RADIOCHEMICAL SEPARATION BY RETENTION ON IONIC PRECIPITATES

ADSORPTION TESTS ON 17 MATERIALS

by

F. GIRARDI, R. PIETRA and E. SABBIONI

1969

Joint Nuclear Research Center
Ispra Establishment - Italy
Chemistry Department
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ABSTRACT

The retention of different radio-ions on columns of seventeen ionic precipitates from different acid media was studied, in view of possible applications for radiochemical separations. The results of about 3,000 adsorption experiments, carried out in a standardized way, are presented schematically in periodic tables.

Twenty-one radiochemical separations, mainly in connection with activation analysis problems, were done to test the practical behaviour of potentially useful materials, and these are briefly described.

KEYWORDS

SEPARATION PROCESSES
IONS
EXTRACTION COLUMNS
PRECIPITATION
ACIDS
ADSORPTION
ACTIVATION ANALYSIS
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4. ACKNOWLEDGEMENTS
1. INTRODUCTION (*)

Inorganic ion exchangers have been applied for a long time in the nuclear energy field particularly when their outstanding resistance to high temperatures and high radiation doses make them preferable to organic ion exchangers (separation of fission products from spent fuel elements, deionization of reactor cooling water at high temperature). Many materials have been developed for such uses (1,2).

They have seldom been used for radiochemical separations applied to neutron activation analysis, despite the fact that a high selectivity for a few ions was often demonstrated.

In a few preceding works (3,4,5,6) we showed that the retention behaviour of many inorganic materials, not usually employed as ion exchangers, was interesting enough for many practical applications in destructive activation analysis.

Indeed, inorganic materials, not especially prepared as ion exchangers, can react with a solution containing trace ions with a number of possible reaction mechanisms, such as isotopic exchange, redox reactions, precipitation, formation of mixed crystals by recrystallization, and, of course, ion exchange. All these mechanisms can contribute to a different extent to the retention of different ions on a column of that material in an adsorption-elution experiment.

(*) Manuscript received on 14 May 1969.
Often, to clarify the actual retention mechanisms and describe them with comprehensive chemical laws can indeed be difficult; and the possibility of extrapolating the behaviour of ionic precipitates to different experimental conditions can be greatly hindered by the intervention of different reaction mechanisms in different media.

This does not prevent, however, the analyst from taking advantage of the unusual retention properties of many ionic precipitates, used in proper media, if he keeps the experimental conditions as close to the original ones as possible.

When a wanted chemical separation is still feasible under standardized working conditions, (the large variety of ionic precipitates which can be applied compensate for a more restricted choice of experimental conditions), then the limitation turns out to be an advantage, as the materials and columns can be prepared in advance and kept in stock and the execution can be left to less specialized personnel. The construction of automated machines can also be greatly simplified.

In this work about 3,000 adsorption-elution cycles were carried out in a standardized way over different ionic precipitates from various media for a preliminary screening of possible useful materials for radiochemical separations applied to activation analysis.
The results were reported schematically in a series of periodic tables. Possible analytical applications can be inferred from the behaviour of different ions in each couple column/medium, and work is being carried out to simplify this task. The experiments were carried out with radioactive tracers which were obtained by neutron activation in a nuclear reactor, with irradiation times ranging from minutes to three weeks, depending on the half-life of the radionuclide. The amount of tracer used was that required to obtain a reasonable counting rate on a gamma spectrometer, by counting the whole of the column or elution volumes. In this way the actual carrier concentration varies greatly from one radiotracer to another depending mainly on its activation cross section. While this is certainly a drawback for understanding the reaction mechanisms, in the practical analytical use it can be considered an advantage, as a relatively close simulation of a destructive activation analysis is thus obtained.

The application of the reported results to actual separation cases is, of course, not entirely straightforward.

The retention capacity of inorganic ion exchangers or ionic precipitates, in general, are usually lower than those of the organic exchangers, and moreover they vary considerably from ion to ion. For example, the retention capacity of hydrated antimony pentoxide for the alkaline ions in 0.1 M HNO₃ (in milliequivalents per gram)
has the following values: \( \text{Na}^+ : 2.61, \text{K}^+ : 1.7, \text{Rb}^+ : 1.45, \text{Cs}^+ : 0.15 \), (7).

As a consequence, ions for which the material has a low retention capacity can exhibit a different behaviour for different ion concentrations. Also ions present in extremely low concentrations can frequently show unusual retention or long elution "tails". The addition of 50-100 \( \mu \text{g} \) of carrier when elution is wanted is a recommended practice to avoid both difficulties.

Elements present in large concentrations (matrix elements) can also alter the behaviour of the exchanger, as it happens with organic exchangers.

Even with these limitations, we found that the behaviour indicated in the tables is very often exactly respected in many practical applications for activation analysis, where ion concentrations are often rather small. Together with the results of the retention experiments we briefly report the results of practical applications, when done.

This report is far from an exhaustive study of analytical applications of ionic precipitates, and indeed it is more an introduction to that study than a real scientific work. However, the possibility of applying directly the data as they are presented here to the development of separation schemes, and the interest at recent congresses arose from their use has convinced us that the presentation of these results was worth while.
The data presented here are not correlate with each other enough to make the detection of possible mistakes easy. The number of data is also high enough to make it possible and even probable the occurrence of such mistakes, even if most of the experiments were repeated at least twice. We shall be grateful to those, who will inform us of such errors.

1.1 References

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Int. Conf. Modern Trends in Activation Analysis Washington
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Unpublished results.
2. EXPERIMENTAL PROCEDURES

The ionic precipitates used in the present work were mostly of commercial origin and the name of the manufacturer and references on the preparation process, when available, are given for each individual material in the next paragraph. In table I the list and addresses of the manufacturers are given.

Most of the materials used in this work were previously known to exhibit ion exchange properties, or they had been briefly studied by us for some special purposes. Others were added on which no information on their possible application was available to us, but could be easily packed into columns with good flow-rate characteristics, and insoluble or only slightly soluble in the proposed medium. Other insoluble materials (SeS₂, Sb₂O₃, MoO₃, TiO₂, BaSO₄, Cr₂O₃, PbO₂, NiF₄, CuO, V₂O₅, CoWO₄, Cu₂O, Co₃O₄) were discarded after a few preliminary assays, as the particle size of the material available to us was too fine for the preparation of columns with good flow-rate behaviour.

Dowex 1 and Dowex 50 were also tested, both in view of their possible use together with inorganic exchangers, and to be used as controls of anion and cation exchange in the study of the reaction mechanisms.

The materials tested were used as received, occasionally after sieving to eliminate finer particles to improve the
elution flow-rate. Preliminary treatments only entailed washing the column with 5 ml of the eluting agent immediately before the adsorption step.

The radioactive tracers (except $^{22}$Na and $^{54}$Mn, which were of commercial origin), were prepared by neutron activation of the stable elements, in form of solid salts, in the Ispra 1 reactor. The irradiation times ranged from a few minutes to several weeks, depending on the activation cross section and the half-life of the radionuclide formed. The irradiated salts were then dissolved and stored, generally as a 1 M HCl solution.

The solutions for the adsorption step were prepared by taking aliquots from the mother solution (generally 10-100 $\lambda$) and diluting them to 30-50 ml with the required acid. From this second stock 5 ml fractions were taken for each tracer experiment.

The tracer used, and the approximate final carrier concentrations, are reported in table II.

The tracer experiments were carried out in the following way: disposable polyethilene columns, prepared by VEMOR (Monvalle, Varese, Italy) (figure 1) were used (internal diameter 7 mm). They include a 15 ml reservoir, and they can be easily stacked one on top of the other when a series of column is needed.

The columns were prepared by putting a quartz wool plug (or a teflon wool plug for the tracer experiments in HF) at the bottom and filling the column with the exchanger up to a 3 cm level.
A preliminary wash with 5 ml of the eluting solution was done before the adsorption step, to wet and settle the column bed. The adsorption step (5 ml) was then carried out, and then two successive 15 ml fractions were passed through. The eluted solutions were collected into two 20 ml polyethylene bottles, the first collecting the adsorption and first 15 ml fraction, the second collecting the successive 15 ml fraction. The second fraction was diluted to 20 ml and both bottles were then analyzed by gamma ray spectroscopy.

The column bed was transferred into a third bottle and analyzed in the same way. An experimental correction factor was used to relate the counting rate of the column to the counting rate of the eluted solutions.

Most of the countings of the tracer experiments were done with a 3" x 3" NaI(Tl) scintillator coupled to a 200 channel LABEN analyzer. In tracer experiment with pure β emitters (32P, 35S, 90Y, 233Th), counting was done with a Geiger counter. The sources were prepared by taking 1 ml of solution and drying it under an infrared lamp.

To count the columns, they were dissolved when possible and treated as the eluted solutions, otherwise they were carefully dried and stirred, and a weighed part was spread over the counting tray. The 1 ml aliquot from the eluted solution was then added to an identical weight of fresh exchanger, which was then stirred, dried and spread over a counting tray in the same way as described above.
Figure 1

WASHING 30 ml
SAMPLE 5 ml

IONIC PRECIPITATE (internal diameter 7 mm)

3 cm

QUARTZ OR TEFLOM WOOL
3. RESULTS

The results of the tracer experiments for each ionic precipitate in various media are reported in the following paragraphs, in form of periodic tables. The following codes were used:

- : element eluted (over 97%)
- : element retained (over 99%)
- : element partially retained (the black area is proportional to the amount retained)
: : behaviour not well reproducible
- : no disk: the element was not tested.

The tables are numbered successively, and the code words of the material and of the medium are indicated in brackets. (see table III for the keys of the code).

Each paragraph is completed by a summarized description of applications made.

Only the part referring to the separation conditions and the results obtained, is given. Details on the irradiation and counting conditions and on the destructive producers used, were generally omitted to avoid repetition of non-essential data.

Reference to published work on the material tested or on materials of similar chemical composition are given for each paragraph.
3.1 Hydrated Manganese Dioxide (HMD)

3.1.1 Material

Commercial HMD ("RA-2", Carlo Erba, Italy), prepared according to reference (19) was used.

3.1.2 Tracer experiments

Carried out in the following media:

a) 0.1 M HNO₃ at 65°C (61 ions): table 1 (HMD/0.1N 65°C)
b) 1 M HNO₃ (50 ions): table 2 (HMD/1N)
c) 1 M HClO₄ (51 ions): table 3 (HMD/1P)
d) 6 M HF (52 ions): table 4 (HMD/6F)
e) 14 M HNO₃ (51 ions): table 5 (HMD/14N)

3.1.3 Applications

HMD was applied to various radiochemical separations, such as removal of Fe and Cr for the determination of Zn and Co in a Fe-Cr alloy, activation analysis of mussel shells (removal of ⁴⁷Ca), and of leaf tissues (separation of Na from Mn and K) (19).

A Tc⁹⁹ᵐ generator based on the use of a HMD column, (20) and the application of HAP, HMD and TDO to the determination of chromium in blood were recently reported (21).
Other applications were:

a) Determination of Cu in steel (total removal of $^{187}$W and partial removal of $^{56}$Mn from 1 M HClO$_4$, determination of $^{64}$Cu in the eluate, figure 2)

b) Determination of Na, K, Mn, Zn, Cs, Ba, Mg, Sr in a fish muscle (see paragraph 3.8.3)

c) Preparation of carrier free Co$^{58}$ from neutron irradiated nickel by the reaction $^{58}$Ni(n,p)$^{58}$Co. Irradiated nickel in a 6 M HF solution was passed over HMD. $^{58}$Co is retained, and it can be eluted with 14M HNO$_3$. The concentration of Ni$^{++}$ in the solution must be lower than 1 mg/ml, or losses in the retention of $^{58}$Co are found.

3.1.4 HMD references

(references 8 to 18 refer to other types of manganese dioxide used as inorganic exchanger, references 19 to 22 refer to HMD in particular).

8) E.Ebler and W.Bender;

9) E.Ebler and W.Bender;
Angew.Chem. 28, I, 25 (1915).

10) N.J.Polissar;


16) V.V. Pushkarev, E.V. Tkachenko, Yv.V. Egorov and A.S. Lyubimov; Radiokhimiya, 4, 1 (1962).


18) P. Guegueniat; Report CEA-R 3284.


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   Int. Conf. Modern Trends in Activation Analysis
HYDRATED MANGANESE DIOXIDE

0.1 M HNO₃ at 65°C

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HYDRATED MANGANESE DIOXIDE

1 M HNO₃

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2(HMD/1N)
HYDRATED MANGANESE DIOXIDE

1 M HClO₄

6 M HF

HYDRATED MANGANESE DIOXIDE

3(HMD/1P)

4(HMD/6P)
HYDRATED MANGANESE DIOXIDE

14 M HNO$_3$

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Figure 2
3.2 Anhydrous Manganese Dioxide (AMD)

3.2.1 Material

AMD was prepared by courtesy of the firm Carlo Erba, Italy. The X-ray powder diffraction pattern corresponded to a pirolusite structure.

3.2.2 Tracer experiments

Carried out in the following media:

a) 1 M HClO₄ (50 ions): table 6 AMD/1P
b) 6 M HF (50 ions): table 7 AMD/6F
c) 14 M HNO₃ (51 ions): table 8 AMD/14N

3.2.3 Applications

a) Determination of Ag in mollusks (Octopus): $^{110m\text{Ag}}$ was fixed on AMD from 6M HF. Fe, Co and Zn were removed (figure 3).

b) Determination of Co and Cs in soil samples. The irradiated soil sample showed a complex $\gamma$-spectra in which $^{46}$Sc and the rare earths radioisotopes were prevailing. The irradiated sample (30 mg) in 5 cc of 6M HF solution was passed on a column of AMD coupled to one with cerous oxalate. The first retained the rare earths, the second retained $^{46}$Sc. $^{60}$Co and $^{137}$Cs were measured in the eluate.
### ANHYDROUS MANGANESE DIOXIDE

**1 M HClO₄**

- \( \text{Li, Be, Na, Mg, Al, Si, P, S, Cl} \)
- \( \text{K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br} \)
- \( \text{Rb, Sr, Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I} \)
- \( \text{Cs, Ba, R.E., Hf, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At} \)
- \( \text{Fr, Ra, Ac, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf} \)
- \( \text{La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu} \)

---

### ANHYDROUS MANGANESE DIOXIDE

**6 M HF**

- \( \text{Li, Be, Na, Mg, Al, Si, P, S, Cl} \)
- \( \text{K, Ca, Sc, Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Ga, Ge, As, Se, Br} \)
- \( \text{Rb, Sr, Y, Zr, Nb, Mo, Tc, Ru, Rh, Pd, Ag, Cd, In, Sn, Sb, Te, I} \)
- \( \text{Cs, Ba, R.E., Hf, Ta, W, Re, Os, Ir, Pt, Au, Hg, Tl, Pb, Bi, Po, At} \)
- \( \text{Fr, Ra, Ac, Th, Pa, U, Np, Pu, Am, Cm, Bk, Cf} \)
- \( \text{La, Ce, Pr, Nd, Pm, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu} \)
ANHYDROUS MANGANESE DIOXIDE

14 M HNO₃
Figure 3
3.3 Silicon Dioxide (SDO)

3.3.1 Material

Commercial Silicon dioxide ("R.P." Reagent grade, Carlo Erba, Italy) was used.

3.3.2 Tracer experiments.

Carried out in the following media:

a) 1M HClO₄ (49 ions): table 9 (SDO/1P)
b) 7M HNO₃ (49 ions): table 10 (SDO/7N)

3.3.3 Applications

SDO does not seem to have a practical interest, at least in the two media of the tracer experiments.
3.4 Hydrated Antimony Pentoxide (HAP)

3.4.1 Material

Commercial HAP (RA-1, Carlo-Erba, Italy) prepared according to reference (23), was used.

3.4.2 Tracer experiments

Carried out in the following media:

a) 1 M HNO₃ (36 ions): table 11 (HAP/1N)
b) 1 M HClO₄ (51 ions): table 12 (HAP/1P)
c) 6 M HCl (58 ions): table 13 (HAP/6Cl)
d) 6 M HClO₄ (36 ions): table 14 (HAP/6P)
e) 6 M HF (53 ions): table 15 (HAP/6F)
f) 7 M HNO₃ (32 ions): table 16 (HAP/7N)
g) 12 M HCl (60 ions): table 17 (HAP/12Cl)
h) 14 M HNO₃ (53 ions): table 18 (HAP/14N)

3.4.3 Applications

Various applications to the selective removal of radiosodium from neutron activated specimens were already reported (22, 23, 24, 25).

Other applications, involving the separation of other radionuclides, were:
a) Determination of Co, Rb, Sr, Sc, Zn, Fe, in mollusks.
   The irradiated sample in 1M HNO₃ with mg amounts of
carriers added for each element was passed on HAP.
Cs, Rb, Sr and Sc are retained. See figure 4.

b) Determination of Ta in Zr: based on the adsorption
   of Ta from 12 M HCl on HAP.
   Carrier-free Nb⁹⁵ from decay of Zr⁹⁵ was mostly
   retained on HAP. The addition of 5 mg Nb carrier
during the dissolution of the sample decreases the
   amount retained by a factor 10². See figure 5.

c) Determination of K, Cs, Sr and Cu in human lung
tissues: the irradiated sample is dissolved in
   fuming HNO₃ and directly passed on a HAP column.
The eluate is diluted to 1 M and passed on another
   HAP column (K, Cs, Sr retained; Cu and Mn eluted,
   see figure 6).

d) Determination of Cu and Zn in leaf tissues (see
   paragraph 3.17.3).

e) Determination of Na, K, Mn, Zn, Cs, Ba, Mg, Sr in
   fish muscle (see paragraph 3.8.3).

f) Preparation of carrier-free Nb⁹⁵ from decay of Zr⁹⁵.
   Neutron activated zirconium (100 mg) is dissolved
   and put in 20 cc of 14 M HNO₃ and passed on HAP. The
   retained Nb is eluted with 6M HF.
g) A Tc\(^{99m}\) isotope generator was formed by adsorbing irradiated Fe (15 mg) on HAP from 30 cc of 14 M HNO\(_3\). Tc\(^{99m}\) was "milked" 10 times successively with 14 M HNO\(_3\) and still no \(^{99m}\)Tc was detected in the eluate.

h) Preparation of carrier free Sc\(^{47}\) from the decay of Ca\(^{47}\). Neutron activated CaCO\(_3\) (1.25 g) was dissolved in 30 cc of 6 M HClO\(_4\) and passed on a PAP column. \(^{47}\)Sc is retained. Elution can be done with 6 M HF.

i) Determination of Mn and Cu in blood and mollusk shells: the irradiated material in 1 M HClO\(_4\) was passed in HAP (Na and K removed). \(^{64}\)Cu and \(^{56}\)Mn are determined in the eluate.

j) Determination of Fe, Cr, Zn in mollusk shells: the irradiated shell in 6M HClO\(_4\) was passed on HAP (\(^{46}\)Sc removed, \(^{59}\)Fe, \(^{51}\)Cr, \(^{63}\)Zn in the eluate).

m) Determination of Fe, Cr, Zn in blood (see paragraph 3.8.3).

n) Determination of Cu and Mn in bone tissue (see paragraph 3.8.3).

3.4.4 References


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1968, paper no 59.
HYDRATED ANTIMONY PENTOXIDE

6 M HCl

HYDRATED ANTIMONY PENTOXIDE

6 M HClO₄
HYDRATED ANTIMONY PENTOXIDE
6 M HF

HYDRATED ANTIMONY PENTOXIDE
7 M HNO₃
HYDRATED ANTIMONY PENTOXIDE
12 M HCl

HYDRATED ANTIMONY PENTOXIDE
14 M HNO₃
Figure 4

IRRADIATED SAMPLE (MOLLUSK)

COUNT

MINERALIZATION

1M HNO₃

COUNT

HAP

WASTE
Figure 5
Figure 6
3.5 Polyantimonic Acid (PAA)

3.5.1 Material

Commercial PAA (Applied Research, Belgium), was used. This material differs from HAP for a higher water content and a lower resistance to strong acid. By heating it to 270°C (drying temperature of HAP) a material identical to HAP is obtained.

3.5.2 Tracer experiments

Carried out in the following media:

a) 6 M HCl (33 ions): table 19 (PAA/6 Cl)

b) 7 M HNO₃ (32 ions): table 20 (PAA/7 N)

c) 14 M HNO₃ (32 ions): table 21 (PAA/14 N)

3.5.3 Applications

Its behaviour is similar to that of HAP. It has a higher retention capacity, but its lower resistance to high HCl and HClO₄ molarities narrows its area of application. No practical application was yet made.

3.5.4 References

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27) Jean Lefebvre;

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30) L.H.Baetsle, D.Huys;
POLYANTIMONIC ACID

6 M HCl

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19(PAA/601)

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POLYANTIMONIC ACID

7 M HNO₃

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20(PAA/7N)
POLYANTIMONIC ACID

14 M HNO₃

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21(FAA/14N)
3.6 Antimony tetroxide (ATO)

3.6.1 Material

ATO was obtained from hydrated antimony pentoxide by heating it at 800-850°C.

3.6.2 Tracer experiments

Carried out in the following media:

a) 1 M HNO₃ (36 ions): table 22 (ATO/1N)
b) 6 M HClO₄ (36 ions): table 23 (ATO/6P)
c) 6 M HF (36 ions): table 24 (ATO/6F)
d) 7 M HNO₃ (36 ions): table 25 (ATO/7N)
e) 10 M HCl (36 ions): table 26 (ATO/10Cl)
f) 14 M HNO₃ (36 ions): table 27 (ATO/14N)

3.6.3 Applications

No special advantages were found over HAP. The retention capacity is lower. No application was therefore made.
**ANTIMONY TETROXIDE**

1 M HNO₃

| Li | Be | Na | Mg | K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br |
|----|----|----|----|---|----|----|----|---|----|----|----|----|----|----|----|----|----|----|----|----|----|
|    |    |    |    |   |    |    |    |   |    |    |    |    |    |    |    |    |    |    |    |    |    |

**ANTIMONY TETROXIDE**

6 M HClO₄

| Li | Be | Na | Mg | K | Ca | Sc | Ti | V | Cr | Mn | Fe | Co | Ni | Cu | Zn | Ga | Ge | As | Se | Br |
|----|----|----|----|---|----|----|----|---|----|----|----|----|----|----|----|----|----|----|----|----|----|
|    |    |    |    |   |    |    |    |   |    |    |    |    |    |    |    |    |    |    |    |    |    |

**ANTIMONY TETROXIDE**

22(ATO/1N)

23(ATO/6P)
### ANTIMONY TETROXIDE

#### 6 M HF

![Periodic Table 6 M HF Image]

#### 7 M HNO₃

![Periodic Table 7 M HNO₃ Image]
ANTIMONY TETROXIDE
10 M HCl

ANTIMONY TETROXIDE
14 M HNO₃
3.7 Cadmium oxide (CDO)

3.7.1 Material

Commercial cadmium oxide (Reagent grade R.P., Carlo Erba, Milan) was used.

CDO is soluble in strong acids. It is only slightly soluble in 0.1 M acids.

3.7.2 Tracer experiments

Carried out only in 0.1 M HNO₃ (59 ions): table 28 (CDO/0.1N).

3.7.3 Applications

CDO was used in the determination of Cs, Sr, and Rb in soils (see 3.10.3) and mollusks (see 3.9.3).
CADMIUM OXIDE

0.1 M HNO₃

28(CDO/0.1N)
3.8 Acid Aluminium Oxide (AAO)

3.8.1 Material

Chromatographic acid aluminium oxide (activity grade 1, Woelm, Germany) was used.

3.8.2 Tracer experiments

Carried out in the following media:

a) 1 M HClO₄ (50 ions): table 29 (AAO/1P)
b) 1 M HClO₄ at 90°C (46 ions): table 30 (AAO/1P 90°C)
c) 1 M HNO₃ at 90°C (42 ions): table 31 (AAO/1N 90°C)
d) 7 M HNO₃ (48 ions): table 32 (AAO/7N)
e) 7 M HNO₃ at 90°C (42 ions): table 33 (AAO/7N 90°C).

3.8.3 Applications

AAO has been used frequently to prepare carrier-free ³²P from irradiated sulphur compounds. The selectivity obtained is good enough to make AAO a suitable filter for removing ³²P from irradiated samples (elimination of ³²P bremsstrahlung from gamma spectra).

Removal of ³²P was made:

a) From irradiated bone tissues, (5 mg) for the determination of Ba and Cr (in 5 cc of 1 M HClO₄ at 90°C), and for the determination of Cu and Mn (in 1 M HClO₄). An HAP column is connected in series to
b) From leaf tissues, for the determination of Hg and Cr (in HNO₃ 1 M and 7 M) (figure 7).

c) From fish muscle, for the determination of Na, K, Mn, Zn, Cs, Ba, Mg, Sr.

The irradiated sample, dissolved in fuming HNO₃ is directly passed on columns of AAO and HAP in series (removal of ³²P and ²⁴Na). The eluate is diluted to 0.1 M and passed on HMD at 70°C (removal of ⁵⁶Mn and ⁴²K). ⁶⁹mZn, 13⁴mCs, ⁷⁷Mg, ¹³⁹Ba and ⁸⁷mSr are determined in the eluate.

3.8.4 References

31) M.Bresesti, R.Lanz and A.M.Del Turco;

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   Report CEA - R 2851.

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ACID ALUMINIUM OXIDE

1 M HClO₄

ACID ALUMINIUM OXIDE

1 M HClO₄ at 90°C
ACID ALUMINIUM OXIDE
1 M HNO₃ at 90°C

ACID ALUMINIUM OXIDE
7 M HNO₃
ACID ALUMINIUM OXIDE

7 M HNO₃ at 90°C

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33(AAO/7N 90°C)
3.9 Tin dioxide (TDO)

3.9.1 Material

Commercial tin dioxide (Reagent grade RP, Carlo Erba, Milan) was used.

3.9.2 Tracer experiments

Carried out in the following media:

- a) 0.1 M HNO$_3$ at 60°C (41 ions): table 34 (TDO/0.1N 60°C)
- b) 1 M HCl (57 ions): table 35 (TDO/1 Cl)
- c) 1 M HNO$_3$ (59 ions): table 36 (TDO/1N)
- d) 1 M HClO$_4$ (47 ions): table 37 (TDO/1P)
- e) 6 M HF (46 ions): table 38 (TDO/6F)
- f) 7 M HNO$_3$ (56 ions): table 39 (TDO/7N)
- g) 14 M HNO$_3$ (46 ions): table 40 (TDO/14N)

3.9.3 Applications

TDO was applied to:

- a) Determination of Cs, Sr, Rb in mollusk soft tissues.

  The irradiated samples in 0.1 M HNO$_3$ are passed over TDO and CDO (removal of $^{59}$Fe, $^{51}$Cr, $^{34}$Sc, $^{65}$Zn, $^{122}$Sb and $^{32}$P). $^{134}$Cs, $^{85}$Sr and $^{86}$Rb are found in the eluate. (figure 8).
b) Determination of Mo in mollusk shells.

The samples in 1 M HNO₃ are passed over TDO, Mo is retained. Major radioactivities are eluted.

c) An application to the determination of Cr in blood was reported in (22).

3.9.4 References


38) M. Csajka; Talanta 14, 1360 (1967).


TIN DIOXIDE

0.1 M HNO₃ at 60°C

34(TDO/0.1N 60°C)

TIN DIOXIDE

1 M HCl

35(TDO/1Cl)
TIN DIOXIDE

14 M HNO₃

- 60 -

40(TDO/14N)
Figure 8

IRRADIATED SAMPLE (MOLLUSK)

MINERALIZATION

0.1 M HNO₃

TDO + CDQ

COUNT

$^{32}P$ bremsstrahlung

$^{65}Zn$

$^{85}Sr$

$^{134}Cs$

$^{134}Cs$

$^{86}Rb$

$^{60}Co$

$^{60}Co$

$E_γ (MeV)$

$10^2$

$10^3$

$10^4$

$10^5$

$10^6$

$cpm$

Figure 8
3.10 Titanium phosphate (TPH)

3.10.1 Material

Acid titanium phosphate for chromatographic applications (Abedem TiA 50-100 S.E.R.A.I., Belgium) was used. The preparation and properties of the material are described in reference (41).

3.10.2 Tracer experiments

Carried out in the following media:

a) 1 M \( \text{H}_2\text{SO}_4 \) (34 ions): table 41 (TPH/1S)
b) 6 M HCl (38 ions): table 42 (TPH/6 Cl)
c) 6 M HClO\(_4\) (38 ions): table 43 (TPH/6P)
d) 7 M \( \text{HNO}_3 \) (41 ions): table 44 (TPH/7N)

3.10.3 Applications

TPH was used with CDO to determine Cs, Sr and Rb in soil samples.

The irradiated sample (50 mg) in 5 cc of 0.1 M \( \text{HNO}_3 \) were passed on CDO (removal of major radioactivities), brought to 7 M \( \text{HNO}_3 \) and passed on TPH (\( ^{86}\text{Rb} \) and \( ^{134}\text{Cs} \) are measured on the column, \( ^{85}\text{Sr} \) in the eluate).

3.10.4 References

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42) Yvonne M. Jones;
    Report BNWL-270.

43) C.B. Amphlett;
    Geneve 1968 session C9 pp. 271.

44) G. Balon, C.A. Beaudet and J. Piret;

45) G. Alberti, G. Giammari and G. Grazzini-Strazza;
    J. Chromatog. 28, 118 (1967).

46) I.J. Gal and O.S. Gal;
TITANIUM PHOSPHATE

6 N HClO₄

7 N HNO₃
3.11 Zirconium phosphate (ZPH)

3.11.1 Materials

Two commercial products for chromatographic applications (ZP BioRad, USA, Zirconium phosphate "Applied Research SPRL", Belgium) were used.

3.11.2 Tracer experiments

Carried out in the following media:

a) $1 \text{ M } \text{H}_2\text{SO}_4$ on BioRad (35 ions): table 45 (ZPH BioRad/1S)
b) $6 \text{ M } \text{HCl}$ on BioRad (32 ions): table 46 (ZPH BioRad/6Cl)
c) $6 \text{ M } \text{HCl}$ on Appl.Res.(33 ions): table 47 (ZPH Appl.Res./6Cl)
d) $6 \text{ M } \text{HClO}_4$ on BioRad (33 ions): table 48 (ZPH BioRad/6P)
e) $6 \text{ M } \text{HClO}_4$ on Appl.Res.(34 ions): table 49 (ZPH Appl.Res./6P)
f) $7 \text{ M } \text{HNO}_3$ on BioRad (36 ions): table 50 (ZPH BioRad/7N)
g) $7 \text{ M } \text{HNO}_3$ on Appl.Res.(34 ions): table 51 (ZPH Appl.Res./7N)

3.11.3 Applications

ZPH has long been used for fission products separation, and much literature is available. No application has yet been made by us.
3. References

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   Energia Nucleare 9, 1 (1962).

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    Report AERE C/R 2755.

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   Report AERE-R-4006.
76) M.J. Riedel; 
77) J. Albertsson; 
78) Zsinka Laszlo, Szirtes Laszlo, 
79) S. Ahrland and J. Albertsson; 
80) E.M. Larsen and D.R. Vissers;  

81) G.H. Nancollas and V. Pekarek;  

82) A.M. Trofinov, G.N. Covalev and V.G. Pitalev;  

83) L. Baetsle, D. Van Deyck, D. Huys and A. Guery;  

84) P.A. Baron;  

85) J.R. Grover and B.E. Chidley;  
    The Industrial Chemist, January 1963.

86) Von Karl-Heinz Konig, H. Shafer and Fritz Hoyer;  
ZIRCONIUM PHOSPHATE (BioRad)

1 M H₂SO₄

45(ZPH BioRad/15)

ZIRCONIUM PHOSPHATE (BioRad)

6 M HCl

46(ZPH BioRad/6Cl)
ZIRCONIUM PHOSPHATE (Appl.Res.)

6 M HCl

ZIRCONIUM PHOSPHATE (BioRad)

6 M HClO₄
ZIRCONIUM PHOSPHATE (Appl.Res.)

6 M HClO₄

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N Ni Pd Pt Cm Dy
Cu Zn Ag Cd Au Hg
Bk Cf Ho Er

ZIRCONIUM PHOSPHATE (BioRad)

7 M HNO₃

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ZIRCONIUM PHOSPHATE (Appl. Res.)

7 M HNO₃

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3.12 Stannic Phosphate (SPH)

3.12.1 Material

Commercial SPH for chromatographic applications (Abedem Sn 50-100 S.E.R.A.I. Belgium) was used.

3.12.2 Tracer experiments

Carried out in the following media:

a) 1 M H₂SO₄ (33 elements): table 52 (SPH/1S)
b) 6 M HClO₄ (33 elements): table 53 (SPH/6P)
c) 7 M HNO₃ (34 elements): table 54 (SPH/7N)

3.12.3 Applications

The behaviour of tin phosphate seems less interesting than that of the Zr and Ti phosphate, at least in the limited amount of experiments done. No practical application was attempted.

3.12.4 References

87) J. Piret, J. Henry, G. Balon and C. Beaudet;

88) Yasushi Inove;

89) Yvonne M. Jones;
Report BNWL 270.

90) E. Merz;
STANNIC PHOSPHATE

7 M HNO₃

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54(SPH/7N)
3.13 Cupric sulphide (CUS)

3.13.1 Material

Reagent grade cupric sulphide (L.P., BDH, United Kingdom) was used. 15 ml of the eluent were used for the preliminary washing of the prepared column, to remove traces of copper sulphate formed by air oxidation of the sulphide.

3.13.2 Tracer experiments

Carried out in the following media:

a) 1 M H₂SO₄ (33 ions): table 55 (CUS/1S)
b) 6 M HCl (34 ions): table 56 (CUS/6Cl)
c) 6 M HClO₄ (34 ions): table 57 (CUS/6P)
d) 7 M HNO₃ (34 ions): table 58 (CUS/7N)

3.13.3 Applications

CUS is fairly selective for retaining Cu, Ag, Au, Se in most media.

Its behaviour is similar to that of CuCl (see 3.14).
CUPRIC SULPHIDE

1 M $H_2SO_4$

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CUPRIC SULPHIDE

6 M HCl

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3.14 Cuprous chloride (CUC)

3.14.1 Material

Reagent grade cuprous chloride ("Analar", BDH, United Kingdom) was used.

15 ml of eluent were used for the preliminary washing of the prepared columns, to remove traces of cupric chloride formed by air oxidation of cuprous chloride.

3.14.2 Tracer experiments

Carried out from the following media:

a) 1 M H₂SO₄ (34 elements): table 59 (CUC/1S)
b) 6 M HClO₄ (36 elements): table 60 (CUC/6F)
c) 6 M HF (36 elements): table 61 (CUC/6F)

3.14.3 Applications

CUC mainly differs from CUS by a different solubility in acid media.

It was used for the determination of copper:

a) in bones (15 mg): from 7 cc of 1 M H₂SO₄ (⁶⁴Cu measured on the column, ²⁴Na removed, a partial precipitation of Ca SO₄ did not affect the separation).
b) in aluminium (50 mg): from 5 cc of 6 M HCl.

(64Cu measured on the column, 24Na, 56Mn, 72Ga
eluted).
GUPROUS CHLORIDE

6 M HF

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61(CUC/6P)
3.15 Cadmium sulphide (CDS)

3.15.1 Material
Reagent grade CdS ("L.R.", B.D.H., United Kingdom) was used.

3.15.2 Tracer experiments
Carried out from 6 M HF (36 elements): table 62 (CDS/6F).

3.15.3 Applications
The behaviour of CDS does not seem particularly interesting. Among all materials which were tested, it was the only one which retained rhenium. No practical application was made.

3.15.4 References
CADMIUM SULPHIDE

6 M HF

62(CDS/6F)
3.16 Lead fluoride (LDF)

3.16.1 Material

Reagent grade PbF ("L.R.", grade, BDH, United Kingdom) was used.

3.16.2 Tracer experiments

Carried out in the following media:

a) 1 M H₂SO₄ (32 ions): table 63 (LDF/1S)
b) 6 M HF (32 ions): table 64 (LDF/6F)

3.16.3 Applications

LDF does not seem particularly interesting. No application was done.
3.17.1 Material

Technical grade cerous oxalate (B.D.H., United Kingdom), was used.

3.17.2 Tracer experiments

Carried out in the following media:

a) 1 M $\text{H}_2\text{SO}_4$ (37 ions): table 65 (COX/1S)

b) 6 M HF (39 ions): table 66 (COX/6F)

3.17.3 Applications

The following applications were done:

a) Determination of Cu and Zn in leaf tissues.

The sample in 6 M HF, was passed on columns of COX and HAP in series. The first retains $^{24}\text{Na}$ and $^{56}\text{Mn}$, the second retains $^{42}\text{K}$. $^{64}\text{Cu}$ and $^{69}\text{Zn}$ are determined in the eluate.

b) Determination of Co and Cs in soils (see 3.2.3).
CEROUS OXALATE

1 M $\text{H}_2\text{SO}_4$

CEROUS OXALATE

6 M HF

65(COX/1S)

66(COX/6F)
3.18 Anion exchange Resin (AER)

3.18.1 Material

Dowex 1 x 8 100-200 Mesh (BioRad, USA) was used. Preliminary conditioning was done with 15 ml of eluent.

3.18.2 Tracer experiments

Carried out in the following media:

a) 6 M HCl  (33 ions): table 67 (AER/6Cl)

b) 6 M HClO₄ (32 ions): table 68 (AER/6F)

c) 6 M HF  (33 ions): table 69 (AER/6F)

d) 7 M HNO₃ (32 ions): table 70 (AER/7N)

3.18.3 Applications

Anion exchange resins are at the base of many reported separation schemes. They couple very well with inorganic exchangers as their behaviour is often different. They afford therefore a means for removing elements which are not affected by most inorganic exchangers tested in the present work, such as Zn.

No application in connection with inorganic exchangers was yet done by us.
ANION EXCHANGE RESIN

6 M HCl

ANION EXCHANGE RESIN

6 M HClO₄
ANION EXCHANGE RESIN

6 M HF

7 M HNO₃
3.19 Cation Exchange Resin (CER)

3.19.1 Material

Dowex 50 x 8, 100-200 mesh (BioRad, USA) was used. Preliminary conditioning was done with 15 ml of eluent.

3.19.2 Tracer experiments

Carried out in the following media:

a) 6 M HCl (32 ions): table 71 (CER/6Cl)
b) 6 M HClO₄ (33 ions): table 72 (CER/6P)
c) 6 M HF (33 ions): table 73 (CER/6F)
d) 7 M HNO₃ (32 ions): table 74 (CER/7N)

3.19.3 Applications

The behaviour of cation exchangers in the experiments done is less interesting than that of the anion exchangers.

No application in connection with inorganic exchangers was yet done by us.
CATION EXCHANGE RESIN

6 M HCl

71(CER/6Cl)

CATION EXCHANGE RESIN

6 M HClO₄

72(CER/6P)
4. ACKNOWLEDGMENTS

The authors gratefully acknowledge the technical assistance of Ludovico Bianchi and Mirella Bianchi. Thanks are also due to Carla Bigliocca, Tommaso Candelieri, Marc Cuypers, Giampaolo Guzzi and Francis Mousty for suggestions and criticism.
TABLE I - Manufactures of ionic precipitates used in this work.

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TABLE II. Radiotracer used and carrier concentrations.

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(*) Uncertain oxidation state.
### Table III. Summary of experiments done and keys of the code used in indexing the tables.

The figures are the number of ions, whose behaviour was tested for the particular couple material/medium.

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| TOTAL                          |      | 102       | 59        | 57      | 181     | 42      | 298     | 46      | 238     | 294     | 345     | 445     | 496     | 42      | 16      | 60      | 269     | 3010    |
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Alfred Nobel
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