Crystal structure of triethyl(5-benzoyl-1*H*-pyrrol-2-yl)methane-tricarboxylate, $C_{21}H_{23}NO_7$

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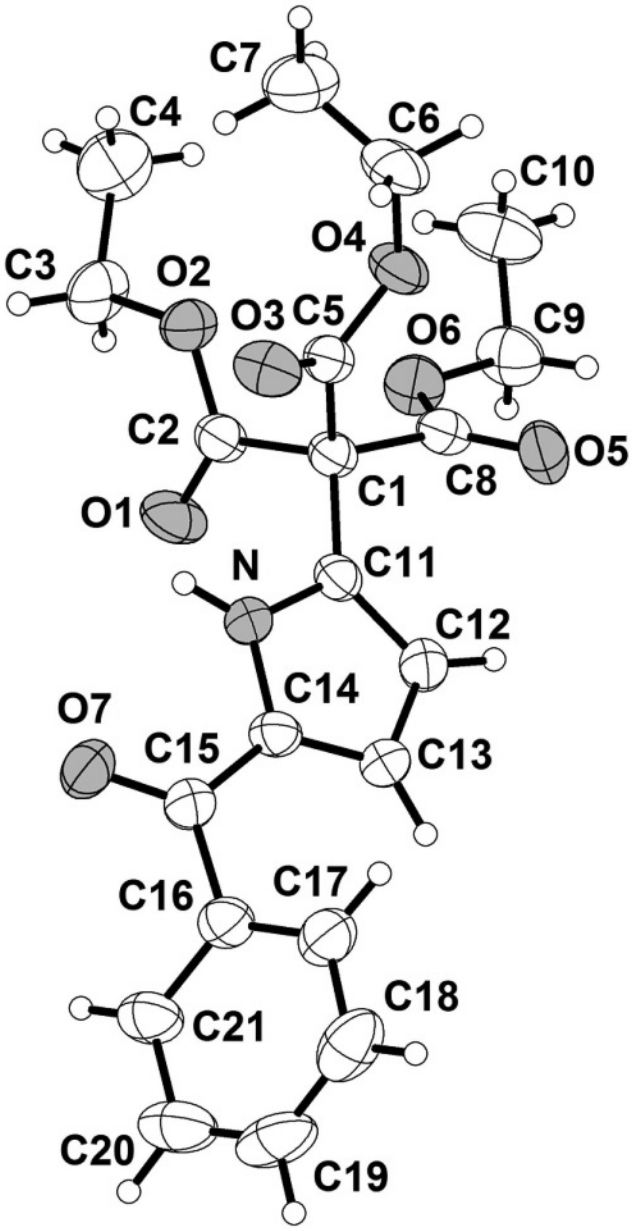

Abstract

$C_{21}H_{23}NO_7$, triclinic, $P\bar{1}$ (no. 2), a = 8.369(2) Å, b = 10.984(2) Å, c = 11.900(2) Å, α = 84.12(3)°, β = 72.05(3)°, γ = 79.81(3)°, V = 1022.9 Å^3, Z = 2, $R_{gt}(F)$ = 0.0584, $wR_{ref}(F^2)$ = 0.1739, T = 293 K.

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Source of material

To a solution of manganese(II) acetate tetrahydrate (6.13 g, 25 mmol) in acetic acid (50mL) was added potassium permanganate (0.99 g, 6.3 mmol). The mixture was stirred at 80 °C for 10 minutes. To the mixture was added acetic anhydride (9.68 g, 75 mmol). The mixture was stirred at 80 °C for 30 minutes and was then cooled to 60 °C. To the reaction mixture was then added sodium acetate (1.64 g, 20 mmol), 2-benzoylpyrrole (1.71 g, 10 mmol) and triethyl methane-tricarboxylate (2.55 g, 11 mmol). The reaction mixture was stirred at 60 °C for 24 hours. The mixture was poured into water (50mL) and extracted with toluene (3×25 mL). The combined toluene extracts were concentrated under reduced pressure. The residue was purified by flash chromatography on silica eluting with hexane/ethyl acetate (90:10) to provide 3.5 g (86%) of the title compound which crystallized upon standing. The product can be further purified by recrystallization from methanol. Elemental analysis calcd. for $C_{21}H_{23}NO_7$: C 62.83, H 5.77, N 3.49%; found C: 61.69, H 5.62, N 3.30%.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.10×0.20×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.98 cm^{-1}
Diffractometer, scan mode:	Nonius cad4, $\omega 2\backslash\theta$
$2\theta_{max}$:	50.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4022, 3744
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2464
$N(param)_{refined}$:	262
Programs:	SHELX [3]

Experimental details

H atoms were positioned geometrically with C–H = 0.93, 0.97 and 0.96 Å for aromatic, methylene and methyl, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (or 1.5 for methyl groups) times U_{eq}(C).

Discussion

Triethyl(5-benzoyl-1*H*-pyrrol-2-yl)-methanetricarboxylate is an important intermediate in the synthesis of ketorolac, which is a an analgesic anti-inflammatory drug [1, 2]. We report herein the synthesis and crystal structure of the title compound. In the crystal structure, intermolecular N–H···O hydrogen bonds link two molecules into dimers.

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(0A)	2*i*	–0.0482	0.1388	0.4356	0.053
H(3A)	2*i*	0.5112	0.2418	0.3084	0.079
H(3B)	2*i*	0.4517	0.3773	0.3529	0.079
H(4A)	2*i*	0.6895	0.3723	0.1886	0.136
H(4B)	2*i*	0.5339	0.4566	0.1596	0.136
H(4C)	2*i*	0.5915	0.3220	0.1146	0.136
H(6A)	2*i*	0.0840	0.3857	–0.0581	0.083
H(6B)	2*i*	0.0640	0.2529	0.0017	0.083
H(7A)	2*i*	0.3360	0.2536	–0.1209	0.131
H(7B)	2*i*	0.3400	0.2259	0.0101	0.131
H(7C)	2*i*	0.3598	0.3584	–0.0501	0.131
H(9A)	2*i*	–0.0430	0.7221	0.2521	0.077
H(9B)	2*i*	0.0535	0.7361	0.3431	0.077
H(10A)	2*i*	0.1893	0.8158	0.1613	0.141
H(10B)	2*i*	0.2109	0.6875	0.1071	0.141
H(10C)	2*i*	0.3073	0.7014	0.1979	0.141
H(12A)	2*i*	–0.3426	0.4598	0.4705	0.058
H(13A)	2*i*	–0.5003	0.3045	0.6087	0.058
H(17A)	2*i*	–0.6153	0.0994	0.5837	0.072
H(18A)	2*i*	–0.8640	0.0400	0.7090	0.093
H(19A)	2*i*	–0.8726	–0.0397	0.8966	0.099
H(20A)	2*i*	–0.6316	–0.0742	0.9556	0.095
H(21A)	2*i*	–0.3763	–0.0344	0.8252	0.074

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	2*i*	–0.1351(2)	0.1949(2)	0.4592(2)	0.036(1)	0.047(1)	0.044(1)	–0.0038(9)	–0.0074(9)	0.0051(9)
O(1)	2*i*	0.1656(2)	0.3317(2)	0.4627(2)	0.060(1)	0.110(2)	0.039(1)	–0.024(1)	–0.0236(9)	0.011(1)
C(1)	2*i*	0.0008(3)	0.3623(2)	0.3241(2)	0.039(1)	0.047(1)	0.034(1)	–0.010(1)	–0.012(1)	0.003(1)
O(2)	2*i*	0.3009(2)	0.3272(2)	0.2684(2)	0.038(1)	0.073(1)	0.044(1)	–0.0111(8)	–0.0149(8)	–0.0047(8)
C(2)	2*i*	0.1652(3)	0.3371(2)	0.3621(2)	0.042(1)	0.054(2)	0.040(1)	–0.012(1)	–0.014(1)	0.006(1)
O(3)	2*i*	0.0417(3)	0.1874(2)	0.2039(2)	0.077(1)	0.057(1)	0.050(1)	–0.021(1)	–0.013(1)	–0.0026(9)
C(3)	2*i*	0.4649(3)	0.3256(3)	0.2882(3)	0.039(2)	0.098(2)	0.070(2)	–0.013(2)	–0.025(1)	–0.008(2)
O(4)	2*i*	0.0670(2)	0.3760(2)	0.1150(2)	0.072(1)	0.062(1)	0.0337(9)	–0.0099(9)	–0.0166(9)	0.0042(8)
C(4)	2*i*	0.5800(4)	0.3733(4)	0.1782(4)	0.057(2)	0.110(3)	0.108(3)	–0.036(2)	–0.023(2)	0.013(2)
O(5)	2*i*	–0.1760(2)	0.5508(2)	0.2834(2)	0.056(1)	0.056(1)	0.066(1)	–0.0083(9)	–0.029(1)	0.0130(9)
C(5)	2*i*	0.0382(3)	0.2962(2)	0.2071(2)	0.038(1)	0.052(2)	0.038(1)	–0.011(1)	–0.013(1)	0.002(1)
O(6)	2*i*	0.0823(2)	0.5612(2)	0.2976(2)	0.055(1)	0.050(1)	0.067(1)	–0.0181(9)	–0.0172(9)	0.0013(9)
C(6)	2*i*	0.1195(4)	0.3243(3)	–0.0024(2)	0.093(2)	0.081(2)	0.035(1)	–0.013(2)	–0.022(2)	0.000(1)
O(7)	2*i*	–0.1795(3)	–0.0251(2)	0.5972(2)	0.050(1)	0.075(2)	0.099(2)	0.011(1)	0.010(1)	0.033(1)
C(7)	2*i*	0.3049(5)	0.2873(4)	–0.0445(3)	0.090(3)	0.097(3)	0.059(2)	–0.007(2)	–0.002(2)	–0.012(2)
C(8)	2*i*	–0.0440(3)	0.5030(2)	0.2998(2)	0.047(2)	0.052(2)	0.035(1)	–0.014(1)	–0.011(1)	0.003(1)
C(9)	2*i*	0.0614(4)	0.6948(3)	0.2730(3)	0.079(2)	0.047(2)	0.065(2)	–0.021(1)	–0.014(2)	0.001(1)
C(10)	2*i*	0.2043(5)	0.7277(4)	0.1767(3)	0.107(3)	0.095(3)	0.074(2)	–0.047(2)	–0.009(2)	0.022(2)
C(11)	2*i*	–0.1454(3)	0.3150(2)	0.4184(2)	0.040(1)	0.045(1)	0.035(1)	–0.008(1)	–0.014(1)	0.004(1)
C(12)	2*i*	–0.3025(3)	0.3760(2)	0.4795(2)	0.043(2)	0.046(2)	0.052(2)	–0.004(1)	–0.013(1)	0.006(1)
C(13)	2*i*	–0.3907(3)	0.2892(2)	0.5573(2)	0.036(1)	0.057(2)	0.045(1)	–0.004(1)	–0.007(1)	0.004(1)
C(14)	2*i*	–0.2841(3)	0.1762(2)	0.5436(2)	0.039(1)	0.049(2)	0.039(1)	–0.012(1)	–0.007(1)	0.004(1)
C(15)	2*i*	–0.3010(3)	0.0578(3)	0.6080(2)	0.043(2)	0.053(2)	0.053(2)	–0.004(1)	–0.007(1)	0.007(1)
C(16)	2*i*	–0.4703(3)	0.0369(2)	0.6912(2)	0.049(2)	0.040(1)	0.053(2)	–0.012(1)	–0.006(1)	0.002(1)
C(17)	2*i*	–0.6172(3)	0.0611(3)	0.6575(3)	0.050(2)	0.056(2)	0.067(2)	–0.006(1)	–0.008(1)	–0.003(1)
C(18)	2*i*	–0.7666(4)	0.0286(3)	0.7333(4)	0.049(2)	0.061(2)	0.116(3)	–0.013(2)	–0.012(2)	–0.011(2)
C(19)	2*i*	–0.7709(5)	–0.0207(3)	0.8445(4)	0.065(2)	0.067(2)	0.092(3)	–0.027(2)	0.024(2)	–0.007(2)
C(20)	2*i*	–0.6268(5)	–0.0421(3)	0.8795(3)	0.092(3)	0.071(2)	0.058(2)	–0.028(2)	0.008(2)	0.004(2)
C(21)	2*i*	–0.4752(4)	–0.0163(3)	0.8026(2)	0.065(2)	0.057(2)	0.055(2)	–0.021(1)	–0.004(1)	0.007(1)

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