NOTES ON REINSCH'S TEST FOR ARSENIC AND ANTIMONY.

BY JAS. LEWIS HOWE AND PAUL S. MERTINS. Received September 12, 1896.

T HAT Reinsch's test for arsenic possesses, in point of convenience, marked advantages over that of Marsh, is generally acknowledged, but it has been questioned both as to delicacy and as to accuracy in distinguishing between arsenic and antimony. As to the former point, Reinsch, in his second article on the test,¹ states that arsenic may be detected in a solution of one part per million. In his original description² of the test he placed the accuracy about one-third of this. Our own experiments show that this accuracy is not overstated. The fact that arsenious oxide and antimonous oxide (Sb₂O₂) are isomorphous in their crystallization has led to the conjecture that antimonous oxide subliming from the copper in the closed tube might appear in the brilliant octahedra, characteristic of arsenic in the test.

Experiments bearing on this point were made as follows:

Reinsch's test was applied to the different compounds of arsenic in this laboratory and in each case several sublimation tubes were used. The test was carried out by boiling the substance with sixteen per cent. hydrochloric acid, in which several strips $(2.5 \times 0.5 \text{ cm.})$ of thin, pure copper were placed. After fifteen minutes (except in cases to be mentioned later) the strips of copper were removed, washed and dried, and after rolling or folding to small compass, placed in open tubes five cm. long and not over five-tenths cm. diameter. These tubes were held in an inclined position in the lowest possible flame of a Bunsen burner until the arsenic sublimed; a second or two usually suffices.

The test was similarly carried out with compounds of antimony and also with various organs of two cats, one killed by six grains of tartar emetic, dying six hours after administration, and the other dying in three days after the administration of the first of six small doses given every twelve hours. Each dose was two grains, but much of this was probably not taken into the system. A perceptibly higher degree of heat was necessary to

¹ H. Reinsch : De l' Essai de l'Arsenic par le Cuivre : J. pharm. Chim., 2, 361, (1842).

² H. Reinsch : Ueber das Verhalten des metallischen Kupfers zu einigen Metalllösungen : *J. prakt. Chem.*, 24, 244, (1842).

954 REINSCH'S TEST FOR ARSENIC AND ANTIMONY.

sublime the antimony than was the case with arsenic; altogether 185 tests were made, most of them furnishing good sublimation tubes. Each tube was numbered as made, and later the whole number were mixed and sorted for arsenic and antimony by examination with a microscope of low power. Reference to the note book showed that in no case had a mistake been made, in fact in every case the arsenic sublimation could easily be distinguished from that of antimony by the naked eye. In no case did the sublimate of antimonous oxide show a trace of crystallization under the microscope used, nor did the arsenious oxide fail in any case to show the characteristic brilliant octahedral crystals.

The evidence that the antimonous oxide cannot appear in crystals which might be mistaken for arsenic is of course negative, but owing to the variety of forms used it must be considered to have the weight of positive evidence.

As regards the substances tested, the following may be recorded :

All arsenious compounds soluble in hydrochloric acid gave the deposit on copper immediately on heating.

Commercial "metallic" arsenic gave the deposit readily.

Freshly sublimed "metallic" arsenic (bright crystals) gave no deposit.

Arsenates gave a deposit only after several minutes boiling.

In the presence of nitric acid or chlorates no test is obtained owing to the solution of the copper.

Whenever aqua regia or potassium chlorate is necessary for solution of an arsenic compound, the solution should be evaporated to dryness with hydrochloric acid. The test can then be carried out as with arsenates.

The presence of organic matter in the arsenic solution does not affect the test, hence it can be applied directly to any organs without any previous destruction of tissue. If much arsenic is present it is best to use but a small portion of the substance, since if much arsenic is deposited on the copper, it will not adhere with firmness.

Antimony is not precipitated on the copper as rapidly as arse-

nic, and the deposit has a decidedly violet tint, very distinct from the iron gray deposit of arsenic.

The following distribution of antimony in the two cats may be added :

Acute poisoning (6 hours). Slow poisoning (72 hours). Stomach.-Heavy deposit and sub-Good tests. limate. Good test with $\frac{1}{100}$ of stomach. Liver .- Not so heavy deposit as stomach. Good sublimate. mate. Heart .- Good deposit after several hours boiling. Good sublimate. Pancreas.-Faintdeposit. No distinct sublimate. Spleen .- Faint deposit. No dis-

- tinct sublimate.
- Kidney .- Faint deposit. No distinct sublimate.
- Intestine .- Good deposit and sublimate.

Muscle.-Faint deposit on two days boiling. No sublimate.

Brain .- No deposit.

Spinal Chord.-No deposit.

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Heavy deposit and good subli-

Good deposit on ninety minutes boiling. Good sublimate.

Good deposit and sublimate.

Good deposit and sublimate.

Good deposit and sublimate.

- Slight violet tinge to copper. No sublimate.
- Marked violet tint to copper. No sublimate.

NOTES ON THE DETERMINATION OF PHOSPHORUS IN STEEL AND CAST IRON.

BY GEORGE AUCHY.

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F the many improvements made in recent years in the method of determining phosphorus in steel, that of Jones -the use of the "reductor"-is not the least. There has been, however, some difference of opinion as to the completeness of the reduction accomplished by its use. Quoting from three most recent publications on the subject: Doolittle and Eavenson consider the reduction of the molybdic acid to be to a point cor-