hexane, toluene and xylene. The chloroform extract of the complex obeyed Beer's law in the range 6–204 ppm of zirconium. To evaluate optimum range and analytical accuracy, the Ringbom's curves were drawn by plotting the percentage transmittance as ordinate and logarithm of the concentration of zirconium as abscissa. The range derived from the maximum slope of the curve is 10–200 ppm of zirconium. The zirconium thiocyanate promethazine complex extracted into chloroform is more stable (14 hr) than zirconium thiocyanate complex (25 min). The molar absorptivity of the ternary complex (1.14×10^3 litre mole^{-1} cm^{-1}) is higher than that of the binary complex (3.2×10^2 litre mole^{-1} cm^{-1}).

The composition of the extractable zirconium-thiocyanate-promethazine complex was studied by Job's method of continuous variations with equimolar solutions. Since three components are involved in the formation and extraction of the complex, two series of experiments were carried out by keeping the concentrations of two components constant and varying the other. Results obtained indicate that the molar ratio of Zr to SCN^- is 1 : 3 and of Zr to promethazine is 1 : 1.

Various cations and anions were tested for the possible interference. The results showed that anions like chloride, fluoride, bromide, iodide, nitrate, sulphate, phosphate, acetate, oxalate and citrate do not interfere. The cations that do not interfere are those of alkali metals, alkaline earth metals and lanthanides. The following ions can be tolerated in concentrations hundred times higher than zirconium: Mn(II), Bi(III), Sn(II), Cr(III), Cr(VI), Pb(II), As(III), In(III), Te(IV), Au(III), Pt(IV), Pd(II), Sc(III), Th(IV), La(III). Ten times higher concentration can be tolerated for Co(II), Fe(III), U(VI), Ti(IV), Mo(VI), Ce(IV), Nb(V), Ta(V) and W(VI). Interference was reduced by pre-extracting W(VI), U(VI), Mo(VI), Ce(IV), and Fe(III) from 6 NHCl.

Analysis of zirconium steel

About 0.5 g of the steel sample was weighed into a 250 ml beaker. Hydrochloric acid (20 ml of 6 M) was then added. The solution was gently warmed and cooled. Perchloric acid (7 ml of 72%) was added and the mixture evaporated to copious fumes. The residue was dissolved with 20 ml of distilled water. Iron content of the mixture was then extracted with three 5 ml portions of diethylether[6]. The aqueous solution was boiled, cooled and diluted to 100 ml. A suitable aliquot of this solution was taken in a separatory funnel and the zirconium content was determined by the procedure outlined earlier. Analysis of steel samples gave a result of 0.128% and 0.280% Zr as against the certified values of 0.132% and 0.302%, respectively.

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ANNOUNCEMENT

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