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# Metalloporphyrin Mixed-Valence $\pi$-Cation Radicals: $[Fe(oxoOEC^{\bullet/2})(Cl)]_2SbCl_6$, Structure, Magnetic Properties, and Near-IR Spectra

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**Table S1.** Complete Crystallographic Details for $[Fe(oxoOEC^{\bullet/2})(Cl)]_2SbCl_6$

| | |
|---|---|
| formula | $C_{37}H_{46}Cl_6FeN_4OSb_{0.50}$ |
| FW, amu | 892.20 |
| $a$, Å | 10.333(2) |
| $b$, Å | 12.555(3) |
| $c$, Å | 15.849(3) |
| $\alpha$, deg | 83.64(3) |
| $\beta$, deg | 77.53(3) |
| $\gamma$, deg | 87.19(3) |
| $V$, Å$^3$ | 1994.6(7) |
| space group | $P\bar{1}$ |
| $Z$ | 2 |
| $D_c$, g/cm$^3$ | 1.486 |
| F(000) | 915 |
| $\mu$, mm$^{-1}$ | 1.146 |
| crystal dimensions, mm | 0.2 × 0.15 × 0.02 |
| radiation | MoK$\alpha$, $\bar{\lambda}$ = 0.71073 Å |
| temperature, K | 127(2) |
| diffractometer | Enraf-Nonius Fast Area Detectdor |
| $\theta$ range for collected data, deg | 2.20–25.50 |
| index range | $-12 \leq h \leq 12$ |
| | $-10 \leq k \leq 15$ |
| | $-19 \leq l \leq 19$ |
| total data collected | 14683 |
| absorption correction | Empirical (DIFFABS) |
| relative transmission coefficients (I) | 1.0 and 0.3477 |
| unique data | 7319 ($R_{int}$ = 0.1391) |
| unique observed data [I > 2$\sigma$(I)] | 4843 |
| refinement method | Full-matrix least-squares on F$^2$ |
| data/restraints/parameters | 7319/0/453 |
| goodness-of-fit (based on $F^2$) | 1.030 |
| final $R$ indices [I > 2$\sigma$(I)] | $R_1$ = 0.0983, $wR_2$ = 0.2397 |
| final $R$ indices (all data) | $R_1$ = 0.1633, $wR_2$ = 0.2935 |

**Table S2.** Atomic Coordinates and Equivalent Isotropic Displacement Parameters (Å$^2$) for $[Fe(oxoOEC^{\bullet/2})(Cl)]_2SbCl_6$[a]

| atom | $x$ | $y$ | $z$ | $U$(eq) |
|---|---|---|---|---|
| Fe(1) | 0.86665(13) | 0.20352(11) | 0.07576(9) | 0.0401(4) |
| Cl(1) | 0.6892(2) | 0.3098(2) | 0.07664(17) | 0.0484(6) |
| O(1) | 0.5883(8) | −0.0728(6) | 0.3321(5) | 0.058(2) |
| N(1) | 0.9778(8) | 0.2896(6) | 0.1358(5) | 0.0379(17) |
| N(2) | 0.8062(8) | 0.1111(6) | 0.1975(5) | 0.0377(17) |
| N(3) | 0.8331(8) | 0.0686(6) | 0.0191(5) | 0.0407(18) |
| N(4) | 0.9945(7) | 0.2511(6) | −0.0386(5) | 0.0393(18) |
| C(a1) | 1.0615(9) | 0.3705(8) | 0.0996(6) | 0.043(2) |
| C(a2) | 0.9688(11) | 0.2905(8) | 0.2245(7) | 0.048(2) |
| C(a3) | 0.8110(9) | 0.1408(8) | 0.2744(6) | 0.041(2) |
| C(a4) | 0.7281(10) | 0.0203(9) | 0.2080(7) | 0.049(3) |
| C(a5) | 0.7534(10) | −0.0170(8) | 0.0582(7) | 0.045(2) |
| C(a6) | 0.8606(9) | 0.0580(8) | −0.0685(7) | 0.045(2) |
| C(a7) | 1.0015(10) | 0.2163(9) | −0.1187(6) | 0.044(2) |
| C(a8) | 1.0713(10) | 0.3405(9) | −0.0546(7) | 0.049(3) |
| C(b1) | 1.1084(10) | 0.4226(8) | 0.1634(7) | 0.044(2) |
| C(b2) | 1.0506(9) | 0.3702(7) | 0.2425(6) | 0.039(2) |
| C(b3) | 0.7321(11) | 0.0730(8) | 0.3506(6) | 0.045(2) |
| C(b4) | 0.6698(10) | −0.0034(9) | 0.3023(7) | 0.049(2) |
| C(b5) | 0.7344(11) | −0.0836(9) | −0.0089(7) | 0.050(2) |
| C(b6) | 0.7991(10) | −0.0383(9) | −0.0846(7) | 0.048(2) |
| C(b7) | 1.0856(10) | 0.2823(9) | −0.1864(6) | 0.045(2) |
| C(b8) | 1.1264(10) | 0.3635(8) | −0.1464(7) | 0.047(2) |
| C(m1) | 0.8850(10) | 0.2245(8) | 0.2883(6) | 0.043(2) |
| C(m2) | 0.7048(9) | −0.0355(8) | 0.1468(6) | 0.042(2) |
| C(m3) | 0.9388(11) | 0.1258(8) | −0.1308(7) | 0.047(2) |
| C(m4) | 1.1010(9) | 0.3957(9) | 0.0097(6) | 0.044(2) |
| C(11) | 1.2064(10) | 0.5099(9) | 0.1442(7) | 0.052(3) |
| C(12) | 1.3483(8) | 0.4684(9) | 0.1153(6) | 0.068(3) |
| C(21) | 1.0755(8) | 0.3840(7) | 0.3290(6) | 0.048(2) |
| C(22) | 1.1633(13) | 0.2934(10) | 0.3622(8) | 0.066(3) |
| C(31) | 0.8251(12) | 0.0080(10) | 0.4032(7) | 0.055(3) |
| C(32) | 0.7588(13) | −0.0709(11) | 0.4782(9) | 0.072(4) |
| C(41) | 0.6211(12) | 0.1351(9) | 0.4056(7) | 0.057(3) |

**Table S2.** Continued

| atom | $x$ | $y$ | $z$ | $U$(eq) |
|---|---|---|---|---|
| C(42) | 0.5294(12) | 0.1997(10) | 0.3521(9) | 0.064(3) |
| C(51) | 0.6524(10) | −0.1808(8) | 0.0125(8) | 0.050(2) |
| C(52) | 0.5029(11) | −0.1581(9) | 0.0325(8) | 0.058(3) |
| C(61) | 0.8036(11) | −0.0758(10) | −0.1728(7) | 0.055(3) |
| C(62) | 0.6774(14) | −0.0556(11) | −0.2070(9) | 0.068(3) |
| C(71) | 1.1222(12) | 0.2630(11) | −0.2813(8) | 0.066(3) |
| C(72) | 1.0563(13) | 0.3420(10) | −0.3386(8) | 0.063(3) |
| C(81) | 1.2214(11) | 0.4514(10) | −0.1870(8) | 0.058(3) |
| C(82) | 1.3643(12) | 0.4244(11) | −0.1839(9) | 0.068(4) |
| Sb | 0.5000 | 0.5000 | 0.5000 | 0.0527(3) |
| Cl(3) | 0.6928(3) | 0.4197(2) | 0.4183(2) | 0.0638(8) |
| Cl(4) | 0.5744(3) | 0.6719(2) | 0.4373(2) | 0.0591(7) |
| Cl(5) | 0.3881(3) | 0.4909(3) | 0.3859(2) | 0.0681(8) |
| C(1) | 0.6982(12) | 0.2185(10) | 0.6281(11) | 0.076(4) |
| Cl(11) | 0.6832(4) | 0.2511(3) | 0.7448(2) | 0.0773(9) |
| Cl(12) | 0.8478(4) | 0.1387(4) | 0.6066(3) | 0.0886(12) |

[a] $U$(eq) is defined as one third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor. The estimated standard deviations of the least significant digits are given in parentheses.

**Table S3.** Bond Lengths for $[Fe(oxoOEC^{\bullet/2})(Cl)]_2SbCl_6$[a]

| bond | length (Å) | bond | length (Å) |
|---|---|---|---|
| Fe(1)–N(4) | 2.047(8) | Sb–Cl(4)#1) | 2.364(3) |
| Fe(1)–N(1) | 2.053(8) | Sb–Cl(3)#1) | 2.375(3) |
| Fe(1)–N(3) | 2.080(8) | Sb–Cl(3) | 2.375(3) |
| Fe(1)–N(2) | 2.124(8) | C(1)–Cl(12) | 1.790(12) |
| Fe(1)–Cl(1) | 2.213(3) | C(1)–Cl(11) | 1.911(16) |
| O(1)–C(b4) | 1.225(12) | C(m1)–H(m1) | 0.9500 |
| N(1)–C(a1) | 1.360(12) | C(m2)–H(m2) | 0.9500 |
| N(1)–C(a2) | 1.390(13) | C(m3)–H(m3) | 0.9500 |
| N(2)–C(a3) | 1.325(13) | C(m4)–H(m4) | 0.9500 |
| N(2)–C(a4) | 1.403(13) | C(11)–H(1a) | 0.9900 |
| N(3)–C(a6) | 1.376(13) | C(11)–H(1b) | 0.9900 |
| N(3)–C(a5) | 1.392(12) | C(12)–H(1c) | 0.9800 |
| N(4)–C(a8) | 1.375(13) | C(12)–H(1d) | 0.9800 |
| N(4)–C(a7) | 1.376(12) | C(12)–H(1e) | 0.9800 |
| C(a1)–C(m4) | 1.399(13) | C(21)–H(2a) | 0.9900 |
| C(a1)–C(b1) | 1.439(14) | C(21)–H(2b) | 0.9900 |
| C(a2)–C(m1) | 1.401(14) | C(22)–H(2c) | 0.9800 |
| C(a2)–C(b2) | 1.432(14) | C(22)–H(2d) | 0.9800 |
| C(a3)–C(m1) | 1.393(14) | C(22)–H(2e) | 0.9800 |
| C(a3)–C(b3) | 1.505(13) | C(31)–H(3a) | 0.9900 |
| C(a4)–C(m2) | 1.326(15) | C(31)–H(3b) | 0.9900 |
| C(a4)–C(b4) | 1.490(15) | C(32)–H(3c) | 0.9800 |
| C(a5)–C(m2) | 1.383(14) | C(32)–H(3d) | 0.9800 |
| C(a5)–C(b5) | 1.473(15) | C(32)–H(3e) | 0.9800 |
| C(a6)–C(m3) | 1.375(14) | C(41)–H(4a) | 0.9900 |
| C(a6)–C(b6) | 1.463(15) | C(41)–H(4b) | 0.9900 |
| C(a7)–C(m3) | 1.386(14) | C(42)–H(4c) | 0.9800 |
| C(a7)–C(b7) | 1.436(13) | C(42)–H(4d) | 0.9800 |
| C(a8)–C(m4) | 1.388(15) | C(42)–H(4e) | 0.9800 |
| C(a8)–C(b8) | 1.446(14) | C(51)–H(5a) | 0.9900 |
| C(b1)–C(b2) | 1.376(14) | C(51)–H(5b) | 0.9900 |
| C(b1)–C(11) | 1.490(14) | C(52)–H(5c) | 0.9800 |
| C(b2)–C(21) | 1.478(13) | C(52)–H(5d) | 0.9800 |
| C(b3)–C(41) | 1.522(16) | C(52)–H(5e) | 0.9800 |

**Table S3.** Continued

| bond | length (Å) | bond | length (Å) |
|---|---|---|---|
| C(b3)–C(b4) | 1.534(14) | C(61)–H(6a) | 0.9900 |
| C(b3)–C(31) | 1.551(15) | C(61)–H(6b) | 0.9900 |
| C(b5)–C(b6) | 1.323(15) | C(62)–H(6c) | 0.9800 |
| C(b5)–C(51) | 1.485(15) | C(62)–H(6d) | 0.9800 |
| C(b6)–C(61) | 1.515(15) | C(62)–H(6e) | 0.9800 |
| C(b7)–C(b8) | 1.385(15) | C(71)–H(7a) | 0.9900 |
| C(b7)–C(71) | 1.513(16) | C(71)–H(7b) | 0.9900 |
| C(b8)–C(81) | 1.507(14) | C(72)–H(7c) | 0.9800 |
| C(11)–C(12) | 1.523(14) | C(72)–H(7d) | 0.9800 |
| C(21)–C(22) | 1.538(14) | C(72)–H(7e) | 0.9800 |
| C(31)–C(32) | 1.522(15) | C(81)–H(8a) | 0.9900 |
| C(41)–C(42) | 1.547(17) | C(81)–H(8b) | 0.9900 |
| C(51)–C(52) | 1.528(15) | C(82)–H(8c) | 0.9800 |
| C(61)–C(62) | 1.517(17) | C(82)–H(8d) | 0.9800 |
| C(71)–C(72) | 1.507(18) | C(82)–H(8e) | 0.9800 |
| C(81)–C(82) | 1.509(17) | C(1)–H(1) | 0.9900 |
| Sb–Cl(5) | 2.363(3) | C(1)–H(2) | 0.9900 |
| Sb–Cl(5)#1) | 2.363(3) | | |
| Sb–Cl(4) | 2.364(3) | | |

[a]The estimated standard deviations of the least significant digits are given in parentheses.

**Table S4.** Bond Angles for $[Fe(oxoOEC^{\bullet/2})(Cl)]_2SbCl_6$[a]

| angle | degree | angle | degree |
|---|---|---|---|
| N(4)–Fe(1)–N(1) | 87.1(3) | Cl(4)#1–Sb–Cl(3)#1 | 90.26(10) |
| N(4)–Fe(1)–N(3) | 87.3(3) | Cl(5)–Sb–Cl(3) | 90.38(12) |
| N(1)–Fe(1)–N(3) | 152.2(3) | Cl(5)#1–Sb–Cl(3) | 89.62(13) |
| N(4)–Fe(1)–N(2) | 155.4(3) | Cl(4)–Sb–Cl(3) | 90.26(10) |
| N(1)–Fe(1)–N(2) | 87.0(3) | Cl(4)#1–Sb–Cl(3) | 89.74(10) |
| N(3)–Fe(1)–N(2) | 86.8(3) | Cl(3)#1–Sb–Cl(3) | 180.0 |
| N(4)–Fe(1)–Cl(1) | 104.9(2) | Cl(12)–C(1)–Cl(11) | 104.1(8) |
| N(1)–Fe(1)–Cl(1) | 101.5(2) | C(b1)–C(11)–H(1a) | 109.1 |
| N(3)–Fe(1)–Cl(1) | 106.2(2) | C(12)–C(11)–H(1a) | 109.1 |
| N(2)–Fe(1)–Cl(1) | 99.7(2) | C(b1)–C(11)–H(1b) | 109.1 |
| C(a1)–N(1)–C(a2) | 103.8(8) | C(12)–C(11)–H(1b) | 109.1 |
| C(a1)–N(1)–Fe(1) | 128.0(6) | H(1a)–C(11)–H(1b) | 107.8 |
| C(a2)–N(1)–Fe(1) | 127.5(6) | C(11)–C(12)–H(1c) | 109.5 |
| C(a3)–N(2)–C(a4) | 110.1(8) | C(11)–C(12)–H(1d) | 109.5 |
| C(a3)–N(2)–Fe(1) | 125.9(6) | H(1c)–C(12)–H(1d) | 109.5 |
| C(a4)–N(2)–Fe(1) | 122.6(6) | C(11)–C(12)–H(1e) | 109.5 |
| C(a6)–N(3)–C(a5) | 105.7(8) | H(1c)–C(12)–H(1e) | 109.5 |
| C(a6)–N(3)–Fe(1) | 125.9(6) | H(1d)–C(12)–H(1e) | 109.5 |
| C(a5)–N(3)–Fe(1) | 126.9(7) | C(b2)–C(21)–H(2a) | 109.0 |
| C(a8)–N(4)–C(a7) | 105.0(8) | C(22)–C(21)–H(2a) | 109.0 |
| C(a8)–N(4)–Fe(1) | 125.9(7) | C(b2)–C(21)–H(2b) | 109.0 |
| C(a7)–N(4)–Fe(1) | 127.8(6) | C(22)–C(21)–H(2b) | 109.0 |
| N(1)–C(a1)–C(m4) | 122.7(9) | H(2a)–C(21)–H(2b) | 107.8 |
| N(1)–C(a1)–C(b1) | 112.7(9) | C(21)–C(22)–H(2c) | 109.5 |
| C(m4)–C(a1)–C(b1) | 124.5(9) | C(21)–C(22)–H(2d) | 109.5 |
| N(1)–C(a2)–C(m1) | 123.9(9) | H(2c)–C(22)–H(2d) | 109.5 |
| N(1)–C(a2)–C(b2) | 111.7(9) | C(21)–C(22)–H(2e) | 109.5 |
| C(m1)–C(a2)–C(b2) | 124.4(10) | H(2c)–C(22)–H(2e) | 109.5 |
| N(2)–C(a3)–C(m1) | 125.4(9) | H(2d)–C(22)–H(2e) | 109.5 |
| N(2)–C(a3)–C(b3) | 114.6(9) | C(32)–C(31)–H(3a) | 108.2 |
| C(m1)–C(a3)–C(b3) | 119.9(9) | C(b3)–C(31)–H(3a) | 108.2 |
| C(m2)–C(a4)–N(2) | 127.9(9) | C(32)–C(31)–H(3b) | 108.2 |
| C(m2)–C(a4)–C(b4) | 124.1(10) | C(b3)–C(31)–H(3b) | 108.2 |
| N(2)–C(a4)–C(b4) | 107.9(9) | H(3a)–C(31)–H(3b) | 107.4 |

**Table S4.** Continued

| angle | degree | angle | degree |
|---|---|---|---|
| C(m2)–C(a5)–N(3) | 123.8(9) | C(31)–C(32)–H(3c) | 109.5 |
| C(m2)–C(a5)–C(b5) | 126.9(9) | C(31)–C(32)–H(3d) | 109.5 |
| N(3)–C(a5)–C(b5) | 109.3(9) | H(3c)–C(32)–H(3d) | 109.5 |
| C(m3)–C(a6)–N(3) | 124.6(9) | C(31)–C(32)–H(3e) | 109.5 |
| C(m3)–C(a6)–C(b6) | 125.4(10) | H(3c)–C(32)–H(3e) | 109.5 |
| N(3)–C(a6)–C(b6) | 109.9(8) | H(3d)–C(32)–H(3e) | 109.5 |
| N(4)–C(a7)–C(m3) | 123.3(9) | C(b3)–C(41)–H(4a) | 108.9 |
| N(4)–C(a7)–C(b7) | 111.6(9) | C(42)–C(41)–H(4a) | 108.9 |
| C(m3)–C(a7)–C(b7) | 125.0(9) | C(b3)–C(41)–H(4b) | 108.9 |
| N(4)–C(a8)–C(m4) | 124.2(9) | C(42)–C(41)–H(4b) | 108.9 |
| N(4)–C(a8)–C(b8) | 111.2(9) | H(4a)–C(41)–H(4b) | 107.7 |
| C(m4)–C(a8)–C(b8) | 124.4(10) | C(41)–C(42)–H(4c) | 109.5 |
| C(b2)–C(b1)–C(a1) | 105.6(9) | C(41)–C(42)–H(4d) | 109.5 |
| C(b2)–C(b1)–C(11) | 128.7(9) | H(4c)–C(42)–H(4d) | 109.5 |
| C(a1)–C(b1)–C(11) | 125.6(9) | C(41)–C(42)–H(4e) | 109.5 |
| C(b1)–C(b2)–C(a2) | 106.2(9) | H(4c)–C(42)–H(4e) | 109.5 |
| C(b1)–C(b2)–C(21) | 128.2(9) | H(4d)–C(42)–H(4e) | 109.5 |
| C(a2)–C(b2)–C(21) | 125.4(9) | C(b5)–C(51)–H(5a) | 108.6 |
| C(a3)–C(b3)–C(41) | 113.4(9) | C(52)–C(51)–H(5a) | 108.6 |
| C(a3)–C(b3)–C(b4) | 99.8(8) | C(b5)–C(51)–H(5b) | 108.6 |
| C(41)–C(b3)–C(b4) | 108.5(9) | C(52)–C(51)–H(5b) | 108.6 |
| C(a3)–C(b3)–C(31) | 110.8(9) | H(5a)–C(51)–H(5b) | 107.6 |
| C(41)–C(b3)–C(31) | 114.0(9) | C(51)–C(52)–H(5c) | 109.5 |
| C(b4)–C(b3)–C(31) | 109.4(9) | C(51)–C(52)–H(5d) | 109.5 |
| O(1)–C(b4)–C(a4) | 123.9(10) | H(5c)–C(52)–H(5d) | 109.5 |
| O(1)–C(b4)–C(b3) | 129.0(10) | C(51)–C(52)–H(5e) | 109.5 |
| C(a4)–C(b4)–C(b3) | 107.2(9) | H(5c)–C(52)–H(5e) | 109.5 |
| C(b6)–C(b5)–C(a5) | 107.2(10) | H(5d)–C(52)–H(5e) | 109.5 |
| C(b6)–C(b5)–C(51) | 130.6(10) | C(b6)–C(61)–H(6a) | 108.4 |
| C(a5)–C(b5)–C(51) | 122.2(10) | C(62)–C(61)–H(6a) | 108.4 |
| C(b5)–C(b6)–C(a6) | 107.8(10) | C(b6)–C(61)–H(6b) | 108.4 |
| C(b5)–C(b6)–C(61) | 126.3(10) | C(62)–C(61)–H(6b) | 108.4 |
| C(a6)–C(b6)–C(61) | 125.8(9) | H(6a)–C(61)–H(6b) | 107.5 |
| C(b8)–C(b7)–C(a7) | 106.1(9) | C(61)–C(62)–H(6c) | 109.5 |

**Table S4.** Continued

| angle | degree | angle | degree |
|---|---|---|---|
| C(b8)–C(b7)–C(71) | 128.3(9) | C(61)–C(62)–H(6d) | 109.5 |
| C(a7)–C(b7)–C(71) | 125.7(9) | H(6c)–C(62)–H(6d) | 109.5 |
| C(b7)–C(b8)–C(a8) | 106.0(9) | C(61)–C(62)–H(6e) | 109.5 |
| C(b7)–C(b8)–C(81) | 127.9(10) | H(6c)–C(62)–H(6e) | 109.5 |
| C(a8)–C(b8)–C(81) | 125.7(10) | H(6d)–C(62)–H(6e) | 109.5 |
| C(a3)–C(m1)–C(a2) | 126.5(10) | C(72)–C(71)–H(7a) | 108.9 |
| C(a4)–C(m2)–C(a5) | 127.2(10) | C(b7)–C(71)–H(7a) | 108.9 |
| C(a6)–C(m3)–C(a7) | 127.6(10) | C(72)–C(71)–H(7b) | 108.9 |
| C(a8)–C(m4)–C(a1) | 126.9(9) | C(b7)–C(71)–H(7b) | 108.9 |
| C(b1)–C(11)–C(12) | 112.6(10) | H(7a)–C(71)–H(7b) | 107.7 |
| C(b2)–C(21)–C(22) | 113.0(8) | C(71)–C(72)–H(7c) | 109.5 |
| C(32)–C(31)–C(b3) | 116.3(10) | C(71)–C(72)–H(7d) | 109.5 |
| C(a3)–C(m1)–H(m1) | 116.7 | H(7c)–C(72)–H(7d) | 109.5 |
| C(a2)–C(m1)–H(m1) | 116.7 | C(71)–C(72)–H(7e) | 109.5 |
| C(a4)–C(m2)–H(m2) | 116.4 | H(7c)–C(72)–H(7e) | 109.5 |
| C(a5)–C(m2)–H(m2) | 116.4 | H(7d)–C(72)–H(7e) | 109.5 |
| C(a6)–C(m3)–H(m3) | 116.2 | C(b8)–C(81)–H(8a) | 108.6 |
| C(a7)–C(m3)–H(m3) | 116.2 | C(82)–C(81)–H(8a) | 108.6 |
| C(a8)–C(m4)–H(m4) | 116.5 | C(b8)–C(81)–H(8b) | 108.6 |
| C(a1)–C(m4)–H(m4) | 116.5 | C(82)–C(81)–H(8b) | 108.6 |
| C(b3)–C(41)–C(42) | 113.3(10) | H(8a)–C(81)–H(8b) | 107.6 |
| C(b5)–C(51)–C(52) | 114.6(9) | C(81)–C(82)–H(8c) | 109.5 |
| C(b6)–C(61)–C(62) | 115.4(10) | C(81)–C(82)–H(8d) | 109.5 |
| C(72)–C(71)–C(b7) | 113.3(11) | H(8c)–C(82)–H(8d) | 109.5 |
| C(b8)–C(81)–C(82) | 114.5(10) | C(81)–C(82)–H(8e) | 109.5 |
| Cl(5)–Sb–Cl(5)#1 | 180.0 | H(8c)–C(82)–H(8e) | 109.5 |
| Cl(5)–Sb–Cl(4) | 89.36(11) | H(8d)–C(82)–H(8e) | 109.5 |
| Cl(5)#1–Sb)–Cl(4) | 90.64(11) | Cl(12)–C(1)–H(1) | 110.9 |
| Cl(5)–Sb–Cl(4)#1 | 90.64(11) | Cl(11)–C(1)–H(1) | 110.9 |
| Cl(5)#1–Sb–Cl(4)#1 | 89.36(11) | Cl(12)–C(1)–H(2) | 110.9 |
| Cl(4)–Sb–Cl(4)#1 | 180.0 | Cl(11)–C(1)–H(2) | 110.9 |
| Cl(5)–Sb–Cl(3)#1 | 89.62(13) | H(1)–C(1)–H(2) | 109.0 |
| Cl(5)#1–Sb–Cl(3)#1 | 90.38(12) | | |
| Cl(4)–Sb–Cl(3)#1 | 89.74(10) | | |

[a]The estimated standard deviations of the least significant digits are given in parentheses.

**Table S5.** Anisotropic Displacement Parameters (Å$^2$) for [Fe(oxoOEC$^{\bullet/2}$)(Cl)]$_2$SbCl$_6$[a]

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| Fe(1) | 0.0370(7) | 0.0464(8) | 0.0377(8) | 0.0022(6) | −0.0114(5) | −0.0073(6) |
| Cl(1) | 0.0430(13) | 0.0515(14) | 0.0529(15) | −0.0043(11) | −0.0159(10) | −0.0001(11) |
| O(1) | 0.065(5) | 0.060(5) | 0.049(4) | 0.000(4) | −0.011(4) | −0.021(4) |
| N(1) | 0.046(5) | 0.038(4) | 0.030(4) | 0.001(3) | −0.012(3) | −0.004(3) |
| N(2) | 0.040(4) | 0.044(4) | 0.033(4) | −0.003(3) | −0.015(3) | −0.003(3) |
| N(3) | 0.035(4) | 0.044(4) | 0.043(5) | 0.004(3) | −0.012(3) | −0.005(3) |
| N(4) | 0.033(4) | 0.045(4) | 0.040(4) | 0.004(3) | −0.013(3) | −0.005(3) |
| C(a1) | 0.037(5) | 0.054(6) | 0.042(5) | −0.004(4) | −0.016(4) | −0.007(4) |
| C(a2) | 0.053(6) | 0.038(5) | 0.058(7) | −0.010(5) | −0.019(5) | 0.002(4) |
| C(a3) | 0.034(5) | 0.048(5) | 0.042(5) | 0.007(4) | −0.012(4) | −0.003(4) |
| C(a4) | 0.042(6) | 0.051(6) | 0.050(6) | 0.010(5) | −0.009(4) | 0.006(5) |
| C(a5) | 0.039(5) | 0.042(5) | 0.052(6) | −0.002(4) | −0.009(4) | 0.004(4) |
| C(a6) | 0.037(5) | 0.047(6) | 0.053(6) | −0.003(5) | −0.012(4) | −0.003(4) |
| C(a7) | 0.042(5) | 0.057(6) | 0.035(5) | 0.000(4) | −0.013(4) | −0.004(4) |
| C(a8) | 0.036(5) | 0.066(7) | 0.044(6) | 0.010(5) | −0.013(4) | −0.006(5) |
| C(b1) | 0.046(6) | 0.044(5) | 0.044(6) | 0.003(4) | −0.021(4) | −0.001(4) |
| C(b2) | 0.039(5) | 0.039(5) | 0.041(5) | −0.002(4) | −0.016(4) | 0.006(4) |
| C(b3) | 0.062(6) | 0.048(6) | 0.027(5) | 0.002(4) | −0.015(4) | −0.005(5) |
| C(b4) | 0.047(6) | 0.048(6) | 0.050(6) | −0.003(5) | −0.007(4) | −0.003(5) |
| C(b5) | 0.056(6) | 0.052(6) | 0.048(6) | −0.010(5) | −0.019(5) | 0.002(5) |
| C(b6) | 0.044(6) | 0.058(6) | 0.042(6) | 0.003(5) | −0.011(4) | −0.008(5) |
| C(b7) | 0.044(5) | 0.055(6) | 0.039(5) | −0.007(4) | −0.009(4) | −0.009(4) |
| C(b8) | 0.039(5) | 0.052(6) | 0.046(6) | 0.010(5) | −0.008(4) | −0.011(4) |
| C(m1) | 0.054(6) | 0.042(5) | 0.036(5) | −0.002(4) | −0.016(4) | 0.000(4) |
| C(m2) | 0.035(5) | 0.049(6) | 0.047(6) | −0.009(4) | −0.018(4) | −0.005(4) |
| C(m3) | 0.058(6) | 0.049(6) | 0.038(5) | −0.012(4) | −0.019(4) | 0.001(5) |
| C(m4) | 0.037(5) | 0.058(6) | 0.036(5) | 0.007(4) | −0.011(4) | −0.013(4) |
| C(11) | 0.051(6) | 0.056(6) | 0.052(6) | 0.004(5) | −0.023(5) | −0.018(5) |
| C(12) | 0.052(7) | 0.081(9) | 0.077(9) | −0.007(7) | −0.025(6) | −0.013(6) |
| C(21) | 0.056(6) | 0.041(5) | 0.051(6) | −0.008(4) | −0.023(5) | 0.001(5) |
| C(22) | 0.077(9) | 0.069(8) | 0.062(8) | −0.007(6) | −0.037(6) | 0.000(6) |
| C(31) | 0.064(7) | 0.062(7) | 0.043(6) | 0.001(5) | −0.021(5) | −0.008(6) |
| C(32) | 0.073(9) | 0.077(9) | 0.067(8) | 0.020(7) | −0.029(6) | −0.020(7) |
| C(41) | 0.068(8) | 0.055(7) | 0.047(6) | −0.002(5) | −0.009(5) | −0.014(6) |

**Table S5.** Continued

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{23}$ | $U_{13}$ | $U_{12}$ |
|---|---|---|---|---|---|---|
| C(42) | 0.055(7) | 0.062(7) | 0.074(8) | −0.013(6) | −0.013(6) | 0.005(6) |
| C(51) | 0.045(6) | 0.046(6) | 0.061(7) | −0.011(5) | −0.013(5) | −0.004(4) |
| C(52) | 0.058(7) | 0.046(6) | 0.073(8) | −0.006(5) | −0.018(6) | −0.015(5) |
| C(61) | 0.060(7) | 0.060(7) | 0.045(6) | −0.009(5) | −0.011(5) | −0.006(5) |
| C(62) | 0.083(9) | 0.070(8) | 0.060(8) | −0.014(6) | −0.031(6) | 0.003(7) |
| C(71) | 0.055(7) | 0.074(8) | 0.064(8) | −0.003(6) | 0.000(5) | −0.019(6) |
| C(72) | 0.072(8) | 0.066(8) | 0.048(7) | 0.002(5) | −0.012(5) | −0.005(6) |
| C(81) | 0.052(7) | 0.069(7) | 0.050(7) | 0.002(5) | −0.006(5) | −0.017(6) |
| C(82) | 0.054(7) | 0.082(9) | 0.065(8) | 0.021(7) | −0.016(6) | −0.019(6) |
| Sb | 0.0543(7) | 0.0491(6) | 0.0497(6) | −0.0035(4) | 0.0012(4) | −0.0096(5) |
| Cl(3) | 0.0561(17) | 0.0584(17) | 0.0672(19) | −0.0072(13) | 0.0099(13) | −0.0067(13) |
| Cl(4) | 0.0585(17) | 0.0487(15) | 0.0647(18) | −0.0013(12) | −0.0012(13) | −0.0121(12) |
| Cl(5) | 0.078(2) | 0.0656(19) | 0.0633(19) | −0.0063(14) | −0.0175(15) | −0.0128(15) |
| C(1) | 0.039(6) | 0.056(7) | 0.135(13) | −0.025(8) | −0.012(7) | 0.003(5) |
| Cl(11) | 0.077(2) | 0.079(2) | 0.074(2) | −0.0157(17) | −0.0091(16) | 0.0043(17) |
| Cl(12) | 0.072(2) | 0.114(3) | 0.089(3) | −0.043(2) | −0.0219(18) | 0.004(2) |

[a]The estimated standard deviations of the least significant digits are given in parentheses. The anisotropic displacement factor exponent takes the form: $-2\,\pi\,[h^2\,U_{11}$ +...+ 2 h k a* b* $U_{12}]$.

**Table S6.** Hydrogen Atom Coordinates and Equivalent Isotropic Displacement Parameters (Å$^2$) for [Fe(oxoOEC$^{\bullet/2}$)(Cl)]$_2$SbCl$_6$[a]

| atom | $x$ | $y$ | $z$ | $U$(eq) |
|---|---|---|---|---|
| H(m1) | 0.8780 | 0.2382 | 0.3469 | 0.052 |
| H(m2) | 0.6484 | −0.0949 | 0.1659 | 0.050 |
| H(m3) | 0.9513 | 0.1087 | −0.1890 | 0.056 |
| H(m4) | 1.1538 | 0.4572 | −0.0093 | 0.053 |
| H(1a) | 1.1997 | 0.5472 | 0.1968 | 0.062 |
| H(1b) | 1.1843 | 0.5627 | 0.0979 | 0.062 |
| H(1c) | 1.3708 | 0.4160 | 0.1609 | 0.102 |
| H(1d) | 1.4089 | 0.5283 | 0.1048 | 0.102 |
| H(1e) | 1.3565 | 0.4340 | 0.0617 | 0.102 |
| H(2a) | 0.9896 | 0.3867 | 0.3711 | 0.057 |
| H(2b) | 1.1189 | 0.4533 | 0.3260 | 0.057 |
| H(2c) | 1.1195 | 0.2248 | 0.3674 | 0.099 |
| H(2d) | 1.1775 | 0.3071 | 0.4192 | 0.099 |
| H(2e) | 1.2490 | 0.2906 | 0.3212 | 0.099 |
| H(3a) | 0.8910 | −0.0322 | 0.3627 | 0.066 |
| H(3b) | 0.8744 | 0.0594 | 0.4267 | 0.066 |
| H(3c) | 0.7055 | −0.0313 | 0.5242 | 0.107 |
| H(3d) | 0.8268 | −0.1148 | 0.5013 | 0.107 |
| H(3e) | 0.7014 | −0.1173 | 0.4575 | 0.107 |
| H(4a) | 0.6607 | 0.1852 | 0.4365 | 0.069 |
| H(4b) | 0.5672 | 0.0842 | 0.4500 | 0.069 |
| H(4c) | 0.5813 | 0.2528 | 0.3099 | 0.096 |
| H(4d) | 0.4591 | 0.2364 | 0.3912 | 0.096 |
| H(4e) | 0.4898 | 0.1508 | 0.3213 | 0.096 |
| H(5a) | 0.6764 | −0.2237 | 0.0634 | 0.060 |
| H(5b) | 0.6746 | −0.2248 | −0.0370 | 0.060 |
| H(5c) | 0.4801 | −0.1114 | 0.0795 | 0.087 |
| H(5d) | 0.4565 | −0.2258 | 0.0504 | 0.087 |
| H(5e) | 0.4763 | −0.1225 | −0.0196 | 0.087 |
| H(6a) | 0.8246 | −0.1538 | −0.1695 | 0.066 |
| H(6b) | 0.8770 | −0.0397 | −0.2153 | 0.066 |
| H(6c) | 0.6062 | −0.0984 | −0.1697 | 0.102 |
| H(6d) | 0.6925 | −0.0761 | −0.2664 | 0.102 |
| H(6e) | 0.6520 | 0.0206 | −0.2072 | 0.102 |

**Table S6.** Continued

| atom | $x$ | $y$ | $z$ | $U$(eq) |
|---|---|---|---|---|
| H(7a) | 1.2196 | 0.2674 | −0.3016 | 0.079 |
| H(7b) | 1.0970 | 0.1896 | −0.2873 | 0.079 |
| H(7c) | 0.9599 | 0.3397 | −0.3180 | 0.094 |
| H(7d) | 1.0800 | 0.3232 | −0.3985 | 0.094 |
| H(7e) | 1.0861 | 0.4144 | −0.3365 | 0.094 |
| H(8a) | 1.1936 | 0.5164 | −0.1568 | 0.069 |
| H(8b) | 1.2149 | 0.4688 | −0.2484 | 0.069 |
| H(8c) | 1.3694 | 0.3904 | −0.1259 | 0.103 |
| H(8d) | 1.4152 | 0.4903 | −0.1963 | 0.103 |
| H(8e) | 1.4011 | 0.3751 | −0.2273 | 0.103 |
| H(1) | 0.6211 | 0.1779 | 0.6228 | 0.092 |
| H(2) | 0.7045 | 0.2848 | 0.5874 | 0.092 |

[a] $U$(eq) is defined as one third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor the estimated standard deviations of the least significant digits are given in parentheses.