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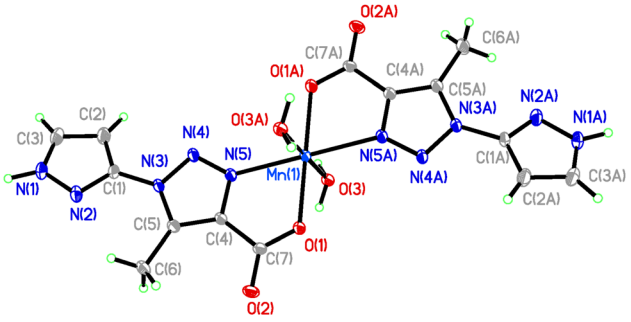
# Crystal structure of diaqua-bis[5-methyl-1-(1*H*-pyrazol-3-*yl*)-1*H*-1,2,3-triazole-4-carboxylato-$\kappa^2N,O$)]manganese(II), $C_{14}H_{16}MnN_{10}O_6$

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**Abstract**

$C_{14}H_{16}MnN_{10}O_6$, triclinic, $P\bar{1}$ (no. 2), $a$ = 6.7549(7) Å, $b$ = 7.1615 (7) Å, $c$ = 11.2405(10) Å, $\alpha$ = 77.624(8)°, $\beta$ = 73.524(9)°, $\gamma$ = 63.834 (10)°, $V$ = 465.48 (9) Å$^3$, $Z$ = 1, $R_{gt}(F)$ = 0.0339, $wR_{ref}(F^2)$ = 0.0860, $T$ = 293 K.

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The figure illustrates the crystal structure, while Table 1 encapsulates the crystallographic data, and Table 2 meticulously details the atomic composition, inclusive of atomic coordinates and displacement parameters.

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

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.28 × 0.25 × 0.22 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 0.77 mm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, |
| $\theta_{max}$, completeness: | 29.1°, >99 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 3,381, 2,121, 0.019 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 1,879 |
| $N(param)_{refined}$: | 143 |
| Programs: | CrysAlis$^{PRO}$,[1] SHELX[2, 3] |

## 1 Source of material

The necessary chemical reagents were procured from commercial vendors and directly employed in the experiment without undergoing any additional purification process. The ligand 5-methyl-1-(1*H*-pyrazol-3-*yl*)-1*H*-1,2,3-triazole-4-carboxylic acid was prepared according to the literature reported.[4–7] A certain amount of $MnCl_2{\cdot}4H_2O$ (0.0198 g, 0.1 mmol) and 5-methyl-1-(1*H*-pyrazol-3-*yl*)-1*H*-1,2,3-triazole-4-carboxylic acid (0.0386 g, 0.2 mmol) was dissolved in 15 mL MeCN, and the mixture was stirred at room temperature for 10 min. Following this, the homogeneous solution was transferred into a Teflon-lined autoclave (25 mL) and heated at 150 °C for 5 days. Following the heating process, the mixture was left to cool down naturally, reaching room temperature without any external intervention. Ultimately, colourless block crystals were isolated and retrieved through the filtration method.

## 2 Experimental details

The crystal structure was determined utilizing the SHELXT[2] program and subsequently refined with the latest SHELXL2014/7 software.[3] All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.95–0.97 Å, N–H = 0.94–0.97 Å and O–H = 0.92–0.93 Å). Their $U_{iso}$ values were set to 1.2 $U_{eq}$ or 1.5 $U_{eq}$ of the parent atoms.

## 3 Comment

With the ongoing exploration and investigation of metal-organic complexes crafted from *N*-heterocyclic carboxylic acid ligands, it has emerged that these complexes boast not only intricate and captivating topological structures

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**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Mn1 | 1.0000 | 0.0000 | 0.0000 | 0.02515 (13) |
| C1 | 0.5552 (3) | 0.2311 (3) | 0.48956 (16) | 0.0264 (4) |
| C2 | 0.7181 (4) | 0.2027 (4) | 0.55226 (19) | 0.0389 (5) |
| H2 | 0.8686 | 0.1801 | 0.5194 | 0.047* |
| C3 | 0.6031 (4) | 0.2160 (4) | 0.67458 (19) | 0.0402 (5) |
| H3 | 0.6622 | 0.2031 | 0.7427 | 0.048* |
| C4 | 0.5632 (3) | 0.2388 (3) | 0.17150 (16) | 0.0231 (4) |
| C5 | 0.4316 (3) | 0.2906 (3) | 0.28752 (17) | 0.0249 (4) |
| C6 | 0.1826 (3) | 0.3888 (4) | 0.3312 (2) | 0.0425 (6) |
| H6A | 0.1420 | 0.3418 | 0.4175 | 0.064* |
| H6B | 0.1126 | 0.3500 | 0.2828 | 0.064* |
| H6C | 0.1320 | 0.5382 | 0.3213 | 0.064* |
| C7 | 0.5043 (3) | 0.2547 (3) | 0.05040 (17) | 0.0240 (4) |
| N1 | 0.3916 (3) | 0.2506 (3) | 0.67733 (15) | 0.0385 (4) |
| H1 | 0.2766 | 0.2667 | 0.7545 | 0.046* |
| N2 | 0.3557 (3) | 0.2594 (3) | 0.56367 (15) | 0.0376 (4) |
| N3 | 0.5857 (3) | 0.2270 (3) | 0.35973 (14) | 0.0253 (3) |
| N4 | 0.8011 (3) | 0.1388 (3) | 0.29333 (14) | 0.0295 (4) |
| N5 | 0.7846 (2) | 0.1471 (3) | 0.17926 (13) | 0.0264 (3) |
| O1 | 0.6651 (2) | 0.1631 (2) | −0.03514 (12) | 0.0302 (3) |
| O2 | 0.3029 (2) | 0.3551 (2) | 0.04163 (13) | 0.0344 (3) |
| O3 | 1.0710 (2) | 0.2801 (2) | −0.08331 (12) | 0.0285 (3) |
| H3A | 0.9489 | 0.4019 | −0.0822 | 0.043* |
| H3B | 1.1530 | 0.2923 | −0.0451 | 0.043* |

alongside a vast array of coordination patterns but also exhibit promising applications.[8]

Azole carboxylic acids, a subclass of *N*-heterocyclic carboxylic acid ligands, are fundamentally constructed by the interlinkage of imidazole, pyrazole, and other organic aromatic carboxylic acids. The inherent presence of N and O atoms in azole carboxylic acids hints at the likelihood of hydrogen bonding and $\pi\cdots\pi$ stacking interactions contributing to the intricate assembly of supramolecular networks during complex formation.[9,10] Mn(II) ions, renowned for their stability and half-filled outermost electron shell, can adopt various coordination modes (ranging from four- to eight-coordination), forming high-spin Mn(II) complexes.[11–13] Due to their exceptional magnetic properties, Mn(II) complexes are highly sought-after in the realm of molecular magnet research.[14]

In light of this, a novel Mn(II) complex $C_{14}H_{16}MnN_{10}O_6$ was synthesized through the reaction between 5-methyl-1-(1*H*-pyrazol-3-*yl*)-1*H*-1,2,3-triazole-4-carboxylic acid and $MnCl_2\cdot 4H_2O$, with a concerted effort to cultivate single crystals.

The molecule structure of $C_{14}H_{16}MnN_{10}O_6$ consists of one Mn(II) ion, two 5-methyl-1-(1*H*-pyrazol-3-*yl*)-1*H*-1,2,3-triazole-4-carboxylate ligands, and two coordinated water molecules, which displays monomeric structure. The ligand 5-methyl-1-(1*H*-pyrazol-3-*yl*)-1*H*-1,2,3-triazole-4-carboxylate forms five-membered rings expressed by Mn–N–C–C–O which displays a irregular pentagon. In this pentagon conformation, the sum of interior angles is almost close to 540°. The Mn(II) ion is coordinated with two N atoms and four O atoms forming a distorted octahedron. The Mn(II) is in the centre of this octahedron with the Mn–N and Mn–O distances are 2.2765 (15) Å and 2.1484 (13)–2.2175 (14) Å, the angles of N–Mn–N, O–Mn–O and O–Mn–N are 180.0°, 89.80 (5)–180.0° and 75.66 (5)–104.34 (5)°, respectively.[15]

The presence of coordinated water molecules within the complex's formula serves a crucial function in stabilizing its entire structure. In the crystal structure, there exists a hydrogen bond O–H···N linking the title complex into a three-dimensional structure.

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**Conflict of interest:** The author hereby declares that there are no conflicts of interest pertaining to this article.

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