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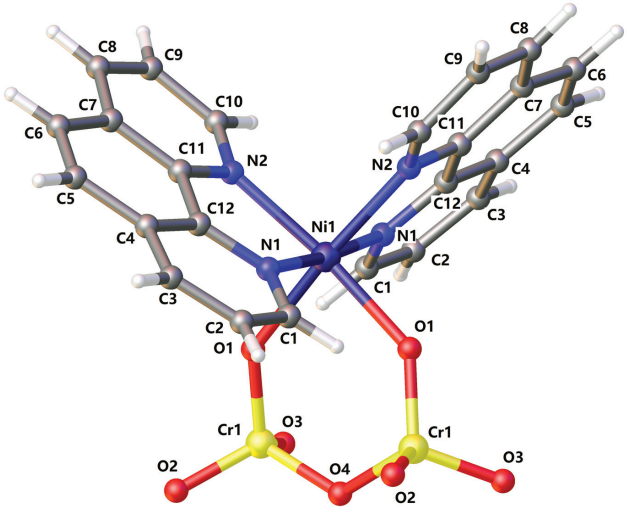
The crystal structure of (dichromato-κ²*O*,*O*′)bis(1,10-phenanthroline-κ²*N*,*N*′)nickel(II), $C_{12}H_{16}N_4O_7Cr_2Ni$



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Abstract

$C_{12}H_{16}N_4O_7Cr_2Ni$, orthorhombic, *Pbcn* (no. 60), $a = 13.7804(5)$ Å, $b = 10.3110(5)$ Å, $c = 16.4118(6)$ Å, $V = 2331.96(17)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0438$, $wR_{ref}(F^2) = 0.1111$, $T = 293(2)$ K.

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The molecular 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.

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Table 1: Data collection and handling.

Crystal:	Red block
Size:	0.25 × 0.15 × 0.15 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.77 mm^{-1}
Diffractometer, scan mode:	Rigaku R-axis Spider, ω
θ_{max}, completeness:	29.2°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	6978, 2753, 0.031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2186
$N(param)_{refined}$:	173
Programs:	CrysAlis^{PRO} [1], Olex2 [2], Il Milione [3], SHELX [4]

Source of material

A mixture containing $K_2Cr_2O_7$ (0.930 g), 1,10-phenanthroline monohydrate (0.640 g), $Ni_2SO_4 \cdot 6H_2O$ (0.310 g) and deionized water (H_2O) (13.0 mL) was prepared by mixing these components and sealed in a 25 mL Teflon-lined stainless steel autoclave (65% of the total volume of the autoclave). Then, the resulting slurry was heated to 433 K in an oven and maintained at the temperature for a week. Then, the red crystals of the title compound (about 50% yield based on Cr) were obtained.

Experimental details

The hydrogen atoms were placed at calculated positions with the SHELX program.

Comment

At present, the studies on organic-inorganic hybrid compounds have been attracting considerable concerns for the diversity of structure and its advantages of performance, such as excellent biocompatibility, flexibility, long life, mechanical properties [5–7]. Organic functional groups can be distributed evenly in the structure of the inorganic matrix, being in favour of developing the physicochemical properties of hybrid materials [8, 9]. Meanwhile, hybrid organic-inorganic monolith has also attracted attention as a potential material with good thermal stability etc. [10]. Therefore, organic-inorganic hybrid materials may be used in many areas because they are easy

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Ni1	0.5000	0.61861(5)	0.7500	0.02363(16)
Cr1	0.56435(4)	0.92105(5)	0.83073(3)	0.02935(16)
O1	0.67825(19)	0.9422(3)	0.81649(19)	0.0662(8)
O2	0.5351(2)	0.9850(3)	0.91573(14)	0.0626(8)
O3	0.5000	0.9966(3)	0.7500	0.0443(9)
O4	0.53769(16)	0.7658(2)	0.83101(10)	0.0321(5)
N1	0.63929(17)	0.6104(2)	0.70156(14)	0.0262(5)
N2	0.48005(17)	0.4791(3)	0.65990(14)	0.0272(6)
C1	0.7190(2)	0.6742(3)	0.72425(19)	0.0356(7)
H1	0.7158	0.7274	0.7699	0.043*
C2	0.8069(2)	0.6648(4)	0.6828(2)	0.0415(8)
H2	0.8609	0.7105	0.7009	0.050*
C3	0.8130(2)	0.5877(4)	0.6150(2)	0.0417(9)
H3	0.8710	0.5820	0.5862	0.050*
C4	0.7313(2)	0.5172(3)	0.58913(17)	0.0341(7)
C5	0.7308(3)	0.4336(4)	0.5201(2)	0.0440(9)
H5	0.7862	0.4270	0.4879	0.053*
C6	0.6513(3)	0.3637(4)	0.5006(2)	0.0443(9)
H6	0.6534	0.3084	0.4559	0.053*
C7	0.5634(2)	0.3727(3)	0.54727(18)	0.0344(7)
C8	0.4794(3)	0.3007(4)	0.5316(2)	0.0436(9)
H8	0.4780	0.2409	0.4892	0.052*
C9	0.3997(3)	0.3194(4)	0.5794(2)	0.0457(9)
H9	0.3436	0.2717	0.5698	0.055*
C10	0.4019(2)	0.4101(3)	0.6428(2)	0.0373(8)
H10	0.3463	0.4223	0.6740	0.045*
C11	0.5608(2)	0.4600(3)	0.61338(16)	0.0267(6)
C12	0.6457(2)	0.5320(3)	0.63496(16)	0.0259(6)

to process and are amenable to design on the molecular scale [11].

Herein, we report a new organic-inorganic hybrid compound, $[Ni(phen)_2]Cr_2O_7$ (phen = 1,10-phenanthroline), i.e. a inorganic complex dichromate anion ($[Cr^{VI}{}_2O_7]^{2-}$) coordinated to a metal-organic coordination ion ($[Ni^{II}(phen)_2]^{2+}$) by two oxygen atoms. The four-coordinated Cr atoms show the bond distances of 1.642(3) Å and 1.7751(18) Å for bridging oxygen atoms (Cr—O_b) and the bond distances of 1.596(3) Å and 1.604(3) Å for terminal oxygen atoms (Cr—O_t). The O—Cr—O bond angles are in the range of 108.54(19)–110.67(16)°. In the six-coordinated Ni(II):$[Ni(N_4O_2)]$. The Ni—O bond length is 2.084 Å and Ni—N bond lengths are 2.078(3) Å and 2.082(3) Å. The O/N—Ni—O/N bond angles are within the normal range of 79.75(10)–175.28(14)°. Bond valence sum calculations [12] on the Cr and Ni sites afford values of 5.94 and 2.27.

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