# Crystal structure of tris(acetylacetonato)iron(III), $C_{15}H_{21}O_6Fe$, *at 20 K*

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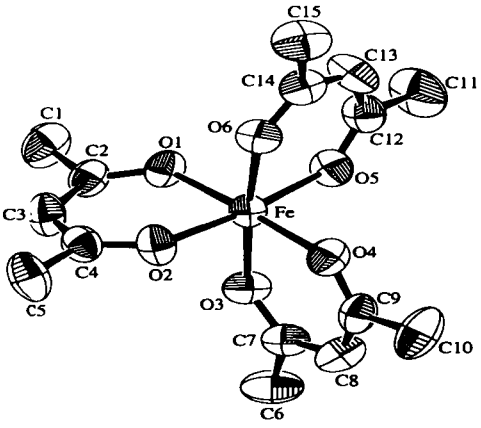


**Abstract**

$C_{15}H_{21}O_6Fe$, , *Pbca* (No. 61), $a$ = 16.561(3) Å, $b$ = 15.434(4) Å, $c$ = 13.578(3) Å, $V$ = 3470.6 Å$^3$, $Z$ = 8, $R_{gt}(F)$ = 0.053, $wR_{obs}(F)$ = 0.059, $T$ = 20 K.

**Source of material**

3 mmol imidazole was dissolved in 10 mL water, and 1 mmol $(NH_4)_2Fe(SO_4)_2 \cdot H_2O$ was also dissolved in 10 mL water. The two solutions were mixed to give green precipitate, then 10 mL acetylacetone was added into above mixture with stirring at room temperature. When the precipitate had entirely dissolved, the solution changed its color from green to deep red, and the reaction mixture was filtered. The filtrate stood for six days until the red single crystals were obtained.

**Experinental details**

The structure was solved by Patterson method followed by Fourier syntheses. H atoms were located in a difference Fourier map, and their coordinates and thermal parameters were fixed during structure refinement.

**Discussion**

Ferric and ferrous ions exist broadly in various organisms, and play important roles in bewildering array of proteins [1]. They relate closely to many functions such as $O_2$ storage in yoglobin, $O_2$ transport in hemoglobin, oxidation of inactivated carbon-hydrogen bonds in cytochrome $P_{450}$, oxygen reduction in cytochromeC oxidase and so on [2, 3]. Consequently, much interest has been focused on their complexes [4]. What is more, the β-diones find a variety of laboratory uses and many industrial applications and have contributed a lot to the present understanding of steric pressure and conformers, prototropy and tautomerism, chelate formation, and intramolecular bonding [5]. So we use ferric ion andacetylacetonato anion as center ion and ligand respectively to synthesize the title complex and report its crystal structure herein.

The molecular structure of the title complex with the atom numbering scheme is illustrated in the figure (left, 50% probability displacement elipsoids). In a molecule of the title complex, six oxygen atoms from three bidentate acac anions coordinate to a ferric ion to complete the coordination geometry of a six-coordinated octahedron around the ferric ion. It is worthy to take a look at the distances and angles around ferric ion in this complex. The bond angles of O(1)–Fe–O(4), O(3)–Fe–O(6), O(2)–Fe–O(5), are 176.4(1)°, 175.0(1)°, 173.5(1)°, respectively, which are all smaller than 180°. On the other hand, the bond distances of all the Fe—O, namely Fe—O(1) [2.004(3) Å], Fe—O(2) [1.996(3) Å], Fe—O(3) [1.995(3) Å], Fe—O(4) [1.977(3) Å], Fe—O(5) [1.985(3) Å], Fe—O(6) [1.992(3) Å ], are not equal to each other. Therefore, the coordination geometry formed by six oxygen atoms is a distorted octahedron. A β-diketonate anion coordinate usually to a metal cation in a chelate fashion [6]. The bond distances of O(1)—C(2), O(2)—C(4), C(2)—C(3) and C(3)—C(4) are 1.274(6) Å, 1.258(6) Å, 1.381(7) Å and 1.390(7) Å, respectively, which are in range between the single and double bond distances. It shows that there exists conjugated structure in each coordinated β-diketonate anion acac, which leads to the delocalization of electron density of the coordinated β-diketonate structure. Whereas, the annular and exocyclic C—C distances are on average 1.385 Å and 1.504 Å, respectively, which are the main deviations from those observed at room temperature (1.377 Å and 1.530 Å, respectively) [7]. The mean values of annular and exocyclic O–C–C angle for the title compound at 20 K are 124.2° and 115.6 , respectively, and those observed previously [7] at room temperature are 125.0° and 114.9 , respectively. The other bond lengths and angles are almost the same in spite of the temperature difference. All in all, three acac coordinates concurrently to a ferric ion to result a distorted octahedron of the title complex with three six-membered rings. The plane through O(1), O(2) and Fe joints to the plane through O(3), O(4) and Fe at an angle of 86.49°, to the plane through O(5), O(6) and Fe at an angle of 78.91°. In the molecular packing, separate molecules insert with each other by the ramose six-membered ring.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | red prismatic, size 0.20 × 0.20 × 0.30 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.7107 Å) |
| μ: | 89.13 cm$^{-1}$ |
| Diffractometer, scan mode: | Rigaku AFC7R, ω/2θ |
| $2\theta_{max}$: | 54.98° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 3944, 3744 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2.5\ \sigma(I_{obs})$, 1920 |
| $N(param)_{refined}$: | 200 |
| Programs: | SHELXS-86 [8], teXsan [9] |

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**Table 2.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | x | y | z | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 8c | 0.4157 | 0.7303 | 0.4703 | 0.140 |
| H(2) | 8c | 0.4525 | 0.7968 | 0.3974 | 0.165 |
| H(3) | 8c | 0.4998 | 0.7132 | 0.4238 | 0.152 |
| H(4) | 8c | 0.5091 | 0.6333 | 0.2803 | 0.095 |
| H(5) | 8c | 0.5226 | 0.5898 | 0.0974 | 0.190 |
| H(6) | 8c | 0.5025 | 0.5021 | 0.1475 | 0.130 |
| H(7) | 8c | 0.4519 | 0.5336 | 0.0582 | 0.192 |
| H(8) | 8c | 0.2741 | 0.4225 | 0.4359 | 0.136 |
| H(9) | 8c | 0.2155 | 0.3564 | 0.3878 | 0.101 |
| H(10) | 8c | 0.1821 | 0.4283 | 0.4565 | 0.157 |
| H(11) | 8c | 0.1343 | 0.3974 | 0.2450 | 0.087 |
| H(12) | 8c | 0.0903 | 0.4097 | 0.0803 | 0.182 |
| H(13) | 8c | 0.0396 | 0.4936 | 0.0913 | 0.159 |
| H(14) | 8c | 0.1115 | 0.4909 | 0.0182 | 0.130 |
| H(15) | 8c | 0.0528 | 0.7582 | 0.3963 | 0.151 |
| H(16) | 8c | 0.0393 | 0.8438 | 0.3388 | 0.184 |
| H(17) | 8c | 0.1083 | 0.8374 | 0.4157 | 0.237 |
| H(18) | 8c | 0.1214 | 0.8773 | 0.1987 | 0.063 |
| H(19) | 8c | 0.1707 | 0.8468 | -0.0042 | 0.103 |
| H(20) | 8c | 0.2030 | 0.9207 | 0.0626 | 0.149 |
| H(21) | 8c | 0.2628 | 0.8564 | 0.0137 | 0.123 |

**Table 3.** Atomic coordinates and displacement parameters (in $Å^2$).

| Atom | Site | x | y | z | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Fe | 8c | 0.25418(4) | 0.64276(4) | 0.23083(5) | 0.0521(4) | 0.0604(4) | 0.0429(4) | 0.0006(4) | -0.0008(3) | -0.0056(3) |
| O(1) | 8c | 0.3324(2) | 0.7029(2) | 0.3203(2) | 0.064(2) | 0.075(2) | 0.052(2) | -0.009(2) | -0.004(2) | -0.019(2) |
| O(2) | 8c | 0.3498(2) | 0.5940(2) | 0.1605(2) | 0.058(2) | 0.092(3) | 0.056(2) | 0.011(2) | -0.003(2) | -0.023(2) |
| O(3) | 8c | 0.2502(2) | 0.5443(2) | 0.3258(2) | 0.088(3) | 0.059(2) | 0.048(2) | -0.003(2) | -0.015(2) | -0.002(2) |
| O(4) | 8c | 0.1786(2) | 0.5767(2) | 0.1469(2) | 0.067(2) | 0.079(2) | 0.052(2) | -0.009(2) | -0.014(2) | 0.001(2) |
| O(5) | 8c | 0.1669(2) | 0.7028(2) | 0.3041(2) | 0.065(2) | 0.080(3) | 0.051(2) | 0.004(2) | 0.016(2) | 0.004(2) |
| O(6) | 8c | 0.2485(2) | 0.7382(2) | 0.1323(2) | 0.078(2) | 0.066(2) | 0.048(2) | 0.004(2) | 0.012(2) | 0.001(2) |
| C(1) | 8c | 0.4476(4) | 0.7386(5) | 0.4122(4) | 0.100(5) | 0.116(5) | 0.085(4) | -0.025(4) | -0.033(4) | -0.012(4) |
| C(2) | 8c | 0.4083(3) | 0.6911(4) | 0.3290(4) | 0.062(4) | 0.078(4) | 0.055(3) | -0.012(3) | -0.012(3) | 0.002(3) |
| C(3) | 8c | 0.4534(3) | 0.6397(4) | 0.2666(4) | 0.053(3) | 0.105(5) | 0.088(4) | 0.004(3) | -0.016(3) | -0.006(4) |
| C(4) | 8c | 0.4231(3) | 0.5970(3) | 0.1844(4) | 0.060(3) | 0.075(4) | 0.066(4) | 0.008(3) | 0.011(3) | 0.002(3) |
| C(5) | 8c | 0.4806(4) | 0.5499(5) | 0.1166(5) | 0.078(4) | 0.136(6) | 0.104(5) | 0.034(4) | 0.023(4) | -0.016(5) |
| C(6) | 8c | 0.2210(4) | 0.4147(3) | 0.4085(4) | 0.145(6) | 0.061(3) | 0.063(3) | 0.000(4) | 0.009(4) | -0.001(3) |
| C(7) | 8c | 0.2119(3) | 0.4740(4) | 0.3217(4) | 0.085(4) | 0.056(3) | 0.048(3) | 0.003(3) | 0.014(3) | -0.005(3) |
| C(8) | 8c | 0.1617(3) | 0.4512(3) | 0.2422(4) | 0.091(4) | 0.074(4) | 0.063(3) | -0.025(3) | 0.005(3) | -0.003(3) |
| C(9) | 8c | 0.1483(3) | 0.5024(4) | 0.1610(4) | 0.054(3) | 0.087(4) | 0.056(3) | -0.010(3) | 0.002(3) | -0.012(3) |
| C(10) | 8c | 0.0917(4) | 0.4724(5) | 0.0806(4) | 0.092(5) | 0.150(7) | 0.080(4) | -0.048(4) | -0.024(4) | -0.016(4) |
| C(11) | 8c | 0.0776(4) | 0.8065(5) | 0.3654(5) | 0.113(6) | 0.134(6) | 0.119(5) | 0.036(5) | 0.063(5) | -0.010(5) |
| C(12) | 8c | 0.1356(3) | 0.7759(4) | 0.2875(4) | 0.067(4) | 0.082(4) | 0.061(4) | 0.008(3) | 0.012(3) | -0.012(3) |
| C(13) | 8c | 0.1507(4) | 0.8249(4) | 0.2055(5) | 0.094(4) | 0.086(4) | 0.080(4) | 0.036(4) | 0.006(4) | 0.010(4) |
| C(14) | 8c | 0.2047(4) | 0.8036(4) | 0.1310(4) | 0.082(4) | 0.071(4) | 0.046(3) | -0.006(3) | -0.011(3) | 0.001(3) |
| C(15) | 8c | 0.2110(4) | 0.8620(4) | 0.0428(4) | 0.136(6) | 0.095(4) | 0.058(3) | -0.003(4) | -0.017(4) | 0.021(3) |

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