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# {2-[2′-(Diethylcarbamoylmethoxy)-1,1′-binaphth-2-yloxy]-*N*,*N*-diethylacetamide-$\kappa^4O$}(2,4,6-trinitrophenolato)manganese(II)

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**Key indicators**

Single-crystal X-ray study
*T* = 296 K
Mean σ(C–C) = 0.004 Å
Disorder in main residue
*R* factor = 0.040
*wR* factor = 0.094
Data-to-parameter ratio = 11.4

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



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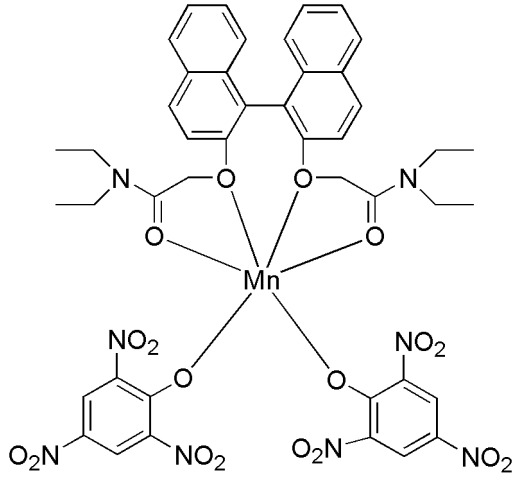
In the title complex, $[Mn(C_6H_2N_3O_7)_2(C_{32}H_{36}N_2O_4)]$, the Mn atom is six-coordinated by four O atoms from the 2-[2′-(diethylcarbamoylmethoxy)-1,1′-binaphth-2-yloxy]-*N*,*N*-diethylacetamide ligand and two O atoms from two monodentate 2,4,6-trinitrophenolate (picrate) ligands. In the crystal structure, the molecule possesses a crystallographically imposed twofold axis of symmetry.

## Comment

Many ligands used in catalysis are under intensive study (Pu & Yu, 2001; Aspinall, 2002; Kubayashi & Ishitani, 1999; Inanaga *et al.*, 2002). These ligands, especially BINOL (1,1′-2,2′-binaphthol), have attracted much attention as chiral ligands for transition metal catalysis (Hanawa *et al.*, 2003; Claver *et al.*, 2000; Keck *et al.*, 1993). They are also used as chiral building blocks in coordination and metallosupramolecular chemistry (Cui *et al.*, 2003; Kesanli & Lin, 2003; Telfer & Kuroda, 2003).




(I)

We have synthesized a new BINOL derivative, 2-[2′-(diethylcarbamoylmethoxy)-1,1′-binaphth-2-yloxy]-*N*,*N*-diethylacetamide, and investigated its reaction with manganese picrate. We present here the crystal structure of the resulting title complex, (I). In (I), the Mn atom is six-coordinated by four O atoms from the ligand and two O atoms from two monodentate picrate ligands (Fig. 1). The coordination polyhedron is a distorted octahedron. The molecule possesses a crystallographically imposed twofold axis of symmetry. Selected bond lengths and angles are given in Table 1. To relieve steric overcrowding, the two naphthalene rings in the molecule are almost perpendicular to each other; the dihedral

angle between them is 91.0°. The distances between the Mn atom and the coordinated ether and carbonyl O atoms are 2.4095 (16) and 2.0859 (17) Å, respectively, indicating that the latter bond is stronger than the former.

## Experimental

To a solution of manganese picrate (0.2 mmol) in ethanol (5 ml) was added dropwise a solution of the ligand (0.2 mmol; Zhang *et al.*, 2003) in ethanol (10 ml). The mixture was stirred at room temperature for 4 h. The precipitated solid complex was filtered, washed with ethanol and dried *in vacuo* over $P_2O_5$ for 48 h. The complex was obtained as a yellow powder. Single crystals of the manganese complex grew from $CH_3Cl$ and $CH_3CH_2OH$ by slow evaporation at room temperature. After about a month, yellow crystals formed from the solution.

*Crystal data*

| | |
|---|---|
| $[Mn(C_6H_2N_3O_7)_2(C_{32}H_{36}N_2O_4)]$ | $D_x$ = 1.455 Mg m$^{-3}$ |
| $M_r$ = 1023.78 | Mo $K\alpha$ radiation |
| Monoclinic, $C2/c$ | Cell parameters from 36 reflections |
| $a$ = 22.802 (4) Å | $\theta$ = 3.2–13.6° |
| $b$ = 14.879 (2) Å | $\mu$ = 0.37 mm$^{-1}$ |
| $c$ = 14.635 (2) Å | $T$ = 296 (2) K |
| $\beta$ = 109.69 (1)° | Block, yellow |
| $V$ = 4675.1 (14) Å$^3$ | 0.56 × 0.40 × 0.40 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Siemens *P*4 diffractometer | $R_{int}$ = 0.015 |
| $\omega$ scans | $\theta_{max}$ = 25.3° |
| Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997) | $h = 0 \rightarrow 27$ |
| | $k = 0 \rightarrow 17$ |
| $T_{min}$ = 0.786, $T_{max}$ = 0.863 | $l = -17 \rightarrow 16$ |
| 4704 measured reflections | 3 standard reflections |
| 4245 independent reflections | every 97 reflections |
| 2312 reflections with $I > 2\sigma(I)$ | intensity decay: 1.8% |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0478P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)]$ = 0.040 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $wR(F^2)$ = 0.094 | $\Delta\rho_{max}$ = 0.35 e Å$^{-3}$ |
| $S$ = 0.82 | $\Delta\rho_{min}$ = −0.21 e Å$^{-3}$ |
| 4245 reflections | Extinction correction: *SHELXL*97 |
| 371 parameters | Extinction coefficient: 0.00184 (14) |
| H-atom parameters constrained | |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Mn—O3 | 2.0413 (19) | Mn—O1 | 2.4095 (16) |
| Mn—O2 | 2.0859 (17) | | |
| O3—Mn—O2 | 102.39 (8) | O2—Mn—O1 | 69.31 (6) |
| O3—Mn—O2$^{i}$ | 98.56 (8) | O3—Mn—O1$^{i}$ | 92.72 (7) |
| O2—Mn—O2$^{i}$ | 147.60 (9) | O2—Mn—O1$^{i}$ | 85.18 (6) |
| O3—Mn—O1 | 166.88 (8) | O1—Mn—O1$^{i}$ | 76.77 (8) |

Symmetry code: (i) $1-x, y, \frac{3}{2}-z$.

H atoms were treated as riding atoms, with C—H distances of 0.96 ($CH_3$), 0.97 ($CH_2$) and 0.93 Å (CH) [$U_{iso}$(H) = 1.2$U_{eq}$(C)]. The five atoms (N2, O4, O5, O8 and O9) of one picrate ligand are disordered, and the refined occupancies for the disordered components are 0.882 (7) and 0.118 (7) for N2, O4 and O5, and 0.625 (18) and 0.375 (18) for O8 and O9. Geometrical restraints were applied to the disordered atoms.

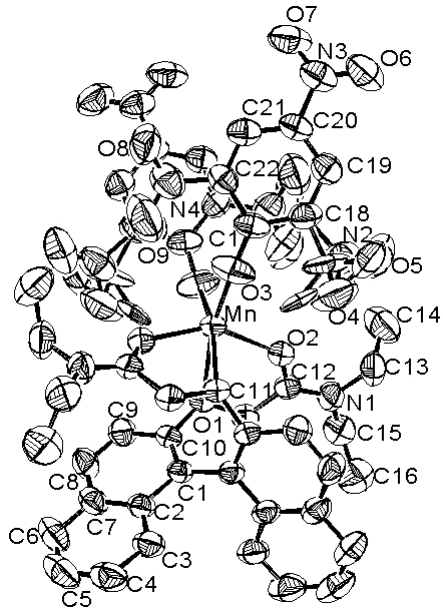



**Figure 1**
A view of (I), showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level. Only major components of the disordered groups are shown. H atoms have been omitted for clarity. Unlabeled atoms are related to labeled atoms by $(1-x, y, \frac{3}{2}-z)$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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