in ether indicated perchromic acid. In acid solution sodium peroxide gave no such oxidation. If a chromic salt were oxidized by sodium peroxide as above, the solution acidified with acetic acid and lead acetate added, in dilute solutions such as were used in this work, no precipitate appeared. On allowing the solution to stand, or more quickly, on warming, the customary yellow precipitate of lead chromate appeared and oxygen continued to be evolved for some time.

These results show the reason of the failure to obtain the tests for chromate of lead in the earlier part of the work where dilute solutions of chrome alum were severally treated with sodium peroxide and bromine water. The sodium peroxide forms an alkaline solution with the evolution of oxygen. The chromic salt is oxidized to the perchromate, the sodium salt being formed in the alkaline solution. This substance is stable and gives a yellow color to the solution. On acidification with acetic acid, lead acetate produces no precipitate, as the lead perchromate is soluble in this medium. On standing or warming, oxygen is given off and the lead perchromate breaks down to the chromate, giving the customary yellow precipitate.

Oxone, the fused form of sodium peroxide, acts the same as the unfused variety, as would be expected. For analytical work it is less desirable, owing to the impurities it contains. In addition to the silica found in the unfused variety, it carries about 1.5 per cent. of copper as the result of several analyses showed. In addition, small amounts of iron were found to be present. The results obtained in this investigation show that sodium peroxide is the best oxidizing agent for chromium in solution. Sodium perchromate is formed by this agent in alkaline solution. This solution is stable, and, on acidification, oxygen is rapidly and continuously evolved. The oxygen can be liberated as fast as desired, thus giving a powerful oxidizing source simply controlled.

The properties of this alkaline perchromate solution will be further investigated.

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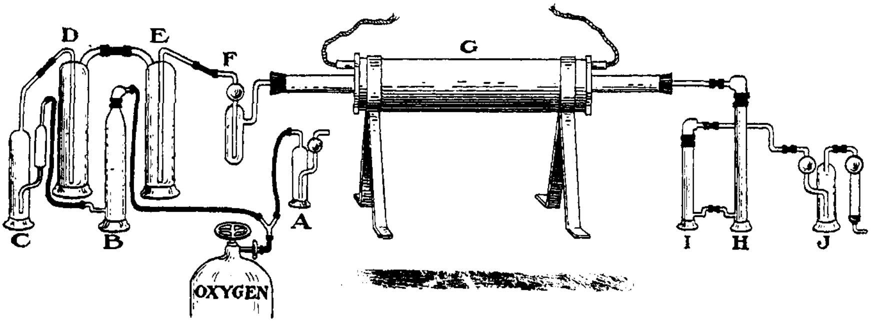
THE DETERMINATION OF CARBON IN STEEL, FERRO-ALLOYS, AND PLUMBAGO BY MEANS OF AN ELECTRIC COMBUSTION FURNACE.

BY C. M. JOHNSON.

Received March 3, 1908.

Several months ago it occurred to the writer that the Hoskins resistance wire could be applied to the heating of combustion tubes. A drawing was prepared for a furnace of a muffle type to heat four tubes lying in the same plane and parallel.

After some correspondence it was agreed, at first, to try a single tube furnace. It consists of a steel tube 295 mm. x 76.3 mm. containing a non-conducting packing of magnesia oxide. In the center is a quartz tube wound with the Hoskins wire.[1] Inside of this tube is placed another of the same material of 19 mm. inside diameter and 600 mm. long, in which the combustions are made.



A. Mercury pressure gauge for detection of leaks and stoppages.
B. Jar for stick potassium hydroxide or for any solid dryer or absorbent.
C. Safety jar for potassium hydroxide solution, preventing solution from backing over into rubber tubing.
D. Calcium chloride jar.
E. Soda lime jar.
F. Mercury valve, to prevent reverse action.
G. Electric combustion furnace.
H. Jar for granular zinc to remove
    Acid fumes,
    Litharge fumes,
    Sulphur fumes,
    Chlorine fumes.
I. Jar for phosphoric anhydride to remove water.
J. Absorbent and weighing apparatus for carbon dioxide.

This furnace was to be durable, if not heated above 1100°, but it burned out in three days. It was then rewired with greater resistance and was guaranteed, if not heated above 1000°. Fearing the furnace might again desert the cause, the writer put in a small 32-ohm rheostat that happened to be at hand. With about one-fourth of this resistance the furnace, on a 220-volt direct current, has been maintaining a constant temperature. To secure complete combustion of steel, it is very essential that the heat be maintained as close to 950° as possible, *i. e.*, as little under that temperature as practicable. If the temperature drops to about 900°, or under, the results obtained are liable to be from 0.01 to 0.10 per cent. too low, unless red lead is mixed with the drillings. Hence, if one

[3] The apparatus can be supplied by the Scientific Materials Co., of Pittsburg.

desires to operate with oxygen alone, the necessity of keeping the temperature from 940° to 960° centigrade cannot be made too emphatic.

The oxygen is purified by passage through jars of stick caustic potash, potassium hydroxide solution, calcium chloride, and soda lime in the order named. The oxygen then passes through a mercury valve into the porcelain or quartz (fused silica) tube, half of which is filled loosely with ignited asbestos. The products of the combustion are purified from acid, sulphur, litharge, or chlorine fumes by passing through a jar of granulated 30-mesh zinc. The water is removed by a jar of phosphoric anhydride.

For steels containing 0.30 to 1.50 per cent. carbon two grams of fine drillings, not over one-fourth mm. thick, are taken. For still lower percentages of carbon 3.0 to 5.0 grams of drillings of not over 20-mesh size are selected.

The sample is weighed into a clay boat. (The boat is molded and burned in the laboratory by a boy at a trifling cost.) The steel begins to burn by the time the stopper of the combustion tube is in place. Two grams of steel are decarbonized in three minutes and five grams in six minutes. The burning is continued for ten minutes more with oxygen passing through the combustion tube at a rapid rate. The weighing apparatus is detached, wiped and weighed. Twenty-five minutes afford ample time for a single combustion, counting all operations.

The weighing apparatus and the jars for the purifying train are the writer's design and were first published, in part, with illustrations in the January *Journal of the Engineers' Society of Western Pennsylvania*, 1906, and more fully in THIS JOURNAL, **28**, 862 (1906). This weighing apparatus (J) is used forty times before it is refilled. As it is always weighed against a duplicate for a tare, after the fortieth combustion its tare is used as an absorber for forty more combustions, so that when a pair has been freshly filled the operator knows he can complete eighty combustions before he needs to refill his weighing outfit.

While no red lead is necessary for steel combustions, some of the alloys such as ferro-chrome, carbonless chrome, and ferro-boron, require that red lead be mixed with the drillings or powder to break the metallic bond and permit of decarbonization. Ferro chrome is the most refractory as from a carbon content of more than 4 per cent. only 0.2 per cent. was obtained by burning as in steels with oxygen alone, at a temperature of 940°. Pig-iron also requires some red lead. In general, about one-half the amount of lead required for decarbonization in a gas furnace is sufficient for the same work in the electric furnace, by reason of the higher heat attainable within the range of durability. A few of the many comparisons made in this laboratory between the combustions in a gas furnace with red lead and oxygen and combustion in oxygen alone are given in Table I:

TABLE I.

Sample.	Method.	Weight of drillings taken.	Amount of red lead used.	Per cent. carbon found.
No. 1 Steel	Electric	4 grams	none	0.09
" 1 "	Red lead	4 "	7 grams	0.09
" 288 "	Electric	2 "	none	1.176
" 288 "	Red lead	2 "	4 grams	1.175
No. 2 Steel	Electric	5 grams	none	0.121
" 2 "	Red lead	4 "	7 grams	0.111
" 3 "	Electric	1½ "	none	0.976
" 3 "	Red lead	1½ "	4 grams	0.967
" 4 "	Electric	3 "	none	0.109
" 4 "	Red lead	5 "	7 grams	0.118
" 5 "	Electric	2 "	none	0.469
" 5 "	Red lead	2 "	4 grams	0.474
" 6 "	Electric	2 "	none	0.736
" 6 "	Red lead	2 "	4 grams	0.737
" 7 "	Electric	3 "	none	0.118
" 7 "	Red lead	4 "	7 grams	0.117
" 8 "	Electric	2 "	none	1.17
" 8 "	Red lead	2 "	4 grams	1.168
" 9 "	Electric	2 "	none	1.15
" 9 "	Red lead	2 "	4 grams	1.16
" 10 "	Electric	5 "	none	0.046
" 10 "	Red lead	4 "	7 grams	0.040

TABLE II.—FERRO-ALLOYS AND PLUMBAGO.

Sample.	Method.	Weight of drillings taken.	Amount of red lead used.	Per cent. carbon found.
Tungsten powder	Electric	2 grams	none	0.003
	Red lead	2 "	4 grams	0.010
Plumbago, No. 153	Electric	0.3 "	none	50.700
	Red lead	0.2 "	4 grams	50.800
Plumbago, No. 356	Electric	0.3 "	none	51.650
	Red lead	0.2 "	4 grams	51.300
Plumbago, No. 1	Electric	0.2 "	none	94.900
	Red lead	0.3 "	4 grams	94.300
68.5 per cent. Ferro-chrome	Electric	1.0 gram	1 gram	4.21
	Red lead	1.0 "	4 grams	4.15
Ferro-vanadium, No. 134.	Electric	1.0 "	none	3.12
	Red lead	1.0 "	4 grams	3.09
Ferro-titanium, No. 1	Electric	2.0 grams	none	0.22
	Red lead	2.0 "	4 grams	0.24
Ferro-boron, No. 1	Electric	1.0 gram	1 gram	1.73
	Red lead	1.0 "	4 grams	1.72
Carbonless chrome No. 9—	Electric	1.0 "	1 gram	0.08
96.0 per cent. chromium—	Red lead	1.0 "	4 grams	0.09
Pig iron	Electric	1.0 "	none	3.20
"B"	Electric	1.0 "	0.5 gram	3.58
	Red lead	1.0 "	4 grams	3.58

The advantages of the electric heating apparatus are obvious. Very little heat is radiated; economy of space is attained; tubes are heated gradually and cooled gradually; time required is the minimum; labor cost is plainly the lowest because of simplicity and rapidity and no expensive platinum tubes or boats or crucibles are used.

Some may say, "Why not burn the steel in air?" The answer is that the cost of oxygen is small, 1/3 cent per combustion, and the steel burns twice as fast. Oxygen can now be had at 5 c. per cu. ft. in 100 cu. ft. cylinders. The method is accurate for all steels. As pointed out in the writer's article in THIS JOURNAL[1] and in his preliminary paper read before the Pittsburg Section in Dec., 1905, one may lose as much as 50 per cent. of the carbon in certain alloy steels by attempting to dissolve the borings in either neutral or acid double chloride of copper and potassium.

The best protection for the bottoms of clay or porcelain boats is a liberal layer of ignited silica sand, such as is used for acid open-hearth furnace bottoms. The silica rock is crushed to about 20-mesh and ignited in a muffle furnace at a bright red heat, cooled, and kept in glass-stoppered bottles.

To secure complete decarbonization it is necessary either that thin drillings be used or, if the sample contains much coarse or bulky material, it should be selected. This can easily be accomplished by pouring the borings on a 20-mesh sieve and shaking all of the steel of 20-mesh size and the still more finely divided dust on to a 60-mesh sieve, which retains only the 20 to 60-mesh material. This *always* represents a good average sample.

Further, the drillings should be placed in as compact a mass as possible. If curly drillings are scattered along the entire length of the boat instead of being put in a deep, compact body, borings that are a little thick will frequently be found to still contain unburned metal. This detail is a very important one. Of course, the reason is that drillings lying in close contact heat each other to incandescence during the burning with oxygen.

Also, during the period when the oxygen is being absorbed in large quantity by the burning metal, the flow of the gas should be regulated so that there is a bare excess and no more. That is, the oxygen should be turned on in sufficient quantity that the gas is bubbling through the weighing apparatus slowly. If the gas is rushed through during this period the steel becomes violently heated and slags with the sand and the sides of the boat, destroying the latter. Worse yet, low results are obtained frequently in this way, probably due to the formation of carbon

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[1] *Loc. cit.*

monoxide, which is driven out of the hot portion of the tube before it is oxidized to the dioxide.

If the oxygen is turned into the tubes in sufficient quantity to maintain a slow stream during the period of the burning, the end point of the combustion is distinctly shown by a sudden increase of the speed of the bubbling through (J). The rush of oxygen is then checked but the rate of flow is still rather rapid for the final ten minutes.

The weighing apparatus (J) is filled not quite to the bend of the inlet tube with a solution of potassium hydroxide made by dissolving 500 grams of the latter in 500 cc. of water. The drying tube at the outlet of J is closely filled with pieces of stick caustic potash cracked to about half the size of a grain of wheat. To prevent the caustic potash from coming in contact with the small rubber stopper in the drying tube a *loose* plug of asbestos is placed at that point. The little bulb of this drying tube is filled about half full of glass wool. If dry sticks of caustic potash are cracked quickly, the small pieces can be conveyed to the drying tube in dry condition and constitute not only a splendid guard against loss of moisture from J but are also equally effective as an absorbent of carbon dioxide.

If a porcelain boat is used, the 15 x 75 mm. Royal Meissen boat is the best shape and most endurable of any porcelain boats that the writer has tried. When putting in the sand bottom, fill the front half of the boat about two-thirds full and then with the butt end of the forceps make a trough in the sand, working it well up the sides of the boat. Pour the drillings from the weighing bottle into this depression. By so doing the drillings are kept in a compact mass and when the combustion is completed the burned steel can be lifted out in a small cake. In this way a boat can be used ten to fifteen times.

When a great many combustions are made daily, the fused silica, or electro quartz, tube is the most durable. The continuous spraying of oxides against the walls of a porcelain tube weaken it and when the current is turned off and the tube is permitted to get cold the contraction causes a rupture.

To prevent the contents of D, B, E from clogging the inlets and outlets, large plugs of cotton are used at these points. Glass wool plugs should be used in H and loose plugs of ignited asbestos in I. Enough mercury is placed in the bottom of F and A to form a seal. The inlet end of the quartz tube heats somewhat and it is better to wrap it several times around with a strip of cheese-cloth, the end of which dips into a 150 cc. beaker of water suspended directly underneath by means of copper wire. During the absorption of carbon dioxide the outlet of J is protected from ingress of moisture or carbon dioxide or fumes from the room by a drying tube not shown in the figure. It is filled with pieces of stick caustic potash broken to the size of a pea.

LABORATORY OF THE PARK STEEL WORKS,
CRUCIBLE STEEL CO. OF AMERICA,
PITTSBURG, PA.

(CONTRIBUTION FROM THE OFFICE OF PUBLIC ROADS, U. S. DEPT. AGRICULTURE.)

THE EXTRACTION OF POTASH FROM FELDSPATHIC ROCK.[1]

BY ALLERTON S. CUSHMAN AND PREVOST HUBBARD.

Received March 4, 1908.

The extraction of potash from native rocks has long been considered one of the most important as well as one of the most difficult problems of industrial chemistry. In spite of the enormous resources of the North American continent, there has not yet been found anywhere on it an available source of potash, thus necessitating the importation from abroad of many hundreds of millions of pounds per annum of the salts and compounds of this important substance. One result of this lack of a native source of supply has been to stimulate the use in agriculture of hard wood ashes, which are even at the present time brought from Canada in considerable quantities to the added devastation of the fast disappearing forests. In addition to this, cotton hull ashes from the South are shipped to the North, which merely robs the soil of one portion of the country to supply the deficit in another. The pegmatitic granites and feldspathic dykes of the eastern and central western United States offer an unlimited source of supply which only awaits an economical method for making it available. Many of these feldspar deposits run as high as 10 per cent. in potash (K_2O) and it follows, therefore, that a quarry only fifty feet square and fifty in depth contains about 2,000,000 pounds of this alkali. Under the stimulus of the rapidly growing cement industry, great advance has been made in the last few years in the art and economics of fine grinding, which must of necessity be the first step in any process which attempts the extraction of potash from feldspar or other minerals. At the present time, in the manufacture of Portland cements, at least two extremely fine grindings as well as a burning at a high temperature are accomplished so economically that the finished product can be packed in bags or barrels and sold in some places for a price equal to about three-tenths of a cent per pound or about six dollars per ton. In view of the fact that a short ton of 10 per cent. feldspar contains about ten dollars' worth of potash at prevailing prices the problem of extraction is not on first thought an unpromising one from an economical standpoint.

There are about twenty well defined rock-forming minerals as com-

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[1] Paper read before the N. Y. Section, Am. Chem. Soc., Feb. 7, 1907. Published with the permission of the Secretary of Agriculture.