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Crystal structure of *N,N'*-bis(4-bromosalicylidene) ethylene-1,2-diaminopropan, $C_{34}H_{32}Br_4N_4O_4$

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Abstract

$C_{34}H_{32}Br_4N_4O_4$, $M_r = 880.27$, monoclinic, $P2_1/n$ (no. 14), $a = 16.3126(5)$ Å, $b = 6.9654(2)$ Å, $c = 29.300(1)$ Å, $\beta = 92.726(1)^\circ$, $V = 3325.41(18)$ Å³, $Z = 4$, $R_{gt}(F) = 0.0349$, $wR_{ref}(F^2) = 0.0799$, $T = 150(2)$ K.

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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:	Yellow block
Size:	0.22 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	4.89 mm ⁻¹
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
θ_{max} , completeness:	26.4°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	54,956, 6827, 0.075
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 4981
$N(param)_{refined}$:	421
Programs:	Bruker [1], Olex2 [2], SHELX [3, 4]

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Source of material

All chemicals were also commercially available and used without further purification. Place 4-bromosalicylaldehyde (0.402 g, 2.0 mmol) in 20 mL methanol and stir at room temperature until dissolved. Then 0.10 mL (0.5 mmol) of 1,2-diaminopropane was added to the above solution. The resulting solution was stirred and refluxed at 80 °C for 3 h, then naturally cooled and filtered. The filtrate was allowed to stand at room temperature for one week to obtain the yellow title compound.

Experimental details

The structure was solved with the Olex2 program [2] as an interface together with the SHELXT and SHELXL programs [3, 4]. All H atoms were placed in geometrically idealized positions, with C-H = 0.99 (methylene), O-H = 0.84 (hydroxyl), 0.95 Å (aryl), and with $U_{iso}(H) = 1.2 U_{eq}(C)$ for H atoms on methylene, hydroxyl and aryl.

Comment

Schiff bases can be easily tuned by the change of amine or carbonyl component under moderate reaction conditions [5, 6]. Schiff bases can also act as ligands to construct different types of complexes [7, 8]. In order to explore the relationship between molecular structure and physicochemical properties of halogenated Schiff-bases, herein we report a new bromo-substituted salen-type ligand.

X-ray diffraction analysis reveals that the asymmetric unit of the title compound contains two crystallographic independent salen-type molecules. As shown in the figure, the two crystallographically independent molecules show a different conformation about the central C–C bond. PLATON software analysis reveals that a self-assembled supramolecular dimer, the adjacent molecular units are linked by weak intermolecular C-H...O hydrogen-bonds.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
Br1	0.49023 (3)	0.17925 (6)	0.54696 (2)	0.05009 (11)
Br2	0.30537 (3)	0.18836 (6)	0.98393 (2)	0.05271 (12)
Br3	0.67421 (3)	−0.39478 (6)	0.54938 (2)	0.04996 (11)
Br4	0.56008 (3)	1.34275 (6)	0.96431 (2)	0.05096 (11)
O1	0.38663 (17)	0.4417 (4)	0.69950 (9)	0.0481 (7)
H1	0.3705	0.5415	0.7124	0.072*
O2	0.38264 (16)	0.4751 (4)	0.82863 (9)	0.0461 (6)
H2	0.3810	0.5758	0.8129	0.069*
O3	0.55090 (16)	−0.0257 (4)	0.68329 (9)	0.0480 (7)
H3A	0.5581	0.0708	0.7002	0.072*
O4	0.66154 (17)	0.9754 (4)	0.82433 (9)	0.0496 (7)
H4	0.6609	0.8678	0.8113	0.074*
N1	0.33810 (18)	0.7944 (4)	0.70670 (11)	0.0427 (7)
N2	0.34572 (18)	0.8243 (4)	0.80694 (11)	0.0417 (7)
N3	0.5991 (2)	0.3182 (4)	0.70434 (11)	0.0505 (8)
N4	0.6158 (2)	0.6304 (4)	0.80629 (12)	0.0515 (9)
C1	0.3851 (2)	0.6649 (5)	0.63712 (13)	0.0397 (8)
C2	0.4000 (2)	0.6964 (5)	0.59127 (13)	0.0426 (9)
H2A	0.3882	0.8192	0.5785	0.051*
C3	0.4313 (2)	0.5558 (5)	0.56369 (13)	0.0430 (9)
H3	0.4412	0.5798	0.5325	0.052*
C4	0.4476 (2)	0.3776 (5)	0.58351 (13)	0.0411 (8)
C5	0.4330 (2)	0.3390 (5)	0.62862 (13)	0.0420 (9)
H5	0.4446	0.2154	0.6410	0.050*
C6	0.4012 (2)	0.4813 (5)	0.65578 (13)	0.0412 (9)
C7	0.3533 (2)	0.8190 (5)	0.66507 (13)	0.0410 (8)
H7	0.3436	0.9416	0.6516	0.049*
C8	0.3043 (2)	0.9554 (5)	0.73195 (14)	0.0464 (9)
H8A	0.2461	0.9281	0.7377	0.056*
H8B	0.3059	1.0726	0.7129	0.056*
C9	0.3507 (2)	0.9931 (5)	0.77727 (13)	0.0442 (9)
H9	0.3240	1.1036	0.7925	0.053*
C10	0.4405 (2)	1.0422 (6)	0.77204 (14)	0.0471 (9)
H10A	0.4660	1.0755	0.8020	0.071*
H10B	0.4447	1.1516	0.7512	0.071*
H10C	0.4688	0.9312	0.7596	0.071*
C11	0.3090 (2)	0.8404 (5)	0.84424 (13)	0.0404 (8)
H11	0.2839	0.9590	0.8513	0.049*
C12	0.3048 (2)	0.6817 (5)	0.87620 (12)	0.0380 (8)
C13	0.3435 (2)	0.5047 (5)	0.86773 (12)	0.0373 (8)
C14	0.3437 (2)	0.3583 (5)	0.89989 (13)	0.0409 (8)
H14	0.3708	0.2403	0.8946	0.049*
C15	0.3037 (2)	0.3874 (5)	0.93963 (13)	0.0405 (8)
C16	0.2627 (2)	0.5566 (5)	0.94857 (13)	0.0440 (9)
H16	0.2342	0.5716	0.9759	0.053*
C17	0.2642 (2)	0.7019 (5)	0.91699 (12)	0.0413 (8)
H17	0.2371	0.8193	0.9230	0.050*
C18	0.6529 (2)	0.1436 (5)	0.64246 (12)	0.0374 (8)
C19	0.6045 (2)	−0.0203 (5)	0.64938 (12)	0.0390 (8)
C20	0.6103 (2)	−0.1807 (5)	0.62189 (12)	0.0389 (8)
H20	0.5771	−0.2905	0.6263	0.047*
C21	0.6659 (2)	−0.1768 (5)	0.58780 (12)	0.0402 (8)
C22	0.7163 (2)	−0.0200 (5)	0.58040 (13)	0.0416 (9)
H22	0.7548	−0.0222	0.5571	0.050*
C23	0.7088 (2)	0.1387 (5)	0.60798 (12)	0.0404 (8)

Table 2: (continued)

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
H23	0.7425	0.2474	0.6034	0.049*
C24	0.6470 (2)	0.3134 (5)	0.67095 (13)	0.0422 (8)
H24	0.6793	0.4230	0.6647	0.051*
C25	0.5962 (3)	0.4946 (6)	0.73169 (15)	0.0598 (12)
H25A	0.5395	0.5461	0.7305	0.072*
H25B	0.6326	0.5929	0.7190	0.072*
C26	0.6234 (3)	0.4514 (6)	0.78026 (15)	0.0548 (11)
H26	0.5879	0.3493	0.7930	0.066*
C27	0.7134 (2)	0.3862 (6)	0.78234 (14)	0.0487 (10)
H27A	0.7475	0.4867	0.7695	0.073*
H27B	0.7186	0.2679	0.7646	0.073*
H27C	0.7316	0.3626	0.8142	0.073*
C28	0.5708 (2)	0.6361 (5)	0.84066 (13)	0.0436 (9)
H28	0.5396	0.5259	0.8479	0.052*
C29	0.5657 (2)	0.8065 (5)	0.86897 (12)	0.0391 (8)
C30	0.5149 (2)	0.8111 (5)	0.90613 (12)	0.0399 (8)
H30	0.4823	0.7019	0.9124	0.048*
C31	0.5109 (2)	0.9711 (5)	0.93397 (13)	0.0431 (9)
H31	0.4750	0.9742	0.9586	0.052*
C32	0.5604 (2)	1.1263 (5)	0.92502 (13)	0.0401 (8)
C33	0.6112 (2)	1.1299 (5)	0.88855 (12)	0.0380 (8)
H33	0.6441	1.2392	0.8830	0.046*
C34	0.6131 (2)	0.9699 (5)	0.86004 (12)	0.0374 (8)

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