The French method deducts from the per cent. of cane sugar five times the ashes, plus the per cent. of inverted sugar. By this method the problem stands :

Cane sugar	85.00%
Ashes × 5 + inverted sugar 1.7 × 5 + 3.2 = 11.7	= 11.70%
Crystallizable sugar	73.30%
Molasses	26.70%
	100.00%

The brand of the sugar makes some difference in value ; for example, a centrifugal sugar of the same color, test and other factors being the same, is preferred to the extent of about $\frac{1}{16}$ to $\frac{1}{8}$ of a cent. For each degree or per cent. of a centrifugal sugar, about $\frac{1}{16}$ of a cent is allowed, so that a centrifugal sugar testing 90 per cent., worth 9 cents, would be worth 9½ cents if it tested 95 per cent., other factors remaining the same.

XLI.—On the Preparation of Methylene Chloride, and Dioxyethyl-Methylene.

By Wm. H. GREENE, M. D.

With the exception of the diethyl ether of methylene glycol, all of the oxyethyl substitution compounds of methane have already been described. Ortho-formic ether, $CH(OC_2H_5)_3$, was studied by Kay and Williamson, and is generally known as Kay's ether. Ortho-carbonic ether, $C(OC_2H_5)_4$, was discovered and described by H. Bassett. Methyl-ethyl ether has long been known.

By a reaction similar to that by which these ethers are formed, I have isolated dioxyethyl-methylene, the reaction between sodium ethylate and methylene chloride taking place as indicated by theory.

The chief difficulty lies in the preparation of pure mythylene chloride. The process described by Perkin, and depending upon the reduction of chloroform by zinc and ammonia, yields only small quantities of methylene chloride, and the direct chlorination of methyl chloride gives equally unsatisfactory results. The method which, after numerous experiments, I have found to answer best, consists in the reduction of an alcoholic solution of chloroform by zinc and hydrochloric acid.

The zinc and chloroform (mixed with several times its volume of alcohol) are placed in a flask connected with a suitable condensing apparatus, and hydrochloric acid is added in small portions. The reaction develops considerable heat, and methylene chloride and chloroform distil over ; when the reaction has somewhat subsided, and no more liquid distils, more hydrochloric acid is added, and a moderate heat applied, if necessary. In any case, the mixture is heated towards the close of the operation, until alcohol begins to distil in quantity. The operation is then arrested, and the product in the receiver is washed, dried and rectified, that portion which passes below about 53° being retained. The residue is returned to the flask, and again submitted to the action of the zinc and hydrochloric acid. By several careful rectifications of the product passing below 53°, pure methylene chloride, boiling at 40–41°, is obtained.

By several operations in this manner, the yield of methylene chloride may be brought up to about twenty per cent. of the chloroform employed.

Little or no advantage is gained by attempting to fractionate the product as it distils from the flask, so that the chloroform may flow back into the reducing mixture, for such distillation necessarily takes place in a stream of hydrogen, which carries with it about as much chloroform as methylene chloride.

Dioxyethyl-methylene.—This compound was prepared by gradually introducing one molecule of sodium into a mixture of one molecule of methylene chloride and about four times the theoretical quantity of absolute alcohol, contained in a flask connected with a reflux condenser. After all of the sodium has been introduced, the mixture is heated on a water-bath for about an hour, and is then distilled. The distillate is fractionated, and the portion which passes below 78° contains all of the diethyl ether. It is agitated with a tolerably concentrated solution of calcium chloride, and the light etherial layer is separated, dried over calcium chloride, and carefully rectified, that portion being retained which passes between 86° and 89°.

Dioxyethyl-methylene, so obtained, is an etherial liquid, having a penetrating, pleasant odor, somewhat recalling that of mint. Its specific gravity at 0° is 0.851, and it boils at 89°, under a pressure of 769 millimeters. It is slightly soluble in water, from which it may be separated by the addition of calcium chloride ; it mixes in all proportions with ether and alcohol, and cannot readily be separated from its alcoholic solution if much alcohol be present ; in such a case, fractional distillation, and treatment of the portion which passes below 78°, with solution of calcium chloride, effect the separation.

XLII.—ACTION OF BENZO-TRICHLORIDE ON PRIMARY AMINES.

BY JAMES H. STEBBINS, JR., B. S.

O. Doebner has shown, by his experiments on "malachite green" (Ber. chem. Gesell., **II**, 1,236), that benzo-trichloride will only unite with tertiary amines, in the presence of zinc or aluminium chlorides.

Now, it occurred to me, that some of the primary amines might unite directly with benzo-trichloride, without the aid of metallic chlorides, to form some new and interesting compounds.

Therefore, acting on this principle, I took para-toluidine as my starting point, as I happened to have quite a quantity of the latter at my disposal, and I think that the following experiments justify (as far as I have proceeded) the above-stated theory.

If equal molecules of benzo-trichloride ($C_6H_5C = Cl_3$) and para-toluidine ($C_6H_4CH_3NH_2$) be allowed to react on one another, at the ordinary temperature, the mixture soon grows very warm, and gives off copious fumes of HCl, mixed with aqueous vapor. The reaction is ended when the fumes are no longer evolved, and the white granulated mass thus obtained is dissolved in strong alcohol, from which it crystallizes on cooling in two forms, namely—those obtained by slow cooling are long, white needles, having a strong, vitreous lustre, and melting at 155° C., and those produced by rapid cooling are small, white, rhombic prisms.

If the substance be heated a little over its melting point, it sublimes, producing small, white needles which appear to be the original compound unaltered.

It is only sparingly soluble in boiling water and HCl, but is quite soluble in acetic acid.

Strong H_2SO_4 dissolves it readily, giving off fumes of hydrochloric acid, and it is precipitated, probably free from chlorine, by the addition of cold water.

This compound, then, is neither a base nor an acid, and its constitution can only be explained by converting it by oxidation or reduction, into a substance of known composition, and I think that this problem can easily be solved by its property of producing, first a red, then a black precipitate, with a solution of potassic permanganate.

The precipitate thus formed is collected on a filter, washed with a little water, and then boiled for a short time with strong alcohol, and filtered rapidly. On cooling, fine, yellow-colored needles make their appearance, the constitution of which, as well as that of the mother substance, I hope to lay before you at the next meeting.