A Single-line source for ^{193}Iridium Mössbauer Spectroscopy

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Summary An Os–Nb alloy suitable for repeated neutron irradiation to yield a ^{193}Ir Mössbauer source is described.

THE I ($\frac{1}{2} \leftrightarrow \frac{3}{2}$) 73 keV Mössbauer transition of ^{193}Ir has a sufficient change in nuclear size, $\delta R/R$, and quadrupole moment of the ground state for isomer shifts and quadrupole splittings to be greater than the natural line width and thus useful to the chemist.[1] The magnetic moments are however too small for magnetic splittings in reasonable external fields to be observed.

The 73 keV excited state is most simply fed as a result of β decay of ^{193}Os ($t_{\frac{1}{2}}$ 31·6 h) prepared by neutron irradiation of the stable ^{192}Os (abundance 41%),[2] but with a pure osmium source the hexagonal structure of Os metal gives a quadrupole splitting of 0·48 mm s^{-1} in the emitted Mössbauer γ-ray,[3] and thus a large apparent line width.

Osmium forms a cubic alloy with niobium up to an Os content of 6 atomic %.[4] Niobium has desirable properties for a host matrix, being mono-isotopic with a low neutron-capture cross section. The capture product is long-lived and also has a low capture cross section. Neutron-irradiated niobium gives no other photon emission in the region of the 73 keV Mössbauer peak and an Os–Nb alloy is thus suitable for repeated neutron irradiation to give a Mössbauer source for ^{193}Ir.

A niobium alloy of composition 5·0 atomic % ^{192}Os (9·8 weight %) was prepared by milling together 925 mg of pure Nb powder (Koch-Light laboratories) with 100 mg of Os powder enriched to 98·7% in ^{192}Os (Oak Ridge National Laboratory). The isotopic enrichment of the osmium is desirable because of the other strong activities produced by neutron irradiation of normal Os. The powder mixture was then compressed by cold compaction, and subsequently fused in an argon arc furnace to produce a small alloy button; this was annealed for 24 h at 1200 °C *in vacuo*. After annealing, filings were examined by X-ray powder photography to confirm that the alloy was cubic and single-phase; the cell parameter a was 3·29 Å (pure Nb, a = 3·30 Å). The annealed pellet was cut into two halves, one of which when irradiated in the A.W.R.E. Herald Reactor, Aldermaston, in a flux of 2×10^{13}n cm^{-2} s^{-2} for 4 days, gave sufficient activity (88% of the saturation value) for a week's use.

With a thin absorber of iridium metal (39 mg cm^{-2}), a single-line spectrum with a line width of 0·64 ± 0·1 mm s^{-1} was observed at liquid-helium temperature; this agrees well with the Heisenberg line width of 0·60 mm s^{-1}. Repeated measurements give a value of +0·84 ± 0·03 mm s^{-1} for the shift of the source relative to Ir metal, corresponding to an absorption at −0·84 mm s^{-1} for an Ir metal absorber. Further annealing had no effect on the line width.

A Ge(Li) detector allowed complete resolution from the K_α X-rays but not the K_β rays which overlap the 73 keV Mössbauer transition. Since the proportion of decays which populate the 73 keV level is fairly low (*ca.* 20%), and a large number of X-rays are emitted as a result of internal conversion,[1] the ratio of γ-ray to X-ray photons detected is also low, so that even compounds with relatively high recoil-free fraction give rather low absorptions. In order to avoid over-long counting times, since the half life of the source is so short, it is advisable to make the absorber rather thick.

Using these sources we have obtained well resolved quadrupole-split spectra even from organometallic compounds which might be expected to give very low recoil-free fractions; *e.g.* $[Ir(Ph_2PCH_2CH_2PPH_2)_2O_2][PF_6]$ gives a quadrupole splitting of 1·49 mm s^{-1}, a line width of 1·20 mm s^{-1}, and an absorption of 2% with a thickness of 173 mg cm^{-2} of natural iridium.

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