

Diazidobis[4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-2-imidazoline-1-oxyl-3-oxide- κ^2 O,N]manganese(II)

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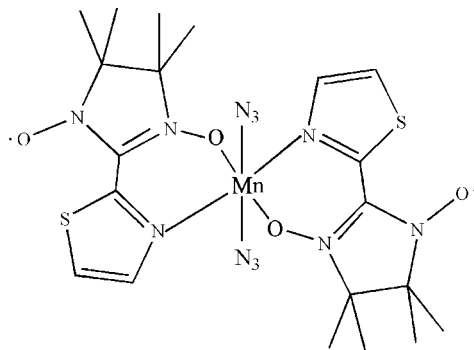
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.039; wR factor = 0.110; data-to-parameter ratio = 16.8.

In the crystal structure of the title compound, $[\text{Mn}(\text{N}_3)_2(\text{C}_{10}\text{H}_{14}\text{N}_3\text{O}_2\text{S})_2]$, the Mn(II) atom exhibits a roughly octahedral coordination geometry. The Mn(II) atom lies on an inversion centre, thus the asymmetric unit comprises one half-molecule. The metal center is six-coordinated by two azide anions and by two chelating 4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-2-imidazoline-1-oxyl-3-oxide nitronyl nitroxide radical ligands, leading to two six-membered chelate rings.

Related literature

For the design and synthesis of molecule-based magnetic materials, see: Aoki *et al.* (2003). For nitronyl nitroxide radicals, see: Minguet *et al.* (2000); Catala *et al.* (2005). For transition metal-radical complexes, see: Wang *et al.* (2005). For paramagnetic metal complexes of nitronyl nitroxide radicals, see: Li *et al.* (2002); Liu *et al.* (2001). For the synthesis, see: Ullman *et al.* (1970, 1972)



Experimental

Crystal data

$[\text{Mn}(\text{N}_3)_2(\text{C}_{10}\text{H}_{14}\text{N}_3\text{O}_2\text{S})_2]$	$V = 1348.1$ (4) Å ³
$M_r = 619.60$	$Z = 2$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 9.9600$ (18) Å	$\mu = 0.70$ mm ⁻¹
$b = 12.272$ (2) Å	$T = 291$ (2) K
$c = 11.353$ (2) Å	$0.45 \times 0.30 \times 0.25$ mm
$\beta = 103.714$ (3)°	

Data collection

Bruker SMART CCD area-detector diffractometer	7966 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3061 independent reflections
$T_{\min} = 0.745$, $T_{\max} = 0.846$	2628 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.036$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$	182 parameters
$wR(F^2) = 0.110$	H-atom parameters constrained
$S = 1.06$	$\Delta\rho_{\text{max}} = 0.37$ e Å ⁻³
3061 reflections	$\Delta\rho_{\text{min}} = -0.25$ e Å ⁻³

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: publCIF (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2202).

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Diazidobis[4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-2-imidazoline-1-oxyl-3-oxide- κ^2O,N]manganese(II)

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Comment

The design and synthesis of molecule-based magnetic materials is one of the major subjects of materials science (Aoki *et al.* 2003). In many different types of organic radicals, research has focused on the nitronyl nitroxide radicals (NITR) family because of their flexibility and functionality (Minguet *et al.* 2000; Catala *et al.* 2005). The nitroxide derivatives can be bound to the metal center through the oxygen atoms of O–N groups, affording a good variety of transition metal–radical complexes (Wang *et al.* 2005;). There have been many magnetic studies on transition metal complexes with nitronyl nitroxide and imino nitroxide radicals and paramagnetic metal complexes of nitronyl nitroxide radicals have been extensively studied (Li *et al.* 2002; Liu *et al.* 2001). In the present paper, we report the synthesis and crystal structure of the title compound $Mn(N_3)_2(NIT2-thz)_2$.

Experimental

NIT2-thz [NIT2-thz = 4,4,5,5-tetramethyl-2-(1,3-thiazol-2-yl)-2-imidazoline-1-oxyl-3-oxide] was synthesized using a method in the literature (Ullman *et al.* 1970; Ullman *et al.* 1972). $Mn(Ac)_2 \cdot 4H_2O$ (1 mmol) and NIT2-thz (2 mmol) were mixed in 30 ml of methanol. An aqueous solution (10 ml) of NaN_3 (2 mmol) was added to this solution. The mixture was stirred for an 1 h and filtered off. The filtrate was kept at room temperature for 1 week, and well formed dark brown crystals of $Mn(N_3)_2(NIT2-thz)_2$ were obtained.

Refinement

The H atoms were positioned geometrically and refined using the riding-model approximation, with C–H = 0.93 or 0.96 Å and $U_{iso}(H) = 1.2U_{eq}(\text{carrier})$ or $U_{iso}(H) = 1.5U_{eq}(\text{methyl carrier})$.

Figures

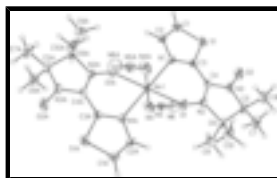


Fig. 1. ORTEP drawing of the title compound with atom labeling. The thermal ellipsoids are drawn at 30% probability level. [symmetry codes: $x, -y, -z + 1$].

supplementary materials

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Crystal data

[Mn(N₃)₂(C₁₀H₁₄N₃O₂S)₂]

$M_r = 619.60$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 9.9600$ (18) Å

$b = 12.272$ (2) Å

$c = 11.353$ (2) Å

$\beta = 103.714$ (3)°

$V = 1348.1$ (4) Å³

$Z = 2$

$F_{000} = 642$

$D_x = 1.526$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 4318 reflections

$\theta = 2.5$ – 28.2 °

$\mu = 0.70$ mm⁻¹

$T = 291$ (2) K

Block, dark brown

$0.45 \times 0.30 \times 0.25$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291$ (2) K

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.745$, $T_{\max} = 0.846$

7966 measured reflections

3061 independent reflections

2628 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.036$

$\theta_{\max} = 27.5$ °

$\theta_{\min} = 2.5$ °

$h = -12 \rightarrow 12$

$k = -15 \rightarrow 15$

$l = -12 \rightarrow 14$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.039$

$wR(F^2) = 0.110$

$S = 1.06$

3061 reflections

182 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0605P)^2 + 0.2559P]$$

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

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.37$ e Å⁻³

$\Delta\rho_{\min} = -0.25$ e Å⁻³

Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes)

are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Mn1	0.0000	0.0000	0.5000	0.03345 (14)
S1	0.25159 (5)	0.27889 (4)	0.36683 (5)	0.05049 (17)
O1	0.17034 (15)	-0.08711 (11)	0.44955 (14)	0.0504 (4)
O2	0.38630 (18)	0.14599 (13)	0.23568 (17)	0.0660 (5)
N1	0.12384 (15)	0.14413 (11)	0.47278 (13)	0.0337 (3)
N2	0.23912 (14)	-0.05108 (11)	0.37542 (13)	0.0336 (3)
N3	0.34509 (15)	0.05765 (13)	0.27421 (14)	0.0407 (4)
N4	-0.1080 (2)	0.00819 (18)	0.31193 (18)	0.0641 (6)
N5	-0.08511 (17)	0.04681 (12)	0.22465 (15)	0.0433 (4)
N6	-0.0640 (3)	0.08223 (18)	0.13715 (19)	0.0761 (7)
C1	0.1569 (2)	0.32772 (15)	0.4616 (2)	0.0495 (5)
H1	0.1479	0.4012	0.4782	0.059*
C2	0.0969 (2)	0.24624 (14)	0.50955 (17)	0.0412 (4)
H2	0.0413	0.2584	0.5635	0.049*
C3	0.20718 (16)	0.14870 (12)	0.39806 (15)	0.0318 (3)
C4	0.25886 (16)	0.05337 (13)	0.34979 (15)	0.0317 (3)
C5	0.40415 (19)	-0.05207 (16)	0.25646 (16)	0.0406 (4)
C6	0.30045 (18)	-0.12873 (14)	0.29998 (16)	0.0382 (4)
C7	0.4089 (3)	-0.0651 (2)	0.12397 (19)	0.0628 (6)
H7A	0.3179	-0.0551	0.0732	0.094*
H7B	0.4417	-0.1367	0.1114	0.094*
H7C	0.4702	-0.0116	0.1039	0.094*
C8	0.5497 (2)	-0.0545 (2)	0.3366 (2)	0.0601 (6)
H8A	0.6029	0.0037	0.3137	0.090*

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H8B	0.5923	-0.1230	0.3269	0.090*
H8C	0.5461	-0.0455	0.4197	0.090*
C9	0.1798 (2)	-0.1673 (2)	0.1994 (2)	0.0595 (6)
H9A	0.1102	-0.1990	0.2346	0.089*
H9B	0.2118	-0.2208	0.1507	0.089*
H9C	0.1413	-0.1064	0.1496	0.089*
C10	0.3643 (3)	-0.22455 (19)	0.3780 (2)	0.0620 (6)
H10A	0.4243	-0.1981	0.4515	0.093*
H10B	0.4167	-0.2680	0.3345	0.093*
H10C	0.2925	-0.2682	0.3974	0.093*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Mn1	0.0362 (2)	0.0333 (2)	0.0373 (2)	-0.00148 (13)	0.02166 (16)	0.00206 (13)
S1	0.0483 (3)	0.0342 (2)	0.0743 (4)	-0.00716 (19)	0.0251 (3)	0.0074 (2)
O1	0.0597 (9)	0.0351 (6)	0.0717 (10)	0.0065 (6)	0.0459 (8)	0.0095 (6)
O2	0.0697 (10)	0.0592 (9)	0.0861 (12)	-0.0037 (8)	0.0525 (10)	0.0169 (8)
N1	0.0365 (7)	0.0299 (7)	0.0381 (7)	0.0019 (5)	0.0156 (6)	-0.0003 (5)
N2	0.0325 (7)	0.0336 (7)	0.0395 (8)	0.0037 (5)	0.0182 (6)	0.0003 (6)
N3	0.0371 (8)	0.0466 (8)	0.0452 (8)	0.0000 (6)	0.0230 (7)	0.0038 (7)
N4	0.0681 (13)	0.0827 (14)	0.0416 (10)	-0.0251 (10)	0.0134 (9)	0.0060 (9)
N5	0.0497 (9)	0.0360 (8)	0.0453 (9)	-0.0042 (6)	0.0138 (7)	0.0000 (7)
N6	0.121 (2)	0.0587 (12)	0.0582 (12)	-0.0128 (12)	0.0400 (13)	0.0104 (10)
C1	0.0490 (11)	0.0305 (8)	0.0668 (13)	0.0003 (8)	0.0093 (10)	-0.0061 (8)
C2	0.0449 (10)	0.0352 (8)	0.0444 (10)	0.0065 (7)	0.0122 (8)	-0.0065 (7)
C3	0.0297 (8)	0.0302 (7)	0.0372 (8)	-0.0012 (6)	0.0113 (6)	0.0028 (6)
C4	0.0277 (8)	0.0361 (8)	0.0335 (8)	0.0006 (6)	0.0118 (6)	0.0029 (6)
C5	0.0346 (9)	0.0556 (11)	0.0361 (9)	0.0076 (7)	0.0170 (7)	-0.0030 (8)
C6	0.0369 (9)	0.0404 (9)	0.0402 (9)	0.0095 (7)	0.0151 (7)	-0.0048 (7)
C7	0.0640 (14)	0.0898 (17)	0.0422 (11)	0.0087 (12)	0.0277 (10)	-0.0064 (11)
C8	0.0358 (10)	0.0856 (16)	0.0599 (13)	0.0054 (10)	0.0133 (10)	-0.0127 (12)
C9	0.0569 (13)	0.0618 (13)	0.0587 (13)	-0.0084 (10)	0.0117 (11)	-0.0173 (10)
C10	0.0695 (15)	0.0542 (12)	0.0703 (15)	0.0292 (11)	0.0324 (13)	0.0111 (11)

Geometric parameters (\AA , $^\circ$)

Mn1—N4 ⁱ	2.153 (2)	C2—H2	0.9300
Mn1—N4	2.153 (2)	C3—C4	1.438 (2)
Mn1—O1 ⁱ	2.1931 (12)	C5—C8	1.518 (3)
Mn1—O1	2.1931 (12)	C5—C7	1.525 (3)
Mn1—N1	2.2194 (14)	C5—C6	1.561 (3)
Mn1—N1 ⁱ	2.2194 (14)	C6—C10	1.518 (3)
S1—C1	1.699 (2)	C6—C9	1.524 (3)
S1—C3	1.7170 (16)	C7—H7A	0.9600
O1—N2	1.2832 (17)	C7—H7B	0.9600
O2—N3	1.273 (2)	C7—H7C	0.9600
N1—C3	1.321 (2)	C8—H8A	0.9600

N1—C2	1.367 (2)	C8—H8B	0.9600
N2—C4	1.339 (2)	C8—H8C	0.9600
N2—C6	1.504 (2)	C9—H9A	0.9600
N3—C4	1.3513 (19)	C9—H9B	0.9600
N3—C5	1.502 (2)	C9—H9C	0.9600
N4—N5	1.168 (2)	C10—H10A	0.9600
N5—N6	1.148 (2)	C10—H10B	0.9600
C1—C2	1.345 (3)	C10—H10C	0.9600
C1—H1	0.9300		
N4 ⁱ —Mn1—N4	180.0	N2—C4—C3	127.70 (14)
N4 ⁱ —Mn1—O1 ⁱ	89.95 (8)	N3—C4—C3	123.31 (15)
N4—Mn1—O1 ⁱ	90.05 (8)	N3—C5—C8	106.60 (17)
N4 ⁱ —Mn1—O1	90.05 (8)	N3—C5—C7	109.34 (17)
N4—Mn1—O1	89.95 (8)	C8—C5—C7	109.88 (17)
O1 ⁱ —Mn1—O1	180.0	N3—C5—C6	100.84 (13)
N4 ⁱ —Mn1—N1	90.68 (6)	C8—C5—C6	114.10 (17)
N4—Mn1—N1	89.32 (6)	C7—C5—C6	115.27 (18)
O1 ⁱ —Mn1—N1	97.92 (5)	N2—C6—C10	109.20 (15)
O1—Mn1—N1	82.08 (5)	N2—C6—C9	105.59 (15)
N4 ⁱ —Mn1—N1 ⁱ	89.32 (6)	C10—C6—C9	110.10 (19)
N4—Mn1—N1 ⁱ	90.68 (6)	N2—C6—C5	100.77 (13)
O1 ⁱ —Mn1—N1 ⁱ	82.08 (5)	C10—C6—C5	115.73 (16)
O1—Mn1—N1 ⁱ	97.92 (5)	C9—C6—C5	114.43 (16)
N1—Mn1—N1 ⁱ	180.0	C5—C7—H7A	109.5
C1—S1—C3	89.35 (9)	C5—C7—H7B	109.5
N2—O1—Mn1	124.73 (10)	H7A—C7—H7B	109.5
C3—N1—C2	110.83 (14)	C5—C7—H7C	109.5
C3—N1—Mn1	125.29 (11)	H7A—C7—H7C	109.5
C2—N1—Mn1	122.15 (11)	H7B—C7—H7C	109.5
O1—N2—C4	126.95 (13)	C5—C8—H8A	109.5
O1—N2—C6	120.47 (13)	C5—C8—H8B	109.5
C4—N2—C6	112.47 (13)	H8A—C8—H8B	109.5
O2—N3—C4	123.82 (15)	C5—C8—H8C	109.5
O2—N3—C5	123.29 (14)	H8A—C8—H8C	109.5
C4—N3—C5	112.24 (14)	H8B—C8—H8C	109.5
N5—N4—Mn1	135.07 (17)	C6—C9—H9A	109.5
N6—N5—N4	178.1 (2)	C6—C9—H9B	109.5
C2—C1—S1	111.16 (14)	H9A—C9—H9B	109.5
C2—C1—H1	124.4	C6—C9—H9C	109.5
S1—C1—H1	124.4	H9A—C9—H9C	109.5
C1—C2—N1	114.81 (16)	H9B—C9—H9C	109.5
C1—C2—H2	122.6	C6—C10—H10A	109.5
N1—C2—H2	122.6	C6—C10—H10B	109.5
N1—C3—C4	123.11 (14)	H10A—C10—H10B	109.5
N1—C3—S1	113.83 (12)	C6—C10—H10C	109.5
C4—C3—S1	123.04 (12)	H10A—C10—H10C	109.5

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N2—C4—N3	108.87 (14)	H10B—C10—H10C	109.5
N4 ⁱ —Mn1—O1—N2	-122.97 (15)	O1—N2—C4—N3	175.59 (17)
N4—Mn1—O1—N2	57.03 (15)	C6—N2—C4—N3	-8.19 (19)
O1 ⁱ —Mn1—O1—N2	105 (8)	O1—N2—C4—C3	-0.6 (3)
N1—Mn1—O1—N2	-32.28 (14)	C6—N2—C4—C3	175.65 (17)
N1 ⁱ —Mn1—O1—N2	147.72 (14)	O2—N3—C4—N2	-178.27 (17)
N4 ⁱ —Mn1—N1—C3	118.52 (15)	C5—N3—C4—N2	-7.24 (19)
N4—Mn1—N1—C3	-61.48 (15)	O2—N3—C4—C3	-1.9 (3)
O1 ⁱ —Mn1—N1—C3	-151.43 (14)	C5—N3—C4—C3	169.12 (16)
O1—Mn1—N1—C3	28.57 (14)	N1—C3—C4—N2	-3.7 (3)
N1 ⁱ —Mn1—N1—C3	24.5 (17)	S1—C3—C4—N2	174.59 (14)
N4 ⁱ —Mn1—N1—C2	-77.84 (15)	N1—C3—C4—N3	-179.39 (16)
N4—Mn1—N1—C2	102.16 (15)	S1—C3—C4—N3	-1.1 (2)
O1 ⁱ —Mn1—N1—C2	12.20 (15)	O2—N3—C5—C8	70.0 (2)
O1—Mn1—N1—C2	-167.80 (15)	C4—N3—C5—C8	-101.13 (18)
N1 ⁱ —Mn1—N1—C2	-171.9 (18)	O2—N3—C5—C7	-48.8 (2)
Mn1—O1—N2—C4	26.1 (2)	C4—N3—C5—C7	140.13 (18)
Mn1—O1—N2—C6	-149.81 (13)	O2—N3—C5—C6	-170.64 (18)
N4 ⁱ —Mn1—N4—N5	-71 (2)	C4—N3—C5—C6	18.27 (18)
O1 ⁱ —Mn1—N4—N5	118.9 (3)	O1—N2—C6—C10	-42.4 (2)
O1—Mn1—N4—N5	-61.1 (3)	C4—N2—C6—C10	141.14 (17)
N1—Mn1—N4—N5	21.0 (3)	O1—N2—C6—C9	76.0 (2)
N1 ⁱ —Mn1—N4—N5	-159.0 (3)	C4—N2—C6—C9	-100.50 (18)
Mn1—N4—N5—N6	137 (8)	O1—N2—C6—C5	-164.65 (15)
C3—S1—C1—C2	-0.76 (16)	C4—N2—C6—C5	18.85 (18)
S1—C1—C2—N1	-0.1 (2)	N3—C5—C6—N2	-20.40 (16)
C3—N1—C2—C1	1.2 (2)	C8—C5—C6—N2	93.45 (18)
Mn1—N1—C2—C1	-164.58 (14)	C7—C5—C6—N2	-138.01 (17)
C2—N1—C3—C4	176.71 (16)	N3—C5—C6—C10	-138.01 (17)
Mn1—N1—C3—C4	-18.1 (2)	C8—C5—C6—C10	-24.2 (2)
C2—N1—C3—S1	-1.76 (19)	C7—C5—C6—C10	104.4 (2)
Mn1—N1—C3—S1	163.46 (8)	N3—C5—C6—C9	92.35 (18)
C1—S1—C3—N1	1.47 (15)	C8—C5—C6—C9	-153.79 (17)
C1—S1—C3—C4	-177.00 (16)	C7—C5—C6—C9	-25.3 (2)

Symmetry codes: (i) $-x, -y, -z+1$.

