}_2 \), which is formed at a higher temperature, it being where the coal is in direct contact with the red hot coals. The carbon at this point burns to \( \text{CO}_2 \) and this passing up through the coal, takes up another atom of carbon forming two molecules of \( \text{CO}_2 \) in place of one of \( \text{CO}_2 \). The production of this carbonic oxide \( \text{CO}_2 \) is a great loss in the amount of heat obtained from a certain amount of coal. These above gases are all volatile at this temperature except the water vapor. 7th Ammonia \( \text{NH}_3 \) and other compounds of nitrogen and sulphur, which are given off at this temperature are also volatile and combustible.

As these gases are given off at a comparatively low-temperature they are to some degree condensed at the fire bridget and may suffer the rise of temperature in the furnace and thus be
burned, to the production of more heat and the true products of combustion, CO₂ and H₂O. But this condensation is not always complete so more or less of these gases escape through the chimney, especially if the draught be strong. Hence this reduces the amount of heat that might be obtained from the coal used.

The residue left from the volatilization of these gases consists of the non-volatile, but combustible portions of the coal called "coke" which consists of the carbon and mineral constituents of the coal. The second step necessitates an elevation of temperature and a supply of air. If the combustion to perfect the products of the second step are:
1st Water vapor 2nd Carbonic acid 3rd Nitric oxide 4th Sulphurous acid. Hence in the water vapor would be all the hygroscopic. In the Carbonic acid all the
carbon; all the nitrogen of the coal would be there mixed with a large portion of the nitrogen of the air, and all the sulphur would be in the sulphurous acid, and as these products or gases are of no use as heat producers they are made to pass out through the chimney and cause no loss of heat except the amounts used to increase their temperatures.

Theoretically this process takes place with an adequate supply of air and high temperature. In practice, however, this change does not take place perfectly, and in place of the CO₂, H₂O, N, and SO₂ the products of perfect combustion. (Theoretical quantity of O supposed to have been used and good coal.) we have besides these above named gases, some unburnt and some partially burned carbon, some unburnt and partially burned products
of "destructive distillation." viz.: hydrogen, hydrocarbons, carbon dioxide, water vapor, carbon, and the excess of air used. This mixture constitutes the constituents of waste chimney gases commonly called smoke.

Smoke is the term applied to the mixture of water vapor and gases that come from the chimney with sufficient quantity of carbon to make it visible. This quantity of visible carbon is vastly over estimated, and it is really small in proportion to the vast quantities of water vapor, carbonic acid, nitrogen, etc., present and even smaller in proportion to the amount of fuel used, is being at its maximum, i.e., when smoke is thickest only 3% of fuel used. The loss it causes however in the heating capacity of the fuel used is far
greater than that of the subcutant hydrogen and hydrocarbons it being 1-4 and proportionately greater than that of the Carbonic oxide.

"History of Gases, principally CO₂, and steps in their discovery."

The term gas, now commonly applied to uniform substances, was first introduced by van Helmont about the middle of the 17th cent. But the scientific use of the term is of more recent origin. Gas, as used scientifically, is a substance possessing perfect elasticity and existing under a constant pressure a uniform rate of expansion for equal increments of temperature. Vapor, in distinction from gas, does not possess a constant rate of expansion for
equal increments of temperature although gas at its maximum density nearly approaches the state of vapor.

Van Helmont (1639–1644) was the first to take up the study of air and substances whose characters is different from atmospheric air and to show that they could be produced artificially. He first showed that the conditions which gases are generated are chiefly, combustion, fermentation, and reactions of acids on metals and calcareous substances but he was not at all acquainted with the distinction of the different gases thus produced. He described carbonic acid as the product of combustion of fuel and stated that it was present in mineral waters, caves, etc. and that it would not support life nor combustion, but goes no further in his account of "gas sylvestre" as he termed it.
In the early part of the 17th cent. the first and most remarkable inquiries on the subject of gas was made by Jean Peyr, a French physician, who first noticed the increase of weight on the calcination of tin and lead. Later he pointed out that the increase was limited and definite. His researches remained, however, for a century and a half floating through chemistry unexplained and unappreciated but to become later the key to the knowledge of gaseous bodies and basis of all present chemical science.

In 1665 Hooke professed in his "Micrographia" a theory which offered an explanation of the necessity for the presence of air in combustion. He considered that air was an all volatile of combustible matter at high temperatures and that this reaction was as rapid as to produce fire, which he considered a mode of violence. That when a limited supply of air would effect no
Combustion after a while that the solvent was saturated and no combustion would take place unless more air was supplied. The constituent of the air which he regarded as taking part in combustion existed in a highly fixed condition in nature, so that a given bulk of air contained vastly more of it than a given bulk of air. Hooke's views were developed later by Mayow in 1669 and by Willis in 1671, in which Willis adds that combustion not only consists in the reaction of the combustible with air but also in the air elimination of the combustible principles from the former.

Boyle in 1674 was the next investigator of note on the principles of combustion. He referred in his "General History of the Air" to the alteration of air in combustion and was satisfied that by combustion some thing was abstracted from the air.
but he defines it as "some vital substance diffused through the air whether it be volatile or some inanimate substance sidereal or subterraneal."

The next important investigator was Hales. In 1727 he was the first to use an apparatus which developed into our modern pneumatic trough. His investigations were mainly on the inflammability of gases and thus power of supporting combustion.

Contemporary with Hales was Black, who made important discoveries in regard to CO₂ when turned fixed air. His researches were based on the absorption and mixture of weight of carbonic acid and at some time was of constant air. He found that when magnesia was turned it lost weight and accompanying results he came to the conclusion that the mixture in case of lime, and decrease, in case of magnesia was due to the absorption of fixed air.
on one hand and loss on the other.

Cavendish in 1766 still further investigated CO₂ as a product of combustion and called it "inflammable gas." In 1783 which is an important period in chemistry much advance was made in the part played by air in combustion and in the chemistry of chimney gas, but as the experiments were not accurate—little detailed account is given.

Among the most recent investigators since 1783 are Priestley, the discoverer of oxygen—Marthi and Thales who investigated Marsh gas. While Gay-Lussac and Humphry are the most recent and accurate experimentors and on whose researches rests most of the structure of the chemistry of gases.
"Analysis of Gases."

The analysis of gases consists mainly in the power of certain reagents to absorb certain constituents of a gas without affecting the other portion. The three methods by which the analysis of a gas is accomplished are:

1. By direct absorption of the different constituents of the gas.
2. By combusting the gas with oxygen or copper oxide thus forming an easily soluble gas and water.
3. By first absorbing all the constituents of the gas except the one to be determined and subsequent combustion with the formation of soluble gases and water. Carbonic acid is usually determined first by absorption in either caustic soda or caustic potash. This absorption is effected by passing a measured
quantity of the gas into a vessel containing \( NaOH \) or \( KOH \), and agitating. The \( CO_2 \) is absorbed and the \( N_2 \) is determined by volume. Calculated by the decrease of volume.

Oxygen, \( O_2 \), is determined by passing the residue after the \( CO_2 \) has been extracted into a vessel or pipette containing either moist phosphorus or alkali.

Pyrogallic or copper and ammonia, agitating and the \( N_2 \) is determined from the decrease in volume.

Heavy Hydrocarbons, - Methane, \( CH_4 \), etc. are determined similarly by the use of fuming sulphuric acid.

Ozone Tones \( O_3 \) is determined by absorption in cuprous chloride in solution in HCl. or \( NH_4 \)OH.

Nitrogen is determined in all cases by the difference of 100% minus the \( N_2 \) of the other gases.

Hydrogen is estimated by burning the residue with twice its volume of oxygen. This combustion is usually effected by the electric current. In this
process the two volumes of H₂ unite with one of O forming water. The contraction is equal to these volumes. If H₂, hence the volume of H₂ is calculated by multiplying the total contraction by \( \frac{1}{2} \).

Methane and Carbonic oxide when present in small quantities together are determined by combustion with Copper oxide with the formation of CO₂ and H₂O. By passing the gas through a solution of Copper Sulphide in solution in HCl the CO₂ is absorbed, and the methane is alone combusted. The vol. of CO₂ formed is equal to the volume of CH₄ present. The volume of CO₂ is determined by first absorbing the methane and the volume of CO₂ formed equals the volume of CO₂ present. In all cases the temperature must be noted, as gas varies \( \frac{1}{273} \) its volume for every degree C., an error of 1°C would cause a large and considerable loss of the
gas present or may increase it to that amount.

"Introduction to Experiments."

My object in the following experimental work is to show the relative amount of loss sustained by the imperfect combustion of coal in the ordinary furnace. It is true that this loss has been investigated but not in the same line as the following researches.

Loss by incomplete combustion may occur in two forms 1st. The visible 2d. The invisible. It is with the latter form of loss that my experiments tends to show i.e. the loss in gases which escape notice and are not in the visible smoke. Many schemes have been introduced to do away with the visible loss...
and convert it into fuel in the form of smoke, abortion, and consumption, but it must not be overlooked that in perfect combustion there is no smoke and the problem of consuming smoke should be solved by securing a more perfect combustion of the fuel.

These schemes are for doing away with the visible portion of smoke (so-called), but no notice is taken of the invisible gases which escape with the visible and are valuable for heating purposes. Hence the problem I have attempted to solve is, What is the relative quantity of loss sustained by a furnace in the escape of these invisible gases? To solve this problem the thorough analysis of the chimney gases should be made, the amount determined and the loss calculated,
These escaping invisible gases are formed under different circumstances which tends to the production of smoke, for instance, when the fire is comparatively low and coal is thrown in the furnace a reaction takes place at the point at which the coal comes in direct contact with the burning coals, first the destructive distillation takes place and the second the burning of the carbon causes with the formation of CO₂. This CO₂ passing up through the heated coal takes up another atom of C, this forming two atoms molecules of CO in place of some of CO₂ a certain percent of this CO formed escapes the further action of the fire and passes up the chimney in a free state and hence is not utilized. Again, the fire being hot and a quantity of fuel added will cause reaction takes place but with the production gases
amount of 20 gases and smoke. Possibly the production may be further decreased by a very strong draught. I had made no experiments—however—in regard to the draught, and all the gases examined were collected under the following conditions: 1st. Moderate firing. 2nd. Average and 1/4 fuel used for moderate firing. 3rd. Large quantity of smoke produced. 4th. Sample taken immediately after firing. 5th. Regular time 2 min. to fill gasometer.

The following method of procedure was taken to make an ultimate analysis of the gases.

Collection of Sample

The manner in which I collected the sample was after the ordinary method by means of a gasometer.
The gasometer A. Fig 1 is filled with water and connections are made with the chimney B by inserting the glass tube C through the orifice Ia in the chimney about 1/2 the distance through as the funnel is near the center of the chimney. From the tube C a rubber tube D is run and is connected with the gasometer and aspirator D by means of a T tube E. The air in the tubes C, D, D' is replaced by chimney gas by means of the aspirator. The furnace is now fired up and the connections are closed with the aspirator and opened with the gasometer, D, D'. As the water
flows from d', the gas from the chimney is drawn in till it is filled as is shown by the indicator E. The stop cocks d and d' are now closed and the reservoir F is filled with water the cock d'' which communicates with the bottom opened and the water mus in forcing the gas under pressure, the gasometer is now transferred to the gas room and a sample is immediately drawn, and a direct analysis is made by the Osval apparatus. Fig II.

TREATMENT OF GAS."

The gas is measured accurately in the graduated cylinder B. Osval's apparatus Fig II and temperature Invited. The gas is then passed into the cylinder C which contains a saturated solution of corrosive to wash out all which remain
the CO₂, it is then run back into T₈ and measured, the decrease in volume notes the volume of CO₂ originally present. The gas is then run into N₂E while contains moist phosphorus in order to remove the oxygen. The gas is then passed back and forth into the phosphorus bulb till there is no further decrease in volume. The gas is then run into the CO₄ bulb to remove acid fumes and finally measured in the cylinder T₈. The gas is now passed into the bulb D which contains fresh Cu₂Cl₂ in solution in HCl to remove the CO₂ present.
The Cu₂Cl₂ absorbs slowly so that in order to get perfect extraction the gas has to be passed in and out the bulb several times. The gas is now freed of acid fumes by passing it into the KOH bulb and it is then measured and decrease of volume noted. Care must be taken that the temperature does not vary as gas expands, \( \frac{1}{273} \) its volume for each increase of 1°C. As a slight moisture would seriously affect the results especially with CO₂ account of its being present in small quantities.

The results of the analysis of gas taken in this manner is as follows:

Table I, p. 28.
The amounts 12.4, 3, and 7 are not recorded in Table I as they were badly in error.

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<th>CaO</th>
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<td>0.9</td>
<td>8.1</td>
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<td>10.1</td>
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<td></td>
<td>8.17</td>
<td>1.05</td>
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<td>1.05</td>
<td>78.68</td>
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<td>1.05</td>
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<td>106</td>
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<td>78.68</td>
<td>106</td>
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The amounts 12.4, 3, and 7 are not recorded in Table I as they were badly in error. The amounts were calculated by the methods of the analytical procedure.
either as saturated or unsaturated hydrocarbons and in small quantities.

The first operation proposed was to extract all the CO₂ and combust the residue over copper oxide to the formation of CO₂ (air in case hydrocarbons were present would be formed) out of the C₂. Secondly to extract all the heavy hydrocarbons and CO₂ and combust residue fuel sparre manner to the formation of CO₂. Thirdly to remove CO₂, CO, and heavy hydrocarbons and combust and determine the proportions of saturated hydrocarbons.

The first operation was carried out by means of an apparatus shown in Fig IV, page 27. It is the gasometer connected by b to two wash bottles containing an strong solution of KOH (a - a'), to remove CO₂. Thence to a thirdottle
e' containing a clear solution of baryta water, as a check on $\mathrm{CO}_2$ and also to absorb it, from here the gas goes through two U tubes $\mathrm{c}$ and $\mathrm{c}'$, containing $\mathrm{KOH}$, then through the tube $\mathrm{b}$ containing a saturated solution of $\mathrm{KOH}$. This tube is about $\frac{3}{4}$ in diameter and is placed at an angle of about $8^\circ$ or $10^\circ$ so the gas must travel up Hill in fine bubbles, hence a more complete extraction of the $\mathrm{CO}_2$. The gas passes from $\mathrm{b}$ down through three wash bottles $\mathrm{d}$, $\mathrm{d'}$, and $\mathrm{d''}$, containing baryta water. They are filled about $\frac{3}{4}$ or $\frac{2}{3}$ full of clear baryta water, now if $\mathrm{CO}_2$ in even very minute quantities pass into baryta water an intermediate white turbidity follows, hence these bottles on check and warrant the absolute complete extraction of the $\mathrm{CO}_2$ if they remain clear. The tube $\mathrm{d''}$ contains dry $\mathrm{CaCO}_3$ to dry it. From $\mathrm{d''}$ the gas passes through another
long tube of same size as B but containing in the lower half 1 dry CaCl₂ and in the upper half 1 solid KOH in order to remove moisture, then through the CaCl₂ tube E into the combustion tube filled with CuO. The gas which enters the tube is dry and free from CO₂. As the gas is combusted the C₂ and C₃H₄ etc. are burned to CO₂ and H₂O which are caught respectively in KOH (H) bulb and H₂SO₄ bulb (G). The gas is drawn through the apparatus by means of the aspirator IV. The object in having the large number of KOH wash bottles is to drive out the CO₂ is all out and by the fact that the baryta bottles remain clear establishes the fact that all the CO₂ is extracted. Duplicate determinations are made and a strong H₂SO₄ spirit acid bulb is nested. Fig IV on page 30, in order to extract the hydrocarbons
and the process is repeated. Next a copper bromide air solution in HCl. Tube is marked E Fig. V on page 32. In order to remove all the CO into and the process repeated. The same sample of gas is used in all six determinations as to get concordant results of the same sample. The following table, Table II, shows the analysis and calculations of the loss obtained. Columns 1-11 simply contain data necessary to finding the correct volume of gas used in the experiments. Col. 11-13 contains data for vol. of HCl, col. 14-16, contains data for vol. of CO in gas after CO + HCl had been extracted. Col. 17, shows page 7 of CO by vol. Col. 18, page 8 CO2 by Orsat method. Col. 19 - the Nag of loss of CO by CO2 Col. 20 the relation amount of heat lost by not having the CO burned to CO2. Col. 21 shows the maximum loss of carbon other gases visible smoke.
*Table II.*

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*Continued on next page.*

The gas was measured under water pressure (col. 1) and then reduced to mercury pressure (col. 2). The corrected volume (col. 10) is under normal temperature and pressure i.e. 0°C at 760 mm pressure.
The percolates are calculated, in case of gases, by volume.

The volume of CO is equal to vol. CO₂ (col. 16).

In calculating the percentage of heat lost by CO₂, the calculation was made from the relative amount of C in CO₂ to C in CO, or C to C. (20)
The law of gases is that all gases present under the same temperature and pressure equal volumes and that all gases present under a constant constant temperature and pressure equal expansion for equal increments of temperature. Now when CO is burned to CO$_2$ it takes up another atom of O forming CO$_2$. The volume of this CO$_2$ formed is equal to the volume of CO originally present in accordance with the above law. As the volume of CO is equal to the volume of CO$_2$ the volume of the gas is calculated from the CO$_2$. This gives an average weight of CO in the gas of 40.2% by volume. The average amount of CO$_2$ in the gas is found to be 6.75%, while the average $S_o$ of loss of carbon (total) by CO$_2$ is 6.98%. The average $S_i$ of heat lost from carbon not being burned to CO$_2$ is 5.43%, and the maximum amount of heat lost by visible smoke is 3%.
As it is found that the total loss of the carbon dioxide used is 9.98% and the total loss of heat going off the chimney is 7.8%. The heat lost by going up the chimney is the heat required to heat the gases to a temperature much higher than that of the surrounding air. If these gases were of the same temperature as the gases in the outside of the chimney, there would be no draught, and a large amount of heat is required to raise these gases to a sufficient temperature so as to cause a draught by the cold outside air pressing down and forcing the heated air up. These two losses of heat aggregate to about 26% of the total heat given off by the coal or fuel.

The average percentage of carbon contained in the coals used as fuel in the furnaces when the gases were collected is between 73.70-80.70. Now if 10% of this carbon is lost by incomplete combustion to the formation of carbon monoxide, the remaining carbon is between 67.70-72.70.
of the coal utilized and this page is further decreased by other losses which makes the total amount of carbon actually utilized for production of heat about 55% of coal carbon as CO.

The manner by which these percentages were obtained is as follows: The inclusion of the actually existing amount of CO₂ originally present in the gases was shown by the first analysis (Arter's method) to the amount of CO₂ formed by the combustion of the CO in the gas was found by dividing the page of CO₂ found by combustion by the page of CO₂ actually present plus the page of CO₂ found by combustion. This gives the page of the carbon lost by the escape of CO₂.

By burning carbon completely, i.e., to CO₂, it is found to yield 8080 heat units or calories, i.e., the amount of heat is given off to raise a mile (1 pound) of water, 10 C with a mile! pm.
of substance. Burning C to CO gives 66.07 heat
miles or by burning the C to only CO there is
lost 8.080 - 66.07 = 24.73 heat miles. Now it is seen
that over 1/3 of the heat is given off to burn carbon
to CO and the other 2/3 is lost. The loss actual
percent being 69.470 % heat miles saved in
burning CO to CO2. The Page of heat by the carburetor
the samples of gases was found by Jupiterly 69.470%
if the Page loss of carbon by CO in what the loss of
total heat of carbon is reduced to an average
of 6.3470 instead of 6.982. If the CO lost
were burned to CO2, the full heating power
minus, that which goes off as visible smoke
and from that that goes off as sensible heat,
would be obtained.

In conclusion will state that the only way to stop this
loss is to secure the perfect combustion of coal which would
eliminate both visible and invisible gases.