HD193

# TRU•Spec and RE•Spec Chromatography: Basic Studies and Applications

**Edmund A. Huff and Doris R. Huff**

Analytical Chemistry Laboratory

Chemical Technology Division

Argonne National Laboratory

# OBJECTIVES

- Determine Distribution Coefficients on TRU•Spec and RE•Spec columns as a function of hydrochloric acid and nitric acid concentrations.
- Apply Distribution Coefficient data to separations of Nuclear Fuel Cycle materials.
- Apply Distribution Coefficient data to separations in the Nuclear Waste and Environmental areas.
- Compare Nuclear Waste Glass dissolution procedures.

# Determination of Distribution Coefficients ($K_d$'s)

- 0.5 g of TRU•Spec or RE•Spec resin were packed into a 10 mL buret by gravity flow.
- Element standards, adjusted to the appropriate acid concentration, were added to the top of the column.
- Solution fractions were collected sequentially.
- Fractions were analyzed by ICP-AES.
- Distribution coefficients were calculated from the equation $K_d$ = **V/m**, where V represents the volume in mL at which an element appears at its maximum concentration (corrected for the interstitial column volume) and m is the weight of the dry packing in grams.

# Extraction Phase Composition

| | CMPO[1], % | TBP[2], % |
|---|---|---|
| TRU•Spec | 13 | 27 |
| RE•Spec | 16 | 24 |

[1]CMPO: Octyl(phenyl)-N,N-diisobutylcarbamoylmethylphosphine oxide

[2]TBP: Tributyl phosphate

# TRU•Spec Column: $K_d$'s as a Function of Nitric Acid Concentration

| Element | Nitric Acid Concentration | | | |
|---|---|---|---|---|
| | 0.2 N | 1 N | 4 N | 8 N |
| Al | <5 | <5 | <5 | <5 |
| Ba | <5 | <5 | <5 | <5 |
| Be | <5 | <5 | <5 | <5 |
| Ca | <5 | <5 | <5 | <5 |
| Cd | <5 | <5 | <5 | <5 |
| Co | <5 | <5 | <5 | <5 |
| Cr | <5 | <5 | <5 | <5 |
| Cu | <5 | <5 | <5 | <5 |
| Fe | <5 | 5 | 45 | >400 |
| K | <5 | <5 | <5 | <5 |
| Li | <5 | <5 | <5 | <5 |
| Mg | <5 | <5 | <5 | <5 |
| Mn | <5 | <5 | <5 | <5 |
| Mo | 8 | 20 | 28 | 180 |
| Na | <5 | <5 | <5 | <5 |
| Ni | <5 | <5 | <5 | <5 |
| Pd | 7 | 5 | <5 | <5 |
| Pb | <5 | <5 | <5 | <5 |
| Rh | <5 | <5 | <5 | <5 |
| Ru | <5 | <5 | <5 | <5 |
| Sn | <5 | <5 | 6 | 14 |
| Sr | <5 | <5 | <5 | <5 |
| Te | <5 | <5 | <5 | <5 |
| Ti | <5 | 7 | 42 | >400 |
| Th | >400 | >400 | >400 | >400 |

| | | | | |
|---|---|---|---|---|
| U | >400 | >400 | >400 | >400 |
| V | <5 | <5 | 5 | 36 |
| Zn | <5 | <5 | <5 | <5 |
| Zr | >400 | >400 | >400 | >400 |

## TRU-Spec Column: $K_d$'s as a Function of Nitric Acid Concentration

| Element | Nitric Acid Concentration | | | |
|---|---|---|---|---|
| | 0.2 N | 1 N | 4 N | 8 N |
| Ce | 50 | 175 | 145 | 75 |
| Dy | 10 | 35 | 64 | 120 |
| Er | <10 | 15 | 40 | 110 |
| Eu | 40 | 100 | 135 | 125 |
| Gd | 16 | 65 | 80 | 100 |
| Ho | <10 | 25 | 50 | 110 |
| La | 35 | 90 | 80 | 35 |
| Nd | 50 | 175 | 145 | 85 |
| Pr | 60 | 175 | 150 | 80 |
| Sm | 50 | 165 | 155 | 120 |
| Tb | 15 | 45 | 72 | 115 |
| Tm | <10 | 15 | 40 | 110 |
| Y | <10 | 17 | 36 | 85 |

# RE•Spec Column: $K_d$'s as a Function of Nitric Acid Concentration

| Element | Nitric Acid Concentration | | | |
|---|---|---|---|---|
| | 0.2 N | 1 N | 4 N | 8 N |
| Al | <5 | <5 | <5 | <5 |
| Ba | <5 | <5 | <5 | <5 |
| Be | <5 | <5 | <5 | <5 |
| Ca | <5 | <5 | <5 | <5 |
| Cd | <5 | <5 | <5 | <5 |
| Co | <5 | <5 | <5 | <5 |
| Cr | <5 | <5 | <5 | <5 |
| Cu | <5 | <5 | <5 | <5 |
| Fe | <5 | 5 | 160 | >400 |
| K | <5 | <5 | <5 | <5 |
| Li | <5 | <5 | <5 | <5 |
| Mg | <5 | <5 | <5 | <5 |
| Mn | <5 | <5 | <5 | <5 |
| Mo | 24 | 44 | 150 | 370 |
| Na | <5 | <5 | <5 | <5 |
| Ni | <5 | <5 | <5 | <5 |
| Pb | <5 | <5 | <5 | <5 |
| Sn | <5 | <5 | <5 | 10 |
| Sr | <5 | <5 | <5 | <5 |
| Ti | <5 | 7 | 42 | >400 |
| Th | >400 | >400 | >400 | >400 |
| U | >400 | >400 | >400 | >400 |
| V | <5 | <5 | 5 | 44 |
| Zn | <5 | <5 | <5 | <5 |
| Zr | >400 | >400 | >400 | >400 |

# RE•Spec Column: Kd's as a Function of Nitric Acid Concentration

| Element | Nitric Acid Concentration | | | |
|---|---|---|---|---|
| | 0.2 N | 1 N | 4 N | 8 N |
| Ce | 60 | 270 | 230 | 145 |
| Dy | 14 | 64 | 130 | >400 |
| Er | <10 | 25 | 80 | >400 |
| Eu | 42 | 250 | 280 | 310 |
| Gd | 24 | 125 | 150 | 310 |
| Ho | 10 | 40 | 95 | >400 |
| La | 36 | 170 | 130 | 76 |
| Nd | 60 | 270 | 250 | 180 |
| Pr | 68 | 290 | 250 | 170 |
| Sm | 58 | 250 | 250 | 310 |
| Tb | 17 | 90 | 150 | >400 |
| Tm | <10 | 19 | 70 | >400 |
| Y | <10 | 22 | 60 | >400 |

# TRU•Spec Column: $K_d$'s as a Function of Hydrochloric Acid Concentration

| Element | Hydrochloric Acid Concentration 0.2 N | 1 N | 4 N | 8 N |
|---|---|---|---|---|
| Al | <10 | <10 | <10 | <10 |
| Ba | <10 | <10 | <10 | <10 |
| Be | <10 | <10 | <10 | <10 |
| Ca | <10 | <10 | <10 | <10 |
| Cd | <10 | 12 | 25 | <10 |
| Co | <10 | <10 | <10 | <10 |
| Cr | <10 | <10 | <10 | <10 |
| Cu | <10 | <10 | <10 | <10 |
| Fe | <10 | 17 | >400 | >400 |
| K | <10 | <10 | <10 | <10 |
| Li | <10 | <10 | <10 | <10 |
| Mg | <10 | <10 | <10 | <10 |
| Mn | <10 | <10 | <10 | <10 |
| Mo | <10 | 40 | >400 | >400 |
| Na | <10 | <10 | <10 | <10 |
| Ni | <10 | <10 | <10 | <10 |
| Pb | <10 | <10 | <10 | <10 |
| Sn | >400 | >400 | >400 | >400 |
| Sr | <10 | <10 | <10 | <10 |
| Ti | <10 | <10 | 15 | >400 |
| Th | <10 | <10 | >400 | >400 |
| U | 250 | >400 | >400 | >400 |
| V | <10 | <10 | <10 | 25 |
| Zn | <10 | 26 | 25 | <10 |
| Zr | <10 | <10 | <10 | >400 |

# RE•Spec Column: $K_d$'s as a Function of Hydrochloric Acid Concentration

| Element | Hydrochloric Acid Concentration 0.2 N | 1 N | 4 N | 8 N |
|---|---|---|---|---|
| Al | <10 | <10 | <10 | <10 |
| Ba | <10 | <10 | <10 | <10 |
| Be | <10 | <10 | <10 | <10 |
| Ca | <10 | <10 | <10 | <10 |
| Cd | <10 | <10 | 26 | <10 |
| Co | <10 | <10 | <10 | <10 |
| Cr | <10 | <10 | <10 | <10 |
| Cu | <10 | <10 | <10 | <10 |
| Fe | <10 | <10 | >400 | >400 |
| K | <10 | <10 | <10 | <10 |
| Li | <10 | <10 | <10 | <10 |
| Mg | <10 | <10 | <10 | <10 |
| Mn | <10 | <10 | <10 | <10 |
| Mo | <10 | 70 | >400 | >400 |
| Na | <10 | <10 | <10 | <10 |
| Ni | <10 | <10 | <10 | <10 |
| Pb | <10 | 15 | <10 | <10 |
| Sn | >400 | >400 | >400 | >400 |
| Sr | <10 | <10 | <10 | <10 |
| Ti | <10 | <10 | 28 | >400 |
| Th | <10 | 15 | >400 | >400 |
| U | 400 | >400 | >400 | >400 |
| V | <10 | <10 | <10 | 50 |
| Zn | <10 | 14 | 26 | <10 |
| Zr | <10 | <10 | 10 | >400 |

# TRU•Spec Column: $K_d$'s as a Function of Hydrochloric Acid Concentration

| Element | Hydrochloric Acid Concentration | | | |
|---|---|---|---|---|
| | 0.2 N | 1 N | 4 N | 8 N |
| Ce | <10 | <10 | <10 | 33 |
| Eu | <10 | <10 | <10 | 38 |
| La | <10 | <10 | <10 | 33 |
| Nd | <10 | <10 | <10 | 35 |
| Pr | <10 | <10 | <10 | 35 |
| Sm | <10 | <10 | <10 | 50 |
| Y | <10 | <10 | <10 | 10 |

# RE•Spec Column: $K_d$'s as a Function of Hydrochloric Acid Concentration

| Element | Hydrochloric Acid Concentration | | | |
|---|---|---|---|---|
| | 0.2 N | 1 N | 4 N | 8 N |
| Ce | <10 | <10 | <10 | 180 |
| Eu | <10 | <10 | <10 | 150 |
| La | <10 | <10 | <10 | 80 |
| Nd | <10 | <10 | <10 | 180 |
| Pr | <10 | <10 | <10 | 175 |
| Sm | <10 | <10 | <10 | 170 |
| Y | <10 | <10 | <10 | 28 |

# Conclusions

- In the nitric acid system, the distribution coefficients for the rare earths are higher for the RE•Spec phases.
- Thorium and uranium are strongly retained by the RE•Spec and TRU•Spec columns at the range of nitric acid concentrations studied.
- Rare earths are not retained by either phase at less than 6N hydrochloric acid.
- Thorium is strongly adsorbed above 4N hydrochloric acid.
- Uranium shows high distribution coefficients over the range of hydrochloric acid concentrations studied.
- The data can be used to devise group separation procedures prior to analytical measurements.
- This approach provides matrices that minimize interelement interferences.

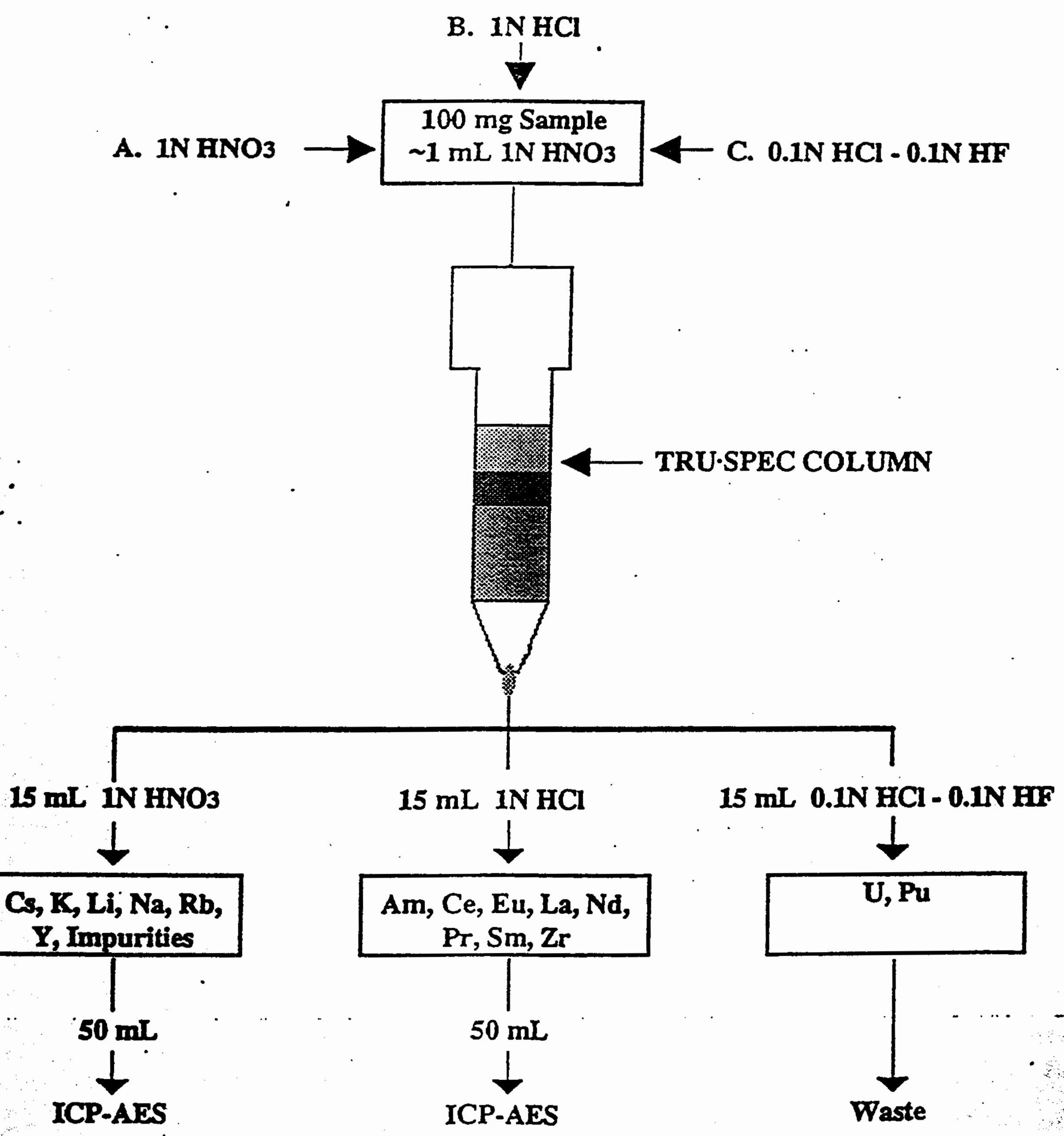
# OBJECTIVES

**Separation Procedures--TRU•Spec Columns:**

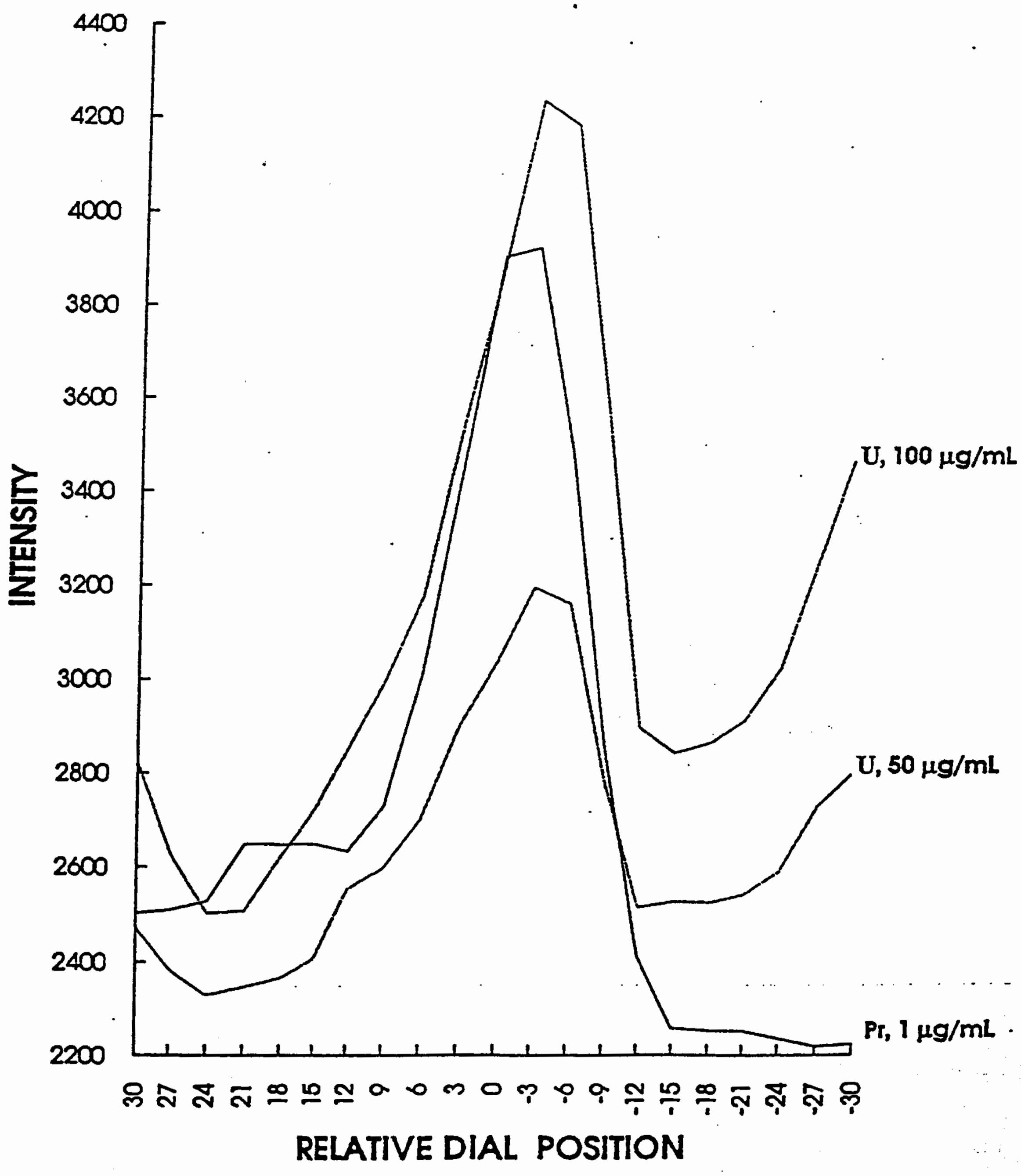
- Document the behavior of impurities as a function of nitric acid concentration.
- Evaluate recoveries for the chosen separation scheme--major and minor elements.
- Assess precision of analytical data.
- Verify that the cesium and rubidium fraction can be analyzed in a non-radioactive facility.
- Optimize the stripping of the actinides.

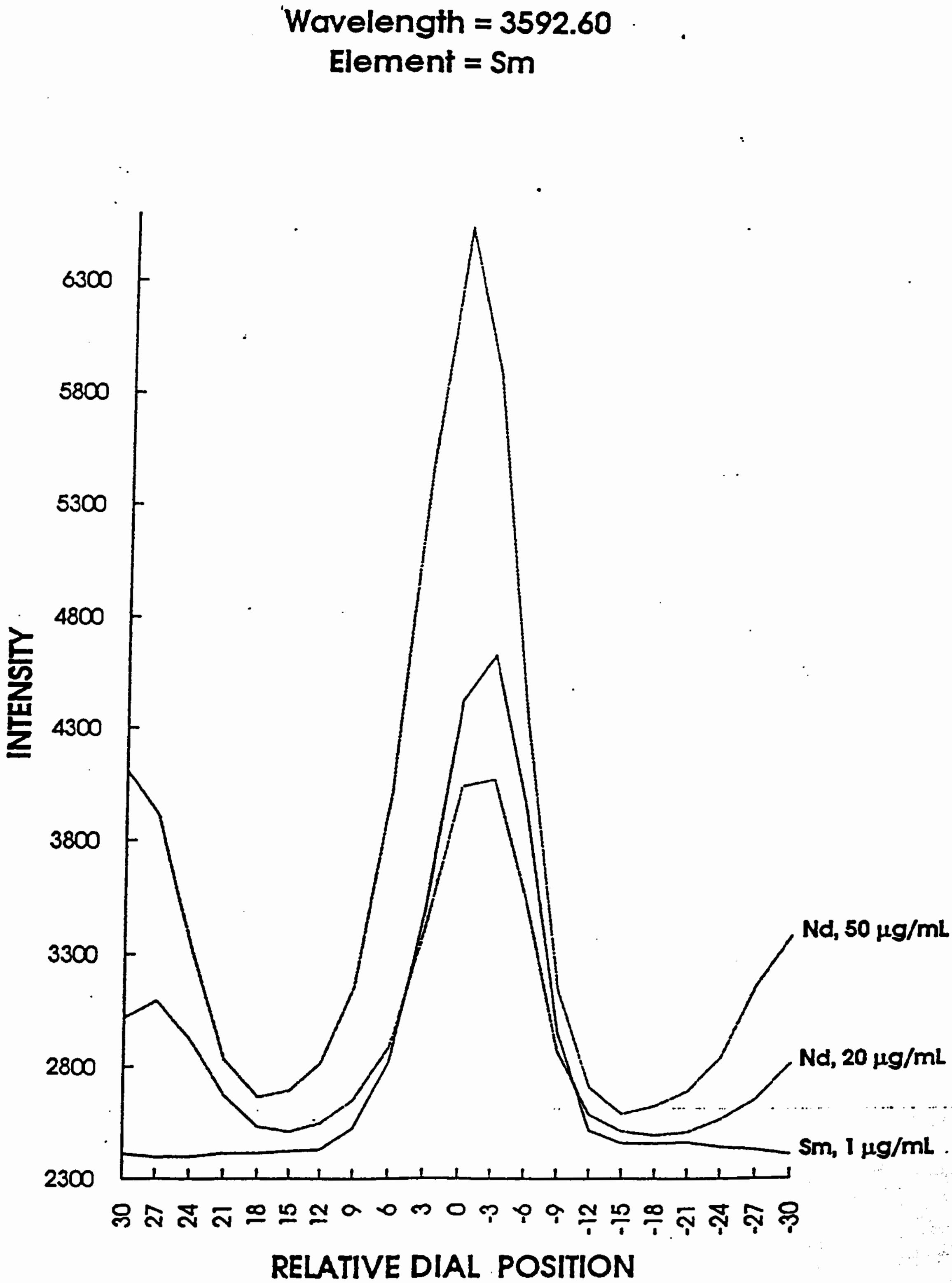
# EXPERIMENTAL

## SEPARATION SCHEME



Wavelength = 3908.44
Element = Pr




Wavelength = 3592.60
Element = Sm
INTENSITY
6300
5800
5300
4800
4300
3800
3300
2800
2300
Nd, 50 µg/mL
Nd, 20 µg/mL
Sm, 1 µg/mL
30 27 24 21 18 15 12 9 6 3 0 -3 -6 -9 -12 -15 -18 -21 -24 -27 -30
RELATIVE DIAL POSITION

# Nominal Composition of KCl-LiCl Standard Reference Material

| Element | Concentration, % |
|---|---|
| Ba | 0.50 |
| Ce | 0.47 |
| Cs | 1.35 |
| Eu | 0.02 |
| K | 21.40 |
| La | 0.23 |
| Li | 5.59 |
| Na | 4.70 |
| Nd | 0.70 |
| Pr | 0.22 |
| Pu | 2.00 |
| Rb | 0.16 |
| Sm | 0.23 |
| Sr | 0.42 |
| U | 1.95 |
| Y | 0.22 |

## Precision Data (1N Nitric Acid Elution)

| Element | Initial (µg/g) | Spike (µg/g) | µg/g IFRB-1 | µg/g IFRC-1 | µg/g IFRD-1 | Ave. ± S. D. | % Rec. |
|---|---|---|---|---|---|---|---|
| Al | <20 | 1000 | 1000 | 985 | 980 | 988 ± 10.4 | 98.8 |
| Ba | 625 | 1000 | 1615 | 1620 | 1615 | 1617 ± 2.9 | 99.2 |
| Be | <5 | 1000 | 950 | 955 | 950 | 952 ± 2.9 | 95.2 |
| Ca | <5 | 1000 | 945 | 950 | 950 | 948 ± 2.9 | 94.8 |
| Cd | <5 | 1000 | 955 | 960 | 955 | 957 ± 2.9 | 95.7 |
| Co | <10 | 1000 | 1030 | 1025 | 1015 | 1023 ± 7.6 | 102.3 |
| Cr | <5 | 1000 | 1000 | 1005 | 1010 | 1005 ± 5.0 | 100.5 |
| Cu | <5 | 1000 | 1015 | 1020 | 1015 | 1017 ± 2.9 | 101.7 |
| Fe | <5 | 1000 | 980 | 985 | 980 | 982 ± 2.9 | 98.2 |
| Mg | <5 | 1000 | 975 | 980 | 975 | 977 ± 2.9 | 97.7 |
| Mn | <2 | 1000 | 955 | 960 | 955 | 957 ± 2.9 | 95.7 |
| Ni | <10 | 1000 | 960 | 960 | 955 | 958 ± 2.9 | 95.8 |
| Pb | <20 | 1000 | 945 | 945 | 940 | 943 ± 2.9 | 94.3 |
| Sn | <20 | 1000 | 1090 | 1120 | 1065 | 1092 ± 27 | 109.2 |
| Sr | <5 | 1000 | 980 | 985 | 975 | 980 ± 5.0 | 98.0 |
| V | <10 | 1000 | 990 | 1000 | 1015 | 1002 ± 12.6 | 100.2 |
| Zn | <5 | 1000 | 965 | 970 | 970 | 968 ± 2.9 | 96.8 |
| Y | <5 | 500 | 493 | 485 | 488 | 489 ± 4.0 | 97.8 |

# Precision Data (1N Hydrochloric Acid Elution)

| Element | Initial (µg/g) | Spike (µg/g) | µg/g IFRB-2 | IFRE-2 | IFRF-2 | IFRG-2 | Ave. ± S. D. | % Rec |
|---|---|---|---|---|---|---|---|---|
| Ce | <50 | 2000 | 1880 | 1880 | 1840 | 1835 | 1859 ± 25 | 93.0 |
| Nd | <50 | 2000 | 2055 | 2035 | 2000 | 1950 | 2010 ± 46 | 100.5 |
| Eu | <5 | 200 | 205 | 196 | 196 | 199 | 199 ±4.2 | 99.5 |
| Pr | <50 | 1000 | 1060 | 1030 | 1025 | 995 | 1027 ± 27 | 102.7 |
| Sm | <20 | 1000 | 1035 | 1015 | 1000 | 965 | 1004 ± 29 | 100.4 |
| La | <10 | 500 | 494 | 482 | 475 | 455 | 477 ± 16 | 95.4 |
| Zr | <5 | 1000 | 880 | 965 | 975 | 1010 | 940 ± 52 | 94.0 |

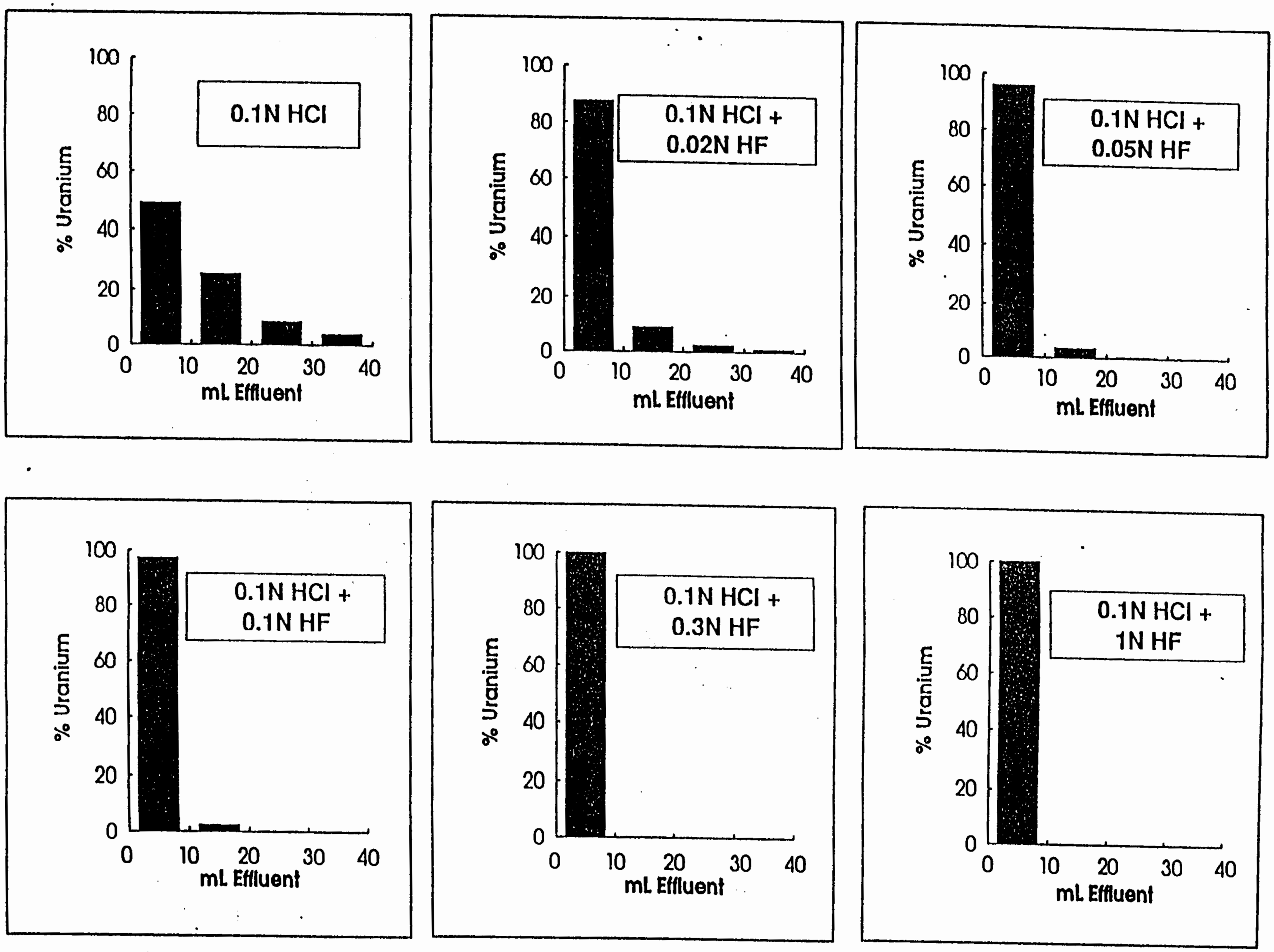
# Lithium and Potassium Recovery Data

| Separated Sample | Potassium, % | | Lithium, % | |
|---|---|---|---|---|
| | 1N $HNO_3$ | 1N HCl | 1N $HNO_3$ | 1N HCl |
| IFR-1 | 29.30 | <.005 | 7.20 | <.001 |
| IFR-2 | 28.95 | <.005 | 7.10 | <.001 |
| IFR-3 | 29.25 | <.005 | 7.10 | <.001 |
| IFR-4 | 29.10 | <.005 | 7.10 | <.001 |
| IFR-5 | 29.10 | <.005 | 7.05 | <.001 |
| IFR-6 | 28.95 | <.005 | 7.05 | <.001 |
| IFR-7 | 29.15 | <.005 | 7.10 | <.001 |
| **Ave.** | **29.11** | | **7.10** | |
| **S.D.** | **0.13** | | **0.05** | |
| **Direct** | **29.10** | | **7.20** | |

# Cesium and Rubidium Recoveries

| Sample | Cesium (µg/g) Added | Cesium (µg/g) Found | % Rec. | Rubidium (µg/g) Added | Rubidium (µg/g) Found | % Rec. |
|---|---|---|---|---|---|---|
| IFR-A1 | 0 | <10 | - | 0 | <25 | - |
| IFR-B1 | 5000 | 5450 | 109 | 1000 | 1050 | 105 |
| IFR-C1 | 5000 | 5450 | 109 | 1000 | 1050 | 105 |
| IFR-D1 | 5000 | 5450 | 109 | 1000 | 1050 | 105 |
| IFR-E1 | 1000 | 1150 | 115 | 200 | 220 | 110 |
| IFR-F1 | 2500 | 2800 | 112 | 500 | 550 | 110 |
| IFR-G1 | 10000 | 10800 | 108 | 2000 | 2100 | 105 |

# Uranium Stripping --TRU-Spec Column

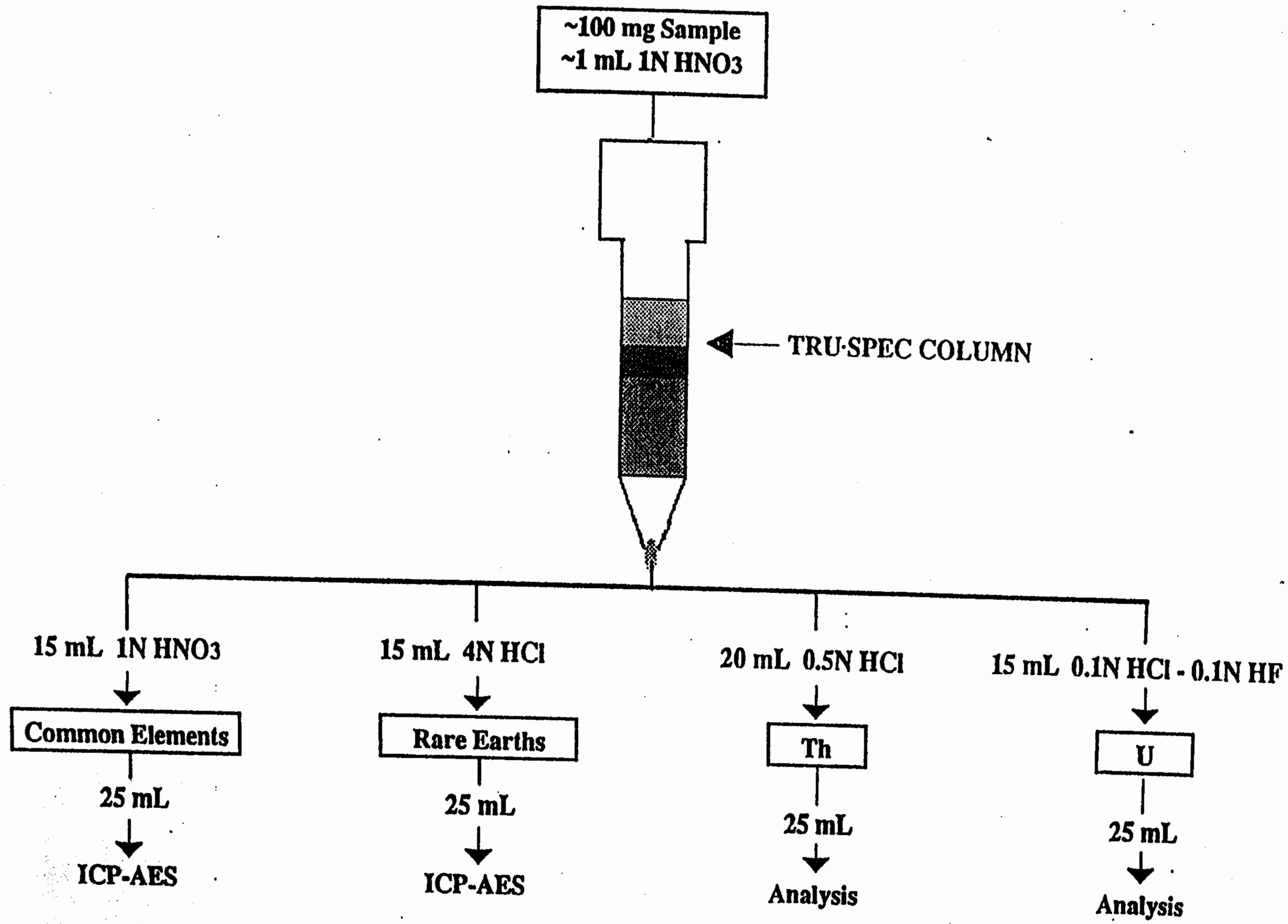


# Recoveries

| Element | Initial (µg/g) | Added (µg/g) | Found (µg/g) 1 N $HNO_3$ | Found (µg/g) 1 N HCl | % Rec. |
|---|---|---|---|---|---|
| As | <100 | 2000 | 1920 | <100 | 96.0 |
| B | <50 | 1000 | 970 | <20 | 97.0 |
| Mo | <50 | 1000 | 110 | 160 | 27.0 |
| Pd | <50 | 5000 | 4890 | <20 | 97.8 |
| Rh | <50 | 5000 | 4930 | <20 | 98.6 |
| Ru | <50 | 5000 | 4640 | <20 | 92.8 |
| Se | <100 | 2000 | 1960 | <100 | 98.0 |
| Te | <100 | 5000 | 4520 | <150 | 90.3 |
| Ti | <10 | 1000 | 990 | <5 | 99.0 |

# EXPERIMENTAL

## SEPARATION SCHEME



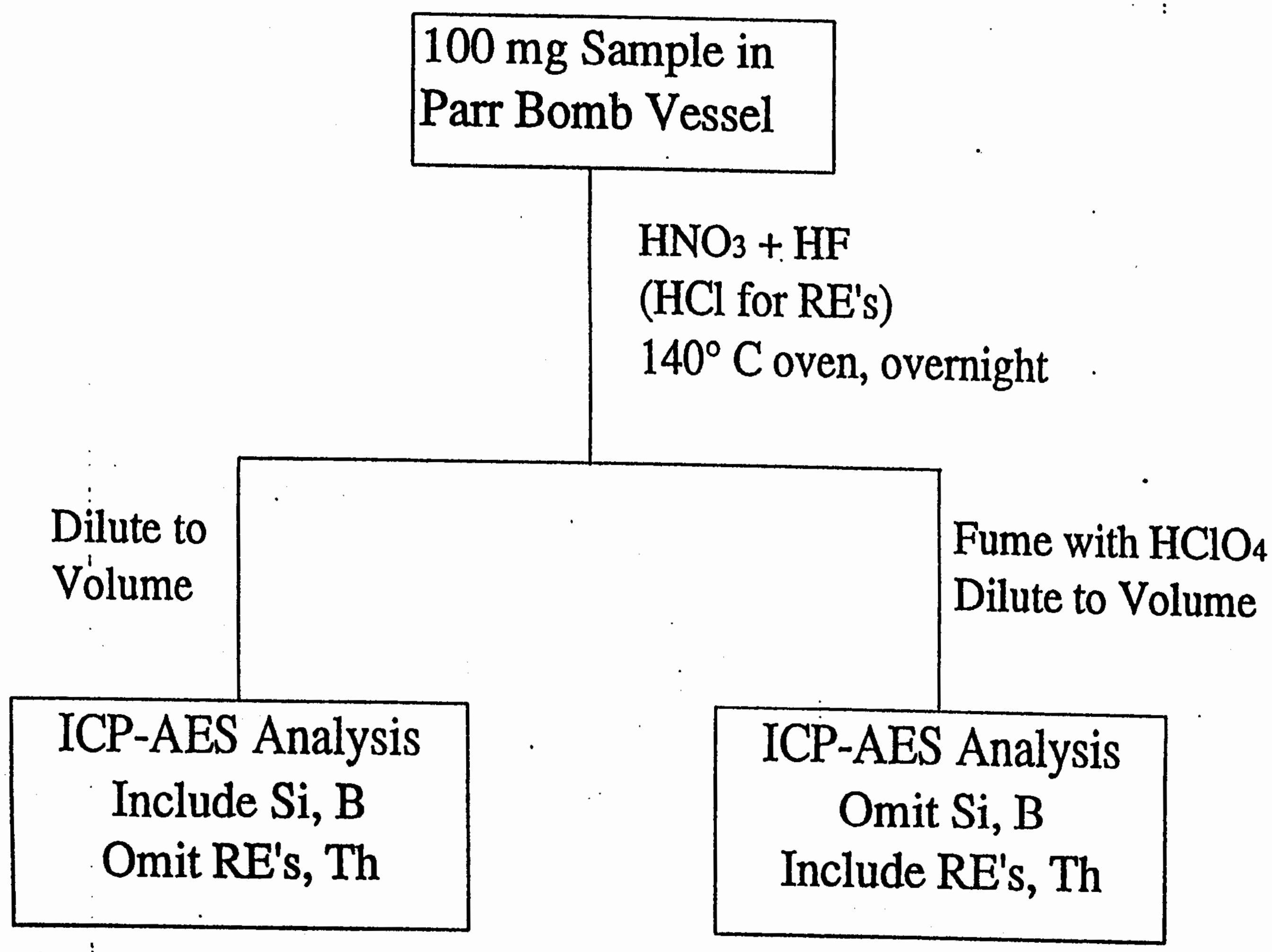
# Analytical Data for Rocky Flats Soil (SRM 4353)

## TRU•Spec Column Separation

| Element | Run 1 | Run 2 | Run 3 | Run 4 | Avg. ± S.D. |
|---|---|---|---|---|---|
| | Concentration, % | | | | |
| Al | 1.90 | 2.07 | 1.72 | 1.88 | 1.90 ±0.14 |
| Ca | 0.219 | 0.241 | 0.257 | 0.265 | 0.246 ±0.020 |
| Fe | 2.43 | 2.47 | 2.41 | 2.44 | 2.44 ±0.03 |
| K | 1.77 | 1.78 | 1.79 | 1.78 | 1.78 ±0.01 |
| Mg | 0.153 | 0.165 | 0.157 | 0.163 | 0.160 ±0.005 |
| Na | 0.531 | 0.508 | 0.502 | 0.517 | 0.515 ±0.013 |
| Ti | 0.177 | 0.183 | 0.187 | 0.181 | 0.182 ±0.004 |
| | Concentration, µg/g | | | | |
| Ag | 5.1 | 5.0 | 5.0 | 5.1 | 5.05 ±0.06 |
| Ba | 406 | 413 | 432 | 435 | 422 ±14 |
| Be | <2 | <2 | <2 | <2 | |
| Cd | <2 | <2 | <2 | <2 | |
| Co | 15.9 | 16.1 | 15.7 | 13.8 | 15.4 ±1.1 |
| Cr | 328 | 364 | 395 | 400 | 372 ±33 |
| Cu | 19.3 | 20.4 | 20.4 | 20.8 | 20.2 ±0.6 |
| La [1] | 27.4 | 31.2 | | | 29.3 |
| Li | 9.8 | 11.8 | 9.6 | 10.3 | 10.4 ±1.0 |
| Mn | 356 | 366 | 376 | 376 | 369 ±10 |
| Mo | 5.4 | 5.4 | 6.2 | 6.0 | 6.0 ±0.4 |
| Ni | 201 | 202 | 205 | 203 | 203 ±2 |
| Pr [1] | <20 | <20 | | | |
| Sm [1] | <10 | <10 | | | |
| Sr | 69.7 | 80.0 | 86.3 | 90.0 | 81.5 ±8.9 |
| Th [2] | 7.4 | 8.5 | | | 8.0 |
| U [3] | <20 | <20 | | | |
| V | 56.2 | 57.9 | 58.6 | 57.1 | 57.5 ±1.0 |
| Zn | 35.1 | 36.4 | 35.6 | 35.7 | 35.7 ±0.5 |
| Zr | 116 | 121 | 122 | 125 | 121 ±4 |

[1] 4N HCl elution
[2] 0.5N HCl elution
[3] 0.1N HCl + 1N HF elution

# Glass Dissolution



# Recovery Data for Rocky Flats Soil (SRM 4353)

| Element | Concentration, µg/g | | | Rec, % |
|---|---|---|---|---|
| | Initial | Added | Found | |
| | 4N HCl Elution | | | |
| La | 29.3 | 193 | 208 | 92.6 |
| | 29.3 | 195 | 214 | 94.7 |
| Pr | <20 | 386 | 393 | 102.0 |
| | <20 | 390 | 388 | 99.5 |
| Sm | <10 | 193 | 188 | 97.4 |
| | <10 | 195 | 193 | 99.0 |
| | 1N HCl Elution | | | |
| Th | 8.0 | 386 | 361 | 91.5 [1] |
| | 8.0 | 390 | 369 | 92.6 |
| | 0.1N HCl + 1N HF Elution | | | |
| U | <20 | 386 | 395 | 102.3 |
| | <20 | 390 | 389 | 99.7 |

[1] Elution with 15 mL of 0.5N HCl is insufficient;
2.1% and 2.5% Th were found in the 0.1N HCL-1N HF fractions.

## Comparative Data for Glass Dissolution

| Element (Oxide) | Weight Percent | | | |
|---|---|---|---|---|
| | Parr Bomb | | Beaker | |
| | Element | Oxide | Element | Oxide |
| Al ($Al_2O_3$) | 3.40 | 6.42 | 3.32 | 6.27 |
| B ($B_2O_3$) | <0.01 | <0.02 | <0.01 | <0.02 |
| Ba (BaO) | 0.038 | 0.042 | 0.043 | 0.048 |
| Ca (CaO) | 1.58 | 2.21 | 1.59 | 2.22 |
| Ce ($CeO_2$) | **0.06** | **0.07** | 0.717 | 0.880 |
| Co (CoO) | <0.005 | <0.01 | <0.005 | <0.01 |
| Cr ($Cr_2O_3$) | 1.34 | 1.96 | 1.24 | 1.81 |
| Cu (CuO) | <0.005 | <0.01 | <0.005 | <0.01 |
| Fe ($Fe_2O$) | 46.5 | 66.5 | 46.3 | 66.8 |
| K ($K_2O$) | 0.674 | 0.812 | ND | |
| Li ($Li_2O$) | <0.001 | <0.002 | ND | |
| Mg (MgO) | 0.486 | 0.806 | 0.487 | 0.807 |
| Mn ($MnO_2$) | 0.028 | 0.044 | 0.029 | 0.046 |
| Mo ($MoO_3$) | <0.005 | <0.01 | <0.005 | <0.01 |
| Na ($Na_2O$) | 0.314 | 0.419 | ND | |
| Ni (NiO) | 0.835 | 1.06 | 0.838 | 1.07 |
| Pb (PbO) | <0.02 | <0.03 | <0.02 | <0.03 |
| Si ($SiO_2$) | 9.45 | 20.2 | **<0.04** | **<0.1** |
| Sn ($SnO_2$) | <0.01 | <0.02 | <0.01 | <0.02 |
| Sr (SrO) | 0.009 | 0.011 | 0.009 | 0.011 |
| Ti ($TiO_2$) | 0.131 | 0.218 | 0.131 | 0.218 |
| V ($V_2O_3$) | 0.010 | 0.013 | 0.009 | 0.011 |
| Zn (ZnO) | 0.014 | 0.017 | 0.012 | 0.015 |
| Zr ($Zr_2O_3$) | 0.009 | 0.012 | 0.007 | 0.009 |
| | Σ Oxides | 101.6 | | |

ND = not determined

9/1-x15

# Comparative Glass Analysis Data

| Element | Concentration, % | | | | Diff., % |
|---|---|---|---|---|---|
| | Direct [1] | TRU-Spec [2] | | | |
| | | Run 1 | Run 2 | Avg. | |
| Al | 4.85 | 4.89 | 4.83 | 4.86 | 0.2 |
| B | 2.52 | 2.33 | 2.32 | 2.33 | 7.8 |
| Ba | 0.321 | 0.350 | 0.344 | 0.347 | 7.8 |
| Ca | 0.235 | 0.237 | 0.234 | 0.236 | 0.4 |
| Dr | 0.489 | 0.538 | 0.531 | 0.534 | 8.8 |
| Cu | 0.043 | 0.051 | 0.048 | 0.050 | 14.9 |
| Fe | 5.38 | 5.54 | 5.40 | 5.47 | 1.7 |
| K | 0.056 | 0.062 | 0.052 | 0.057 | 1.8 |
| Li | 2.33 | 2.32 | 2.32 | 2.32 | 0.4 |
| Mg | 0.600 | 0.604 | 0.596 | 0.600 | 0.0 |
| Mn | 1.13 | 1.16 | 1.14 | 1.15 | 1.7 |
| Na | 7.78 | 7.93 | 7.65 | 7.79 | 0.1 |
| Ni | 0.626 | 0.638 | 0.631 | 0.634 | 1.3 |
| Si | **26.6** | **0.53** | **0.59** | | |
| Sr | 0.011 | 0.012 | 0.012 | 0.012 | 8.3 |
| Ti | 0.042 | 0.046 | 0.044 | 0.045 | 6.8 |
| Zn | 0.022 | 0.030 | 0.028 | 0.029 | 28.0 |
| Zr | 0.970 | 1.04 | 1.03 | 1.03 | 6.0 |

[1] Parr bomb dissolution only

[2] Parr bomb dissolution and fluoride elimination

} close up

# Recovery Data

| Element | Concentration, % | | | Rec.,% |
|---|---|---|---|---|
| | Initial | Added | Found | |
| Ce | <0.003 | 0.0974 | 0.0964 | 99.0 |
| Nd | <0.003 | 0.0974 | 0.1042 | 106.7 |
| Eu | <0.001 | 0.0243 | 0.0247 | 101.6 |
| Th | <0.003 | 0.487 | 0.380 | 78.0 [1] |
| U | 0.071 | 0.487 | 0.557 | 99.8 |

[1] Fluoride may not have been completely eliminated

# Waste Glass Analysis (High Fe)

| Element | Concentration, % Run 1 | Run 2 | Avg., % |
|---|---|---|---|
| Al | 4.25 | 4.09 | 4.17 |
| Ba | 0.030 | 0.029 | 0.030 |
| Ca | 1.51 | 1.45 | 1.48 |
| Ce | 0.506 | 0.403 | 0.454 |
| Cr | 1.11 | 1.17 | 1.14 |
| Cu | 0.006 | 0.005 | 0.006 |
| Fe | 40.5 | 38.9 | 39.7 |
| Mg | 0.443 | 0.429 | 0.436 |
| Mn | 0.025 | 0.024 | 0.025 |
| Ni | 0.769 | 0.741 | 0.755 |
| Pb | 1.34 | 1.30 | 1.32 |
| Sr | 0.008 | 0.008 | 0.008 |
| Ti | 0.114 | 0.110 | 0.112 |
| Zn | 0.012 | 0.012 | 0.012 |

# Recovery Data

| | Concentration, % Initial | Added | Found | Rec.,% |
|---|---|---|---|---|
| Th | <0.01 | 0.491 | 0.501 | 102.0 |
| U | <0.03 | 0.491 | 0.403 | 82.1 |

# Conclusions

- Chemical separation of the actinides is needed for the best figures of merit.
- A TRU•Spec column elution method was developed that meets programmatic objectives.
- Recovery data document the acceptability of the procedure for the applications studied.
- Interferences in alpha-spectrometry can be minimized by group separations.
- The separations procedures should be applicable to geological matrices for the determination of rare earths and actinides.