Crystal structure of triethylammonium 3-carboxy-7-oxa-bicyclo-[2.2.1]-hept-5-ene-2-carboxylate urea monosolvate, $(C_6H_{16}N)^+(C_8H_7O_5)^-\cdot CH_4N_2O$, $C_{15}H_{27}N_3O_6$

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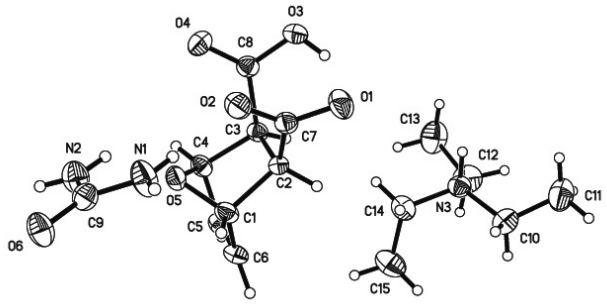

Abstract

$C_{15}H_{27}N_3O_6$, monoclinic, $P2_1/c$ (no. 14), a = 14.293(1) Å, b = 7.6426(6) Å, c = 16.346(2) Å, β = 96.326(1)°, V = 1774.6 Å^3, Z = 4, $R_{gt}(F)$ = 0.0481, $wR_{ref}(F^2)$ = 0.1494, T = 298 K.

Source of material

All the reagents and solvents from commercial sources were used without further purification. A mixture of exo-7-oxa-bicyclo[2,2,1]hept-5-ene-2,3-dicarboxylic anhydride (0.332 g, 2 mmol), urea (0.120 g, 2 mmol) and 5 drops of triethylamine in methanol (10 mL) was stirred for 5 h at room temperature, and then refluxed for 1 h. After cooling, the solution was left for crystallization at room temperature. The single crystal suitable for *X*-ray determination was obtained by evaporation from the methanol solution within 5 d.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.20×0.40×0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.00 cm^{-1}
Diffractometer, scan mode:	CCD area detector, φ and ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8552, 3107
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1667
$N(param)_{refined}$:	220
Programs:	SHELXS-97 [3]

Discussion

7-Oxa-bicyclo-[2,2,1]-hept-5-ene-2,3-dicarboximide and its *N*-substituent compounds have recently become an intense research topic in heterocyclic chemistry because of their antitumor, antivirus, analgesic, sedative and fungicidal activities [1,2]. In the present work, the reaction of exo-7-oxa-bicyclo-[2,2,1]-hept-5-ene-2,3-dicarboxylic anhydride and urea in methanol is expected to form 7-oxa-bicyclo-[2,2,1]-hept-5-ene-2,3-dicarboximide, but instead the title compound formed. In this paper, the crystal structure of the title compound is reported.

The asymmetric unit of the title compound contains one triethylammonium cation, one 3-carboxy-7-oxa-bicyclo-[2.2.1]-hept-5-ene-2-carboxylate anion and one urea solvent molecule. In the anion of the title compound, the cyclohexane ring tends towards a boat conformation, and the tetrahydrofuran ring and the dihydrofuran ring adopt envelope conformations. The dihedral angles formed by the plane (C1/C2/C3/C4) and the mean planes of the carboxylate and methoxycarbonyl groups are 77.3(2) and 46.0(2)°, respectively. The bond lengths and angles are in expected ranges. The crystal structure is stabilized by N–H···O and O–H···O hydrogen bonds.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.9290	0.4288	0.6961	0.081
H(1B)	4*e*	0.8573	0.5323	0.6435	0.081
H(2A)	4*e*	0.9400	0.9241	0.6844	0.074
H(2B)	4*e*	0.8636	0.8224	0.6366	0.074
H(3)	4*e*	0.2289	0.5699	0.5343	0.060
H(3A)	4*e*	0.5104	0.6498	0.4434	0.085
H(1)	4*e*	0.7071	0.6672	0.7155	0.054
H(2)	4*e*	0.5460	0.6016	0.6316	0.044
H(3B)	4*e*	0.5035	0.8324	0.5526	0.044
H(4)	4*e*	0.6298	1.0733	0.5775	0.049
H(5)	4*e*	0.5299	1.0712	0.6937	0.064
H(6)	4*e*	0.5794	0.8195	0.7802	0.067
H(10A)	4*e*	0.2447	0.4046	0.6550	0.086
H(10B)	4*e*	0.1542	0.5000	0.6776	0.086
H(11A)	4*e*	0.0659	0.3724	0.5713	0.131
H(11B)	4*e*	0.1229	0.2251	0.6214	0.131
H(11C)	4*e*	0.1555	0.2921	0.5385	0.131
H(12A)	4*e*	0.1072	0.7853	0.6025	0.093
H(12B)	4*e*	0.0734	0.6457	0.5355	0.093
H(13A)	4*e*	0.1864	0.9345	0.5101	0.132
H(13B)	4*e*	0.0817	0.9093	0.4721	0.132
H(13C)	4*e*	0.1610	0.7900	0.4432	0.132
H(14A)	4*e*	0.3105	0.8010	0.5860	0.072
H(14B)	4*e*	0.3438	0.6322	0.6350	0.072
H(15A)	4*e*	0.2245	0.8862	0.6912	0.137
H(15B)	4*e*	0.3298	0.8565	0.7264	0.137
H(15C)	4*e*	0.2538	0.7145	0.7399	0.137

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Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	4*e*	0.9072(2)	0.5281(4)	0.6781(2)	0.057(2)	0.059(2)	0.082(2)	–0.005(2)	–0.020(2)	–0.001(2)
N(2)	4*e*	0.9137(2)	0.8255(3)	0.6710(2)	0.050(2)	0.060(2)	0.072(2)	0.003(1)	–0.009(1)	0.006(2)
N(3)	4*e*	0.2094(2)	0.6141(3)	0.5813(2)	0.046(2)	0.057(2)	0.047(2)	0.003(1)	0.006(1)	–0.002(1)
O(1)	4*e*	0.6072(2)	0.3835(3)	0.5391(1)	0.061(1)	0.045(1)	0.070(2)	–0.005(1)	0.005(1)	–0.017(1)
O(2)	4*e*	0.7376(2)	0.5370(3)	0.5587(1)	0.044(1)	0.066(1)	0.055(1)	0.002(1)	0.005(1)	–0.012(1)
O(3)	4*e*	0.5510(2)	0.6970(3)	0.4193(1)	0.072(2)	0.065(1)	0.034(1)	–0.013(1)	0.007(1)	–0.005(1)
O(4)	4*e*	0.6627(2)	0.8886(3)	0.4455(1)	0.064(2)	0.080(2)	0.054(1)	–0.019(1)	0.014(1)	0.008(1)
O(5)	4*e*	0.7076(1)	0.8801(2)	0.6394(1)	0.041(1)	0.044(1)	0.047(1)	–0.007(1)	–0.0052(9)	0.0010(9)
O(6)	4*e*	1.0222(2)	0.6759(3)	0.7534(1)	0.046(1)	0.058(1)	0.071(2)	0.002(1)	–0.014(1)	–0.001(1)
C(1)	4*e*	0.6636(2)	0.7445(4)	0.6820(2)	0.056(2)	0.043(2)	0.033(2)	–0.002(2)	–0.006(1)	0.002(1)
C(2)	4*e*	0.6016(2)	0.6530(3)	0.6108(2)	0.041(2)	0.041(2)	0.029(1)	–0.006(1)	0.004(1)	0.001(1)
C(3)	4*e*	0.5718(2)	0.8163(3)	0.5555(2)	0.033(1)	0.043(2)	0.033(2)	0.004(1)	0.000(1)	–0.002(1)
C(4)	4*e*	0.6218(2)	0.9641(4)	0.6074(2)	0.047(2)	0.034(2)	0.041(2)	0.001(1)	–0.003(1)	–0.002(1)
C(5)	4*e*	0.5712(2)	0.9827(4)	0.6824(2)	0.060(2)	0.053(2)	0.048(2)	0.001(2)	0.005(2)	–0.018(2)
C(6)	4*e*	0.5978(2)	0.8465(4)	0.7289(2)	0.069(2)	0.064(2)	0.033(2)	–0.012(2)	0.006(2)	–0.014(2)
C(7)	4*e*	0.6531(2)	0.5145(4)	0.5670(2)	0.045(2)	0.041(2)	0.033(2)	–0.000(2)	–0.001(1)	0.006(1)
C(8)	4*e*	0.6002(2)	0.8046(4)	0.4696(2)	0.047(2)	0.042(2)	0.037(2)	0.002(2)	0.002(1)	0.003(1)
C(9)	4*e*	0.9507(2)	0.6760(4)	0.7039(2)	0.039(2)	0.059(2)	0.054(2)	0.001(2)	0.009(2)	–0.003(2)
C(10)	4*e*	0.1863(3)	0.4588(4)	0.6320(2)	0.080(3)	0.070(2)	0.065(2)	–0.010(2)	0.014(2)	0.008(2)
C(11)	4*e*	0.1276(3)	0.3256(5)	0.5869(3)	0.087(3)	0.068(2)	0.109(3)	–0.013(2)	0.024(3)	0.002(2)
C(12)	4*e*	0.1251(3)	0.7224(5)	0.5551(2)	0.057(2)	0.090(3)	0.084(3)	0.022(2)	0.006(2)	0.005(2)
C(13)	4*e*	0.1399(3)	0.8504(5)	0.4893(3)	0.089(3)	0.059(2)	0.115(3)	0.009(2)	0.004(3)	0.021(2)
C(14)	4*e*	0.2915(2)	0.7127(4)	0.6235(2)	0.058(2)	0.061(2)	0.060(2)	–0.000(2)	0.001(2)	–0.008(2)
C(15)	4*e*	0.2732(3)	0.8004(5)	0.7023(2)	0.110(3)	0.088(3)	0.074(3)	–0.001(3)	0.006(2)	–0.031(2)

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