

**cyclo-Decakis( $\mu$ -2,2-diphenylacetato-*O,O'*)-  
dodecakis( $\mu$ -methoxo)decairon(III)) mixed solvate****Huub Kooijman,<sup>a\*</sup> Anthony L. Spek,<sup>a</sup> Elisabeth Bouwman,<sup>b</sup> Fabrizio Miccichè,<sup>b</sup> Sabine T. Warzeska<sup>b</sup> and Jan Reedijk<sup>b</sup>**<sup>a</sup>Bijvoet Center for Biomolecular Research, Department of Crystal and Structural Chemistry, Utrecht University, Padualaan 8, 3584 CH Utrecht, The Netherlands, and <sup>b</sup>Leiden Institute of Chemistry, Gorlaeus Laboratories, Leiden University, PO Box 9502, 2300 RA Leiden, The Netherlands

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The title compound,  $[\text{Fe}_{10}(\text{C}_{14}\text{H}_{11}\text{O}_2)_{10}(\text{CH}_3\text{O})_{20}]$ , takes the form of a so-called 'ferric wheel', consisting of ten Fe cations, linked by carboxylate and methoxo anions. The complex is ring-shaped with the moiety formula  $[\text{Fe}^{\text{III}}(\text{carboxylate})(\text{methoxo})_2]_{10}$  and is located on a crystallographic inversion centre. The unit cell contains two independent regions filled with disordered solvent of unknown composition.

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**Comment**

The title compound, (I), was obtained as a side product in the study of the coordination behaviour of a tridentate nitrogen-containing organic ligand in the presence of various carboxylates (Mimmi *et al.*, 2002).

**Key indicators**

Single-crystal X-ray study

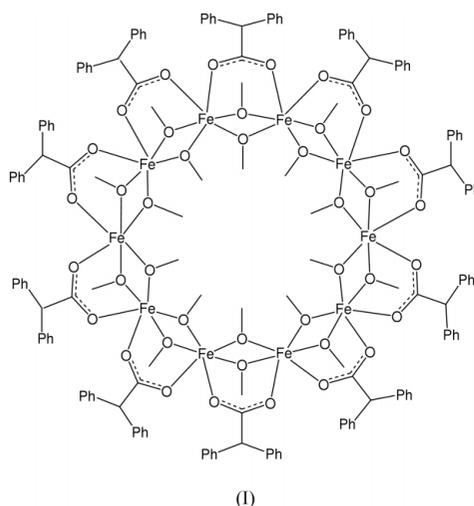
 $T = 150 \text{ K}$ Mean  $\sigma(\text{C}-\text{C}) = 0.015 \text{ \AA}$ 

Disorder in main residue

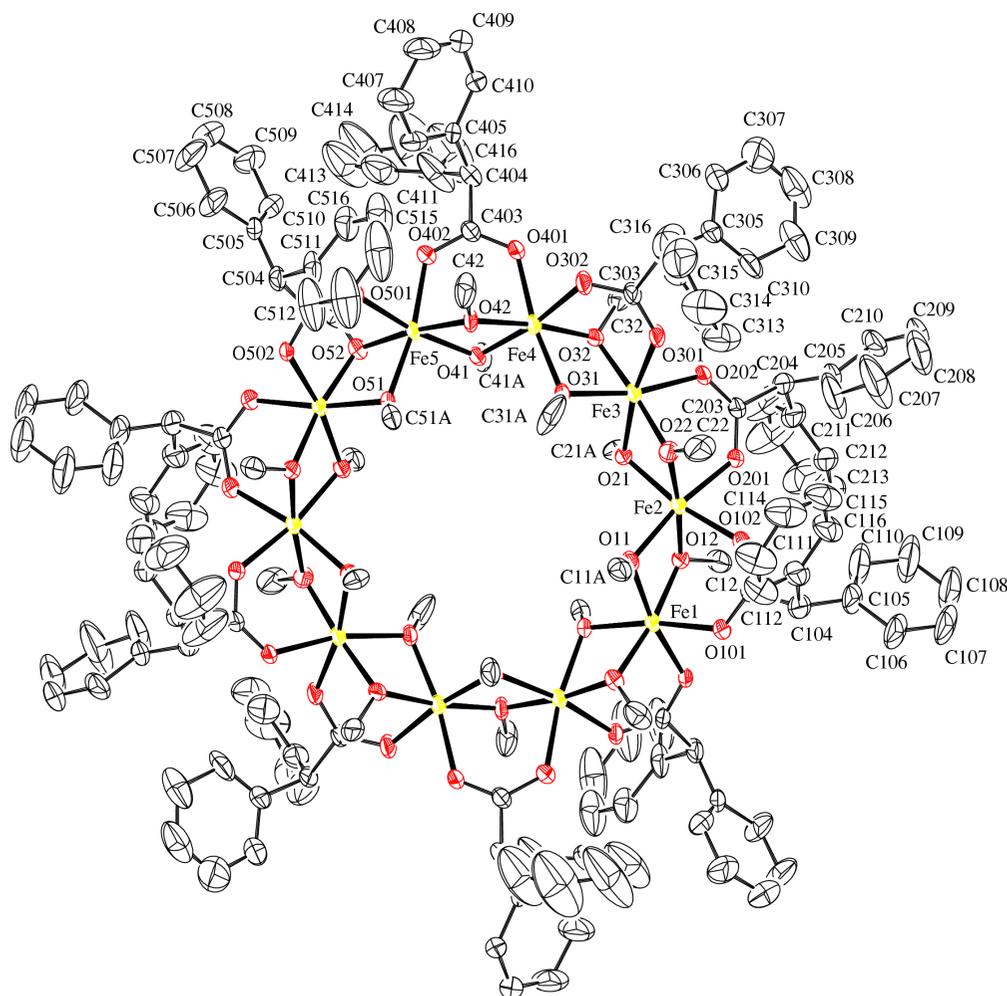
 $R$  factor = 0.078 $wR$  factor = 0.173

Data-to-parameter ratio = 16.5

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.



The decairon complex presented here is a so-called 'ferric wheel', a circular array containing ten segments (see Fig. 1). Each segment consists of an iron cation, connected to the next Fe cation by a triple link formed by two methoxo anions and one carboxylate anion. The moiety formula of the wheel is therefore  $[\text{Fe}^{\text{III}}(\text{carboxylate})(\text{methoxo})_2]_{10}$ . The wheel is located on a crystallographic inversion centre with coordinates  $(1/2, 0, 0)$ . The  $\text{FeO}_6$  chromophores display a distorted octahedral coordination sphere, with deviations from ideal angles up to  $12^\circ$ . All Fe cations lie in one plane [maximum deviation of the least-squares plane through the ten Fe ions is  $0.030 (1) \text{ \AA}$ ]. Similar structures have been reported with other carboxylate anions: chloroacetate (Taft *et al.*, 1994), acetate (Benelli *et al.*, 1996) and 3-(4-methylbenzoyl)propionate (Frey *et al.*, 2000). In all these structures, the triple link between pairs of adjacent iron cations is formed by one carboxylate


**Figure 1**

View of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level. The minor disorder component and H atoms have been omitted for clarity.

anion and two methoxy anions. The Fe···Fe distances and Fe···Fe···Fe angles in (I) lie in the same ranges as found for the previously reported ferric wheel structures.

The methoxy ions pointing inward (*i.e.* to the ‘axle’ of the wheel) are disordered over two positions, which can be characterized as axial or equatorial with respect to the Fe<sub>10</sub> plane. In both orientations, the angle between the vector O—Me and the plane Fe—O—Fe is equal, however. An all-equatorial conformation is not possible due to the occurrence of collisions between the methyl moieties. The phenyl moieties display high anisotropy in their atomic displacement parameters, indicative of slight disorder (see Fig. 1). Attempts to refine two-site disorder models to describe this disorder gave unsatisfactory results. The crystal structure contains two separate regions filled with disordered solvent molecules, each located on a crystallographic inversion centre. The nature of the solvent could not be established unambiguously (see *Experimental*).

## Experimental

The title compound was obtained as a side product in the preparation of complexes of Fe<sup>III</sup> cations with *N,N*-bis(2-ethyl-5-methylimidazol-

4-ylmethyl)aminopropane in the presence of 2,2-diphenylacetate anions. Crystals were obtained by vapor diffusion of diethyl ether and toluene into a solution of the complex mixture in methanol.

### Crystal data

[Fe<sub>10</sub>(C<sub>14</sub>H<sub>11</sub>O<sub>2</sub>)<sub>10</sub>(CH<sub>3</sub>O)<sub>20</sub>]  
 $M_r = 3291.47$   
 Triclinic,  $P\bar{1}$   
 $a = 12.4268$  (12) Å  
 $b = 18.8782$  (16) Å  
 $c = 19.5576$  (16) Å  
 $\alpha = 82.980$  (10)°  
 $\beta = 77.686$  (10)°  
 $\gamma = 82.656$  (10)°  
 $V = 4424.4$  (7) Å<sup>3</sup>

$Z = 1$   
 $D_x = 1.235$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 408 reflections  
 $\theta = 2.0$ – $25.0$ °  
 $\mu = 0.86$  mm<sup>-1</sup>  
 $T = 150$  K  
 Block, brown  
 0.20 × 0.15 × 0.10 mm

### Data collection

Nonius KappaCCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans with  $\kappa$  offset  
 Absorption correction: none  
 91822 measured reflections  
 16169 independent reflections

8452 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.119$   
 $\theta_{\text{max}} = 25.4$ °  
 $h = -14 \rightarrow 14$   
 $k = -22 \rightarrow 22$   
 $l = -23 \rightarrow 23$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.078$

$wR(F^2) = 0.173$

$S = 1.11$

16169 reflections

977 parameters

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 5P]$$

where  $P = (F_o^2 + 2F_c^2)/3$

$$(\Delta/\sigma)_{\max} = 0.008$$

$$\Delta\rho_{\max} = 0.79 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.52 \text{ e } \text{\AA}^{-3}$$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Fe1—Fe2	3.0170 (11)	Fe4—Fe5	3.0172 (11)
Fe2—Fe3	3.0175 (12)	Fe5—Fe1 <sup>i</sup>	3.0161 (11)
Fe3—Fe4	3.0196 (12)		
Fe5 <sup>i</sup> —Fe1—Fe2	141.90 (4)	Fe3—Fe4—Fe5	142.30 (4)
Fe1—Fe2—Fe3	145.88 (4)	Fe4—Fe5—Fe1 <sup>i</sup>	143.88 (4)
Fe2—Fe3—Fe4	145.94 (4)		

Symmetry code: (i)  $1 - x, -y, 2 - z$ .

The disorder of the Me moieties of the methoxo ions located on the inside of the ring is described with a two-site model. The occupation factor of the major component refined to a value of 0.696 (4). Disordered solvent is present in two voids per unit cell, each located on a crystallographic inversion centre. Solvents used in synthesis and crystallization (toluene, diethyl ether and methanol) could not be recognized in the disordered electron density. The contribution of the disordered solvent to the diffraction pattern was therefore incorporated in the model using *PLATON/SQUEEZE* (van der Sluis & Spek, 1990; Spek, 2002). A total of 75 e were found in a void of  $453 \text{ \AA}^3$  [located at (0,0,0)], whereas 92 e were found in a void of  $323 \text{ \AA}^3$  [located at (0,1/2,0)]. Methyl moieties were described as rigid

groups with the C atom as carrier and were allowed to rotate around the C—O bond. All other H atoms were included at calculated positions, riding on their carrier atoms. Isotropic displacement parameters of the H atoms were coupled to the equivalent isotropic displacement parameter of their carrier atoms by a fixed factor.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2002); software used to prepare material for publication: *PLATON*.

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