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The crystal structure of hexakis(1-ethylimidazole- κ^1N)nickel(II) dichloride – 1-ethylimidazole (1/2), $C_{40}H_{64}Cl_2NiN_{16}$

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Abstract

$C_{40}H_{64}Cl_2NiN_{16}$, monoclinic, $P2_1/c$ (no. 14), $a = 8.2320(9)$ Å, $b = 16.2902(16)$ Å, $c = 17.3843(17)$ Å, $\alpha = 90^\circ$, $\beta = 101.773(3)^\circ$, $\gamma = 90^\circ$, $V = 2282.2(4)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0448$, $wR_{ref}(F^2) = 0.1181$, $T = 298(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Green block
Size:	0.21 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.59 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX II, φ and ω
θ_{max} , completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	11,230, 4011, 0.041
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 3098
$N(param)_{refined}$:	292
Programs:	Bruker [1], Shelx [2]

Source of material

In a 50 mL flask, nickel(II) chloride hexahydrate (0.5 mmol) and 1-ethylimidazole (20 mmol) were successively added to 15 mL H₂O, and then stirred for 12 h at 60 °C. After cooling down to room temperature, the filtrate was left to slowly evaporate at room temperature for five days, and then the green block crystals were filtered off. Yield: 37.6%. **Anal.** **Calcd.** for $C_{40}H_{64}Cl_2NiN_{16}$: C, 53.46; H, 7.18; N, 24.94; found: C, 53.37; H, 7.24; N, 24.88.

Experimental details

Hydrogen atoms were assigned with common isotropic displacement factors $U_{iso}(H) = 1.2$ times $U_{eq}(C, \text{imidazole ring and methylene})$ and $U_{iso}(H) = 1.5$ times $U_{eq}(C, \text{methyl carbon})$. All the H atoms were refined as riding on their parent atom.

Comment

Transition metal complexes based on imidazole derivatives as solvent, base and ligands are attractive owing to their structural diversity and potential applications in the fields of superoxide dismutase activities, and catalysis, and so on [3–5]. The combination of versatile imidazole ligands and various nickel salts result in Ni complexes which provide interesting properties and thus

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
Ni1	0.500000	0.500000	0.500000	0.03136 (15)
N1	0.2053 (3)	0.29588 (15)	0.54248 (15)	0.0470 (6)
N2	0.3789 (3)	0.39457 (13)	0.53303 (12)	0.0362 (5)
N3	0.8888 (3)	0.47453 (16)	0.70334 (14)	0.0463 (6)
N4	0.7024 (3)	0.47330 (14)	0.59449 (13)	0.0366 (5)
N5	0.3719 (3)	0.56810 (14)	0.57269 (13)	0.0371 (5)
N6	0.2934 (3)	0.66329 (15)	0.64528 (15)	0.0506 (7)
N7	0.5281 (4)	0.4166 (2)	0.9152 (2)	0.0842 (10)
N8	0.6889 (5)	0.3902 (3)	1.0279 (2)	0.0995 (12)
Cl1	0.17660 (11)	0.22856 (5)	0.79457 (5)	0.0570 (2)
C1	0.2666 (4)	0.34578 (17)	0.49480 (17)	0.0411 (7)
H1	0.232647	0.345625	0.440387	0.049*
C2	0.2830 (4)	0.3140 (2)	0.61627 (18)	0.0516 (8)
H2	0.266012	0.289495	0.662327	0.062*
C3	0.3897 (4)	0.37438 (19)	0.60995 (17)	0.0475 (7)
H3	0.461041	0.399009	0.652007	0.057*
C4	0.0749 (5)	0.2346 (2)	0.5180 (3)	0.0735 (11)
H4A	-0.026658	0.253547	0.532328	0.088*
H4B	0.053943	0.228931	0.461314	0.088*
C5	0.1194 (6)	0.1549 (3)	0.5540 (3)	0.1102 (17)
H5A	0.221588	0.136634	0.541223	0.165*
H5B	0.033154	0.116055	0.534405	0.165*
H5C	0.132738	0.159455	0.609958	0.165*
C6	0.2123 (4)	0.55357 (19)	0.58118 (17)	0.0459 (7)
H6	0.147315	0.509417	0.559350	0.055*
C7	0.4154 (4)	0.63492 (18)	0.61263 (17)	0.0441 (7)
H7	0.518776	0.659785	0.617747	0.053*
C8	0.1642 (4)	0.6119 (2)	0.62524 (18)	0.0503 (8)
H8	0.061430	0.616291	0.639337	0.060*
C9	0.3041 (6)	0.7350 (2)	0.6977 (3)	0.0834 (12)
H9A	0.397467	0.768702	0.691075	0.100*
H9B	0.204209	0.767630	0.682394	0.100*
C10	0.3234 (8)	0.7134 (4)	0.7774 (3)	0.136 (2)
H10A	0.226720	0.684251	0.785257	0.204*
H10B	0.337137	0.762237	0.809024	0.204*
H10C	0.419380	0.679065	0.792390	0.204*
C11	0.7871 (4)	0.40103 (19)	0.60245 (18)	0.0517 (8)
H11	0.768481	0.357902	0.566676	0.062*
C12	0.7687 (4)	0.51571 (17)	0.65639 (16)	0.0404 (7)
H12	0.735857	0.568554	0.666555	0.048*
C13	0.9005 (4)	0.4013 (2)	0.66907 (19)	0.0552 (8)
H13	0.973658	0.359023	0.688213	0.066*
C14	0.9922 (5)	0.5058 (2)	0.7755 (2)	0.0713 (11)
H14A	0.950665	0.559091	0.787233	0.086*
H14B	1.104413	0.513318	0.767324	0.086*
C15	0.9964 (6)	0.4509 (3)	0.8434 (2)	0.0885 (13)
H15A	0.885740	0.443109	0.851776	0.133*
H15B	1.063969	0.474946	0.889384	0.133*
H15C	1.042250	0.398763	0.833116	0.133*
C16	0.4792 (6)	0.3453 (3)	0.9393 (3)	0.0846 (12)
H16	0.392411	0.312895	0.913079	0.102*
C17	0.5758 (7)	0.3295 (3)	1.0066 (3)	0.0883 (12)
H17	0.568406	0.282651	1.036289	0.106*
C18	0.6547 (6)	0.4417 (3)	0.9709 (3)	0.1005 (14)
H18	0.711451	0.490681	0.968728	0.121*

Table 2: (continued)

Atom	x	y	z	U_{iso}^*/U_{eq}
C19 ^a	0.4674 (18)	0.4329 (7)	0.8142 (9)	0.099 (3)
H19A ^a	0.536765	0.403915	0.784286	0.118*
H19B ^a	0.351506	0.419913	0.794191	0.118*
C20 ^a	0.500 (2)	0.5226 (7)	0.8181 (12)	0.131 (5)
H20A ^a	0.611942	0.532510	0.844907	0.197*
H20B ^a	0.424826	0.548762	0.846220	0.197*
H20C ^a	0.483002	0.544571	0.765880	0.197*
C19 ^b	0.4373 (12)	0.4659 (8)	0.8567 (6)	0.097 (3)
H19C ^b	0.411254	0.516884	0.880361	0.116*
H19D ^b	0.333138	0.438620	0.835292	0.116*
C20 ^b	0.5187 (17)	0.4855 (10)	0.7923 (6)	0.109 (4)
H20D ^b	0.590027	0.532066	0.806259	0.163*
H20E ^b	0.436598	0.497956	0.746141	0.163*
H20F ^b	0.583483	0.439251	0.781999	0.163*

^aOccupancy: 0.453(11), ^boccupancy: 0.547(11).

the synthesis of new functionalized imidazole-based Ni complexes is highly desirable [6, 7]. For example, Cetinkaya and the coworker described the synthesis and crystal structures of three new cyano-bridged tetracyanonickelate(II) complexes with 1-ethylimidazole ligand [8]. Lately, Dou et al. [9] reported the 1-ethylimidazole-based Ni-polyoxovanadates $[\text{Ni}(\text{1-eIm})(\text{H}_2\text{O})_4]_2(\text{V}_{10}\text{O}_{28})(\text{1-eImH})_2 \cdot 2\text{H}_2\text{O}$ with excellent heterogeneous solvent-free catalytic activity in Knoevenagel condensation at mild reaction conditions. Some efforts involving 1-ethylimidazole-decorated nickel complexes have been reported. Newly designed 1-ethylimidazole-coordinated Ni complexes are long-sought-after yet still unmet.

The asymmetric unit of the title crystal structure is shown in the figure. Single crystal X-ray diffraction analysis reveals that the asymmetric unit of the nickel complex structure consist of one half of a nickel(II) cation, three coordinated 1-ethylimidazole organic ligands, one free 1-ethylimidazole molecule and one chloride anion. In the crystal structure, the Ni(II) cation is coordinated by six N atoms from 1-ethylimidazole molecules and displayed the hexa-coordinated mode. The Ni–N bond lengths are within the range of 2.118(2) and 2.132(2) Å, which are in agreement with those of the reported hexakis(1-ethyl-1*H*-imidazole-*N*₃)-nickel(II) diperchlorate and hexakis(1-ethyl-1*H*-imidazole)-nickel(II) bis(bis(trifluoromethanesulfonyl) amide) [10, 11]; and similar to related metal complexes [12]. Meanwhile, the N(5)–Ni(1)–N(2), N(5)–Ni(1)–N(4) and N(2)–Ni(1)–N(4) bond angles are 86.78(8)°, 93.05(8)°, and 87.87(8)°, respectively, which suggested that Ni adopts a distorted octahedral geometry. Furthermore, C19 and C20 atoms of the title complex are disordered and both corresponding occupancies of (C19, C19[′]) and (C20, C20[′]) were (0.453, 0.547).

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