ferrocyanide and consequently a homogeneous substance could not be obtained. I then tried to bring about the above reaction by shaking freshly precipitated mercurous chloride with a solution of potassium ferrocyanide, but no reaction took place. The following method, which was tried, might also be mentioned.

Mercurous carbonate was prepared by adding an excess of potassium bicarbonate solution to a mercurous nitrate solution. After a few days the mercurous carbonate is filtered and washed. The greater portion of the precipitate is transferred to an Erlenmeyer flask and acetic acid added in excess. To the turbid liquid obtained, a potassium ferrocyanide solution is added. A heavy precipitate is obtained on shaking, but analyses do not agree with the expected compound of the formula $Hg_4Fe(CN)_6$. The analysis of the substances obtained by the first and last method of preparation approach more closely the formula $Hg_3Fe(CN)_6$, which is the same compound obtained when potassium ferricyanide is added to mercurous nitrate. This seems to show that in the above-mentioned cases oxidation of the ferrocyanide occurred. The preparation of the compound $Hg_4Fe(CN)_6$ will therefore only become possible when a suitable solvent for mercurous chloride is found.

MICHIGAN COLLEGE OF MINES,
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THE PREPARATION OF BORON CARBIDE IN THE ELECTRIC FURNACE.

BY S. A. TUCKER AND H. J. W. BLISS.

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THE compound B_6C was first isolated by Joly[1] in some of the products prepared in connection with admantine boron, and Moissan[2] gives three methods for the preparation of the carbide.

(1) An arc is passed between carbon electrodes which are bound together with a mixture of boric oxide and aluminum silicate. Under these conditions the product is contaminated with silicon carbide.

(2) By heating amorphous boron with carbon to a temperature of about 3000° C., either by placing directly in the arc, or by exposing it to the radiant heat from the arc.

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[1] Compt. rend. 97, 456.

[2] "Le Four Électrique."

(3) The solution of boron and carbon in certain metals at electric furnace temperatures yields boron carbide, another method which was found by Moissan the most suitable for its preparation.

Further, Mühlhäuser[1] prepared and described a boride of carbon BC, and his method for its preparation consisted in heating a mixture of boric oxide and carbon in an electric furnace. The resulting product consisted of shining black globules of metallic lustre, and, according to the author, scarcely distinguishable from the admixed graphite. After treatment with acids and heating, the product blackened the fingers, but behaved in its action towards reagents similarly to Moissan's carbide.

The purpose of the present investigation was to prepare the carbide in the pure form, using boron oxide as the raw material, and thus to avoid the necessity of using amorphous boron which is obtained with some difficulty and considerable expense.

The first experiments were carried out in the resistance type of electric furnace, constructed entirely of carbon or graphite so as to eliminate the presence of silica. The mixture composed of boric oxide and petroleum coke in molecular proportions was placed about a carbon core or rod raised to a high temperature by the current.

Some metallic-looking globules were thus obtained, but the yield was small and from the fact that the largest portion was found in contact with the carbon rod, it appeared evident that the highest temperatures were necessary for its formation.

The type of furnace was therefore changed to that of the arc, in which one pole was made the graphite crucible composing the body of the furnace while the other consisted of a graphite rod hung vertically through the opening of the crucible. This arrangement enables a powerful arc to be operated in a comparatively small space. For purposes of heat insulation the outside of the crucible was surrounded with a heat-conserving layer such as magnesia. The yield of carbide in such a furnace is decidedly better than in the case of a resistance furnace. The chief difficulty met with was from the volatility of the boric oxide which was to some extent driven off without taking part in the reaction.

Attempts were made to reduce this tendency by the addition

[1] Z. anorg. Chem. 5, 92.

of lime or magnesia, so that these borates might be formed first, but such additions did not improve the yield appreciably.

The method which was finally adopted was to place the petroleum coke in a layer at the bottom of the furnace, and then add the powdered boric oxide through a perforation in the vertical electrode. This, in a large measure, hindered the volatilization of the oxide, as it was thus brought directly in contact with carbon heated to the temperature of the arc.

PHYSICAL AND CHEMICAL PROPERTIES OF THE PRODUCT.

The residual black powder obtained contained no constituent which could be distinguished from the excess of petroleum coke, even under the microscope. The larger globules and the layer found on the core of the resistance furnace resembled fused magnetite, were brittle, easily crushed, and had a brilliant crystalline structure.

In all cases where a layer of the carbide was found attached to the electrode there was also a layer of graphite of fine quality, probably formed by its decomposition. This graphite was very similar to that obtained by the decomposition of silicon carbide. In some cases, however, the boron carbide seemed to melt under the influence of the great heat and thus coat the surface of carbon articles placed in the furnace.

The globules were found to conduct electricity, had a hardness greater than that of silicon carbide and resisted the action of all the usual acids. Fused alkali decomposed it with evolution of carbon monoxide; strong oxidizing agents, such as hot nitric acid with potassium chlorate, and boiling sulphuric acid were without action upon it. The substance is unaffected by heating to redness in the air.

The material obtained from the furnace, consisting of the globules together with other material which appeared to consist of the carbide, was powdered to pass a 30-mesh sieve and then treated after the manner indicated by Moissan, by repeated boiling with nitric acid in conjunction with potassium chlorate, and then with boiling sulphuric acid; in this way the graphite was eliminated and a product of uniform density was obtained which is determined in a liquid separating-column. The compound, after fusion with the alkaline carbonates, was analyzed by Gladding's method.[1] The first lot analyzed gave over 70 per

[1] This Journal, 20, 288.

cent. boron, and was therefore again treated with nitric acid and the chlorate several times. A product was finally obtained which gave, on fusion and analysis, a boron content of 82.8 per cent., allowing for a small undissolved residue, which proved to be carbon.

Since some of this carbon was probably oxidized in the fusion, this result would be a little low. The theoretical boron content of B_6C is 84.6 per cent. Therefore, this analysis together with the general properties of substance identify it with Joly's and Moissan's carbide. The conclusions reached by the foregoing are that Mühlhäuser actually obtained a mixture of the carbide with graphite, owing to the difficulty of separating the latter, but that he failed to obtain any large masses of the product because of the very high temperature necessary.

For the preparation of the carbide from boric oxide in considerable quantities a special form of electric furnace would have to be designed, so that means could be provided for the condensation of the boric oxide fumes—possibly this could be accomplished by using some sort of shaft furnace, whereby these fumes would be retained by the descending charge.

COLUMBIA UNIVERSITY,
NEW YORK.

ALUMINUM PHENOLATE.

BY ALFRED N. COOK.

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ALUMINUM phenolate was first prepared by Gladstone and Tribe[1] about twenty years ago, but no study was made of the compound except to distil it. While engaged in the study of phenyl ether and its preparation from aluminum phenolate it appeared to the author that a further study of the compound might be interesting and profitable.

Preparation.—The mode of operation found best in its preparation is to heat from 100 to 500 grams of phenol in a liter flask fitted with a return Liebig condenser. A simple air condenser is not sufficient in manufacturing a large quantity, since the escaping hydrogen keeps the tube hot and carries with it a large part of the phenol. In two or three instances in the earlier ex-

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[1] J. Chem. Soc. 41, 7.