WEAK GAMMA TRANSITION OF 0.89 MeV OF $^{207}$Pb

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The $^{207}$Bi isotope decays through electron capture to $^{207}$Pb which further de-exites to ground state with the emission of gamma rays. Many workers have studied the decay of $^{207}$Pb by using coincidence techniques. Besides the well-established gammas of $^{207}$Pb, controversial reports appear in literature about some of its weak gamma transitions.$^{1,2}$ Recent studies have reported$^3$ the weak gamma transitions of 1.440 MeV and 1.460 MeV. However, they failed to detect the 0.8978 MeV gamma transition as reported earlier$^{1,2}$. This communication describes the attempts to detect the weak gammas by analysing the single conversion electron spectrum with a surface barrier Si-detector.

We have used an old and chemically pure $^{207}$Bi source of about 1 μCi which was obtained from Amer sham (U.K.). An old source was preferred to ensure its purity. The conversion electron spectrum was measured with an Ortec surface barrier Si-detector. The system resolution was 18 keV (FWHM) at 0.978 MeV. The energies of the conversion lines were determined by the internal calibration of the Ortec multichannel analyser using the conversion electrons in the decay of $^{207}$Bi. As the source was very weak it was placed very close (about 1 cm) to the detectors. The method of least square fit was applied to draw the calibration curve.

Figure 1 shows the single conversion electron spectrum measured with surface barrier Si-detector. A weak K0.8978 conversion electron line can be observed. The 0.8978 MeV transition is a weakly converted gamma ray, as 99% of this transition goes to ground level with multipole mixture of M1 (0.8%) and E2 (0.14%). The upper limit of the intensity of this line is of the order of $10^{-2}$ compared to that of the K1.06 conversion electron line. The line intensity of the conversion electron was calculated by determining the area of the line and allowing subtraction of the beta background. The gamma energies used here were from Jardine$^4$.

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SPECTROPHOTOMETRIC STUDIES OF IRON (III) AND URANIUM (VI) COMPLEXES WITH 4,5,7-TRIHYDROXY-3-(p-METHOXYPHENYL) COUMARIN

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4,5,7-Trihydroxy-3-(p-methoxyphenyl) coumarin (scheme 1) has been reported by Dhar et al.$^4$ as a useful chromatographic spray reagent for the detection of metal ions in micro and macro quantities. Its colour reactions with V(V), Fe(II), Fe(III), Cu(II), Ce(III) and UO$_2$(II) are especially sensitive. In the present communication the use of the coumarin derivative for the spectrophotometric determination of Fe(III) and UO$_2$(II) ions is reported.

The reagent m.p. 308°C (Lit. 308-310°C) was synthesised$^5$. Metal ion solutions were prepared from AnalaR (BDH) grade FeCl$_3$ and UO$_2$(NO$_3$)$_2$·6H$_2$O salts. Stock solutions $1 \times 10^{-3}$ M of the reagent in pure methanol and of the metal ions in methanol (80%) were prepared. The optical density was measured using a Pye Unicam SP8-100 spectrophotometer. The pH was measured using Elico digital pH meter, model L1-120 employing sodium acetate-acetic acid buffer mixture. Mostly sodium, potassium, ammonium
The stoichiometry of the complexes in solution was determined employing $1 \times 10^{-3}$ M solutions. Both Fe$^{3+}$ and UO$_2^{2+}$ ions gave 1:2 (metal:reagent) complexes. The complex species in solution could therefore be represented as: $[\text{Fe}(C_9H_7NO_5)_2(CH_3OH)_2]^{+}$ and $[\text{UO}_2(C_9H_7NO_5)_2(CH_3OH)_2]$ and tentatively the structures of these species can be shown as in scheme 2.

\[ \text{WHERE } M = \text{Fe}^{3+}, m^+ = 1 \\
M = \text{UO}_2^{2+}, m^+ = 0 \]

To determine iron (III) the reagent compares favourably in terms of sensitivity with the known reagents, tartaric acid$^7$, 1,10-phenanthroline$^8$, phenyl-2-pyridylketoxime$^8$ and 2,2',2'-terpyridine$^8$. For uranium(VI) the reagent appears to be better and more sensitive than the already existing reagents aresazo III$^9$, dibenzoylethane$^9$, mercaaptocetic acid$^9$ and ammonium thiocyanate$^9$ cited in literature. The reagent is nearly as sensitive as acetylacetone$^7$, 8-hydroxyquinoline$^7$ and sodium diethylthiocarbaminate$^7$. The selectivity of the reagent is Fe$^{3+}$ and UO$_2^{2+}$ ions can be determined fairly well even in the presence of a number of other ions.

The procedure recommended for the determination using this reagent is: To 4.0–20 ppm of the metal ion in about 5 ml of methanolic solution (80%) the reagent is added in excess. The pH is adjusted to the required value after making up the volume with methanol (80%) and then the absorbance of the resulting solution measured after nearly 12 hr against the reagent blank.

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A NEW ONE STEP PREPARATION OF PICROPODOPHYLLONE FROM PODOPHYLLOTOXIN

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During our studies on the synthesis of analogues of podophyllotoxin (I), a lignan antitumor and cytotoxic spindle poison, we required picropodophyllone (II), a cis ketolactone, an isomer of the trans ketolactone, podophyllotoxone (III). The known method of preparing picropodophyllone (II) is a two-step process, the first by isomerizing podophyllotoxin I by bases to the picropodophyllinen (IV) and then oxidizing the hydroxy lactone (IV) to the ketolactone (II) by manganese dioxide. Though the yield in first step is very good, it was very low in the second step.

We had earlier used successfully, chloramine-T (trihydrate) in nonaqueous medium such as dimethyl sulphoxide and dioxan to oxidize some benzyl alcohols such as benzyl alcohol, benzoin, and furon and therefore we considered utilizing both the base properties and the oxidizing ability of chloramine-T, to convert the podophyllotoxin (I) to picropodophyllone (II). We report here the successful conversion of podophyllotoxin to picropodophyllone with a very much improved yield of the desired product in a single step, using DMSO as nonaqueous medium.

The product obtained in the above reaction was identical with the product obtained by the two-step synthesis in mp, COTLC in 5 different solvent systems and products showed no change in mixed mp.

In a typical experiment a mixture of podophyllotoxin (I) (0.5 g) and chloramine-T trihydrate (1 g) was kept stirred in dry DMSO (50 ml) at 60-65°C for 30 hr. The reaction mixture was diluted with water (Ca 100 ml) and then with 10% NaOH (Ca 50 ml) and stirred well and extracted into ether. The ether layer was successively washed with water and dried over sodium sulphate. The solid residue obtained after evaporation of ether was purified by column chromatography using chloroform: acetone (7:1) yield of the product: (0.27, 54%); mp 151-152°C (lit 5 mp 153-154°C).

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