

Microprobe Analysis of WL 754 Sapphirine

29 June 1976

analysis of different grains of WL 754 to determine homogeneity using grain 1-1 Morse's analysis as standard.

Each analysis represents an average of 3 individual analyses except where

| | <u>FeO^T</u> | <u>Al₂O₃</u> | <u>SiO₂</u> | <u>MgO</u> | <u>Total</u> | <u>(+ MnO + TiO₂)</u> |
|----------------|------------------------|------------------------------------|------------------------|------------|--------------|----------------------------------|
| Morse Analysis | 10.67 | 57.04 | 14.19 | 16.84 | 98.74 | 0.38 |
| S-1 | 10.88 | 56.95 | 13.38 | 17.43 | 98.63 | |
| 1-1 | 10.18 | 57.62 | 13.25 | 17.94 | 98.99 | |
| 2-1 | 10.75 | 56.12 | 13.66 | 17.79 | 98.32 | |
| 3-1 | 10.82 | 56.61 | 13.40 | 17.60 | 98.43 | |
| S-2 | 10.79 | 56.75 | 12.57 | 17.77 | 97.88 | |
| 4-1 | 10.43 | 57.30 | 12.63 | 17.66 | 98.01 | |
| 5-1 | 11.06 | 56.30 | 13.29 | 17.70 | 98.35 | |
| 6-1 | 10.62 | 56.98 | 12.96 | 17.67 | 98.23 | |
| S-3 | 10.85 | 56.86 | 13.14 | 17.61 | 98.45 | |
| 7-1 | 10.28 | 57.88 | 13.06 | 17.82 | 99.03 | |
| 8-1 | 10.25 | 57.02 | 13.34 | 18.07 | 98.68 | |
| 9-1 | 10.53 | 57.21 | 13.00 | 17.73 | 98.46 | |
| * S-4 | 10.66 | 56.66 | 12.96 | 17.54 | 97.82 | |
| S-5 | 10.87 | 56.36 | 13.03 | 17.66 | 97.92 | |
| ** S-6 | 11.00 | 56.18 | 13.11 | 17.76 | 98.04 | |
| Average | 10.66 | 56.85 | 13.12 | 17.72 | | |

* - three analyses on same spot

** - one analysis

~~The following analysis was performed on 7/25/76 using a different MgO standard. The results are as follows:~~

:SAPPHIRINE ANALYSIS JUN 29 76:

REFERENCE CURRENT 150

KEY PRESET TIME(SEC.) AND COUNT:20,30000:

WHAT PROGRAM NEXT?

:ANALYSIS PROGRAM

NO. OF STD. REPEATS AND CORRECTION TYPE:3,0:

CH,EL NAME,PEAK POS,BF,OFFSET,CONC,BETA,GRF,BASE,WINDOW,STD NAME

:1,FE,19343,1.00,500,C

:2,AL,31498,1.00,500,C

:3,SI,17111,1.00,500,C

from John Heegins

Sapphirine WL 754 Wilson Lake, Labrador, Canada

from: DR. S.A. Morse, Univ. of Mass.

Mossbauer Analysis by: R.G. Burns

| peaks | isomer shifts | Quadrupole Splitting | % Area | Assignment |
|-------|---------------|----------------------|--------|---------------------------------------|
| 1+8 | 1.12 mm/sec | 2.68 mm/sec | 35.3 | Fe ²⁺ in M5 + M6 ← |
| 2+7 | 1.06 | 2.25 | 13.8 | Fe ²⁺ in M4 |
| 4+6 | 1.15 | 1.49 | 7.0 | Fe ²⁺ in M3 |
| 3+5 | 0.37 | 0.825 | 43.9 | Fe ³⁺ in predominantly oct |

Chemical Analysis:

| | wt. % | at. wt. | at. pt. | oxygens | cations | |
|--------------------------------|-------|---------|---------------------|---------|---------|----------------|
| SiO ₂ | 14.19 | 60.08 | .2362 | .4724 | 1.714 | 1.714 |
| Al ₂ O ₃ | 57.04 | 101.96 | .5594 | 1.678 | 8.120 | 4.286 3.834 |
| TiO ₂ | 0.10 | 79.90 | .0012 | .0024 | .0008 | |
| Fe ₂ O ₃ | 5.16 | 159.69 | .0323 ³⁺ | .0969 | .4689 | |
| FeO | 6.05 | 71.85 | .0842 ²⁺ | .0842 | .6111 | |
| MnO | 0.28 | 70.94 | .0039 | .0039 | .0283 | |
| MgO | 16.84 | 40.31 | .4178 | .4178 | 3.0324 | |
| total | 99.66 | | | 2.7556 | | |

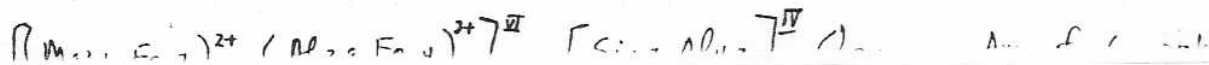
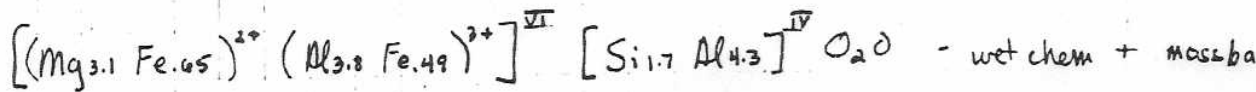
$$20/2.7556 = 7.2579$$

total Fe = 8.3 wt%

total FeO = 10.67 wt%

from Mossbauer results 43.5% of Fe is in ferric state

i.e. Fe₂O₃ = 5.16% , FeO = 6.05%



Microprobe Analysis of WL 754 sapphirine

28 June 1976

Standards - *M. olivine* - Fe, Mg
 Sillimanite - Al, Si

SAPPHIRINE ANALYSIS

SUBTITLE?:1-1:

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| C1 | 8.67 | 26.97 | 6.40 | 10.55 | |
| BC | 11.16 | 50.96 | 13.70 | 17.49 | |
| AC | 11.07 | 58.27 | 14.46 | 17.30 | 101.09 |
| FM | 1.107 | 8.228 | 1.729 | 3.087 | 14.151 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| C1 | 8.87 | 27.14 | 6.36 | 10.48 | |
| BC | 11.41 | 51.27 | 13.59 | 17.38 | |
| AC | 11.32 | 58.63 | 14.35 | 17.21 | 101.50 |
| FM | 1.127 | 8.249 | 1.712 | 3.061 | 14.149 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| C1 | 8.59 | 26.67 | 6.43 | 10.65 | |
| BC | 11.05 | 50.39 | 13.75 | 17.66 | |
| AC | 10.96 | 57.69 | 14.50 | 17.46 | 100.61 |
| FM | 1.102 | 8.183 | 1.745 | 3.129 | 14.158 |

RESPONSE:N:

AVERAGE COMP.

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| SD | .14 | .23 | .04 | .09 | |
| C1 | 8.71 | 26.93 | 6.39 | 10.56 | |
| BC | 11.20 | 50.88 | 13.68 | 17.51 | |
| AC | 11.11 | 58.19 | 14.44 | 17.32 | 101.06 |
| FM | 1.111 | 8.218 | 1.730 | 3.091 | 14.150 |

Mg is too high with olivine std.

SUBTITLE?:2-1:

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.64 | 26.15 | 6.54 | 11.00 | |
| BC | 11.11 | 49.41 | 13.98 | 18.23 | |
| AC | 11.03 | 56.75 | 14.73 | 18.03 | 100.5 |
| FM | 1.110 | 8.062 | 1.775 | 3.240 | 14.18 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.73 | 26.14 | 6.40 | 11.10 | |
| BC | 11.23 | 49.40 | 13.70 | 18.41 | |
| AC | 11.14 | 56.81 | 14.45 | 18.21 | 100.6 |
| FM | 1.122 | 8.076 | 1.741 | 3.273 | 14.21 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.73 | 26.33 | 6.37 | 11.14 | |
| BC | 11.23 | 49.75 | 13.63 | 18.48 | |
| AC | 11.14 | 57.20 | 14.38 | 18.27 | 101.0 |
| FM | 1.117 | 8.098 | 1.727 | 3.271 | 14.21 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.64 | 26.30 | 6.27 | 11.03 | |
| BC | 11.12 | 49.70 | 13.41 | 18.28 | |
| AC | 11.04 | 57.11 | 14.16 | 18.07 | 100.3 |
| FM | 1.114 | 8.139 | 1.710 | 3.257 | 14.21 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.61 | 26.37 | 6.41 | 11.08 | |
| BC | 11.08 | 49.83 | 13.71 | 18.37 | |
| AC | 11.00 | 57.23 | 14.47 | 18.15 | 100.8 |
| FM | 1.103 | 8.104 | 1.737 | 3.250 | 14.19 |

RESPONSE: A:

AVERAGE COMP.

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| SD | .05 | .11 | .09 | .06 | |
| C1 | 8.67 | 26.26 | 6.39 | 11.07 | |
| BC | 11.15 | 49.61 | 13.68 | 18.35 | |
| AC | 11.07 | 57.02 | 14.43 | 18.14 | 100.6 |
| FM | 1.113 | 8.094 | 1.734 | 3.254 | 14.20 |

Microprobe Analysis of WZ 754 sapphirine

28 June 1976

standards - K. pyrope for Si, Al, Mg, Fe.

SAPPHIRINE ANALYSIS

SUBTITLE?:2-1:

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| C1 | 8.67 | 30.59 | 5.90 | 10.59 | |
| BC | 11.15 | 57.80 | 12.62 | 17.56 | |
| AC | 11.11 | 56.65 | 14.07 | 17.15 | 98.98 |
| FM | 1.136 | 8.180 | 1.722 | 3.132 | 14.170 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| C1 | 8.67 | 30.50 | 5.99 | 10.45 | |
| BC | 11.15 | 57.63 | 12.82 | 17.32 | |
| AC | 11.11 | 56.43 | 14.28 | 16.92 | 98.74 |
| FM | 1.139 | 8.170 | 1.752 | 3.097 | 14.158 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| C1 | 8.84 | 29.88 | 5.93 | 10.59 | |
| BC | 11.37 | 56.45 | 12.68 | 17.55 | |
| AC | 11.32 | 55.46 | 14.12 | 17.18 | 98.08 |
| FM | 1.171 | 8.098 | 1.749 | 3.171 | 14.189 |

RESPONSE:A:

- AVERAGE COMP.

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|--------|
| SD | .10 | .39 | .05 | .08 | |
| C1 | 8.72 | 30.32 | 5.94 | 10.54 | |
| BC | 11.22 | 57.30 | 12.70 | 17.47 | |
| AC | 11.18 | 56.18 | 14.15 | 17.08 | 98.59 |
| FM | 1.149 | 8.153 | 1.742 | 3.133 | 14.177 |

→ Converting to total Fe as FeO to correct proportions of FeO and Fe₂O₃

Total FeO = 11.13

Total Fe = 8.69

Fe³⁺ = 8.69 × .435 = 3.78

Fe²⁺ = 4.91

Fe³⁺ × 1.4297 = Fe₂O₃ - 3.78 × 1.4297 = 5.40 % Fe₂O₃

Fe²⁺ × 1.2865 = FeO - 4.91 × 1.2865 = 6.32 % FeO

SAPPHIRINE ANALYSIS

SUBTITLE?:2-2: = 1-1 grain

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.78 | 30.71 | 5.83 | 10.50 | |
| BC | 11.30 | 58.02 | 12.47 | 17.41 | |
| AC | 11.25 | 56.85 | 13.91 | 17.02 | 99.0 |
| FM | 1.151 | 8.215 | 1.706 | 3.109 | 14.11 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.24 | 30.79 | 5.88 | 10.47 | |
| BC | 10.60 | 58.18 | 12.57 | 17.36 | |
| AC | 10.57 | 56.87 | 14.03 | 16.90 | 98.3 |
| FM | 1.086 | 8.242 | 1.725 | 3.096 | 14.14 |

RESPONSE::

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| C1 | 8.60 | 30.77 | 5.87 | 10.39 | |
| BC | 11.06 | 58.14 | 12.55 | 17.23 | |
| AC | 11.03 | 56.88 | 14.00 | 16.82 | 98.7 |
| FM | 1.131 | 8.229 | 1.718 | 3.076 | 14.15 |

RESPONSE:A:

AVERAGE COMP.

| | FE | AL | SI | MG | TOTAL |
|----|-------|-------|-------|-------|-------|
| SD | .27 | .05 | .02 | .06 | |
| C1 | 8.54 | 30.76 | 5.86 | 10.45 | |
| BC | 10.98 | 58.12 | 12.53 | 17.33 | |
| AC | 10.95 | 56.86 | 13.98 | 16.91 | 98.7 |
| FM | 1.121 | 8.230 | 1.715 | 3.095 | 14.16 |

SELECT ANALYSIS POSITION AND PUSH BUTTON WHEN READY

DATA REDUCTION BY METHOD OF BENCE & ALBEE:

ON SPECIMEN: WILSON LAKE SAPPHIRINE

| ELEMENT | WEIGHT% (OXIDE) | STD.DEVI. (PERCENT) | FORMULA | K-RATIO | UNKN PEAK (COUNTS) | UNKN BKGD (COUNTS) | COUNTING TIME(SEC) | STD P (COUN | |
|---------|--------------------|------------------------|---------|----------------|-----------------------|-----------------------|-----------------------|----------------|-------|
| TI | KA | 0.011 | 186.07 | 0.001 | 0.0003 | 700.0 | 480.0 | 10.00 | 72828 |
| MN | KA | 0.397 | 12.59 | 0.049 | 0.0053 | 217.0 | 80.0 | 10.00 | 25981 |
| NA | KA | 0.010 | 132.56 | 0.003 | 0.0008 | 46.0 | 39.0 | 10.00 | 8590 |
| CA | KA | 0.026 | 67.49 | 0.004 | 0.0013 | 257.5 | 225.0 | 10.00 | 25522 |
| SI | KA | 13.333 | 3.02 | 1.941 | 0.2680 | 1439.7 | 20.0 | 10.00 | 5316 |
| AL | KA | 58.737 | 1.97 | 10.076 | 1.4383 | 6373.7 | 18.0 | 10.00 | 4437 |
| K | KA | 0.000 | 141.16 | 0.000 | 0.0000 | 157.2 | 170.0 | 10.00 | 31765 |
| MG | KA | 17.308 | 1.17 | 3.755 | 0.3714 | 10338.5 | 100.0 | 10.00 | 27663 |
| FE | KA | 9.806 | 2.03 | 1.194 | 0.1360 | 2979.0 | 80.0 | 10.00 | 21398 |
| TOTAL | | 99.627 | | 24.000 OXYGENS | | | | ITERATIONS= 3 | |

16-APR-79 15:34:13

ANOTHER PT (Y,YES,K,NO) ?Y

SELECT ANALYSIS POSITION AND PUSH BUTTON WHEN READY

DATA REDUCTION BY METHOD OF BENCE & ALBEE:

ON SPECIMEN: WILSON LAKE SAPPHIRINE

| ELEMENT | WEIGHT% (OXIDE) | STD.DEVI. (PERCENT) | FORMULA | K-RATIO | UNKN PEAK (COUNTS) | UNKN BKGD (COUNTS) | COUNTING TIME(SEC) | STD P (COUN | |
|---------|--------------------|------------------------|---------|----------------|-----------------------|-----------------------|-----------------------|----------------|-------|
| TI | KA | 0.007 | 272.45 | 0.001 | 0.0002 | 693.6 | 680.0 | 10.00 | 72828 |
| MN | KA | 0.408 | 12.32 | 0.050 | 0.0054 | 221.0 | 80.0 | 10.00 | 25981 |
| NA | KA | 0.017 | 84.05 | 0.005 | 0.0013 | 50.2 | 39.0 | 10.00 | 8590 |
| CA | KA | 0.000 | 143.72 | 0.000 | 0.0000 | 210.5 | 225.0 | 10.00 | 25522 |
| SI | KA | 14.193 | 2.95 | 2.041 | 0.2862 | 1535.8 | 20.0 | 10.00 | 5316 |
| AL | KA | 58.297 | 1.97 | 9.879 | 1.4230 | 6306.1 | 18.0 | 10.00 | 4437 |
| K | KA | 0.000 | 137.20 | 0.000 | 0.0000 | 156.8 | 170.0 | 10.00 | 31765 |
| MG | KA | 17.512 | 1.16 | 3.753 | 0.3736 | 10396.7 | 100.0 | 10.00 | 27663 |
| FE | KA | 10.763 | 1.94 | 1.294 | 0.1494 | 3263.9 | 80.0 | 10.00 | 21398 |
| TOTAL | | 101.196 | | 24.000 OXYGENS | | | | ITERATIONS= 3 | |

16-APR-79 15:35:21

ANOTHER PT (Y,YES,K,NO) ?

WHEN READY

EE:

| ID | UNKN PEAK (COUNTS) | UNKN BKGD (COUNTS) | COUNTING TIME(SEC) | STD PEAK (COUNTS) | STD BKGD (COUNTS) | COUNTING TIME(SEC) | STANDARD FILENAME | OXIDE FORMULA |
|----|-----------------------|-----------------------|-----------------------|----------------------|----------------------|-----------------------|----------------------|------------------|
| 3 | 700.0 | 680.0 | 10.00 | 72828.8 | 680.0 | 10.00 | XSPHN | TI02 |
| 3 | 217.0 | 80.0 | 10.00 | 25981.9 | 80.0 | 10.00 | XTEPH | HNO |
| 3 | 46.0 | 39.0 | 10.00 | 8590.3 | 39.0 | 10.00 | XTAB | HA20 |
| 0 | 257.5 | 225.0 | 10.00 | 25522.5 | 225.0 | 10.00 | XAN | CA0 |
| 0 | 1439.7 | 20.0 | 10.00 | 5316.7 | 20.0 | 10.00 | XAN | SI02 |
| 3 | 6373.7 | 18.0 | 10.00 | 4437.0 | 18.0 | 10.00 | XAN | AL203 |
| 0 | 157.2 | 170.0 | 10.00 | 31765.3 | 170.0 | 10.00 | XORTH | K20 |
| 4 | 10338.5 | 100.0 | 10.00 | 27663.9 | 100.0 | 10.00 | XHARJ | H00 |
| 0 | 2979.0 | 80.0 | 10.00 | 21398.1 | 80.0 | 10.00 | XFAY | FE0 |

NS ITERATIONS= 3

WHEN READY

EE:

| ID | UNKN PEAK (COUNTS) | UNKN BKGD (COUNTS) | COUNTING TIME(SEC) | STD PEAK (COUNTS) | STD BKGD (COUNTS) | COUNTING TIME(SEC) | STANDARD FILENAME | OXIDE FORMULA |
|----|-----------------------|-----------------------|-----------------------|----------------------|----------------------|-----------------------|----------------------|------------------|
| 2 | 693.6 | 680.0 | 10.00 | 72828.8 | 680.0 | 10.00 | XSPHN | TI02 |
| 4 | 221.0 | 80.0 | 10.00 | 25981.9 | 80.0 | 10.00 | XTEPH | HNO |
| 3 | 50.2 | 39.0 | 10.00 | 8590.3 | 39.0 | 10.00 | XTAB | HA20 |
| 0 | 210.5 | 225.0 | 10.00 | 25522.5 | 225.0 | 10.00 | XAN | CA0 |
| 2 | 1535.8 | 20.0 | 10.00 | 5316.7 | 20.0 | 10.00 | XAN | SI02 |
| 0 | 6306.1 | 18.0 | 10.00 | 4437.0 | 18.0 | 10.00 | XAN | AL203 |
| 0 | 156.8 | 170.0 | 10.00 | 31765.3 | 170.0 | 10.00 | XORTH | K20 |
| 6 | 10396.7 | 100.0 | 10.00 | 27663.9 | 100.0 | 10.00 | XHARJ | H00 |
| 4 | 3263.9 | 80.0 | 10.00 | 21398.1 | 80.0 | 10.00 | XFAY | FE0 |

NS ITERATIONS= 3