Comparing the amounts of coke with fixed carbon and ash of the proximate analysis, it will be seen that there is considerable difference; due, doubtless, to incomplete distillation as the temperature of 830° is evidently not sufficient to drive off all of the volatil matter in some of the samples (see Porter and Ovitz, Bureau of Mines, *Bull.* I). Distillation was apparently complete, however, in the Alberta lignite.

The following table will show the difference between the coke left after the distillation and the fixed carbon obtained by the proximate analysis:

	N. Dakota.	Saskatchewan.	Alberta.
Distillation, coke including ash	52.50	53.20	49.40
Fixed carbon, including ash	47.60	49.65	49.30
	4.90	3.55	0.10

The following table shows the relative amounts of hygroscopic water as compared with the total amount found by destructive distillation:

	N. Dakota.	Saskatchewan.	Alberta.
Per cent. of water obtained by distillation	36.30	28.00	32.70
Per cent. of hygroscopic water	12.95	15.96	22.50
Water produced by distillation	23.35	12.04	10.20
Per cent. of H present (by ultimate anal.)	4.30	3.77	4.21

Calculating the amounts of water found to the dry analysis, the North Dakota lignite would consume 2.98 per cent. of the H to form water, the Saskatchewan 1.59 and the Alberta 1.47, leaving respectively, 1.32, 2.18 and 2.80 per cent. of H to be evolved either as free hydrogen or in the form of hydrocarbons.

From the above data and from the moisture determinations in samples from different localities[1] it is apparent that the composition of the various lignites depends somewhat upon the locality. The southern samples contain more moisture than those from farther north. The total amount of volatil constituents, however, increases slightly as we go northward. The individual gases seem to vary but not regularly. Carbon monoxide is practically constant and marsh gas changes but little. There is, however, an increase in the amount of hydrogen as we pass northward. On the whole, there is but little difference in the volatil constituents from the different samples, although there is a distinct difference in the general physical appearance.

MINNEAPOLIS, MINN.

NOTES.

Preparation of Stannic Iodide and Its Solubility in Certain Organic Solvents.—In the preparation of stannic iodide for use in this laboratory

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[1] Part I, THIS JOURNAL, **29**, 1488.

recently, recourse was had to the method of Schneider,[1] depending on the reaction of tin and iodine in a medium of carbon disulfide. It was thought desirable to avoid, if possible, the use of the latter volatil and unpleasant reagent, and accordingly, after a little experimentation, an attempt was made to substitute carbon tetrachloride therefor. It has been found that the tetrachloride forms an excellent substitute for the disulfide in this process, and that it offers some further advantage in the matter of the purification of the product by crystallization from solution. The following is the method finally adopted: In a long-necked Kjeldahl flask there were placed twelve to fifteen grams of tin and forty grams of iodine, and these were covered with about 75 cc. of carbon tetrachloride. The neck of the flask was fitted with a cork stopper bearing a small reflux condenser. The bottom of the flask was then heated gently with a burner until the tetrachloride commenced to boil, when the flame was lowered until just sufficient to maintain the ebullition. The reaction between the tin and the iodine usually begins before the boiling point of the medium is reached, and continues of itself, supplying considerable heat. As the concentration of the iodine becomes less, the end point may be hastened by heating the solution more strongly. The end of the reaction is indicated by the complete change of the violet color of the solution to deep orange, and the absence of violet fumes of iodine above the liquid. A slight excess of tin is desirable to avoid the necessity of removing an excess of iodine by distillation after the reaction is over.

The stannic iodide is much more soluble in the boiling carbon tetrachloride than at ordinary temperatures. Accordingly, the boiling solution is decanted through a folded filter, preferably in a steam-jacketed funnel, the filter washed with boiling tetrachloride, and the filtrate cooled in ice-water. An abundant precipitation of small crystals of stannic iodide takes place; the mother liquor may be drained by centrifugation or otherwise, and evaporated for the production of a second small crop of crystals. The crystals may be further purified for use by again dissolving in hot tetrachloride and recrystallizing, or by sublimation as used by Fitzgerald.[2]

Two determinations of the solubility of stannic iodide in carbon tetrachloride gave the following figures:

Temp.	Sp. gr. of solution.	Grams of SnI_4 in 100 cc. solution.	Grams of SnI_4 in 100 grams solution.
22.4	1.59	8.35	5.25
50.0	1.63	20.47	12.50

It has also been found that chloroform may be used in place of the tetrachloride, but its lower boiling point offers one objection to its use.

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[1] *Pogg. Ann.*, 127, 624 (1886).

[2] THIS JOURNAL, 29, 1694 (2907).

The range of solubility of stannic iodide in chloroform is of the same order as in carbon tetrachloride, and the iodide may be crystallized from it in the same manner.

The following figures were obtained for the solubility of stannic iodide in chloroform and in benzene:

Solvent.	Temp. °C.	Sp. gr. of solution.	Grams of SnI_4 in 100 cc. solution.	Grams of SnI_4 in 100 grams solution.
$CHCl_3$	28.0	1.50	12.32	8.21
C_6H_6	20.2	0.95	12.02	12.65

Stannic iodide appears to be fairly stable in contact with the air during ordinary manipulations, and while in solution in carbon tetrachloride, chloroform, etc. Under the prolonged action of moist air, however, it forms the oxide.

An analogous procedure may be used for the preparation of the corresponding bromide. The heat evolved in the reaction with bromine is considerably more than with iodine, and it is advizable to place the tin alone in the flask, covered with a layer of tetrachloride, and to add the bromine subsequently, dissolved in a further amount of tetrachloride, in small portions at a time, through the open end of the condenser. The reaction takes place spontaneously, without the addition of external heat; the reaction flask may be kept in a bath of cold water with advantage. The bromide is much more sensitive to the action of moist air than the iodide, and on this account much more care is necessary in handling the product to assure exclusion of water vapor.

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Lecture Table Experiment on Nascent Hydrogen.—Many experiments have been proposed to convince students that hydrogen in the process of formation upon the surface of a metal is especially active. The general principle of the following experiment has of course no claim to originality, but it has been found that the particular reagents suggested make the demonstration especially rapid and striking.

A large Erlenmeyer flask is partly filled with sulfuric acid, 15 cc. of the concentrated acid to the liter. A few grams of magnesium ribbon are first introduced, and then enough of a dilute solution of ferric chloride and potassium ferricyanide to impart to the mixture a decided color. After agitating the flask for a short time, it is seen to be filled with a finely divided deep blue precipitate. Next it is proved in the usual manner that the several reagents separately do not produce a similar effect: (*a*) magnesium is introduced into a dilute (neutral) solution of ferric chloride and potassium ferricyanide; (*b*) dilute sulfuric acid and magnesium sulfate are added to a second portion; (*c*) hydrogen is bubbled through a third portion.

The concentration of the acid should not vary too widely from that suggested. If the acid is much more dilute, the action is too slow; if much more concentrated, the reaction products are pale green rather than deep blue.

The point at issue can easily be understood by a class without any statement of the composition of the brown solution or that of the blue reaction products.

G. S. Forbes.

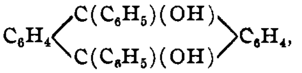
[Contribution from the Chemical Laboratory of Harvard University.]

THE ACTION OF ETHYL MAGNESIUM BROMIDE ON ANTHRAQUINONE.[1]

By Latham Clarke and Paul Whittier Carleton.

Received August 30, 1911.

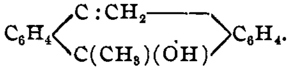
It has been shown by Haller and Guyot[2] that an excess of phenyl magnesium bromide reacts with anthraquinone forming 9,10-diphenyl-9,10-dihydroxydihydroanthracene,



$$C_6H_4\left\langle\begin{array}{c}C(C_6H_5)(OH)\\C(C_6H_5)(OH)\end{array}\right\rangle C_6H_4,$$

while an excess of the quinone gives phenyloxanthrone (or phenyloxanthranol).

Guyot and Staehling[3] prepared analogous compounds from methyl anthraquinone, using phenyl and naphthyl magnesium bromides. They also treated anthraquinone with methyl magnesium bromide, which produced 9,10-dimethyl-9,10-dihydroxydihydroanthracene, and this compound readily lost one molecule of water, giving 9-methene-10-methylhydroxydihydroanthracene,



$$C_6H_4\left\langle\begin{array}{c}C:CH_2\text{———}\\C(CH_3)(OH)\end{array}\right\rangle C_6H_4.$$

The action of ethyl magnesium bromide on anthraquinone was studied in this laboratory in 1907 by Clarke and Victor Cobb, who found that the normal action was to give 9,10-diethyl-9,10-dihydroxydihydroanthracene, but certain other compounds were formed by side reactions. These last we were prevented from studying, on account of the departure of Mr. Cobb from Cambridge.

In the autumn of 1910 this study was taken up by the present writers. We have found that with an excess or ethyl magnesium bromide, the anthraquinone is converted into the di-ol; and when an excess of the quinone is used, the product is ethyloxanthranol. The di-ol is very sus-

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[1] The work described in this paper formed part of a thesis presented to the Faculty of Arts and Sciences of Harvard University for the degree of Doctor of Philosophy by Paul Whittier Carleton.

[2] *Compt. rend.*, 138, 327, 1251 (1904); *Bull. soc. chim.*, 31, 795, 979 (1904).

[3] *Bull. soc. chim.*, 33, 375 1104 (1905).