Determination of Thyroid Hormone in Serum by Activation Analysis

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Activation analysis was performed for measuring protein-bound iodine (PBI) in serum and trace amounts of iodine. The samples of human were passed through an ion-exchange column, and to which were added small amounts of ^{131}I for recovery check. Acid digestion was then carried out by adding chromium trioxide and sulphric acid; and distillation followed after adding phosphorous acid and hydrogen peroxide. The iodine evolved was trapped in potassium hydroxide. An aliquot of the distillate was placed in a polyethylene ampoule, and was irradiated in the pile at a thermal neutron flux of 4×10^{12} n/cm^2/sec. for either 15 or 30 minutes. The induced ^{128}I with added carriers was oxidized with laundry beach, reduced with $Na_2S_2O_5$, and then extracted with carbon tetrachloride after adding sodium nitrite. The carbon tetrachloride layer was washed with dil-H_2SO_4 and distilled water.

Then ^{128}I was reduced to iodide with $Na_2S_2O_5$, precipitated with silver nitrate, and assayed in a multi-channel pulse height analyzer and a Geiger-Müller Counter. This post-irradiation chemistry could be performed in approximately 30 minutes. The gamma ray spectrum and decay curve indicated the abscence of nuclides other than ^{128}I and ^{131}I. PBI was determined on sera obtained from patients with thyroid diseases and normal subjects, and the values obtained were nearly the same as those by the chemical method. The duplicate determinations indicated good reproducibility. By this procedure, we could measure 2.9×10^{-9} g of iodine.

Preparation of Radioactive-Iodide-labeled Compounds Related to Thyroxine

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Our new radioactive iodide labeling method of ^{135}I-Thyroxine, which had been reported at the 5th annual meeting of this association, was applied to other related compounds. Triiodothyronine, diiodotyrosine and monoiodotyrosine were labeled and good results were obtained.

The procedure was devided into the following three steps; oxidation of $Na^{125}I$ (^{131}I) to $^{125}I_2$ ($^{131}I_2$), exchange labeling reaction and separation of products.

Oxidation of $Na^{125}I$. To 1 ml of a solution of $Na^{125}I$ in a centrifuge tube, 15 μg of KI-carrier was added and after overlayering with 1 ml of ether, 1 drop of conc.HCl and 2 drops of 30% H_2O_2 were added. The tube was added. The tube was corked and allowed to stand for 2 hours with occational shaking. Then the lower layer was removed and the ether layer was washed two times with distilled water. More than 90% of initial radioactivity was extracted in this either as $^{125}I_2$.

Exchange reaction. To this ether solution of $^{125}I_2$, 1.2 ml of a pH 4-5 buffered (0.02N acetate buffer) 50% ethanol solution of 1 mg of substrate compound was added. The mixture became a single layer solution. After it was placed in a hot water bath at 40°C for 60-90 minutes, a stream of air was blowed over the surface. Ether was removed and the quantity of the solution decreased. The yield of the exchange reaction was approximately 70% in thyroxine and triiodothyronine, 95% diiodotyrosine and monoiodotyrosine.