meet this need,[1] and the results relating to gas purification thereby obtained are given below. All samples for analysis were taken under steady-state conditions.

Experimental

Oxygen Removal by Hot Copper.—In repeated attempts, no oxygen could be detected by this method in hydrogen passed at 110 cc./min. through a fused silica tube (diameter 7 cm., length, 22 cm.) filled with copper turnings at 600°. (The glass purification train, in which all joints were sealed, also contained sodium hydroxide pellets to remove hydrogen sulfide.)

With the copper at 230° and other conditions the same, the purified hydrogen contained 0.0017% oxygen by volume.

Oxygen removal in this way from nitrogen is facilitated by adding hydrogen so that the end product is water, not copper oxides.[2] Oxygen determinations were carried out on nitrogen thus purified in a pilot plant, the copper being near 400°. In one case, 0.0009% oxygen was found in the purified gas, which contained 25% by volume of hydrogen. In a second installation, the oxygen content was 0.0002% at the exit of the purifier with only 3% hydrogen present. Higher purity was not required in the pilot plant, and these oxygen contents probably are not valid lower limits.

Contamination in Transit.—The oxygen content of "oxygen-free" hydrogen being passed through about 10 feet of rubber tubing jumped to 0.0026%. At a distance from the purifier in a steel system believed to be tight, the second nitrogen mentioned above contained 0.0035% oxygen.

These data have been presented in the hope that some one will use the analytical method in a systematic study of oxygen removal, for it is capable of giving quantitative results on gases that could well appear oxygen-free by other quantitative methods.

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(1) Winslow and Liebhafsky, *Ind. Eng. Chem.*, *Anal. Ed.*, **18**, 565 (1946).

(2) So long as the metal is exposed, oxygen removal by copper at red heat can be very effective though repeated contact appears to be necessary for complete removal; *v.* Moser, "Die Reindarstellung von Gasen," Ferdinand Enke, Stuttgart, 1920, p. 79. When, owing to oxide formation, diffusion has become the rate-determining step oxygen removal necessarily suffers. There is no doubt that the addition of hydrogen *facilitates* the process. Hydrogen seems first to have been used for this purpose by Hulett, THIS JOURNAL, **27**, 1415 (1905) who, however, used essentially an oxyhydrogen torch to prepare nitrogen from air.

Though equilibrium favors the formation of ammonia under our experimental conditions, the rate of formation is almost certainly negligible since the homogeneous reaction is too slow and since copper at 400° does not adsorb appreciable nitrogen; *v.*, Frankenburger, *Z. Elektrochem.*, **39**, 276 (1933), and Dew and Taylor, *J. Phys. Chem.*, **31**, 281 (1927).

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RECEIVED JULY 19, 1946

The Reaction of Sodium 1-Dodecanesulfinate with Mercuric Chloride[1]

BY C. S. MARVEL, C. E. ADAMS AND RAYNER S. JOHNSON

Reports[2] indicate that German investigators thought their activated Buna-S recipes called Redox formulas were dependent in part on the presence of small amounts of sulfinic acid salts in their emulsifier, Mersolat. In seeking methods of identifying and determining alkylsulfinic acids in the presence of alkylsulfonic acids, we have investigated the reaction of sodium 1-dodecanesulfinate with mercuric chloride. The formation of arylmercury chlorides from arylsulfinates by heating with aqueous mercuric chloride is well known[3] although the analogous reaction of alkylsulfinates has apparently not been reported. We have found that sodium 1-dodecanesulfinate does react readily with mercuric chloride in boiling aqueous solution to give 1-dodecylmercury chloride in 49% yield. Thus, the reaction is demonstrated to be a useful one in the aliphatic sulfinic acid series.

Experimental

Sodium 1-Dodecanesulfinate.—This salt was prepared by the method of Allen.[4] The crude salt showed at least 74% sulfinate content on potentiometric titration in acetic acid with sodium nitrite.[5,6] An anhydrous sample of the salt was also prepared and analyzed both for the elements and for sulfinate content.

Anal.[7] Calcd. for $C_{12}H_{25}SO_2Na$: C, 56.24; H, 9.83; Na, 8.97. Found: C, 56.30; H, 9.99; Na (as Na_2SO_4), 8.88.

The anhydrous material showed over 94% sulfinate by potentiometric titration.[6]

1-Dodecylmercury Chloride.—Using a modification of the procedure of Whitmore, Hamilton and Thurman,[8] 1 g. (0.0037 mole) of pure dry sodium 1-dodecanesulfinate was added with mechanical stirring to a boiling solution of 50 cc. of water and 1 g. (0.0037 mole) of mercuric chloride. A flocculent white precipitate formed immediately and the stirring and heating were continued for two hours. The insoluble product was separated from the reaction mixture by filtration, dried and extracted three times with 20-cc. portions of boiling benzene. Evaporation of the solvent from the filtered benzene extracts gave 0.73 g. (0.0018 mole, 49.3% of the theoretical amount) of crude 1-dodecylmercury chloride, m. p. 106–111°. After crystallization twice from benzene and once from ethanol the product melted at 113–113.5°.

Anal.[7] Calcd. for $C_{12}H_{25}HgCl$: C, 35.55; H, 6.21. Found: C, 35.73; H, 6.51.

Meals[9] has reported the melting point of 1-dodecylmercury chloride as 114–114.5°.

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(1) This investigation was carried out under the sponsorship of the Office of Rubber Reserve, Reconstruction Finance Corporation, in connection with the Government Synthetic Rubber Program.

(2) Swann and Elias, Off. Pub. Bd., U. S. Dept. Commerce, Report PB 1636, 1945.

(3) Connor in Gilman's "Organic Chemistry," 2nd ed., Vol. 1, John Wiley and Sons, Inc., New York, N. Y., 1943, p. 918.

(4) Allen, *J. Org. Chem.*, **7**, 23 (1942).

(5) Koenigs, *Ber.*, **11**, 615 (1878).

(6) We are indebted to Dr. H. A. Laitinen, Mrs. Nancy Fritz and Mrs. Rita Leubner for these titrations.

(7) Microanalyses were done by Mr. H. S. Clark, Illinois State Geological Survey.

(8) Whitmore, Hamilton and Thurman, "Organic Syntheses," 2nd ed., Coll. Vol. 1, John Wiley and Sons, Inc., New York, 1941, p. 519.

(9) Meals, *J. Org. Chem.*, **9**, 211 (1944).

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