EUR 587.e

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

# THE CHROMATOGRAPHIC CONCENTRATION AND THE SPECTROGRAPHIC DETERMINATION OF TRACES OF CADMIUM, YTTRIUM, SCANDIUM AND LANTHANONS IN URANIUM METAL

by

Ch. A. BERTHELOT, S. HERRMANN, K.F. LAUER
and R.A.A. MUZZARELLI

1964

Joint Nuclear Research Center
Geel Establishment - Belgium

Central Nuclear Measurements Bureau - C.N.B.M.

## LEGAL NOTICE



The authors' names are listed in alphabetical order.

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The limits of detection are :

0.5 ppb for Yb and Sc ;
2 ppb for Dy, Er, Eu and Y ;
5 ppb for the other rare earth metals and Cd in uranium.

The problem of lanthanum in uranium when used as internal standard is discussed.

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## 1. INTRODUCTION

For certain measurements in nuclear physics, eg. pile oscillation, the concentration of cadmium and of some of the rare earths have to be known even if these elements are only present in parts per billion (ppb). As there is no direct method for determining such small amounts of impurities in presence of uranium, methods for separation and concentration of these trace elements have been developed.

Depending on the type of equipment available and the method used there is always a certain lower limit of detection for each element. It is best to express this lower limit in micrograms element detectable when using a specific procedure. This lower limit then fixes indirectly the quantity of uranium that has to be used for one determination. If one uses 10 g. of uranium for the separation we have at a concentration of 1 ppm in uranium a corresponding weight of $10^{-5}$ g. of trace element and at 1 ppb a corresponding weight of $10^{-8}$ g.

The present study was made in order to determine cadmium and rare earths in uranium with a limit of 0,5 ppb for Sc and Yb, a limit of 2 ppb for Dy, Er, Eu, Y and a limit of 5 ppb for the other rare earths and cadmium. The samples taken were : for the determination of cadmium 10 g. of uranium, and for the determination of rare earths 20 g. of uranium.

Studies concerning chemical absorption of rare earths by cellulose, (1, 2, 3, 4) have suggested that other cations could also be fixed by the acid groups of cellulose when it is in suitable solvents or has been treated with special reagents (5).

Several tests have therefore been made to verify this hypothesis and to see to what extent cadmium in uranium can be concentrated

and whether it is possible to provide suitable conditions for the spectrographic determination.

The method developed has the advantage of permitting determination of both cadmium and rare earths on the same cellulose ashes.

20 g. of the cellulose that was used gave on calcination at 500° C only 2 mg. of ashes. The impurities found in this ash are given in table I.

Milligram quantities of uranium that are not eluted from the cellulose column do not disturb the spectrographic measurements.

Table I

Impurities of 20 g. of cellulose treated with 50 ml. of sulfuric acid solution.

| $10^{-6}$ g. | elements |
|---|---|
| 6 | Fe |
| 1 | Pb |
| 0,6 | B, Cr, Mn |
| 0,3 | Cu |
| 0,2 | Al, Mg, Sn, Mo |
| not found | Cd, Y, Sc and Lanthanons |

# 2. EXPERIMENTAL

## 2.1. Determination of cadmium

### 2.1.1. Reagents for cadmium and rare earths

- Genuine Whatman ashless cellulose powder, chemically prepared, standard grade. Whatman ashless filter paper.
- Ether-nitric acid solution :
  Ethyl ether RP (Carlo Erba) containing 5 % (vol.) of bidistilled nitric acid (Merk).
- Sulfuric acid 1 : 10 (Merk).

### 2.1.2. Spectrographic products for cadmium

- Metallic cadmium p.A. (Merk)
- Zinc oxide (Specpure Johnson Matthey) : 1 g. in 1000 ml. of $H_2SO_4$ 1 : 10.

### 2.1.3. Apparatus

(see figure no. 1, Scale 1 : 2).

### 2.1.4. Preparation of the column

Place a porcelain filter disc into the bottom of the tube ; close the stopcock and add 200 cc of ether-nitric acid solution. Add a little cellulose powder ; allow the dispersed powder to settle and then press it down with the plunger to form a uniform pad free of air bubbles. Repeat this procedure until the pad is 5 cm high (20 g. of cellulose powder). Do not allow fibres to remain on the neck of the tube. Introduce a 4 cm. diameter filter paper in such a way that it forms a level and uniform upper surface of the pad. If some air is included in the cellulose column, it is impossible to obtain a good elution of uranyl nitrate.

As cations in aqueous medium are not quantitatively absorbed on cellulose, all organic solvents must be reasonably free of water.

2.1.5. Recovery of cadmium

The first experiments were aimed at showing that cadmium is fixed on the cellulose and that it is possible to recover it quantitatively after combustion. Although it is possible to elute the cadmium retained on the cellulose by means of 2 to 6 N, hydrochloric acid, combustion was prefered.

In fact, the elution of cadmium from the cellulose column using nitric and chloridric acids aqueous solutions was tried, but the recovery was not satisfactory.

2 ml. of cadmium solution in concentrated nitric acid (see table II) were added to 3 ml. nitric acid and 95 ml. of diethyl ether.

This solution was passed through the cellulose column and followed with 100 ml. of ether-nitric acid washing solution ; slight suction was applied to the bottom of the column to remove all the ether.

The cellulose was transfered to a platinum dish and 50 ml. of zinc-sulfuric acid solution were used to remove the small particles of cellulose from the walls of the column and were then poured into the dish so as to wet all the cellulose.

The cellulose was then dried at 120° C, burned at 300° C and ignited at 500° C. The sulfuric acid forms $CdSO_4$, the fusion point and vapour pressure of which insure good stability of the product during calcination at 500° C.

2.1.6. Spectrographic method for cadmium

The spectrographic standards are based on zinc oxide as spectroscopic matrix and contain 500-300-100-30-10-3 and 1 ppm of cadmium.

By using the above cited procedure the ashes and the zinc oxide are intimately mixed and the zinc oxide also stabilises the arc. The zinc lines are used as reference lines.
The moving-plate spectrograms show that all cadmium is volatilized after an arcing of 30 seconds ; we adopted however a longer exposure time (see figure no. 2).

2.1.7. Operating conditions for spectrochemical analysis of cadmium.

| | |
|---|---|
| spectrograph | Jarrell-Ash, 3.4 m Ebert mount |
| grating | 15.000 grooves/inch |
| dispersion | 5 Å mm ; 1st order |
| slit width | 10 μ |
| spectral range | 2200-4300 Å |
| line pairs (Cd < 1000 ppm in ZnO) | Cd - 2288.02 Å or Cd - 2288.02 Å<br>Zn - 2530,10 Å Zn - 2542.32 |
| (Cd < 500 ppm in ZnO) | Cd - 3261.06 Å or Cd - 3261.06 Å<br>Zn - 3276.55 Å Background |
| uniform illumination | 3-lens method with intermediate diaphragm |
| Type of carbon electrodes : | |
| 1. anode with a center post | Ringsdorff RWO no. 5 |
| 2. cathode | Carbone-Lorraine Ø 3,2 mm. |
| analytical gap | 5 mm. |
| sample weight | 50 mg. |
| standards zinc oxide base | (300, 100, 30, 10, 3 and 1 ppm) |
| excitation conditions | dc-arc 10 amperes |
| exposure time | 35 secondes |
| emulsion | Kodak S.A. 1 |
| development | J.A. Photoprocessor, 4 ½ mn. at 19.5° C. (Eastman D 19) |

emulsion calibration 3-step sector ($^{Io}/I = 3$)

The sensitivity of the method, is better than $5.10^{-8}$ g. of cadmium.

Characteristic results of such tests are shown in the table II :

2.1.8. Results

Table II

1 ml. Cd solution = $7,18.10^{-6}$ g. Cd.

| Cd $10^{-6}$ g. | | | |
|---|---|---|---|
| Calculated | found | Relative Difference % | Mean rel. diff. % |
| 1.065 * | 1.369 | + 22.2 | |
| 1.049 | 1.089 | + 4.5 | + 8.90 |
| 1.207 | 1.207 | 0 | |
| 3.491 * | 2.992 | - 16.7 | |
| 3.161 | 2.133 | - 21.1 | - 18.90 |
| 1.300 * | 1.400 | + 7.1 | |
| 1.307 | 1.508 | + 13.3 | + 10.20 |
| 2.750 * | 3.000 | + 9.1 | |
| 2.650 | 2.700 | + 1.9 | + 5.50 |
| | | Mean Dispersion | 10.87 |

* The ashes of one separation were quantitatively divided into 3 and 2 samples for spectrographic analysis.

## 2.2. Determination of yttrium, scandium and lanthanons

### 2.2.1. Description of the method common to cadmium and rare earths

A sample of about 30 g. of uranium metal, was placed in the fused silica container to dissolve the uranium.
This allows the use of a minimum quantity of bidistilled nitric acid. The solution was progressively transferred into an Erlenmeyer flask.
The solution is divided into two parts, one of which has double the weight of the other. The two fractions are crystallized separately by evaporating the solution in a current of nitrogen until some crystals appear ; slight stirring during the crystallisation helps the production of small crystals.
The bigger fractions serves for the determination of the rare earths. The uranyl nitrate is dissolved in 95 ml. of diethyl ether. Then 5 ml. of nitric acid are added. After having opened the stopcock, this solution is carefully poured into the column in such a way that the superficial strata of the cellulose are not disturbed.
The flow rate should be such that the drops are falling almost uninterrupted. When the level of the solution reaches the cellulose surface, add 120 ml. of ether nitric acid solution in several portions, beginning with very small fractions (2-3 ml.). Wash the sides of the column. Apply gentle suction to free the column as completely as possible from ether. Blow the cellulose by means of a rubber blowing bulb into a large platinum dish and add 50 ml. sulfuric acid 1 : 10. Mix the powder and the liquid, heat first at 50° C and then at 120° C.
Ignite in a muffle furnace at 500° C until all the carbonaceous matter has been removed. (Time : 2 - 3 hours).

### 2.2.2. Spectrographic products for rare earths

| | |
|---|---|
| = Europium oxide | Specpure Johnson Matthey |
| = Ytterbium oxide | Specpure Johnson Matthey |
| = Lanthanum oxide | Specpure Johnson Matthey |
| = Lithium Carbonate | Specpure Spex. |
| = Rare earths Spex. Mix. | (16 elements at 5.28 %) |

### 2.2.3. Spectrographic Method for Rare Earths

The rare earths Spex. Mix, is mixed with Specpure lithium carbonate to give the following concentrations : 500-100-50-10-5-2,5 and 1 ppm of Ce, Dy, Er, Eu, Gd, Ho, Lu, La, Nd, Pr, Sm, Sc, Tb, Tm, Yb and Y. To determine the sensivity of the lines suitable for quantitative micro-photometry, the standards are then volatilized in a 10 d.c. arc, and their spectra photographed with a Jarrel-Ash 3,4 m. Ebert grating spectrograph working in the $1^{st}$ and $6^{th}$ order. (6).

Table III gives the lines of each of the rare earths suitable for quantitative spectrochemical analysis.

## Table III

### Rare earths lines suitable for quantitative spectrochemical analysis

| Element | Wavelength (Å) | Concentration limit for quantitative micro-photometry ($10^{-8}$ g) |
|---|---|---|
| Lanthanum | La II - 3949.11 | 10 |
| | La II - 3988.52 | 10 |
| Cerium | Ce II - 4040.76 | 10 |
| Praseodymium | Pr II - 4179.42 | 10 |
| | Pr II - 4225.33 | |
| Neodymium | Nd II - 4303.58 | 10 |
| Samarium | Sm II - 3634.29 | 10 |
| | Sm II - 4424.34 | 10 |
| | Sm II - 4434.32 | 10 |
| Europium | Eu II - 3971.96 | 4 |
| | Eu I - 4627.22 | 4 |
| | Eu I - 4661.88 | 10 |
| Gadolinium | Gd II - 3081.99 | 10 |
| | Gd II - 3350.47 | 10 |
| | Gd II - 3422.47 | 10 |
| | Gd II - 3646.19 | 10 |
| Terbium | Tb II - 3676.35 | 10 |
| | Tb II - 3702.85 | 10 |
| Dysprosium | Dy II - 3645.42 | 10 |
| | Dy I - 4045.99 | 4 |
| Holmium | Ho II - 3398.98 | 10 |
| | Ho II - 3891.02 | 10 |
| Erbium | Er II - 3372.76 | 4 |
| | Er I - 4007.97 | 4 |
| Thulium | Tm II - 3462.20 | 10 |
| | Tm I - 3717.92 | 10 |
| | Tm II - 3761.33 | 10 |

| Element | Wavelength (Å) | Concentration limit for quantitative microphotometry ($10^{-8}$ g.) |
|---|---|---|
| Ytterbium | Yb II - 3289.37 | 1 |
| | Yb II - 3694.19 | 4 |
| | Yb I - 3987.98 | 1 |
| Lutetium | Lu II - 2615.42 | 10 |
| | Lu II - 2911.39 | 10 |
| | Lu I - 3359.56 | 10 |
| Scandium | Sc I - 3911.81 | 4 |
| | Sc I - 4020.40 | 1 |
| | Sc I - 4023.69 | 1 |
| Yttrium | Y II - 3242.28 | 10 |
| | Y II - 3600.73 | 10 |
| | Y II - 3633.12 | 4 |
| | Y II - 3710.30 | 4 |

After ignition of the activated cellulose, the ashes are ground and mixed with specpure lithium carbonate and lanthanum oxide. The mixture is then volatilized in a d.c. arc between graphite electrodes. The choice of the base substance, as spectrographic matrix, is very important for the intensity of the background and cyanogen bands.

## 2.2.4. Operating conditions for spectrochemical analysis of rare earths

| | |
|---|---|
| Spectrograph | Jaco Ebert 3,4 m. |
| grating | 7,500 grooves/inch |
| dispersion | 1.6 Å / mm ($6^{th}$ order) |
| slit width | .010 mm |
| weight of sample | 10 mg. or 20 mg. |
| graphite preformed electrodes | National Spectroscopic Products |
| 1. lower electrode (anode) | AGKSP - L 4006 (10 mg.) |
| | or |
| | " L 4000 (20 mg.) |
| 2. upper electrode | " L 4036 |
| arc gap | 5 mm. |
| d.c. arc current | 10 amperes |
| complete burn | 45 secondes |
| spectroscopic standards | 333-100-33-11- 3.6 and 1.2 ppm |
| Li carbonate base | of 15 rare earth elements |
| internal standard | 1000 ppm of La in the standards |
| | and in the sample |
| photographic plates | Kodak S.A. 1 |
| emulsion calibration | 3-step sector ($I_{o/I}$ = 3) |
| development | J.A. photoprocessor, Eastman D 19 |
| | 4 ½ mn. at 19.5° C. |

### 2.2.5. The problem of lanthanum in uranium, when lanthanum is used as internal standard.

The great similarity in physical and chemical properties of most of the rare earth metals (volatilization behavior, dissociation energy of the oxides, excitation and ionization potentials, rate of diffusion in the discharge column...) suggests that lanthanum can be used as internal standard to compensate to a high degree the uncontrolled variations in excitation conditions and the wandering of the cathode spot. Lanthanum is also chosen, because the spectrum of lanthanum is simpler than the spectrum of other rare earth elements, with exception of europium, thulium, ytterbium and lutetium (7).

Analytical line pairs were selected to satisfy the rules of spectrochemical analysis :

a. the two lines have approximately the same wavelength.

b. " " " " " " " excitation potential.

For example, analytical line pairs for 6 rare earths, their excitation potentials and their limit of detection are tabulated in Table IV.

Table IV

| Line pairs Å | Excitation potentials (eV) | | | Limit of detection (µg) for 10 mg of charge in a L-4006 electrode |
|---|---|---|---|---|
| Sc I - 4020.40 | 3.08 | | | 0.01 |
| La I - 4015.39 | 3.22 | | | |
| Y II - 3710.30 | 6.38 + 3.52 | = | 9.90 | less than 0.04 |
| La II - 3735.85 | 5.61 + 3.54 | | 9.15 | |
| Eu II - 3971.96 | 5.67 + 3.33 | = | 9.00 | 0.04 |
| La II - 3949.10 | 5.61 + 3.54 | | 9.15 | |
| Dy II - 4045.99 | 6.2 + >3.0 | => | 9.20 | 0.04 |
| La II - 4042.91 | 5.61 + 3.99 | | 9.60 | |
| Er I - 4007.97 | ? | | | 0.04 |
| La I - 4015.39 | 3.22 | | | |
| Yb I - 3987.99 | 3.11 | | | less than 0.01 |
| La I - 4015.39 | 3.22 | | | |
| Yb II - 3289.37 | 6.2 + 3.77 | = | 9.97 | 0.01 |
| La II - 3337.49 | 5.61 + 4.11 | | 9.72 | |

We found however, that the intensity ratios

Yb I - 3987.99 / La II - 3988.52 , Er I - 4007.97 / La II - 4031.69 and Sc I - 4020.40 / La II - 3995.75 were also consistent.

For separation of trace amounts of scandium, yttrium and lanthanons from 20 g. uranium metal, the spectrochemical method gives a limit of detection in uranium of :

0.5 ppb for Sc and Yb.

2 " " Dy, Er, Eu and Y.

5 " " the other rare earths metals in uranium.

Because of its low neutron cross section lanthanum can be tolerated in uranium if its concentration is less than 500 ppm. This is a concentration which can easily be analysed without separation. Besides of this, natural pure uranium never contains such quantities of lanthanum.

The lanthanum used for the preparation of the standards contains less than 50 ppm of praseodymium and its utilization introduces therefore about 0.05 ppm of praseodymium into the sample (lithium carbonate mixture) to be analysed. As for praseodymium the tolerance limit calculated by using its neutron cross section also lies at about 400 ppm in uranium, we can neglect this amount in our case. The concentration limit calculated for a contribution in neutron absorption of less than 1/1000 of the absorption of thermal neutrons by natural uranium is :

$$\frac{N_i}{N_U} \leqslant 10^{-3} \cdot \frac{s_U}{s_i} \qquad \text{or} \qquad \text{concentration (ppm)} \leqslant 10^{3} \cdot \frac{A_i}{A_U} \cdot \frac{s_U}{s_i}$$

$N_i$ = Number of atoms of element i.

Table V gives the tolerance limit for lanthanum and praseodymium in natural uranium.

Table V

| Element | cross-section s barns (2200 m/s) | atomic weight A | concentration limit (ppm) |
|---|---|---|---|
| Uranium | 7.68 | 238 | |
| Lanthanum | 8.9 | 138.9 | 504 |
| Praseodymium | 11.3 | 140.9 | 403 |

Lanthanum can also be replaced by yttrium as internal standard (8) or by any other rare earth (9).

### 2.2.6. Recovery of europium and ytterbium. Results.

Characteristic results of such tests are shown in table VI and VII.

Table VI - Europium

| Analytical lines : Eu II - 3971.96 Å / La II - 3949.10 Å<br>1 ml. Eu solution = $3.75.10^{-6}$ g. Eu. | | |
|---|---|---|
| Eu $10^{-6}$ g. | | |
| calculated | found | Relative difference % |
| 1.777 | 1.408 | + 16.4 |
| 1.162 | 1.056 | - 9.1 |
| 1.830 | 1.893 | + 3.4 |
| | Mean dispersion | 9.6 % |

## Table VII - Ytterbium

1 ml. Yb solution : $1,12.10^{-6}$ g. Yb

Analytical lines Yb II - 3289.37 Å

La II - 3337.49 Å

| Yb $10^{-7}$ g. | | |
|---|---|---|
| calculated | found | relative difference % |
| 3.6298 | 4.0835 | + 12.50 |
| 3.6298 | 3.1760 | - 12.50 |
| 3.9562 | 3.4907 | - 11.70 |
| 3.9562 | 3.4907 | - 11.70 |
| | | Mean dispersion 12.1 % |

The method of arcing the sample to completion in absence of lanthanum as internal standard, gives results that have somewhat greater variation.

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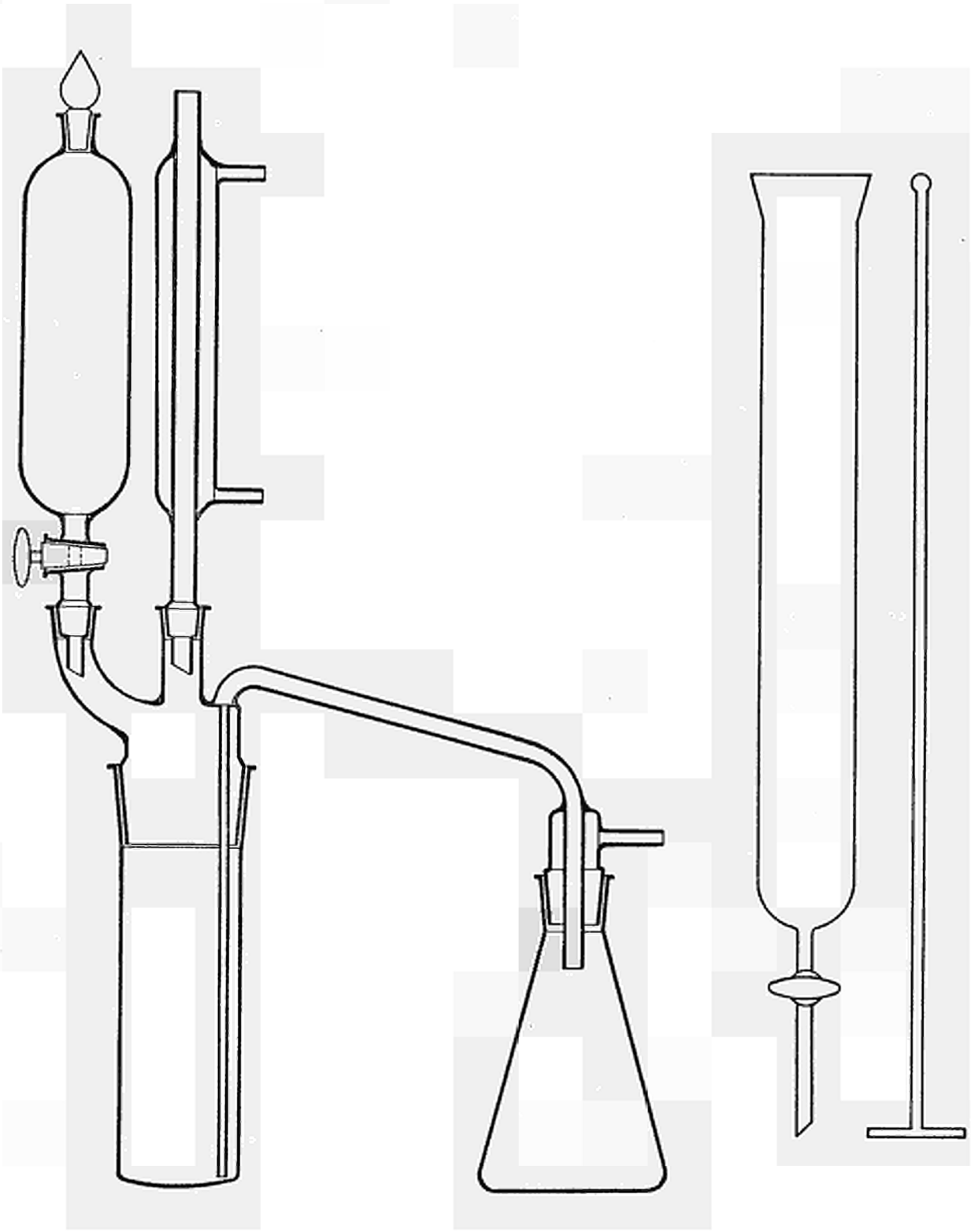

Fig.1 Apparatus for dissolution of Uranium metal, and chromatographic column.

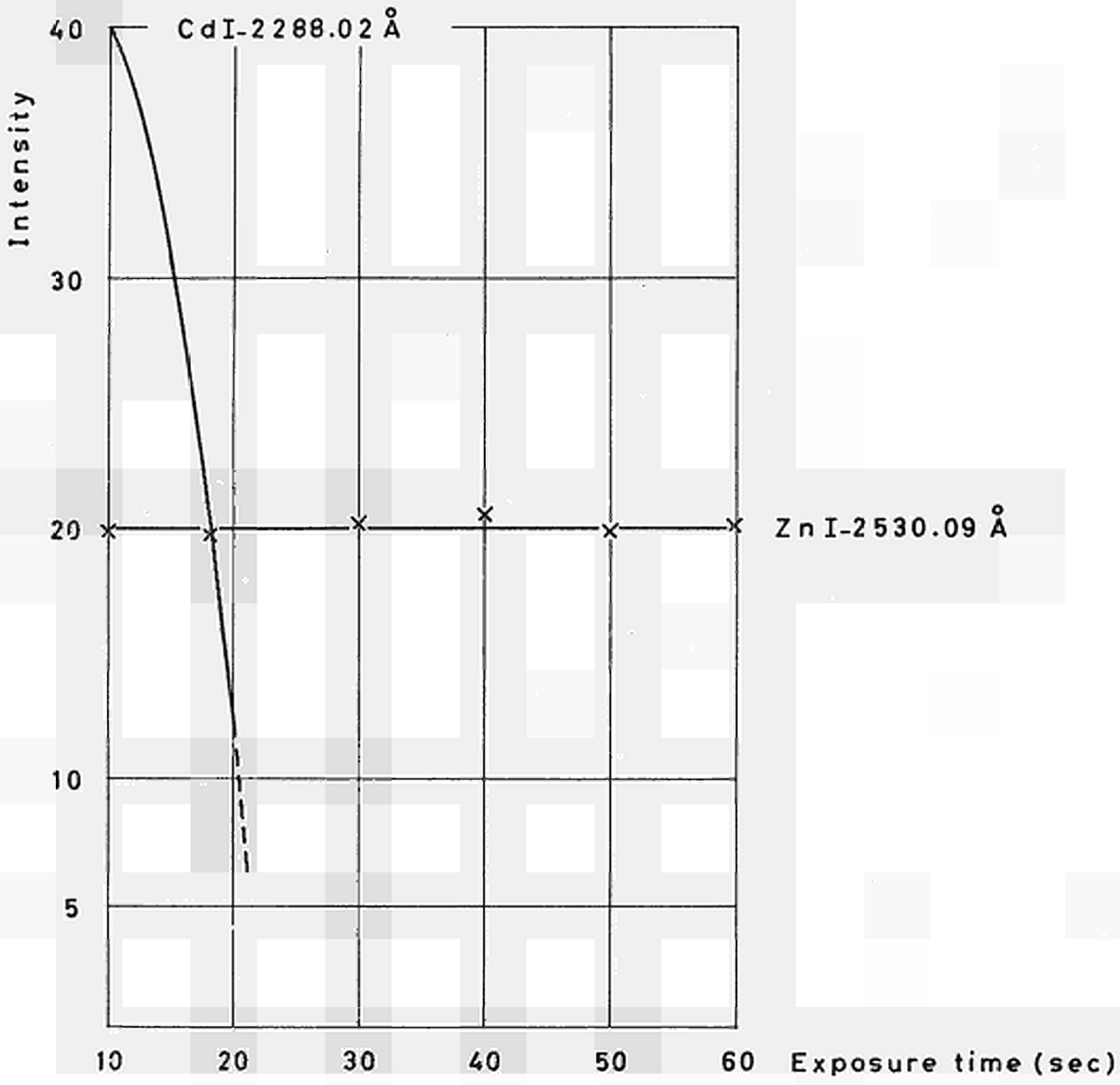


Fig.2 Variation of line intensity during the vaporization period for 500 ppm of Cd in ZnO ( dc-arc 10 amperes with preformed center-post electrode Ringsdorff RWO )

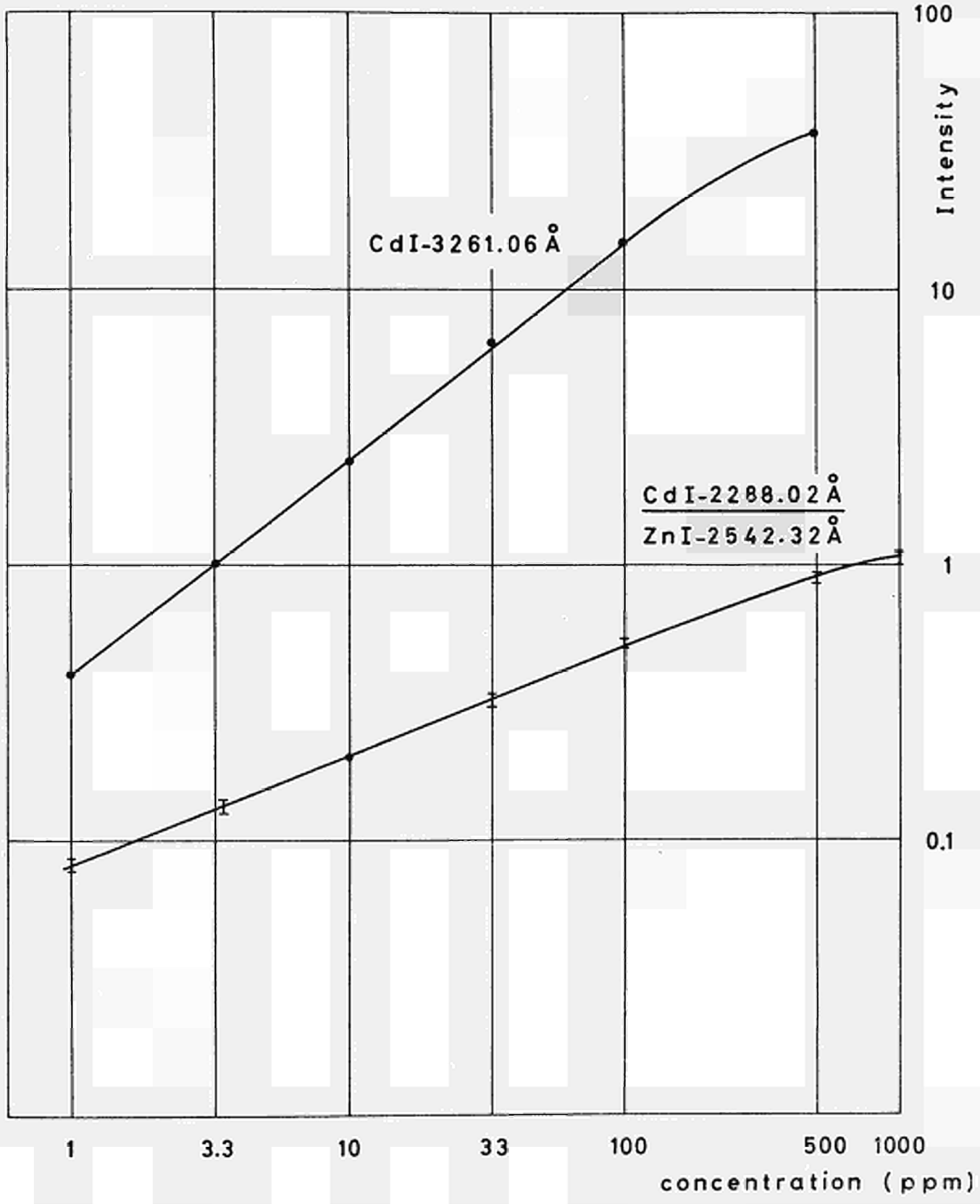


Fig. 3 Analytical curves for the determination of Cadmium.

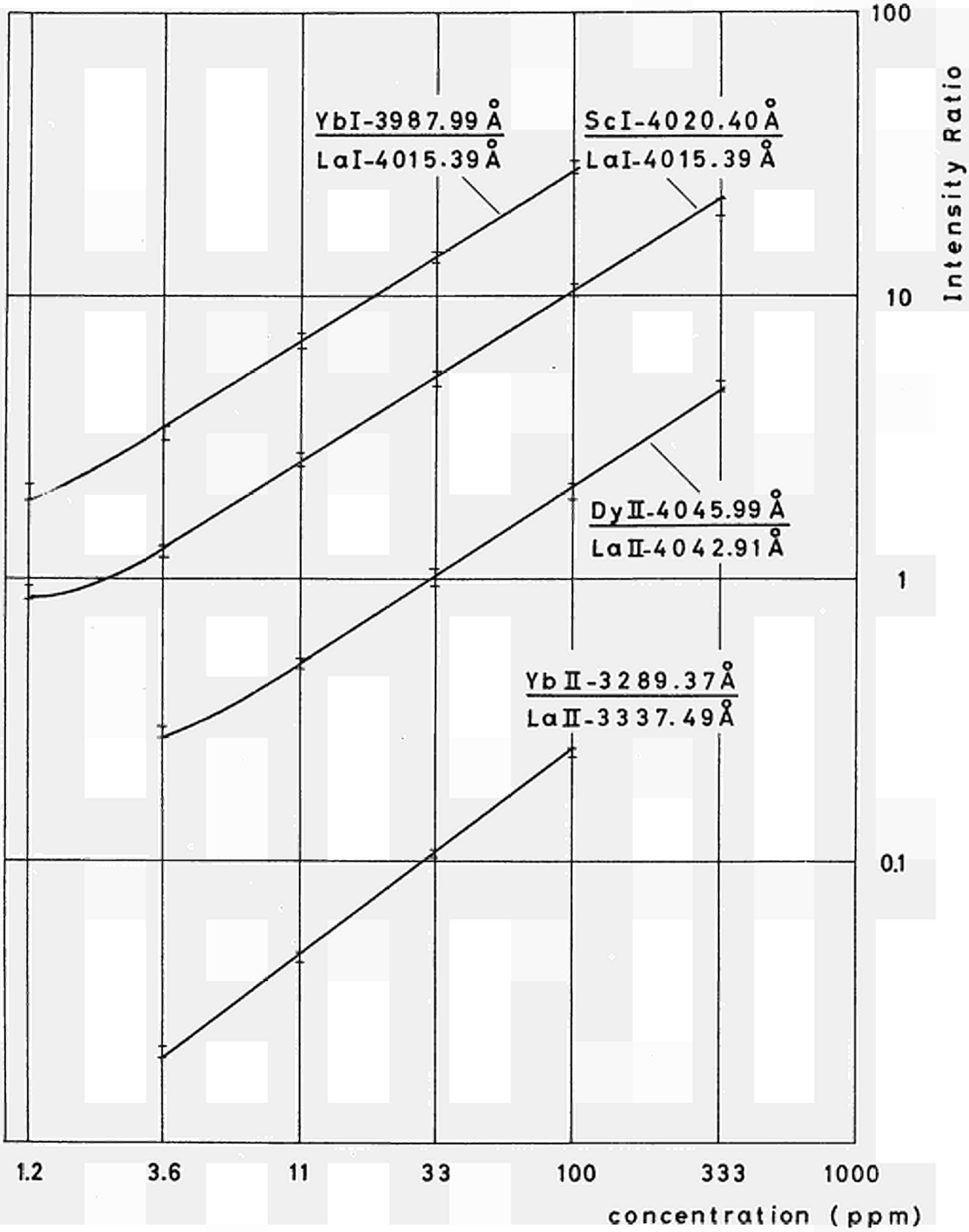


Fig.4 Analytical curves for the spectrographic determination of Ytterbium, Scandium and Dysprosium with Lanthanum as internal standard (1000 ppm in $Li_2Co_3$).

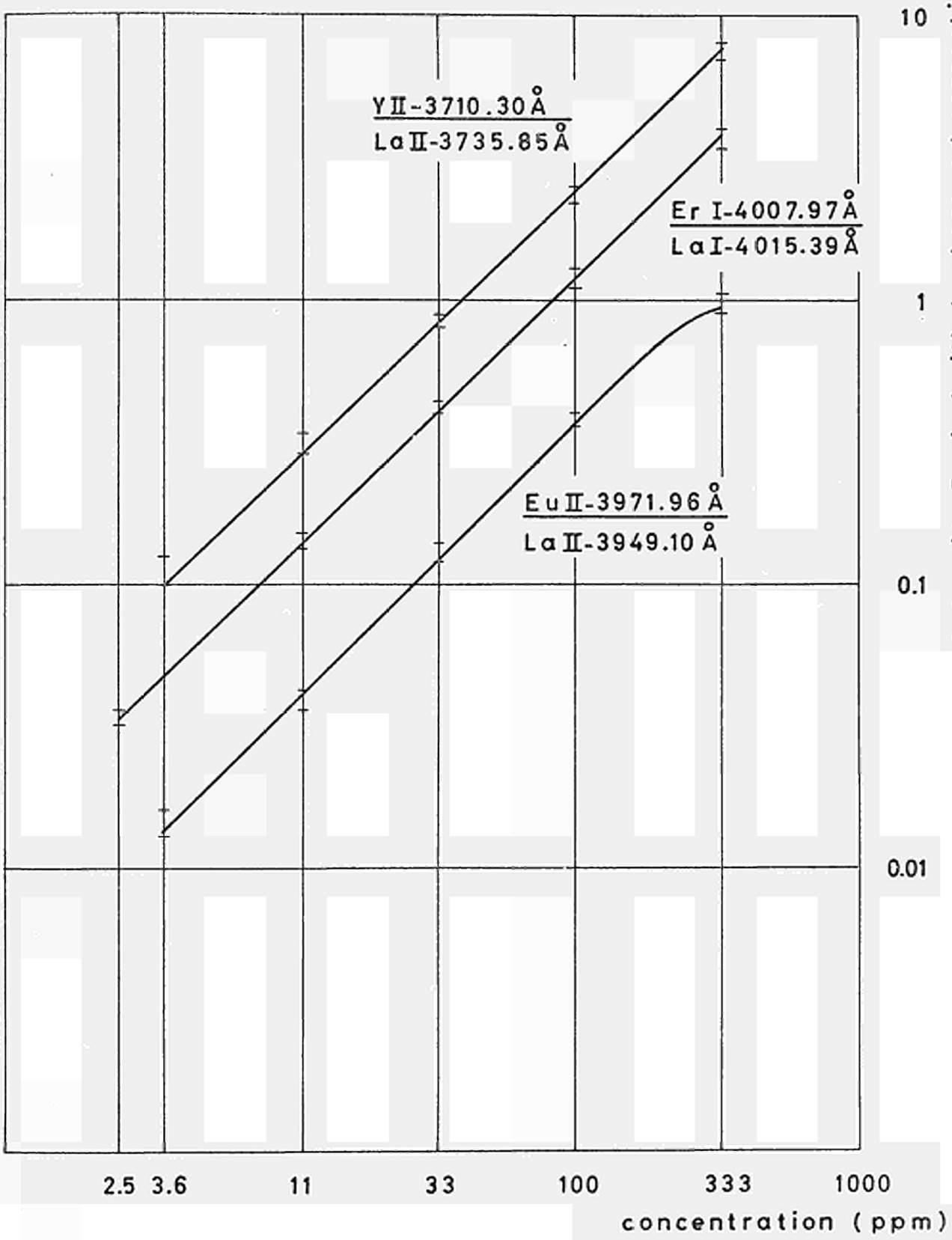


Fig. 5 Spectrographic determination of Yttrium, Erbium and Europium in Lithium carbonate with Lanthanum as internal standard (1000 ppm in $Li_2Co_3$)