

TABLE 3. (on deposit). Crystal and structure refinement data of tsilaisite.

| | | |
|--|------------------|-------------------------|
| a (Å) | 15.9461(5) | |
| c | 7.1380(3) | |
| V (Å ³) | 1571.9(1) | |
| Space group | $R3m$ | |
| Z | 3 | |
| D_{calc} (g cm ⁻³) | 3.133 | |
| Radiation | MoK α | |
| Monochromator | graphite | |
| Set of measured reflections | 1682 | |
| | <u>Standard*</u> | <u>Split-site SREF*</u> |
| $wR2$ (%) | 5.16 | 4.90 |
| $R1$ (%) for $I_0 > 2\sigma(I_0)$ | 2.05 | 1.91 |
| $R1$ (%) for all reflections | 2.12 | 1.97 |
| GooF | 1.122 | 1.135 |
| Extinction coefficient | 0.0071(3) | 0.0070(3) |
| Flack parameter | 0.13(2) | 0.13(2) |
| Larg. diff. peak-hole (e Å ⁻³) | 1.19 and -0.45 | 0.37 and -0.53 |

*Standard and Split-site SREF denote, respectively, structural refinements carried out with the O1 site at (0,0, z) and the O2 site at ($x,2x,z$), and with O1 at ($x,2x,z$) and O2 at (x,y,z) to allow for positional disorder, as indicated by the high U_{eq} values (Burns *et al.*, 1994).

TABLE 4. (on deposit). Final fractional atomic coordinates and equivalent displacement parameters (\AA^2).

| Site | Standard* | | | | Split-site SREF* | | | |
|------|-------------|-------------|-------------|------------------------|------------------|-------------|-------------|------------------------|
| | <i>x</i> | <i>y</i> | <i>z</i> | <i>U</i> _{eq} | <i>x</i> | <i>y</i> | <i>z</i> | <i>U</i> _{eq} |
| X | 0 | 0 | 0.22856(40) | 0.0269(8) | 0 | 0 | 0.22897(37) | 0.0261(8) |
| Y | 0.12399(4) | <i>x</i> /2 | 0.62408(9) | 0.0113(1) | 0.12400(4) | <i>x</i> /2 | 0.62407(8) | 0.0114(1) |
| Z | 0.29803(3) | 0.26129(3) | 0.61115(7) | 0.0060(1) | 0.29801(3) | 0.26127(3) | 0.61120(3) | 0.0060(1) |
| B | 0.10993(8) | 2 <i>x</i> | 0.45431(29) | 0.0067(3) | 0.10999(7) | 2 <i>x</i> | 0.45429(27) | 0.0070(3) |
| T | 0.19190(2) | 0.18997(3) | 0 | 0.00479(8) | 0.19191(2) | 0.18997(2) | 0 | 0.00470(8) |
| O1 | 0 | 0 | 0.77938(51) | 0.044(1) | 0.02087(23) | <i>x</i> /2 | 0.77961(47) | 0.0078(8)† |
| O2 | 0.06139(6) | 2 <i>x</i> | 0.48181(26) | 0.0197(4) | 0.07115(16) | 0.12293(12) | 0.48192(23) | 0.0101(4)† |
| O3 | 0.26843(13) | <i>x</i> /2 | 0.51001(22) | 0.0115(3) | 0.26862(12) | <i>x</i> /2 | 0.51004(20) | 0.0114(2) |
| O4 | 0.09351(6) | 2 <i>x</i> | 0.07011(21) | 0.0085(2) | 0.09348(5) | 2 <i>x</i> | 0.07006(19) | 0.0082(2) |
| O5 | 0.18698(11) | <i>x</i> /2 | 0.09173(20) | 0.0084(2) | 0.18697(10) | <i>x</i> /2 | 0.09176(19) | 0.0083(2) |
| O6 | 0.19732(7) | 0.18723(7) | 0.77527(15) | 0.0079(2) | 0.19727(7) | 0.18721(7) | 0.77531(14) | 0.0079(2) |
| O7 | 0.28539(7) | 0.28583(7) | 0.08002(14) | 0.0063(2) | 0.28533(7) | 0.28581(6) | 0.08008(13) | 0.0062(2) |
| O8 | 0.21011(7) | 0.27092(8) | 0.44140(15) | 0.0080(2) | 0.21009(7) | 0.27095(7) | 0.44150(14) | 0.0078(2) |
| H3 | 0.2594(27) | <i>x</i> /2 | 0.4005(51) | 0.01720† | 0.2559(24) | <i>x</i> /2 | 0.4085(48) | 0.01706† |

* Standard and Split-site SREF as in Table 3.

† Isotropic displacement parameter; for H3 was constrained to $1.5U_{eq}(O3)$

TABLE 5. (on deposit). Anisotropic displacement parameters (\AA^2) of tsilaisite.

| Site | Standard* | | | | | | Split-site SREF* | | | | | |
|------|-----------|-----------|-----------|------------|------------|-----------|------------------|-----------|-----------|------------|------------|-----------|
| | U^{11} | U^{22} | U^{33} | U^{23} | U^{13} | U^{12} | U^{11} | U^{22} | U^{33} | U^{23} | U^{13} | U^{12} |
| X | 0.030(1) | 0.030(1) | 0.021(1) | 0 | 0 | 0.0149(5) | 0.029(1) | 0.029(1) | 0.021(1) | 0 | 0.00 | 0.0143(5) |
| Y | 0.0108(2) | 0.0080(2) | 0.0160(2) | -0.0013(1) | -0.0026(2) | 0.0054(1) | 0.0108(2) | 0.0081(2) | 0.0162(2) | -0.0013(1) | -0.0027(1) | 0.0054(1) |
| Z | 0.0063(2) | 0.0073(2) | 0.0047(2) | 0.0005(1) | -0.0001(1) | 0.0035(1) | 0.0063(2) | 0.0072(2) | 0.0046(2) | 0.0006(1) | -0.0001(1) | 0.0035(1) |
| B | 0.0072(5) | 0.0059(7) | 0.0065(7) | 0.0003(6) | 0.0002(3) | 0.0029(4) | 0.0074(5) | 0.0071(7) | 0.0063(6) | 0.0002(5) | 0.0001(3) | 0.0035(4) |
| T | 0.0045(2) | 0.0048(1) | 0.0046(1) | -0.0004(1) | -0.0000(1) | 0.0029(1) | 0.0048(1) | 0.0047(1) | 0.0046(1) | -0.0004(1) | 0.0000(1) | 0.0023(1) |
| O1 | 0.064(2) | 0.064(2) | 0.005(1) | 0 | 0 | 0.032(1) | 0 | 0 | 0 | 0 | 0 | 0 |
| O2 | 0.0305(8) | 0.0060(6) | 0.0144(7) | 0.0005(5) | 0.0003(3) | 0.0030(3) | 0 | 0 | 0 | 0 | 0 | 0 |
| O3 | 0.0237(8) | 0.0104(4) | 0.0047(5) | -0.0003(3) | -0.0006(5) | 0.0119(4) | 0.0235(7) | 0.0105(4) | 0.0044(5) | -0.0003(2) | -0.0005(5) | 0.0118(4) |
| O4 | 0.0069(4) | 0.0125(6) | 0.0079(5) | -0.0013(5) | -0.0007(2) | 0.0063(3) | 0.0068(4) | 0.0116(6) | 0.0078(5) | -0.0013(4) | -0.0006(2) | 0.0058(3) |
| O5 | 0.0142(6) | 0.0066(4) | 0.0071(5) | 0.0009(2) | 0.0018(5) | 0.0071(3) | 0.0145(6) | 0.0064(3) | 0.0067(5) | 0.0007(2) | 0.0014(4) | 0.0072(3) |
| O6 | 0.0088(4) | 0.0095(4) | 0.0043(3) | -0.0004(3) | 0.0007(3) | 0.0038(3) | 0.0087(4) | 0.0096(4) | 0.0041(3) | -0.0007(3) | 0.0004(3) | 0.0037(3) |
| O7 | 0.0061(4) | 0.0053(4) | 0.0052(4) | -0.0006(3) | 0.0005(3) | 0.0013(3) | 0.0058(3) | 0.0054(3) | 0.0053(3) | -0.0006(3) | 0.0005(3) | 0.0013(3) |
| O8 | 0.0064(4) | 0.0116(4) | 0.0073(4) | 0.0027(3) | 0.0005(3) | 0.0055(3) | 0.0063(4) | 0.0115(4) | 0.0069(3) | 0.0027(3) | 0.0006(3) | 0.0055(3) |

* Standard and Split-site SREF as in Table 3.

TABLE 6. (on deposit). Relevant bond distances (Å), and mean atomic numbers (m.a.n.) of tsilaisite

| | Standard* | Split-site SREF* | | |
|------------|------------|------------------|------------|------------|
| B-O2 | 1.3551(27) | 1.3634(26) | | |
| B-O8 (× 2) | 1.3867(15) | 1.3854(14) | | |
| <B-O> | 1.376 | 1.378 | | |
| m.a.n. B | 5 | 5 | | |
| T-O4 | 1.6250(6) | 1.6250(5) | | |
| T-O5 | 1.6373(7) | 1.6374(6) | | |
| T-O7 | 1.6145(10) | 1.6140(9) | | |
| T-O6 | 1.6082(10) | 1.6078(10) | | |
| <T-O> | 1.621 | 1.621 | | |
| m.a.n. T | 14 | 14 | | |
| X-O2 (× 3) | 2.4784(27) | 2.4831(24) | | |
| X-O4 (× 3) | 2.8195(19) | 2.8020(18) | | |
| X-O5 (× 3) | 2.7607(18) | 2.7616(17) | | |
| <X-O> | 2.686 | 2.682 | | |
| m.a.n. X | 8.36(9) | 8.30(8) | | |
| | | Y1 | Y2 | Y3 |
| Y-O1 | 2.0398(20) | 2.1776(24) | 2.1776(24) | 1.8058(33) |
| Y-O2 | 1.9836(12) | 1.8715(20) | 2.1021(21) | 1.8715(20) |
| Y-O2 | 1.9836(12) | 2.1021(21) | 2.1021(21) | 1.8715(20) |
| Y-O3 | 2.1545(18) | 2.1566(17) | 2.1566(17) | 2.1566(17) |
| Y-O6 | 2.0456(11) | 2.0455(10) | 2.0455(10) | 2.0455(10) |
| Y-O6 | 2.0456(11) | 2.0455(10) | 2.0455(10) | 2.0455(10) |
| <Y-O> | 2.042 | 2.071 | 2.117 | 1.950 |
| m.a.n. Y | 16.46(5) | 16.56(4) | | |
| Z-O3 | 1.9732(8) | 1.9725(9) | | |
| Z-O6 | 1.8572(11) | 1.8575(9) | | |
| Z-O8 | 1.9163(11) | 1.9166(10) | | |
| Z-O7 | 1.9573(10) | 1.9582(9) | | |
| Z-O7' | 1.8786(10) | 1.8797(9) | | |
| Z-O8' | 1.8828(11) | 1.8828(10) | | |
| <Z-O> | 1.911 | 1.911 | | |
| m.a.n. Z | 13.22(5) | 13.23(4) | | |

*Standard and Split-site SREF as in Table 3.