Crystal structure of dimethyl 2-(*tert*-butylamino)-5,7,7-trimethyl-5,6,7,8-tetrahydro-4*H*-chromene-3,4-dicarboxylate, $C_{20}H_{30}NO_5$

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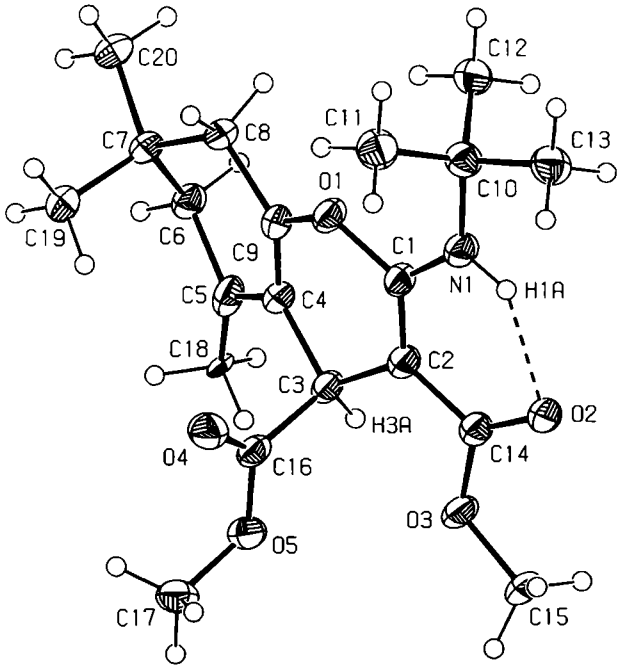

Abstract

$C_{20}H_{30}NO_5$, monoclinic, $P12_1/c1$ (no. 14),
$a = 13.454(5)$ Å, $b = 8.999(4)$ Å, $c = 15.719(6)$ Å,
$\beta = 90.232(9)°$, $V = 1903.1$ Å^3, $Z = 4$,
$R_{gt}(F) = 0.073$, $wR_{ref}(F^2) = 0.161$, $T = 120$ K.

Source of material

The title compound was synthesized by the reaction of t-butyl isocyanide and dimethyl acetylene dicarboxylate with 3,3,5-trimethyl cyclohexanone in the presence of piperidine as a base in cold dichloromethane, then purified by column chromatography on silica-gel using a mixture of ethylacetate and hexane (*v/v* 30:70) as eluent. Recrystallization from methanol gave crystals suitable for X-ray structure analysis.

Discussion

Some of 2-amino-4*H*-pyran derivatives have fairly low stability, for example in the presence of little amounts of H^+ ions or NaOH, the ring transformation process take place and furan and pyridine derivatives are produced [1,2]. Some substituted 2-amino-4*H*-pyrans contain electron withdrawing groups, and fused heterocyclic rings are stable and have been shown to exhibit biological activities [3]. There are some new reports on the crystal structure of 2-aminio-4*H*-pyrans with electron withdrawing groups [4-8].

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The crystal structure analysis of the title compound was performed in view of its good stability. The molecule is stabilized by one intramolecular N–H···O type of hydrogen bonding. Details of N1–H1A···O2 are d(N1—H1A) = 0.88 Å; d(H1A···O2) = 2.05 Å, d(N1···O2) = 2.718 Å, ∠N1–H1A···O2 = 132.45°.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.07 × 0.11 × 0.13 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.91 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	52.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10717, 3694
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2201
$N(param)_{refined}$:	242
Program:	SHELXTL [9]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	0.1070	1.1143	0.3005	0.042
H(3A)	4*e*	0.3395	0.7789	0.3952	0.036
H(6A)	4*e*	0.5812	0.6596	0.1881	0.041
H(6B)	4*e*	0.5621	0.8345	0.1960	0.041
H(8A)	4*e*	0.4180	0.9517	0.1007	0.037
H(8B)	4*e*	0.3396	0.8339	0.0642	0.037
H(11A)	4*e*	0.0118	1.0259	0.1311	0.067
H(11B)	4*e*	0.1197	0.9979	0.0916	0.067
H(11C)	4*e*	0.0535	1.1341	0.0587	0.067
H(12A)	4*e*	0.2615	1.1771	0.1292	0.062
H(12B)	4*e*	0.2379	1.3174	0.1882	0.062
H(12C)	4*e*	0.1948	1.3105	0.0933	0.062
H(13A)	4*e*	−0.0177	1.2400	0.2267	0.064
H(13B)	4*e*	0.0223	1.3509	0.1549	0.064
H(13C)	4*e*	0.0704	1.3536	0.2482	0.064
H(15A)	4*e*	0.1336	0.7719	0.5907	0.063
H(15B)	4*e*	0.0641	0.8900	0.5429	0.063
H(15C)	4*e*	0.1620	0.9446	0.5918	0.063
H(17A)	4*e*	0.2340	0.3392	0.4567	0.062
H(17B)	4*e*	0.2353	0.3400	0.3549	0.062
H(17C)	4*e*	0.1429	0.4095	0.4047	0.062
H(18A)	4*e*	0.4336	0.6220	0.3659	0.027
H(18B)	4*e*	0.5446	0.6835	0.3563	0.027
H(18C)	4*e*	0.5110	0.5371	0.3061	0.027
H(19A)	4*e*	0.4780	0.5045	0.0924	0.057
H(19B)	4*e*	0.3892	0.5767	0.0385	0.057
H(19C)	4*e*	0.3788	0.5576	0.1393	0.057
H(20A)	4*e*	0.5713	0.8696	0.0432	0.059
H(20B)	4*e*	0.5030	0.7754	−0.0207	0.059
H(20C)	4*e*	0.5927	0.6965	0.0289	0.059

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}
O(1)	4*e*	0.2494(2)	0.9353(3)	0.1905(1)	0.037(1)	0.038(1)	0.028(1)	0.008(1)	0.007(1)	−0.001(1)
O(2)	4*e*	0.1159(2)	1.0500(3)	0.4251(1)	0.045(1)	0.034(1)	0.038(1)	0.007(1)	0.014(1)	−0.000(1)
O(3)	4*e*	0.1904(2)	0.8447(3)	0.4803(1)	0.045(2)	0.043(2)	0.027(1)	0.008(1)	0.014(1)	0.002(1)
O(4)	4*e*	0.1899(2)	0.5877(3)	0.2812(2)	0.056(2)	0.041(2)	0.037(1)	−0.005(1)	−0.003(1)	0.004(1)
O(5)	4*e*	0.2642(2)	0.5394(3)	0.4066(1)	0.044(2)	0.039(2)	0.032(1)	0.001(1)	0.006(1)	0.006(1)
N(1)	4*e*	0.1402(2)	1.0876(3)	0.2549(2)	0.037(2)	0.035(2)	0.032(1)	0.007(1)	0.008(1)	0.001(1)
C(1)	4*e*	0.2044(2)	0.9745(4)	0.2660(2)	0.033(2)	0.032(2)	0.029(2)	−0.002(2)	0.008(1)	−0.005(1)
C(2)	4*e*	0.2270(3)	0.9038(4)	0.3407(2)	0.034(2)	0.033(2)	0.028(2)	0.003(2)	0.005(1)	−0.001(1)
C(3)	4*e*	0.2984(2)	0.7742(4)	0.3421(2)	0.033(2)	0.034(2)	0.024(2)	0.003(2)	0.005(1)	−0.002(1)
C(4)	4*e*	0.3664(2)	0.7844(4)	0.2653(2)	0.033(2)	0.032(2)	0.026(2)	0.002(2)	0.007(1)	−0.001(1)
C(5)	4*e*	0.4634(3)	0.7100(4)	0.2672(2)	0.033(2)	0.041(2)	0.037(2)	0.000(2)	0.008(2)	−0.013(2)
C(6)	4*e*	0.5289(3)	0.7369(4)	0.1899(2)	0.032(2)	0.038(2)	0.032(2)	0.004(2)	0.008(1)	−0.002(2)
C(7)	4*e*	0.4706(3)	0.7343(4)	0.1061(2)	0.034(2)	0.035(2)	0.029(2)	0.001(2)	0.012(1)	−0.000(1)
C(8)	4*e*	0.3884(3)	0.8524(4)	0.1103(2)	0.036(2)	0.034(2)	0.023(2)	−0.001(2)	0.003(1)	0.000(1)
C(9)	4*e*	0.3356(2)	0.8520(4)	0.1943(2)	0.033(2)	0.030(2)	0.030(2)	0.004(2)	0.005(1)	−0.004(1)
C(10)	4*e*	0.1174(3)	1.1737(4)	0.1766(2)	0.038(2)	0.034(2)	0.036(2)	0.003(2)	0.001(2)	0.003(2)
C(11)	4*e*	0.0715(3)	1.0740(5)	0.1084(2)	0.047(2)	0.044(2)	0.043(2)	0.001(2)	−0.003(2)	0.001(2)
C(12)	4*e*	0.2114(3)	1.2518(4)	0.1439(2)	0.045(2)	0.036(2)	0.043(2)	0.001(2)	0.009(2)	0.003(2)
C(13)	4*e*	0.0413(3)	1.2900(4)	0.2041(2)	0.041(2)	0.039(2)	0.049(2)	0.007(2)	0.006(2)	0.005(2)
C(14)	4*e*	0.1725(2)	0.9426(4)	0.4167(2)	0.030(2)	0.034(2)	0.030(2)	−0.001(2)	0.003(1)	−0.003(1)
C(15)	4*e*	0.1328(3)	0.8644(4)	0.5577(2)	0.052(2)	0.044(2)	0.030(2)	0.005(2)	0.020(2)	−0.001(2)
C(16)	4*e*	0.2444(3)	0.6245(4)	0.3386(2)	0.034(2)	0.037(2)	0.025(2)	0.008(2)	0.008(1)	0.001(1)
C(17)	4*e*	0.2151(3)	0.3952(4)	0.4056(2)	0.040(2)	0.042(2)	0.042(2)	−0.000(2)	0.009(2)	0.011(2)
C(18)	4*e*	0.4894(2)	0.6346(3)	0.3267(2)	0.021(1)	0.025(2)	0.008(1)	0.016(1)	0.003(1)	0.004(1)
C(19)	4*e*	0.4250(3)	0.5792(4)	0.0929(2)	0.041(2)	0.037(2)	0.035(2)	0.001(2)	0.010(2)	−0.004(2)
C(20)	4*e*	0.5407(3)	0.7724(4)	0.0328(2)	0.039(2)	0.046(2)	0.034(2)	−0.001(2)	0.013(2)	0.000(2)

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