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著者	KUBO, Kanji
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Crystal Structure of 2-Amino-7-methoxytropone

Kanji KUBO

Abstract — Structure of 2-amino-7-methoxytropone (**1**), $C_8H_9NO_2$, was determined by X-ray crystallographic analysis. It crystallizes in the space group $P\bar{1}$ (#2) with cell parameters a=10.6612(10) Å, b=10.7491(12) Å, c=7.5468(10) Å, α=102.118(5)°, β=109.960(7)°, γ=90.324(3)°, Z=4, and V=791.97(16) Å^3. The structure of **1** contains two crystallographically independent molecules. The NH_2 groups of 2-aminotropones participate in the N—H···O intra- and intermolecular hydrogen bonds. The crystal of **1** had some intermolecular N—H···O, C—H···O, and C—H···π interactions.

1. Introduction

Molecular self-assembly often results in a number of thermodynamically interesting states, such as those of crystals, liquid crystals, gels, and colloids. Numerous studies have been dedicated to the structural investigation and the determination of molecular aggregation mechanisms[1, 2]. Troponoids, being a remarkable class of non-benzenoid π-conjugated systems, have been used as a building block of molecular assemblies such as liquid crystals and organogelators[3, 4]. Tropone and tropolone frequently play an important role as entities determining the specific properties of molecular assemblies. Recently, we have prepared liquid crystals with a tropone core such as tropone[5], cyanotropone[6], nitrotropone[7], bitorpone[8], phenytropone[9] *N,N'*-di(tropon-2-yl)piperazine[10] and phenylazotropone[11]. While the crystal structures of some troponoid core such as tropone[13], tropolone[14], 5-cyanotropolone[15], 5-nitrotropolone[15], 5-(acetyloxy)tropolone[16], 5-methoxytropolone[17], 5-methyltropolone,[18] 2-amino-5-hexyloxytropone,[19] 2-aminotropone,[20] *N,N'*-di(tropon-2-

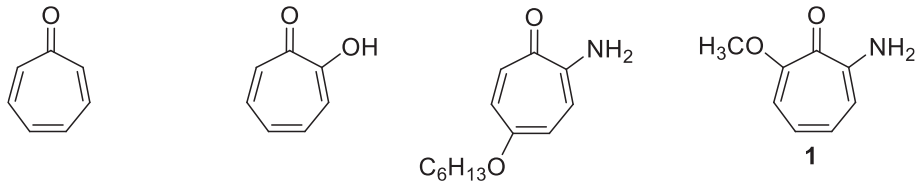

Fig. 1. Chemical structures of title compound (**1**).

yl)piperazine,[20] and 5-(4-ethoxyphenylazo)tropolone.[20] have been elucidated. However the crystal structure of 7-methoxy-2-aminotropone (**1**) has not been elucidated. In this paper, the crystal structure of 2-amino-7-methoxytropone (**1**) is elucidated in order to obtain the information about intermolecular interactions, which are controlling appearance of molecular assembly.

2. Experimental

2.1. Data Collection

A yellow prism crystal of $C_8H_9NO_2$ having approximate dimensions of 0.40×0.35×0.30 mm was mounted on a glass fiber. All measurements were made on Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-$K\alpha$ radiation. Indexing was performed from 2 oscillations that were exposed for 300 seconds. The crystal-to-detector distance was 127.40 mm. Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions: a=10.6612(10) Å, b=10.7491(12) Å, c=7.5468(10) Å, α=102.118(5)°, β=109.960(7)°, γ=90.324(3)°, and V=791.97(16) Å^3. For Z=4 and MW=151.16, the calculated density is 1.268 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be $P\bar{1}$ (#2). The data were collected at a temperature of 25±1°C to a maximum 2θ value of 55.0°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ=0.0°. The exposure rate was 120.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in 5.0° step, at χ=45.0° and φ=180.0°. The exposure rate was 120.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

2.2. Data Reduction

Of the 3532 reflections that were collected, 3532 were unique (R_{int}=0.000); equivalent reflections were merged. The linear absorption coefficient, μ, for Mo-$K\alpha$ radiation radiation is 0.919 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.764 to 0.973. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction[21] was applied (coefficient=0.349260).

2.3. Structure Solution and Refinement

The structure was solved by direct methods[22] and expanded using Fourier techniques.[23] The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically and the rest were refined using the riding model. The final cycle of full-matrix least-

squares refinement ($\Sigma w(F_0^2-F_c^2)^2$ where w=least-squares weights) on F^2 was based on 3532 observed reflections and 217 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of: $R_1=\Sigma||F_0|-|F_c||/\Sigma|F_0|=0.0648$, $wR_2=[\Sigma(w(F_0^2-F_c^2)^2)/\Sigma w(F_0^2)^2]^{1/2}=0.2021$. The standard deviation of an observation of unit weight was 1.05. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.39 and -0.36 e^-/A^3, respectively.

Neutral atom scattering factors were taken from Cromer and Waber.[24] Anomalous dispersion effects were included in F_{calc};[25] the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley.[26] The values for the mass attenuation coefficients are those of Creagh and Hubbell.[27] All calculations were performed using the *CrystalStructure*[28] crystallographic software package except for refinement, which was performed using *SHELXL97*.[29] Fractional atomic coordinates and equivalent isotropic displacement parameters were shown in Table 1. Anisotropic displacement parameters were shown in Table 2. Crystallographic data have been deposited with Cambridge Crystallographic Data Centre: Deposition number CCDC1856365 for **1**. Copies of the data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge, CB2 1EZ, UK; Fax: +44 1223 336033; e-mail: deposit@ccdc.cam.ac.uk).

Table 1. Fractional atomic coordinates, equivalent isotropic displacement parameters (B_{iso}/B_{eq})

atom	x	y	z	B_{eq}
O1	−0.40992(17)	0.10489(12)	−0.29357(18)	4.98(3)
O2	−0.40054(19)	0.26687(13)	0.0051(2)	5.54(3)
O3	0.34150(13)	0.41326(12)	0.4022(2)	4.96(3)
O4	0.12687(16)	0.28585(16)	0.3386(3)	6.70(4)
N1	−0.3630(2)	−0.12988(17)	−0.3389(2)	4.92(3)
N2	0.38126(18)	0.63581(17)	0.3478(2)	4.87(3)
C1	−0.37255(17)	0.06543(16)	−0.1393(2)	3.66(3)
C2	−0.34085(17)	−0.06836(16)	−0.1552(2)	3.74(3)
C3	−0.2901(2)	−0.1339(2)	−0.0061(2)	4.64(3)
C4	−0.2666(2)	−0.0956(2)	0.1899(3)	5.21(4)
C5	−0.2859(2)	0.0191(2)	0.2921(2)	5.04(4)
C6	−0.3292(2)	0.12742(19)	0.2243(2)	4.50(3)
C7	−0.36441(18)	0.15006(16)	0.0400(2)	3.88(3)
C8	−0.4062(3)	0.3664(2)	0.1571(3)	6.45(5)
C9	0.23648(17)	0.47004(17)	0.3452(2)	3.95(3)
C10	0.25403(18)	0.59853(17)	0.3146(2)	4.06(3)
C11	0.1557(2)	0.6836(2)	0.2599(3)	5.39(4)
C12	0.0199(2)	0.6700(3)	0.2175(3)	6.69(6)
C13	−0.0579(2)	0.5689(3)	0.2147(4)	7.25(7)
C14	−0.0173(2)	0.4507(2)	0.2546(3)	6.44(5)
C15	0.1087(2)	0.4069(2)	0.3102(3)	4.86(4)
C16	0.0162(3)	0.1961(3)	0.2889(5)	8.71(8)

H1	−0.409(2)	−0.088(2)	−0.439(3)	5.9(5)
H2	−0.343(2)	−0.211(2)	−0.362(3)	5.2(4)
H3	−0.2688	−0.2164	−0.0459	5.57
H4	−0.2331	−0.1561	0.2621	6.25
H5	−0.2678	0.0253	0.4234	6.05
H6	−0.3357	0.1962	0.3180	5.40
H7	−0.4946	0.3638	0.1632	7.74
H8	−0.3852	0.4471	0.1340	7.74
H9	−0.3426	0.3558	0.2775	7.74
H10	0.445(2)	0.576(2)	0.391(3)	5.4(4)
H11	0.400(3)	0.723(2)	0.328(4)	7.4(6)
H12	0.1880	0.7618	0.2511	6.47
H13	−0.0255	0.7405	0.1861	8.03
H14	−0.1490	0.5790	0.1824	8.70
H15	−0.0865	0.3925	0.2416	7.72
H16	−0.0117	0.2013	0.3984	10.45
H17	0.0413	0.1117	0.2517	10.45
H18	−0.0563	0.2144	0.1830	10.45

$B_{eq}=8/3\pi^2(U_{11}(aa^*)^2+U_{22}(bb^*)^2+U_{33}(cc^*)^2+2U_{12}(aa^*bb^*)\cos\gamma+2U_{13}(aa^*cc^*)\cos\beta+2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Anisotropic displacement parameters (Å^2)

Atom	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0969(10)	0.0440(6)	0.0449(6)	0.0059(6)	0.0192(6)	0.0123(5)
O2	0.1094(12)	0.0464(7)	0.0586(8)	0.0136(7)	0.0351(8)	0.0102(6)
O3	0.0501(7)	0.0443(6)	0.0916(10)	0.0076(5)	0.0219(6)	0.0149(6)
O4	0.0629(9)	0.0646(9)	0.1276(15)	−0.0082(7)	0.0391(9)	0.0139(9)
N1	0.0844(12)	0.0462(8)	0.0529(9)	0.0135(8)	0.0210(8)	0.0091(6)
N2	0.0614(9)	0.0498(8)	0.0736(11)	0.0077(7)	0.0185(8)	0.0220(7)
C1	0.0506(8)	0.0427(8)	0.0455(8)	0.0002(6)	0.0158(6)	0.0110(6)
C2	0.0474(8)	0.0463(8)	0.0491(8)	0.0044(6)	0.0168(6)	0.0122(6)
C3	0.0606(10)	0.0556(10)	0.0602(10)	0.0165(8)	0.0171(8)	0.0196(8)
C4	0.0696(12)	0.0725(13)	0.0598(11)	0.0185(10)	0.0180(9)	0.0311(10)
C5	0.0692(11)	0.0776(13)	0.0465(9)	0.0073(9)	0.0177(8)	0.0217(9)
C6	0.0636(10)	0.0614(10)	0.0462(9)	−0.0001(8)	0.0231(7)	0.0066(7)
C7	0.0527(9)	0.0450(8)	0.0503(9)	0.0003(6)	0.0199(7)	0.0093(6)
C8	0.114(2)	0.0551(11)	0.0757(14)	0.0180(12)	0.0382(13)	0.0058(10)
C9	0.0501(9)	0.0470(8)	0.0485(8)	0.0071(6)	0.0164(7)	0.0026(6)
C10	0.0572(9)	0.0507(9)	0.0410(8)	0.0107(7)	0.0129(7)	0.0062(6)
C11	0.0728(13)	0.0667(12)	0.0584(11)	0.0234(10)	0.0129(9)	0.0158(9)
C12	0.0702(14)	0.0974(18)	0.0798(15)	0.0373(14)	0.0154(11)	0.0229(13)
C13	0.0535(12)	0.127(2)	0.0877(17)	0.0336(14)	0.0165(11)	0.0208(16)
C14	0.0508(11)	0.1040(19)	0.0823(15)	0.0022(11)	0.0246(10)	0.0030(13)
C15	0.0535(10)	0.0657(11)	0.0620(11)	0.0032(8)	0.0233(8)	0.0022(8)
C16	0.0893(19)	0.102(2)	0.134(2)	−0.0357(16)	0.0370(18)	0.0194(19)

The general temperature factor expression:
$\exp(-2\pi^2(a^{*2}U_{11}h^2+b^{*2}U_{22}k^2+c^{*2}U_{33}l^2+2a^*b^*U_{12}hk+2a^*c^*U_{13}hl+2b^*c^*U_{23}kl))$

3. Result & Discussion

The crystal structure of **1** was shown in Fig. 2. 2-Amino-7-methoxytropone contains two crystallographically independent molecules (**1a** and **1b**) in the crystal lattice. The data-collection and refinement parameters are listed in Table 3.

The selected geometric parameters were shown in Table 4. The planarity of the seven-membered ring is fairly good; the deviations of atoms from the least squares planes defined by atoms C1/C2/C3/C4/C5/C6/C7 and C9/C10/C11/C12/C13/C14/C15 are within 0.032 Å for **1a** and 0.021 Å for **1b**, respectively.

As shown in Table 5, the C-C bond lengths of seven-membered ring, apart from the C1-C2 and C8-C9 bonds, show no apparent bond alternation, in contrast to what has been observed for tropone[13] and tropolone[14]. The average value of the C-C bond lengths of **1a** and **1b**, apart from the C1-C2 and C9-C10 bonds, is 1.393 Å, which agrees with the C-C bond length (1.390 Å) of benzene[30]. The C1-C2 and C9-C10 bonds are significantly longer than all other bonds in the ring; the same structural peculiarity had been noted in the tropolone and 2-amino-5-hexyloxytropone.[19]

There is an intramolecular N—H···O hydrogen bond of 2-aminotropone moiety. However there is no intramolecular C—H···O hydrogen bond of methoxy group. Intermolecular N—H···O hydrogen bonds, C—H···O, and C—H···π interactions are observed as shown in Fig. 3 and 4.

The NH_2 group of 2-aminotropone unit participates in the N—H···O intermolecular hydrogen

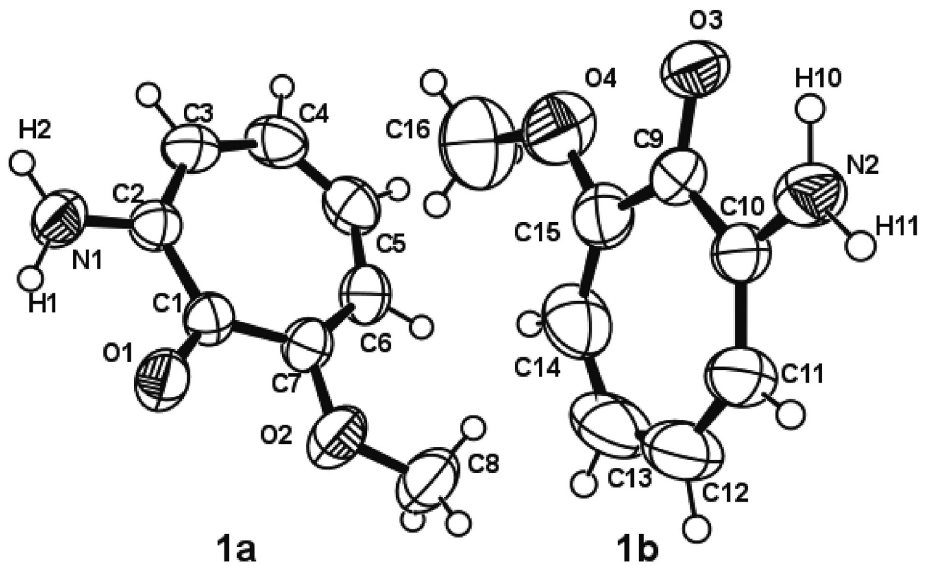

Fig. 2. ORTEP drawings of **1** showing 50% probability displacement ellipsoids.

Table 3. Crystal and experimental data

Chemical formula: $C_8H_9NO_2$	
Formula weight＝151.16	
T＝298K	
Crystal system: Triclinic	Space group: $P\bar{1}$
a＝10.6612(10) Å	α＝102.118(5)°
b＝10.7491(12) Å	β＝109.960(7)°
c＝7.5468(10) Å	γ＝90.324(3)°
V＝791.97(16) Å^3	Z＝4
D_x＝1.268 g/cm^3	
Radiation: Mo K_α (λ＝0.71069 Å) graphite monochromated	
μ(Mo K_α)＝0.919 cm^{-1}	F(000)＝320.00
Crystal size＝0.40×0.35×0.30 mm^3	
$2\theta_{max}$＝55° with Mo K_α	
Data/Restraints/Parameters＝3532/0/217	
R indices [$I \geq 2\sigma(I)$]: $R1$＝0.0648	
R indices (all data): $R1$＝0.0765, $wR2$＝0.2021	
Goodness-of-fit on F^2＝1.052	
$(\Delta/\sigma)_{max}$＝0.000	
$(\Delta\rho)_{max}$＝0.39 eÅ^{-3}	$(\Delta\rho)_{min}$＝−0.36 eÅ^{-3}
Measurement: Rigaku RAXIS-RAPID diffractometer	
Program System: *CrystalStructure 3.8*	
Data collection & cell refinement program: *CrystalStructure 3.8*	
Data reduction program: *CrystalStructure 3.8*	
Structure solving program: *SIR92*	
Structure refinement program: *SHELXL97*	

bonds (Table 6). The N···O distances are close to the intermolecular N—H···O distances [N···O: 2.877(4) and 3.312(4) Å] of 2-amino-5-hexyloxytropone.[19] Some intermolecular C—H···π interactions are observed in **1** as shown in Fig. 3 and Table 6. The H···C distance (2.826 Å) is within the range associated with C—H···π interaction (2.8–3.1 Å).[14–20]

An intermolecular C—H···O hydrogen bond is observed in the crystal structure of **1b** (Table 6 and Fig. 4). The H···O distances are close to this type interaction (2.5–2.7 Å).[14–20]

In conclusion, the crystal structure of 2-amino-7-methoxytropone (**1**) was elucidated by X-ray crystallographic analysis. Thus, the crystal structure of **1** has intermolecular C—H···π interaction, C—H···O, and N—H···O hydrogen bonds.

4. References and Notes

1） K. Kubo, *Kagaku* **59**, 56–57 (2004).
2） K. Kubo, A. Mori, S. Ujiie, and C. Tschierske: *J. Oleo Sci.,* **53**, 575–579 (2004).

Table 4. Selected geometric parameters (Å,°)

O1-C1	1.260(2)	O2-C7	1.367(2)
O2-C8	1.413(3)	O3-C9	1.265(2)
O4-C15	1.367(2)	O4-C16	1.413(3)
N1-C2	1.343(2)	N2-C10	1.335(2)
C1-C2	1.469(2)	C1-C7	1.438(2)
C2-C3	1.405(2)	C3-C4	1.382(3)
C4-C5	1.368(3)	C5-C6	1.387(3)
C6-C7	1.386(2)	C9-C10	1.469(2)
C9-C15	1.434(2)	C10-C11	1.407(3)
C11-C12	1.371(3)	C12-C13	1.356(4)
C13-C14	1.403(4)	C14-C15	1.381(3)
N1-H1	0.95(2)	N1-H2	0.89(2)
N2-H10	0.96(2)	N2-H11	1.01(3)
C7-O2-C8	120.12(17)	C15-O4-C16	120.8(2)
O1-C1-C2	116.71(15)	O1-C1-C7	119.85(15)
C2-C1-C7	123.43(16)	N1-C2-C1	112.39(16)
N1-C2-C3	119.26(16)	C1-C2-C3	128.34(15)
C2-C3-C4	130.52(19)	C3-C4-C5	128.7(2)
C4-C5-C6	128.1(2)	C5-C6-C7	130.63(18)
O2-C7-C1	108.81(16)	O2-C7-C6	121.23(16)
C1-C7-C6	129.95(16)	O3-C9-C10	116.68(16)
O3-C9-C15	119.65(18)	C10-C9-C15	123.65(16)
N2-C10-C9	113.32(16)	N2-C10-C11	118.51(19)
C9-C10-C11	128.16(18)	C10-C11-C12	130.5(2)
C11-C12-C13	129.5(2)	C12-C13-C14	127.7(2)
C13-C14-C15	130.5(2)	O4-C15-C9	108.71(17)
O4-C15-C14	121.4(2)	C9-C15-C14	129.9(2)
C2-N1-H1	117.8(15)	C2-N1-H2	118.8(16)
H1-N1-H2	123(2)	C10-N2-H10	115.5(16)
C10-N2-H11	117.2(17)	H10-N2-H11	127(2)
C8-O2-C7-C1	177.4(2)	C8-O2-C7-C6	−1.8(3)
C16-O4-C15-C9	−171.4(2)	C16-O4-C15-C14	7.9(3)
O1-C1-C2-N1	−2.7(2)	O1-C1-C2-C3	176.0(2)
O1-C1-C7-O2	−0.7(2)	O1-C1-C7-C6	178.5(2)
C2-C1-C7-O2	−179.85(17)	C2-C1-C7-C6	−0.7(3)
C7-C1-C2-N1	176.54(18)	C7-C1-C2-C3	−4.8(3)
N1-C2-C3-C4	−175.6(2)	C1-C2-C3-C4	5.9(3)
C2-C3-C4-C5	−0.7(4)	C3-C4-C5-C6	−2.8(4)
C4-C5-C6-C7	0.1(3)	C5-C6-C7-O2	−177.7(2)
C5-C6-C7-C1	3.3(3)	O3-C9-C10-N2	1.6(2)
O3-C9-C10-C11	−177.15(19)	O3-C9-C15-O4	−3.2(2)
O3-C9-C15-C14	177.6(2)	C10-C9-C15-O4	175.39(18)
C10-C9-C15-C14	−3.9(3)	C15-C9 C10 N2	−177.02(18)
C15-C9-C10-C11	4.2(3)	N2-C10-C11-C12	179.9(2)
C9-C10-C11-C12	−1.4(3)	C10-C11-C12-C13	−0.8(4)
C11-C12-C13-C14	−0.3(4)	C12-C13-C14-C15	1.6(5)
C13-C14-C15-O4	−178.6(2)	C13-C14-C15-C9	0.5(4)

Table 5. Selected Bond Lengths (Å) of **1**, 2-amino-5-hexyloxytropone, tropolone, and tropone.

1a		**1b**		2-amino-5-hexyloxytropone	Tropolone	Tropone	
O1-C1	1.260(2)	O3-C9	1.265(2)	1.250(3)	1.261(3)	1.259	1.257
C1-C2	1.469(2)	C9-C10	1.469(2)	1.474(3)	1.454(4)	1.449	1.434
C1-C7	1.438(2)	C9-C15	1.434(2)	1.423(4)	1.410(3)	1.459	1.450
C2-C3	1.405(2)	C10-C11	1.407(3)	1.385(4)	1.379(4)	1.363	1.348
C3-C4	1.382(3)	C11-C12	1.371(3)	1.382(4)	1.393(4)	1.415	1.435
C4-C5	1.368(3)	C12-C13	1.356(4)	1.376(4)	1.341(4)	1.347	1.327
C5-C6	1.387(3)	C13-C14	1.403(4)	1.409(4)	1.410(4)	1.430	1.432
C6-C7	1.386(2)	C14-C15	1.381(3)	1.353(4)	1.373(4)	1.358	1.362
N1-C2	1.343(2)	N2-C10	1.335(2)	1.340(4)			
N1-H1	0.95(2)	N2-H10	0.96(2)	0.89(4)			
N1-H2	0.89(2)	N2-H11	1.01(3)	0.84(4)			
O2-C2					1.333(3)		
O2-H2					0.94(3)		

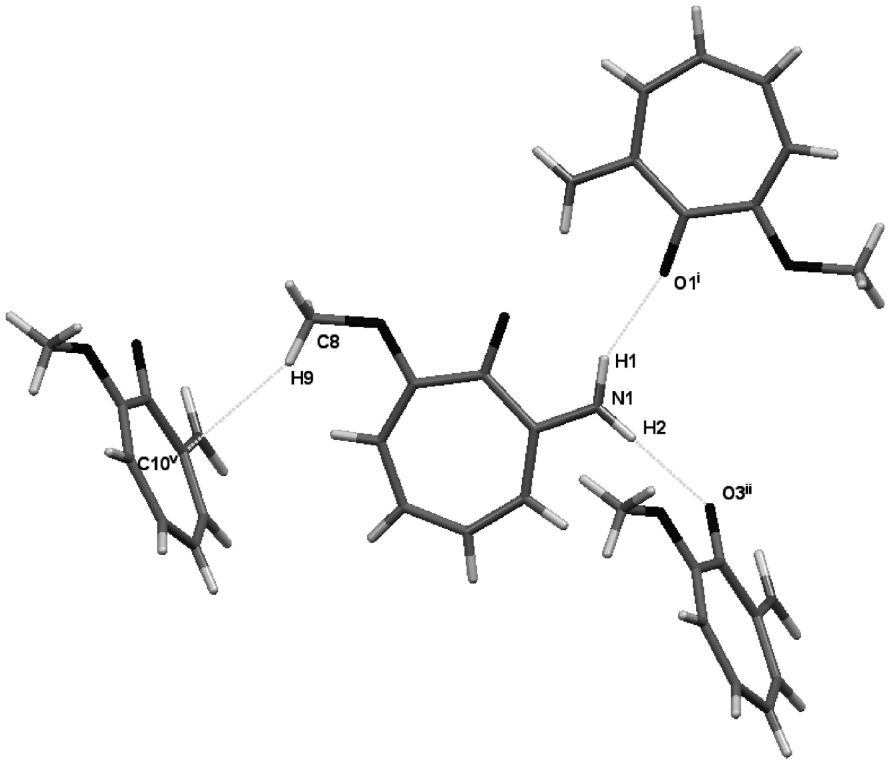

Fig. 3. Intermolecular N—H···O hydrogen bonds and C—H···π interactions of **1a**. Symmetry codes: (i) -1-x, -y, -1-z, (ii) -x, -y, -z, (v) -x, 1-y, 1-z.

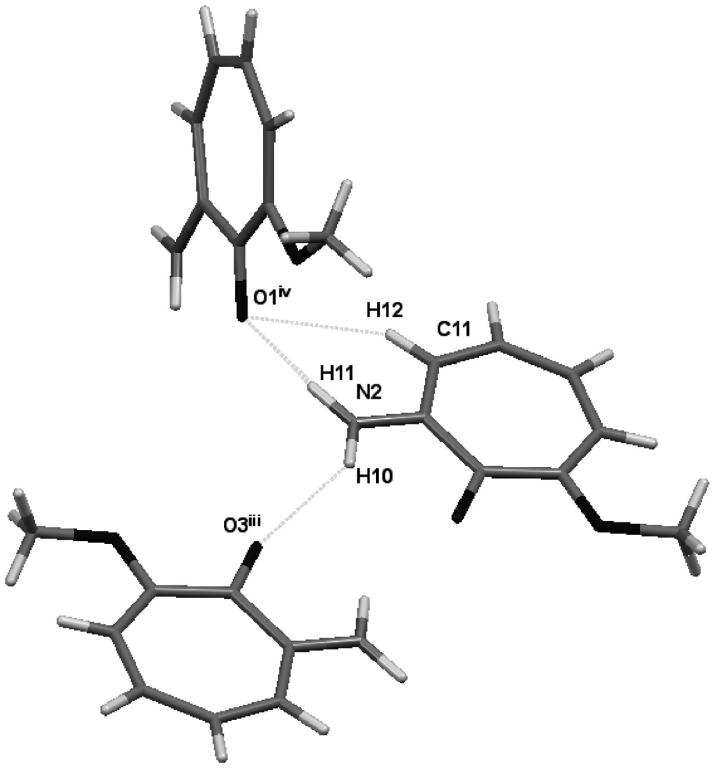

Fig. 4. Intermolecular N—H···O hydrogen bonds and C—H···O interactions of **1b**. Symmetry codes: (iii) 1-x, 1-y, 1-z, (iv) -x, 1-y, -z.

Table 6. Hydrogen bond geometry (Å,°) of **1**

D	H	A	D-H	H···A	D···A	D-H···A	Symmetry codes
N1	H1	O1	0.95(2)	2.13(2)	2.550(2)	105(2)	
N1	H1	O1^{i}	0.95(2)	2.24(2)	3.060(2)	144(2)	(i) -1-x, -y, -1-z
N1	H2	O3^{ii}	0.89(2)	2.14(2)	3.005(2)	165(2)	(ii) -x, -y, -z
N2	H10	O3	0.96(2)	2.10(2)	2.567(2)	109(2)	
N2	H10	O3^{iii}	0.96(2)	2.26(2)	3.034(2)	136(2)	(iii) 1-x, 1-y, 1-z
N2	H11	O1^{iv}	1.01(3)	1.93(3)	2.930(2)	171(2)	(iv) -x, 1-y, -z
N2	H11	C1^{iv}	1.01(3)	2.90(3)	3.850(3)	159(2)	(iv) -x, 1-y, -z
C8	H9	C10^{v}	0.960	2.826	3.690(3)	150	(v) -x, 1-y, 1-z
C11	H12	O1^{iv}	0.930	2.647	3.435(3)	143	(iv) -x, 1-y, -z

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