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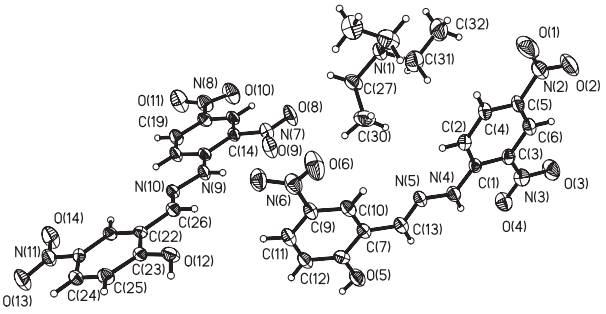
Crystal structure of (*E*)-2-((2-(2,4-dinitrophenyl) hydrazono)methyl)-4-nitrophenol — triethylamine (2/1), $C_{32}H_{33}N_{11}O_{14}$



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Abstract

$C_{32}H_{33}N_{11}O_{14}$, Monoclinic, $P2_1/c$ $a = 12.3339(18)$ Å, $b = 37.764(5)$ Å, $c = 8.2077(12)$ Å, $\alpha = 90°$, $\beta = 109.053(2)°$, $\gamma = 90°$, $V = 3613.6(9)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0621$, $wR_{ref}(F^2) = 0.1574$, $T = 296(2)$ K.

CCDC no.: 1446315

Table 1: Data collection and handling.

Crystal:	Red, block,
Size:	0.25×0.32×0.41 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.17 cm^{-1}
Diffractometer, scan mode:	Bruker SMART APEX, ϕ and ω scans
$2\theta_{max}$:	51°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	18974, 6644
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2654
$N(param)_{refined}$:	527
Programs:	SHELX [5, 6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(5)	4*e*	0.8907	0.1342	0.8754	0.114
H(12)	4*e*	1.0577	0.1506	−0.0041	0.090
H(2)	4*e*	0.4092	0.0305	0.3972	0.058
H(4)	4*e*	0.2417	0.0021	0.2563	0.064
H(6)	4*e*	0.1851	−0.0139	0.7037	0.066
H(10)	4*e*	0.6107	0.0793	0.3554	0.065
H(11)	4*e*	0.8420	0.1517	0.3291	0.074
H(12A)	4*e*	0.9136	0.1536	0.6262	0.074
H(13)	4*e*	0.6740	0.0794	0.8095	0.065
H(15)	4*e*	0.3955	0.2649	0.2478	0.061
H(16)	4*e*	0.7142	0.2961	0.1024	0.063
H(19)	4*e*	0.5987	0.3389	0.1518	0.072
H(20)	4*e*	0.9412	0.2762	0.0034	0.051
H(24)	4*e*	1.2187	0.2458	−0.1128	0.063
H(25)	4*e*	1.1653	0.1880	−0.1095	0.066
H(26)	4*e*	0.8331	0.1912	0.0458	0.056
H(27A)	4*e*	0.2869	0.1423	0.0584	0.085
H(27B)	4*e*	0.2708	0.1518	0.2345	0.085
H(28A)	4*e*	0.1214	0.1165	−0.2317	0.145
H(28B)	4*e*	0.1579	0.0782	−0.2668	0.145
H(28C)	4*e*	0.2504	0.1050	−0.1580	0.145
H(29A)	4*e*	0.0778	0.0733	−0.0485	0.106
H(29B)	4*e*	0.2096	0.0659	0.0335	0.106
H(30A)	4*e*	0.3749	0.1031	0.3628	0.161
H(30B)	4*e*	0.4474	0.1247	0.2720	0.161
H(30C)	4*e*	0.3833	0.0903	0.1856	0.161
H(31A)	4*e*	0.1908	0.0804	0.3173	0.110
H(31B)	4*e*	0.1527	0.1186	0.3506	0.110
H(32A)	4*e*	−0.0336	0.1058	0.1753	0.173
H(32B)	4*e*	0.0063	0.0796	0.3319	0.173
H(32C)	4*e*	0.0053	0.0673	0.1490	0.173
H(9A)	4*e*	0.683(3)	0.2092(9)	0.104(5)	0.03(1)
H(4A)	4*e*	0.517(4)	0.051(1)	0.833(5)	0.08(2)

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

A methanol solution (10 mL) containing 5-nitro-salicyl aldehyde (0.038 g, 0.2 mmol) and 2,4 (2,4-dinitrophenyl)hydranzine (0.037 g, 0.2 mmol) is mixed with

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.1626(3)	0.11296(9)	0.1062(5)	0.056(2)	0.046(2)	0.078(3)	−0.006(2)	0.030(2)	−0.001(2)
N(2)	4*e*	0.0908(4)	−0.0259(1)	0.3766(7)	0.066(3)	0.076(3)	0.089(4)	−0.024(2)	0.023(3)	−0.003(3)
N(3)	4*e*	0.3580(4)	0.0170(1)	0.9129(5)	0.068(3)	0.062(3)	0.060(3)	0.000(2)	0.030(2)	−0.004(2)
N(4)	4*e*	0.4984(3)	0.04651(9)	0.7162(5)	0.052(2)	0.056(2)	0.054(3)	−0.010(2)	0.023(2)	−0.001(2)
N(5)	4*e*	0.5552(3)	0.06121(9)	0.6134(4)	0.046(2)	0.046(2)	0.065(2)	−0.008(2)	0.026(2)	−0.002(2)
N(6)	4*e*	0.6719(4)	0.1144(1)	0.1297(6)	0.080(3)	0.092(4)	0.066(3)	−0.020(3)	0.040(3)	−0.012(3)
N(7)	4*e*	0.4938(4)	0.2087(1)	0.2030(5)	0.058(3)	0.059(3)	0.072(3)	−0.014(2)	0.032(2)	−0.004(2)
N(8)	4*e*	0.4147(4)	0.3330(1)	0.2442(6)	0.063(3)	0.069(3)	0.103(4)	0.004(3)	0.042(3)	−0.006(3)
N(9)	4*e*	0.6977(3)	0.2287(1)	0.1109(5)	0.045(2)	0.043(3)	0.071(3)	−0.006(2)	0.030(2)	0.002(2)
N(10)	4*e*	0.7911(3)	0.23933(9)	0.0662(4)	0.035(2)	0.058(2)	0.048(2)	−0.003(2)	0.017(2)	0.003(2)
N(11)	4*e*	1.1180(3)	0.3027(1)	−0.0595(5)	0.052(3)	0.056(3)	0.089(3)	−0.001(2)	0.036(2)	0.012(2)
O(1)	4*e*	0.0606(4)	−0.0268(1)	0.2203(6)	0.124(4)	0.178(5)	0.077(3)	−0.086(3)	0.008(3)	−0.014(3)
O(2)	4*e*	0.0354(3)	−0.0388(1)	0.4613(5)	0.084(3)	0.116(3)	0.122(3)	−0.052(2)	0.036(3)	−0.005(3)
O(3)	4*e*	0.2976(3)	0.0017(1)	0.9847(4)	0.091(3)	0.117(3)	0.078(2)	−0.023(2)	0.051(2)	0.005(2)
O(4)	4*e*	0.4417(3)	0.03491(9)	0.9957(4)	0.086(3)	0.081(2)	0.060(2)	−0.022(2)	0.027(2)	−0.014(2)
O(5)	4*e*	0.8368(3)	0.12036(9)	0.8417(4)	0.065(2)	0.082(3)	0.077(3)	−0.030(2)	0.020(2)	−0.005(2)
O(6)	4*e*	0.6100(4)	0.0896(1)	0.0642(5)	0.165(4)	0.141(4)	0.076(3)	−0.094(3)	0.050(3)	−0.039(3)
O(7)	4*e*	0.6946(3)	0.1388(1)	0.0468(4)	0.101(3)	0.102(3)	0.074(2)	−0.024(2)	0.046(2)	0.003(2)
O(8)	4*e*	0.4099(3)	0.20368(8)	0.2465(5)	0.068(2)	0.075(2)	0.133(3)	−0.016(2)	0.064(2)	0.007(2)
O(9)	4*e*	0.5529(3)	0.18428(9)	0.1767(5)	0.080(3)	0.054(2)	0.134(3)	−0.007(2)	0.066(2)	0.001(2)
O(10)	4*e*	0.3434(3)	0.3238(1)	0.3108(5)	0.087(3)	0.094(3)	0.144(4)	0.005(2)	0.080(3)	−0.012(2)
O(11)	4*e*	0.4290(3)	0.3636(1)	0.2087(6)	0.102(3)	0.062(3)	0.194(5)	0.008(2)	0.086(3)	−0.002(3)
O(12)	4*e*	0.9994(2)	0.16258(7)	−0.0236(4)	0.039(2)	0.049(2)	0.090(2)	0.001(1)	0.017(2)	0.001(2)
O(13)	4*e*	1.2100(3)	0.30911(8)	−0.0812(5)	0.070(2)	0.066(2)	0.148(3)	−0.008(2)	0.061(2)	0.011(2)
O(14)	4*e*	1.0532(3)	0.32612(8)	−0.0433(5)	0.086(3)	0.049(2)	0.173(4)	0.012(2)	0.077(3)	0.014(2)
C(1)	4*e*	0.4003(3)	0.0282(1)	0.6382(5)	0.043(3)	0.032(2)	0.058(3)	−0.001(2)	0.018(2)	0.003(2)
C(2)	4*e*	0.3638(4)	0.0223(1)	0.4600(5)	0.053(3)	0.045(3)	0.050(3)	−0.002(2)	0.022(2)	0.004(2)
C(3)	4*e*	0.3305(4)	0.0137(1)	0.7288(6)	0.054(3)	0.037(3)	0.055(3)	0.000(2)	0.024(2)	0.000(2)
C(4)	4*e*	0.2645(4)	0.0051(1)	0.3752(6)	0.061(3)	0.041(3)	0.061(3)	−0.010(2)	0.023(3)	0.001(2)
C(5)	4*e*	0.1979(4)	−0.0078(1)	0.4689(6)	0.055(3)	0.041(3)	0.069(3)	−0.014(2)	0.021(3)	0.000(2)
C(6)	4*e*	0.2299(4)	−0.0043(1)	0.6429(6)	0.056(3)	0.047(3)	0.070(4)	−0.007(2)	0.031(3)	0.002(3)
C(7)	4*e*	0.7072(3)	0.0969(1)	0.5891(6)	0.042(3)	0.045(3)	0.059(3)	−0.004(2)	0.021(2)	−0.004(2)
C(8)	4*e*	0.8008(4)	0.1190(1)	0.6693(6)	0.038(3)	0.055(3)	0.061(3)	−0.001(2)	0.012(2)	−0.007(3)
C(9)	4*e*	0.7191(4)	0.1154(1)	0.3159(6)	0.050(3)	0.055(3)	0.061(3)	−0.009(2)	0.030(3)	−0.005(3)
C(10)	4*e*	0.6698(4)	0.0948(1)	0.4113(6)	0.046(3)	0.046(3)	0.074(3)	−0.006(2)	0.025(3)	−0.011(3)
C(11)	4*e*	0.8097(4)	0.1378(1)	0.3948(7)	0.055(3)	0.060(3)	0.079(4)	−0.014(3)	0.033(3)	−0.002(3)
C(12)	4*e*	0.8513(4)	0.1391(1)	0.5715(7)	0.044(3)	0.058(3)	0.086(4)	−0.014(2)	0.026(3)	−0.004(3)
C(13)	4*e*	0.6463(4)	0.0785(1)	0.6897(6)	0.049(3)	0.055(3)	0.064(3)	−0.007(2)	0.026(2)	−0.005(2)
C(14)	4*e*	0.5283(3)	0.2448(1)	0.1855(5)	0.042(3)	0.054(3)	0.041(3)	−0.010(2)	0.019(2)	0.000(2)
C(15)	4*e*	0.4597(3)	0.2710(1)	0.2181(5)	0.038(3)	0.070(3)	0.051(3)	−0.008(2)	0.022(2)	−0.001(2)
C(16)	4*e*	0.6502(3)	0.2894(1)	0.1312(5)	0.042(3)	0.053(3)	0.071(3)	−0.007(2)	0.030(2)	0.003(2)
C(17)	4*e*	0.6276(3)	0.2533(1)	0.1418(5)	0.034(2)	0.055(3)	0.044(3)	−0.004(2)	0.017(2)	−0.003(2)
C(18)	4*e*	0.4865(4)	0.3057(1)	0.2067(6)	0.044(3)	0.059(3)	0.064(3)	0.003(2)	0.024(2)	−0.005(2)
C(19)	4*e*	0.5818(4)	0.3151(1)	0.1614(6)	0.057(3)	0.049(3)	0.081(3)	−0.006(2)	0.030(3)	0.001(3)
C(20)	4*e*	0.9864(3)	0.2581(1)	−0.0166(5)	0.035(2)	0.053(3)	0.044(3)	0.006(2)	0.018(2)	0.001(2)
C(21)	4*e*	1.0838(3)	0.2662(1)	−0.0554(5)	0.041(3)	0.042(3)	0.046(3)	−0.001(2)	0.017(2)	0.007(2)
C(22)	4*e*	0.9550(3)	0.2232(1)	−0.0072(5)	0.030(2)	0.045(3)	0.043(2)	0.002(2)	0.011(2)	0.003(2)
C(23)	4*e*	1.0242(3)	0.1959(1)	−0.0376(5)	0.037(2)	0.042(3)	0.050(3)	−0.004(2)	0.010(2)	0.003(2)
C(24)	4*e*	1.1525(3)	0.2400(1)	−0.0879(5)	0.042(3)	0.062(3)	0.060(3)	0.006(2)	0.025(2)	0.009(3)
C(25)	4*e*	1.1214(4)	0.2056(1)	−0.0827(5)	0.047(3)	0.056(3)	0.067(3)	0.008(2)	0.025(2)	−0.002(3)
C(26)	4*e*	0.8530(3)	0.2148(1)	0.0378(5)	0.042(3)	0.050(3)	0.048(3)	−0.005(2)	0.015(2)	−0.002(2)
C(27)	4*e*	0.2757(4)	0.1322(1)	0.1605(7)	0.051(3)	0.052(3)	0.101(4)	−0.005(2)	0.015(3)	−0.009(3)
C(28)	4*e*	0.1725(5)	0.0971(1)	−0.1844(7)	0.108(5)	0.091(4)	0.103(5)	−0.006(3)	0.053(4)	−0.019(4)
C(29)	4*e*	0.1533(4)	0.0840(1)	−0.0193(7)	0.075(4)	0.060(3)	0.122(5)	0.002(3)	0.024(4)	−0.025(4)
C(30)	4*e*	0.3797(4)	0.1106(1)	0.2536(8)	0.058(4)	0.085(4)	0.154(6)	0.007(3)	0.000(4)	0.019(4)
C(31)	4*e*	0.1385(5)	0.0996(1)	0.2666(7)	0.092(4)	0.072(4)	0.122(5)	0.017(3)	0.051(4)	0.014(4)
C(32)	4*e*	0.0185(5)	0.0870(2)	0.2272(8)	0.094(5)	0.130(6)	0.138(6)	−0.005(4)	0.060(4)	0.038(4)

an aqueous solution (10 mL) of $Zn(NO_3)_2{\cdot}6H_2O$, 0.2 mmol (0.061 g). After stirring for 20 min in air, the pH value was adjusted to 5.5, and the mixture was further stirred for 6 h at room temperature, then the mixture was cooled over a period of 12 h at a rate 5° C/h. Colorless crystals of title complex were obtained suitable for X-ray diffraction analysis. For title complex, yield: 0.0682 g (44°) based on 5-nitro-salicyl aldehyde. Elemental analysis (%): calcd for $C_{32}H_{31}N_{11}O_{14}$: C 48.43, H 3.93, N 19.41, found: C 48.72, H 3.90, N 19.54.

Experimental details

Positions of hydrogen atoms at N atoms were located from the difference Fourier syntheses and refined. All U_{iso} values were restrained on U_{eq} values of the parent atoms.

Discussion

It is well-known that Schiff base compounds have continued to enjoy extensive interests owing to their structural diversity and potential applications in pharmacology and catalysis [1, 2]. The chelating salicylaldehyde derivatives ligands containing O and N donor atoms show broad biological activity, especially with one or more halogen atoms added in the aromatic ring (e.g. antitumor, antibacterial and antifungal activities [3, 4]). Substituted Schiff base can be employed to construct functional covalent organic frameworks (COF). In order to develop environmentally benign methods, a new salicyl aldehyde-type Schiff base compound has been prepared through transition metal catalysis. The X-ray diffraction analysis reveals that title compound consists of three discrete molecules: two similar Schiff base and one triethylamine molecule. The Zn(II) cation is not present in the final product, this is also confirmed by the bond length of C–O linker by hydroxyl rather in ketone group form, which is just is 1.34 Å. The perspective view of the title compound with atom labeling is illustrated in the figure.

All the atoms of this Schiff base are nearly coplanar. For the first benzene ring of C1—C2—C3—C4—C5—C6, carbon atom C5 is beyond the least square plane at 0.0118 Å, while, the for second benzene ring of C7—C8—C9—C10—C11—C12, C7 atom is beyond the least square plane at just 0.0183 Å, and for the two Schiff base compounds the dihedral angles between the two benzene rings within same mioety are 1.59°, 13.59°, respectively. Interestingly, the nitro groups show different bond lengths although they displayed the same configuration in the Schiff base compound, in which the bond distance of N(2)—O(1), N(2)—O(2), N(3)—O(3), N(6)—O(6), N(6)—O(7) are found to be 1.210(5), 1.224(5), 1.234(4), 1.217(5), 1.232(5) Å, respectively. The triethylamine is involved in hydrogen bonding between the hydroxyl oxygen and tert-amine nitrogen, such as, O(12)—H(12)···N(1)#2 [O(12)—N(1)#2 = 2.702(5), O(12)—H(12)···N(1)#2 = 155.7°]. Among the three ethyl groups, only one terminal carbon extended along the opposite direction to the two others. There is another hydrogen bond found between the two adjacent Schiff base units, which is, O(5)—H(5)···O(12)#1, [O(5)···O(12)#1 = 2.517(4), O(5)—H(5)···O(12)#1 = 168.7].

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