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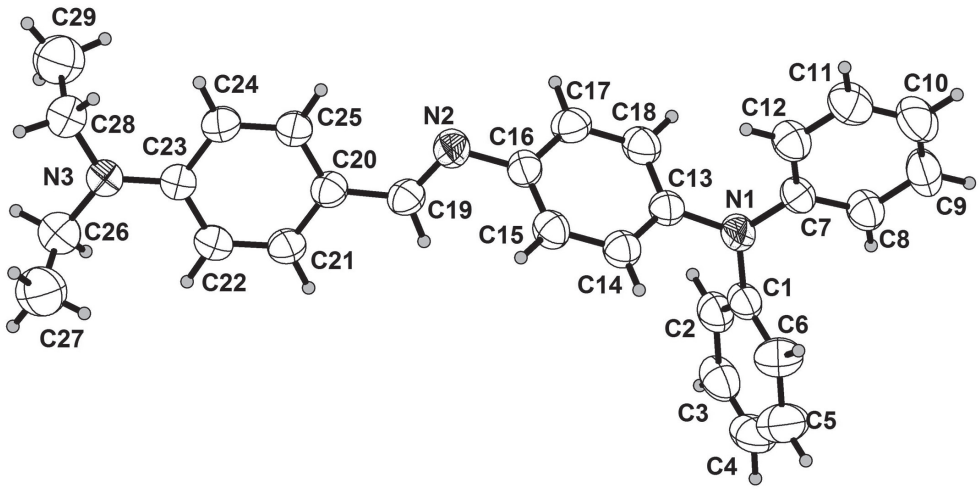
The crystal structure of (*E*)-4-((4-(diethylamino) benzylidene)amino)-*N*,*N*-diphenylaniline, $C_{29}H_{29}N_3$



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Abstract

$C_{29}H_{29}N_3$, triclinic, $P\bar{1}$ (no. 2), $a = 10.677(4)$ Å, $b = 11.325(4)$ Å, $c = 21.530(8)$ Å, $\alpha = 88.026(5)°$, $\beta = 75.646(5)°$, $\gamma = 71.504(4)°$, $V = 2389.0(15)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0663$, $wR_{ref}(F^2) = 0.2002$, $T = 293(2)$ K.

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One of two crystallographically independent molecules of the title 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:	Black needle
Size:	0.30 × 0.10 × 0.10 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ and ω
θ_{max}, completeness:	25.0°, 99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17204, 8351, 0.136
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4965
$N(param)_{refined}$:	581
Programs:	Bruker [1], SHELX [2, 3]

Source of material

Reagents and solvents used were of commercially available quality. The ethanol solution of *N*,*N*-diphenylbenzene-1,4-diamine (0.26 g, 1 mmol) was added to a solution containing 4-(diethylamino)benzaldehyde (0.18 g, 1 mmol) in 5 mL of absolute ethanol and acetic acid (1 day) under heating and stirring. The mixture was then refluxed for 3 h. Afterwards the mixture was cooled to room temperature. It was pured by ethanol recrystallization in 82% yield as yellow solid. Then the resulting solution had to stand in air. On slow evaporation of the solvent, some black block crystals were obtained after 5 days. ^{1}H NMR (400 MHz, $(CD_3)_2CO$) δ [ppm] 8.39 (d, $J = 5.0$ Hz, 1H), 7.75–7.65 (m, 2H), 7.30–7.22 (m, 4H), 7.20–7.11 (m, 2H), 7.07–6.93 (m, 8H), 6.82–6.69 (m, 2H), 3.46 (q, $J = 7.1$ Hz, 4H), 1.16 (t, $J = 7.0$ Hz, 6H). ^{13}C NMR (100 MHz, $(CD_3)_2CO$) δ [ppm] 158.47, 150.13, 148.43, 148.07, 144.95, 130.55, 129.34, 125.31, 124.13, 123.64, 122.59, 121.98, 111.01, 44.18, 12.03. MALDI-TOF: ($[M + H]^+$), 516.85. IR (KBr pellet, cm^{-1}): 3447m,

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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}
C1	0.0858(3)	1.3253(2)	0.23642(11)	0.0568(6)
C2	−0.0257(3)	1.3344(3)	0.21327(11)	0.0659(7)
H2	−0.0551	1.2654	0.2131	0.079*
C3	−0.0944(3)	1.4449(3)	0.19034(12)	0.0773(9)
H3	−0.1696	1.4502	0.1746	0.093*
C4	−0.0520(4)	1.5462(3)	0.19080(14)	0.0889(10)
H4	−0.0980	1.6206	0.1751	0.107*
C5	0.0581(4)	1.5388(3)	0.21428(17)	0.0960(10)
H5	0.0861	1.6085	0.2149	0.115*
C6	0.1275(3)	1.4289(3)	0.23684(14)	0.0787(8)
H6	0.2027	1.4242	0.2524	0.094*
C7	0.1808(2)	1.1991(2)	0.31988(10)	0.0515(6)
C8	0.1000(3)	1.2881(3)	0.36818(12)	0.0647(7)
H8	0.0302	1.3554	0.3598	0.078*
C9	0.1231(3)	1.2769(3)	0.42888(13)	0.0812(9)
H9	0.0685	1.3373	0.4610	0.097*
C10	0.2247(4)	1.1787(3)	0.44250(15)	0.0855(9)
H10	0.2396	1.1725	0.4834	0.103*
C11	0.3042(3)	1.0897(3)	0.39519(15)	0.0780(8)
H11	0.3732	1.0222	0.4040	0.094*
C12	0.2822(3)	1.0996(3)	0.33455(12)	0.0646(7)
H12	0.3366	1.0382	0.3029	0.078*
C13	0.2159(2)	1.1067(2)	0.21203(11)	0.0539(6)
C14	0.3095(2)	1.1167(2)	0.15639(12)	0.0584(6)
H14	0.3389	1.1863	0.1511	0.070*
C15	0.3590(2)	1.0232(2)	0.10872(12)	0.0584(6)
H15	0.4207	1.0309	0.0711	0.070*
C16	0.3183(2)	0.9183(2)	0.11608(11)	0.0533(6)
C17	0.2316(3)	0.9060(2)	0.17374(12)	0.0611(7)
H17	0.2082	0.8333	0.1807	0.073*
C18	0.1796(3)	0.9999(2)	0.22080(12)	0.0635(7)
H18	0.1196	0.9913	0.2588	0.076*
C19	0.3485(2)	0.8595(2)	0.01129(12)	0.0561(6)
H19	0.3091	0.9445	0.0075	0.067*
C20	0.3887(2)	0.7798(2)	−0.04595(11)	0.0544(6)
C21	0.3597(3)	0.8327(2)	−0.10230(12)	0.0600(6)
H21	0.3161	0.9182	−0.1018	0.072*
C22	0.3930(3)	0.7635(2)	−0.15826(12)	0.0649(7)
H22	0.3701	0.8021	−0.1945	0.078*
C23	0.4610(3)	0.6356(2)	−0.16165(12)	0.0649(7)
C24	0.4921(3)	0.5812(2)	−0.10483(13)	0.0725(8)
H24	0.5375	0.4962	−0.1053	0.087*
C25	0.4559(3)	0.6525(2)	−0.04931(12)	0.0660(7)
H25	0.4771	0.6144	−0.0126	0.079*
C26	0.4371(4)	0.6177(3)	−0.27436(14)	0.0891(9)
H26A	0.4263	0.5514	−0.2977	0.107*
H26B	0.3481	0.6794	−0.2587	0.107*
C27	0.5279(4)	0.6747(3)	−0.31668(17)	0.1066(11)
H27A	0.5381	0.7405	−0.2934	0.160*
H27B	0.4902	0.7084	−0.3520	0.160*
H27C	0.6153	0.6131	−0.3326	0.160*
C28	0.5802(3)	0.4315(3)	−0.22361(14)	0.0821(9)
H28A	0.6298	0.4083	−0.2680	0.098*
H28B	0.6460	0.4178	−0.1979	0.098*

Table 2 (continued)

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C29	0.4929(4)	0.3554(3)	−0.20192(18)	0.1034(11)
H29A	0.4568	0.3677	−0.1563	0.155*
H29B	0.5448	0.2692	−0.2125	0.155*
H29C	0.4192	0.3788	−0.2226	0.155*
C30	0.5116(3)	0.2899(3)	0.56125(12)	0.0624(7)
C31	0.4147(3)	0.3507(3)	0.52904(14)	0.0909(10)
H31	0.4404	0.3583	0.4849	0.109*
C32	0.2801(3)	0.4001(4)	0.56180(19)	0.1088(12)
H32	0.2155	0.4415	0.5397	0.131*
C33	0.2403(3)	0.3888(3)	0.62673(17)	0.0936(10)
H33	0.1490	0.4224	0.6486	0.112*
C34	0.3340(3)	0.3289(3)	0.65868(14)	0.0791(8)
H34	0.3071	0.3212	0.7027	0.095*
C35	0.4707(3)	0.2784(3)	0.62644(12)	0.0694(7)
H35	0.5346	0.2368	0.6489	0.083*
C36	0.7548(2)	0.2454(3)	0.55679(11)	0.0572(7)
C37	0.7520(3)	0.3548(3)	0.58315(14)	0.0804(9)
H37	0.6808	0.4276	0.5824	0.096*
C38	0.8554(4)	0.3572(4)	0.61108(16)	0.1030(12)
H38	0.8532	0.4316	0.6290	0.124*
C39	0.9607(4)	0.2504(5)	0.61222(16)	0.1033(12)
H39	1.0294	0.2518	0.6313	0.124*
C40	0.9640(3)	0.1438(4)	0.58559(16)	0.0923(10)
H40	1.0359	0.0714	0.5860	0.111*
C41	0.8631(3)	0.1401(3)	0.55784(13)	0.0747(8)
H41	0.8676	0.0653	0.5394	0.090*
C42	0.6871(2)	0.1799(3)	0.46658(11)	0.0573(6)
C43	0.6324(2)	0.0916(3)	0.45444(11)	0.0613(7)
H43	0.5686	0.0712	0.4872	0.074*
C44	0.6700(3)	0.0323(3)	0.39461(11)	0.0604(7)
H44	0.6326	−0.0283	0.3876	0.072*
C45	0.7635(2)	0.0626(2)	0.34501(10)	0.0518(6)
C46	0.8215(3)	0.1485(3)	0.35790(12)	0.0633(7)
H46	0.8877	0.1669	0.3256	0.076*
C47	0.7836(3)	0.2079(3)	0.41755(12)	0.0680(7)
H47	0.8227	0.2670	0.4250	0.082*
C48	0.7295(3)	−0.0331(2)	0.25923(11)	0.0539(6)
H48	0.6425	−0.0240	0.2847	0.065*
C49	0.7677(2)	−0.0916(2)	0.19576(10)	0.0501(6)
C50	0.6774(2)	−0.1315(2)	0.17253(11)	0.0548(6)
H50	0.5911	−0.1215	0.1990	0.066*
C51	0.7104(3)	−0.1854(2)	0.11165(11)	0.0552(6)
H51	0.6469	−0.2118	0.0982	0.066*
C52	0.8389(3)	−0.2007(2)	0.06994(10)	0.0516(6)
C53	0.9305(3)	−0.1596(2)	0.09359(11)	0.0565(6)
H53	1.0167	−0.1683	0.0673	0.068*
C54	0.8953(2)	−0.1069(2)	0.15440(11)	0.0555(6)
H54	0.9584	−0.0808	0.1684	0.067*
C55	0.7805(3)	−0.2984(2)	−0.01588(12)	0.0671(7)
H55A	0.7985	−0.2903	−0.0620	0.080*
H55B	0.6872	−0.2475	0.0030	0.080*
C56	0.7961(3)	−0.4314(3)	−0.00145(17)	0.0946(10)
H56A	0.8877	−0.4824	−0.0210	0.142*
H56B	0.7333	−0.4576	−0.0182	0.142*
H56C	0.7772	−0.4396	0.0442	0.142*

Table 2 (continued)

Atom	x	y	z	U_{iso}*/U_{eq}
C57	1.0035(3)	−0.2605(2)	−0.03587(12)	0.0679(7)
H57A	1.0279	−0.3291	−0.0670	0.081*
H57B	1.0736	−0.2782	−0.0124	0.081*
C58	0.9995(3)	−0.1429(3)	−0.07073(13)	0.0781(8)
H58A	0.9297	−0.1245	−0.0937	0.117*
H58B	1.0863	−0.1537	−0.1004	0.117*
H58C	0.9801	−0.0754	−0.0404	0.117*
N1	0.1570(2)	1.20921(19)	0.25841(9)	0.0642(6)
N2	0.3626(2)	0.82293(19)	0.06678(10)	0.0622(6)
N3	0.4965(3)	0.5650(2)	−0.21777(11)	0.0933(9)
N4	0.6504(2)	0.2397(2)	0.52836(9)	0.0714(7)
N5	0.8085(2)	0.0066(2)	0.28226(9)	0.0588(5)
N6	0.8730(2)	−0.2525(2)	0.00897(9)	0.0628(6)

3413s, 1596s, 1586m, 1525m, 1404w, 1317m, 1270m, 1317m, 1174s, 835m, 820m, 745m, 699s, 592m.

Experimental details

All H-atoms were placed in calculated positions and treated as riding: C—H = 0.93–0.97 Å, with $U_{iso}(H) = 1.2$ or 1.5 U_{eq} (parent C-atom).

Comment

Schiff bases bearing a bridged C=N structure have a wide range of applications in detection of metal ions, because they always have weak fluorescence signals for the C=N isomerization, but when the C=N group is coordinated with metal ions to form the complexes, the C=N isomerization is inhibited that results in much stronger fluorescence signals [4–6]. Among these compounds, the excellent electronic and optical properties of triphenylamine-based probes are studied widely for their butterfly-like structure, low ionization potential, high electron affinity and good UV light harvesting features [6, 7]. As part of an ongoing study concerning the optical properties of Schiff bases [8], the title compound is reported here.

The asymmetric unit of the title structure consists of two independent molecules of title molecule. In both independent molecules, the C=N bond adopts *E* or *trans* configurations. These core fragments of the title molecule are almost planar with torsion angles C20–N19–N2–C16 and C49–N48–N5–C45 of 178.7(2)° and −179.9(2)°, respectively, the dihedral angles between the mean planes of the phenyl rings are 55.3(2)° and 151.1(1)°, respectively. Geometric parameters are in general as expected [9].

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