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NOTE

Synthesis and Crystal Structure of Symmetrical Organic Compound

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An unexpected symmetrical organic compound has been synthesized with higher yield under mild reaction conditions. The crystal structure of the title compound has been determined by single-crystal X-ray diffraction. It crystallizes in the trigonal space group $P3_22_1$ with a = 8.679 (9) Å, b = 8.679 (9) Å, c = 17.714 (17) Å, β = 90° X, Z = 6.

Keywords: Symmetrical structure, Crystal Structure, Synthesis.

Schiff bases compounds are poentially multidentate ligands, because they can accommodate one[1,2], two or more transition metal centers and form metal complexes with interesting properties[3-6], such as catalytic activity for epoxidation or aziridination[7], models of reaction centers of metalloenzymes[8], non-linear optical materials[9] and for molecular recognition and biological activity[10].

3-Methoxysalicylaldehyde was purchased from Alfa Aesar and used without further purification. *o*-Aminoacetophenone oxime was synthesized according to an analogous method reported earlier[11]. The other reagents were analytical grade reagents from Tianjin Chemical Reagent Factory. C, H and N analyses were carried out with a GmbH Variuo EL V3.00 automatic elemental analyzer. X-ray single crystal structure was determined on a Bruker Smart APEX CCD area detector. Melting points were obtained by use of a microscopic melting point apparatus made by Beijing Taike Instrument Limited Company and are uncorrected.

To an ethanolic solution (2 mL) of 3-methoxysalicylaldehyde (153.4 mg, 1.01 mmol) was added an ethanolic solution (3 mL) of *o*-aminoacetophenone oxime (150.2 mg, 1 mmol). After stirring at 55-60 °C for 12 h, the mixture was filtered and the precipitate was collected on a suction filter to afford the unexpected organic compound (215.4 g, 85.8 %) as pale yellow powder. m.p. 196-197 °C. Anal. calcd. for $C_{11}NO$ (%): C, 81.49; N, 8.64; O, 9.87. Found (%): 81.46; N, 8.68; O, 9.86.

The compound is dissolved with methylene chloride and *n*-hexane, the color of the solution is pale yellow, the mixture was filtered and the filtrate was allowed to stand at room temperature for about two weeks. The solvent was partially evaporated and obtained pale-yellow block-like single crystals.

X-Ray structure determination: The single crystal of the title compound, with approximate dimensions of 0.21 × 0.10 × 0.08 mm was placed on a Bruker Smart 1000 diffractometer equipped with Apex CCD area detector. The diffraction data were collected using a graphite monochromated MoK_α radition (λ = 0.71073 Å) at 288.9 (2) K. Data reduction and cell refinement were performed using SAINT[12]. The structure was solved by the direct method (SHELXS-97[13]) and fourier difference techniques and refined by full-matrix least-squares method[14,15] on F^2 using SHELXL-97. Anisotropic thermal parameters were assigned to all atoms. Details of the data collection and refinements of title compound are given in Table-1.

X-ray crystallographic analysis revealed the crystal structure of the title compound. And the structure of the compound is shown in Fig. 1. Details of relevant bond lengths and angles of the title compound are summarized in Table-2. The crystal structure of the title compound is built up by only the $C_{11}NO$ molecules and all bond lengths and angles are in normal ranges. The title molecule crystallizes possessing a crystallographically imposed center of symmetry at the shaft of the C8-C8 bond. The packing arrangement of the unit cell of the title compound are shown in Fig. 2.

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TABLE-1
CRYSTAL DATA AND STRUCTURE
REFINEMENT FOR THE TITLE COMPOUND

Empirical formula	$C_{11}NO$
Formula weight, g mol^{-1}	162.12
Temperature, K	288.9(2)
Wavelength, Å	0.71073
Crystal system	Trigonal
Space group	P3,21
Cell dimensions (Å, °)	a = 8.679 (9), b = 8.679 (9), c = 17.714 (17), α = 90.00, β = 90, γ = 120.00
Volume, $Å^3$	1155.54(3)
Z	6
Density (calculated), mg/m^3	1.397
Absorption coefficient, mm^{-1}	0.093
F(000)	486
Index ranges	-11 ≤ h ≤ 10, -10 ≤ k ≤ 11, -22 ≤ 1 ≤ 22
Reflections collected	1700/1656 [$R_{(int)}$ = 0.0307]
Independent reflection	1656
Data/restraints/parameters	1728/0/118
Goodness of fit indicator	1.084
R [I> 2σ(I)]	R_1 = 0.0545, wR_2 = 0.1042
Largest diff. peak and hole, $eÅ^3$	0.360 and -0.308

TABLE-2
SELECTED BOND LENGTHS (Å) AND
ANGLES (°) OF THE TITLE COMPOUND

Bond	Lengths	Bond	Lengths	Bond	Lengths
O(1)-C(1)	1.81(2)	N(1)-C(6)	1.38(2)	C(2)-C(3)	1.642(18)
O(1)-C(2)	1.286(19)	C(1)-C(7)	1.40(2)	C(2)-C(10)	1.37(2)
C(3)-C(4)	1.439(18)	C(4)-C(5)	1.50(2)	C(5)-C(6)	1.33(3)
C(3)-C(8)	1.455(12)	C(4)-C(7)	1.44(2)	C(5)-C(11)	1.31(2)
C(8)-C(8)	1.430(16)	C(8)-C(9)	1.405(18)	C(9)-C(10)	1.43(3)
Bond	**Angles**	**Bond**	**Angles**	**Bond**	**Angles**
C(2)-O(1)-C(1)	145.8(13)	C(7)-C(1)-O(1)	94.8(12)	O(1)-C(2)-C(10)	122.2(14)
O(1)-C(2)-C(3)	120.4(13)	C(10)-C(2)-C(3)	117.1(12)	C(4)-C(3)-C(8)	122.4(9)
C(4)-C(3)-C(2)	112.5(11)	C(8)-C(3)-C(2)	105.5(9)	C(3)-C(4)-C(7)	121.3(13)
C(3)-C(5)-C(5)	121.0(12)	C(7)-C(4)-C(5)	117.7(13)	C(11)-C(5)-C(6)	118.5(15)
C(11)-C(5)-C(4)	120.8(15)	C(6)-C(5)-C(4)	119.8(14)	C(5)-C(6)-N(1)	116.7(16)
C(1)-C(7)–C(4)	122.1(15)	C(9)-C(8)-C(8)	113.6(11)	C(9)-C(8)-C(3)	126.6(11)
C(8)-C(8)-C(3)	119.4(9)	C(8)-C(9)-C(10)	124.5(17)	C(2)-C(10)-C(9)	118.0(15)

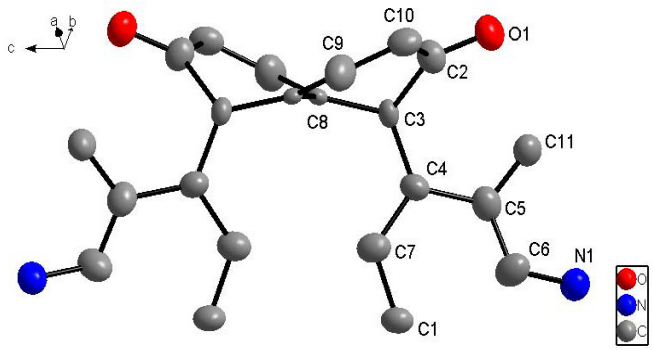

Fig. 1. Molecule structure of the compound with atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30 % probability level

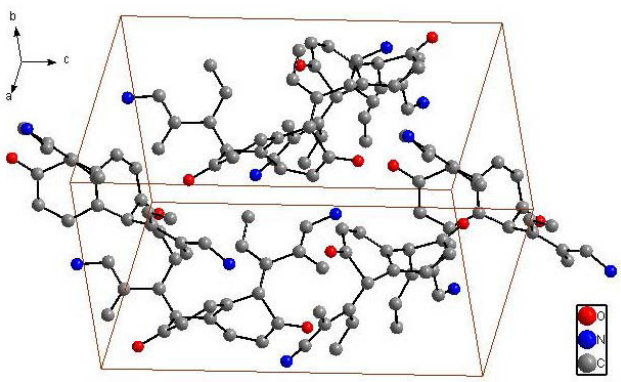

Fig. 2. Packing arrangement of the unit cell of the title compound

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