Corrado TESTA

My fifty year experience in Extraction Chromatography

JOURNAL OF CHROMATOGRAPHY

CHROMATOGRAPHY OF SOME CATIONS BY MEANS OF PAPER TREATED WITH A LIQUID ANION EXCHANGER

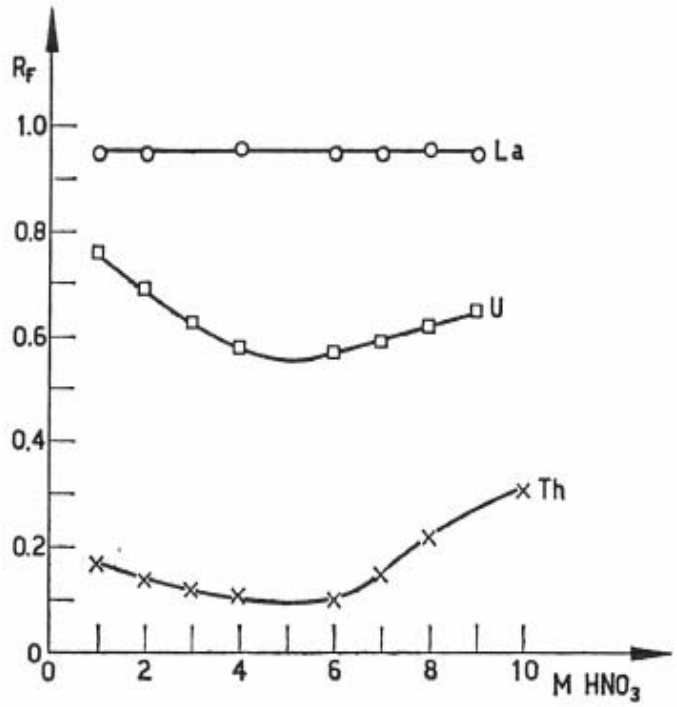
C. TESTA

Laboratori C.I.S.E., Milan (Italy)

(Received July 21st, 1960)

SEPARATION OF Fe^{3+}–Co^{2+}–Ni WITH 4 *N* HCl

Element	E_a° *(from ref.* [1]*)*	R_F	*Developer*
Fe^{3+}	1000	0	KCNS
Co^{2+}	< 1	0.50	8-Hydroxyquinoline
Ni	0	0.97	Dimethylglyoxime



R_F values on paper treated with 0.1 *M* TNOA in benzene *vs.* molarity of HNO_3 in the eluent.

THE USE OF PAPER TREATED WITH TRI-*n*-OCTYLPHOSPHINE OXIDE FOR THE CHROMATOGRAPHIC SEPARATION OF METAL IONS

E. CERRAI AND C. TESTA

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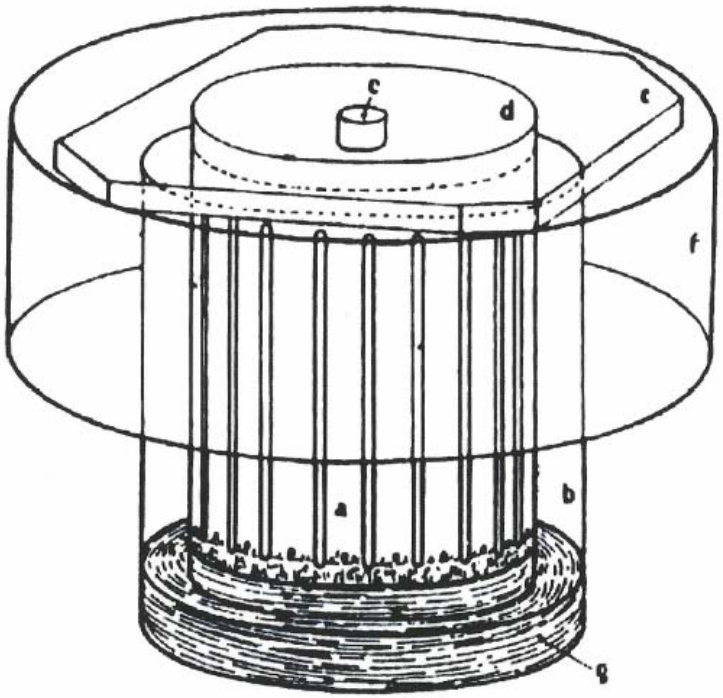
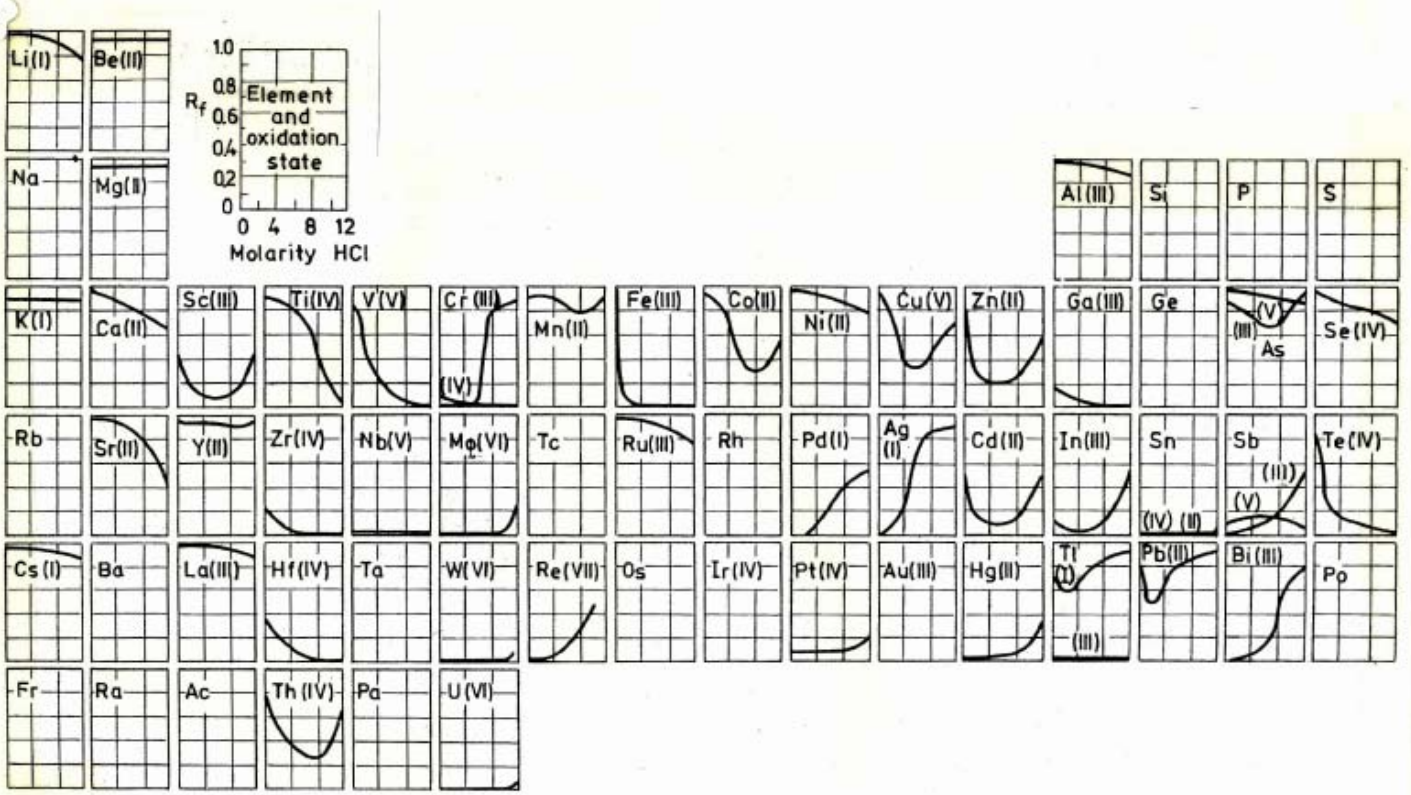
(Received February 27th, 1961)

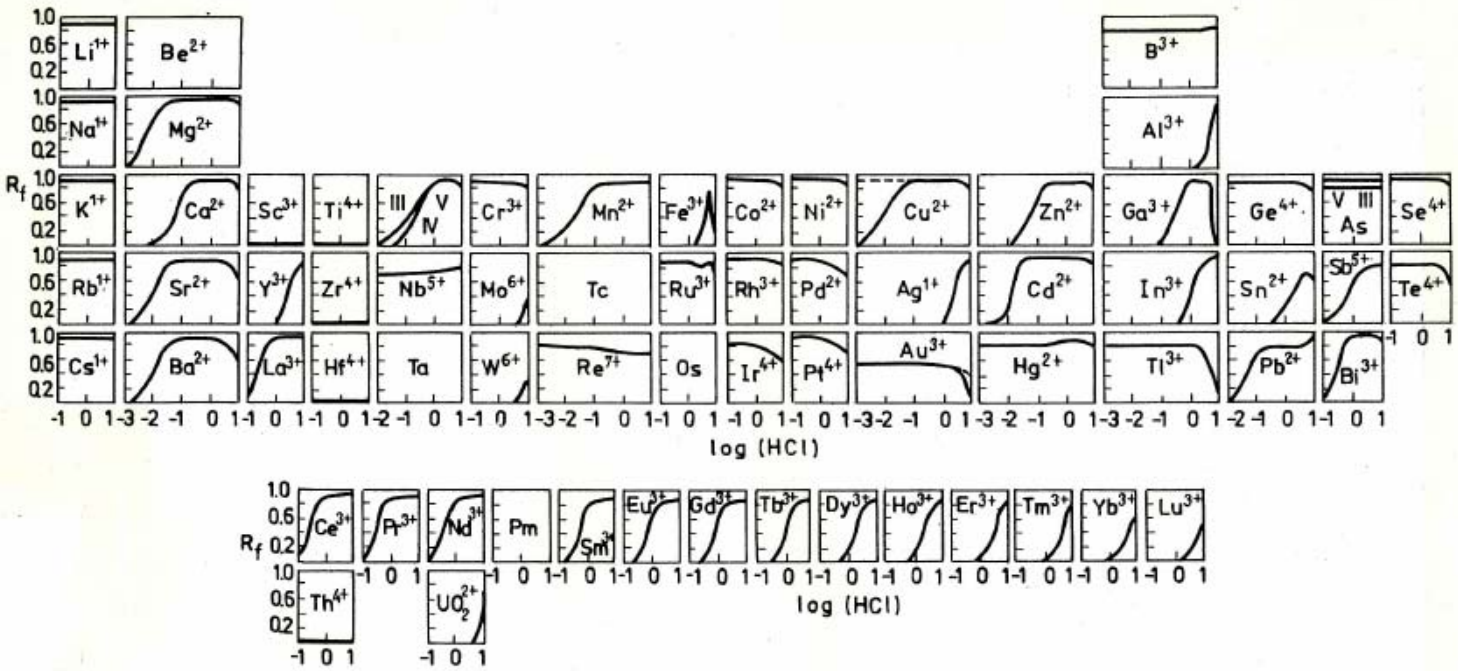


Apparatus for ascending chromatography. (a) Chromatographic paper CRL/1 type; (b) glass container; (c) perspex lid; (d) internal plate of the lid; (e) central hole; (f) glass crystallizing dish; (g) eluent solution.

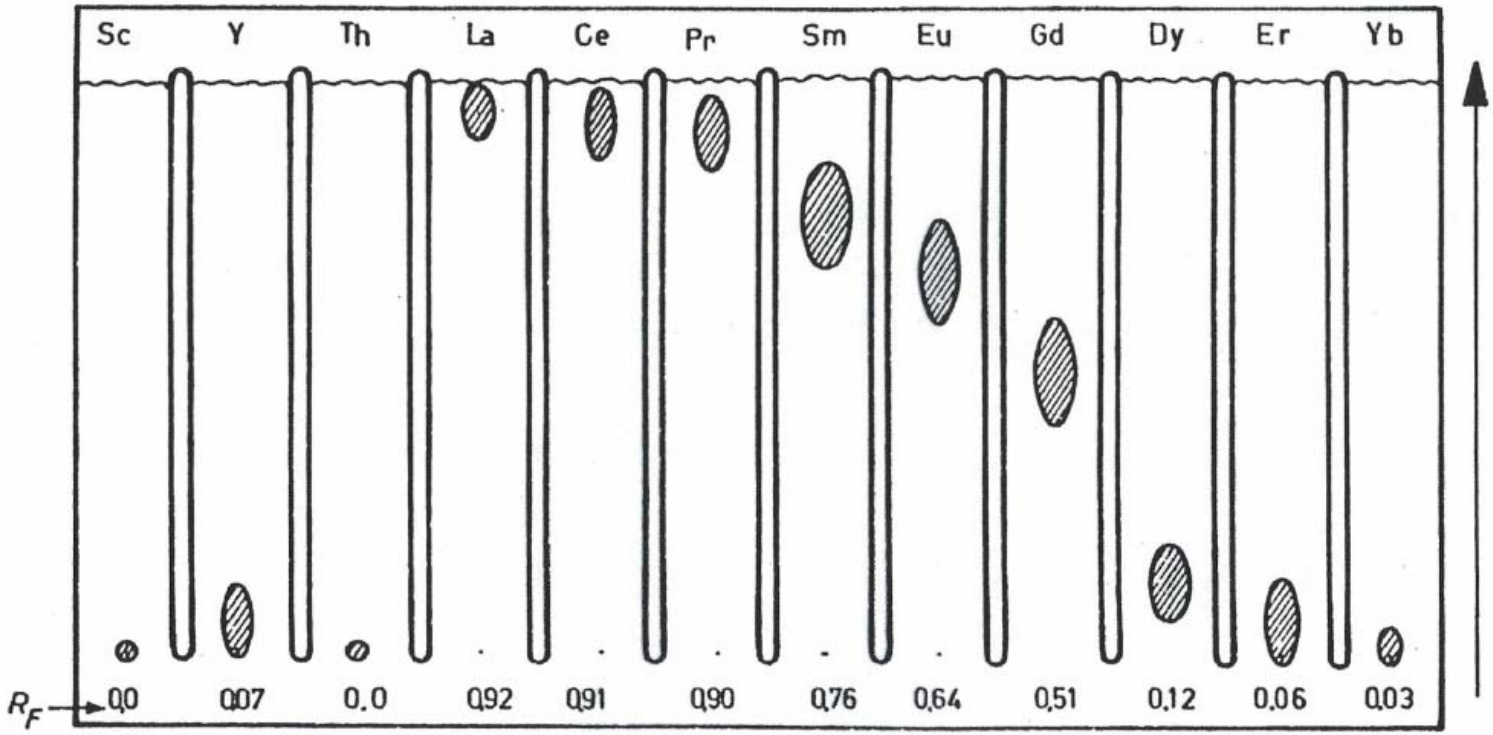
Cerrai, Testa, 1962



R_f spectra of elements eluted with HCl on paper treated with TOPO (reprinted from Ref.[4], p. 115, by courtesy of Elsevier Publ. Co.)

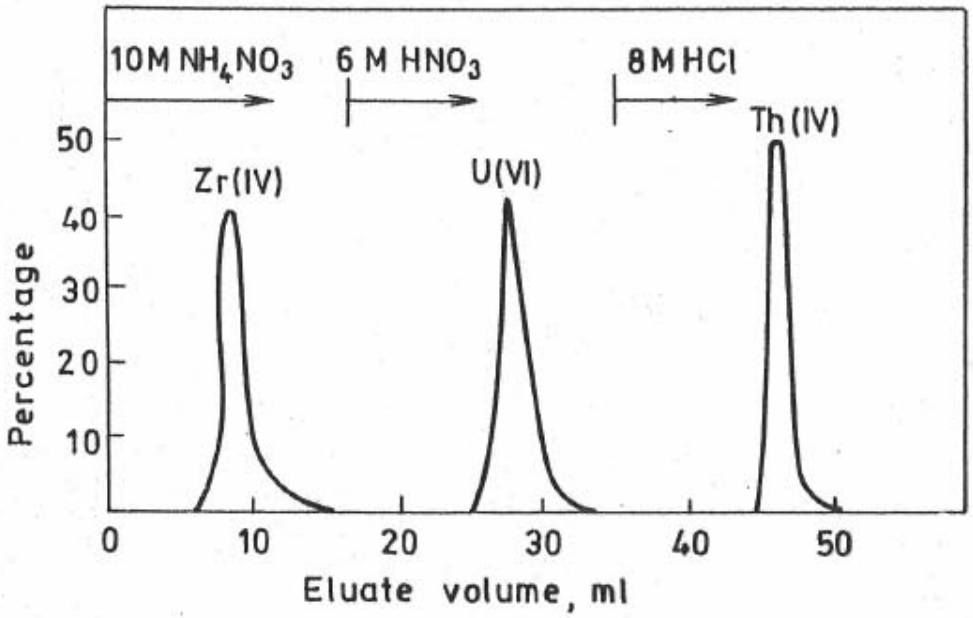


R_f spectra of elements eluted with HCl on paper treated with HDEHP (reprinted from Ref.[3], p. 386–7, by courtesy of Elsevier Publ. Co.)



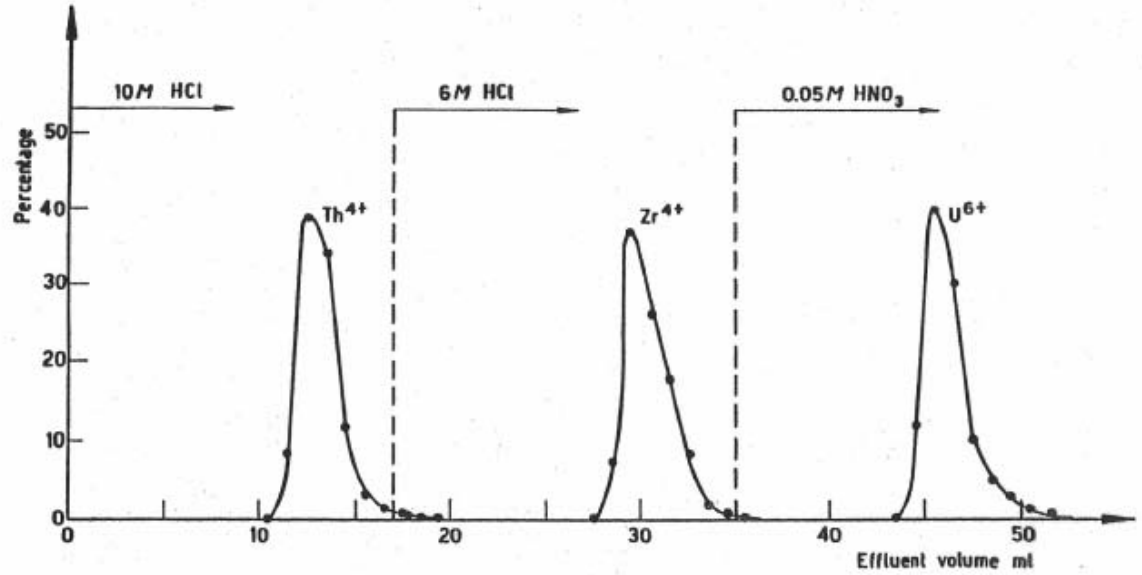
Example of ascending chromatogram on type CRL/1 paper with nine rare earths and three additional elements. Paper treated with 0.1 *M* HDEHP, elution with 1 *M* HCl.

Cerrai, Testa, 1962



Separation of Zr (10 mg), U (5 mg) and Th (1 mg).
Column: TNOA–Cellulose (0.045 M), 10x150 mm; Flow rate: 0.25 ml·cm^{-2}·min^{-1} (from Ref. [65], by courtesy of the author)

Cerrai, Testa, 1961



Separation of Th (1 mg + ^{234}Th) – Zr (10 mg) – U (5 mg). Percentage of initial amount detected in the effluent *vs.* effluent volume. Bed: 15 cm × 1 cm^2; elution rate 0.25 ml/min.cm^2.

Cellulose powder - TNOA

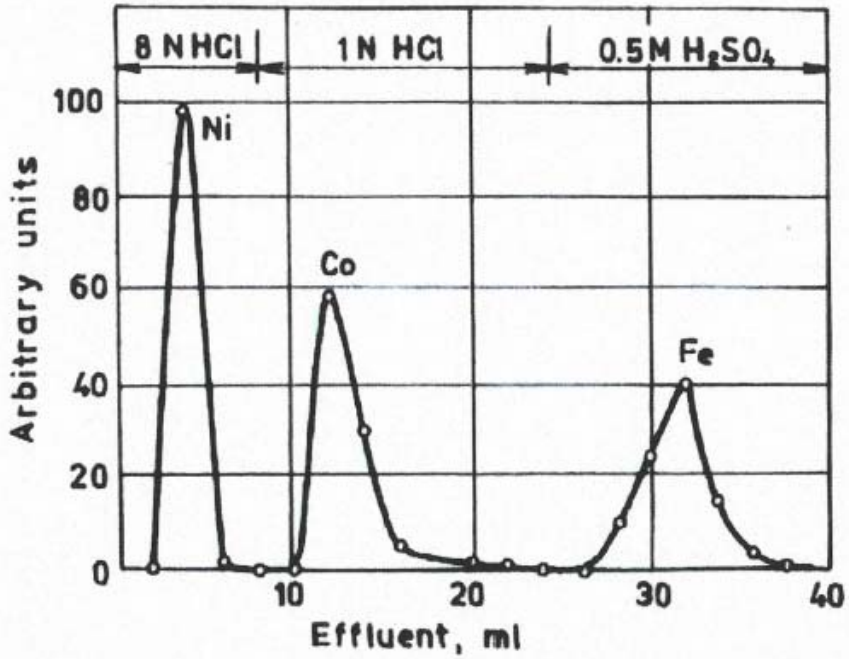
Cerrai, Testa, 1961

SUPPORTI INERTI MICROPOROSI

* KEL-F	$[\text{-CFCl-CF}_2\text{-}]$	policlorotrifluoretilene
ALGOFLON	$[\text{-CF}_2\text{-CF}_2\text{-}]_n$	politetrafluoroetilene
MOPLEN	$[\text{-CH(CH}_3\text{)-CH}_2\text{-}]_n$	polipropilene
VIPLA, PVC	$[\text{-CHCl-CH}_2\text{-}]_n$	polivinilcloruro
* MICROTENE	$[\text{-CH}_2\text{-CH}_2\text{-}]_n$	polietilene

MICROTHENE-710

50÷100 MESH A BASSA DENSITA'



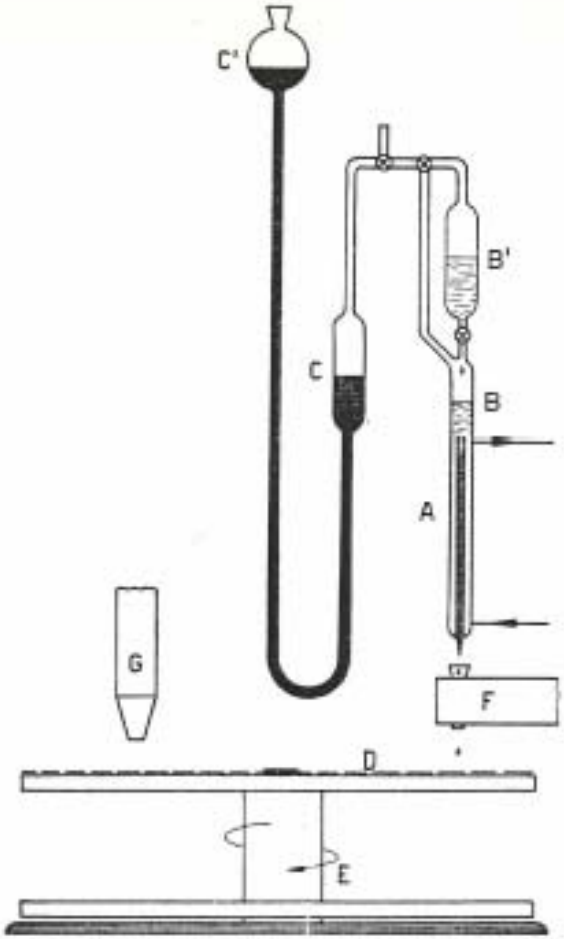
Separation of iron sub-group elements[25].

Kel-F, low density type, TOPO–HCl, H_2SO_4. Flow rate : 0.5 ml·min^{-1}; Column size : ϕ = 8.6 mm; Concentration : Ni = Co = Fe = 5 mg. (By courtesy of the authors)

E. Cerrai , C. Testa , 1962

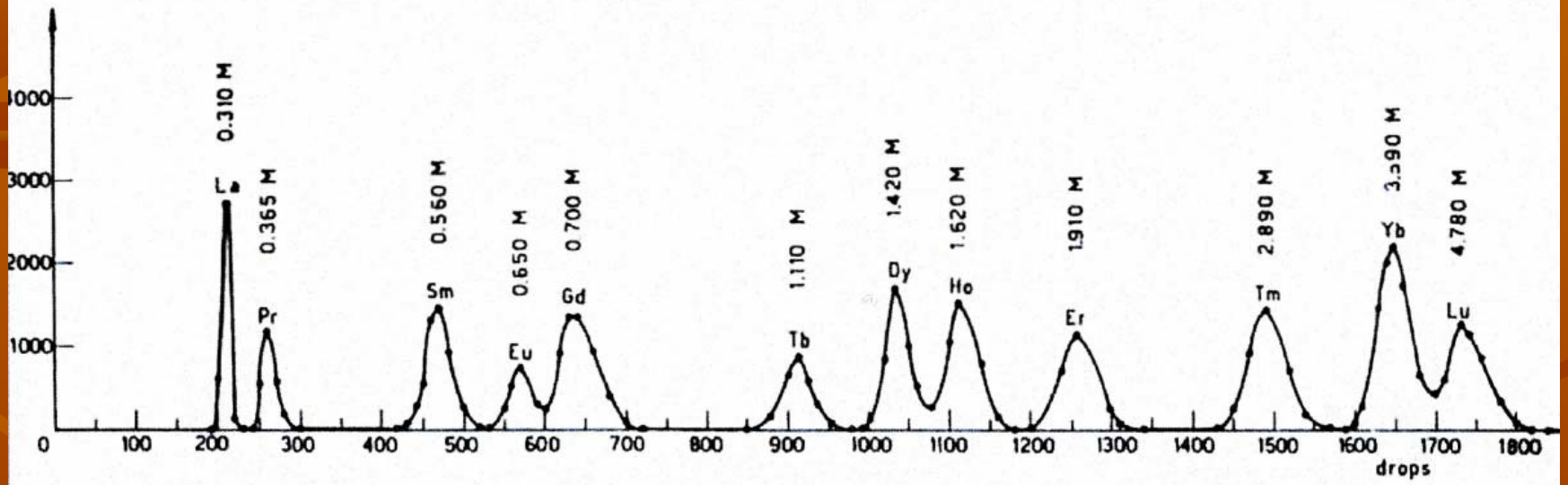
SEPARATION OF RARE EARTHS BY MEANS OF SMALL COLUMNS OF KEL-F SUPPORTING DI(2-ETHYLHEXYL)ORTHOPHOSPHORIC ACID

E. CERRAI and C. TESTA
Laboratori CISE Segrate (Milano)*



Diagrammatic sketch of the apparatus used for column separation of rare earth elements. *A*: jacketed column kept at a constant temperature; *B*: lower container of the gradient elution system; *B'*: higher container of the gradient elution system; *C* and *C'*: mercury containing bulbs; *D*: paper disk; *E*: rotating fraction collector; *F*: photocell device for elution control; *G*: warm air blower.

Kd-F — HDEHP Column.

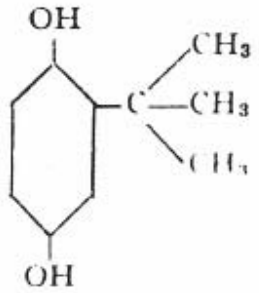


Separation of La-Pr-Sm-Eu-Gd-Tb-Dy-Ho-Er-Tm-Yb-Lu at 85 ± 1°C. Feed solution: 0·04 ml of a solution containing the elements at a tracer level. Gradient elution with HCl; the molarity is quoted in the plot. Flow-rate 0·06 ml/min (4·8 drops/min).

Cerrai, Testa, 1963

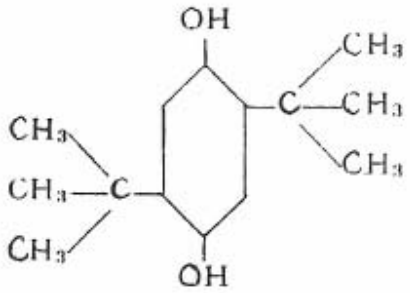
ORGANIC COMPOUNDS INVESTIGATED

Compound A



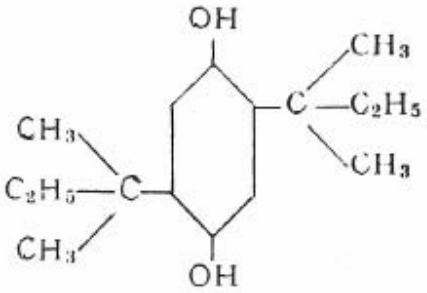
Tert.-butylhydroquinone (M.W. = 166)

Compound B



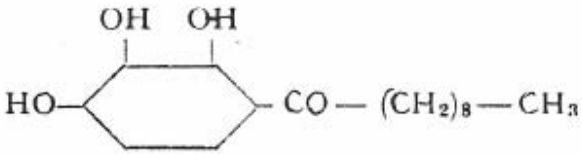
2,5-Di-*tert.*-butylhydroquinone (M.W. = 222)

Compound C



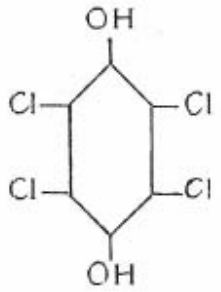
2,5-Di-*tert.*-amylhydroquinone (M.W. = 250)

Compound D

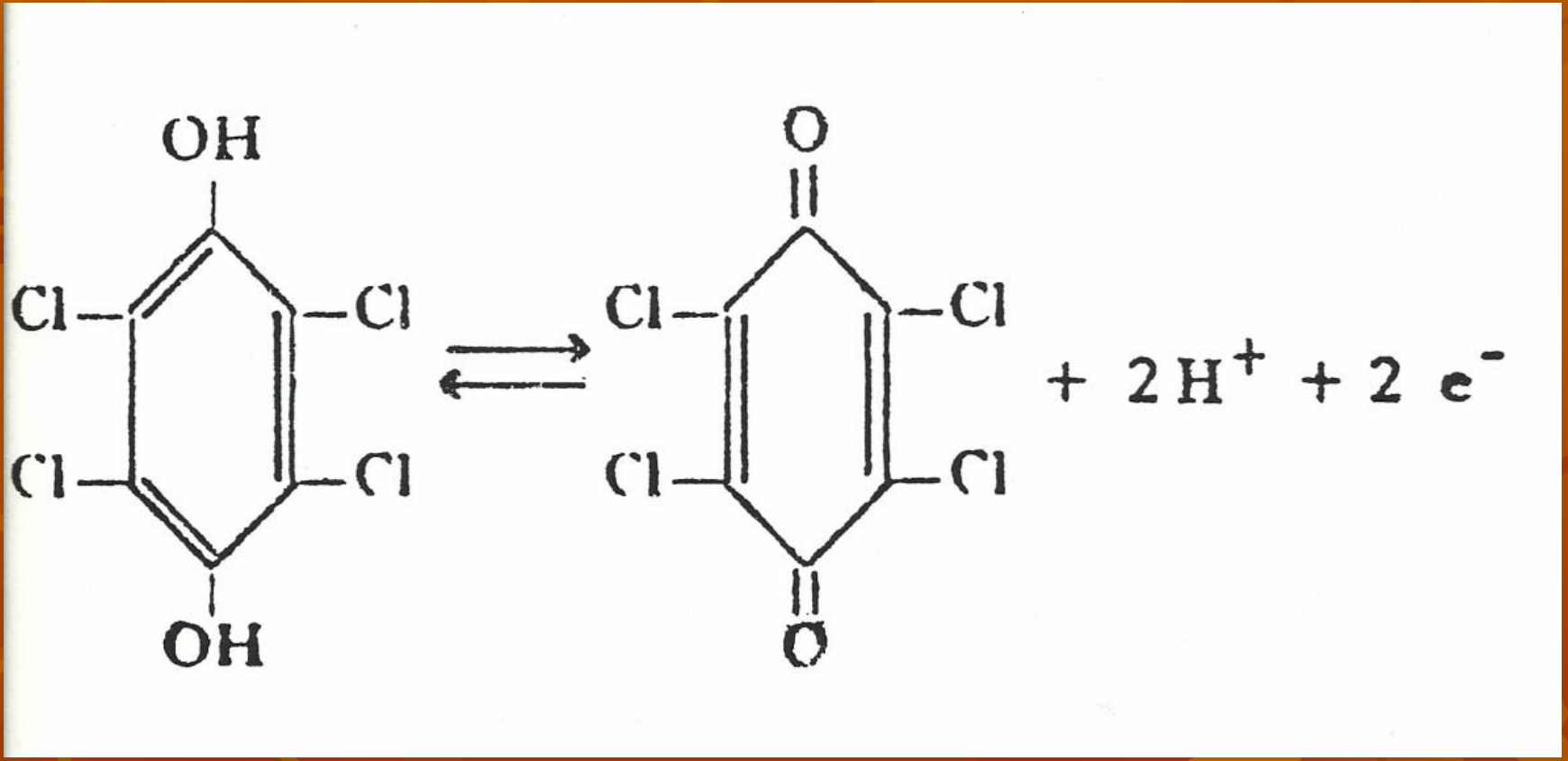


2,3,4-Trihydroxydecanophenone (M.W. = 280)

Compound E



Tetrachlorohydroquinone (M.W. = 248)


OH
Cl
Cl
Cl
Cl
OH
O
Cl
Cl
Cl
Cl
O
+ 2 H+ + 2 e-

REDOX REACTIONS OBTAINED WITH COLUMN I

Reduced form			*Oxidized form*		
Cycle	*Reaction*	*Bed capacity (meq)*	*Cycle*	*Reaction*	*Bed capacity (meq)*
1	$Fe^{3+} \rightarrow Fe^{2+}$	9.80[a]	2	$I^- \rightarrow I$	9.60[b]
3	$I \rightarrow I^-$	10.96	4	$Sn^{2+} \rightarrow Sn^{4+}$	10.70
5	$V^{5+} \rightarrow V^{4+}$	—	6	$Sn^{2+} \rightarrow Sn^{4+}$	—
7	$Ce^{4+} \rightarrow Ce^{3+}$	10.80	8	$Cu^{+} \rightarrow Cu^{2+}$	12.00[c]
9	$Fe^{3+} \rightarrow Fe^{2+}$	—	10	Ascorbic ac. → dehydroasc. ac.	10.90
11	$I \rightarrow I^-$	10.70	12	$I^- \rightarrow I$	10.50
13	$Ce^{4+} \rightarrow Ce^{3+}$	11.00			

[a] Incomplete saturation of the bed.
[b] The previous oxidation was incomplete.
[c] Cu^{+} partially oxidized to Cu^{2+} by atmospheric oxygen.

ELECTRON-EXCHANGE PROCESSES ON SIMPLE COLUMNS OF KEL-F SUPPORTING TETRACHLOROHYDROQUINONE

E. CERRAI AND C. TESTA

*Laboratori CISE - Segrate, Milano (Italy)**

(Received September 1st, 1962)

THE APPLICATION OF COLUMN REDOX–EXTRACTION CHROMATOGRAPHY TO THE SEPARATION OF SOME ACTINIDE ELEMENTS

A. DELLE SITE and C. TESTA

Radiotoxicological Laboratory, Medical Service, CSN-CASACCIA, CNEN, Rome (Italy)

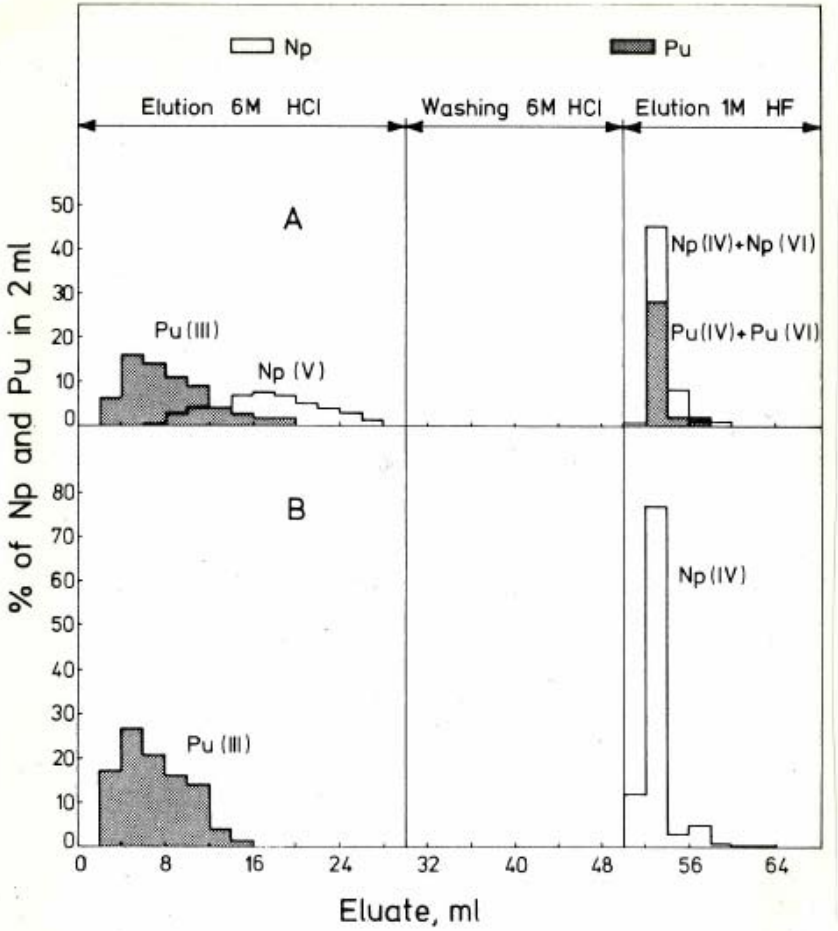


Fig. 2. Separation of neptunium and plutonium in 6 *M* HCl. (A) Microthene–TOPO column; (B) Microthene–TOPO–DPHQ column.

USE OF EXTRACTION CHROMATOGRAPHY IN RADIOTOXICOLOGY

C. TESTA

Determinations of radionuclides in urine by means of extraction chromatography techniques

Radio-nuclide[a]	Pretreat-ment[b]	HNO_3 Molarity in urine	Inert support[c]	Stationary phase[d]	Chroma-tog. pro-cedure[e]	Eluting agent	Final recov-ery, %	Sensitivity limit	Detec-tion system[f]	Time, hrs.	Ref.
Th	W.M.	4.0 M	K.	0.1 M TOPO	C.C.	0.5 M HCl	97.5	1 µg/l	Col.	8	[9]
	W.M.	4.0 M	M.	0.5 M TOPO	B.E.	0.3 M H_2SO_4	98.2	0.2 µg/l	Col.	4	[17]
U	W.M.	7.5 M	K.	conc. TBP	C.C.	Water	91.1	2 dpm/l	ZnS	4	[12]
	W.M	4.0 M	M.	0.5 M TOPO	B.E.	1 M HF	70.0	1 dpm/l	ZnS	4	[17]
Pu	P.P	2.0 M	K.	1 M TNOA	C.C.	conc. H_2SO_3	90.5	0.04 pCi/l	SSD	16	[14]
	W.M	4.0 M	M.	0.3 M TOPO	B.E.	6 M HCl·0.01 M HI	70.5	0.07 pCi/l	SSD	8	[25]
	W.M.	2.0 M	M.	0.3 M TOPO	B.E.	6 M HCl·0.1 M HI	76.5	0.10 pCi/l	ZnS	4	[20]
	P.O.	2.0 M	M.	0.3 M HX70	C.C	2 M HNO_3·0.15% hydroquinone	73.5	0.08 pCi/l	SSD	16	[29]
Np	W.M.	6.0 M	M.	0.1 M TOPO	E.E.	6 M HCl · Cl_2	83.2	0.05 pCi/l	SSD	8	[26]
	P.P.	2.0 M	M.	0.3 M HX70	C.C.	0.1 M oxalic acid	82.3	0.04 pCi/l	SSD	16	[29]
Am (Cm)	W.M.	0.001 M	M.	1.5 M HDEHP	B.E.	3 M HNO_3	85.9	0.05 pCi/l	SSD	8	[33]
Y	W.M.	0.3 M	M.	1.5 M HDEHP	B.E.	6 M HCl	92.0	5 pCi/l	β	4	[35]

[a] Th = Natural thorium; U = enriched uranium; Pu = ^{239}Pu, ^{240}Pu; Np = ^{237}Np; Am = ^{241}Am; Y = ^{90}Y, for the determination of ^{90}Sr
[b] W.M. = Wet mineralization; P.P. = Phosphates precipitation
[c] K. = Kel-F; M. = Microthene-710
[d] TOPO = Tri-n-octylphosphine oxide; TBP = Tri-n-butylphosphate; TNOA = Tri-n-octylamine; HX70 = Neo-tridecanohydroxamic acid; HDEHP = Di(2-ethylhexyl) phosphoric acid
[e] C.C. = Column chromatography; B. E. = Batch extraction
[f] Col. = Colorimetry; ZnS = Alpha counting with a ZnS(Ag) detector; SSD = Alpha counting with a solid state detector after electroplating; β = low background beta counting

Mineralizzazione del campione (1-1,5 L) con HNO_3 e H_2O_2

⇩

Precipitazione dell'Uranio con H_3PO_4

⇩

Separazione dell'Uranio su colonna cromatografica (polietilene microporoso + tri-ottilfosfina ossido)

⇩

Preparazione della sorgente alfa per elettrodeposizione

⇩

Conteggio mediante rivelatore al silicio a barriera superficiale

Extraction Chromatography in Radioecology

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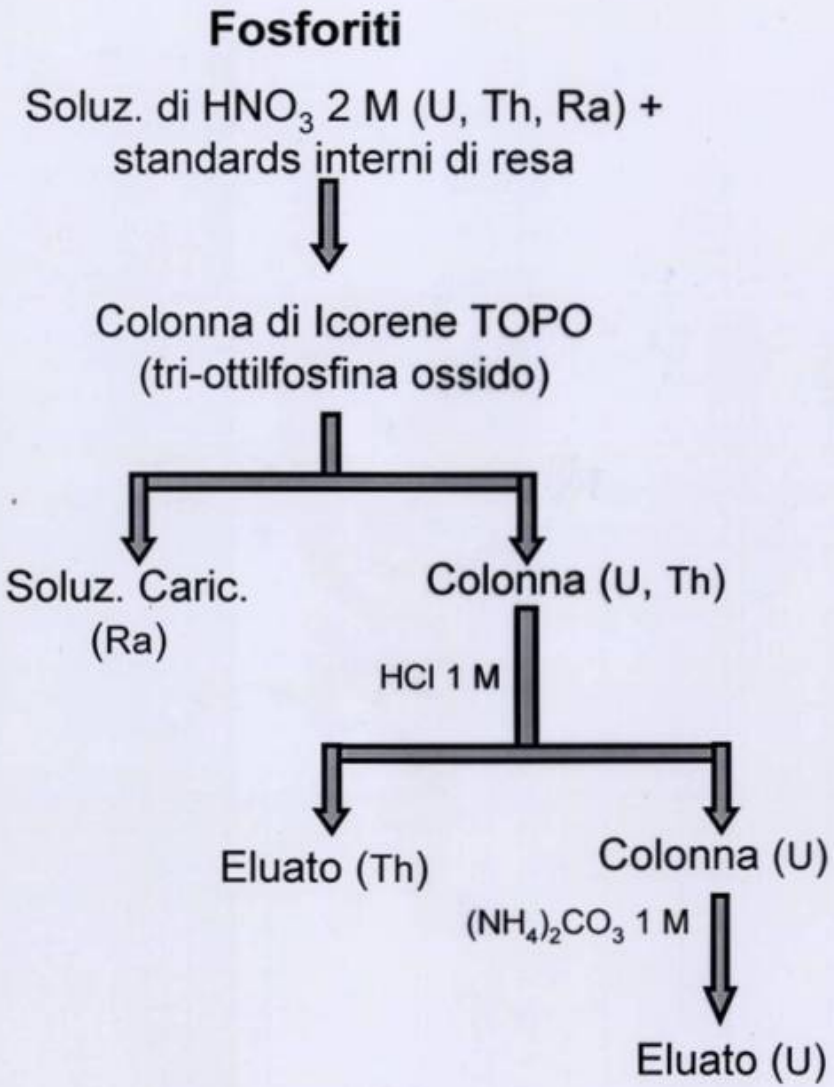
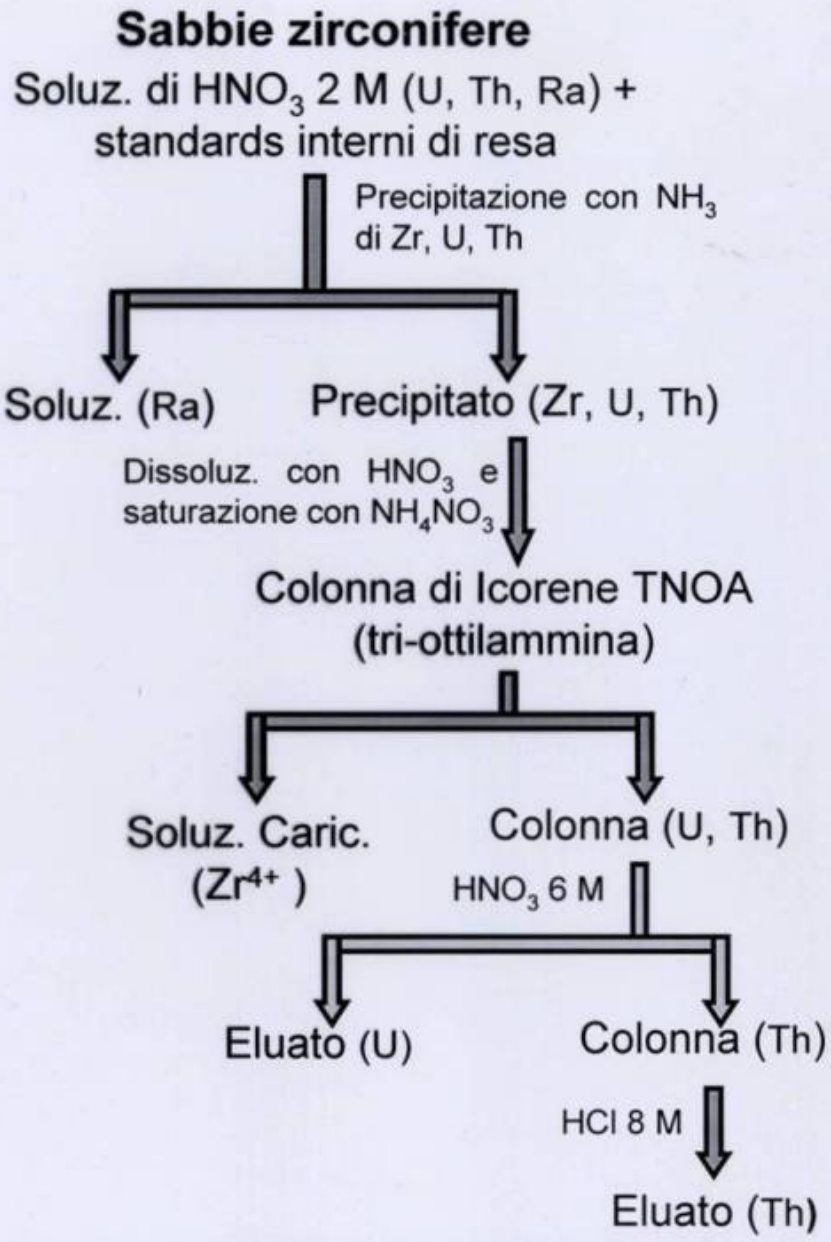
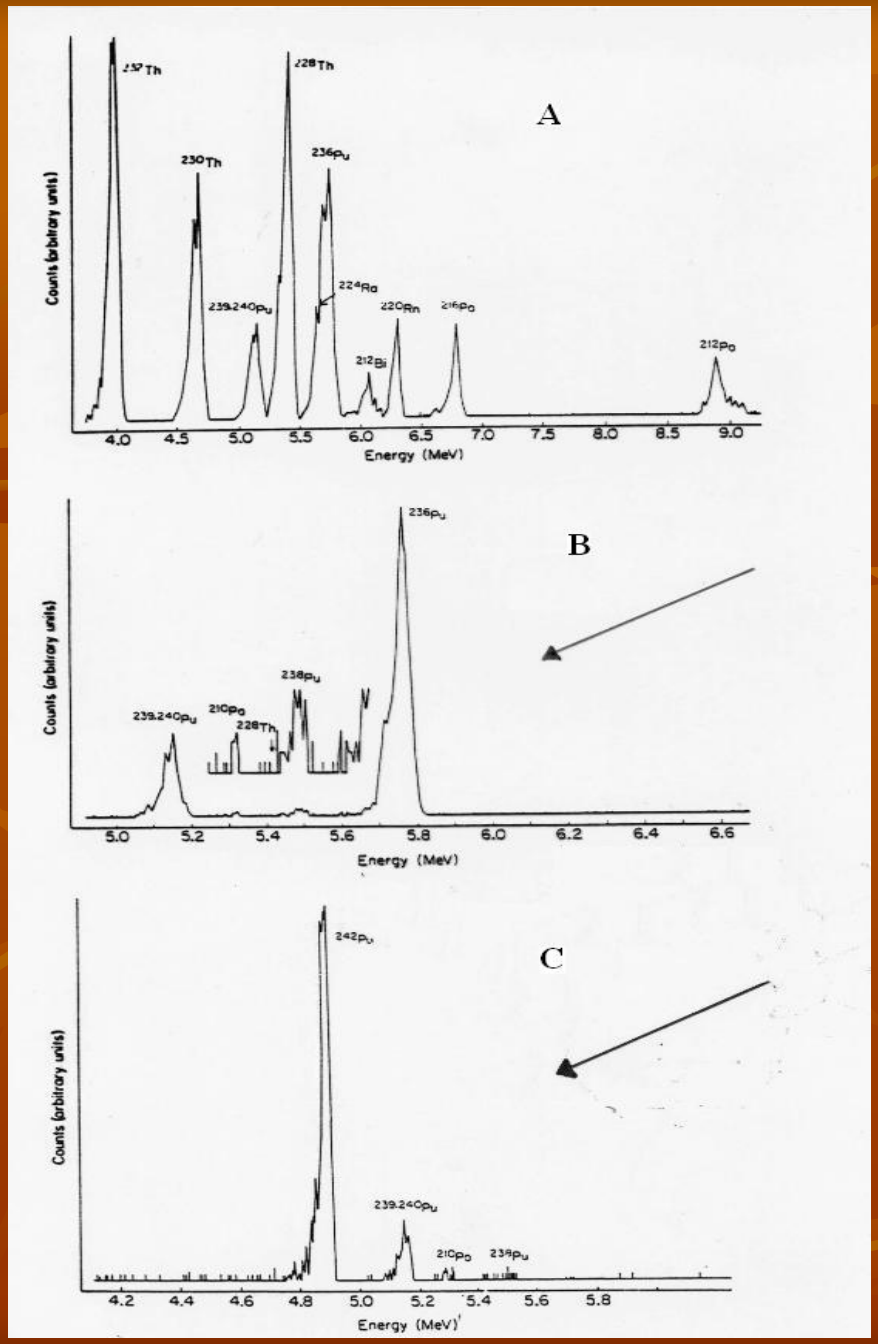
Institute of General Chemistry
University of Urbino, Italy

Rad. & Rad.
1991


Fosforiti
Soluz. di HNO_3 2 M (U, Th, Ra) + standards interni di resa
Colonna di Icorene TOPO (tri-ottilfosfina ossido)
Soluz. Caric. (Ra)
Colonna (U, Th)
HCl 1 M
Eluato (Th)
Colonna (U)
$(NH_4)_2CO_3$ 1 M
Eluato (U)


Sabbie zirconifere
Soluz. di HNO_3 2 M (U, Th, Ra) + standards interni di resa
Precipitazione con NH_3 di Zr, U, Th
Soluz. (Ra)
Precipitato (Zr, U, Th)
Dissoluz. con HNO_3 e saturazione con NH_4NO_3
Colonna di Icorene TNOA (tri-ottilammina)
Soluz. Caric. (Zr^{4+})
Colonna (U, Th)
HNO_3 6 M
Eluato (U)
Colonna (Th)
HCl 8 M
Eluato (Th)



The α–spectra of

(A) a sediment sample after one extraction step with TOPO;

(B) sediment sample after double extraction with TOPO;

(C) a marine organism sample (*Merluccius merluccius*) after double extraction with TOPO.

CONCENTRATION AND SPECIATION OF PLUTONIUM, AMERICIUM, URANIUM, THORIUM, POTASSIUM AND Cs-137 IN A VENICE CANAL SEDIMENT SAMPLE

C. Testa, D. Desideri, F. Guerra, M.A. Meli, C. Roselli

Institute of General Chemistry, Urbino University, Urbino, Italy

S. Degetto

ICTIMA-CNR, Padua, Italy

VERTICAL PROFILES OF Pu-239 (240), Pu-238, AND Am-241 IN SOME PECULIAR ITALIAN MOSSES

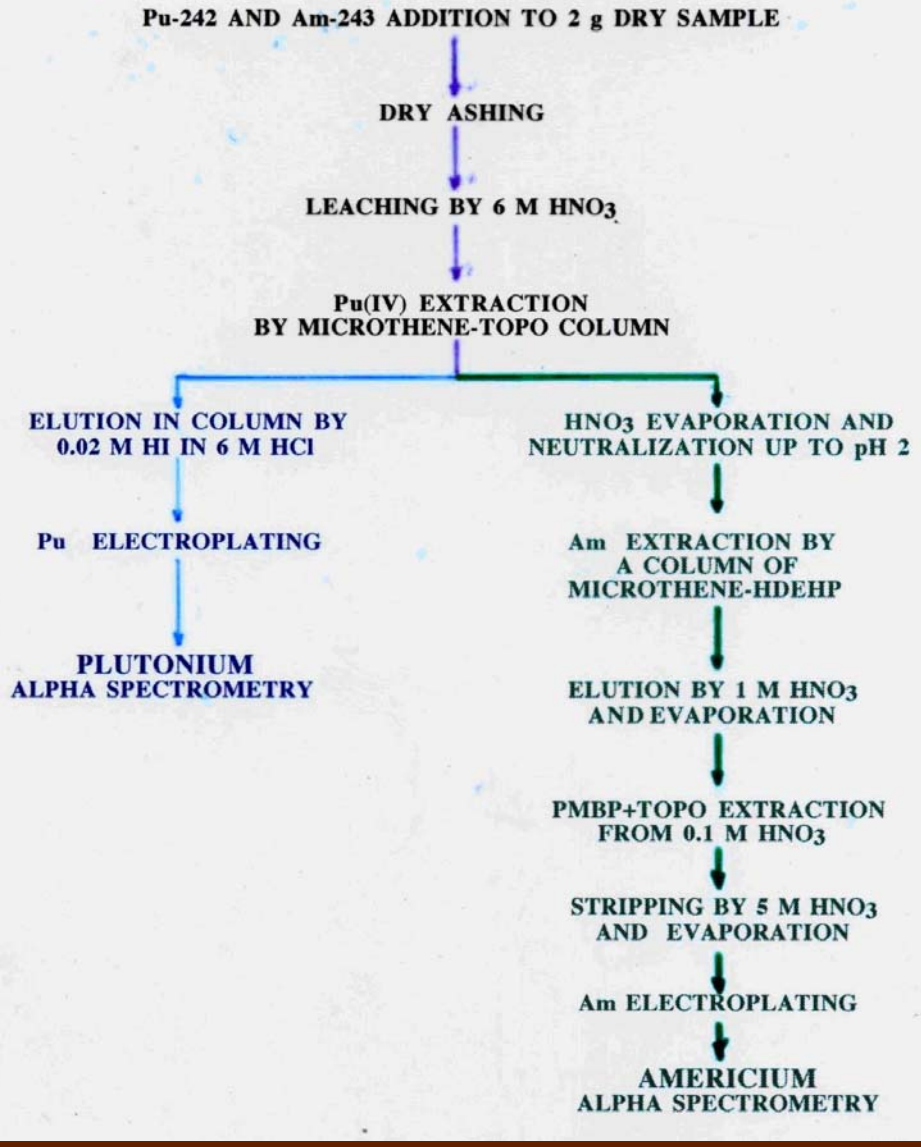
C. Testa[1], G. Jia[2], S. Degetto[3], D. Desideri[1], F. Guerra[1], M.A. Meli[1], C. Roselli[1].

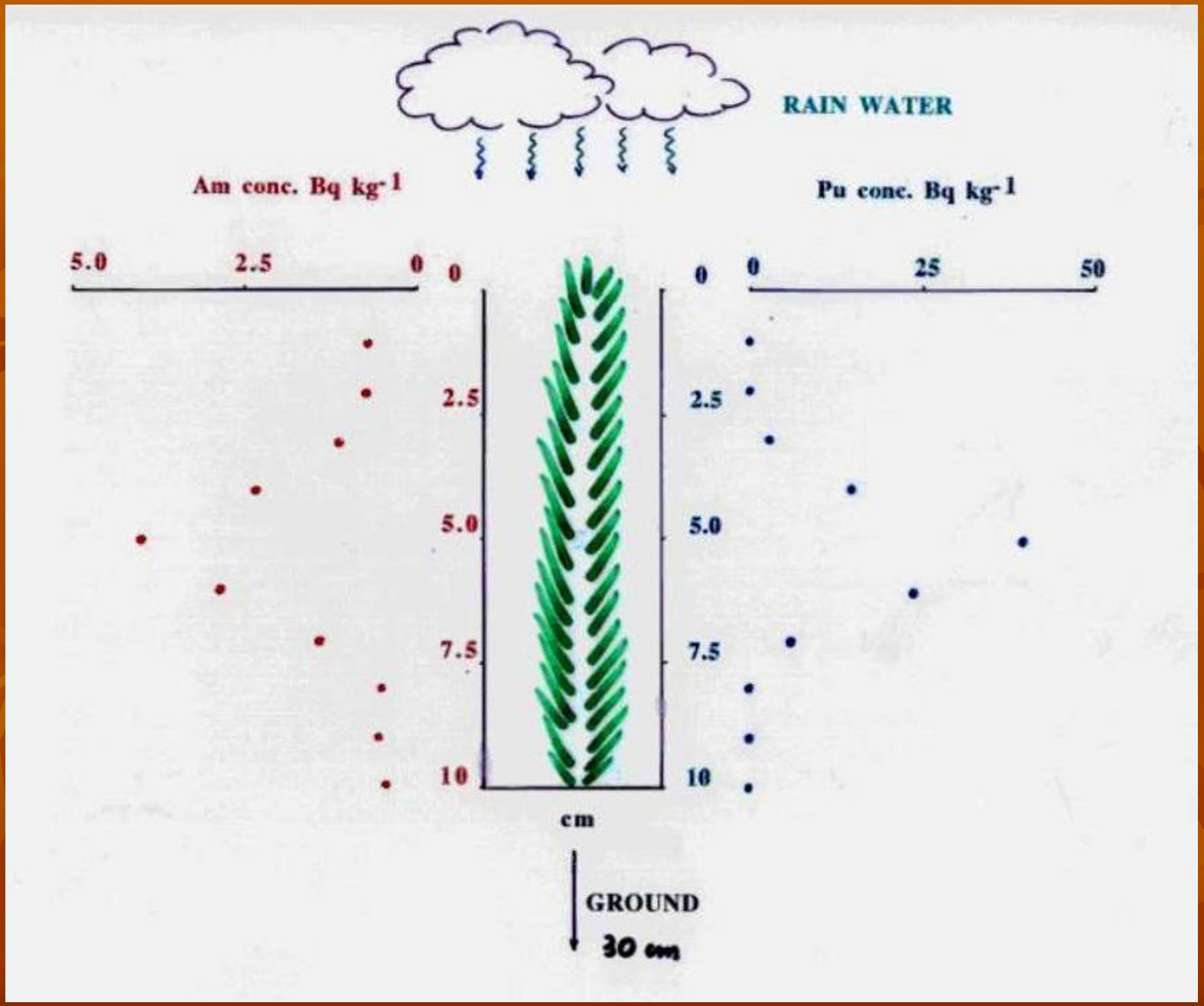
1. Urbino University, Italy

2. Institute of Atomic Energy, Beijing, P. R. of China

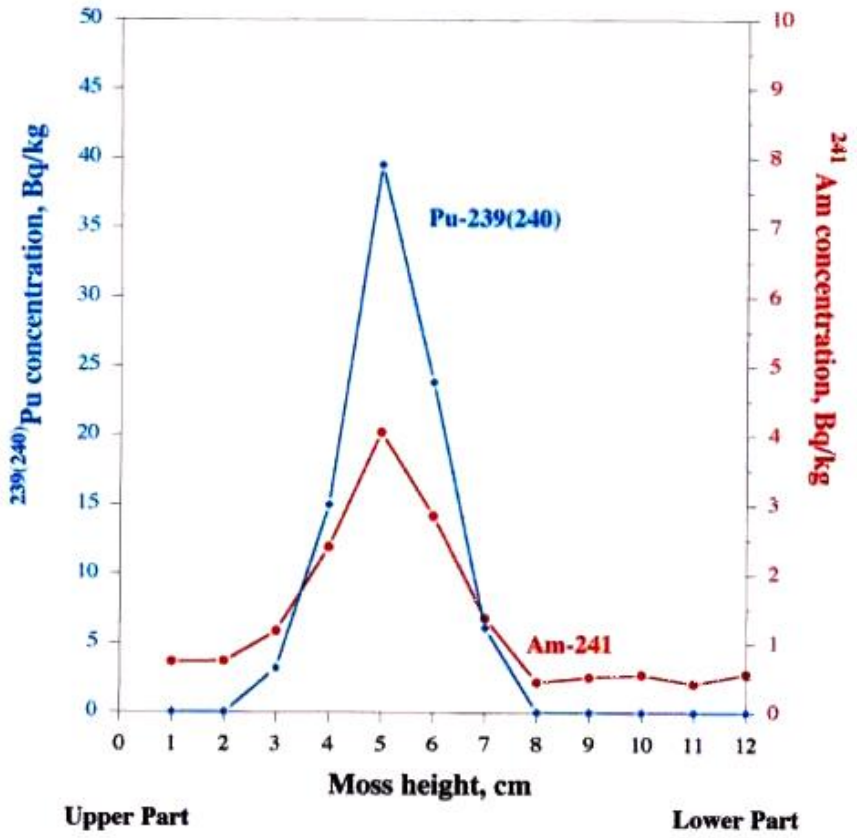
3. CNR - ICTIMA, Padua, Italy

RADIOANALYTICAL METHOD FOR THE DETERMINATION OF PLUTONIUM AND AMERICIUM IN MOSSES AND LICHENS

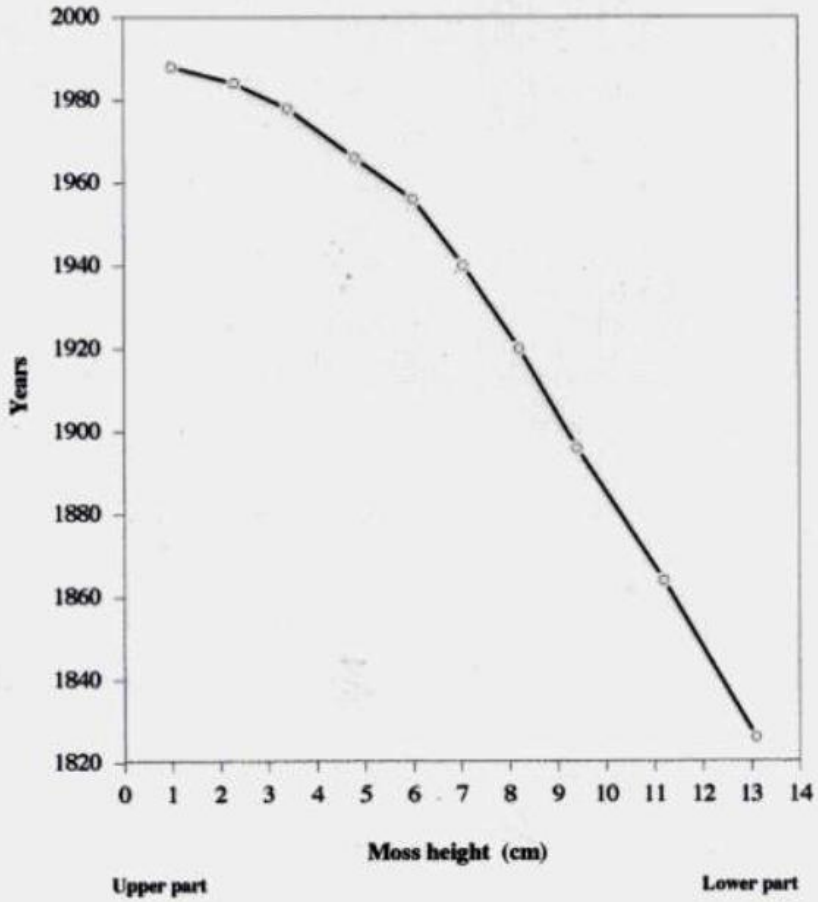



RAIN WATER
Am conc. Bq kg-1
Pu conc. Bq kg-1
5.0
2.5
0
0
2.5
5.0
7.5
10
0
2.5
5.0
7.5
10
0
25
50
cm
GROUND
30 cm

Vertical distribution of $^{239(240)}$Pu and ^{241}Am in a terrestrial moss core (Sphagnum Compactum) drawn on the Alps (Northern Italy)



SPHAGNUM COMPACTUM SECTIONS DATATION BY THE Pb-210 METHOD

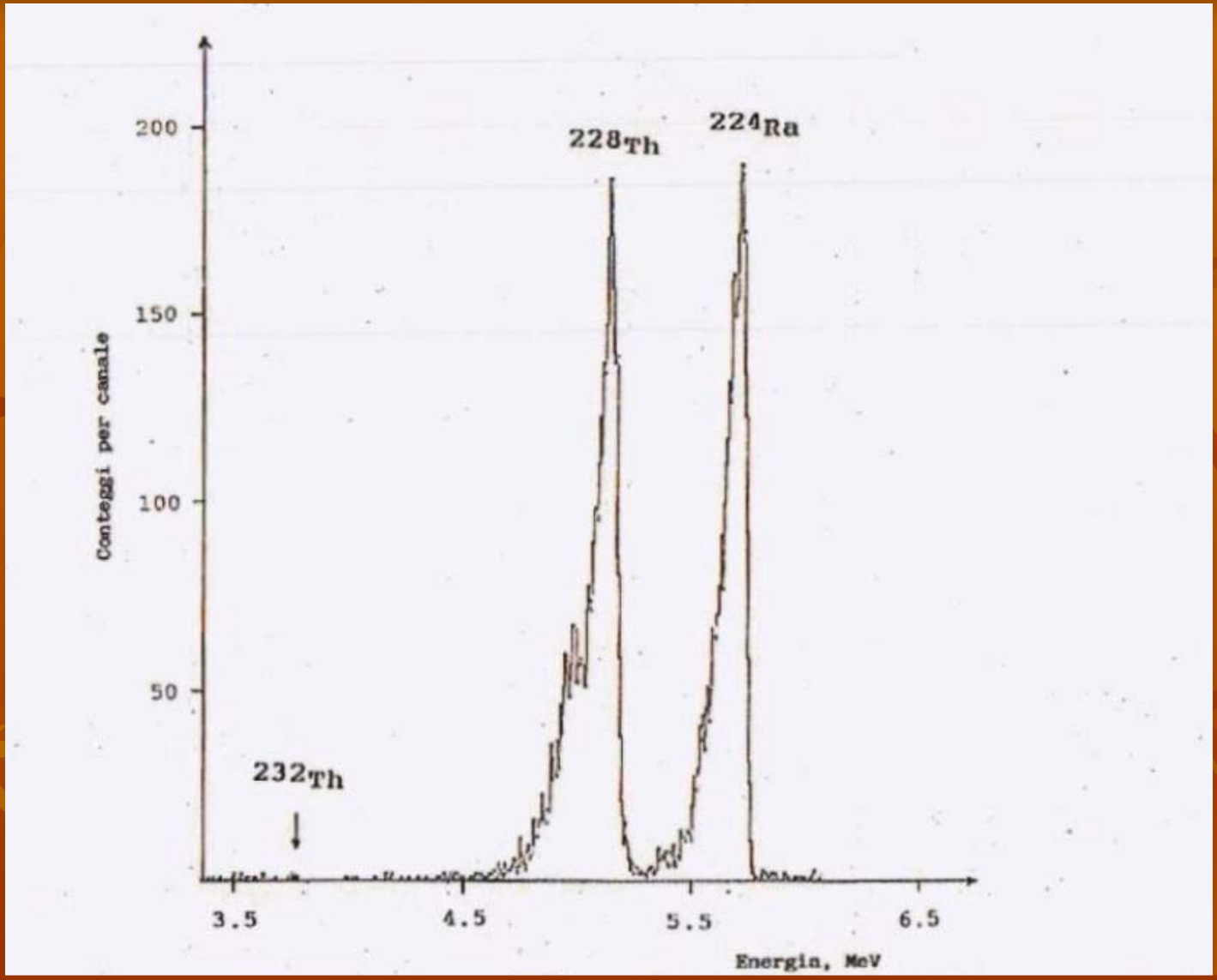


EXTRACTION CHROMATOGRAPHY FOR THE DETERMINATION OF NATURAL RADIONUCLIDES IN SOME MATRIXES CONNECTED WITH HYDROCARBONS EXPLOITATION.

C. Testa, D. Desideri, F. Guerra, M. A. Meli, C. Roselli

General Chemistry Institute, University of Urbino (Italy)

- TYPICAL L.S.A. SCALE FORMATION



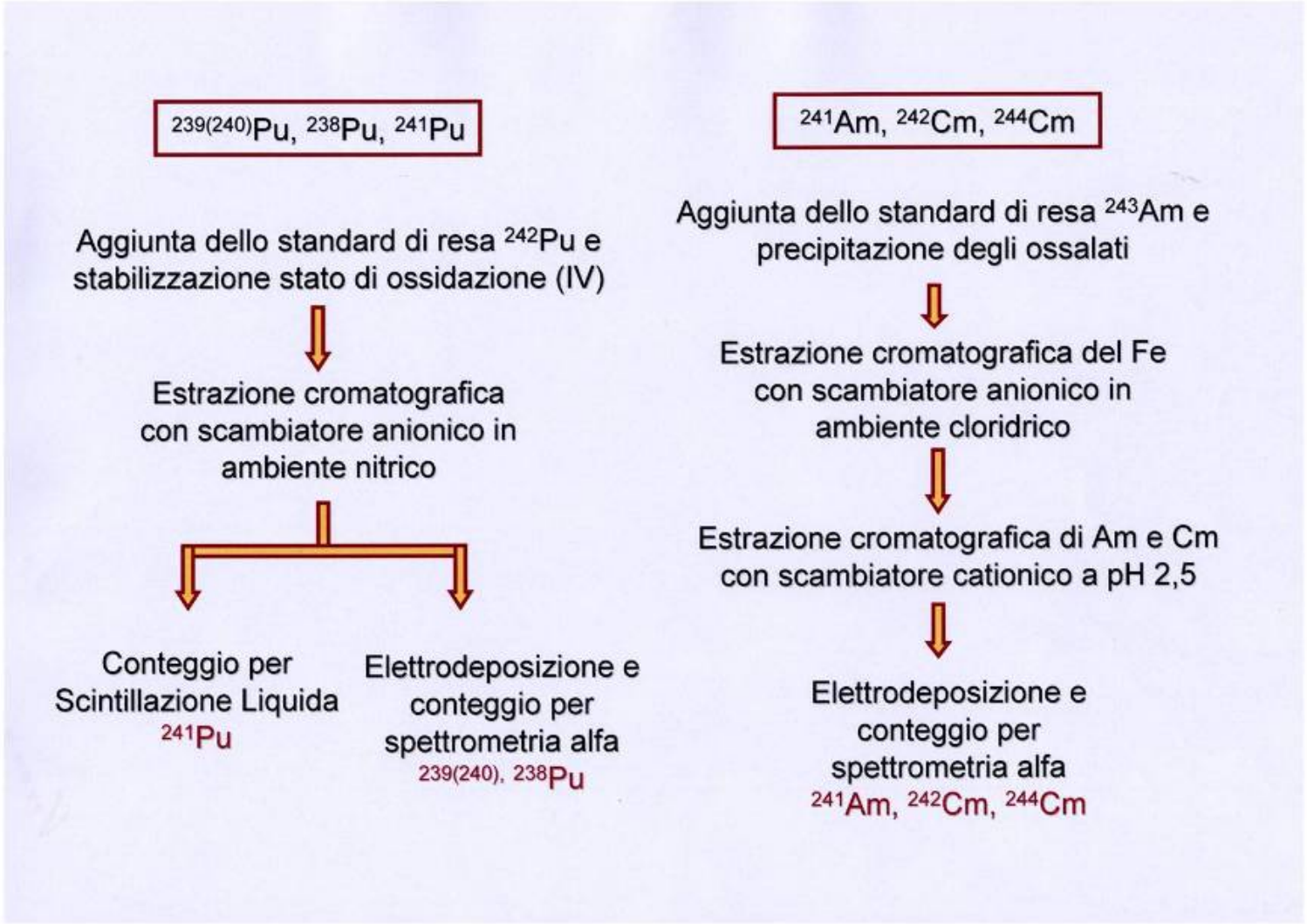
Spettro alfa del Torio isolato da un'incrostazione proveniente da un impianto di estrazione di idrocarburi sito in Tunisia; si noti l'enorme disequilibrio tra il ^{232} Th e il ^{228} Th

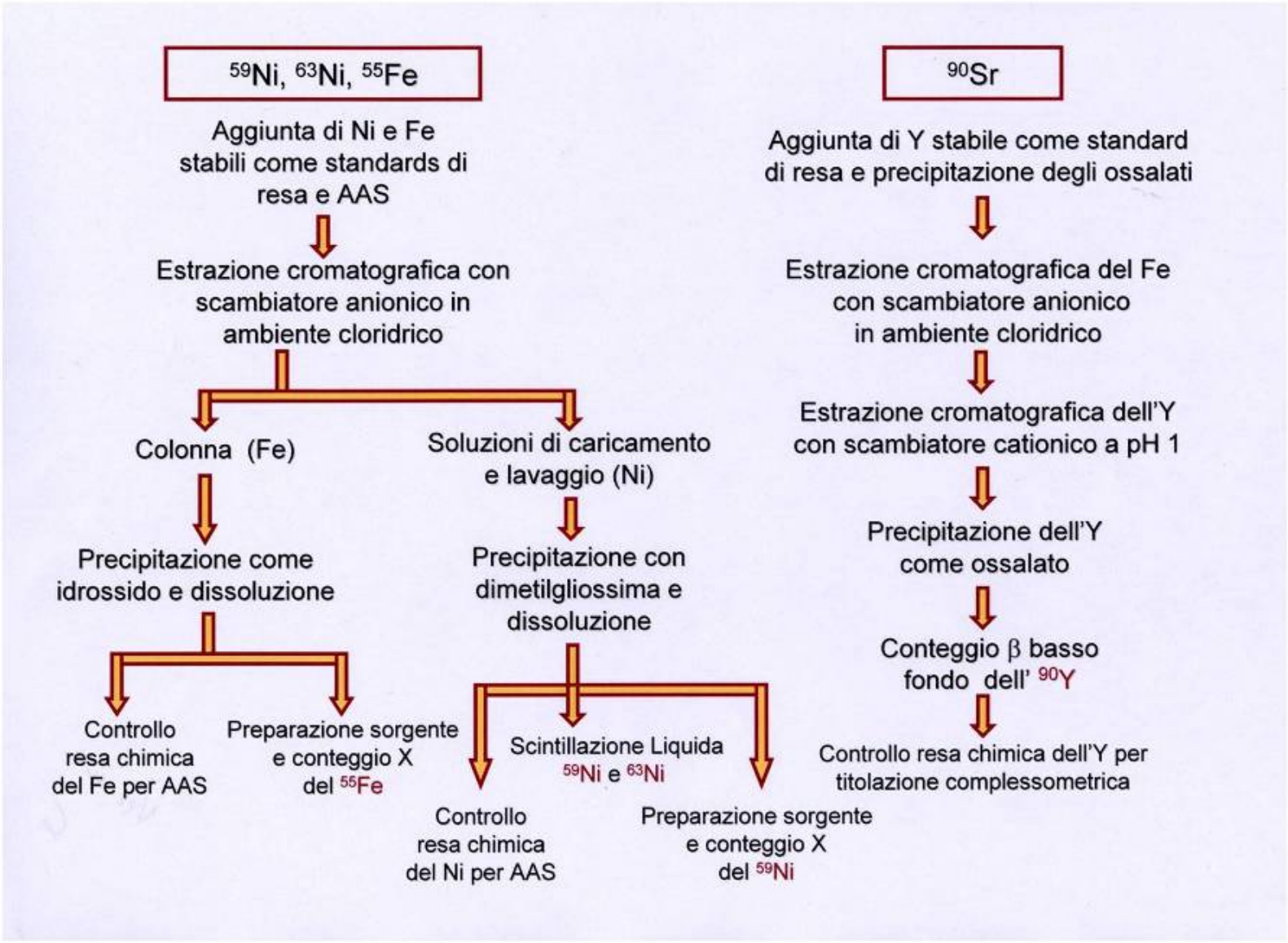
Corrado Testa
Centro di Radiochimica Applicata
Università di Urbino "Carlo Bo"

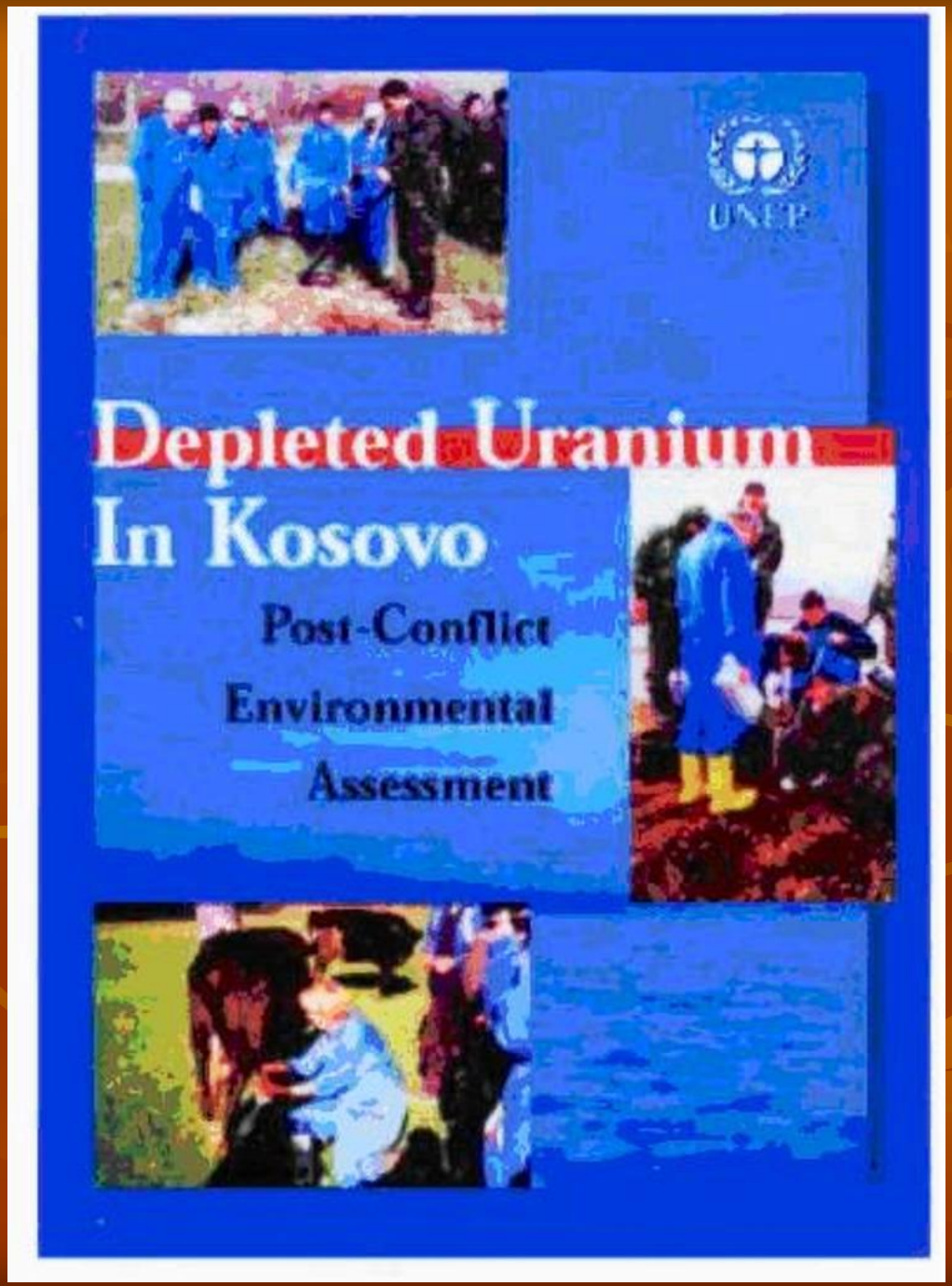
L'IMPORTANZA DELLA RADIOCHIMICA IN CAMPO AMBIENTALE E PER LA CARATTERIZZAZIONE RADIOLOGICA DI MATERIALI PROVENIENTI DA CENTRALI NUCLEARI IN DECOMMISSIONING.

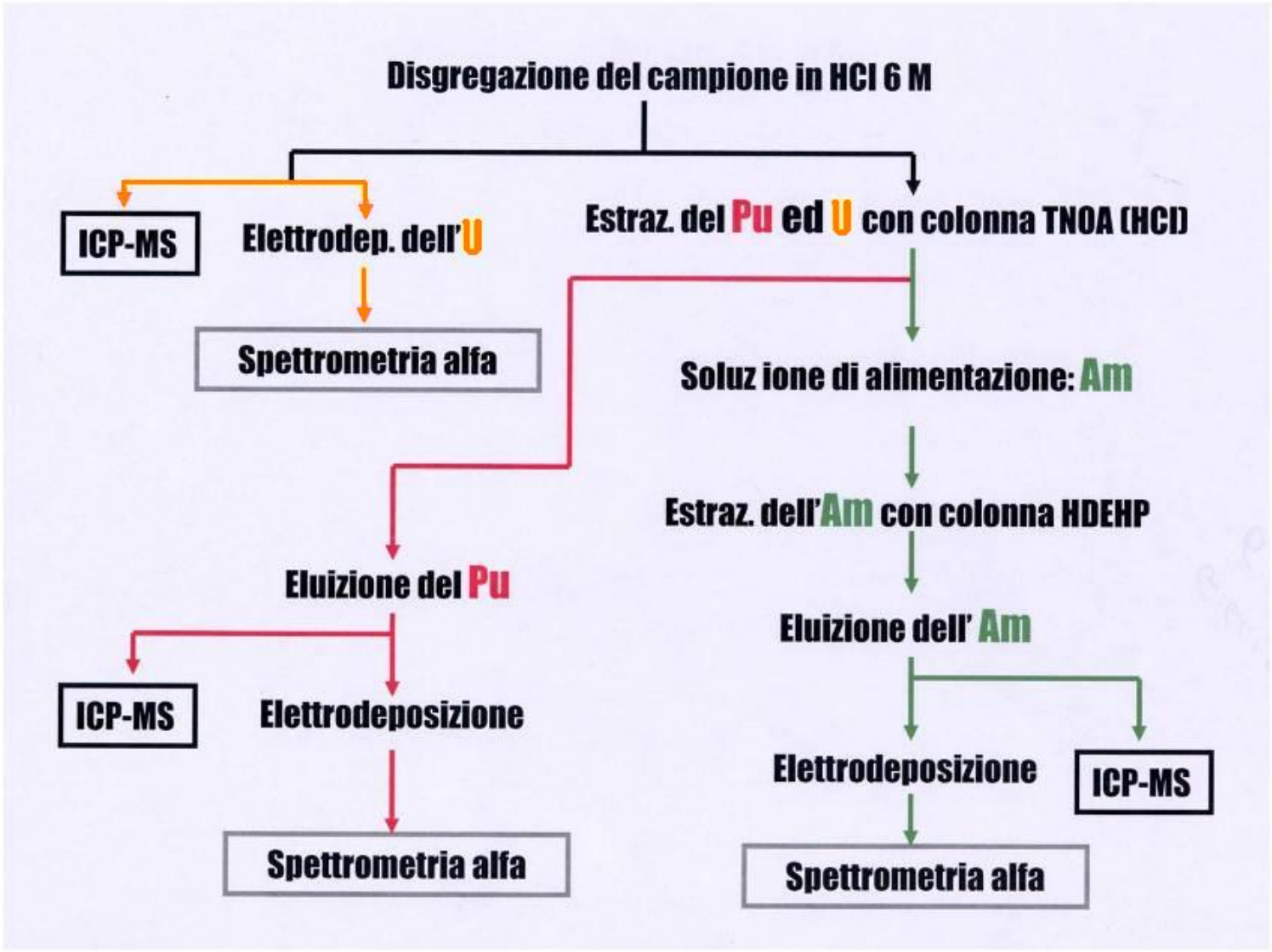
Convegno: "Il Sistema T.E.R.N.I. Research."
Workshop: "Progetti di ricerca applicata sulle radiazioni ionizzanti"

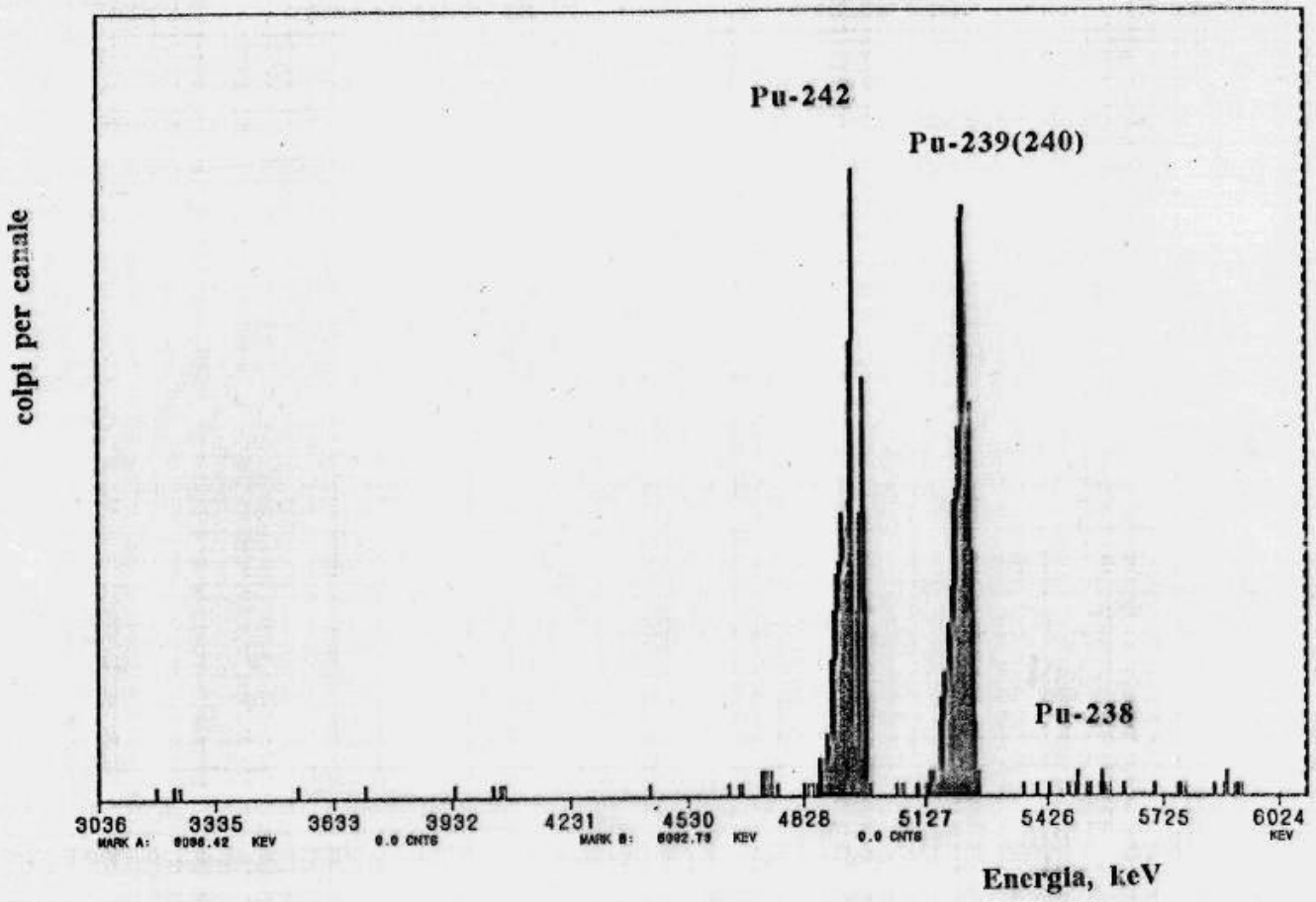
Narni, 10 Giugno 2005


$^{239(240)}$Pu, ^{238}Pu, ^{241}Pu
Aggiunta dello standard di resa ^{242}Pu e stabilizzazione stato di ossidazione (IV)
Estrazione cromatografica con scambiatore anionico in ambiente nitrico
Conteggio per Scintillazione Liquida ^{241}Pu
Elettrodeposizione e conteggio per spettrometria alfa $^{239(240)}$, ^{238}Pu
^{241}Am, ^{242}Cm, ^{244}Cm
Aggiunta dello standard di resa ^{243}Am e precipitazione degli ossalati
Estrazione cromatografica del Fe con scambiatore anionico in ambiente cloridrico
Estrazione cromatografica di Am e Cm con scambiatore cationico a pH 2,5
Elettrodeposizione e conteggio per spettrometria alfa ^{241}Am, ^{242}Cm, ^{244}Cm

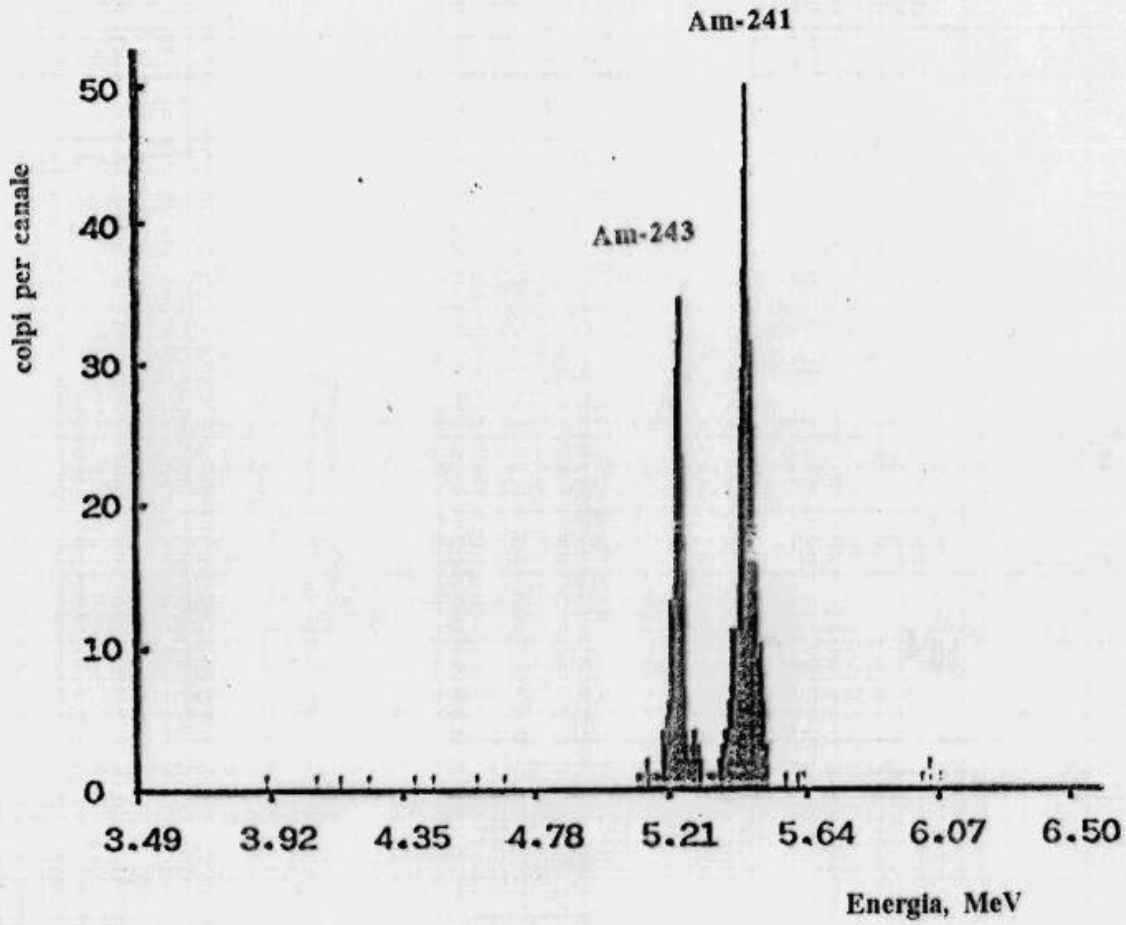

^{59}Ni, ^{63}Ni, ^{55}Fe
Aggiunta di Ni e Fe stabili come standards di resa e AAS
Estrazione cromatografica con scambiatore anionico in ambiente cloridrico
Colonna (Fe)
Soluzioni di caricamento e lavaggio (Ni)
Precipitazione come idrossido e dissoluzione
Precipitazione con dimetilgliossima e dissoluzione
Controllo resa chimica del Fe per AAS
Preparazione sorgente e conteggio X del ^{55}Fe
Scintillazione Liquida ^{59}Ni e ^{63}Ni
Controllo resa chimica del Ni per AAS
Preparazione sorgente e conteggio X del ^{59}Ni
^{90}Sr
Aggiunta di Y stabile come standard di resa e precipitazione degli ossalati
Estrazione cromatografica del Fe con scambiatore anionico in ambiente cloridrico
Estrazione cromatografica dell'Y con scambiatore cationico a pH 1
Precipitazione dell'Y come ossalato
Conteggio β basso fondo dell' ^{90}Y
Controllo resa chimica dell'Y per titolazione complessometrica


UNEP
Depleted Uranium
In Kosovo
Post-Conflict
Environmental
Assessment


Disgregazione del campione in HCl 6 M
ICP-MS
Elettrodep. dell'U
Spettrometria alfa
Estraz. del Pu ed U con colonna TNOA (HCl)
Soluz ione di alimentazione: Am
Estraz. dell'Am con colonna HDEHP
Eluizione dell' Am
Elettrodeposizione
ICP-MS
Spettrometria alfa
Eluizione del Pu
ICP-MS
Elettrodeposizione
Spettrometria alfa



Spettro alfa del plutonio



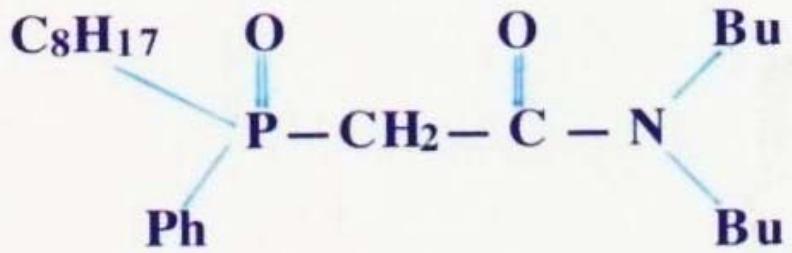
Spettro alfa dell'americio

EICHROM RESINS

Sr • Spec	**>**	**Sr - Pb**
Tru • Spec	**>**	**Attinidi e Lantanidi**
U/TEVA • Spec	**>**	**U, Th, Pu, Np**
TEVA • Spec	**>**	**Th, Pu, Np, Tc**

TRU • SPEC

Estraente:



[CMPO] [ottil (fenil)-N,N-diisobutilcarbametilfosfinaossido]

Diluente: Tri-n-butil fosfato

$Am^{3+} + 3\,NO_3^- + 3\,E \; < \; > Am(NO_3)_3 \cdot E_3$
(Ln^{3+})

$Pu^{4+} + 4\,NO_3^- + 2\,E \; < \; > Pu(NO_3)_4 \cdot E_2$
(Np^{4+})

$UO_2^{2+} + 2\,NO_3^- + 2\,E \; < \; > UO_2(NO_3)_2 \cdot E_2$

TEVA • SPEC

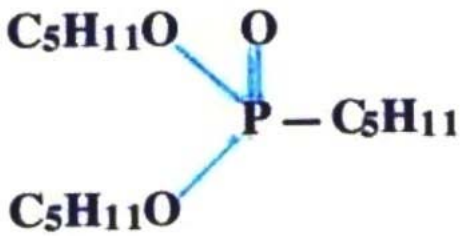
Estraente:

Diluente: nessuno

$$Pu^{4+} + 4\,NO_3^- + 2\,E \cdot NO_3 < \quad > E_2^{+} \cdot Pu(NO_3)_6^{2-}\ (Th^{4+}, Np^{4+})$$

U/TEVA • SPEC

Estraente:



DP[PP] Diamilamilfosfonato

Diluente: nessuno

$$UO_2^{2+} + 2\ NO_3^- + 2\ \overline{E} < \quad > UO_2(NO_3)_2 \cdot E_2$$

$$Th^{4+} + 4\ NO_3^- + 3\ \overline{E} < \quad > Th(NO_3)_4 \cdot E_3$$

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