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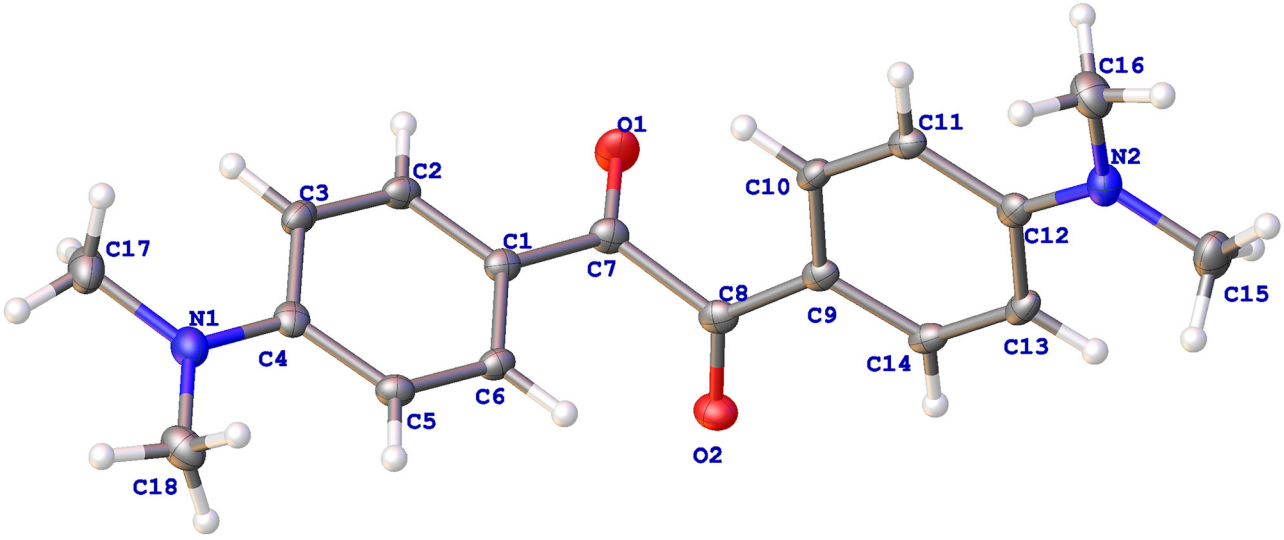
The crystal structure of 1,2-bis(4-(dimethylamino) phenyl)ethane-1,2-dione. $C_{18}H_{20}N_2O_2$



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Abstract

$C_{18}H_{20}N_2O_2$, triclinic, $P\bar{1}$ (no. 2), a = 9.9301(5) Å, b = 10.6446(5) Å, c = 15.2690(8) Å, α = 77.743(2)°, β = 87.460(2)°, γ = 89.139(2)°, V = 1575.60(14) Å^3, Z = 4, $R_{gt}(F)$ = 0.0465, $wR_{ref}(F^2)$ = 0.1188, T = 150 K.

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A part of 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:	Yellow block
Size:	0.15 × 0.12 × 0.11 mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ:	0.66 mm^{-1}
Diffractometer, scan mode:	Bruker,
θ_{max}, completeness:	68.4°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	30,030, 5722, 0.041
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 5146
$N(param)_{refined}$:	405
Programs:	Olex2,[1] SHELX[2,3]

1 Source of material

1,2-bis(4-(dimethylamino)phenyl)ethane-1,2-dione was synthesized according to the classical procedure.[1] The crystal for X-ray data collection was obtained by recrystallization from N, N-dimethylformamide.

2 Experimental details

Using Olex2, the structure was solved with the SHELXS structure solution program using Direct Methods and

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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}
O1	1.23894 (12)	0.78017 (13)	0.68555 (8)	0.0455 (3)
O2	1.13676 (12)	0.53098 (11)	0.81983 (8)	0.0378 (3)
N1	0.82578 (15)	0.61685 (13)	0.42010 (9)	0.0366 (3)
N2	0.83089 (14)	0.91823 (14)	1.02825 (9)	0.0364 (3)
C1	1.05585 (14)	0.68721 (14)	0.62821 (10)	0.0257 (3)
C2	1.08659 (15)	0.74582 (14)	0.53854 (11)	0.0292 (3)
H2	1.162068	0.801407	0.524804	0.035*
C3	1.01085 (16)	0.72522 (15)	0.47009 (10)	0.0311 (3)
H3	1.034048	0.767286	0.410116	0.037*
C4	0.89825 (15)	0.64186 (14)	0.48770 (10)	0.0283 (3)
C5	0.86580 (15)	0.58495 (14)	0.57858 (10)	0.0281 (3)
H5	0.789520	0.530499	0.593051	0.034*
C6	0.94294 (14)	0.60729 (14)	0.64611 (10)	0.0261 (3)
H6	0.919061	0.567511	0.706455	0.031*
C7	1.13906 (15)	0.71170 (15)	0.69876 (11)	0.0299 (3)
C8	1.10577 (14)	0.64524 (14)	0.79635 (11)	0.0283 (3)
C9	1.04526 (14)	0.72190 (14)	0.85620 (10)	0.0259 (3)
C10	0.99421 (15)	0.84651 (14)	0.82431 (10)	0.0263 (3)
H10	1.007460	0.885635	0.762602	0.032*
C11	0.92575 (15)	0.91271 (14)	0.88037 (10)	0.0277 (3)
H11	0.891642	0.996366	0.856863	0.033*
C12	0.90528 (15)	0.85770 (15)	0.97304 (10)	0.0279 (3)
C13	0.96391 (15)	0.73503 (15)	1.00579 (10)	0.0293 (3)
H13	0.957029	0.697806	1.068120	0.035*
C14	1.03008 (14)	0.67011 (14)	0.94847 (10)	0.0272 (3)
H14	1.066853	0.587502	0.971818	0.033*
C15	0.8113 (2)	0.8599 (2)	1.12282 (12)	0.0466 (5)
H15A	0.768948	0.775654	1.129384	0.070*
H15B	0.752894	0.915351	1.151957	0.070*
H15C	0.898666	0.849356	1.150966	0.070*
C16	0.7570 (2)	1.03634 (18)	0.99356 (13)	0.0454 (4)
H16A	0.820900	1.105342	0.968744	0.068*
H16B	0.701725	1.060398	1.042274	0.068*
H16C	0.698649	1.022699	0.946350	0.068*
C17	0.8564 (2)	0.68052 (18)	0.32760 (12)	0.0444 (4)
H17A	0.830981	0.771508	0.318866	0.067*
H17B	0.805708	0.639827	0.287595	0.067*
H17C	0.953195	0.673190	0.313923	0.067*
C18	0.70707 (18)	0.53593 (17)	0.43806 (13)	0.0418 (4)
H18A	0.731355	0.451534	0.473840	0.063*
H18B	0.671490	0.525446	0.381203	0.063*
H18C	0.638236	0.576177	0.471258	0.063*
O3	0.79844 (11)	0.17197 (11)	0.74342 (8)	0.0392 (3)
O4	0.52753 (13)	0.03587 (11)	0.81602 (8)	0.0415 (3)
N3	0.35184 (13)	0.15516 (13)	0.40725 (9)	0.0333 (3)
N4	0.53808 (14)	0.63604 (13)	0.90466 (10)	0.0350 (3)
C19	0.40304 (15)	0.14698 (14)	0.48922 (10)	0.0276 (3)
C20	0.51740 (16)	0.21841 (15)	0.50091 (10)	0.0299 (3)
H20	0.559755	0.273459	0.450372	0.036*
C21	0.56813 (15)	0.20930 (14)	0.58439 (10)	0.0287 (3)
H21	0.643536	0.260126	0.590568	0.034*
C22	0.51133 (15)	0.12686 (14)	0.66060 (10)	0.0266 (3)
C23	0.34462 (16)	0.06484 (14)	0.56680 (10)	0.0289 (3)
H23	0.267089	0.016180	0.561565	0.035*
C24	0.39855 (16)	0.05486 (14)	0.64904 (10)	0.0281 (3)
H24	0.358647	−0.002221	0.699566	0.034*
C25	0.56669 (16)	0.11399 (14)	0.74874 (10)	0.0300 (3)
C26	0.68209 (15)	0.20315 (15)	0.76078 (10)	0.0294 (3)
C27	0.64576 (15)	0.31497 (14)	0.79775 (10)	0.0263 (3)
C28	0.74333 (15)	0.38696 (15)	0.82865 (10)	0.0289 (3)
H28	0.835446	0.362410	0.825047	0.035*
C29	0.70920 (15)	0.49209 (15)	0.86399 (10)	0.0292 (3)
H29	0.777804	0.538060	0.885003	0.035*
C30	0.57329 (15)	0.53297 (14)	0.86958 (10)	0.0264 (3)
C31	0.47490 (15)	0.46142 (14)	0.83694 (10)	0.0279 (3)
H31	0.382894	0.486925	0.838567	0.033*
C32	0.51113 (15)	0.35578 (14)	0.80303 (10)	0.0275 (3)
H32	0.442994	0.308762	0.782479	0.033*
C33	0.39906 (18)	0.68030 (18)	0.90801 (14)	0.0425 (4)
H33A	0.373398	0.722801	0.847612	0.064*
H33B	0.390178	0.741261	0.947868	0.064*
H33C	0.339923	0.606659	0.930819	0.064*
C34	0.63866 (19)	0.71408 (18)	0.93383 (13)	0.0443 (4)
H34A	0.688265	0.661171	0.982591	0.067*
H34B	0.594076	0.785391	0.955158	0.067*
H34C	0.701534	0.748360	0.883417	0.067*
C35	0.41232 (19)	0.23843 (19)	0.32758 (11)	0.0439 (4)
H35A	0.401954	0.328365	0.332598	0.066*
H35B	0.367340	0.225435	0.274370	0.066*
H35C	0.508362	0.217546	0.322140	0.066*
C36	0.23161 (17)	0.08491 (17)	0.39588 (12)	0.0368 (4)
H36A	0.248203	−0.007577	0.415473	0.055*
H36B	0.209345	0.104323	0.332515	0.055*
H36C	0.156266	0.110661	0.431994	0.055*

refined with the SHELXL refinement package. The C-bound H atoms were geometrically placed (C–H = 0.95–0.98 Å) and refined as riding with U_{iso}(H) = 1.2–1.5 U_{eq}(C).[2–4]

3 Comment

Benzil is one of the important building blocks, with two adjacent carbonyl groups, which can be easily transformed into various heterocycles. These heterocycles and benzil derivatives are widely used in pharmaceuticals, polymer, and material chemistry. Such as biologically active compounds,[5] fluorescence probes,[6] dyes,[7] sensitizers in organic photovoltaics,[8] as well as organic nonlinear optical materials.[9] Benzils can be obtained through several synthetic methods, for instance, Friedel–Crafts acylation,[10] oxidation of benzoins,[11] oxidation of diarylalkenes,[12] oxidation of diarylalkynes,[13] etc. In benzils, some crystal structures of alkoxy group, halogenated or nitro group substitution on phenyl ring have been reported.[9,14,15]

The crystal structure of 1,2-bis(4-(dimethylamino)phenyl) ethane-1,2-dione displays approximate bond lengths and angles compared to the similar structure.[8] The torsion angle of O(1)–C(7)–C(8)–O(2) is –101.53(18)° indicating that there is no conjugated π-bond between the two carbonyl groups. The torsion angle of C(2)–C(1)–C(7)–O(1) is –1.4(2)°, exhibiting the carbonyl almost coplanar with the associated aromatic ring. The torsion angles of O(2)–C(8)–C(9)–C(14) and C(1)–C(7)–C(8)–C(9) are 5.9(2)° and –106.40(16)°, respectively. The dimethylamino segments are approximately coplanar with the corresponding associated aromatic rings due to the p-π conjugation.

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