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Ringa Danielsson and Risto Saikkonen

# Chemical analysis of USGS-W-2, USGS-DNC-1 and USGS-BIR-1 standard rocks

Espoo 1985

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# CHEMICAL ANALYSIS OF USGS-W-2, USGS-DNC-1 AND USGS-BIR-1 STANDARD ROCKS

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Three U.S. Geological Survey mafic rock standards were analysed using wet chemical methods for the main components and emission spectroscopy for minor and trace elements. Lead was determined by isotope dilution mass spectrometry. The results are presented together with the ''best estimates'' given by F. J. Flanagan.

Key words: standard rocks, basalts, diabase, chemical analysis, wet methods, spectroscopy, Iceland, United States.

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## CONTENTS

## INTRODUCTION

Three new U.S. Geological Survey (USGS) mafic rock standards kindly provided by Dr. F. J. Flanagan were analysed at the Geochemistry Department, Geological Survey of Finland, during 1978—1979. The basalt BIR-1 was from near Reykjavik, Iceland, and the diabases DNC-1 and W-2 from the vicinity of Durham, North Carolina, and the Bull Run Quarry near Centreville, Virginia, respectively.

The experimental design was as described in USGS Professional Paper 840 aiming primarily to confirm the homogeneity of the rock standard samples. In this work each sample consisted of three bottles; two portions were taken from each bottle to make a set of six analytical portions for evaluating the precision and accuracy of the methods.

# ANALYTICAL METHODS

## Main components

The weight loss at 105°C was measured, after which six 1-g portions from each sample were fused with sodium carbonate. Silica was separated from the hydrochloric acid solution by double dehydration after which aliquots were taken and aluminium, calcium and magnesium were determined by atomic absorption spectrometry (AAS). The residual silica retained in the $R_2O_3$ group was recovered by dehydration from the sulphuric acid solution of this group. One aliquot of the solution containing the $R_2O_3$ group was taken for photometric determination of total iron by the 1.14-HCl method and one aliquot for colorimetric determination of titanium using Tiron. Aluminium was calculated by difference, calcium was precipitated with oxalic acid and magnesium with dibasic ammonium phosphate.

Another set of six portions (0.5 g) from each sample were decomposed by treatment with hydrofluoric-nitric-sulphuric acid, and aliquots were taken to measure manganese and phosphorus colorimetrically: manganese with potassium periodate, and phosphorus as the yellow molybdovanadophosphoric acid complex. Sodium was determined by flame emission spectrophotometry (AES), and potassium by atomic absorption spectrophotometry. A Jarrel-Ash 810 atomic absorption spectrophotometer was used for the atomic absorption and emission determinations. An air-acetylene flame was used for sodium, potassium and magnesium, and a nitrous oxide-acetylene flame for calcium and aluminium.

Ferrous iron was determined titrimetrically by the Pratt method. Ferric iron was calculated from values for total iron and ferrous iron. Combined water was obtained employing the Penfield method using lead oxide as a flux. The scheme for the main component analysis is shown in figure 1.

Each analyst analysed the six portions of his sample at the same time.

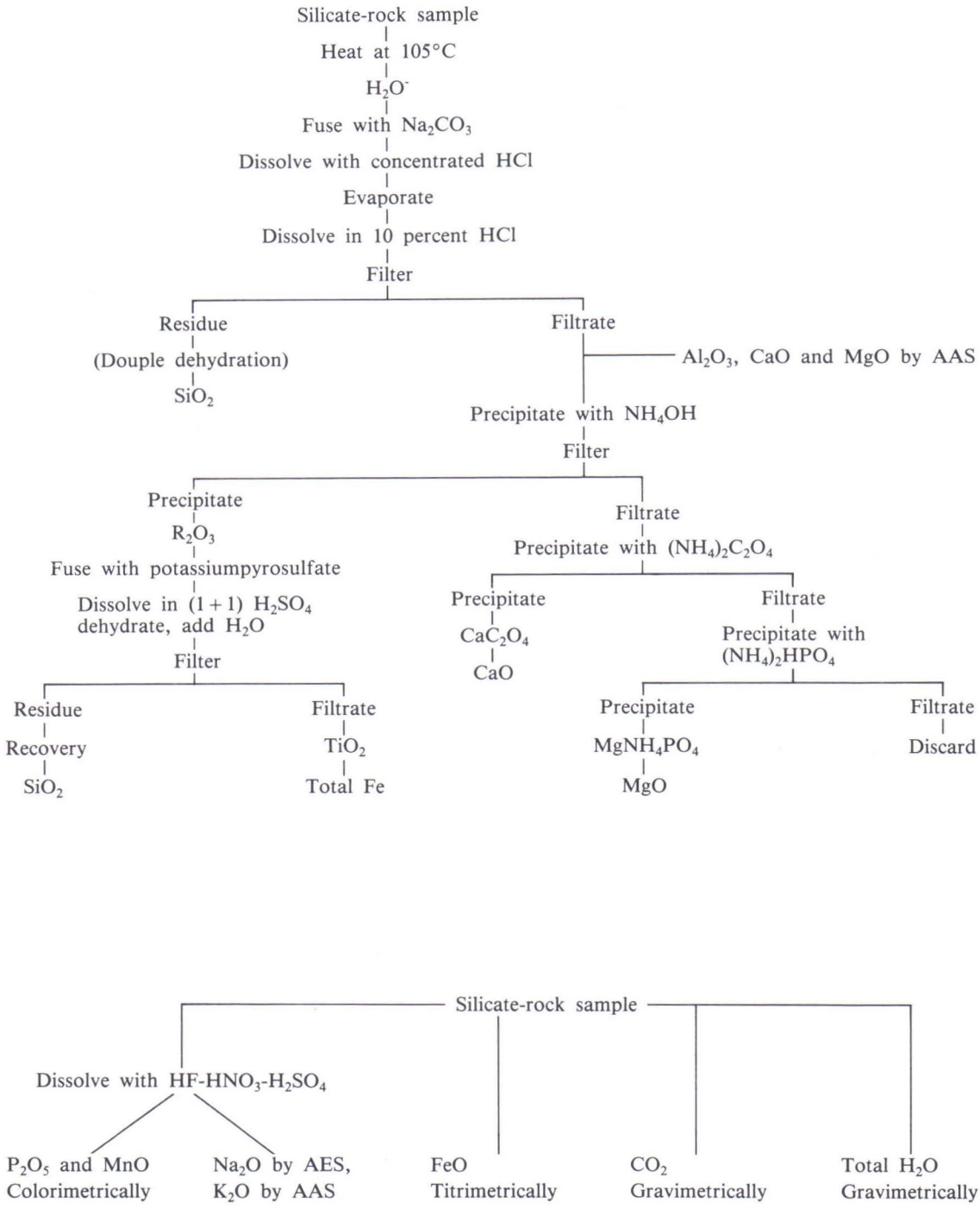
## Minor and trace elements

Lead was determined by isotope dilution mass spectrometry. Cobalt, chromium, copper, gallium, nickel, scandium, strontium, vanadium and yttrium were determined by optical emission spectroscopy. Table 8 lists the analytical and reference lines employed.

Six portions from each sample were weighed together with an equal amount of graphite, to which palladium had been added as an internal standard. The mixtures were packed into graphite electrodes and burned in the d-c arc of a Jaco Ebert plane grating 3.4 m spectrograph. The spectra were recorded on Kodak analysis plates and evaluated in a Jarrell-Ash microdensitometer. The "working curves" were drawn from spectra of well documented USGS, French and Canadian silicate-rock reference samples.

This procedure was performed by two analysts treating their own portions from each bottle. In this manner eighteen ($22 \times 3 \times 3$) spectra of each rock standard sample were recorded and evaluated during a period of three months, the analysts not knowing from which of the rock samples the portions originated.

**Figure 1.** The scheme for the main component analysis of silicate rock in this work.



## RESULTS AND DISCUSSION

### Main components

Tables 1, 2 and 3 give the arithmetic means ($\bar{x}$), the standard deviations (s), the coefficients of variation (v) and the methods used for bulk chemical analysis together with the "best estimates" of W-2, DNC-1 and BIR-1 (Flanagan 1984 and Abbey 1983).

The agreement between the results of the present study and the "best estimates" is good ($|\bar{x}-\bar{\bar{x}}| < s_{\bar{\bar{x}}}$, where $\bar{x}$ = the arithmetic mean, $\bar{\bar{x}}$ = the "best estimate" given by F. J. Flanagan, and $s_{\bar{\bar{x}}}$ = the standard deviation of the "best estimate") for 22 components and fairly good ($|\bar{x}-\bar{\bar{x}}| < 2s\bar{\bar{x}}$) for ten components. Some large differences are noted. In table 2, sample DNC-1, our value for $H_2O^+$ is 0.60% (the "best estimate" given by Flanagan is 0.73%). This difference is probably due to the fact that all the water had not escaped from the sample during the ignition in the Penfield method. There is a great numerical difference between our value 1.41% and the "best estimate" 1.65% for $Fe_2O_3$ (Table 1); we are not able to explain the reason for our low value. The lack of agreement between the MgO result of the present study, 6.72% (Table 1, gravimetric method) and the "best estimate", 6.39%, may be due to the carbon entrapped in our $Mg_2P_2O_7$ precipitate (Maxwell 1968, p. 165).

Analysis of variance did not show any significant variations between the contents in the three bottles of the same rock standards.

### Minor and trace elements

Tables 4, 5 and 6 give the arithmetic means (x), the standard deviations (s) and the coefficients of variation (v) together with the "best estimates" of W-2, DNC-1 and BIR-1 (Flanagan 1984 and Abbey 1983). Two chromium outliers from BIR-1 were discarded. A DNC-1 scandium line was abortive. All scandium values obtained, however, are reported.

Table 7 lists the lead contents of W-2, DNC-1 and BIR-1 determined by isotope dilution mass spectrometry.

The analytical lines of gallium and yttrium are embedded in a background. Hence the measurements done by different analysts may differ, aggravating the discrepancies of their results. All the samples are high in iron; BIR-1 and W-2 are also high in titanium. The iron interferes with gallium and the titanium with yttrium. The "old" USGS standard, W-1, widely used as a reference, would have been an excellent calibrating aid, but it is depleted. The good results obtained for vanadium are probably due to the availability of suitable rock standards. Our data are not sufficient for a t-test comparison of the results with the "best estimates" given by Flanagan. The lead values obtained by O. Kouvo are the only ones mentioned by Flanagan (Personal communication, 1983).

## ACKNOWLEDGEMENTS

We are indebted to Dr. Francis J. Flanagan of the U.S. Geological Survey for providing the samples, to the Geological Survey of Finland for providing the facilities, and to Mr. Christer Ahlsved, Dr. Olavi Kouvo, Mr. Ari Puisto and Mrs. Mervi Wiik for performing their part in the analytical work.

**Table 1.** Bulk chemical analyses of diabase W-2. Analyst Risto Saikkonen. $\bar{x}$ = arithmetic mean, s = standard deviation, v = coefficient of variation. Methods = methods used in this study. $\bar{\bar{x}}$ = the "best estimate", $s_{\bar{\bar{x}}}$ = standard deviation of the "best estimate", n = number of laboratory means from which $\bar{\bar{x}}$ was calculated (Flanagan 1984, Abbey 1983).

| | This study | | | | The "best estimates" | | | |
|---|---|---|---|---|---|---|---|---|
| | $\bar{x}$ % (dry basis) | s | v | Methods | $\bar{\bar{x}}$ % (dry basis) | $\bar{\bar{x}}$ % | n | $s_{\bar{\bar{x}}}$ |
| $SiO_2$ | 52.60 | 0.10 | 0.2 | Grav. | 52.81 | 52.68 | 18 | 0.29 |
| $TiO_2$ | 1.04 | 0.01 | 1.0 | Color. | 1.06 | 1.062 | 19 | 0.013 |
| $Al_2O_3$ | 15.51 | 0.03 | 0.2 | AAS | 15.49 | 15.45 | 17 | 0.16 |
| " | 15.41 | 0.13 | 0.8 | Grav. | | | | |
| $Fe_2O_3$ | 1.41 | 0.04 | 2.8 | Color. | 1.65 | 1.53 | 6 | 0.087 |
| FeO | 8.39 | 0.02 | 0.3 | Titr. | 8.36 | 8.34 | 6 | 0.093 |
| MnO | 0.16 | 0.00 | 0.0 | Color. | 0.167 | 0.167 | 20 | 0.004 |
| MgO | 6.63 | 0.01 | 0.2 | AAS | 6.39 | 6.37 | 13 | 0.058 |
| " | 6.82 | 0.06 | 0.9 | Grav. | | | | |
| CaO | 10.74 | 0.02 | 0.2 | Grav. | 10.89 | 10.86 | 11 | 0.078 |
| " | 10.68 | 0.14 | 1.3 | AAS | | | | |
| $Na_2O$ | 2.11 | 0.01 | 0.2 | AES | 2.21 | 2.20 | 17 | 0.037 |
| $K_2O$ | 0.63 | 0.01 | 1.0 | AAS | 0.63 | 0.626 | 20 | 0.012 |
| $H_2O^+$ | 0.58 | 0.02 | 3.0 | Grav. | 0.55 | 0.55 | 6 | 0.036 |
| $P_2O_5$ | 0.11 | 0.00 | 0.0 | Color. | 0.14 | 0.141 | 18 | 0.116 |
| $H_2O^-$ | | | | | | 0.25 | 3 | 0.018 |

**Table 2.** Bulk chemical analyses of diabase DNC-1. Analyst Mervi Wiik. $\bar{x}$ = arithmetic mean, s = standard deviation, v = coefficient of variation. Methods = methods used in this study. $\bar{\bar{x}}$ = the "best estimate", $s_{\bar{\bar{x}}}$ = standard deviation of the "best estimate", n = number of laboratory means from which $\bar{\bar{x}}$ was calculated (Flanagan 1984, Abbey 1983).

| | This study | | | | The "best estimates" | | | |
|---|---|---|---|---|---|---|---|---|
| | $\bar{x}$ % (dry basis) | s | v | Methods | $\bar{\bar{x}}$ % (dry basis) | $\bar{\bar{x}}$ % | n | $s_{\bar{\bar{x}}}$ |
| $SiO_2$ | 47.39 | 0.04 | 0.1 | Grav. | 47.29 | 47.15 | 11 | 0.21 |
| $TiO_2$ | 0.50 | 0.00 | 0.0 | Color. | 0.49 | 0.484 | 16 | 0.007 |
| $Al_2O_3$ | 18.54 | 0.05 | 0.2 | AAS | 18.39 | 18.34 | 16 | 0.169 |
| " | 18.76 | 0.09 | 0.5 | Grav. | | | | |
| $Fe_2O_3$ | 1.78 | 0.04 | 2.4 | Color. | 1.80 | 1.79 | 4 | 0.107 |
| FeO | 7.33 | 0.01 | 0.1 | Titr. | 7.34 | 7.32 | 4 | 0.062 |
| MnO | 0.14 | 0.00 | 0.0 | Color. | 0.148 | 0.148 | 14 | 0.003 |
| MgO | 10.32 | 0.01 | 0.1 | AAS | 10.16 | 10.13 | 15 | 0.112 |
| " | 10.21 | 0.15 | 1.5 | Grav. | | | | |
| CaO | 11.20 | 0.02 | 0.2 | AAS | 11.52 | 11.49 | 9 | 0.073 |
| " | 11.43 | 0.06 | 0.5 | Grav. | | | | |
| $Na_2O$ | 1.93 | 0.01 | 0.7 | AES | 1.89 | 1.886 | 21 | 0.057 |
| $K_2O$ | 0.23 | 0.00 | 0.0 | AAS | 0.23 | 0.234 | 15 | 0.009 |
| $H_2O^+$ | 0.60 | 0.04 | 6.0 | Grav. | 0.73 | 0.73 | 4 | 0.040 |
| $P_2O_5$ | 0.09 | 0.00 | 0.0 | Color. | 0.07 | 0.070 | 9 | 0.005 |
| $H_2O^-$ | | | | | | 0.29 | 3 | 0.047 |

**Table 3.** Bulk chemical analyses of basalt BIR-1. Analyst Christer Ahlsved. $\bar{x}$ = arithmetic mean, s = standard deviation, v = coefficient of variation. Methods = methods used in this study. $\bar{\bar{x}}$ = the "best estimate", $s_{\bar{\bar{x}}}$ = standard deviation of the "best estimate", n = number of laboratory means from which $\bar{\bar{x}}$ was calculated (Flanagan 1984, Abbey 1983).

| | This study | | | | The "best estimates" | | | |
|---|---|---|---|---|---|---|---|---|
| | $\bar{x}$ % (dry basis) | s | v | Methods | $\bar{\bar{x}}$ % (dry basis) | $\bar{\bar{x}}$ % | n | $s_{\bar{\bar{x}}}$ |
| $SiO_2$ | 47.85 | 0.06 | 0.1 | Grav. | 48.00 | 47.96 | 13 | 0.19 |
| $TiO_2$ | 0.96 | 0.00 | 0.0 | Color. | 0.96 | 0.96 | 15 | 0.010 |
| $Al_2O_3$ | 15.72 | 0.00 | 0.0 | AAS | 15.54 | 15.53 | 12 | 0.15 |
| " | 16.21 | 0.29 | 1.9 | Grav. | | | | |
| $Fe_2O_3$ | 2.08 | 0.00 | 0.0 | Color. | 2.06 | 2.06 | 5 | 0.104 |
| FeO | 8.30 | 0.01 | 0.1 | Titr. | 8.35 | 8.34 | 5 | 0.097 |
| MnO | 0.17 | 0.00 | 0.0 | Color. | 0.175 | 0.175 | 13 | 0.003 |
| MgO | 9.84 | 0.02 | 0.2 | AAS | 9.71 | 9.70 | 10 | 0.079 |
| " | 9.84 | 0.03 | 0.3 | Grav. | | | | |
| CaO | 13.30 | 0.02 | 0.1 | AAS | 13.33 | 13.32 | 16 | 0.12 |
| " | 13.33 | 0.03 | 0.2 | Grav. | | | | |
| NaO | 1.78 | 0.00 | 0.0 | AES | 1.82 | 1.820 | 16 | 0.045 |
| $K_2O$ | 0.03 | 0.00 | 0.0 | AAS | 0.03 | 0.030 | 11 | 0.003 |
| $H_2O^+$ | 0.06 | 0.01 | 8.2 | Grav. | 0.086 | 0.086 | 5 | 0.025 |
| $P_2O_5$ | 0.02 | 0.00 | 0.0 | Color. | 0.021 | 0.021 | 4 | 0.014 |
| $H_2O^-$ | | | | | | 0.078 | 2 | 0.016 |

**Table 4.** Analysis of diabase W-2 by optical emission spectroscopy. Analysts Ari Puisto, AP, and Ringa Danielsson, RD. $\bar{x}$ = arithmetic mean, s = standard deviation, v = coefficient of variation. $\bar{\bar{x}}$ = the "best estimate", $s_{\bar{\bar{x}}}$ = standard deviation of the "best estimate", n = number of laboratory means from which $\bar{\bar{x}}$ was calculated (Flanagan 1984, Abbey 1983).

| | | This study | | | The "best estimates" | | |
|---|---|---|---|---|---|---|---|
| Element | | $\bar{x}$ ppm | s | v | $\bar{\bar{x}}$ ppm | n | $s_{\bar{\bar{x}}}$ |
| Co | AP | 39.1 | 4.96 | 12.6 | | | |
| | RD | 37.7 | 3.74 | 9.9 | | | |
| | Tot | 38.4 | 4.39 | 11.4 | 43.15 | 21 | 2.11 |
| Cr | AP | 143 | 18.0 | 12.6 | | | |
| | RD | 100 | 8.75 | 8.7 | | | |
| | Tot | 122 | 14.2 | 11.6 | 91.5 | 19 | 4.45 |
| Cu | AP | 116 | 18.6 | 16 | | | |
| | RD | 132 | 10.8 | 8.2 | | | |
| | Tot | 124 | 15.2 | 12.2 | 106.2 | 10 | 4.88 |
| Ga | AP | 27.8 | 3.67 | 13 | | | |
| | RD | 15.6 | 1.64 | 11 | | | |
| | Tot | 21.7 | 2.84 | 13 | 16.8 | 4 | 0.89 |
| Ni | AP | 86.6 | 7.21 | 8.3 | | | |
| | RD | 85.0 | 5.17 | 6.1 | | | |
| | Tot | 85.8 | 6.28 | 7.3 | 70.4 | 10 | 2.46 |
| Sc | AP | 44.1 | 10.3 | 23.4 | | | |
| | RD | 37.4 | 9.08 | 24.3 | | | |
| | Tot | 40.8 | 9.72 | 23.8 | 35.7 | 9 | 1.06 |
| V | AP | 262 | 13.9 | 5.3 | | | |
| | RD | 266 | 12.4 | 4.7 | | | |
| | Tot | 264 | 13.2 | 5 | 259 | 13 | 12.27 |
| Y | AP | 33.4 | 3.91 | 11.7 | | | |
| | RD | 27.1 | 4.46 | 16.5 | | | |
| | Tot | 30.3 | 4.19 | 13.3 | 23.0 | 7 | 1.63 |

**Table 5.** Analysis of diabase DNC-1 by optical emission spectroscopy. Analysts Ari Puisto, AP, and Ringa Danielsson, RD. $\bar{x}$ = arithmetic mean, s = standard deviation, v = coefficient of variation. $\bar{\bar{x}}$ = the "best estimate", $s_{\bar{\bar{x}}}$ = standard deviation of the "best estimate", n = number of laboratory means from which $\bar{\bar{x}}$ was calculated (Flanagan 1984, Abbey 1983).

| | | This study | | | The "best estimates" | | |
|---|---|---|---|---|---|---|---|
| Element | | $\bar{x}$ ppm | s | v | $\bar{\bar{x}}$ ppm | n | $s_{\bar{\bar{x}}}$ |
| Co | AP | 50.0 | 4.87 | 9.7 | | | |
| | RD | 50.5 | 4.43 | 8.8 | | | |
| | Tot | 50.3 | 4.66 | 9.9 | 56.75 | 18 | 2.19 |
| Cr | AP | 403 | 44.8 | 11 | | | |
| | RD | 303 | 19.1 | 6 | | | |
| | Tot | 356 | 35.2 | 10 | 270.1 | 12 | 8.48 |
| Cu | AP | 112 | 13.8 | 12.3 | | | |
| | RD | 97.7 | 7.87 | 8.0 | | | |
| | Tot | 105 | 11.3 | 10.8 | 99.7 | 7 | 2.64 |
| Ga | AP | 18.9 | 2.67 | 14.1 | | | |
| | RD | 12.6 | 2.83 | 22.5 | | | |
| | Tot | 15.9 | 2.75 | 17.3 | 14.7 | 5 | 0.92 |
| Ni | AP | 326 | 27.4 | 8.4 | | | |
| | RD | 311 | 41.3 | 13.3 | | | |
| | Tot | 318 | 35.1 | 11.0 | 247 | 12 | 11.78 |
| Sc | AP | 34.1 | 5.67 | 16.6 | | | |
| | RD | 35.0[1] | 6.30 | 18 | | | |
| | Tot | 34.5 | 5.97 | 17.9 | 31.4 | 10 | 0.98 |
| Sr | AP | — | — | — | | | |
| | RD | 145.7 | 22.7 | 15.6 | 144 | 7 | 1.77 |
| | Tot | — | — | — | | | |
| V | AP | 143 | 7.07 | 4.9 | | | |
| | RD | 148 | 7.07 | 4.8 | | | |
| | Tot | 146 | 7.07 | 4.8 | 147.5 | 14 | 8.32 |
| Y | AP | 26.0 | 3.57 | 13.7 | | | |
| | RD | 26.4 | 4.19 | 15.9 | | | |
| | Tot | 26.2 | 3.89 | 18.7 | 18.5 | 4 | 0.82 |

[1] Calculated from eight measuring results.

**Table 6.** Analysis of basalt BIR-1 by optical emission spectroscopy. Analysts Ari Puisto, AP, and Ringa Danielsson, RD. $\bar{x}$ = arithmetic mean, s = standard deviation, v = coefficient of variation. $\bar{\bar{x}}$ = the "best estimate", $s_{\bar{\bar{x}}}$ = standard deviation of the "best estimate", n = number of laboratory means from which $\bar{\bar{x}}$ was calculated (Flanagan 1984, Abbey 1983).

| | | This study | | | The "best estimates" | | |
|---|---|---|---|---|---|---|---|
| Element | | $\bar{x}$ ppm | s | v | $\bar{\bar{x}}$ ppm | n | $s_{\bar{\bar{x}}}$ |
| Co | AP | 49.3 | 4.37 | 8.7 | | | |
| | RD | 48.6 | 2.40 | 4.9 | | | |
| | Tot | 49.0 | 3.53 | 7.2 | 51.6 | 18 | 1.88 |
| Cr | AP | 491[1] | 36.4 | 7.4 | | | |
| | RD | 405[1] | 44.2 | 10.9 | | | |
| | Tot | 448 | 40.5 | 9.0 | 372.5 | 7 | 8.25 |
| Cu | AP | 116 | 11.3 | 9.7 | | | |
| | RD | 128 | 12.0 | 9.4 | | | |
| | Tot | 122 | 11.7 | 9.6 | 124.7 | 11 | 3.71 |
| Ga | AP | 19.0 | 2.50 | 13.2 | | | |
| | RD | 13.6 | 2.17 | 16.0 | | | |
| | Tot | 16.3 | 2.34 | 14.4 | 15.0 | 5 | 1.0 |
| Ni | AP | 222 | 14.8 | 6.9 | | | |
| | RD | 221 | 16.9 | 7.65 | | | |
| | Tot | 222 | 15.9 | 7.2 | 166.4 | 9 | 5.88 |
| Sc | AP | 51.8 | 7.97 | 15.4 | | | |
| | RD | 52.9 | 8.01 | 15.1 | | | |
| | Tot | 52.3 | 7.99 | 15.3 | 43.4 | 8 | 1.13 |
| Sr | AP | — | — | — | | | |
| | RD | 110 | 12.5 | 11.3 | | | |
| | Tot | — | — | — | 107.2 | 7 | 1.49 |
| V | AP | 307 | 18.7 | 6.1 | | | |
| | RD | 336 | 23.6 | 7.0 | | | |
| | Tot | 321 | 21.3 | 6.4 | 311.6 | 12 | 11.47 |
| Y | AP | 25.0 | 3.96 | 15.8 | | | |
| | RD | 19.6 | 6.86 | 35.0 | | | |
| | Tot | 22.1 | 5.69 | 25.75 | 15.8 | 4 | 0.92 |

[1] Calculated from eight measuring results.

**Table 7.** Lead contents of W-2, DNC-1 and BIR-1. Analyst Dr. Olavi Kouvo. Lead was determined by isotope dilution mass spectrometry. These values are identical to those of the "best estimate" given by Dr. Flanagan (Flanagan 1984).

| W-2 | DNC-1 | BIR-1 |
|---|---|---|
| 7.66 ppm | 6.21 ppm | 3.11 ppm |

**Table 8.** Spectral lines used for the determination of minor and trace elements by optical emission spectroscopy.

| Element | λ (Ångström) |
|---|---|
| Co | 3453.50 |
| Cr | 2843.25, 4254.35 |
| Cu | 3273.96 |
| Ga | 2943.64 |
| Ni | 3414.76, 3492.96 |
| Sc | 4246.83 |
| Sr | 4077.71 |
| V | 3185.40 |
| Y | 3710.30 |
| (internal standard) | |
| Pd | 3242.70 (reference line) |

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