

XI.—*Note on the Preparation of Carbonate of Amyl.*

BY JOHN A. BRUCE,

OF THE ROYAL COLLEGE OF CHEMISTRY, LONDON.

Carbonate of amyl has been lately examined by Mr. Medlock, whose experiments have been described to the Chemical Society.\* He obtained this compound by a method which had not yet been used for the preparation of carbonic ethers, namely, by the action of phosgene gas upon fusel-oil, and subsequent decomposition of the chlorocarbonate formed, under the influence of heat, in the presence of water. The process which originally had furnished the carbonate of the ethyl-series, and which consists in distilling the oxalate with potassium, has never been applied to the amyl-series.

The reaction of potassium upon the ether-oxalates being still but

\* Chem. Soc. Qu. J. I, 368.

imperfectly understood, Dr. Hofmann requested me to repeat the experiment with oxalate of amyl, in order to obtain some additional data for a more correct interpretation of the phenomena attending the decomposition.

Potassium, when coming in contact with perfectly dry oxalate of amyl, is rapidly attacked, the liquid becomes warm, and a brown mass forms around the metallic globules, whilst a colourless inflammable gas is disengaged. The reaction, however, soon slackens, and only commences again on heating the liquid. On distilling the fluid, a brown residue was obtained, consisting chiefly of carbonate of potash and charcoal, while a pale-yellow distillate passed over; this liquid, when rectified with a thermometer, was found to consist of not less than four different substances. It began to boil at  $130^{\circ}$  C. ( $266^{\circ}$  F.), at which temperature chiefly fusel-alcohol distilled; the boiling-point then rapidly rose to about  $225^{\circ}$  C. ( $433\cdot4$  F.), when it became stationary for a very considerable time, indeed until three-fourths of the whole had passed over. The liquid distilling at this temperature was pure carbonate of amyl, as proved by the analyses hereafter to be detailed. The mercury then again rose until the temperature was  $260^{\circ}$  C. ( $500^{\circ}$  F.), when it became once more stationary, but only for a short period, evidently because a small portion of undecomposed oxalate of amyl, which boils at this temperature, was still present in the liquid. After this had been evaporated, a viscous, somewhat dark residue, having a very powerful odour, remained in the retort.

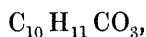
The portion which had been collected between  $223^{\circ}$  and  $226^{\circ}$  C. ( $433\cdot4^{\circ}$  and  $438\cdot8$  F.) was repeatedly redistilled, until a liquid of a constant boiling-point at  $226^{\circ}$  C. ( $438\cdot8$  F.) was obtained, which at  $15\cdot5^{\circ}$  C. ( $60^{\circ}$  F.) had a sp. gr. of  $0\cdot9065$ . When submitted to combustion, it gave the following results :

- I.  $1\cdot91$  grains of substance gave
  - $4\cdot60$  „ „ carbonic acid, and
  - $1\cdot95$  „ „ water.
- II.  $2\cdot05$  „ „ substance gave :
  - $4\cdot90$  „ „ carbonic acid, and
  - $2\cdot10$  „ „ water.

Percentage-composition :

	I.	II.
Carbon. . .	$65\cdot68$	$65\cdot12$
Hydrogen. .	$11\cdot30$	$11\cdot31$

These numbers agree pretty closely with the formula,



as may be seen in the following comparison of the theoretical numbers with the results of the experiments :

		Theory.		Mean of the Experiments.
11 eqs. Carbon . .	66	65·34		65·40
11 „ Hydrogen .	11	10·89		11·35
3 „ Oxygen . .	24	23·77		—
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		101	100·00	

Carbonate of amyl may be produced by the action of potassium or sodium (the latter metal was indeed employed in most of my experiments) upon oxalate of amyl with great facility, and to any extent ; its preparation in this reaction is not attended with the same difficulties which we experience in performing the experiment in the ethyl-series. The actual process, however, which gives rise to its formation remains still doubtful, although the preceding experiments have furnished a new element for its correct interpretation, namely, the observation of a large quantity of amyl-alcohol being reproduced during the reaction—a reproduction which has escaped notice in the preparation of the carbonic ether *par excellence*, on account of the solubility of the ethyl-alcohol in water.