

irradiating spectroscopically pure gold targets in the INCHASS 2 MW Research Reactor for different periods. The irradiated sample was left to cool for an appropriate time and converted to soluble HAuCl_4 when suitable aliquots were subjected to gamma-ray measurement. A multichannel spectrometer was used with a $1.5'' \times 1''$ $\text{Na}(\text{Tl})$ crystal, the relative photopeak efficiency of which had been determined over the whole energy range concerned by measuring gamma-spectra of standard sources with well known decay schemes [2, 3].

From the compound spectrum of the mixed isotopes (Fig. 1) the photo peaks corresponding to the 158 and

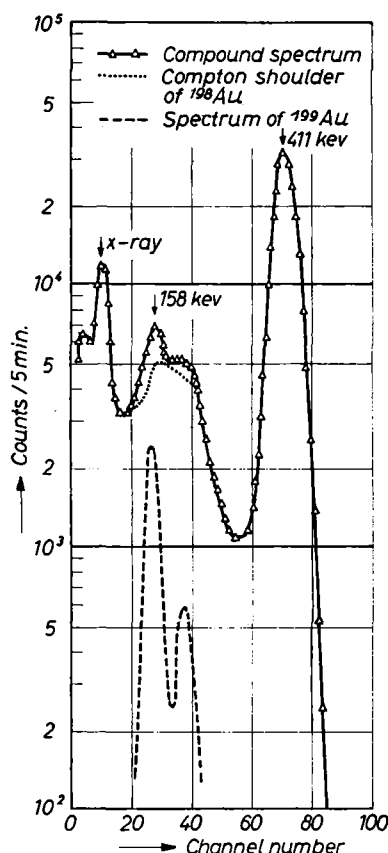


Fig. 1. Compound γ -ray spectrum of $^{198}\text{Au} + ^{199}\text{Au}$. The spectrum of ^{199}Au is obtained with good accuracy from the difference between this curve and the compton shoulder due to pure ^{198}Au , obtained by fitting the spectrum of this nuclide to the 411 keV peak in the figure.

208 KeV gamma rays of ^{199}Au are derived by subtracting the compton shoulder values due to the 411 KeV gamma-ray of ^{198}Au . (The scintillation spectrum of pure ^{198}Au was obtained from a gold sample irradiated for 3 hours at 10^8 n/cm^2 sec.) In table 1 our observed results are listed together with the corresponding figures calculated from the cross-section given above.

Table 1.

Irradiation time	Neutron Flux in n/cm^2 sec.	Ratio of ^{199}Au to ^{198}Au in percent		
		theoretical	experimental from 158 KeV γ	experimental from 208 KeV γ
5 h	1.1×10^{13}	0.159	0.165	0.177
31 h	1.3×10^{13}	1.12	1.12	1.13
48 h	1.3×10^{13}	1.97	1.87	1.97
48 h	1.3×10^{13}	1.97	1.97	2.04
48 h	1.5×10^{13}	2.23	2.27	2.28
50 h	1.1×10^{13}	1.72	1.66	1.79

The experimental data are calculated back to the end of the irradiation period. The thermal neutron flux was calculated from the ^{198}Au activity produced.

It may be concluded that in routinely produced radio-gold samples – as are used normally for medical purposes – the ^{199}Au to ^{198}Au ratio can be determined by means of the gamma spectrometric technique with an accuracy of 5% or better.

We have also proved that the value of 26000 barns for the cross-section for neutron capture in ^{198}Au is correct within a few percent.

Acknowledgement

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Stability of ^{32}P -labelled Pyrophosphate

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With 1 figure. (Received October 24, 1963)

The stability of pyrophosphate (^{32}P) under different conditions was studied. Radioactive pyrophosphate was prepared by igniting $\text{Na}_2\text{H}^{32}\text{PO}_4$ in a platinum crucible at 550° for 5 hours. More than 97% of the ^{32}P is then present as pyrophosphate. The radiochemical

purity of pyrophosphate decreased when amounts of labelled orthophosphate smaller than 15 micromoles were utilized for the preparation. Aqueous solutions containing 2.5 micromoles of pyrophosphate per ml were prepared with different pH-values and stored for

various periods of time. A pH 10.0 solution heated for one hour at 100°C in a sealed ampoule showed no evidence of decomposition.

All the analyses were carried out by paper partition chromatography with hydrochloric acid washed WHATMAN No 1 paper and isopropanol-isobutanol-water-ammonia (40-20-39-1) solvent.

The commonly used solvents, isopropanol-water-trichloroacetic acid-ammonia (75-25-5-2,5) and isopropanol-water-trichloroacetic acid (70-30-5), were also tested. These solvents appear to modify the composition of the mixture during the development of the chromatograms. In fact, it was found that after a two-dimensional run with the same solvent, the pyrophosphate (³²P) isolated in the first run was partially decomposed to orthophosphate (³²P) in the second one. Consequently the use of these solvents was abandoned. Active spots were located by radioautography and their chemical identity was established by adding in the same run a chromatographically pure inactive sample. The chemically detectable spots coincided with the radioactive ones.

The results are shown in Fig. 1. They indicate that it is possible to store alkaline aqueous solutions of pyro-

phosphate (³²P) at a pH = 8.5 or higher, without any significant change in its radiochemical purity, over normal periods of use.

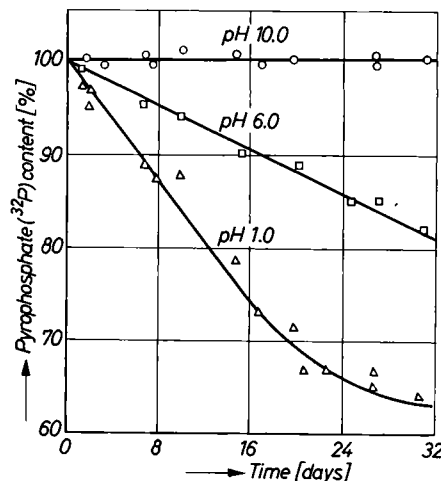


Fig. 1. Decomposition of pyrophosphate (³²P) aqueous solution at room temperature. The graph for pH = 8.5 was a horizontal straight line and coincided with that for pH = 10.0

KURZE MITTEILUNG

Effect of Crystal Structure on Annealing of ⁶⁴Cu Recoil Atoms in Copper Phthalocyanine

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The effect of crystal structure on annealing of recoil atoms has scarcely been investigated so far. Many publications appeared on hot-atom chemistry of copper phthalocyanine since HERR and GÖTTE [1] firstly treated this material with success. Among them, COOK [2] reported a difference in initial retentions between α - and β -crystals after the (n, γ) reaction, but he did not mention any difference in annealing behavior between these crystals. APERS and CAPRON [3] studied the annealing of ⁶⁴Cu atoms in β -copper phthalocyanine, but they did not compare it with that in the α -form. The present authors have found a striking effect of crystal structure on the annealing of ⁶⁴Cu recoil atoms produced by (n, γ) and (γ, n) reactions; that is, recoil atoms in irradiated β -crystals are very sensitive to annealing at least if expressed as a fraction of the total activity produced while those in α -crystals are relatively insensitive.

α -Copper phthalocyanine was purified by repeated precipitation of a reagent made by the Dainihon Ink Manufacturing Co. followed by repeated washing with sodium hydroxide solution and hydrochloric acid. β -Copper phthalocyanine was obtained by sublimation of the purified α -crystals. Both samples were tested by X-ray diffraction method and were proved to be pure.

Neutron irradiation was done in a nuclear reactor, JRR-1 (neutron flux: $5 \times 10^{11} n/cm^2/sec.$) for 5 minutes at dry ice temperature or at reactor temperature (20-50°C). γ -Rays were generated by an electron linear accelerator (maximum energy: 20 Mev, $\sim 10^6 r/min.$), and samples were bombarded for 5 minutes at dry ice temperature or at room temperature (20-60°C).

Table 1. Effect of crystal structure on initial retentions in α - and β -copper phthalocyanines

Nuclear reaction	Irradiation temperature	Retention (%)	
		α -form	β -form
(n, γ)	Reactor temp.	6.7 ± 0.2 (12)	39.6 ± 0.6 (12)
	Dry ice temp.	6.1 ± 0.1 (14)	23.5 ± 0.7 (14)
(γ, n)	Room temp.	6.6 ± 0.1 (7)	64.8 ± 0.4 (6)
	Dry ice temp.	6.2 ± 0.1 (14)	23.6 ± 0.8 (14)

Samples were irradiated for 5 minutes, and parentheses mean numbers of experiments.

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