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THE MANUFACTURE OF CALCIUM CARBIDE.[1]

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SO universal is the interest in acetylene gas and so different the estimates and opinions as to the cost of calcium carbide as a source of the cheap production of acetylene gas, that we have thought it desirable to place on record the data from our actual experience in the production of calcium carbide in quantities. The works of the Willson Aluminum Company have been running night and day since May 1st, 1895, producing calcium carbide. These works are daily duplicating the results here given and can expand indefinitely. Each individual step, except water power, as taken at Spray, N. C., is capable of being changed in the direction of reducing the cost of the output, as these efforts have been attended with the clumsiness, lack of adaptability and excessive cost that is incident to all efforts along an untrodden path. Still we can produce calcium carbide at less than $25.00 per ton, including wear and tear and interest on capital.

Beyond looking after the dynamos, no special training is necessary, as neither metallurgical nor chemical skill is required in the operations. We grind and mix coke and lime, start the water wheel, see that the arc is formed, shovel in the mixture of lime and coke and the volt- and ammeter show when to lower or

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[1] Read Sept. 3d, before the Springfield meeting of the A. A. A. S. by one of us (M). We have made since then several additions, so as to make the article complete up to the present time.

raise the carbon pencils, which is done by means of a screw located in the dynamo room, away from the furnace. We can measure with an ordinary yard stick on this screw the height of the piece of carbide in the furnace. We stop when we have raised the carbon pencils thirty-three inches, switch the current off to another furnace and repeat the operation. The carbide in the former furnace, as soon as cooled and brought in contact with water, is all ready to do perfect work in generating acetylene gas ; it will proceed with this work without help and will make room therefor in spite even of bands of steel.

Water power costs us $6.00 per horse power. Water in the raceways ready for the water wheels is now offered in enormous quantities to the Willson Aluminum Company at the rate of $5.00 per horse power per year. These powers are located at different places, where coke and lime can be had cheaply, and also cheap transportation for the carbide to the market.

The technical description of our process which follows herewith was written by G. de Chalmot, who has had for some time personal supervision of the operations of the Willson Aluminum Company.

In the year 1888 Mr. T. L. Willson started a series of experiments with a view of reducing refractory ores in the electric furnace, and among other valuable things he made calcium carbide.[1]

We will first give a short description of the furnace and a general outline of the process, then enlarge somewhat on the details. The furnace used in Spray, N. C., is built of ordinary brick (a sectional front view is given in figure 1). The front side is formed by four iron doors, the one above the other. The upper two remain closed usually. The chimney is attached near the top of the furnace, and commences with a flue m in the corner. The furnace measures at the bottom inside two and one-half by three feet. The electric current enters at the bottom

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[1] We will note here that Moissan, who discovered this process for making carbide, independently of Mr. Willson, communicated incidentally at the meeting of the French Academy of December 12th, 1892, (*Compt. Rend.*, 115, 1033) that a carbide of calcium is formed if calcium oxide is heated in an electric furnace with carbon electrodes. He investigated the compound much later (*Compt. Rend.*, 118, 50). Mr. Willson, who sent during the summer of 1892 samples of carbide for examination to Lord Kelvin, of the Glasgow University, clearly antedates Moissan. See Journal of Franklin Institute of 1895, page 333.—Note.

and top. The bottom electrode is an iron plate a covered with eight inches of carbon b. For this covering we use pieces of carbon pencils or a mixture of coke and coal tar. Sixteen copper cables of 0.75 inch in diameter c convey the electricity from the dynamos to the bottom electrode.

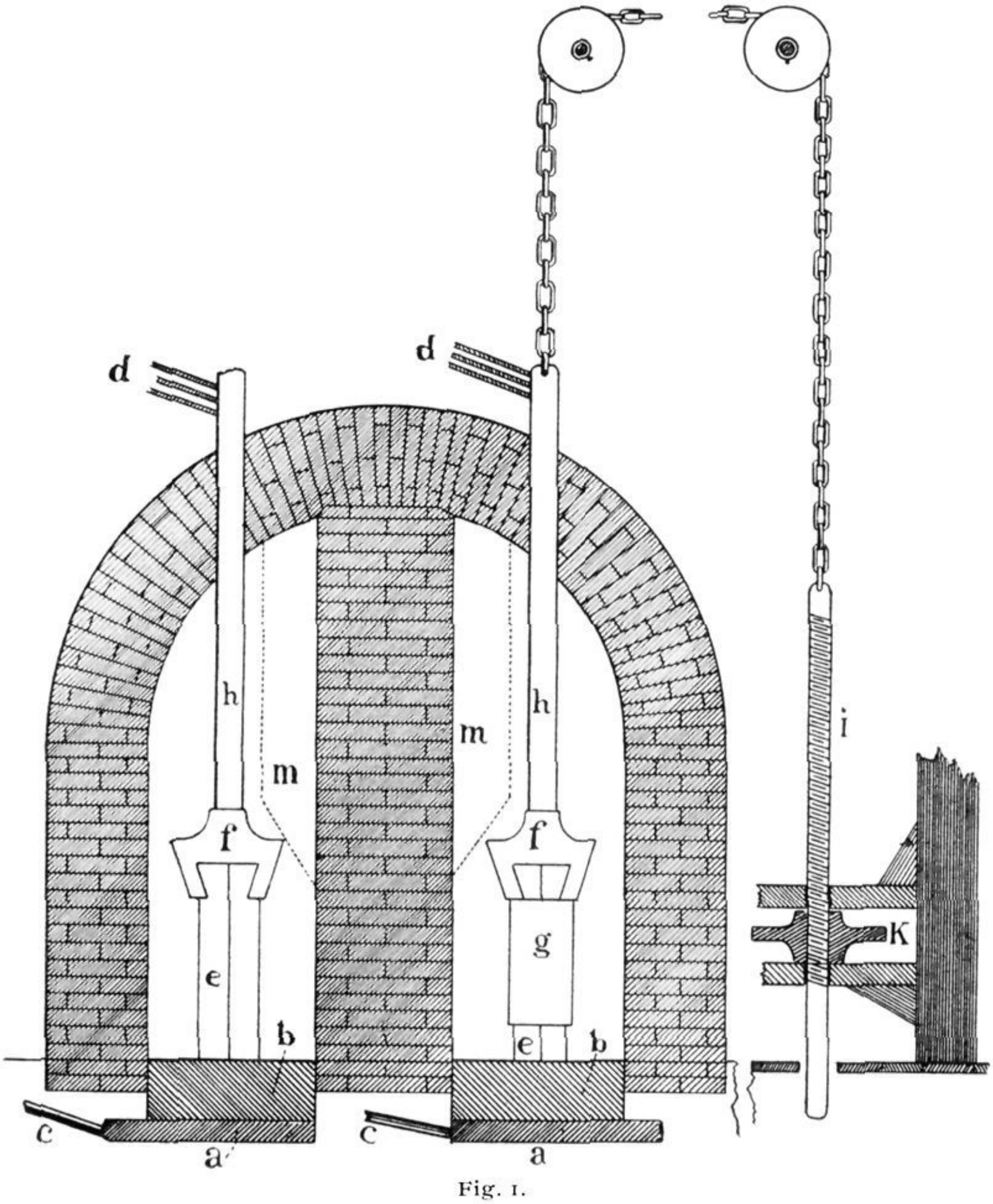

Fig. 1.

Sixteen other cables are connected with the top electrode d. The top electrode is composed of six carbon pencils e, each four inches square and thirty-six inches long. Six pencils are arranged in three pairs behind each other and are cut out at the top so as to fit in the carbon holder f. They are enveloped together by a sheet of iron g, which is shown in the right handed furnace of figure 1. They really form one pencil. The carbon holder is screwed to a copper bar h, which is three inches square and to which the copper cables are connected. This bar is fastened by a chain that runs over two pulleys to a long upright screw i. On this screw is a nut which forms the center of a wheel k. By turning the wheel the screw can be raised or lowered. The man who attends to the wheel has the volt- and ammeter before him. The electric current is generated in two dynamos to which transformers are connected, and which can give a current of from fifty to 100 volts. The power is furnished by a water wheel of 300 horse power under twenty-eight feet fall.

Two of the furnaces have been working for twelve months and they have given satisfaction, except for working not sufficiently economically. In the furnaces built for the Niagara Falls Carbide plant, many changes which we suggested, have been adopted, looking to economy of production. We give here a short description of these furnaces (figures 2 and 3).

In Spray it is necessary to allow the furnace to cool before emptying it. In order to use one and the same furnace continuously, the bottom of the furnace is replaced by an iron car a which runs on a track and in which carbide is formed. When the car is filled the pencils b have been lifted entirely out of it. The current is then shut off, door c is opened, the full car is run out and replaced by an empty car. The pencils are lowered again to the bottom of the car and a new run is commenced.

The bottom of the car is covered with from four to eight inches of carbon. When the contents of the car have sufficiently cooled outside the furnace, which will take from six to twelve hours, the body of the car is lifted from the track by the trunnions d and turned over. The contents are dropped on a grate formed of iron bars, on which the piece of carbide remains, while the unreduced material falls through into a lower room where it is collected to be used again for the formation of carbide. The mixture of lime and coke is fed into the car through the flues e, which extend along the whole length of the car. The rods f which bear four blades, extend through the whole breadth of

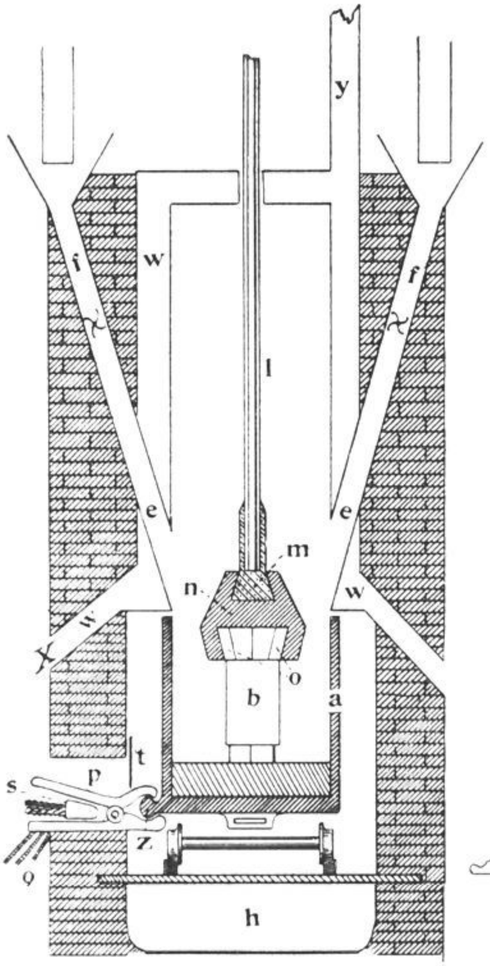

Fig. 2.

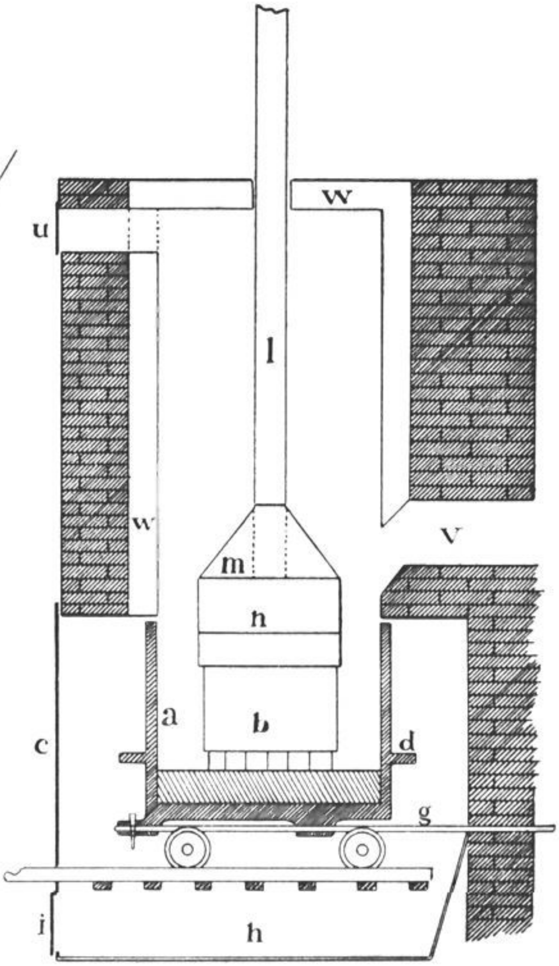

Fig. 3.

the feeding flues. These rods are turned automatically, and the faster they turn, the more material is fed into the car. In order to stoke the furnace automatically, the car is attached to an iron bar g by two hangers and a coupling in front of the car. Bar g extends through the back wall of the furnace, and is automatically moved forward and backward for about two inches and about twenty times per minute. The car is thus also rolled backward and forward on the track for about two inches each time. Every time that the car stops or starts it gets a little jerk which is sufficient to fill up the holes made by the escaping gases in the loose material. This motion of the car further prevents the arc being located for a longer time at one point, for which the arc has always a great tendency. This will materially increase the efficient use of the heat of the arc. Under the track of the car is the bin h in which the unreduced material is collected that will fall from the car when this is taken out. This material can from time to time be taken out through the door i. The carbon holder is more complicated than in the Spray furnace. Twelve carbons are used and the holder is therefore about twice as heavy. It is not advisable to suspend this carbon holder from a copper bar, which moreover becomes rather hot in this closed furnace. The carbon holder is therefore attached to a rod l, which is composed of three slabs. The inner one is of copper and measures six by one and one-half inches, and the outer ones are of iron and are six inches by one. Since it is not practical to attach the twelve carbons in their iron casing to the carbon holder in the furnace, the holder itself is composed of two pieces, m and n, which slide into each other. The aggregate of pencils is connected to piece n outside the furnace and the whole is placed in the car a. Rod l is so far lowered that piece m will easily slide into piece n, and the connection can easily be effected. Iron plates o are placed between the carbon holder proper and the pencils. These plates o are about one inch thick. They are fastened to the inside of the carbon holder by pins which are inserted in the holder, and fit in holes of the plates. These plates can be easily removed and replaced. It will sometimes happen that a small arc is started between the pencils and the inside of the carbon holder and a part of the carbon holder will melt. In the case that the plates o are used, one can simply replace these plates. The car a forms one of the electrodes and is connected with the bottom cables q by two clamps p. The lower clamp is stationary and the upper one can be opened. The clamps are tightened around the appendage z of the car by a wedge and screw s. When the clamps are fastened the slide t is lowered so as to shut the opening. The electric connection with the car can also be and is better made through the bar g, which in that case is composed of an iron and a copper slab. It may also be made by two copper bars which run alongside of the car and are pressed against it with springs. The furnace is entirely closed.

When it is started the door c is shut, but the door u is kept open till the carbon monoxide, which is formed in the reaction, has replaced the air in the furnace. This point is reached when the flame comes out of this door. Door u is then also closed and the gases escape through the chimney v. The use of door u prevents explosions of the carbon monoxide in the closed furnace. Chimney v begins just over the car. The carbon holder and the rod l are therefore not in the current of the hot gases. The upper part of the furnace is cooled moreover by an air jacket w through which a draught of air is maintained. The cold air enters through openings x and the warm air is led off by chimney y. The warm air may be utilized for heating the building. The chimney gases pass through flues or rooms, in which the lime dust is collected by proper means. Owing to valuable suggestions of our superintendent, Mr. J. C. King, this furnace is called the King Furnace. Besides these two types of furnaces, several others have been proposed.

In order to start our present furnace, we shut the lower iron door and lower the pencils to the bottom of the furnace. The current is turned on and the mixture of coke and lime fed in, the arc being kept covered with the mixture as high as one foot around the pencils. It is then easier to keep the arc steady. It is necessary to stoke from time to time, for the gases which are formed in the arc constantly make channels through the material, and especially if unslacked lime is used. These channels will not fall in and less material will come into the arc. The feeding in of the material is continued for several hours. If the attendant at the hand wheel sees that the voltage becomes low, he raises the pencils. If the arc should be broken the amperage becomes zero and the voltage high, and in that case the pencils are quickly lowered. After shutting off the current it is well to allow the furnace to cool two or three hours before emptying it.

The carbide is always found in one piece between the pencils and the bottom. It has a conical form, being broader at the base and can be two and one-half feet high in our furnace. It however never has so great a diameter as to fill up the whole capacity of the furnace. The carbide is therefore entirely surrounded by a cover of the mixture of lime and coke. This mixture is so bad a conductor of heat that the brick walls of the furnace are not attacked. It is very easy to separate the carbide from the loose mixture, for the latter never melts together, while the carbide is hard and solid. The pieces of carbide are covered with a thin coating which is a little thicker at the top of the piece, and the same may be reground and again used. This coating contains mainly carbon, but also carbide and calcium oxide. It seldom yields more than a half cubic foot of gas per pound, but in some cases it yields 1.77 and even 2.10 cubic feet. This coating, however, is of little importance. If the mixture is well made this coating seldom exceeds from twenty to thirty pounds on a piece of carbide of from 300 to 400 pounds. The carbide itself is crystalline. The crystals are especially well developed near the top and are more perfect with an excess of coke, low voltage and when allowed to cool slowly. The center of the piece of carbide stays liquid for some time after the electric current has been shut off. The liquid part, however, is of the same quality as the rest of the piece. We have in fact tapped out of the furnace, carbide which was very pure and yielded 5.59 cubic feet of gas per pound. We do not wish to express an opinion as to the practicability of tapping the carbide as soon as it is formed. We will only mention that Mr. Price, in Newark, has, with a view of tapping the carbide, constructed and patented a new furnace, and that one of us (C.) has also devised a furnace for the same purpose.

Carbide of average quality (about five cubic feet of gas per pound) often has a reddish color, especially if it has been made with a current of high voltage. Carbide of bad quality is often grayish or blackish, or will show streaks of graphite. Pure carbide yields more than 5.90 cubic feet of gas per pound. It has, however, been found to be more economical to produce carbide that yields only about five cubic feet of gas per pound. Samples of carbide of different qualities contained :

TABLE I.

Cubic feet gas per pound.	Carbide. Per cent.	Free calcium oxide. Per cent.	Carbon. Per cent.	Other impurities. Per cent.
5.7	96.6	0.6	....	2.8
5.5	93.2	4.2	....	2.6
5.1	86.4	9.5	....	4.1
5.025	84.7	10.7	1.6	3.0
3.6	61.0	27.5	3.2	8.3
3.45	58.5	1.1	25.6	14.8

The upper part of a piece of carbide is often purer than the under part.

The coke to be used should not contain much ash. Our coke contains about seven per cent. of ash. The carbide obtained with a coke of from ten to eleven per cent. of ash was perceptibly inferior to that obtained with our usual coke. It was found impracticable to make a good quality of carbide with a coke of twenty-seven per cent. ash. It is well that there should not be more than ten per cent. of ash in the coke. The coke should be ground very fine, and it should pass through a fifty mesh sieve. The lime need not be as fine as the coke. The largest pieces should pass through a ten mesh sieve. If the lime is coarser the quality of the carbide becomes inferior. That the state of the pulverization of the lime is important, can be seen by a comparison of the average amount of gas per pound (4.97 cubic feet), obtained with unslacked lime (Table II), and that obtained with air slacked lime (5.27 cubic feet Table III). The unslacked lime was in several instances not quite as fine as the slacked lime. Unslacked lime is decidedly preferable to air slacked lime, as we will see afterwards.

The lime which we use contains one and one-half per cent. magnesia and one per cent. of other impurities. The anhydrous lime should contain ninety-five per cent. calcium oxide, and no more than five per cent. impurities. The presence of magnesia is especially detrimental to the production of carbide. We could not obtain a good quality of carbide with a lime in the following analysis: Insoluble. 0.24 per cent.; silica, 0.78 per cent.; ferric oxide and alumina, 0.68 per cent.; calcium oxide, 92.83 per cent.; magnesium oxide, 5.47 per cent. Further experiments showed that two and one-half per cent. of magnesia in the mixture has a marked influence on the production. The lime used for making carbide should not contain over three per cent. of magnesia. That magnesia has such a bad influence upon the formation of carbide is probably due to its forming a veil between the carbon and the lime particles preventing their combination. Magnesia does not unite either with lime or with carbon. The latter fact was first shown by Moissan,[1] and our own experiments in this line fully confirm his results. The lime and the coke must be mixed very well or the carbide will be of inferior quality and there will be much coating. Besides the carbide some mixture remains in the furnace. More carbon than lime burns out or volatilizes in an open furnace. It is therefore necessary to add carbon to this mixture before using it again. The amount to be added is calculated from the result of an analysis of the mixture. If coke is added in the proper proportions, the unsmelted portion of the material can be returned at least three times into the furnace, and still yield good carbide. The impurities of the lime and the coke ashes remain as well in the carbide as in the residual mixture. It is therefore a good practice to add charcoal instead of coke to the mixture, so as not perceptibly to increase the amount of ash. The mixture that comes from the furnace is red hot and it will stay hot for days. It will lose a large amount of carbon if allowed to lay in heaps in the air. It is better to mix in the necessary amount of carbon and use the mixture at once again. One can also keep the mixture in air-tight sheet-iron tanks. If the lime has been unslacked the mixture cools much quicker and does not lose as much carbon after it has been taken from the furnace. In the case of slacked lime, water gas is probably formed in large amounts. The carbon pencils must be well cared for in order that they last for a long time. If sufficient coke is put in the mixture they are not attacked much at the end. They will shorten from 0.05 to 0.10 inch for every hour running. They become thinner for being exposed to the air when hot. They are mainly attacked after the electric current has been shut off, for if the furnace is working the gases from the arc come up around the carbons and shut the air off. In order to save the

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[1] *Compt. Rend.*, 118, 506.

carbons best it is therefore well to keep the furnaces running with as little interruption as possible. In the closed furnace, which we have described, the carbons will be surrounded by non-oxidizing gases, which will save them materially. In the open furnaces in Spray we surround the carbons with a sheet iron cover that reaches from the carbon holder to within four inches of the bottom end of the carbons. This jacket is fastened with iron wires to the carbon holder. The space between the carbons and the jacket is packed with a mixture of coke and coal tar or pitch. This mixture is baked by surrounding the carbons and jacket with the red hot material that comes from the furnace or by placing them in a fire. The jacket will generally last as long as the carbons. One set of the carbons in an open furnace and with interrupted operations will last on an average about 100 hours. These figures hold good where a current of from 1700 to 2000 amperes is used. The voltage has no perceptible influence on the result. Working with say 1700 amperes and 100 volts and generating about 225 horse power, the production of carbide per hour can be reckoned to be easily eighty-five pounds, and one set of carbons can therefore make at least 8500 pounds of carbide, even in an open furnace. If the furnace is used continuously the carbons will last at least from 200 to 300 hours, and the cost of pencils for one ton of carbide will be about $1.00.

The analytical part of our work has been very simple. After the piece of carbide has been broken open with a hammer, two or more samples, representing as nearly as possible the average quality of the carbide and of about eight ounces each, are taken. These samples are broken in pieces of about one-half inch in diameter and from two to three ounces are taken for one gas test. The material is put into a dry bottle of about one quart capacity, which is provided with a rubber stopper, through which two glass tubes pass. The one tube bears a stop cock and drop funnel, the other tube conducts the gas through a series of U tubes and then through a small gas-meter. The funnel is filled with water, and by opening the stop cock, water is allowed to drop slowly on the carbide. The acetylene gas is generated and is cooled in the U tubes before it passes to the gas-meter. Much water vapor is condensed in the U tubes, for the gases generated in the bottle are hot. We make a correction for the temperature of the gas as it passes the gas-meter. We do not take into consideration the small amount of gas which passes through the gas-meter by the expansion of the gas in the bottle when the latter becomes hot, and because a part of the bottle becomes filled with water. The error arising herefrom is of no consequence, for the volume of the bottle is only one quart and the volume of the gas which passes from the gas-meter is from one-half to one cubic foot. The water, moreover becomes saturated with acetylene. Our figures show the amount of moist gas at the temperature of 60° F.

In order to determine the lime in the mixture, two and five-tenths grams are boiled with a slight excess of hydrochloric acid of known strength in a 250 cc. bottle. The bottle is cooled and filled up. The liquid is filtered and in fifty cc. of the filtrate the excess of acid is determined by titration. The coke is determined by boiling two grams of mixture with twenty-five cc. of twelve per cent. hydrochloric acid and filtering off the coke on a Gooch crucible. These methods do not make a claim to absolute accuracy, but they can be quickly executed and give a good estimate of the relation in which the coke and lime are present in the mixture, as the following figures show. The coke used for the original mixture contained 7.33 per cent. of ash. The coke that remained from the mixture that had been boiled with twelve per cent. hydrochloric acid contained six and eight-tenths per cent. of ash, and the coke which remained by the same treatment from a similar mixture that had been once in the furnace contained seven and eight-tenths per cent. ash. The amount of lime found in mixtures by titration and that found by gravimetric analysis varied only by from one-half to three-quarters per cent. when the small amount of magnesia in the lime was known and taken into consideration. In controlling the different runs we have proceeded as follows:

The carbide was weighed and the coating on it determined either by taking it off and weighing it or by estimating it on small and clean pieces. By deducting the weight of the coating from the weight of the piece of carbide we obtain the net yield of carbide. The gas therein is determined, the figure accepted being the average of the result of the analyses of at least two samples. In order to determine the power used, we multiply the voltage by the amperage and divide the product by 746 to obtain the number of horse power generated by the dynamos. In order to make a more proper comparison we found it necessary to deduct the loss of voltage sustained in the carbon pencils. Our pencils were made in different factories and had a different resistance. We therefore determined the difference in voltage as indicated by the usual reading of our meter and the voltage at the end of the carbon pencils. We touch the end of each pencil alternately with an iron rod that is connected with the volt meter by a copper wire. We call net power the power generated by the dynamos less the average loss in the six carbon pencils. Our meters are placed in the primary circuit and we have not taken into account the losses of amperage in the transformers and those sustained by leakage. We have further found that the readings of our meters are about six per cent. higher than those of standard Weston meters. It may therefore be safely relied upon that all our estimates for the production of carbide per horse power are too low. The error is, however, in all cases in the same direction, so that it cannot have materially influenced our deductions, which are based upon a comparison of our results.

In the carbide there is also a considerable loss of voltage and therefore of power. We found, for example, sixty-five volts in the bottom cables and only fifty volts at the top of a two and a half feet high piece of carbide just under the arc. This makes a loss of six volts for each one foot of carbide. The average production during six to eight hours of continuous working is as large as that during two or three hours at the same power. It is, however, not advisable to make the carbide pieces higher than two and a half feet, since then the resistance of the carbide will begin to materially reduce the quantity of the production.

Taking into account the weight of the product, the time in which it has been produced and the number of horse power used, we calculate for each run the amount of pounds produced per horse power in twenty-four hours. By multiplying these figures by the number of cubic feet of gas produced per pound we obtained the number of cubic feet of gas produced per horse power in twenty-four hours. In the following table we give the results of experiments wherein everything has been determined, wherein both unslacked and slacked lime have been used and voltage, amperage and duration of runs were varied. Since these results were obtained we have had many visitors from all parts of the country and for each party we have made a test run. The results of these runs have all confirmed our previous results, with one exception, which was due to the presence of five and a half per cent. magnesia in the lime.

TABLE II. UNSLACKED LIME.

Date.	Time of experiment.	Volts.	Amperes.	Loss of voltage in the pencils.	Horse power.	Production in twenty-four hours including slag.	Net production.	Cubic feet of gas per hour.	Cubic feet of gas per horse power in twenty-four hours.
	Hours.			Per cent.		Pounds.	Pounds.		
June 27th	2.50	100	1700	7.0	214	9.87	9.42	4.83	45.50
July 2nd	3.00	100	1666	8.0	205	10.34	9.76	5.25	51.24
" 1st	2.25	100	1700	10.0	205	10.66	10.10	4.66	47.06
June 24th	3.20	100	1600	7.0	214	11.50	10.73	4.93	52.90
" 28th	2.50	100	1700	10.0	205	11.70	11.10	4.75	52.72
July 18th	3.00	65	2000	5.0	165	9.63	9.15	4.95	45.29
" 19th	3.00	65	1900	5.0	158	10.40	9.62	4.83	46.46
" 5th	3.75	65	2000	5.0	165	8.38	8.15	5.40	44.01
" 9th	4.50	65	2000	5.0	165	9.50	9.05	4.99	45.16
Aug. 10th	6.00	65	1800	8.0	144	9.34	9.00	5.39	48.51
" 13th	6.00	65	1800	8.0	144	10.83	10.44	4.82	50.32
July 31st	7.00	75	1800	8.5	166	11.44	10.53	4:83	50.86
						Average,	9.75	4.97	48.33

TABLE III. AIR-SLACKED LIME.

Date.	Time of experiment. Hours.	Volts.	Amperes.	Loss of voltage in the pencils. Per cent.	Horse power.	Production per horse power in twenty-four hours, including slag. Pounds.	Net production. Pounds.	Cubic feet of gas per pound.	Cubic feet of gas per horse power in twenty-four hours.
June 25th	5.00	100	1700	7.0	214	8.34	7.96	5.30	42.19
" 29th	4.00	100	1700	10.0	205	8.78	8.34	4.98	41.53
" 19th	5.50	100	1700	7.0	214	9.25	...	4.89	....
" 22nd	4.00	100	1600	7.0	199	9.80	9.65	4.74	45.75
Ang. 14th	4.50	75	1700	7.0	159	7.88	7.13	5.50	39.22
July 12th	3.75	85	1800	3.5	198	...	6.33	5.55	35.13
" 26th	8.00	75	1800	5.0	172	8.40	7.23	5.33	38.54
" 12th	5.50	85	1775	8.0	185	...	7.32	5.32	38.94
" 6th	5.00	80	1020	3.0	200	8.78	8.16	5.11	41.70
" 23rd	3.00	65	1800	5.0	150	6.83	6.40	5.78	56.99
" 22nd	2.00	65	1800	5.0	150	7.13	6.40	5.62	35.97
" 22nd	2.00	65	1800	5.0	150	7.20	6.40	5.64	36.09
" 25th	9.00	65	1800	5.0	150	7.72	7.27	5.54	40.28
" 20th	4.00	65	1800	5.0	150	8.60	8.00	5.01	40.08
" 23rd	5.00	65	1800	5.0	150	9.02	8.00	5.07	40.56
" 24th	8.00	65	1800	5.0	150	9.30	8.03	4.97	39.91
					Average,		7.51	5.27	39.52

It is obvious that the results obtained from unslacked lime are far better than those with air-slacked lime. This is undoubtedly due to a loss of power used in decomposing the hydrated lime. The unslacked lime used by us contained, after being ground, from five to nine per cent. of water. In practice it is necessary to use the mixture that comes from the furnace again. This mixture always contains some carbonate of lime, but if it be mixed when still hot with the necessary amount of carbon and put again into the furnace the lime has no opportunity to slack. The unslacked lime has the further advantage that it weighs less and is much less bulky, and that the mixtures made from it cool much faster than those made from slacked lime. The only disadvantages of unslacked lime are, that it must be ground and that mixtures made from it require more stoking if put into the furnace. The mixtures of unslacked lime can stand up against the sides of the furnace under a very steep incline and they can leave a hole all around the pencils. The mixture to be used should, on an average, contain 100 parts of lime and sixty-four to sixty-five parts of carbon in order to obtain a carbide of about five cubic feet of gas per pound. If the voltage is increased to 100 it is better to take a little more carbon (100 lime and sixty-six to sixty-seven carbon). If the voltage is sixty-five or less, sixty-three to sixty-four parts of carbon are sufficient. If the amount of carbon is increased the carbide becomes purer, but there is often more coating.

The largest amount of gas per horse power is obtained if the carbide yields about five cubic feet of gas per pound. The yield of carbide in pounds varies inversely with the quality. In the following table we give the results of a series of experiments made with slacked lime and with a current of sixty-five volts and from 1700 to 2000 amperes. Several of these experiments have not been taken up in Table II, because the amount of slag on all the pieces of carbide has not been determined.

TABLE IV.

Date.	Production per horse power including slag. pounds.	Cubic feet gas per pound.
July 23rd	6.85	5.78
June 14th	7.10	5.80
July 22nd	7.13	5.62
" 22nd	7.20	5.64
‘ 25th	7.72	5.54
Aug. 14th	7.88	5.50
May 21st	8.10	5.20
" 22nd	8.30	5.10
July 26th	8.40	5.33
June 4th	8.46	5.52
July 20th	8.60	5.01
June 5th	8.76	4.94
May 28th	8.80	5.20
" 23rd	8.82	5.10
July 23rd	9.02	5,07
June 8th	9.06	5.10
" 24th	9.30	4.97
July 11th	9.30	4.33
Aug. 12th	9.44	4.51
May 31st	9.87	4.30
Aug. 8th	10.52	4.23

Carbide has been made successfully in Spray by the use of both the direct and the alternating current. We cannot express an opinion as to what current can be used to the best advantage, for we are not able to compare results. All of the results communicated in this paper have been obtained by the use of the alternating current. That electrolysis plays a part in the carbide manufacturing process of Mr. Willson is therefore out of the question, and we do not need to use a furnace of the Moissan construction to prove this. It is not desirable to increase the amperage over 2000 if only six carbons of four inches square are used. The higher the amperage the greater the loss of voltage in the pencils and therewith that of power. The carbons will also last longer if the amperage is low, because they do not become so hot. Lastly we did not obtain as great a yield per horse power if the amperage was high and the voltage correspondingly low. We obtained the best yield of gas per horse power by using a current of 100 volts, which can be seen by comparing the average of the results given in Table II.

TABLE V.

	Volts.	Horse power.	No. of experiment.	Average cubic feet of gas per horse power in twenty-four hours.
Unslacked lime..	100	205–214	5	49.88
	65–75	144–165	7	47.23
Slacked lime.....	100	200–214	3	43.15
	75–85	159–100	5	38.71
	65	150	7	38.55

It must be taken into account that we measured the primary current and that the losses of amperage in the transformers probably have been higher when we did not use the highest voltage, *i. e.*, 100. We do not know in how far it would be advisable to increase the voltage over 100, since our dynamos cannot give us a current of more than 100 volts. We believe, however, that the heat yielded by an arc of 100 volts and from 1700 to 2000 amperes is about the largest amount to be profitably used for the production of carbide in one furnace with six pencils, as it is used in Spray. We base our assumption on the following facts : The quality of the carbide becomes better if the voltage decreases. We experienced some trouble in obtaining large carbide crystals with an arc of 100 volts and 1700 amperes, and in order to obtain a carbide that yields more than five cubic feet of gas per pound the mixture should contain an excess of carbon. If a current of 100 volts and 1700 amperes is used the furnace requires more attendance and stoking than if a lower power and especially a lower voltage is used. The higher the voltage the faster the pencils must be raised, for if the voltage is low (fifty or sixty-five) the carbide spreads out much more than if the voltage is high (100 volts). In the latter case the carbide builds up as a long thin piece and it is oftener necessary to empty the furnace. As to the time that one furnace should be used continuously, we wish to say that we did not perceive a difference in the quantity and quality of the product whether we ran three hours or from three to nine hours. We must, however, remark that in the case where we used 100 volts and 1700 amperes with mixtures of unslacked lime we could not continue running for much more than three hours because the construction of the furnace did not admit of raising the pencils quite three feet. With slacked lime we made also with this high power very satisfactory runs of five and five and a half hours. During the first hour the production is somewhat lower. It seems that more heat is lost probably for heating up the furnace.

The mixture used in all of the following experiments contained lime, 50.08 per cent., and coke,[1] 39.22 per cent. The current was of sixty-five volts and 1800 amperes, the loss of voltage in the pencils five per cent. and the net horse power 150.

TABLE VI.

Time of experiment. hours.	Production per hour in pounds.	Cubic feet of gas per pound.	Cubic feet of gas per hour.
1	37	5.63	208.31
2	40	5.62	224.80
2	40	5.64	225.60
3	40	5.78	237.20

We have now still to consider a very important question, namely, how much coke and lime are necessary to produce one pound of carbide. The formation of carbide taking place according to the following formula :

$$\underset{56}{CaO}+\underset{36}{3C}=\underset{28}{CO}+\underset{64}{CaC_2}$$

0.563 pound of carbon and 0.875 pound of calcium oxide are

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[1] Of 92.17 per cent. of carbon.

theoretically necessary to yield one pound of carbide. The carbide of five cubic feet of gas per pound contains, however, free calcium oxide. We therefore might expect that more calcium oxide and less carbon are used. In practice, however, some calcium oxide and carbon are volatilized or burned. For a succession of experiments we have weighed the mixture that was put into the furnace and that which was taken out and analyzed both. We have experienced considerable trouble in weighing the red hot material accurately and in obtaining fair samples. We have therefore not been able to observe as to how far the consumption of calcium oxide and of carbon is influenced by the circumstances that alter the quantity and quality of the product. We found, however, that less mixture is used if properly stoked and if the arc is kept covered. We found also that the losses of carbon are always more considerable than those of calcium oxide.

TABLE VII.

Number of experiment.	Amount of CaO put into the furnace.	Amount of C put into the furnace.	CaO obtained from the furnace.	C obtained from the furnace.	Amount of CaO used.	Amount of C used.	Production of clean carbide.	Amount of CaO per pound of carbide.	Amount of C used per pound of carbide.
	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lbs.	Lb.
1	675	448	417	270	258	178	195	1.32	0.91
2	739	463	415	242	324	221	285	1.13	0.78
3	859	557	601	375	258	182	190	1.36	0.96
4	612	402	415	262	197	140	190	1.04	0.74
5	851	491	498	282	353	209	280	1.26	0.75
6	906	577	478	280	428	297	375	1.14	0.79
7	1067	699	420	238	647	461	555	1.17	0.83
8	552	375	253	167	299	208	223	1.34	0.95
9	374	211	207	115	167	96	132	1.27	0.73
10	675	461	244	134	431	327	345	1.25	0.95
							Average	1.228	0.837

TABLE VIII.

Number of experiment.	Mixture into the furnace.		Mixture out of the furnace.	
	CaO. Per cent.	C. Per cent.	CaO. Per cent.	C. Per cent.
1	54.70	36.32	57.01	36.89
2	54.51	34.18	57.29	33.36
3	56.23	36.46	57.00	35.62
4	55.66	36.51	55.47	35.02
5	59.70	34.43	58.16	32.94
6	56.65	36.09	55.60	32.61
7	55.44	36.32	54.93	31.09
8	50.64	34.43	51.81	34.29
9	52.08	29.39	52.09	28.90
10	49.34	33.85	41.45	28.27

The average figures of table VII are rather high, for where much coke and lime have been used this is certainly partly due to losses of material by weighing into and out of the furnace and also by insufficient stoking. In the King furnace, the under part of which shuts hermetically tight and excludes draught, and which is stoked mechanically, the amount of coke and lime necessary for making one pound of carbide will certainly be much reduced. In the figures given in table VII we have left the outside coating out of the calculation. In a plant where the acetylene gas is generated at once from the carbide it would pay to use this coating also for making gas. From table VII we see that a very large percentage of the mixture is not acted on by the arc. We have, however, reduced this amount to one-third of the mixture and could reduce it still more without either injuring the furnace, the quantity and quality of the carbide, and without increasing the amount of carbon and lime necessary for making one pound of carbide. In the furnace used in Spray the inside is square instead of octagonal and the dimensions are rather too large. We therefore feed more material into the furnace than is necessary.

Besides coke we have used several other carbonaceous materials for making carbide. We have used soft coal, anthracite, charcoal, pitch, tar, rosin, and asphalt, and obtained in all cases carbide. Most of these materials are not of sufficient importance to be taken into consideration and we will only add some words about the first three.

Charcoal, owing to its small percentage of ash, yields a very pure carbide. The only drawback, besides its price, is that it is so light that the gases carry if off to a considerable amount. It is therefore necessary to add from five to ten per cent. more carbon to the mixture if charcoal is used than if coke is used.

We used a soft coal which contained volatile matter 19.84 per cent. and ash 1.48 The mixture with soft coal gave a terrific blaze. The carbide was covered with a large amount of very porous slag in which there was much graphite. The average of results of two runs are : 6.41 pounds per horse power in twenty-four hours and 4.33 cubic feet of gas per pound, which equals 27.75 cubic feet of gas per horse power in twenty-four hours.

We used anthracite coal, which contained volatile matter 7.95 per cent. and ash 4.02 per cent. We made two runs with slacked and two with unslacked lime. There was no appreciable difference in the use of slacked and unslacked lime. The average result of the four runs was : 7.64 pounds per horse power in twenty-four hours and 4.03 cubic feet of gas per pound, which equals 30.79 cubic feet per horse power in twenty-four hours. These results are much lower than those obtained with coke. We can not therefore recommend the use of either anthracite or soft coal for making carbide. The superiority of coke and charcoal over anthracite is probably due to the porosity of the former materials, which must facilitate the volatilization of the carbon in the electric arc, which probably must precede the formation of carbide.

ON THE ACTION OF WAGNER'S REAGENT UPON CAFFEINE AND A NEW METHOD FOR THE ESTIMATION OF CAFFEINE.

BY M. GOMBERG.

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THE use of iodine in potassium iodide as a general qualitative reagent for alkaloids dates as far back as 1839.[1] It was, however, R. Wagner[2] who first employed it for the quantitative estimation of vegetable bases, and this solution has since been known as Wagner's reagent. He based his con-

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[1] Bouchardat: *Compt. Rend.*, 9, 475.

[2] *Dingl. poly. J.*, 161, 40; *Ztschr. anal. Chem.*, 1, 102.