A SEMIQUANTITATIVE SPECTROGRAPHIC
METHOD FOR THE ANALYSIS OF MINERALS,
ROCKS, AND ORES (II)

By
C. L. Waring
C. A. Annell

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CHEMISTRY

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A SEMIQUANTITATIVE SPECTROGRAPHIC METHOD FOR THE
ANALYSIS OF MINERALS, ROCKS, AND ORES (II)*

by

C. L. Waring and C. A. Annell

ABSTRACT

The scope of the semiquantitative spectrographic method for
the analysis of minerals, rocks, and ores previously described as
determining 55 elements, has now been increased to 68 elements which
can be estimated in one exposure of a 10-mg sample. Fluorine, the
69th element, requires a separate exposure for some materials. The
method has been used to complete about 185,000 determinations in
the past two years. Listed in this report are 336 chemical check
analyses that indicate approximately 8 percent disagreements in
the magnitude of one 10 percent bracket. No chemical and spectro­
graphic results differ by a factor of more than 10.

*This report concerns work done on behalf of the Division of
INTRODUCTION

In the program of investigating radioactive minerals, rocks, and ores conducted by the Geological Survey on behalf of the Atomic Energy Commission, it is often desirable to know the trace-elements content and the major constituents of a very large number of samples in a limited time.

Trace Elements Investigations Report 143, "A semiquantitative spectrographic method for the analysis of minerals, rocks, and ores," by C. L. Waring and C. A. Annell, presents a procedure for the rapid analysis of solid samples of varied constituents. These investigations are being continued in order to afford reliable basic data leading to better analytical methods applicable to these diversified materials.

The purpose of this paper is to describe advances in the semiquantitative method which have resulted in increasing the number of elements determined from 55 to 69 (see tables 1 and 2) and to compare chemical and spectrographic analytical data on a large number of samples.

The method has been applied to the following materials:

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<td>Schroekingerite</td>
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## DISCUSSION

No significant changes, except the addition of elements, have been introduced into the method since the original description.

The estimation of fluorine requires a separate exposure on samples of low calcium content. Two milligrams of calcium as calcium chloride solution are added to the electrodes and dried prior to loading the sample. The addition of calcium aids in the formation of the calcium fluoride molecule which produces the molecular band effect recorded by the photographic emulsion.

In analyzing the minerals euxenite and samarskite a 2-minute arcing time instead of the customary 1-minute period gives higher accuracy for such elements as Cb, Ta, Er, and U. These and other elements or their...
compounds having high boiling points constitute over 50 percent of the weight of the two minerals and therefore require a longer arcing time to vaporize into the arc stream.

The time required for an analysis varies with the type of material under test and the skill of the analyst. A trained person can complete the analysis of 14 samples (300 elements) of phosphate rock in 16 hours. However, this rate of speed is not recommended as a daily practice as eye fatigue may result. Samples producing more complicated spectra require additional time. Analysis time on 14 samples is broken down approximately as follows:

- Quarter and weigh samples and proper references: 4 hr
- Exposure: 1½ hr
- Development: 45 min
- Plate interpretation: 10 hr

The ease or difficulty with which a spectrum can be analyzed will depend upon the number, kind, and quantity of elements present in the sample. Samples containing relatively large amounts of the transition elements often give very complex spectra with heavy background. For some materials this results in serious interference with good analysis lines of the less abundant elements and necessitates referring to less sensitive lines. This procedure may involve similar scrutiny for a large number of elements and thus prolong the analysis.

Additional testing will be necessary to explain the following observed effects:

1.0 percent Ni in calcium matrices seems to be enhanced.
10.0 percent Ni in calcium phosphate matrices seems to be depressed.
1.0 percent Ni in feldspar matrices seems to be depressed, and
slightly enhanced at less than 1.0 percent.
In schroeckingerite Ca and U seem to be depressed.

RESULTS AND TABLES

The method has been used to complete about 185,000 determinations
during the past two years. Comparisons of chemical and spectrographic
results for 336 determinations are shown in tables 3 - 13. The dis­
agreements are approximately 8 percent in the magnitude of one bracket.
Approximately 4 percent of the disagreements are regarded as borderline
cases because of doubt as to which of two adjacent brackets an element
belonged.

Noted in the chemical analyses were several disagreements especial­
ly for the following elements: Pb, Mn, Mg, V, Al, Fe, Na, Zr, and Ca.
Standard plates for these elements have been remade and better agree­
ment with the chemical analyses has been observed.
Table 1.—Standard sensitivities for the elements determined by the semiquantitative method.\(^1\)/

Note: It is possible to detect some elements below the values listed, as the standard reference plates were prepared on the basis of 10 percent increments.

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<td>Nd - 0.01</td>
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<td>B - 0.001</td>
<td>Ni - 0.01</td>
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<td>Cd - 0.01</td>
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<td>Ce - 0.1</td>
<td>Rb - 0.01 (^2/) (10.0)</td>
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<td>Co - 0.01</td>
<td>Re - 0.1</td>
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<td>Cr - 0.001</td>
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\(^1/\) Better sensitivity for many of these elements may be obtained by special methods.

\(^2/\) A second exposure is required for the high sensitivity listed.

\(^3/\) A third exposure is required for the fluorine estimation.
Table 2.—Arc lines used in the semiquantitative method

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Table 3.—Comparison of chemical and spectrographic analyses for phosphorus of various phosphate rocks (chemical results as oxides converted to element).

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<tr>
<th>Sample no.</th>
<th>Percent P chemical</th>
<th>Percent P spectrographic</th>
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<tr>
<td>128</td>
<td>2.3</td>
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<tr>
<td>129</td>
<td>13.8</td>
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<tr>
<td>130</td>
<td>15.6</td>
<td>10. +</td>
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<td>131</td>
<td>3.8</td>
<td>1.0 - 10.</td>
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<tr>
<td>132</td>
<td>13.7</td>
<td>10. +</td>
</tr>
<tr>
<td>133</td>
<td>14.9</td>
<td>10. +</td>
</tr>
<tr>
<td>134</td>
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<td>1.0 - 10.</td>
</tr>
<tr>
<td>135</td>
<td>9.9</td>
<td>10. +</td>
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<td>0.1 - 1.0</td>
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<tr>
<td>27B-2A</td>
<td>2.7</td>
<td>1.0 - 10.</td>
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<tr>
<td>27B-2B</td>
<td>2.3</td>
<td>1.0 - 10.</td>
</tr>
<tr>
<td>27B-2C</td>
<td>6.3</td>
<td>1.0 - 10.</td>
</tr>
<tr>
<td>27B-3</td>
<td>8.</td>
<td>1.0 - 10.</td>
</tr>
<tr>
<td>27B-4</td>
<td>8.3</td>
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</tr>
<tr>
<td>27B-5A</td>
<td>6.2</td>
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<tr>
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<td>6.0</td>
<td>1.0 - 10.</td>
</tr>
<tr>
<td>PC-1</td>
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<td>1.0 - 10.</td>
</tr>
<tr>
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</tr>
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<td>PC-3</td>
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<td>PC-4A</td>
<td>0.24</td>
<td>0.1 - 1.0</td>
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<tr>
<td>PC-4B</td>
<td>6.1</td>
<td>1.0 - 10.</td>
</tr>
<tr>
<td>PC-5A</td>
<td>5.4</td>
<td>1.0 - 10.</td>
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<td>PC-5B</td>
<td>4.8</td>
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<td>1.9</td>
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1/ Lot 712, TWS 345, TWC 1376
Lot 850, TWS 346, TWC 1378
Lot 851, TWS 347, TWC 1379

Several materials comprise "Florida phosphates." The typical ore is a classic phosphorite with admixed quartz and clay. It has a high content of Ca and P. Above the main ore in the deposit is a "leached zone," consisting mainly of quartz but with some aluminous phosphate and only minor amounts of calcium.
Table 3.--Continued.

<table>
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<th>Sample no. 2/</th>
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<th>Percent P spectrographic</th>
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<tr>
<td>M5</td>
<td>16.2</td>
<td>10.0</td>
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<tr>
<td>M6</td>
<td>15.8</td>
<td>10.0</td>
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<td>M7</td>
<td>16.4</td>
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<td>M8</td>
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<td>M9</td>
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<td>M10</td>
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<tr>
<td>M11</td>
<td>16.0</td>
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</tr>
<tr>
<td>M12</td>
<td>16.0</td>
<td>10.0</td>
</tr>
<tr>
<td>N-1</td>
<td>16.0</td>
<td>10.0</td>
</tr>
<tr>
<td>N-2</td>
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<td>10.0</td>
</tr>
<tr>
<td>N-13</td>
<td>10.4</td>
<td>1.0 - 10.0</td>
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Fossil manatee bones from the leached zone of the Bone Valley formation, Hillsborough County, Florida, which consist mainly of apatite.

<table>
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<th>Percent P spectrographic</th>
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<td>M-1-A</td>
<td>0.23</td>
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<td>M-1-B</td>
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</tr>
<tr>
<td>M-1-C</td>
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<tr>
<td>M-2</td>
<td>0.17</td>
<td>Not detected</td>
</tr>
<tr>
<td>M-3</td>
<td>18.22</td>
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<td>M-4</td>
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<tr>
<td>M-5</td>
<td>17.51</td>
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<tr>
<td>M-6</td>
<td>11.57</td>
<td>10.0</td>
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<tr>
<td>M-7-A</td>
<td>16.32</td>
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<tr>
<td>M-7-B</td>
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<tr>
<td>M-8</td>
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Samples from caves on Mona Island, West Indies, varying from red and white limestone to almost pure earthy phosphatic apatite.

2/ Lot 669, TWS 173, Lot 0, TWS 238
3/ Lot 535, TWS 298
4/ Detection limit of P is approximately 0.1 percent.
Table 4.--Comparison of chemical and spectrographic percentage analyses of phosphate rock from Florida with chemical results as oxides converted to elements. (For description of Florida phosphates, see first part of table 3.)

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<th>No. 4</th>
<th>No. 5</th>
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<td>10.+</td>
<td>15.5</td>
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<tr>
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<td>10.+</td>
<td>17.7</td>
<td>10.+</td>
<td>19.4</td>
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1/ Lot 524, TWS 61, TWC 908
Table 4—Continued.

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<td>5.7 1.0-10.0</td>
<td>5.1 1.0-10.0</td>
<td>3.1 1.0-10.0</td>
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<td>0.08 0.01-0.1</td>
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<td>0.00 0.001-0.01</td>
<td>0.01 0.001-0.01</td>
<td>0.00 0.001-0.01</td>
<td>0.01 0.001-0.01</td>
<td>0.01 0.001-0.01</td>
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<td>0.44 0.1-1.0</td>
<td>0.69 0.1-1.0</td>
<td>0.50 0.1-1.0</td>
<td>0.40 0.1-1.0</td>
<td>0.24 0.1-1.0</td>
<td>0.21 0.1-1.0</td>
<td>0.20 0.1-1.0</td>
</tr>
<tr>
<td>Cr</td>
<td>0.03 0.01-0.1</td>
<td>0.11 0.01-0.1</td>
<td>0.13 0.01-0.1</td>
<td>0.06 0.01-0.1</td>
<td>0.06 0.01-0.1</td>
<td>0.05 0.01-0.1</td>
<td>0.04 0.01-0.1</td>
</tr>
<tr>
<td>V</td>
<td>0.01 0.001-0.01</td>
<td>0.01 0.001-0.01</td>
<td>0.00 0.001-0.01</td>
<td>0.01 0.001-0.01</td>
<td>0.00 0.001-0.01</td>
<td>0.00 0.001-0.01</td>
<td>0.00 0.001-0.01</td>
</tr>
</tbody>
</table>

1/ Lot 2 - 23, TWS 86, TWC 780
Northwest phosphates are from the Phosphoria formation mainly in Montana and Idaho. They are dark-colored pelletal phosphorites, consisting of carbonate fluorapatite, with minor admixed calcite and/or dolomite, clays, mica, and organic matter. When samples representing phosphatic facies are analyzed, the relative amounts of minor constituents will be greater.

Table 5.—Comparison of chemical and spectrographic percentage analyses of northwest phosphates from Montana and Idaho (chemical results: as oxides converted to elements). 1/

<table>
<thead>
<tr>
<th>Element</th>
<th>RAH-47-10</th>
<th>RAH-47-46</th>
<th>LES-47-36</th>
<th>VEM-47-253</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>chem.</td>
<td>spec.</td>
<td>chem.</td>
<td>spec.</td>
</tr>
<tr>
<td>Si</td>
<td>7.06</td>
<td>1.0-10.0</td>
<td>8.28</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Cr</td>
<td>0.11</td>
<td>0.1-1.0</td>
<td>0.13</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>V</td>
<td>0.09</td>
<td>0.1-1.0</td>
<td>0.04</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>Ti</td>
<td>0.09</td>
<td>0.01-0.1</td>
<td>0.11</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>P</td>
<td>11.5</td>
<td>1.0-10.0</td>
<td>12.2</td>
<td>10.+</td>
</tr>
<tr>
<td>Mn</td>
<td>0.04</td>
<td>0.001-0.01</td>
<td>0.03</td>
<td>0.001-0.01</td>
</tr>
<tr>
<td>Ca</td>
<td>26.6</td>
<td>10.+</td>
<td>29.0</td>
<td>10.+</td>
</tr>
<tr>
<td>Mg</td>
<td>0.20</td>
<td>0.1-1.0</td>
<td>0.22</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Fe</td>
<td>0.70</td>
<td>1.0-10.0</td>
<td>0.55</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Al</td>
<td>1.46</td>
<td>1.0-10.0</td>
<td>1.44</td>
<td>1.0-10.0</td>
</tr>
</tbody>
</table>

1/ Lot 1202, TWS 124, TWC 964; Lot 1204, TWS 125, TWC 965; Lot 1205, TWS 126, TWC 966; Lot 1206, TWS 127, TWC 967.
<table>
<thead>
<tr>
<th>Element</th>
<th>RAH-82-47</th>
</tr>
</thead>
<tbody>
<tr>
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<td>chem. spec.</td>
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<tr>
<td>Al</td>
<td>RAH-87-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>Ca</td>
<td>RAH-100-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>Mg</td>
<td>LES-117-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>Si</td>
<td>LES-179-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>V</td>
<td>VEM-111-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
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<tbody>
<tr>
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<tr>
<td>Al</td>
<td>VEM-488-47</td>
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<td>chem. spec.</td>
</tr>
<tr>
<td>Ni</td>
<td>RMC-127-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>Si</td>
<td>RAH-47-179</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>V</td>
<td>VEM-519-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td></td>
<td>VEM-523-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Element</th>
<th>VEM-485-47</th>
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</thead>
<tbody>
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<td>chem. spec.</td>
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<tr>
<td>Al</td>
<td>VEM-488-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>Ni</td>
<td>RMC-127-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>Si</td>
<td>RAH-47-179</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td>V</td>
<td>VEM-519-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
<tr>
<td></td>
<td>VEM-523-47</td>
</tr>
<tr>
<td></td>
<td>chem. spec.</td>
</tr>
</tbody>
</table>

Lot 1134, TWS 223, TWC 1134; Lot 1209, TWS 130; Lot 1211, TWS 131, TWC 1286
Table 6.--Comparison of chemical and spectrographic analyses of a vanadium mineral (chemical results as oxides converted to elements).

Red-brown vanadium mineral associated with hummerite, Hummer mine, Jo Dandy group, Paradox Valley, Montrose County, Colo. This is probably a new mineral but it is mixed with fine-grained clay from which it cannot be separated mechanically.

<table>
<thead>
<tr>
<th>Element</th>
<th>Chemical percent</th>
<th>ADW no. 79 1/</th>
<th>Spectrographic percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>2.5</td>
<td></td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Fe</td>
<td>5.5</td>
<td></td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Mg</td>
<td>2.5</td>
<td></td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>K</td>
<td>2.0</td>
<td></td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Al</td>
<td>0.5</td>
<td></td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Na</td>
<td>1.1</td>
<td></td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Sr</td>
<td>0.6</td>
<td></td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Ca</td>
<td>0.64</td>
<td></td>
<td>0.01-0.1</td>
</tr>
</tbody>
</table>

1/ Lot 0-20, TWS 270, TWC 1421
Table 7.—Comparison of chemical and spectrographic percentage analyses of miscellaneous samples
(chemical results as oxides converted to elements).

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>1.59 1.0-10.0</td>
<td>0.10 0.1-1.0</td>
<td>0.054 0.1-1.0</td>
<td>0.07 0.1-1.0</td>
<td>9.1 1.0-10.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>6.8 1.0-10.0</td>
<td>0.03 0.01-0.1</td>
<td>3.88 1.0-10.0</td>
<td>24.4 10.0</td>
<td>4.6 1.0-10.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>5.5 1.0-10.0</td>
<td>0.007 0.01-0.1</td>
<td>3.3 1.0-10.0</td>
<td>0.96 0.1-1.0</td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3.6 1.0-10.0</td>
<td></td>
<td>0.047 0.01-0.1</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>2.82 1.0-10.0</td>
<td>0.31 0.1-1.0</td>
<td>0.014 0.01-0.1</td>
<td>17.3 10.0</td>
<td>0.23 0.1-1.0</td>
<td>.82 0.1-1.0</td>
<td>30. 10.0</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.072 0.1-1.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>51.7 10.0</td>
<td>37.4 10.0</td>
<td>47.5 10.0</td>
<td></td>
<td>45.9 10.0</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

/ Bitter Creek mine, Montrose County, Colorado
/ Jo Dandy mine, Montrose County, Colorado
/ Hand-picked sample from ore-bearing sandstone, May Day mine, Mesa County, Colorado
/ Olmstedville, Essex County, New York
/ Jo Dandy mine, Montrose County, Colorado
/ Euxenite sample from Kragero, Norway, U.S.N.M. R7144
/ Great Bear pitchblende, Canada
Table 8.—Comparison of chemical and spectrographic analyses of silicate rocks for zirconium (chemical results as oxides converted to elements).  

The samples were radioactive Tertiary intrusives associated pitchblende deposits of the Central City district, Colorado.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Percent Zr chemical</th>
<th>Percent Zr spectrographic</th>
</tr>
</thead>
<tbody>
<tr>
<td>P - 9</td>
<td>&lt; 0.01</td>
<td>0.001 - 0.01</td>
</tr>
<tr>
<td>P - 14</td>
<td>0.14</td>
<td>0.01 - 0.1</td>
</tr>
<tr>
<td>P - 21</td>
<td>0.06</td>
<td>0.1 - 1.0</td>
</tr>
<tr>
<td>P - 23A</td>
<td>&lt; 0.01</td>
<td>0.001 - 0.01</td>
</tr>
<tr>
<td>P - 23B</td>
<td>&lt; 0.01</td>
<td>0.001 - 0.01</td>
</tr>
<tr>
<td>P - 27</td>
<td>0.05</td>
<td>0.01 - 0.1</td>
</tr>
<tr>
<td>P - 29</td>
<td>0.06</td>
<td>0.01 - 0.1</td>
</tr>
<tr>
<td>P - 94</td>
<td>0.05</td>
<td>0.001 - 0.01</td>
</tr>
<tr>
<td>P - 118</td>
<td>0.10</td>
<td>0.01 - 0.1</td>
</tr>
</tbody>
</table>

1/ Lot 0-50, TWS 336, TWC 1336
Table 9.—Comparison of chemical and spectrographic percentage analyses of ash from Dakota lignite samples (chemical results as oxides converted to elements). 1/ Auger hole samples from southwest North Dakota.

<table>
<thead>
<tr>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>5.35 1.0-10.0</td>
<td>13.1 10.+</td>
<td>15. 10.+</td>
<td>7.1 1.0-10.0</td>
<td>4.45 1.0-10.0</td>
<td>10.2 1.0-10.0</td>
</tr>
<tr>
<td>Fe</td>
<td>8.0 1.0-10.0</td>
<td>23.6 10.+</td>
<td>21.9 10.+</td>
<td>24.2 10.+</td>
<td>15.8 10.+</td>
<td>8.7 1.0-10.0</td>
</tr>
<tr>
<td>Ca</td>
<td>11.1 1.0-10.0</td>
<td>2.9 1.0-10.0</td>
<td>7.8 1.0-10.0</td>
<td>8.6 1.0-10.0</td>
<td>14.4 1.0-10.0</td>
<td>10.2 1.0-10.0</td>
</tr>
<tr>
<td>Mg</td>
<td>4.4 1.0-10.0</td>
<td>0.126 0.1-1.0</td>
<td>0.126 0.1-1.0</td>
<td>0.138 0.1-1.0</td>
<td>0.072 0.1-1.0</td>
<td>0.108 0.1-1.0</td>
</tr>
<tr>
<td>Na</td>
<td>0.04 0.01-0.1</td>
<td>0.2 0.1-1.0</td>
<td>0.78 0.1-1.0</td>
<td>0.11 0.01-0.1</td>
<td>0.20 0.1-1.0</td>
<td>0.37 0.1-1.0</td>
</tr>
<tr>
<td>K</td>
<td>6.75 1.0-10.0</td>
<td>10.1 10.+</td>
<td>11.6 10.+</td>
<td>9.3 1.0-10.0</td>
<td>7.8 1.0-10.0</td>
<td>12.6 10.+</td>
</tr>
<tr>
<td>Ti</td>
<td>0.06 0.01-0.1</td>
<td>0.36 0.1-1.0</td>
<td>0.36 0.1-1.0</td>
<td>0.06 0.01-0.1</td>
<td>0.06 0.01-0.1</td>
<td>0.06 0.01-0.1</td>
</tr>
<tr>
<td>Mn</td>
<td>3.2 1.0-10.0</td>
<td>0.67 0.1-1.0</td>
<td>0.67 0.1-1.0</td>
<td>1.28 1.0-10.0</td>
<td>0.92 1.0-10.0</td>
<td>0.92 0.1-1.0</td>
</tr>
<tr>
<td>Cr</td>
<td>0.118 0.1-1.0</td>
<td></td>
<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>0.20 0.1-1.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mg</td>
<td>0.04 0.1-1.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>P</td>
<td>0.45 0.1-1.0</td>
<td></td>
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<td></td>
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<td></td>
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<tr>
<td>Si</td>
<td>0.27 0.1-1.0</td>
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<td></td>
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<td></td>
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</table>

1/ Lot 0-99, TWS 473, TWC 1587
Table 10.—Comparison of chemical and spectrographic percentage analyses of the lead content of carnotite-bearing sandstones from various localities (chemical results as oxides reduced to elements) 1/

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Locality</th>
<th>Chemical Pb</th>
<th>Spectrographic Pb</th>
</tr>
</thead>
<tbody>
<tr>
<td>LRS-6-48</td>
<td>Mine D, Montrose County, Colo.</td>
<td>0.10</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>7</td>
<td>Butterfly mine, Montrose County, Colo.</td>
<td>0.16</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>21</td>
<td>Radium No. 5 mine, San Miguel County, Colo.</td>
<td>0.11</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>22</td>
<td>Raven mine, San Miguel County, Colo.</td>
<td>0.003</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>26</td>
<td>Calamity No. 13 mine, Mesa County, Colo.</td>
<td>0.013</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>32</td>
<td>Vanadous No. 1 mine, San Miguel County, Colo.</td>
<td>0.004</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>33B</td>
<td>Bear Creek mine, San Miguel County, Colo.</td>
<td>0.010</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>34</td>
<td>Primus claim, San Miguel County, Colo.</td>
<td>0.22</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>35A</td>
<td>Dunning-Greysill mine, San Juan County, Colo.</td>
<td>0.004</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>39A</td>
<td>Stone No. 1 claim, Montrose County, Colo.</td>
<td>0.10</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>43</td>
<td>Bitter Creek mine, Montrose County, Colo.</td>
<td>0.12</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>60</td>
<td>Club mine, Montrose County, Colo.</td>
<td>0.045</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>67</td>
<td>Wild Steer mine, Montrose County, Colo.</td>
<td>0.11</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>69</td>
<td>Eastside mine, Navajo Indian Reservation, Ariz.</td>
<td>0.011</td>
<td>0.01-0.1</td>
</tr>
</tbody>
</table>

1/ Lot 0-21, TWS 175
Table 11.--Comparison of chemical and spectrographic percentage analyses of red and gray clays from the Colorado Plateau (chemical results as oxides converted to elements).1/

Red and gray clays consisting chiefly of hydro-mica, quartz, and clacite from a zone underlying vanadium-bearing ore at Bitter Creek mine, Montrose County, Colorado.

<table>
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<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Ca</td>
<td>1.7</td>
<td>1.0-10.0</td>
<td>1.8</td>
<td>1.0-10.0</td>
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<tr>
<td>Si</td>
<td>28.0</td>
<td>10 +</td>
<td>30.0</td>
<td>10 +</td>
</tr>
<tr>
<td>Fe</td>
<td>3.84</td>
<td>1.0-10.0</td>
<td>1.7</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Al</td>
<td>5.6</td>
<td>1.0-10.0</td>
<td>4.7</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Mg</td>
<td>2.46</td>
<td>1.0-10.0</td>
<td>2.42</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Cu</td>
<td>1.37</td>
<td>1.0-10.0</td>
<td>1.45</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Na</td>
<td>0.03</td>
<td>0.1-1.0</td>
<td>0.07</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>K</td>
<td>5.0</td>
<td>1.0-10.0</td>
<td>4.6</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Ti</td>
<td>0.35</td>
<td>0.1-1.0</td>
<td>0.36</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>V</td>
<td>0.04</td>
<td>0.01-0.1</td>
<td>0.04</td>
<td>0.01-0.1</td>
</tr>
</tbody>
</table>

1/ Lot 0-20, TWS 118
Table 12.-- Comparison of chemical and spectrographic percentage analyses of Hyatt pegmatite samples, Larimer County, Colo. (chemical results as oxides reduced to elements). 1/

Hyatt pegmatite samples from NE1/4NW1/4 sec. 28, T. 6 N., R. 71 W., Larimer County, Colorado. Samples from the plagioclase-perthite-quartz wall zone of the pegmatite. The purpose of analyzing the samples was to determine the presence in the wall zone of minor elements that crystallized in relative abundance in the next inner zone.

<table>
<thead>
<tr>
<th>Element</th>
<th>B6090-A chemical</th>
<th>B6090-B chemical</th>
<th>B6090-A and B spectrographic</th>
</tr>
</thead>
<tbody>
<tr>
<td>Si</td>
<td>33.8</td>
<td>--</td>
<td>10. +</td>
</tr>
<tr>
<td>Al</td>
<td>8.25</td>
<td>8.24</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>Na</td>
<td>4.35</td>
<td>4.28</td>
<td>1.0-10.0</td>
</tr>
<tr>
<td>K</td>
<td>3.22</td>
<td>3.3</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Fe</td>
<td>0.50</td>
<td>0.51</td>
<td>0.1-1.0</td>
</tr>
<tr>
<td>Ca</td>
<td>0.25</td>
<td>0.24</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>Mg</td>
<td>0.07</td>
<td>0.08</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>V</td>
<td>0.02</td>
<td>0.02</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>Ti</td>
<td>0.006</td>
<td>0.006</td>
<td>0.001-0.01</td>
</tr>
<tr>
<td>Mn</td>
<td>0.031</td>
<td>0.031</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>Ba</td>
<td>0.045</td>
<td>0.045</td>
<td>0.01-0.1</td>
</tr>
</tbody>
</table>

1/ Lot 1521, TWS 316, TWC 1303
Table 13.--Comparison of chemical and spectrographic percentage analyses of modern and fossil manatee bones (chemical results as oxides converted to elements).1/

Samples M4, M8, and M12 were fossil manatee ribs from the Bone Valley formation of Florida. Samples M13 A and B were modern manatee rib. The analyses were used to compare the modern and fossil bones.

<table>
<thead>
<tr>
<th>Sample no.</th>
<th>Mg chem.</th>
<th>Mg spec.</th>
<th>Sr chem.</th>
<th>Sr spec.</th>
</tr>
</thead>
<tbody>
<tr>
<td>M - 4</td>
<td>0.2</td>
<td>0.1-1.0</td>
<td>0.17</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>M - 8</td>
<td>0.82</td>
<td>0.1-1.0</td>
<td>0.14</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>M - 12</td>
<td>0.3</td>
<td>0.1-1.0</td>
<td>0.14</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>M - 13A</td>
<td>0.6</td>
<td>0.1-1.0</td>
<td>0.13</td>
<td>0.01-0.1</td>
</tr>
<tr>
<td>M - 13B</td>
<td>0.6</td>
<td>0.1-1.0</td>
<td>0.14</td>
<td>0.01-0.1</td>
</tr>
</tbody>
</table>

1/ TWS 173, 238, 321, TWC 1256, TWC 1257
APPENDIX

Composition of standard solutions

The following standard solutions were made from compounds and elements available in the laboratory. Many of the compounds and elements used were Johnson, Matthey and Co. "Specpure" grade (J and M). The compounds were dissolved in distilled water unless otherwise noted.

<table>
<thead>
<tr>
<th>Element standardized</th>
<th>Compound used</th>
<th>Solution</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ag</td>
<td>AgNO₃, reagent</td>
<td></td>
</tr>
<tr>
<td>Al</td>
<td>AlCl₃·6H₂O, C.P.</td>
<td>Compound dried in oven at 140°C and dissolved in cold acidified H₂O.</td>
</tr>
<tr>
<td>As</td>
<td>As₂O₃, Nat. Bur. St. No. 83a</td>
<td>1:1 HNO₃, heated. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Au</td>
<td>Au, metal, J and M</td>
<td>Aqua regia. Boiled down several times with HCl (conc.) to drive off HNO₃. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>B</td>
<td>H₃BO₃, C.P.</td>
<td></td>
</tr>
<tr>
<td>Ba</td>
<td>BaCl₂·2H₂O, C.P.</td>
<td></td>
</tr>
<tr>
<td>Be</td>
<td>Be, metal, J and M</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Bi</td>
<td>Bi, metal, J and M</td>
<td>1:1 HNO₃, diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Ca</td>
<td>CaCl₂·2H₂O, anal. reag.</td>
<td></td>
</tr>
<tr>
<td>Cb</td>
<td>Cb, metal, J and M</td>
<td>48 percent HF. Diluted to volume with HNO₃, conc.</td>
</tr>
<tr>
<td>Cd</td>
<td>CdCl₂·2H₂O, C. P.</td>
<td></td>
</tr>
<tr>
<td>Element standardized</td>
<td>Compound used</td>
<td>Solution</td>
</tr>
<tr>
<td>----------------------</td>
<td>-----------------------</td>
<td>--------------------------------------------------------------------------</td>
</tr>
<tr>
<td>Ce</td>
<td>CeO₂, J and M</td>
<td>H₂SO₄, conc., heated to form amber, Ce(SO₄)₂. 6 percent H₂SO₃ added to form colorless Ce₂(SO₄)₃. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Co</td>
<td>CoCl₂·6H₂O, C.P.</td>
<td></td>
</tr>
<tr>
<td>Cr</td>
<td>Cr, metal J and M</td>
<td>1:1 H₂SO₄</td>
</tr>
<tr>
<td>Cs</td>
<td>CsCl, C.P.</td>
<td></td>
</tr>
<tr>
<td>Cu</td>
<td>CuO, reagent J and M</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Dy</td>
<td>Dy₂O₃, J and M</td>
<td>1:1 HCl. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Er</td>
<td>Er₂O₃, J and M</td>
<td>1:1 HCl. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Eu</td>
<td>Eu₂O₃, J and M</td>
<td>1:1 HCl, heated. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>F</td>
<td>CaCl₂, C.P. and NaF, C.P.</td>
<td>H₂O (2 solutions).</td>
</tr>
<tr>
<td>Fe</td>
<td>Fe, metal J and M</td>
<td>Dilute H₂SO₄.</td>
</tr>
<tr>
<td>Ga</td>
<td>Ga, metal, C.P.</td>
<td>Aqua regia. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Ge</td>
<td>GeO₂, C.P.</td>
<td>HF, 48 percent. H₂SO₄, conc., added and heated to drive off HF. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Gd</td>
<td>Gd₂O₃, J and M</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Hf</td>
<td>HfO₂, J and M</td>
<td>Dilute H₂SO₄, heated and H₂O₂, 3 percent, added until dissolved. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Hg</td>
<td>HgCl₂, reagent</td>
<td></td>
</tr>
<tr>
<td>Ho</td>
<td>Ho₂O₃, J and M</td>
<td>1:1 HCl, heated. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Element standardized</td>
<td>Compound used</td>
<td>Solution</td>
</tr>
<tr>
<td>----------------------</td>
<td>---------------</td>
<td>----------</td>
</tr>
<tr>
<td>In</td>
<td>In, metal, J and M</td>
<td>HNO₃, conc. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Ir</td>
<td>Ir metal powder, C.P.</td>
<td>Fused with 3 parts KOH and 1 part KNO₃. Fusion dissolved in aqua regia. SiO₂ filtered off. Filtrate boiled down to small volume. Crystals of K₂IrCl₆ separate upon cooling and dissolve in H₂O.</td>
</tr>
<tr>
<td>K</td>
<td>HKC₆H₄O₄, Nat. Bur. Stand.</td>
<td></td>
</tr>
<tr>
<td>La</td>
<td>La₂O₃, J and M</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Li</td>
<td>Li₂CO₃, reagent</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Lu</td>
<td>Lu₂O₃, J and M</td>
<td>1:1 HCl, heated. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Mg</td>
<td>Mg, metal, J and M</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Mn</td>
<td>MnCl₂·4H₂O, C.P.</td>
<td></td>
</tr>
<tr>
<td>Mo</td>
<td>Mo, metal, J and M</td>
<td>Aqua regia, heated. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Na</td>
<td>NaCl, reagent</td>
<td></td>
</tr>
<tr>
<td>Nd</td>
<td>Nd₂O₃, J and M</td>
<td>1:1 HCl. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Ni</td>
<td>Ni, metal, J and M</td>
<td>1:1 HNO₃, heated. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Os</td>
<td>Os metal powder, C.P.</td>
<td>Os metal powder heated with aqua regia in flask fitted with reflux condenser.</td>
</tr>
<tr>
<td>P</td>
<td>NaH₂PO₄·H₂O C.P.</td>
<td></td>
</tr>
<tr>
<td>Pb</td>
<td>Pb(NO₃)₂, C.P.</td>
<td></td>
</tr>
<tr>
<td>Pd</td>
<td>Pd, wire, J and M</td>
<td>Aqua regia. Diluted to volume with H₂O.</td>
</tr>
<tr>
<td>Element standardized</td>
<td>Compound used</td>
<td>Solution</td>
</tr>
<tr>
<td>----------------------</td>
<td>---------------</td>
<td>----------</td>
</tr>
<tr>
<td>Pr</td>
<td>Pr&lt;sub&gt;6&lt;/sub&gt;O&lt;sub&gt;11&lt;/sub&gt;, J and M</td>
<td>1:1 HCl. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Pt</td>
<td>Pt, sheet</td>
<td>Aqua regia. Boiled down several times with HCl, conc., to drive off HNO&lt;sub&gt;3&lt;/sub&gt;. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Rb</td>
<td>RbCl, J and M</td>
<td></td>
</tr>
<tr>
<td>Re</td>
<td>Re, metal, J and M</td>
<td>HNO&lt;sub&gt;3&lt;/sub&gt;, conc. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Rh</td>
<td>RhCl&lt;sub&gt;3&lt;/sub&gt;, dry, C.P.</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Ru</td>
<td>(NH&lt;sub&gt;4&lt;/sub&gt;)&lt;sub&gt;2&lt;/sub&gt;RuCl&lt;sub&gt;5&lt;/sub&gt;, J and M</td>
<td>Hot H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Sb</td>
<td>SbI&lt;sub&gt;3&lt;/sub&gt;, C.P.</td>
<td>Acetone + HCl, dil.</td>
</tr>
<tr>
<td>Sc</td>
<td>Sc&lt;sub&gt;2&lt;/sub&gt;(SO&lt;sub&gt;4&lt;/sub&gt;)&lt;sub&gt;3&lt;/sub&gt;·5H&lt;sub&gt;2&lt;/sub&gt;O, J and M</td>
<td></td>
</tr>
<tr>
<td>Si</td>
<td>SiO&lt;sub&gt;2&lt;/sub&gt;, pure</td>
<td>Na&lt;sub&gt;2&lt;/sub&gt;CO&lt;sub&gt;3&lt;/sub&gt; fusion. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Sm</td>
<td>Sm&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;, J and M</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Sn</td>
<td>SnCl&lt;sub&gt;2&lt;/sub&gt;·2H&lt;sub&gt;2&lt;/sub&gt;O, reagent</td>
<td></td>
</tr>
<tr>
<td>Sr</td>
<td>SrCO&lt;sub&gt;3&lt;/sub&gt;, reagent</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Ta</td>
<td>Ta, metal, J and M</td>
<td>48 percent HF + HNO&lt;sub&gt;3&lt;/sub&gt;, conc. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Tb</td>
<td>Tb&lt;sub&gt;4&lt;/sub&gt;O&lt;sub&gt;7&lt;/sub&gt;, J and M</td>
<td>1:1 HCl, heated. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Te</td>
<td>H&lt;sub&gt;2&lt;/sub&gt;TeO&lt;sub&gt;4&lt;/sub&gt;·2H&lt;sub&gt;2&lt;/sub&gt;O, C.P.</td>
<td>1:6 HNO&lt;sub&gt;3&lt;/sub&gt;, heated.</td>
</tr>
<tr>
<td>Th</td>
<td>Th(NO&lt;sub&gt;3&lt;/sub&gt;)&lt;sub&gt;4&lt;/sub&gt;·4H&lt;sub&gt;2&lt;/sub&gt;O, C.P.</td>
<td></td>
</tr>
<tr>
<td>Ti</td>
<td>TiO&lt;sub&gt;2&lt;/sub&gt;, C.P.</td>
<td>48 percent HF + H&lt;sub&gt;2&lt;/sub&gt;O. H&lt;sub&gt;2&lt;/sub&gt;SO&lt;sub&gt;4&lt;/sub&gt;, conc., added and heated to drive off HF. Diluted to volume with H&lt;sub&gt;2&lt;/sub&gt;O.</td>
</tr>
<tr>
<td>Element standardized</td>
<td>Compound used</td>
<td>Solution</td>
</tr>
<tr>
<td>----------------------</td>
<td>--------------</td>
<td>----------</td>
</tr>
<tr>
<td>Tm</td>
<td>Tm$_2$O$_3$, J and M</td>
<td>1:1 HCl, heated. Diluted to volume with H$_2$O.</td>
</tr>
<tr>
<td>Tl</td>
<td>TlNO$_3$, C.P.</td>
<td></td>
</tr>
<tr>
<td>U</td>
<td>(UO$_2$)(C$_2$H$_3$O$_2$)$_2$·2H$_2$O C.P.</td>
<td></td>
</tr>
<tr>
<td>V</td>
<td>NH$_4$VC$_3$, C.P.</td>
<td>1:1 HCl. Diluted to volume with H$_2$O.</td>
</tr>
<tr>
<td>W</td>
<td>W, metal</td>
<td>48 percent HF + HNO$_3$, conc., heat. Diluted to volume with H$_2$O.</td>
</tr>
<tr>
<td>Y</td>
<td>Y$_2$O$_3$, J and M</td>
<td>1:1 HCl and heat. Diluted to volume with H$_2$O.</td>
</tr>
<tr>
<td>Yb</td>
<td>Yb$_2$O$_3$, J and M</td>
<td>1:1 HCl, heated. Diluted to volume with H$_2$O.</td>
</tr>
<tr>
<td>Zn</td>
<td>ZnO, reagent</td>
<td>Dilute HCl.</td>
</tr>
<tr>
<td>Zr</td>
<td>ZrOCl$_2$·8H$_2$O, C.P.</td>
<td></td>
</tr>
</tbody>
</table>