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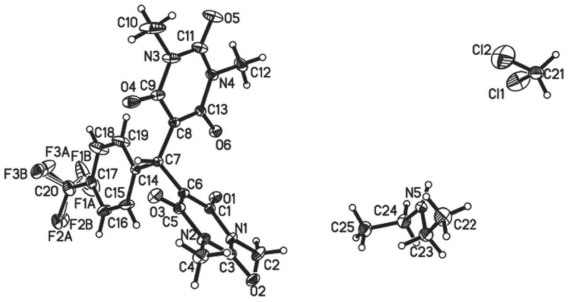
Crystal structure of 5,5′-((4-(trifluoromethyl)-phenyl)methylene)bis(1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione) – diethylamine – dichloromethane (1/1/1) $C_{25}H_{32}Cl_2F_3N_5O_6$



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Abstract

$C_{25}H_{32}Cl_2F_3N_5O_6$, orthorhombic, $P2_12_12_1$ (no. 19) $a = 11.0768(4)$ Å, $b = 15.6620(5)$ Å, $c = 16.6830(5)$ Å, $V = 2894.25(16)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0509$, $wR(F_2) = 0.1498$, $T = 100$ K.

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Table 1: Data collection and handling.

Crystal:	Pink, rod, size 0.24×0.34×0.69 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.92 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8 Venture, φ and ω scans
$2\theta_{max}$:	65.26°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	93047, 10576
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 9190
$N(param)_{refined}$:	398
Programs:	SHELX [12]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*a*	0.7279	0.1218	0.3115	0.048
H(2B)	4*a*	0.8623	0.1542	0.2937	0.048
H(2C)	4*a*	0.7598	0.2211	0.3185	0.048
H(4A)	4*a*	1.0428	0.2326	0.6131	0.048
H(4B)	4*a*	1.0042	0.3073	0.5529	0.048
H(4C)	4*a*	1.1061	0.2403	0.5272	0.048
H(7A)	4*a*	0.7821	0.0144	0.6398	0.020
H(10A)	4*a*	0.5554	0.0607	0.8536	0.091
H(10B)	4*a*	0.4198	0.0255	0.8497	0.091
H(10C)	4*a*	0.4451	0.1260	0.8452	0.091
H(12A)	4*a*	0.3008	0.0597	0.5124	0.050
H(12B)	4*a*	0.2562	0.1318	0.5736	0.050
H(12C)	4*a*	0.2187	0.0342	0.5876	0.050
H(15A)	4*a*	0.8810	−0.0635	0.4896	0.042
H(16A)	4*a*	0.8997	−0.2069	0.4542	0.045
H(18A)	4*a*	0.6115	−0.2633	0.5912	0.061
H(19A)	4*a*	0.5929	−0.1201	0.6256	0.051
H(5A)	4*a*	0.3600	0.4522	0.2278	0.027
H(21A)	4*a*	−0.1252	0.7719	0.1636	0.048
H(21B)	4*a*	−0.2595	0.7759	0.1987	0.048
H(22A)	4*a*	0.5993	0.5943	0.2710	0.070
H(22B)	4*a*	0.5053	0.5374	0.3205	0.070
H(22C)	4*a*	0.4583	0.5991	0.2507	0.070
H(23A)	4*a*	0.5592	0.5145	0.1546	0.036
H(23B)	4*a*	0.6063	0.4526	0.2244	0.036
H(24A)	4*a*	0.4659	0.3826	0.0943	0.039
H(24B)	4*a*	0.3513	0.3411	0.1379	0.039
H(25A)	4*a*	0.5154	0.2458	0.1425	0.064
H(25B)	4*a*	0.4806	0.2742	0.2317	0.064
H(25C)	4*a*	0.5952	0.3157	0.1882	0.064

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(1A)[a]	4*a*	0.681(2)	−0.361(2)	0.435(2)	0.077(6)	0.038(6)	0.10(1)	0.001(4)	−0.023(6)	−0.036(7)
F(2A)[a]	4*a*	0.874(1)	−0.3624(9)	0.460(1)	0.037(4)	0.017(3)	0.045(4)	0.012(2)	−0.001(3)	0.000(2)
F(3A)[a]	4*a*	0.747(4)	−0.392(1)	0.550(2)	0.15(1)	0.017(3)	0.077(9)	0.012(7)	0.070(8)	0.007(4)
F(1B)[b]	4*a*	0.6684(9)	−0.3688(7)	0.4590(9)	0.076(4)	0.035(2)	0.129(7)	0.018(3)	−0.066(4)	−0.034(4)
F(2B)[b]	4*a*	0.859(1)	−0.3588(5)	0.4530(5)	0.085(4)	0.031(2)	0.044(2)	0.026(2)	0.029(3)	0.002(2)
F(3B)[b]	4*a*	0.7733(5)	−0.3919(5)	0.5630(4)	0.055(3)	0.022(1)	0.041(2)	0.0053(9)	0.009(2)	0.0066(9)
O(1)	4*a*	0.6762(1)	0.04905(7)	0.42100(6)	0.0242(5)	0.0249(5)	0.0184(4)	−0.0059(4)	−0.0001(4)	−0.0021(4)
O(2)	4*a*	0.9561(1)	0.26080(8)	0.40294(8)	0.0316(6)	0.0222(5)	0.0305(6)	−0.0058(4)	0.0068(5)	0.0057(4)
O(3)	4*a*	0.9136(1)	0.12902(9)	0.64371(7)	0.0293(6)	0.0335(6)	0.0232(5)	−0.0113(5)	−0.0061(4)	0.0027(4)
O(4)	4*a*	0.6846(1)	0.0423(1)	0.75685(8)	0.0265(6)	0.066(1)	0.0187(5)	−0.0060(6)	0.0006(4)	−0.0025(6)
O(5)	4*a*	0.2842(1)	0.0962(1)	0.7278(1)	0.0250(6)	0.0536(9)	0.057(1)	−0.0012(6)	0.0171(6)	−0.0154(8)
O(6)	4*a*	0.4942(1)	0.03307(8)	0.50329(7)	0.0226(5)	0.0317(6)	0.0217(5)	−0.0034(4)	−0.0025(4)	0.0007(4)
N(1)	4*a*	0.8216(1)	0.15118(8)	0.41183(8)	0.0274(6)	0.0191(5)	0.0175(5)	−0.0032(4)	0.0028(5)	0.0020(4)
N(2)	4*a*	0.9373(1)	0.19206(8)	0.52214(8)	0.0238(6)	0.0208(5)	0.0235(6)	−0.0072(4)	0.0014(4)	0.0008(4)
N(3)	4*a*	0.4839(1)	0.0639(1)	0.74229(9)	0.0246(6)	0.0472(9)	0.0235(6)	−0.0067(6)	0.0092(5)	−0.0116(6)
N(4)	4*a*	0.3913(1)	0.05934(9)	0.61702(9)	0.0160(5)	0.0214(5)	0.0332(7)	−0.0012(4)	0.0033(5)	−0.0009(5)
C(1)	4*a*	0.7589(1)	0.09136(8)	0.45723(8)	0.0204(6)	0.0165(5)	0.0181(5)	−0.0004(4)	0.0026(5)	−0.0005(4)
C(2)	4*a*	0.7903(2)	0.1631(1)	0.3267(1)	0.044(1)	0.0339(8)	0.0180(7)	−0.0069(7)	0.0003(6)	0.0053(6)
C(3)	4*a*	0.9083(1)	0.20448(9)	0.44287(9)	0.0234(6)	0.0164(5)	0.0251(6)	0.0007(5)	0.0049(5)	0.0007(5)
C(4)	4*a*	1.0303(2)	0.2477(1)	0.5567(1)	0.0300(8)	0.0289(8)	0.0374(9)	−0.0141(6)	−0.0028(7)	0.0006(7)
C(5)	4*a*	0.8801(1)	0.13240(9)	0.57204(9)	0.0202(6)	0.0182(5)	0.0215(6)	−0.0031(5)	0.0010(5)	0.0001(5)
C(6)	4*a*	0.7884(1)	0.08112(8)	0.53768(8)	0.0168(5)	0.0165(5)	0.0174(5)	−0.0027(4)	0.0021(4)	−0.0006(4)
C(7)	4*a*	0.7289(1)	0.01524(8)	0.59144(8)	0.0168(5)	0.0175(5)	0.0169(5)	−0.0014(4)	0.0020(4)	0.0001(4)
C(8)	4*a*	0.6058(1)	0.03886(9)	0.62547(8)	0.0182(5)	0.0172(5)	0.0185(6)	−0.0018(4)	0.0026(4)	−0.0017(4)
C(9)	4*a*	0.5982(1)	0.0483(1)	0.71029(9)	0.0221(6)	0.0296(7)	0.0198(6)	−0.0048(5)	0.0045(5)	−0.0030(5)
C(10)	4*a*	0.4753(2)	0.0695(3)	0.8300(1)	0.042(1)	0.110(2)	0.0295(9)	−0.016(1)	0.0166(9)	−0.026(1)
C(11)	4*a*	0.3807(2)	0.0746(1)	0.6979(1)	0.0217(6)	0.0290(7)	0.0372(8)	−0.0035(6)	0.0099(6)	−0.0081(6)
C(12)	4*a*	0.2828(2)	0.0723(1)	0.5687(1)	0.0183(6)	0.0353(8)	0.047(1)	0.0019(6)	−0.0022(6)	0.0013(7)
C(13)	4*a*	0.5016(1)	0.04370(9)	0.58100(9)	0.0173(5)	0.0154(5)	0.0241(6)	−0.0020(4)	0.0017(5)	0.0000(5)
C(14)	4*a*	0.7346(1)	−0.07684(8)	0.56113(8)	0.0174(5)	0.0169(5)	0.0189(5)	0.0011(4)	0.0020(4)	0.0013(4)
C(15)	4*a*	0.8252(2)	−0.1040(1)	0.5104(1)	0.0402(9)	0.0207(6)	0.044(1)	0.0022(6)	0.0259(8)	0.0026(6)
C(16)	4*a*	0.8364(2)	−0.1896(1)	0.4891(1)	0.052(1)	0.0211(7)	0.0399(9)	0.0070(7)	0.0271(9)	0.0025(7)
C(17)	4*a*	0.7563(2)	−0.24911(9)	0.5182(1)	0.0282(7)	0.0186(6)	0.0319(8)	0.0051(5)	−0.0014(6)	−0.0001(5)
C(18)	4*a*	0.6664(2)	−0.2227(1)	0.5697(2)	0.0346(9)	0.0170(7)	0.101(2)	−0.0054(6)	0.033(1)	−0.0078(9)
C(19)	4*a*	0.6558(2)	−0.1373(1)	0.5904(2)	0.0327(9)	0.0186(6)	0.076(2)	−0.0026(6)	0.030(1)	−0.0051(8)
C(20)	4*a*	0.7652(2)	−0.3413(1)	0.4959(1)	0.042(1)	0.0211(7)	0.045(1)	0.0056(7)	−0.0017(8)	−0.0049(7)
Cl(1)	4*a*	−0.1372(1)	0.70119(9)	0.28151(5)	0.1090(8)	0.1267(9)	0.0392(3)	0.0061(7)	−0.0203(4)	0.0129(4)
Cl(2)	4*a*	−0.23235(9)	0.65568(6)	0.12563(5)	0.0857(6)	0.0799(5)	0.0596(4)	−0.0366(5)	−0.0005(4)	−0.0122(4)
N(5)	4*a*	0.4261(1)	0.43903(9)	0.20135(8)	0.0204(5)	0.0235(6)	0.0244(6)	0.0010(4)	0.0012(4)	0.0006(5)
C(21)	4*a*	−0.1895(2)	0.7378(2)	0.1897(1)	0.0366(9)	0.049(1)	0.0348(9)	0.0035(8)	0.0062(8)	0.0020(8)
C(22)	4*a*	0.5242(2)	0.5615(2)	0.2678(2)	0.047(1)	0.0330(9)	0.060(1)	−0.0085(9)	−0.004(1)	−0.0096(9)
C(23)	4*a*	0.5389(2)	0.4902(1)	0.2077(1)	0.0245(7)	0.0348(8)	0.0318(8)	−0.0033(6)	−0.0037(6)	0.0018(7)
C(24)	4*a*	0.4335(2)	0.3640(1)	0.1468(1)	0.0431(9)	0.0297(8)	0.0241(7)	0.0008(7)	0.0004(7)	−0.0042(6)
C(25)	4*a*	0.5133(3)	0.2937(1)	0.1802(1)	0.064(1)	0.0278(8)	0.037(1)	0.0114(9)	0.0122(9)	−0.0005(7)

[a]Disordered, occupancy factor: 0.31; [b]Disordered, occupancy factor: 0.69.

The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

The chemical reagents and solvents used in this study are commercially available. The synthesis of the title compound follows a published protocol [1].

Yield: 90%; m.p. 120 °C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ7.55 (2H, d, $J = 8.8$ Hz, C_6H_4), 7.15 (2H, d, $J = 8.8$ Hz, C_6H_4), 5.32 (1H, t, $J = 6.6$ Hz, C_6H_4CH), 4.22 (2H, d, $J = 6.6$ Hz, COCHCO), 3.35 (6H, s, $2CH_3$), 3.28 (s, 6H, $2CH_3$), 3.04 (4H, q, $J = 7.3$ Hz, CH_2CH_3), 1.30 (6H, t, $J = 7.3$ Hz, CH_2CH_3); ^{13}C-NMR (100 MHz, $CDCl_3$): δ169.5, 155.7, 153.1, 150.0, 138.9, 136.5, 133.0, 131.0, 130.3, 127.0, 122.0, 49.6, 39.1, 29.0, 28.8, 28.5, 28.4, 11.9.

Experimental details

The trifuoromethyl group (C20) is disordered over two positions with an occupancy ratio of 0.69:0.31 (see the figure).

Discussion

The pyrimidine-trione moiety was found to be a key pharmacophore of many biological and therapeutic targets. Thus, several substituted pyrimidine-triones were reported to exhibit marked biological activities such as HIV-1 and HIV-2 protease inhibitors, anticonvulsant sedative-hypnotic, antioxidant, anti-inflammatory, and anticancer agents [2–11]. In the asymmetric unit of the title structure one pyrimidine-trione core, a diethylamine and a dichloromethane molecule are present. The target molecule contains three planar rings: N1/N2/C1/C3/C5/C6, N3/N4/C8—C9/C11/C13 and C14—C19. The dihedral angle between two pyrimidine rings (N1/N2/C1/C3/C5/C6 and N3/N4/C8—C9/C11/C13) was found to be 75.87(3)°. The phenyl ring appeared to be twisted with an angles of 71.08(2)° and 81.93(2)° with respect to the planes of two pyrimidine-trione rings (N1/N2/C1/C3/C5/C6 and N3/N4/C8—C9/C11/C13), respectively. The diethylamine ($NHEt_2$) and dichloromethane (DCM) solvate molecules (Figure) play a crucial role in the stabilization of the crystal structure. In the structure, the target molecule is connected to the diethylamine by a N—H···O hydrogen bond.

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