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The crystal structure of hexaquamagnesium(II) bis (3,4-dinitro-1H-pyrazol-1-ide), $C_6H_{14}MgN_8O_{14}$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.08 × 0.05 × 0.03 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.20 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$\theta_{\max}$, completeness:	27.5°, 99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5219, 1907, 0.032
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1585
$N(\text{param})_{\text{refined}}$:	144
Programs:	Bruker [1], SHELX [2, 3]

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Abstract

$C_6H_{14}MgN_8O_{14}$, monoclinic, $P\bar{1}$ (no. 2), $a = 6.1154(3)$ Å, $b = 7.9529(4)$ Å, $c = 9.0459(5)$ Å, $\alpha = 77.744(2)^\circ$, $\beta = 82.589(2)^\circ$, $\gamma = 81.134(3)^\circ$, $V = 422.67(4)$ Å³, $Z = 1$, $R_{\text{gt}}(F) = 0.0454$, $wR_{\text{ref}}(F^2) = 0.1112$, $T = 150$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

An amount of 0.80 g (0.02 mol) of NaOH was dissolved in 20 mL of water and added to a 100 mL stirred four-necked flask, and the temperature was raised to 323 K; then 3.22 g of 3,4-dinitro-1H-pyrazole (0.02 mol) was added, and the solution turned yellow. After 15 minutes of reaction at 323 K, a 1 mol/L aqueous solution of magnesium chloride

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2710(3)	0.1697(3)	0.4541(2)	0.0180(4)
H1	0.146708	0.118351	0.440068	0.022*
C2	0.3458(3)	0.1654(2)	0.5936(2)	0.0164(4)
C3	0.5296(3)	0.2575(2)	0.5543(2)	0.0146(4)
Mg1	0.000000	0.500000	1.000000	0.0151(2)
N1	0.6724(3)	0.3094(2)	0.64597(19)	0.0173(4)
N2	0.2555(3)	0.0712(2)	0.7345(2)	0.0211(4)
N3	0.5609(3)	0.3119(2)	0.40470(19)	0.0168(4)
N4	0.3998(3)	0.2566(2)	0.34256(19)	0.0186(4)
O1	0.3585(3)	0.0396(3)	0.84619(19)	0.0426(5)
O2	0.0746(3)	0.0189(2)	0.73468(18)	0.0285(4)
O3	0.6203(3)	0.2903(2)	0.78291(18)	0.0341(4)
O4	0.8370(2)	0.3756(2)	0.58192(17)	0.0221(3)
O5	0.0939(3)	0.5185(2)	0.77102(17)	0.0247(4)
H5A	−0.005070	0.481037	0.729455	0.037*
H5B	0.193(5)	0.568(4)	0.719(4)	0.044(9)*
O6	0.0889(4)	0.7394(2)	0.9901(2)	0.0436(5)
H6A	0.109415	0.794104	0.894639	0.065*
H6B	0.220977	0.728654	1.023288	0.065*
O7	−0.3164(3)	0.6131(3)	0.95056(19)	0.0396(5)
H7A	−0.366998	0.687268	1.009801	0.059*
H7B	−0.353(7)	0.660(5)	0.862(5)	0.072(12)*

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hexahydrate (2.03 g, 0.01 L) was added dropwise, and the reaction was carried out for 2 h at 323 K. The solution turned dark yellow, and the reaction solution was slowly evaporated

at room temperature in a quiet environment to obtain a transparent block crystal.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms. All the non-hydrogen atoms were refined anisotropically.

Comment

The nitrogen heterocyclic compound in general has a high formation enthalpy due to a large amount of N–N, C–N, N=N, C=N bonds in the structure. Pyrazoles are one of the important classes of compounds [4–7]. These compounds can be widely used in medicine, pesticides, photosensitive materials, fine chemicals [8–10].

As shown in the figure, the crystal structure of the title compound consists of a hexaqua complex and two organic counter cations. Compared with potassium 3,4-dinitropyrazolate dihydrate and sodium 3,4-dinitropyrazolate dihydrate [11], potassium and sodium do not form a coordination with water. It was owing to potassium and sodium cannot provide lone pair electrons. The ring of the organic anion is planar. The least-squares has angles to the plane through the five atoms (C1, C2, C3, N3, N4) and angles to the two nitro groups of 14.2° (O1, N2, O2) and 9.2° (O3, N1, O4). These findings are in total accord with the literature [12].

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