

## A NEW OXYACID OF NITROGEN.

ALTHOUGH rapid strides have been made recently in the chemistry of nitrogen and its inorganic derivatives, since the discovery of hyponitrous acid by Dr. Divers in 1871, no new oxyacid of nitrogen has been described. In the current number of the *Gazzetta Chimica Italiana* (July 31), there is an account by Dr. A. Angeli of the preparation and properties of the sodium and barium salts of a new acid,  $H_2N_2O_3$ , which fills the gap between hyponitrous and nitrous acids. The method of obtaining this acid is simple and elegant. An alcoholic solution of free hydroxylamine is prepared in the usual manner from hydroxylamine hydrochloride and sodium ethylate, an excess of the latter being taken, and to the solution after filtering off the precipitated salt is cautiously added the theoretical quantity of ethyl nitrate. The reaction proceeds according to the equation  $C_2H_5ONO_2 + NH_2OH = C_2H_5OH + H_2N_2O_3$ , the white sodium salt of the new acid commencing to separate out at once. From this salt, which on analysis proved to be  $Na_2N_2O_3$ , the barium salt is readily obtained in a pure state by adding barium chloride to the dilute aqueous solution. These salts are both moderately stable in the dry state, but are easily decomposed, on boiling the aqueous solution, into the hydrate of the metal and nitric oxide. The same gas is given off quantitatively on acidifying the aqueous solution, and hence all attempts to isolate the free acid have failed. As regards the composition of this acid, from its mode of formation, the formula  $(NO_2)NH.OH$ , or nitro-hydroxylamine, naturally follows; and this is to some extent confirmed by the fact that the yellow silver salt, momentarily obtainable from the aqueous solution, is reduced in a few seconds to metallic silver, and Fehling's solution is also readily reduced. The existence of an acid of this composition has already been indicated by Dr. A. Thum, who showed that hydroxylamine salts when oxidised by potassium permanganate in hot alkaline solution take up exactly as much oxygen as corresponds to the formation of  $Na_2N_2O_3$ , the formula of which

might be  $O \begin{matrix} \diagup N.O.Na \\ \diagdown N.ONa \end{matrix}$ . This salt, not yet

isolated, is clearly isomeric with the sodium salt above described, since it suffers no further oxidation on boiling with excess of potassium permanganate. It would also yield only one ethyl derivative, whilst nitro-hydroxylamine might be expected to give the isomers  $(NO_2).NEt.(OH)$  and  $(NO_2).NH.OEt$ , which would be readily distinguishable, and this point is being followed up by Dr. Angeli. He has also applied the same reaction to amyl nitrite and to nitrobenzene (*Berichte*, July 27). The amyl nitrite formed a sodium salt, the aqueous solution of which gave a yellow silver salt resembling silver hyponitrite. Nitrobenzene gave a substance which is probably  $C_6H_5.NO : N(OH)$ , identical with the nitroso-phenylhydroxylamine,  $C_6H_5.N(NO).OH$  of Bamberger. The further development of this work, which may be expected to throw light on the constitution of Traube's isonitramine derivatives, Frankland's dinitroethylic acid, and of Pelouze's salt, will be looked for with much interest.

G. N. H.

A RESEARCH ON THE LIQUEFACTION OF HELIUM.<sup>1</sup>

MY experiments on the liquefaction of helium were carried out with a sample of that gas, sent to me by Prof. Ramsay from London, in a sealed glass tube holding about 140 ccm. I take this opportunity of rendering him my most sincere thanks. In his letter Prof. Ramsay informed me that the gas had been obtained from the mineral clèveite, and that it was quite free from nitrogen and other impurities, which could be removed by circulation over red-hot magnesium, oxide of copper, soda-lime, and pentoxide of phosphorus. The density of the gas was 2.133 and the ratio of its specific heats ( $C_p/C_v$ ) 1.652; the latter figure indicating that the molecule of helium was monatomic, as had already been found to be the case with argon.

<sup>1</sup> Translated from the original paper, by Prof. K. Olszewski, in the *Bulletin de l'Académie des Sciences de Cracovie* for June 1896, "Ein Versuch, das Helium zu verflüssigen," by Morris Travers.

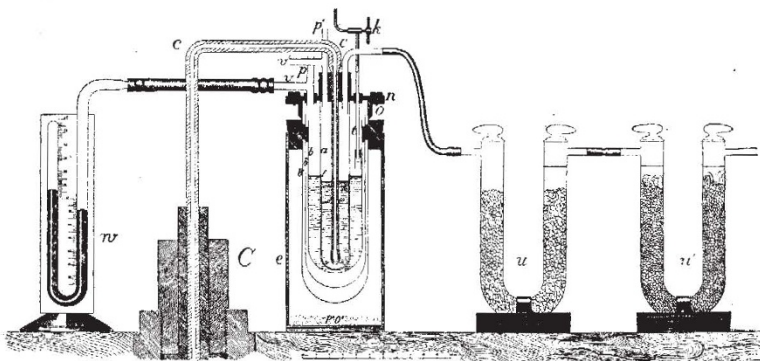
NO. 1399, VOL. 54]

Prof. Ramsay further informed me that the gas was only very slightly soluble in water; 100 ccm. of water dissolving scarcely 0.7 ccm. of helium.

From the results of my earlier experiments I had been led to expect that it would be only possible to liquefy helium at a very low temperature; the small values obtained for the density and solubility of the gas, together with the fact that its molecule is monatomic, indicating a very low boiling-point. For this reason I did not consider it necessary to use liquid ethylene as a preliminary cooling agent, but proceeded directly to conduct my experiments at the lowest temperature that could be produced by means of liquid air. The apparatus employed in these investigations is figured in the accompanying diagram.

The helium was contained in the glass tube, *c*, of the Cailletet's apparatus, *C*. The tube, *c*, reached to the bottom of a glass vessel, *a*, which was intended to contain the liquid air. The vessel, *a*, was surrounded by three glass cylinders, *b*, *b'*, and *b''*, closed at the bottom and separated from one another. The outer vessel, *b''*, was made just large enough to fit into the brass collar, *o*, which supported the lid, *u*, of the apparatus. The tube, *a*, fitted into an opening in the centre of the lid; the tube, *z*, connected with an apparatus delivering liquid oxygen, passed through a hole on the right. The vessel, *b*, was also connected with a mercury manometer and air-pump by means of a T-tube, *p*, *v*, one arm of which passed through the third hole in the lid of the apparatus. The tube, *a*, was closed by a stopper, through which passed the tube, *c*, of the Cailletet's apparatus, a tube connected with the drying apparatus, *u*, *u'*, and one limb of a T-tube, by means of which the manometer and air-pump could be put in connection with the interior of the vessel. The lower part of the whole apparatus was enclosed in a thick-walled vessel, *e*, containing a layer of phosphorus pentoxide.

By turning the valve, *k*, the vessel, *b*, could be partially filled



with liquid oxygen, which, under a pressure of 10 mm. of mercury, boiled at about  $-210^{\circ} C$ . Almost immediately the gaseous air began to condense and collect in the tube, *a*; a supply of fresh air was constantly maintained through the drying tubes, *u* and *u'*, which were filled with sulphuric acid and soda-lime respectively. When the quantity of liquid air ceased to increase, the tap on the U-tube, *u*, was closed, the T-tube, *p' v'*, was connected with the manometer and air-pump, and the liquid air was made to boil under a pressure of 10 mm. of mercury. In order to protect the liquid air from its warmer surroundings, a very thin, double wall tube, *f*, reaching to the level of the liquid in the outer vessel, was placed inside the tube *a*. When, as in some of my experiments, liquid oxygen was used in the inner vessel, this part of the apparatus was dispensed with.

Using the apparatus I have just described, I carried out two series of experiments, in which liquid air and liquid oxygen were employed as cooling agents. The tube of the Cailletet's apparatus was thoroughly exhausted by means of a mercury pump, and then carefully filled with dry helium. In the first series of experiments the helium, confined under a pressure of 125 atmospheres, was cooled to the temperature of oxygen boiling, first under atmospheric pressure ( $-182^{\circ}.5$ ), and then under a pressure of 10 mm. of mercury ( $-210^{\circ}$ ). The helium did not condense under these conditions, and even when, as in subsequent experiments, I expanded the gas till the pressure fell to twenty atmospheres, and in some cases to one atmosphere, I