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ARTHUR A. NOYES, Editor.

REVIEWERS: Analytical Chemistry, H. P. Talbot and W. H. Walker; Biological Chemistry, A. G. Woodman; Carbohydrates, G. W. Rolfe; General Chemistry, A. A. Noyes; Geological and Mineralogical Chemistry, W. O. Crosby and M. L. Fuller; Inorganic Chemistry, Henry Fay; Metallurgical Chemistry and Assaying, H. O. Hofman; Organic Chemistry, J. F. Norris; Physical Chemistry, H. M. Goodwin; Sanitary Chemistry, E. H. Richards; Industrial Chemistry, A. H. Gill and F. H. Thorp.

INORGANIC CHEMISTRY.

HENRY FAY, REVIEWER.

Note on the Preparation of Metallic Lithium. By LOUIS KAHLLENBERG. *J. Phys. Chem.*, 3, 602-603.—Lithium may be obtained in white metallic form from solutions of lithium chloride in pyridine by electrolysis at room temperature. By electrolyzing a concentrated solution of the chloride, using a carbon plate for anode and an iron plate for cathode, a current of from 0.2 to 0.3 ampere per 100 sq. cm. of kathode area, and a difference of potential of but 14 volts between the electrodes, a dense, well-adhering, silver white coating of metallic lithium will soon be deposited.

The Supposed Isomeric Potassium Sodium Sulphites. By GEO. S. FRAPS. *Am. Chem. J.*, 23, 202-214.—According to Röhrig two isomeric sodium potassium sulphites may exist, the one being formed by neutralization of acid sodium sulphite with potassium carbonate, the other by neutralization of acid potassium sulphite with sodium carbonate. Schwicker claims to have obtained two double salts, $4\text{C}_2\text{H}_5\text{SO}_3\text{Na} + \text{KI}$, and $4\text{C}_2\text{H}_5\text{SO}_3\text{K} + \text{NaI}$, by heating the isomeric sulphites with ethyl iodide and recrystallizing from alcohol, the former by starting from acid sodium sulphite and potassium carbonate, and the latter by starting from acid potassium sulphite and sodium carbonate. This would indicate the existence of the isomeric salts $\text{K}-\text{SO}_2-\text{ONa}$ and $\text{Na}-\text{SO}_2-\text{OK}$. The work of Schwicker was repeated; the two salts which were supposed to be isomeric were heated with ethyl iodide, the time of heating and the strength of alcohol with which the product was extracted being varied. The product was analyzed for sodium and potassium. If the salts were isomeric there should be a difference in the ratio of sodium to potassium. The variation in the ratio Na:K in the different experiments is

ascribed to the difference in the conditions. The author found that recrystallization from alcohol, and the use of absolute alcohol increased the ratio, but in seven sets of experiments no values were found which would indicate the isomerism of the two salts.

Preparation and Properties of the So-Called "Nitrogen Iodide." By F. D. CHATTAWAY AND K. J. P. ORTON. *Am. Chem. J.*, 23, 363-368.—The most convenient method of preparing nitrogen iodide, $N_2H_3I_3$ is by bringing together ammonia solution and a cold solution of iodine monochloride, made by treating iodine with a mixture of hydrochloric and nitric acids to which ice has been added. The black precipitate of nitrogen iodide which at once separates may be filtered off with safety, and washed with dilute ammonia. The salt in crystalline form may be obtained by adding 15 cc. tenth-normal iodine monochloride to 100 cc. half-normal potassium hydroxide, and then adding, as rapidly as possible, 100 cc. of ammonia of sp. gr. 0.88. Glittering copper-colored crystals separate after a few moments; they are small, flattened, orthorhombic needles, dichroic, and have a specific gravity of 3.5. Pure nitrogen iodide is without action on litmus, does not give the reaction of iodine with chloroform; but in contact with water it is slowly decomposed. It may be dried in an atmosphere of ammonia over lime without decomposition, if light, to which it is extremely sensitive, be excluded. Nitrogen is slowly given off in diffused light from the compound suspended in water; in direct sunlight effervescence takes place. The partially decomposed substance is more explosive than the pure salt.

The Action of Reducing Agents upon Nitrogen Iodide. By F. D. CHATTAWAY AND H. P. STEVENS. *Am. Chem. J.*, 23, 369-376.—All ordinary reducing agents when brought in contact with nitrogen iodide suspended in water rapidly decompose it, and the quantity of reducing agent oxidized is found in every case to be exactly double the amount equivalent to the hydriodic acid produced. The reducing agents investigated were sodium sulphite, stannous chloride, hydrogen sulphide, sulphurous, arsenious, and antimonious acids. Nitrogen iodide prepared by different methods was used, and in each case the ratio of the amount of reducing agent oxidized, to that of the hydriodic acid produced was 2 : 1. The influence of the action of light upon the ratio was very marked.

On the Cause of the Evolution of Oxygen when Oxidizable Gases are Absorbed by Permanganic Acid. By H. N. MORSE AND H. G. BYERS. *Am. Chem. J.*, 23, 313-322.—It was suggested in a former paper (*this Rev.*, 5, 1) that the evolution of

oxygen which occurs when hydrogen or carbon monoxide is absorbed by acidified solutions of potassium permanganate is due to the action of the peroxide which is formed upon the excess of permanganic acid; and, further that the cause of this action of the oxide upon the acid may be a tendency on the part of the simpler peroxide molecules to polymerize to more complex ones at the expense of the acid. The experiments show, as regards carbon monoxide, that when this gas is absorbed by neutral solutions of potassium permanganate there is no evolution of oxygen; but when an acid is afterwards added, there is an evolution of oxygen similar in all respects to that which is observed when an equal volume of the gas is absorbed by an acidified solution of permanganate of the same concentration. If the reduction of the acid is due to the tendency to form complex molecules, then the evolution of oxygen should gradually diminish in rapidity as the polymerization increases. This has been shown to be the case, and further it has been shown that when the gas is absorbed in acid solution the period of rapid evolution does not terminate with the disappearance of the gas, as it should if the gas is the direct cause of the liberation of oxygen. Polymerization should also require, if the quantity of the gas absorbed remains constant, that the volume of oxygen liberated within a given time increases with increasing concentration of the permanganic acid. This has also been proved to be the case. The experiments were carried out in a tube holding 100 cc. which was agitated at a nearly constant temperature.

Permanganic Acid by Electrolysis. BY H. N. MORSE AND J. C. OLSEN. *Am. Chem. J.*, 23, 431-443.—In order to obtain manganese dioxide entirely free from potassium and other impurities the authors have devised an apparatus by which they can electrolyze potassium permanganate and obtain pure permanganic acid. The cell is composed of a beaker holding 1800 cc., placed in a galvanized iron tank through which water is kept passing. In the beaker is a porous cup of 250 cc. capacity in which is placed the negative electrode, made of a silver plate 50 mm. square, and a siphon through which the alkali may be drawn off. In the beaker the platinum positive electrode dips into a potassium permanganate solution containing about 40 grams of the salt in a liter. As a rule, the cells were found to have a low resistance in the beginning, amounting to from 5 to 10 ohms on the first day, rising to 25 or 30 ohms on the second day, and declining on the third and fourth days to 10 or 14 ohms. Different porous cells were tried, and it was found that the more porous, the smaller was the yield of acid. Using an ordinary battery cup the yield of acid has usually been from 87 to 92 per cent. of the theoretically possible amount. The concentration of

the acid was usually 9-10 per cent. Advantage was taken of the phenomenon of "electrical endosmose" for the concentration of the acid. With a current varying between 1 and 1.5 amperes, about 500 cc. of water passes out of the permanganate solution into the cup. By replacing the water thus drawn off by a 4 per cent. permanganate solution, 20 grams of the salt could be added each day. The electrical conductivity of an acid made from silver permanganate was measured and was found to agree more closely with the previous determinations of Lovén, than with those of Franke. Experiments were also made with other permanganates and with potassium dichromate which, it was found, can be electrolyzed in a similar manner.

A Contribution to the Knowledge of Tellurium. By F. D. CRANE. *Am. Chem. J.*, 23, 408.—The author has studied the different methods of precipitation, purification, and determination of tellurium. The crude material was obtained by extracting with hydrochloric acid the residue from the electrolytic refining of copper, and precipitating by means of acid sodium sulphite. It was found that the tellurium may be precipitated by means of metallic magnesium, the excess of this metal being converted to oxide by boiling, and subsequently dissolved by acetic acid. A convenient means of removing selenium from solution was found to be by the addition of freshly precipitated tellurium which throws out of solution all of the selenium; and it is stated that it will also throw out the more metallic elements which usually accompany tellurium, but no experiments are given to confirm this statement. It was shown that tellurium may be detected in very dilute solutions by acid sodium sulphite, and that ferrous sulphate is an equally sensitive reagent for selenium (see *this Rev.*, 6, 63) even in the presence of tellurium tetrachloride, but not in that of the dichloride. In order to avoid certain suspected sources of error, and to add nothing but hydrochloric acid as a reagent, a method of dissolving tellurium by electrolytic chlorine was devised. For the determination of tellurium a special form of apparatus was devised, whereby it might be filtered, washed, and dried in a current of hydrogen. Inasmuch as five of the six results given are high, and as tellurium has a remarkable tendency to absorb other substances when precipitated as metal—the precipitation having been made with acid sodium sulphite—the apparatus seems to the reviewer to be an unnecessary refinement. A yellow, crystalline form of tellurium dioxide was obtained by pouring a solution of tellurium tetrachloride in hydrochloric acid into boiling water.

On Chlorine Heptoxide. By ARTHUR MICHAEL AND WALLACE T. CONN. *Am. Chem. J.*, 23, 444-446.—Perchloric acid

was prepared by bringing together 25 grams of perchlorate and 100 grams of sulphuric acid (sp. gr. 1.839 at 15°) in a fractionating flask whose low lateral tube was connected with a second flask placed in a freezing-mixture. The mixture in the fractionating flask was heated under 10-20 mm. pressure in a paraffin bath. The reaction starts at 90°, and in an hour the temperature may be raised to 160°. The yield of slightly colored acid was 85-90 per cent. In contact with paper or wood it explodes, carbonizing the material; but it may be mixed with well-cooled alcohol without explosion. To prepare chlorine heptoxide, perchloric acid was brought slowly into contact with phosphorus pentoxide in a retort cooled below -10°. The retort is allowed to stand in the freezing-mixture for a day, then connected with a well-cooled receiver, and slowly warmed in a water-bath to 85°, when the oxide passes over. The freshly prepared substance may be redistilled under ordinary pressure. Chlorine heptoxide is a colorless, very volatile oil, which on standing turns yellowish green with liberation of a greenish gas. Brought into contact with a flame it explodes violently, but in contact with most substances it is remarkably stable. It may be poured on wood, paper, or similar organic substances with impunity. No reaction takes place with stick sulphur even after standing several days. It may likewise be poured on a cooled piece of phosphorus and remain several days without being attacked. A slow reaction takes place when it is brought into contact with benzene or iodine. With cold water it sinks to the bottom of the vessel, and slowly passes over into perchloric acid.

Some New Tellurium Compounds. BY VICTOR LENHER. *J. Am. Chem. Soc.*, 22, 136-141.—The chlortellurates and the bromtellurates of methyl, and ethylamine, aniline, pyridine, and quinoline, and the bromtellurate of trimethylamine were prepared by bringing together solutions of their salts in the corresponding halogen acids. The precipitate thus formed was purified by recrystallization from an acid solution. Since the author has not mentioned the preparations in which the salts were brought together, we are led to infer that only the 2 : 1 type of salt is capable of existence, but it seems more than probable that others of not so simple a type could have been isolated if the proportions of the two salts had been varied.

The Thermochemistry of Iron and Steel. BY E. D. CAMPBELL. *J. Am. Chem. Soc.*, 22, 205-206.—This paper is a withdrawal of a former article entitled "A Preliminary Thermochemical Study of Iron and Steel" (*this Rev.*, 3, 2). It is now shown that the solution of carbide of iron in double chloride of copper and ammonium is much accelerated when a small quantity of

free hydrochloric acid is present, but that the amount of free acid up to 4 per cent. does not appreciably influence the amount of heat evolved. The heat of solution of steel in slightly acid copper ammonium chloride is not changed more than 2 per cent. by variation in the heat or mechanical treatment of the sample.

Notes on Selenium and Tellurium. BY EDWARD KELLER. *J. Am. Chem. Soc.*, 22, 241-245.—In a previous paper (*this Rev.*, 4, 4) the author pointed out the difference of behavior of selenium and tellurium in the dioxide state, towards sulphur dioxide and ferrous sulphate in solutions of hydrochloric acid. These reactions have now been studied with the idea of using them for the quantitative separation of the two elements. It has been found that selenium is most readily precipitated by sulphur dioxide in strong hydrochloric acid solution while tellurium is not precipitated until the acidity of the solution is lowered to 80 per cent. Details are given for the application of this method to the quantitative determination of selenium and tellurium in copper. The author has found that in commercial work the criticism against determining tellurium in the metallic state on account of oxidation is unjustified.

Upon Bismuth Cobalticyanide. BY J. A. MATHEWS. *J. Am. Chem. Soc.*, 22, 274-277.—The quantitative precipitation of bismuth cobalticyanide was studied. The precipitate is crystalline, settles and filters well, and retains water below 165°. The author remarks that it has not been found useful in the analysis of pig-lead, as anticipated, "but it may still be found useful in the analysis of lead-bismuth alloys, 'fusible metals' such as Wood's alloy, etc." His results do not, however, in the least indicate its usefulness. Below a concentration of 0.0079 gram of bismuth per cubic centimeter the precipitation was under no conditions complete, and at ten times that dilution no precipitate was produced even on long standing.

METALLURGICAL CHEMISTRY.

H. O. HOFMAN, REVIEWER.

The Zellweger Roasting Kiln. BY J. ZELLWEGER. *Eng. Min. J.*, 69, 260-262.—This is a straight-line, single-hearth, mechanical reverberatory roasting furnace, 15 × 135 feet, with external fire places. The new feature is the rolling stirrer which turns over the ore and moves it slowly from feed to discharge. It consists of a heavy shaft carried by wheels, 6 feet in diameter, rolling on tracks in the depressed wheel-pits on either side of the hearth. The shaft carries a number of collars on which are mounted projecting V-shaped blades. While travelling from feed to dis-