

Bis(μ -2-methylquinolin-8-olato)- $\kappa^3N,O; \kappa^3O:N, O$ -bis[(acetato- κO)-(methanol- κO)zinc(II)]

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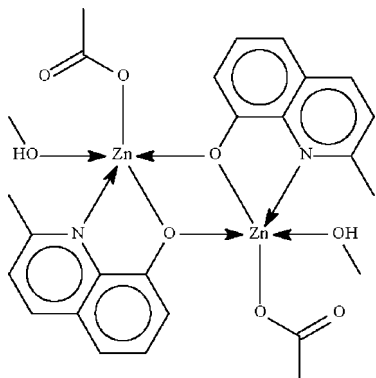
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.008$ Å; R factor = 0.076; wR factor = 0.230; data-to-parameter ratio = 16.3.

The reaction of zinc acetate and 2-methyl-8-hydroxyquinoline in methanol yielded the centrosymmetric dinuclear title compound, $[Zn_2(C_{10}H_8NO)_2(CH_3CO_2)_2(CH_3OH)_2]$, which has the Zn atom within a distorted NO_4 trigonal-bipyramidal coordination geometry. Methanol-acetate $O-H \cdots O$ hydrogen bonds link the dinuclear units into a linear supramolecular chain extending parallel to $[100]$.

Related literature

Unlike 8-hydroxyquinoline, which yields a large number of metal derivatives, 2-methyl-8-hydroxyquinoline forms only a small number of metal chelates. Besides a related chloride salt (Sattarzadeh *et al.*, 2009), there is only one crystal structure report of another zinc derivative; for aquabis(2-methylquinolin-8-ato)zinc, see: da Silva *et al.* (2007).



Experimental

Crystal data

$[Zn_2(C_{10}H_8NO)_2(C_2H_3O_2)_2 \cdot (CH_3O)_2]$
 $M_r = 629.26$
 Triclinic, $P\bar{1}$
 $a = 6.9496$ (1) Å
 $b = 9.6262$ (2) Å
 $c = 9.8232$ (2) Å
 $\alpha = 75.241$ (1)°

$\beta = 89.688$ (1)°
 $\gamma = 86.596$ (1)°
 $V = 634.32$ (2) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 1.95$ mm⁻¹
 $T = 100$ K
 $0.38 \times 0.28 \times 0.18$ mm

Data collection

Bruker SMART APEX diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.525$, $T_{max} = 0.721$

5601 measured reflections
 2855 independent reflections
 2534 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.042$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.076$
 $wR(F^2) = 0.230$
 $S = 1.13$
 2855 reflections

175 parameters
 H-atom parameters constrained
 $\Delta\rho_{max} = 3.72$ e Å⁻³
 $\Delta\rho_{min} = -1.85$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H \cdots A$ | $D-H$ | $H \cdots A$ | $D \cdots A$ | $D-H \cdots A$ |
|---------------------|-------|--------------|--------------|----------------|
| $O4-H4 \cdots O3^i$ | 0.84 | 1.88 | 2.602 (6) | 143 |

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); 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: TK2424).

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 Westrip, S. P. (2009). publCIF. In preparation.

supplementary materials

Acta Cryst. (2009). E65, m554 [doi:10.1107/S1600536809014214]

Bis(μ -2-methylquinolin-8-olato)- κ^3 N,O:O; κ^3 O:N,O-bis[(acetato- κ O)(methanol- κ O)zinc(II)]

E. Sattarzadeh, G. Mohammadnezhad, M. M. Amini and S. W. Ng

Experimental

Zinc acetate (0.17 g, 0.75 mmol) and 2-methyl-8-hydroxyquinoline (0.24 g, 1.5 mmol) were loaded into a convection tube; the tube was filled with dry methanol and kept at 333 K. Crystals were collected from the side arm after several days. Although well formed, all specimens had a slightly blemished interior.

Refinement

The crystal used in the study was a multiply twinned crystal. The diffraction intensities were separated with the RLATT routine of the data collection software, and that component that diffracted to the highest 2θ limit was selected for integration. Although the specimen diffracted strongly, with a high proportion of 'observeds', there was serious overlapping between the main component and the minor components, particularly at low angles.

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.98 Å; O—H 0.84 Å) and were included in the refinement in the riding model approximation, with $U(\text{H})$ set to 1.2 to 1.5 $U(\text{C}, \text{O})$.

The final difference Fourier map had a large peak/deep hole in the vicinity of the Zn1 atom. These could not be reduced even with the 2θ maximum was lowered to 50 °.

Figures

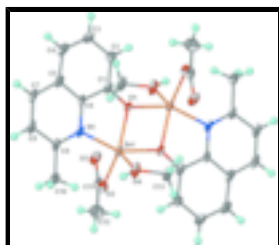


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of dinuclear $[\text{Zn}(\text{C}_{10}\text{H}_8\text{NO})(\text{CH}_3\text{OH})(\text{CH}_3\text{CO}_2)]_2$; ellipsoids are drawn at the 70% probability level and H atoms of arbitrary radius. The unlabelled atoms are related by a centre of inversion.

Bis(μ -2-methylquinolin-8-olato)- κ^3 N,O:O; κ^3 O:N,O- bis[(acetato- κ O)(methanol- κ O)zinc(II)]

Crystal data

$[\text{Zn}_2(\text{C}_{10}\text{H}_8\text{NO})_2(\text{C}_2\text{H}_3\text{O}_2)_2(\text{CH}_4\text{O})_2]$

$M_r = 629.26$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.9496(1) \text{ \AA}$

$Z = 1$

$F_{000} = 324$

$D_x = 1.647 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 3551 reflections

supplementary materials

$b = 9.6262$ (2) Å
 $c = 9.8232$ (2) Å
 $\alpha = 75.241$ (1)°
 $\beta = 89.688$ (1)°
 $\gamma = 86.596$ (1)°
 $V = 634.32$ (2) Å³

$\theta = 2.2$ – 28.3 °
 $\mu = 1.95$ mm⁻¹
 $T = 100$ K
Block, yellow
 $0.38 \times 0.28 \times 0.18$ mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
 $T = 100$ K
 ω scans
Absorption correction: Multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.525$, $T_{\max} = 0.721$
5601 measured reflections

2855 independent reflections
2534 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.042$
 $\theta_{\max} = 27.5$ °
 $\theta_{\min} = 2.1$ °
 $h = -9 \rightarrow 9$
 $k = -12 \rightarrow 12$
 $l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.076$
 $wR(F^2) = 0.230$
 $S = 1.13$
2855 reflections
175 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
 $w = 1/[\sigma^2(F_o^2) + (0.1574P)^2 + 1.7954P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 3.72$ e Å⁻³
 $\Delta\rho_{\min} = -1.85$ e Å⁻³
Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|-------------|-------------|-------------|----------------------------------|
| Zn1 | 0.57254 (8) | 0.63131 (6) | 0.87572 (5) | 0.0148 (3) |
| O1 | 0.5349 (5) | 0.4205 (4) | 0.9193 (4) | 0.0174 (8) |
| O2 | 0.4331 (6) | 0.8211 (4) | 0.8160 (4) | 0.0201 (8) |
| O3 | 0.1619 (6) | 0.7137 (5) | 0.8017 (4) | 0.0236 (9) |
| O4 | 0.8356 (6) | 0.6830 (5) | 0.9340 (4) | 0.0233 (9) |
| H4 | 0.9089 | 0.7231 | 0.8695 | 0.028* |
| N1 | 0.6717 (6) | 0.5859 (5) | 0.6847 (5) | 0.0165 (9) |
| C1 | 0.5872 (7) | 0.3551 (6) | 0.8200 (5) | 0.0169 (10) |
| C2 | 0.5764 (8) | 0.2097 (6) | 0.8320 (6) | 0.0184 (10) |
| H2 | 0.5313 | 0.1493 | 0.9168 | 0.022* |
| C3 | 0.6315 (9) | 0.1497 (6) | 0.7196 (6) | 0.0229 (11) |

| | | | | |
|------|------------|------------|------------|-------------|
| H3 | 0.6238 | 0.0492 | 0.7303 | 0.028* |
| C4 | 0.6955 (8) | 0.2334 (6) | 0.5956 (6) | 0.0232 (11) |
| H4A | 0.7301 | 0.1912 | 0.5207 | 0.028* |
| C5 | 0.7104 (8) | 0.3827 (6) | 0.5786 (6) | 0.0191 (10) |
| C6 | 0.6596 (7) | 0.4431 (6) | 0.6917 (5) | 0.0150 (9) |
| C7 | 0.7707 (8) | 0.4776 (6) | 0.4547 (6) | 0.0196 (11) |
| H7 | 0.8029 | 0.4427 | 0.3748 | 0.023* |
| C8 | 0.7834 (8) | 0.6205 (6) | 0.4485 (5) | 0.0208 (11) |
| H8 | 0.8248 | 0.6846 | 0.3646 | 0.025* |
| C9 | 0.7341 (8) | 0.6730 (6) | 0.5687 (5) | 0.0178 (10) |
| C10 | 0.7542 (9) | 0.8281 (6) | 0.5643 (6) | 0.0227 (11) |
| H10A | 0.7336 | 0.8427 | 0.6586 | 0.034* |
| H10B | 0.6582 | 0.8879 | 0.4988 | 0.034* |
| H10C | 0.8838 | 0.8552 | 0.5326 | 0.034* |
| C11 | 0.2496 (8) | 0.8207 (6) | 0.8104 (5) | 0.0179 (10) |
| C12 | 0.1384 (9) | 0.9585 (7) | 0.8161 (7) | 0.0297 (13) |
| H12A | 0.2244 | 1.0381 | 0.7939 | 0.045* |
| H12B | 0.0864 | 0.9488 | 0.9108 | 0.045* |
| H12C | 0.0322 | 0.9780 | 0.7474 | 0.045* |
| C13 | 0.9045 (8) | 0.6565 (7) | 1.0750 (6) | 0.0233 (11) |
| H13A | 1.0426 | 0.6285 | 1.0786 | 0.035* |
| H13B | 0.8350 | 0.5787 | 1.1356 | 0.035* |
| H13C | 0.8832 | 0.7441 | 1.1079 | 0.035* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| Zn1 | 0.0190 (4) | 0.0192 (4) | 0.0083 (4) | -0.0030 (2) | 0.0016 (2) | -0.0068 (2) |
| O1 | 0.027 (2) | 0.0212 (18) | 0.0063 (16) | -0.0035 (15) | 0.0078 (14) | -0.0071 (14) |
| O2 | 0.025 (2) | 0.0206 (19) | 0.0146 (17) | -0.0033 (15) | 0.0021 (15) | -0.0033 (14) |
| O3 | 0.023 (2) | 0.032 (2) | 0.020 (2) | -0.0040 (16) | 0.0045 (16) | -0.0146 (17) |
| O4 | 0.0186 (19) | 0.041 (2) | 0.0123 (18) | -0.0090 (16) | 0.0026 (14) | -0.0094 (16) |
| N1 | 0.018 (2) | 0.024 (2) | 0.0098 (19) | -0.0017 (16) | -0.0004 (15) | -0.0079 (16) |
| C1 | 0.018 (2) | 0.024 (3) | 0.012 (2) | -0.0030 (19) | 0.0016 (18) | -0.0084 (19) |
| C2 | 0.023 (3) | 0.019 (2) | 0.015 (2) | -0.0026 (19) | 0.0028 (19) | -0.0058 (19) |
| C3 | 0.030 (3) | 0.021 (3) | 0.021 (3) | 0.001 (2) | -0.002 (2) | -0.012 (2) |
| C4 | 0.025 (3) | 0.029 (3) | 0.020 (3) | -0.001 (2) | 0.000 (2) | -0.015 (2) |
| C5 | 0.017 (2) | 0.027 (3) | 0.016 (2) | 0.000 (2) | -0.0011 (19) | -0.012 (2) |
| C6 | 0.017 (2) | 0.021 (2) | 0.008 (2) | 0.0006 (18) | 0.0004 (17) | -0.0055 (18) |
| C7 | 0.018 (2) | 0.032 (3) | 0.012 (2) | 0.002 (2) | 0.0005 (19) | -0.012 (2) |
| C8 | 0.020 (3) | 0.033 (3) | 0.010 (2) | -0.001 (2) | -0.0003 (19) | -0.008 (2) |
| C9 | 0.018 (2) | 0.027 (3) | 0.009 (2) | -0.003 (2) | 0.0009 (18) | -0.0058 (19) |
| C10 | 0.035 (3) | 0.023 (3) | 0.012 (2) | -0.006 (2) | 0.003 (2) | -0.006 (2) |
| C11 | 0.020 (2) | 0.024 (3) | 0.010 (2) | 0.0004 (19) | 0.0012 (18) | -0.0057 (19) |
| C12 | 0.027 (3) | 0.027 (3) | 0.034 (3) | 0.002 (2) | 0.002 (2) | -0.006 (2) |
| C13 | 0.023 (3) | 0.035 (3) | 0.013 (2) | -0.004 (2) | 0.001 (2) | -0.009 (2) |

supplementary materials

Geometric parameters (Å, °)

| | | | |
|-------------------------|-------------|---------------|-----------|
| Zn1—O1 | 1.997 (4) | C4—C5 | 1.414 (8) |
| Zn1—O2 | 1.968 (4) | C4—H4A | 0.9500 |
| Zn1—O1 ⁱ | 2.092 (3) | C5—C7 | 1.402 (8) |
| Zn1—O4 | 2.045 (4) | C5—C6 | 1.413 (7) |
| Zn1—N1 | 2.134 (4) | C7—C8 | 1.369 (8) |
| O1—C1 | 1.328 (6) | C7—H7 | 0.9500 |
| O1—Zn1 ⁱ | 2.092 (3) | C8—C9 | 1.431 (7) |
| O2—C11 | 1.277 (7) | C8—H8 | 0.9500 |
| O3—C11 | 1.250 (7) | C9—C10 | 1.497 (7) |
| O4—C13 | 1.423 (6) | C10—H10A | 0.9800 |
| O4—H4 | 0.8400 | C10—H10B | 0.9800 |
| N1—C9 | 1.319 (7) | C10—H10C | 0.9800 |
| N1—C6 | 1.366 (7) | C11—C12 | 1.508 (8) |
| C1—C2 | 1.381 (7) | C12—H12A | 0.9800 |
| C1—C6 | 1.435 (7) | C12—H12B | 0.9800 |
| C2—C3 | 1.412 (7) | C12—H12C | 0.9800 |
| C2—H2 | 0.9500 | C13—H13A | 0.9800 |
| C3—C4 | 1.366 (9) | C13—H13B | 0.9800 |
| C3—H3 | 0.9500 | C13—H13C | 0.9800 |
| O1—Zn1—O1 ⁱ | 75.2 (2) | C6—C5—C4 | 119.1 (5) |
| O1—Zn1—O2 | 142.5 (2) | N1—C6—C5 | 122.8 (5) |
| O1—Zn1—O4 | 114.7 (2) | N1—C6—C1 | 116.8 (4) |
| O1—Zn1—N1 | 79.8 (2) | C5—C6—C1 | 120.4 (5) |
| O1 ⁱ —Zn1—O2 | 95.8 (2) | C8—C7—C5 | 120.2 (5) |
| O1 ⁱ —Zn1—O4 | 94.5 (2) | C8—C7—H7 | 119.9 |
| O1 ⁱ —Zn1—N1 | 155.0 (2) | C5—C7—H7 | 119.9 |
| O2—Zn1—O4 | 102.1 (2) | C7—C8—C9 | 119.9 (5) |
| O2—Zn1—N1 | 104.7 (2) | C7—C8—H8 | 120.1 |
| O4—Zn1—N1 | 95.0 (2) | C9—C8—H8 | 120.1 |
| C1—O1—Zn1 | 116.2 (3) | N1—C9—C8 | 120.7 (5) |
| C1—O1—Zn1 ⁱ | 139.0 (3) | N1—C9—C10 | 119.2 (5) |
| Zn1—O1—Zn1 ⁱ | 104.81 (16) | C8—C9—C10 | 120.1 (5) |
| C11—O2—Zn1 | 116.0 (3) | C9—C10—H10A | 109.5 |
| C13—O4—Zn1 | 125.3 (3) | C9—C10—H10B | 109.5 |
| C13—O4—H4 | 117.3 | H10A—C10—H10B | 109.5 |
| Zn1—O4—H4 | 117.3 | C9—C10—H10C | 109.5 |
| C9—N1—C6 | 119.7 (4) | H10A—C10—H10C | 109.5 |
| C9—N1—Zn1 | 130.0 (4) | H10B—C10—H10C | 109.5 |
| C6—N1—Zn1 | 110.3 (3) | O3—C11—O2 | 123.5 (5) |
| O1—C1—C2 | 124.6 (5) | O3—C11—C12 | 120.0 (5) |
| O1—C1—C6 | 117.0 (5) | O2—C11—C12 | 116.5 (5) |
| C2—C1—C6 | 118.4 (5) | C11—C12—H12A | 109.5 |
| C1—C2—C3 | 120.8 (5) | C11—C12—H12B | 109.5 |
| C1—C2—H2 | 119.6 | H12A—C12—H12B | 109.5 |

| | | | |
|--|------------|----------------|------------|
| C3—C2—H2 | 119.6 | C11—C12—H12C | 109.5 |
| C4—C3—C2 | 121.2 (5) | H12A—C12—H12C | 109.5 |
| C4—C3—H3 | 119.4 | H12B—C12—H12C | 109.5 |
| C2—C3—H3 | 119.4 | O4—C13—H13A | 109.5 |
| C3—C4—C5 | 120.1 (5) | O4—C13—H13B | 109.5 |
| C3—C4—H4A | 119.9 | H13A—C13—H13B | 109.5 |
| C5—C4—H4A | 119.9 | O4—C13—H13C | 109.5 |
| C7—C5—C6 | 116.7 (5) | H13A—C13—H13C | 109.5 |
| C7—C5—C4 | 124.2 (5) | H13B—C13—H13C | 109.5 |
| O2—Zn1—O1—C1 | 102.0 (4) | C6—C1—C2—C3 | -1.3 (8) |
| O4—Zn1—O1—C1 | -89.7 (4) | C1—C2—C3—C4 | -0.5 (9) |
| O1 ⁱ —Zn1—O1—C1 | -177.9 (5) | C2—C3—C4—C5 | 0.9 (9) |
| N1—Zn1—O1—C1 | 1.1 (4) | C3—C4—C5—C7 | -178.6 (6) |
| O2—Zn1—O1—Zn1 ⁱ | -80.1 (3) | C3—C4—C5—C6 | 0.5 (8) |
| O4—Zn1—O1—Zn1 ⁱ | 88.17 (19) | C9—N1—C6—C5 | -0.4 (8) |
| O1 ⁱ —Zn1—O1—Zn1 ⁱ | 0.0 | Zn1—N1—C6—C5 | 178.2 (4) |
| N1—Zn1—O1—Zn1 ⁱ | 179.0 (2) | C9—N1—C6—C1 | -178.9 (5) |
| O1—Zn1—O2—C11 | 7.8 (5) | Zn1—N1—C6—C1 | -0.3 (6) |
| O4—Zn1—O2—C11 | -161.4 (4) | C7—C5—C6—N1 | -1.5 (8) |
| O1 ⁱ —Zn1—O2—C11 | -65.4 (4) | C4—C5—C6—N1 | 179.3 (5) |
| N1—Zn1—O2—C11 | 100.1 (4) | C7—C5—C6—C1 | 176.9 (5) |
| O2—Zn1—O4—C13 | 107.2 (4) | C4—C5—C6—C1 | -2.3 (8) |
| O1—Zn1—O4—C13 | -65.5 (5) | O1—C1—C6—N1 | 1.2 (7) |
| O1 ⁱ —Zn1—O4—C13 | 10.3 (4) | C2—C1—C6—N1 | -178.8 (5) |
| N1—Zn1—O4—C13 | -146.6 (4) | O1—C1—C6—C5 | -177.3 (5) |
| O2—Zn1—N1—C9 | 36.2 (5) | C2—C1—C6—C5 | 2.7 (8) |
| O1—Zn1—N1—C9 | 178.0 (5) | C6—C5—C7—C8 | 1.8 (8) |
| O4—Zn1—N1—C9 | -67.7 (5) | C4—C5—C7—C8 | -179.1 (5) |
| O1 ⁱ —Zn1—N1—C9 | -179.6 (4) | C5—C7—C8—C9 | -0.3 (8) |
| O2—Zn1—N1—C6 | -142.2 (3) | C6—N1—C9—C8 | 2.1 (8) |
| O1—Zn1—N1—C6 | -0.4 (3) | Zn1—N1—C9—C8 | -176.2 (4) |
| O4—Zn1—N1—C6 | 113.9 (3) | C6—N1—C9—C10 | -177.6 (5) |
| O1 ⁱ —Zn1—N1—C6 | 1.9 (6) | Zn1—N1—C9—C10 | 4.1 (8) |
| Zn1—O1—C1—C2 | 178.4 (4) | C7—C8—C9—N1 | -1.8 (8) |
| Zn1 ⁱ —O1—C1—C2 | 1.5 (9) | C7—C8—C9—C10 | 177.9 (5) |
| Zn1—O1—C1—C6 | -1.6 (6) | Zn1—O2—C11—O3 | -20.5 (7) |
| Zn1 ⁱ —O1—C1—C6 | -178.5 (4) | Zn1—O2—C11—C12 | 159.0 (4) |
| O1—C1—C2—C3 | 178.7 (5) | | |

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---------------------------------------|-------|-------------|-------------|---------------|
| O4—H4 ⁱⁱ —O3 ⁱⁱ | 0.84 | 1.88 | 2.602 (6) | 143 |

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

Fig. 1

