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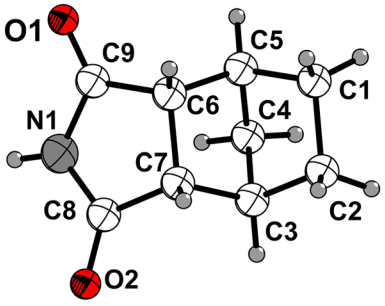
The crystal structure of (3a*S*, 4*R*, 7*S*, 7a*R*)-hexahydro-4, 7-methano-1*H*-isoindole-1, 3-(2*H*)-dione, $C_9H_{11}NO_2$



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Abstract

$C_9H_{11}NO_2$, monoclinic, $P2_1/c$ (no. 14), a = 11.293(4) Å, b = 6.998(3) Å, c = 10.932(4) Å, β = 104.175(7)°, V = 837.6(5) Å^3, Z = 4, $R_{gt}(F)$ = 0.0578, $wR_{ref}(F^2)$ = 0.1330, T = 296 K.

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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.

1 Source of materials

All chemicals were obtained from commercial sources and were used as purchased. A mixture of *cis*-norbornene-exo-2,3-dicarboximide (16.3 g, 0.1 mol) and 10 % palladium on carbon (3.6 g) in methanol (300 ml) were stirred under hydrogen for overnight at room temperature and atmospheric pressure. The reaction monitored by HPLC and the target product was obtained after filtration, washing and concentration under reduced pressure. Colorless block crystals of the title compound were obtained by slow evaporation of a solution in ethyl acetate at room temperature. Melting point: 220–221 °C. ^{1}H NMR (600 MHz, DMSO-d_6) d 11.17 (s, 1H), 2.58 (s, 2H), 2.45 (s, 2H), 1.61–1.47 (m, 2H), 1.30–1.22 (m, 2H), 1.17 (d, J = 10.7 Hz, 1H), 1.10 (d, J = 10.7 Hz, 1H); ^{13}C NMR (150 MHz, DMSO-d_6) d 180.2, 49.5, 38.9, 32.8, 27.3.

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.26 × 0.23 × 0.18 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	0.09 mm^{-1}
Diffractometer, scan mode:	φ and ω
θ_{max}, completeness:	25.0°, 99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	3549, 1468, 0.061
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 949
$N(param)_{refined}$:	109
Programs:	Bruker [1], SHELX [2, 3], WinGX/ORTEP [4]

2 Experimental details

Hydrogen atom was placed in their geometrically idealized positions and constrained to ride on their parent atoms. Their U_{iso} values were set to 1.2U_{eq} of the parent atoms.

3 Comment

Psychiatric disorders such as schizophrenia and bipolar depression had a serious impact on patients in their daily life and work. Antipsychotics have always been a hot and difficult topic in the field of drug research and development [5, 6]. As an important intermediate for the synthesis of antipsychotic drugs lurasidone and tandospirone [7, 8], the title compound has high research value. In the molecule of the title compound bond lengths and angles within *cis*-norbornane-exo-2,3-dicarboximide are very similar to those given in the literature [9, 10]. All non-hydrogen atoms of the molecule are on four planes. The dihedral angles between the C3–C4–C5, C1–C2–C3–C5 and the C3–C5–C6–C7 are 56.1°, 55.5° and 68.3°, respectively. The dihedral angle betweetn C3–C5–C6–C7 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}
C1	0.8518 (3)	0.5595 (5)	0.8549 (3)	0.0645 (9)
H1A	0.8311	0.5449	0.9354	0.077*
H1B	0.8989	0.6757	0.8563	0.077*
C2	0.9227 (3)	0.3838 (5)	0.8250 (3)	0.0671 (9)
H2A	1.0017	0.4205	0.8125	0.081*
H2B	0.9343	0.2898	0.8921	0.081*
C3	0.8392 (2)	0.3067 (4)	0.7029 (3)	0.0525 (8)
H3	0.8796	0.2233	0.6535	0.063*
C4	0.7865 (3)	0.4907 (4)	0.6365 (3)	0.0580 (9)
H4A	0.8486	0.5741	0.6182	0.070*
H4B	0.7227	0.4677	0.5605	0.070*
C5	0.7370 (3)	0.5616 (4)	0.7458 (3)	0.0514 (8)
H5	0.6949	0.6851	0.7314	0.062*
C6	0.6569 (2)	0.3939 (4)	0.7645 (2)	0.0401 (7)
H6	0.6449	0.3913	0.8503	0.048*
C7	0.7281 (2)	0.2186 (4)	0.7380 (2)	0.0393 (6)
H7	0.7521	0.1358	0.8123	0.047*
C8	0.6418 (2)	0.1189 (4)	0.6306 (2)	0.0381 (6)
C9	0.5374 (2)	0.3851 (4)	0.6666 (2)	0.0413 (7)
N1	0.53751 (19)	0.2223 (3)	0.59621 (18)	0.0400 (6)
H1	0.4768	0.1891	0.5357	0.048*
O1	0.45431 (18)	0.4969 (3)	0.64948 (18)	0.0629 (6)
O2	0.66114 (18)	−0.0289 (3)	0.58013 (18)	0.0532 (6)

succinimide group formed by C6–C9–N1 is 60.8°. The complete set of X-ray diffraction data for the title compound was deposited to the Cambridge Crystallographic Data Center (CCDC entry no. 2322260).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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