Calcd. for $C_{25}H_{24}O_{10}N_4$: C, 55.55; H, 4.48; N, 10.37. Found: C, 55.4; H, 4.60; N, 10.3.

Quinine salt of PCA: m.p. 207–209°. *Anal.* Calcd. for $C_{25}H_{31}O_5N_3$: C, 66.20; H, 6.89; N, 9.27. Found: C, 66.3; H, 6.90; N, 9.3.

Quinine salt of Me N-PCA: m.p. 159–160°. *Anal.* Calcd. for $C_{51}H_{62}O_{10}N_6$: C, 66.65; H, 6.80; N, 9.15. Found: C, 66.5; H, 6.76; N, 9.1.

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Reaction of Tantalum with Hydrogen Chloride, Hydrogen Bromide and Tantalum Pentachloride; Action of Hydrogen on Tantalum Pentachloride

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Hydrogen Chloride and Tantalum.—When hydrogen chloride gas is passed over tantalum metal at 410° and above, white tantalum pentachloride is formed and can be collected upon a cold-finger condenser. When the temperature is elevated to 600–750°, a thin film of metal, containing dissolved hydrogen, is formed on the heated tube walls. This metal is probably formed by the reduction or thermal decomposition of the pentachloride or some lower chloride, which may begin to form and immediately disproportionate (*cf.* Ruff and Thomas,[1,2] on $TaCl_3$) at the higher temperature. After the reaction has proceeded (600–750°) for a few hours, it is slowed markedly and an olive colored powder forms on the surface of the unreacted metal. Chemical properties and analyses indicate that the olive powder is tantalum dichloride as described by Ruff and Thomas.[1,2]

The tantalum dichloride powder (mixed with metal) was treated with 1 *M* potassium hydroxide and the residual tantalum was filtered away. When the solution was heated, hydrogen bubbled off and a dark flocculent precipitate (presumably $TaO_2 \cdot xH_2O$) formed. The tantalum was oxidized with nitric acid, the pentoxide thus formed was filtered off and chlorine was determined as silver chloride in the filtrate (*Anal.* Cl/Ta = 1.97).

The metallic tantalum films were fused with sodium carbonate and, during the fusion, considerable adsorbed hydrogen gas escaped and burned. The cooled melts were dissolved in water and the solutions were acidified with nitric acid. The weights of pentoxide thus formed indicated the metallic films were tantalum (99.9% Ta or higher.)

Hydrogen Bromide and Tantalum.—If hydrogen bromide is passed over metallic tantalum at 375°, tantalum pentabromide is formed and if the temperature is maintained at 550° or above, some tribromide begins to form, apparently mixed with a still lower bromide or perhaps the metal. *Anal.* Calcd. for $TaBr_3$: Ta, 43.0; Br, 57.0. Found: Ta, 44.7; Br, 55.5.

At higher temperatures (up to 800°) tantalum metal is deposited in the apparatus, probably as the result of reduction or thermal decomposition of the penta- or tribromide. This reaction is also slowed by the formation of a green powder (tantalum tribromide) on the surface of the unreacted metal.

Tantalum Pentachloride and Tantalum.—Schäfer and Pietruck[3] have shown that tantalum pentachloride is not reduced by the metal at 350–400° in an evacuated tube, an observation which was also made in the course of the present work. In the range 475–500°, however, a small amount of a green chloride is formed. If the unreacted pentachloride is sublimed away *in vacuo* and the green material is dissolved in water, one obtains a green solution in which the chlorine to tantalum ratio is 3.35 and the average oxidation state of the tantalum is 3.29. This latter value was obtained electrometrically by use of platinum and saturated calomel electrodes and a potassium nitrate–agar salt bridge. Ceric sulfate was the oxidant. When the inflection in the plot of e.m.f. *vs.* volume of titrant was noted, a brown flocculent precipitate (*cf.* $TaO_2 \cdot xH_2O$) was present in the solution. It was assumed that the tantalum was then in oxidation state (IV). These analyses would indicate the presence of some tantalum in a lower oxidation state (probably III) and possibly a compound such as Ta_3Cl_{10}.

Hydrogen and Tantalum Pentachloride.—Although niobium pentachloride is readily reduced with hydrogen to form the trichloride,[4] Schäfer and Pietruck[3] have shown that tantalum pentachloride is not affected at temperatures up to 400°. Studies in the course of the present work resulted in the same conclusions. However, if the reaction is carried out at 500°, some reduction of the pentachloride does occur and a film of the product, tantalum metal, containing dissolved hydrogen, is deposited on the heated walls of the reduction tube. No lower chlorides could be detected. This is in contrast to the reduction of the pentabromide by hydrogen. The formation of tantalum tribromide has been reported by Van Haagen[5] and by Young and Hastings.[6]

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O^3,O^4,N-Triacetyl-(–)-epinephrine

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The action of excess acetic anhydride on an aqueous solution of ephedrine in the presence of bicarbonate yields the N-monoacetyl derivative.[1] When essentially the same procedure is applied to (–)-epinephrine (Ia), there is obtained a quantitative yield of a crystalline, levorotatory triacetyl

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[1] L. H. Welsh, *J. Am. Pharm. Assoc.*, **36**, 373 (1947).