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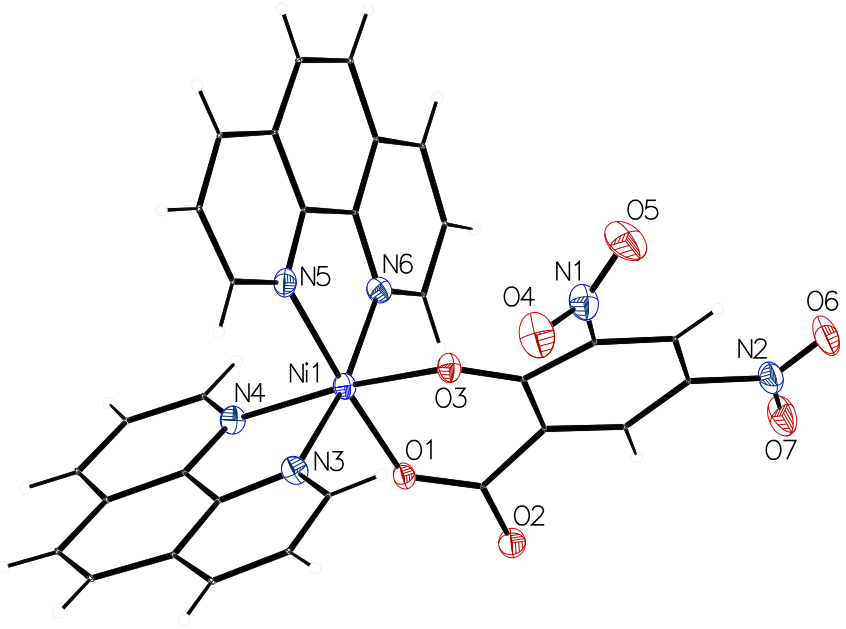
# Crystal structure of (bis(1,10-phenanthroline-$\kappa^2N,N'$))-(3,5-dinitrosalicylato-$\kappa^2O,O'$)nickel(II), $C_{31}H_{18}N_6NiO_7$

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**Abstract**

$C_{31}H_{18}N_6NiO_7$, monoclinic, $P2_1/c$ (no. 14), $a$ = 7.6619(15) Å, $b$ = 18.470(4) Å, $c$ = 19.113(4) Å, $\beta$ = 90.53(3)°, $V$ = 2704.6(9) Å$^3$, $Z$ = 4, $R_{gt}(F)$ = 0.0608, w$R_{ref}(F^2)$ = 0.1656, $T$ = 293 K.

**CCDC no.:** 1575939

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.

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

| | |
|---|---|
| Crystal: | Green block |
| Size: | 0.29 × 0.26 × 0.25 mm |
| Wavelength: | Mo $K\alpha$ radiation (0.71073 Å) |
| $\mu$: | 0.78 mm$^{-1}$ |
| Diffractometer, scan mode: | Rigaku R-AXIS RAPID IP, $\omega$ |
| $\theta_{max}$, completeness: | 27.5°, 99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 23970, 6113, 0.068 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 3799 |
| $N(param)_{refined}$: | 406 |
| Programs: | Rigaku [1], SHELX [2], Olex2 [3] |

## Source of material

A mixture of $Ni(CH_3COO)_2$ (0.1 mmol), phenanthroline (0.1 mmol), 3,5-dinitrosalicylic acid (0.2 mmol) and distilled water (10 mL) was put into a Teflon-lined autoclave (20 mL) and then heated at 150 °C for 72 h. Green block-like crystals of (I) were collected from the reaction mixture.

## Experimental details

The H atoms were positioned geometrically and were included in the refinement in the riding-model approximation [C–H = 0.93 Å and $U_{iso}$(H) = 1.2 $U_{eq}$(C)].

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Ni1 | 0.35079 (6) | 0.31838 (3) | 0.61711 (2) | 0.04189 (17) |
| O1 | 0.2001 (3) | 0.40312 (15) | 0.63745 (13) | 0.0488 (6) |
| O2 | 0.0553 (4) | 0.48007 (18) | 0.70211 (16) | 0.0732 (9) |
| O3 | 0.4681 (3) | 0.32986 (15) | 0.71195 (14) | 0.0500 (7) |
| O4 | 0.7456 (5) | 0.3292 (3) | 0.8049 (2) | 0.1003 (14) |
| O5 | 0.6540 (5) | 0.2695 (3) | 0.8922 (2) | 0.1174 (17) |
| O6 | 0.1596 (5) | 0.3831 (2) | 1.00660 (17) | 0.0943 (13) |
| O7 | −0.0331 (5) | 0.4350 (2) | 0.94308 (19) | 0.0929 (12) |
| N1 | 0.6293 (5) | 0.3103 (3) | 0.8437 (2) | 0.0733 (12) |
| N2 | 0.1057 (5) | 0.4040 (2) | 0.9497 (2) | 0.0661 (10) |
| N3 | 0.5393 (4) | 0.38084 (19) | 0.56876 (17) | 0.0503 (8) |
| N4 | 0.2553 (4) | 0.31852 (18) | 0.51439 (17) | 0.0473 (8) |
| N5 | 0.4861 (4) | 0.2219 (2) | 0.59916 (17) | 0.0514 (8) |
| N6 | 0.1742 (4) | 0.24259 (18) | 0.65661 (17) | 0.0492 (8) |
| C1 | 0.1569 (5) | 0.4286 (2) | 0.6959 (2) | 0.0453 (9) |
| C2 | 0.2329 (4) | 0.3972 (2) | 0.76307 (19) | 0.0411 (8) |
| C3 | 0.3905 (5) | 0.3546 (2) | 0.7649 (2) | 0.0440 (8) |
| C4 | 0.4557 (5) | 0.3419 (2) | 0.8344 (2) | 0.0524 (10) |
| C5 | 0.3656 (5) | 0.3553 (3) | 0.8948 (2) | 0.0566 (11) |
| H5 | 0.4095 | 0.3415 | 0.9383 | 0.068* |
| C6 | 0.2076 (5) | 0.3901 (2) | 0.8883 (2) | 0.0507 (10) |
| C7 | 0.1450 (5) | 0.4120 (2) | 0.8234 (2) | 0.0471 (9) |
| H7 | 0.0403 | 0.4374 | 0.8208 | 0.057* |
| C8 | 0.6768 (5) | 0.4126 (3) | 0.5982 (3) | 0.0628 (12) |
| H8 | 0.6939 | 0.4078 | 0.6462 | 0.075* |
| C9 | 0.7954 (6) | 0.4524 (3) | 0.5603 (3) | 0.0834 (17) |
| H9 | 0.8897 | 0.4745 | 0.5825 | 0.100* |
| C10 | 0.7726 (7) | 0.4587 (3) | 0.4902 (3) | 0.0909 (18) |
| H10 | 0.8512 | 0.4856 | 0.4640 | 0.109* |
| C11 | 0.6305 (6) | 0.4249 (3) | 0.4574 (3) | 0.0711 (14) |
| C12 | 0.6000 (9) | 0.4247 (4) | 0.3839 (3) | 0.105 (2) |
| H12 | 0.6795 | 0.4474 | 0.3547 | 0.126* |
| C13 | 0.4568 (10) | 0.3917 (4) | 0.3552 (3) | 0.116 (3) |
| H13 | 0.4406 | 0.3917 | 0.3069 | 0.140* |
| C14 | 0.3316 (7) | 0.3573 (3) | 0.3991 (3) | 0.0819 (16) |
| C15 | 0.1778 (8) | 0.3238 (4) | 0.3737 (3) | 0.103 (2) |
| H15 | 0.1519 | 0.3246 | 0.3261 | 0.124* |
| C16 | 0.0687 (7) | 0.2905 (3) | 0.4183 (3) | 0.0892 (18) |
| H16 | −0.0340 | 0.2692 | 0.4022 | 0.107* |
| C17 | 0.1126 (5) | 0.2888 (3) | 0.4882 (2) | 0.0607 (11) |
| H17 | 0.0374 | 0.2654 | 0.5187 | 0.073* |
| C18 | 0.3631 (5) | 0.3531 (3) | 0.4696 (2) | 0.0549 (10) |
| C19 | 0.5150 (5) | 0.3878 (2) | 0.4994 (2) | 0.0516 (10) |
| C20 | 0.6363 (6) | 0.2122 (3) | 0.5653 (2) | 0.0663 (13) |
| H20 | 0.6913 | 0.2523 | 0.5460 | 0.080* |
| C21 | 0.7126 (7) | 0.1456 (4) | 0.5580 (3) | 0.0860 (17) |
| H21 | 0.8156 | 0.1407 | 0.5330 | 0.103* |
| C22 | 0.6380 (8) | 0.0879 (4) | 0.5868 (4) | 0.0951 (19) |
| H22 | 0.6902 | 0.0427 | 0.5825 | 0.114* |
| C23 | 0.4827 (7) | 0.0947 (3) | 0.6232 (3) | 0.0743 (14) |
| C24 | 0.4030 (10) | 0.0387 (4) | 0.6547 (4) | 0.106 (2) |
| H24 | 0.4562 | −0.0066 | 0.6548 | 0.127* |
| C25 | 0.2434 (10) | 0.0473 (3) | 0.6869 (4) | 0.102 (2) |
| H25 | 0.1932 | 0.0081 | 0.7096 | 0.122* |
| C26 | 0.1523 (7) | 0.1164 (3) | 0.6860 (2) | 0.0698 (13) |
| C27 | −0.0134 (7) | 0.1288 (3) | 0.7127 (3) | 0.0789 (15) |
| H27 | −0.0767 | 0.0910 | 0.7321 | 0.095* |
| C28 | −0.0825 (6) | 0.1972 (3) | 0.7103 (3) | 0.0719 (14) |
| H28 | −0.1942 | 0.2061 | 0.7269 | 0.086* |
| C29 | 0.0164 (5) | 0.2531 (3) | 0.6826 (2) | 0.0573 (11) |
| H29 | −0.0298 | 0.2996 | 0.6824 | 0.069* |
| C30 | 0.2406 (6) | 0.1744 (2) | 0.6575 (2) | 0.0547 (10) |
| C31 | 0.4094 (6) | 0.1638 (2) | 0.6260 (2) | 0.0538 (10) |

# Comment

Complexes of salicylic acid ($H_2$sal) and salicylates are of general interest [4, 5]. 1,10–Phenanthroline (phen) often acts as a chelating ligand due to its property. Many complexes with salicylates and phen were found displaying diverse structure types [6, 7]. The syntheses of metal-organic supramolecular assemblies employing organic ligands with $NO_2$ groups are limited [7]. Up to now, some complexes of the 3,5-dinitrosalicylate ligand with diverse architectures have been reported [8–10].

In the crystal structure of the title compound, Ni atom adopts an octahedral coordination geometry, defined by four N atoms of two phen ligands and two O atoms from one 3,5-$(NO_2)_2$ sal ligand. The Ni–O bond lengths are 1.987(3) and 2.026(3) Å, respectively. The Ni–O (phen) bond lengths are from 2.074(3) to 2.096(3) Å. The 3,5-$(NO_2)_2$ sal ligand is coordinated to Ni(II) via one oxygen atom of the ($O_2$) and one oxygen atom from the phenolate group ($O_3$). The adjacent mononuclear units are further connected to each other by C–H···O hydrogen bonds formed between the oxygen atoms of $NO_2$ groups and carboxylate groups of 3,5-$(NO_2)_2$ sal ligands and phenyl hydrogens of phen.

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**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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