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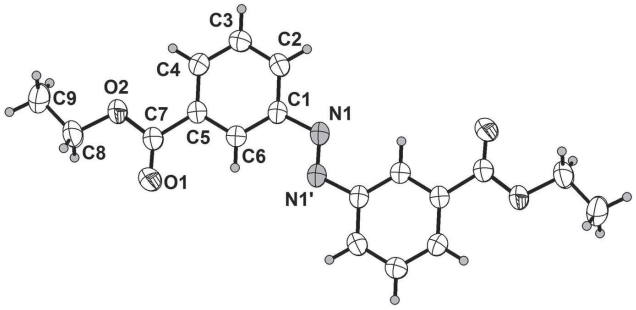
Crystal structure of diethyl 3,3′-(diazene-1,2-diyl)(*E*)-dibenzoate, $C_{18}H_{18}N_2O_4$



DOI 10.1515/ncrs-2016-0372
Received November 22, 2016; accepted April 25, 2017; available online May 10, 2017

Abstract

$C_{18}H_{18}N_2O_4$, monoclinic, $P2_1/n$ (no. 14), $a = 5.2291(2)$ Å, $b = 10.5932(4)$ Å, $c = 15.3775(6)$ Å, $\beta = 96.619(2)°$, $V = 846.13(6)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0479$, $wR_{ref}(F^2) = 0.1438$, $T = 293(2)$ K.

CCDC no.: 1545390

The molecular structure of the title compound is shown in the figure (' = 2−*x*, 2−*y*, 2−*z*). Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

All reagents and solvents were commercially available and used as received without further purification. Azobenzene-3,3′-dicarbonylchloride (3 mmol) was added to a solution of ethanol (25 mL) and 1,2-dichloroethane (15 mL). The reaction mixture was stirred at 80 °C for 3 h, then the solution was naturally cooled to room temperature. After filtration orange flakes of the title compound were obtained with 94.6% yield. Crystals suitable for X-ray diffraction were obtained by slow evaporation of a solution including 0.05 g of the product in methanol (5 mL) and chloroform (5 mL).

Table 1: Data collection and handling.

Crystal:	Orange block
Size:	0.20 × 0.20 × 0.20 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	0.9 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
$2\theta_{max}$, completeness:	57°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	20762, 209, 0.022
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1866
$N(param)_{refined}$:	110
Programs:	Bruker programs [1, 2], SHELX [3], DIAMOND [4]

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
O2	0.10504(18)	0.65799(9)	0.85455(6)	0.0555(3)
C6	0.6243(2)	0.86064(10)	0.94331(7)	0.0418(3)
H6	0.6232	0.9208	0.8992	0.050*
C1	0.8062(2)	0.86682(11)	1.01687(7)	0.0419(3)
C5	0.4449(2)	0.76376(10)	0.93660(7)	0.0405(3)
C4	0.4443(2)	0.67457(12)	1.00307(8)	0.0498(3)
H4	0.3222	0.6104	0.9986	0.060*
O1	0.2532(2)	0.83270(11)	0.79680(7)	0.0799(4)
C2	0.8067(3)	0.77771(13)	1.08252(8)	0.0538(3)
H2	0.9289	0.7822	1.1314	0.065*
C7	0.2592(2)	0.75768(11)	0.85532(8)	0.0468(3)
C3	0.6257(3)	0.68168(13)	1.07583(9)	0.0606(4)
H3	0.6262	0.6220	1.1202	0.073*
C8	−0.0801(3)	0.64201(16)	0.77739(9)	0.0657(4)
H8A	0.0084	0.6303	0.7259	0.079*
H8B	−0.1891	0.7161	0.7685	0.079*
C9	−0.2372(3)	0.52945(14)	0.79195(11)	0.0662(4)
H9A	−0.1291	0.4560	0.7969	0.099*
H9B	−0.3687	0.5193	0.7435	0.099*
H9C	−0.3159	0.5401	0.8449	0.099*
N1	1.00062(18)	0.96177(9)	1.03032(6)	0.0463(3)

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Experimental details

H atoms were subsequently treated as riding atoms with distances C—H = 0.96 (CH_3), 0.97 (CH_2), 0.93 Å (ArH) and O—H = 0.85 Å.

Comment

In recent years, the study of liquid crystalline materials has been of interest due to their potentially wide range of applications, such as in electrical [5], optical [6], and biological medical fields [7]. In this respect, the preparation of liquid crystalline materials containing azobenzene moieties appears to be very promising, because the photoinduced *trans-cis* isomerization of azobenzene chromophores can give rise to photochromic and optical dichroic effects [8]. Liquid crystalline polymers containing azobenzene derivatives have been widely investigated for the photo-controlled release of drugs [9], the preparation of holographic optical memories [10], and non-linear optical materials [11]. In order to enlarge the number of azobenzene derivatives, the synthesis and crystal structure of the title compound was investigated.

The title crystal structure is centrosymmetric and contains one half of a title molecule in the asymmetric unit. Consequently the title molecule is located around an inversion center in the monoclinic space group $P2_1/n$ (*cf*. the figure). Thus the intersection angle between two aryl rings is 0°, which is the same to that of azobenzene [12]. The crystal packing does not exhibit classical hydrogen bond interactions.

Acknowledgements: This work was supported by the Foundation of Anyang Institute of Technology.

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