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MODERN TRENDS IN ACTIVATION ANALYSIS

PRACTICAL ASPECTS OF NEUTRON ACTIVATION DETERMINATION OF THE PLATINUM METALS

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SUMMARY.

Triple gamma coincidence counting of ^{192}Ir allowed to determine Ir by instrumental neutron activation analysis down to 1 p.p.b. in ultra-basic rocks and down to ca. 20 p.p.b. in some high-furnace slags; the limiting factor for the latter matrix was the presence of ^{124}Sb. Radiochemical neutron activation analysis of the U.S.G.S. standard rocks revealed that the Ir contents are up to three orders of magnitude lower than previously reported, except for the ultra-basic rocks. The factor of merit of several scintillation and semiconductor gamma-ray detectors was determined for the neutron activation determination of Pd, Pt and Os. In the case of radiochemically pure sources, a NaI(Tl) wafer was preferred; in the presence of high-energy gamma emitters, a Ge(Li) low-energy photon detector was superior.

INTRODUCTION.

Neutron activation analysis can be useful in the field of the platinum metals, not only because of its great sensitivity for some of these elements (e.g. Ir, Pd and Os), but also for its being an intrinsically accurate technique, which allows it to be used as a standardization method. A positive interest for application of NAA

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in this field appears from some recent conferences, such as "Activation analysis of noble metals" (Tashkent, Uz.SSR, 1970), "Symposium on nuclear analysis techniques in the production and industrial use of noble metals" (Brussels, November 1971), and "Symposium on the analytical chemistry of the platinum group metals" (Johannesburg, February 1972). The National Institute for Metallurgy (Johannesburg, South Africa) is now distributing a "platinum ore standard" to laboratories all over the world (1), and the Bureau Eurisotop (Brussels) is sponsoring the development of a copper matrix containing standardized platinum metal contents.

A number of general statements on the neutron activation determination of platinum metals have already been made elsewhere by one of the authors (2,3). Some practical aspects will be discussed below.

NEUTRON ACTIVATION DETERMINATION OF IRIDIUM.

Although the reaction $^{193}Ir(n,\gamma)^{194}Ir$ is very sensitive, most activation analysts have utilized the reaction $^{191}Ir(n,\gamma)^{192}Ir$, apparently because of the longer half-life (74.2 d) of the latter radionuclide, so that it can be measured after a long cooling time. ^{192}Ir is usually counted after radiochemical separations, and very low amounts can then be determined ($<10^{-12}$g), even when using a Ge(Li) detector.

From the decay scheme of ^{192}Ir appears that various coincidence measurements are feasible, which offer an alternative for the specific counting of radiochemically separated, but not radiochemically pure ^{192}Ir (4,5).

Gamma-gamma coincidence counting has been applied for the instrumental neutron activation determination of Ir in meteorites, where the concentrations are sufficiently high (p.p.m. range)(6-10). Similar results can, however, be obtained by Ge(Li) gamma-ray spectrometry (9,11,12).

Instrumental neutron activation determination of Ir in ultrabasic rocks and some high-furnace slags.

Triple gamma coincidence counting via the 308.43 - 295.6 - 316.5 keV gamma-ray cascade allows the instrumental neutron activation determination of Ir down to p.p.b. concentrations (5,10). Our set-up is shown in Fig.1. It consisted of three 7.62 cm x 7.62 cm NaI(Tl) detectors, coupled to EMI 9531A or 9578 photomultipliers, horizontally mounted at 120° angles (which discriminates against $\beta^+\gamma$ emitters such as ^{58}Co), and shielded with lead to avoid cross-talk. The detectors were usually placed at 5 cm from the radioactive source, or farther if the gross activity was too high.

Each chain consisted of the following electronic circuitry : a preamplifier NE 5281, an amplifier NE 4603, a pulse height analyzer NE 4602, and a timing discriminator NE 4616, connected to a fast coincidence module NE 4619 (resolving time τ from 2 to 100 ns). The output of the coincidence module was connected to the delayed coincidence input of an Intertechnique SA-40 multichannel analyzer, via a variable delay line AD-YU, 552 CI (0-1.5 μs) and a pulse-shaping circuit. This set-up allowed the triple coincident gamma spectrum to be observed (Fig.2).

The amplifier was used in the double delay line differentiation mode, and the timing discriminator in the zero cross-over mode. With the circuitry described, a resolving time of 40 ns allowed to detect all true coincidences "seen" by the detectors. With the counting chains gated on the 308.43 - 295.6 - 316.5 keV gamma-ray cascade, the system is specific to ^{192}Ir.

There will, of course, be a smooth background contribution in the 300-keV region, from Compton scattering of higher-energy triple coincident gamma-rays (e.g. from ^{124}Sb or ^{110m}Ag), and possibly some double coincident events (e.g. from ^{124}Sb, ^{110m}Ag, ^{46}Sc or ^{60}Co), combined with backscattering into the third detector. This does not interfere with the ^{192}Ir complex 0.31 MeV peak area determination by Covell's method (13).

The contribution of "semi-random" (10) and random triple coincidences was explicitly checked for a particular type of samples, by observing the coincident count rate after maladjustment of the timing discriminator delay line in chain I, or in chains I and II, respectively (cfr Fig.1). By keeping single detector integral pulse rates below 10^4c.p.s. (by chosing appropriate sample size, neutron dose and/or sample-detector distance), it was found that problems associated with (semi)random coincidences, and also pulse pile-up and related problems, such as multichannel analyzer dead-time problems (even when operated in the delayed coincidence mode) could be kept under control. Nevertheless, an addition method of analysis was used throughout (14), resulting in very similar counting conditions (same gross activities) for samples and standards.

The above method was applied to some ultrabasic rocks and some types of high-furnace slags, using a sample size of 1 gram. The rocks were irradiated for 32 hours at a flux of 5.10^{11} n.cm^{-2}.s^{-1}, and counted for ca. 24 hours, 20 to 30 days after irradiation. The high-furnace slags were irradiated for 6 hours only, in order to keep the gross activity within the limits discussed above, and also counted after ca. 1 month cooling.

The rocks were all peridotites, similar in composition to the well-known U.S.G.S. standard rock PCC-1 (15). The slags had the following typical composition : 26.5-29% SiO_2; 20-24% Fe; 11.5-18% CaO; 7.5-9% Zn; 0.8-1.8% Pb; 0.7-0.9% BaO; 0.5% Cu; 0.1-0.4% Sn; 0.15-0.45% Cr; 0.05-0.2% Co; 0.01-0.1% Sb, and the gamma spectrum showed the following major activities after 1 month cooling : ^{46}Sc, ^{60}Co, ^{59}Fe, ^{51}Cr, ^{124}Sb, ^{182}Ta, ^{131}Ba, ^{75}Se, ^{110m}Ag.

Results are given in Table I, column 2. Column 3 shows the Ir content found for the very same samples, but after radiochemical separation according to a procedure described elsewhere (4), followed by Ge(Li) gamma spectrometry. The agreement is quite satisfactory. Sample PCC-1 has been analyzed for Ir by 6 different laboratories (cfr. Table II) with an average value of about 5.2 p.p.b., implying that the above instrumental technique gives accurate results.

In the experimental conditions described above, the following approximate detection limits can be obtained : for ultrabasic rocks ca. 1 ng Ir/g, for slags ca. 20 ng Ir/g (assuming an Sb content of 0.1%). For the slags investigated here, Compton scattering from triple coincident ^{124}Sb gamma rays appears to be the limiting factor (cfr. Fig.2), implying that the instrumental technique is not selective enough, if the Sb content exceeds the Ir content by a factor of 500 or more. For the peridotite rocks, the background source has not unequivocally been identified, but does not hamper the Ir determination.

In spite of the long counting times, triple gamma coincidence counting is still fast in terms of man-work hours. It is ideally applicable to peridotite rocks, and may be useful in the case of "rich" high-furnace slags.

Radiochemical neutron activation analysis of Ir in silicate rocks.

Earlier data on U.S.G.S. standard rocks suggest that Ir contents in silicate rocks are typically in the p.p.b. range. More recent results, however, tend to be 1 or 2 orders of magnitude lower, except for the ultrabasic rocks (peridotite PCC-1, dunite DTS-1), where the data remain relatively consistent. See Table II, which is adapted from a compilation by Flanagan (16).

The radiochemical separation method followed in this work is similar to that described earlier (4). In order to obtain higher sensitivities, the samples were, however, subjected to a higher neutron dose (3.10^{19} n.cm^{-2}). The chemical procedure was consequently somewhat modified, to avoid the necessity of evaporating highly active solutions.

Procedure.

After at least 1 week cooling, the quartz ampoules were cleaned with nitric acid, and scouring powder, and finally rinsed with water. The vials were opened, the rock powder (0.5 g) transferred to a new nickel crucible containing 10 mg Ir sponge, and covered with 10 g sodium peroxide. After fusion, the melt was cooled, and disintegrated with water; 30 ml HCl 12 N was carefully added and the total volume brought to 100 ml with water. The solution was heated in the presence of 1 ml hydrogen peroxide in order to oxidize Ir to the valency IV. After cooling, the solution was transferred to a small Dowex-1X8 column, pretreated with 50 ml HCl 0.5 N containing a few drops of H_2O_2. A dark brown band of Ir(IV) formed at the top of the column. The resin was washed with HCl 0.5 N (typically 2000 ml), and then with water (typically 250 ml), until no further activities were eluted. The eluates were discarded to the radioactive waste. The resin was extruded and boiled in the presence of 35 ml HCl 8 N + 1 g hydrazinhydrochloride, until the brown Ir(IV) chlorocomplex had disappeared from the resin. The resin was collected in a filter paper, and subjected to a second batch extraction. The combined filtrates were evaporated after adding 10 mg Si carrier. The residue was taken up in 30 ml cold water, and SiO_2 immediately filtered, washed with 1% HCl and discarded (contains ^{233}Pa, ^{182}Ta, ^{95}Zr-^{95}Nb ... activities). The filtrate was evaporated to less than 5 ml, and transferred to a standard counting vial for counting with a Ge(Li) detector.

In several cases, the decontamination was not yet sufficient at this stage, requiring a second ion-exchange separation and/or SiO_2 scavenging. The chemical yield was determined by reactivation of an aliquot of the final solution, spotted on very pure titania. Yields were typically ca. 60%.

Results and Discussion.

The elutions are somewhat time-consuming, and less elegant than the fire-assay separation described by Greenland et al. (5). Several samples can, however, be treated simultaneously, without constant attendance being required and without special equipment. Even after two anion-exchange separations, the final Ir fractions were not radiochemically pure, the main contaminants usually being $^{233}Pa(Th)$, ^{46}Sc, ^{110m}Ag, $^{131}I(U)$, ^{95}Zr-^{95}Nb, ^{181}Hf, ^{124}Sb, ^{60}Co, $^{182+183}Ta$, ^{59}Fe and some others. Ge(Li) gamma-ray spectrometry or coincidence counting is therefore required; in the former case, the counting time was typically 12 to 15 hours. The ^{233}Pa contamination is particularly annoying for the ^{192}Ir counting, because of its photo peaks at 300.2 and 311.9 keV, but can considerably be reduced by a SiO_2 scavenging.

Results are summarized in the last line of Table II. The data for DTS-1 and PCC-1 agree well with those from all other authors. For the other silicate rocks, our results tend to confirm the low concentrations reported by Greenland et al. (5) and Anders et al. (19). Our data for BCR-1 are not very reproducible (sample heterogeneity?); this is also apparent from the results of Anders et al. (19). The high values of ref.(9) have already been discussed by Greenland et al.(5) : a possible cause is the long half-life of ^{192}Ir, which can give rise to radioactive contamination problems, as indicated by Kimberlin et al.(24), for instance. The discrepancies might also be caused by extreme sample heterogeneity with respect to Ir at the sub-p.p.b. level, but the present limited amount of data does not permit such a conclusion to be drawn.

In the present work, special attention was paid to the contamination problem. Samples were taken from freshly opened U.S.G.S. bottles, and immediately sealed in carefully cleaned quartz tubes. Only new crucibles, glass ware, ion-exchange columns, etc. were used in the chemical treatment following irradiation. Moreover, a number of unirradiated rock samples were carried through the radiochemical procedure, and the separated Ir fractions counted for 65 hours : no ^{192}Ir could, however, be detected, implying that no radioactive ^{192}Ir contamination had occurred. For each irradiation series, the Ir standards (50 ng Ir) were processed only after the samples had been completely finished, to minimize the risk of contamination.

RECOMMENDED RADIATION DETECTORS FOR NEUTRON IRRADIATED PALLADIUM, PLATINUM AND OSMIUM.

Taking into account natural isotopic abundances, activation cross sections, decay schemes and half-lives, it appears (3) that the analytically most interesting radionuclides, formed upon neutron activation of Pd, Pt and Os are 13.47 h ^{109}Pd, 18 h ^{197}Pt and 15.0 d ^{191}Os. It is often advisable to chose gamma counting, because of its greater specificity as compared to beta counting, thus requiring less radiochemical purity of the separated fractions. The fact that low-energy photons or X-rays dominate the gamma spectrum of neutron-irradiated Pd, Pt and Os, makes it quite worthwile to consider which detector is best suited.

Instead of using the "minimal-Q criterion" as a figure of merit for the detector (25), it was decided, for reasons summarized elsewhere (14), to rely upon criteria described by Currie (26). The working expression of "limit for quantitative determination " L_Q ("paired observations") was considered the most appropriate for the present purpose :

$$L_Q = 50\left[1 + (1 + 0.08\,\mu_B)^{\frac{1}{2}}\right] \text{(counts)} \qquad (1)$$

where L_Q = minimal number of counts due to the radioactive source which will have a fractional standard deviation of 10% (due to counting statistics) when recorded by means of a counter with a background of μ_B counts in the same time.

Although this formula was derived for integral countings, it will be applied here to a photo peak upon a continuous background (natrral background or some interfering activity, here 4.7 µCi ^{22}Na).

The statistics of decision, detection and determination (26) apply directly to the observations (activity) rather than to the underlying quantity. Statistical conclusions drawn in terms of the net signal may be extended to the related physical quantity by means of a calibration factor, e.g. $m_Q = f\,L_Q$, where f is expressed in µg/count. The factor f was experimentally determined by irradiating a known amount of the element.

The following detectors were considered : a 7.62 cm x 7.62 cm NaI(Tl) scintillator (window 0.5 mm stainless steel), a coaxial Ge(Li) detector (efficiency 7.8% relative to a 7.62 cm x 7.62 cm NaI(Tl) crystal for ^{60}Co; FWHM 2.2 keV at 1332.5 keV; Al window), a 2.5 cm x 0.2 cm NaI(Tl)"wafer" (window 0.2 mm Be), and a planar 10 mm x 5 mm Ge(Li) low-energy photon detector ("LEPD", window 0.13 mm Be), described in detail elsewhere (27).

The following irradiations were performed : 35 µg Pd, 340 µg Pt and 160 µg $(NH_4)_2OsCl_6$, all "specpure" quality (Johnson-Matthey), for 5 hours at $5.10^{11} n.cm^{-2}.s^{-1}$. The radioactive sources were counted as close to the detectors as physically possible, ca. 12 hours after irradiation for Pd and Pt, and 3 weeks after irradiation for Os.

Alternatively, the samples were counted in the immediate presence of a 4.7 µCi ^{22}Na source, at such a distance from the detector that the multichannel analyzer fractional dead time did not exceed 10%. The NaI(Tl) detectors were coupled to a 400-channel analyzer Intertechnique SA-40, and the Ge(Li) detectors to a 4000-channel analyzer Didac.

Detailed results for Pd are shown in Table III. In the case of radiochemically pure fractions, counting of the Ag X-rays with a thin NaI(Tl) crystal, having a Be window, is clearly the best choice: the efficiency is high, and the background low, resulting in $m_Q = 7 ng$; this is a factor of 60 better than a conventional Ge(Li) detector, but also a factor of 25 more favourable than the 7.62 cm x 7.62 cm NaI(Tl) crystal. For the latter two detectors the ca. 22-keV Ag X-rays cannot penetrate the window. The next best choice is the LEPD.

The limit for quantitative determination becomes considerably worse when counting the sample in the presence of high-energy gamma emitters, such as ^{22}Na. (Table III, last column). The deterioration is less pronounced with the LEPD, and the data show that the latter is superior for instrumental activation analysis, or after incomplete radiochemical separations (not radiochemically pure sources). This effect would have been even more pronounced with a stronger interfering source.

Similar conclusions can be drawn for ^{197}Pt and ^{191}Os (Table IV). Another obvious advantage of the LEPD is its superior resolution, as compared to all the other detectors considered (Fig.3, 4 and 5). Fig.3 shows a complication in LEPD spectra of very low-energy gamma emitters, namely Ge X-ray escape peaks. Fig.3 gives also evidence for fluorescence X-rays in the Pd sample, caused by Ag K_{β} X-rays (from ^{109}Pd-^{109m}Ag), as could be confirmed by diluting the sample. This implies that samples and standards should have the same physical and chemical form, when counting X-rays following β^{-} decay, in order to avoid systematic errors.

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TABLE I.

Iridium contents in peridotite rocks and high-furnace slags (in p.p.b.)

Sample	INAA (^{192}Ir, γγγ)	RNAA* (^{192}Ir)
Peridotite rocks		
PCC-1 (a)	6.7	7.1
SAL-91 (b)	3.8	3.6
SAL-93 (b)	5.0	5.0
High-furnace slags		
1	ND; ND; ND	0.4; 0.6; 0.5
2	ND; ND; ND	0.9; 0.8
3	ND; ND	0.6; 0.1
4	137; 140; 153; 140; 134	–; 132; 163; –; –.
DB	26; 28; 32	29; 24; –

* Radiochemical separation performed on same sample of γγγ-coincidence counting (instrumental analysis).

(a) Sample from Guano Valley, Lake County, Oregon; U.S.G.S. standard rock; for results by other authors, see Table II.

(b) Samples from Island of Sal (Cape Verde Archipelago), taken by P.De Paepe, Geological Institute, University of Ghent, Belgium.

ND : not detected

– : not determined

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TABLE II.

Iridium in U.S.G.S. standard rocks (in p.p.b.).

Granite G-2	Granodiorite GSP-1	Andesite AGV-1	Basalt BCR-1	Dunite DTS-1	Peridotite PCC-1	Ref.
–	–	–	<0.1	–	–	17
17.7	1.1	0.7	1.1	0.5	6.6	7
0.12(2)	–	–	–	–	–	18
–	–	–	0.012;4.0; ≤0.06;≤0.05; 0.007	–	–	19
0.07	≤0.16	≤0.20	≤0.12	0.56	5.7	20
–	–	–	<0.1	–	–	21
ND	6; 3; 2; 3	5; 3; 5; 9	3; 1; ND; ND	0.6;0.6; 0.6;0.3	2; 8; 4; 5	22
0.005; 0.009; 0.028; 0.039	0.005; 0.009; 0.012	0.003; 0.004; 0.006; 0.029	0.002; 0.004; 0.005; 0.006	0.34 to 4.5(14) (Ø:1.12)	2.6; 2.8; 3.6; 3.1	5
–	–	–	–	–	5.6; 5.4	23
–	–	–	–	0.5;1.5	6.6; 6.7	4
–	–	–	–	–	6.7	(a)
0.003; 0.007; 0.007	0.021	0.007; 0.007	0.003; 0.20	0.65	4.2; 7.1	(b)

All results obtained by neutron activation analysis, except ref.(17): spark source mass spectrometry.

Between brackets: number of analyses.

ND = not detected.

(a) : this work, instrumental determination via $\gamma\gamma\gamma$-coincidence counting, cf. Table I.

(b) : this work, after radiochemical separation.

TABLE III.

Counting of ^{109}Pd with various gamma detectors (*).

Detector	Radiation measured	Pd alone on top of detector			Pd + 4.7 μCi ^{22}Na so that FDT $\leq$ 10%			$\frac{m'_Q}{m_Q}$
		S	B	m_Q(μg)	S'	B'	m'_Q(μg)	
Ge(Li) LEPD	Ag K_α X	38600	7	0.10	37500	4200	0.90	8.9
	88.0 keV	4200	14	1.02	4200	2200	5.96	5.8
Ge(Li) coax.	88.0 keV	10000	18	0.45	650	9000	75.0	167
NaI(Tl) wafer	Ag X	612000	16	0.007	25800	10800	2.06	286
	88.0 keV	71700	9	0.056	1960	18300	35.4	632
NaI(Tl) 7.6x7.6cm	88.0 keV	45600	200	0.20	400	22700	190	970

(*) 35 μg Pd, irradiated for 5 hours at 5.10^{11}n.cm^{-2}.s^{-1}; counted for 5 min., 12 hours after irradiation.

S = net number of counts per 5 min. under peak specified in column 2

B = number of background counts per 5 min. in same energy range

m_Q= micrograms Pd which can be determined quantitatively ($\pm$ 10%)

TABLE IV.

Counting of ^{197}Pt and ^{191}Os with various gamma detectors (*)

Detector	^{197}Pt				^{191}Os			
	Radiation measured	m_Q (µg)	m'_Q (µg)	$\frac{m'_Q}{m_Q}$	Radiation measured	m_Q (µg)	m'_Q (µg)	$\frac{m'_Q}{m_Q}$
Ge(Li) LEPD	77.7 keV (^{197}Pt)	4.6	27.4	6.0	Ir K_{α_1} 129.4 keV	2.1 5.0	14.4 33.2	6.9 6.6
Ge(Li) coax.	77.7 keV +Ir,Pt,Au, Hg K_β X-rays	2.1	257	122	Ir,Re K_α 129.4 keV	0.94 0.91	149 113	158 124
NaI(Tl) wafer	77.7 keV +Ir,Pt,Au, Hg X-rays	0.15	57.5	383	Ir,Re X 129.4 keV	0.086 0.30	25.3 144	294 480
NaI(Tl) 7.6x7.6cm	77.7 keV +Ir,Pt,Au, Hg X-rays	0.49	422	861	Ir,Re X 129.4 keV	0.41 0.63	226 635	551 1079

(*) 340 µg Pt, and 160 µg $(NH_4)_2OsCl_6$, irradiated for 5 hours at $5.10^{11} n.cm^{-2}.s^{-1}$, counted for 5 min., 12 hours (Pt) or 3 weeks (Os) after irradiation.

m_Q, m'_Q : cf Table III.

LIST OF FIGURES.

Fig.1 Set-up for triple gamma coincidence counting of ^{192}Ir.
HV = high voltage supply; Pb = lead shielding;
NaI = NaI(Tl) detector; PM = photomultiplier;
PA = preamplifier; AMP = amplifier (double delay line clipping);
PHA = pulse height analyzer; TD = timing discriminator (cross-over pick-off); COINC = fast coincidence module;
SCAL = scaler; TIM = timer; MCA = multichannel analyzer.

Fig.2 Triple gamma coincident spectrum of 1 g high-furnace slag, containing 28 p.p.b. Ir, irradiated for 6 hours at 5.10^{11} n. $cm^{-2}.s^{-1}$ and counted during 20 hours, 20 days after irradiation. Channels II and III gated at 0.3 MeV (Fig.1), channel I gated from 0.1-1 MeV : this allows the ^{124}Sb interference to be detected.

Fig.3 Gamma spectrum of ^{109}Pd-^{109m}Ag, taken with the low-energy photon detector (LEPD). 35 μg Pd, irradiated for 5 hours at $5.10^{11} n.cm^{-2}s^{-1}$, measured for 5 minutes against the Be window, 1 day after irradiation.

Fig.4 Gamma spectrum taken with the LEPD. 340 μg Pt, irradiated and measured in the same conditions as Fig.3.
(^{197}Pt, ^{191}Pt, ^{193m}Pt, ^{195m}Pt, ^{199}Au).

Fig.5 Gamma spectrum taken with the LEPD. 160 μg $(NH_4)_2OsCl_6$ irradiated and measured in the same conditions as Fig.3, except that the cooling time was 3 weeks.
(^{191}Os, ^{185}Os, some ^{193}Os).

sults.

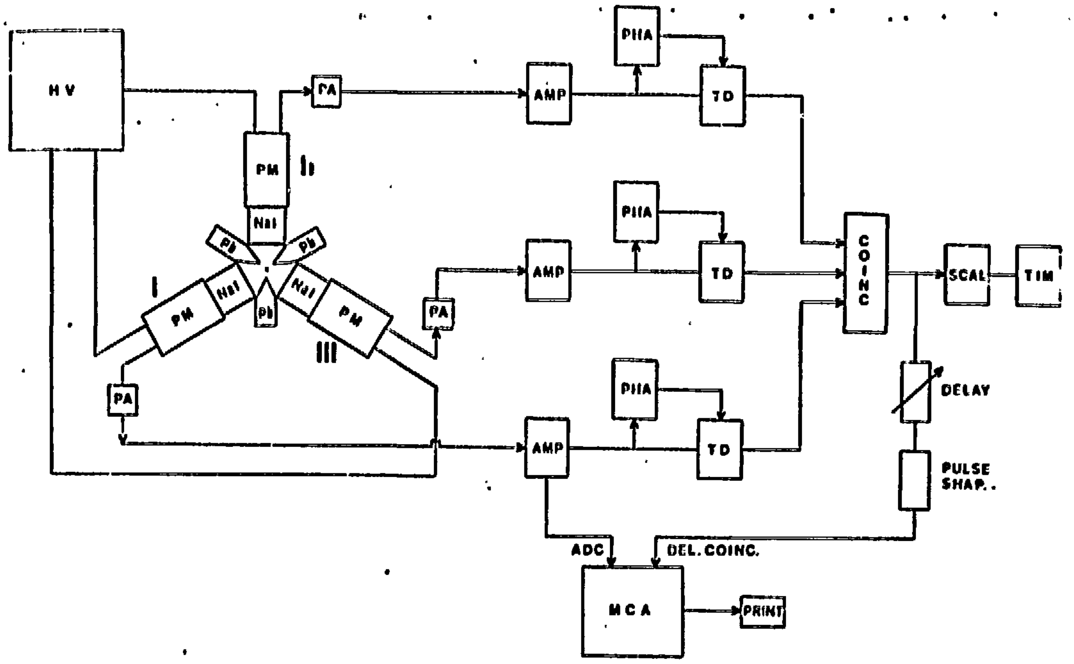

Fig 1.

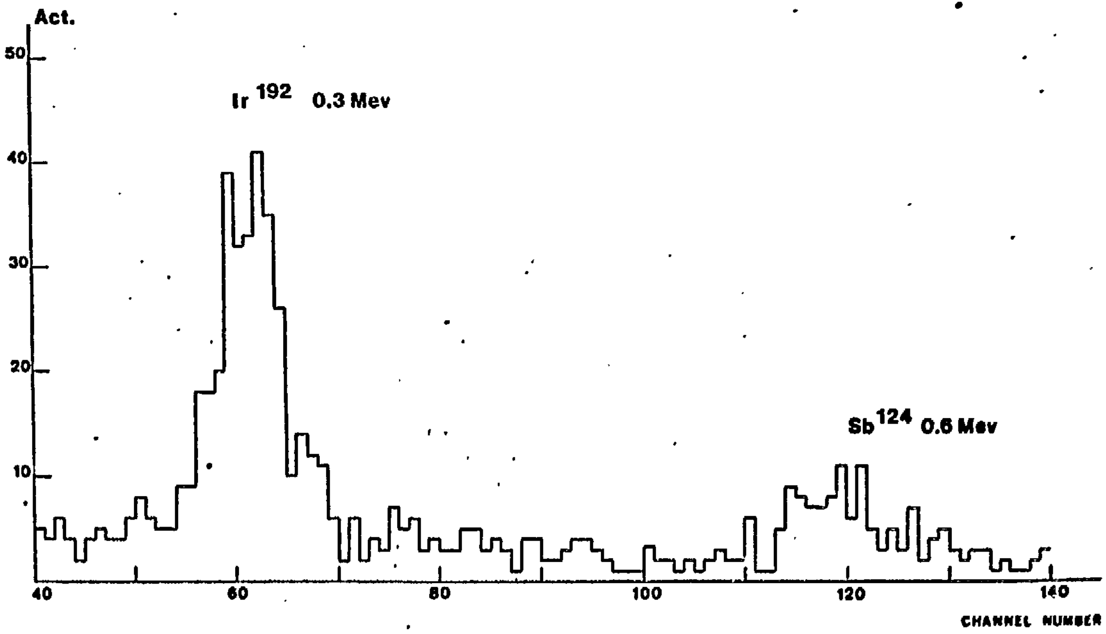

Fig 2.

with 10 g sodium peroxide. After fusion, the melt was cooled, and disintegrated with water; 30 ml HCl 12 N was carefully added and

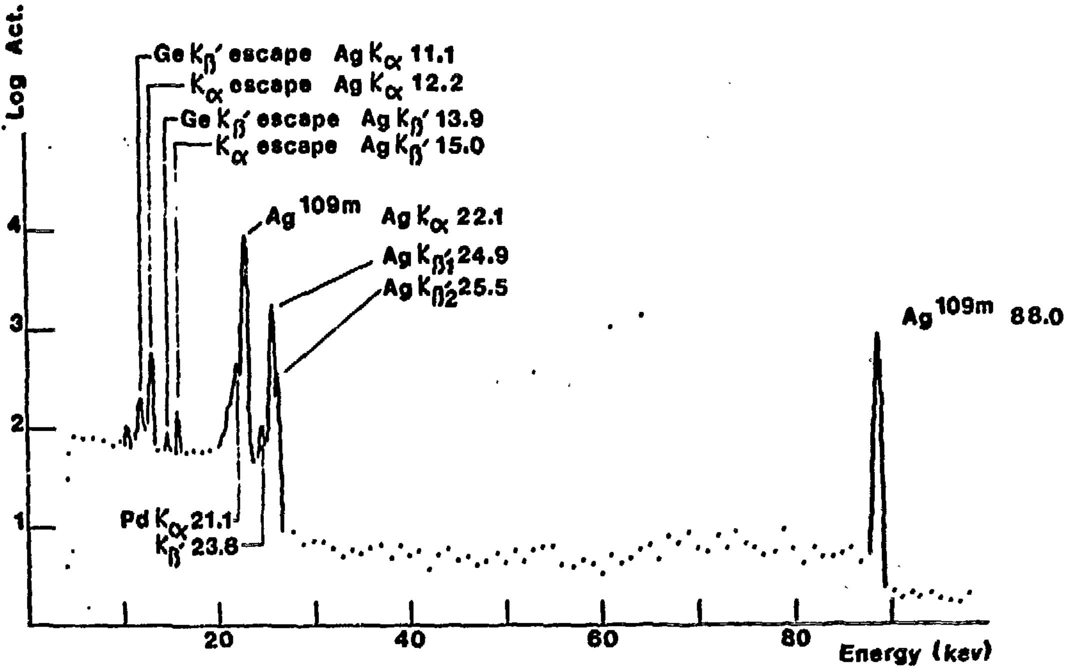

Fig. 3.

M 30

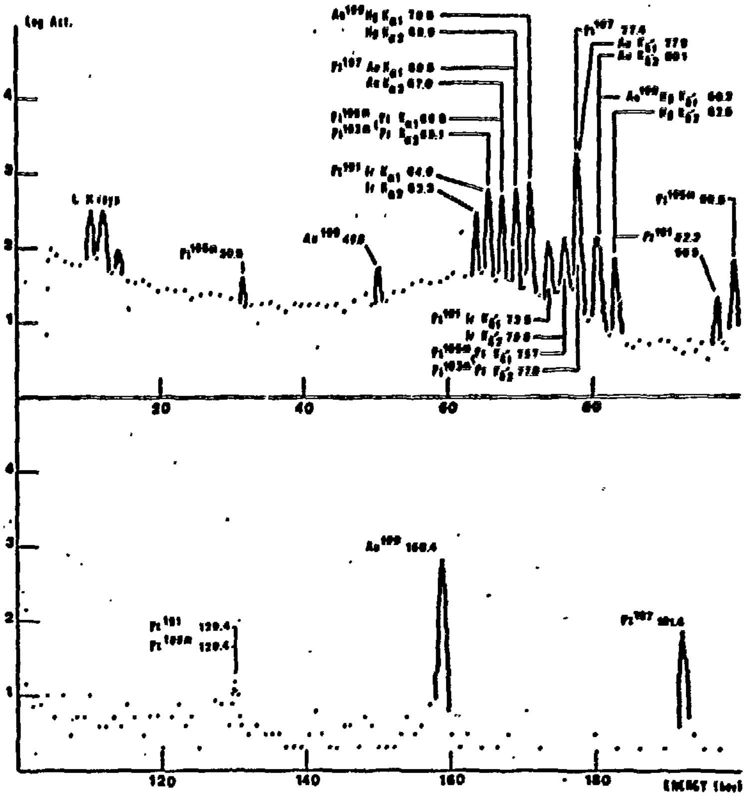
Log Act.
4
3
2
1
L X rays
20
40
60
80
4
3
2
1
Au¹⁹⁹ 158.4
Pt¹⁹⁷ 191.4
120
140
160
180
ENERGY (keV)

Fig. 4.

15.0 d ^{191}Os. It is often advisable to chose gamma counting, because

M 30

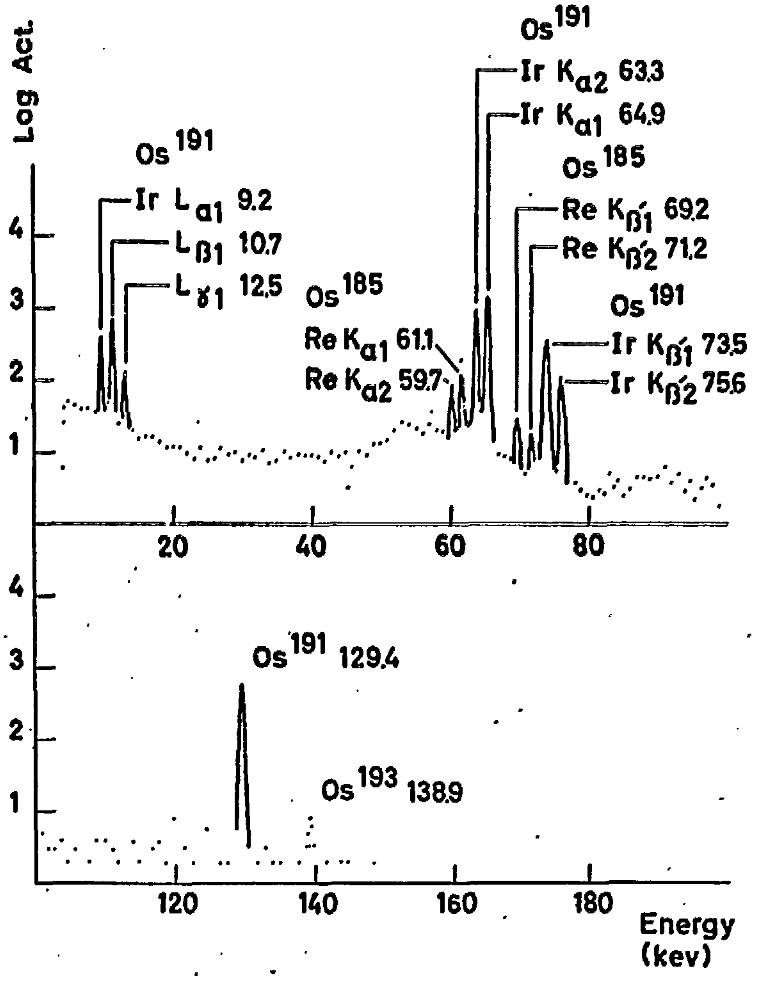

Fig. 5.