Crystal structure of 3-(2,6-dichlorophenyl)-4,5-dihydro-7H-pyrano[4,3-d]isoxazol-7-one, C₁₂H₇Cl₂NO₃

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Abstract

C₁₂H₇Cl₂NO₃, monoclinic, *P*12₁/*n*1 (No. 14), *a* = 7.499(4) Å, *b* = 14.450(4) Å, *c* = 21.559(3) Å, β = 96.89(2)°, *V* = 2319.3 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.054, *wR*_{ref}(*F*²) = 0.134, *T* = 293 K.

Source of material

The compound was prepared from the reaction of 2,6-dichlorobenzonitrile oxide with 3-bromo-5,6-dihydro-4*H*-pyran-2-one [1]. Crystals were isolated from an ethyl acetate/light petroleum solution of the compound (mp 379 K – 380 K).

Discussion

The structure was investigated in order to determine the regioisomers prepared in [1] and to show that the bromine in 3-bromo-5,6-dihydro-4*H*-pyran-2-one can be used as a steric auxiliary, to reverse the regioselectivity of nitrile oxide cycloaddition, as discussed in refs [2,3]. Two independent molecules comprise the asymmetric unit with the only significant dif-

ference between them being in the rotation about the C7—C71 bond. The structure has the carbonyl group lying to the opposite side of the molecule with reference to the aryl group.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.03 × 0.16 × 0.40 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71069 Å)
μ:	5.56 cm ^{−1}
Diffractometer, scan mode:	Rigaku AFC6R, ω/2θ
2θ _{max} :	50.6°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	4581, 4236
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 1626
<i>N</i> (<i>param</i>) _{refined} :	325
Programs:	teXsan [4], SHELXS-86 [5], SHELXL-97 [6], DIFABS [7], ORTEPII [8]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(4a1)	4e	−0.0406	0.5128	−0.1478	0.082
H(4a2)	4e	0.0093	0.6000	−0.1854	0.082
H(4b1)	4e	0.1794	0.9361	0.5485	0.073
H(4b2)	4e	0.1554	0.8803	0.4857	0.073
H(5a1)	4e	−0.1286	0.6531	−0.1033	0.069
H(5a2)	4e	−0.0001	0.5941	−0.0553	0.069
H(5b1)	4e	0.1369	0.7427	0.5375	0.061
H(5b2)	4e	0.3054	0.7947	0.5715	0.061
H(73a)	4e	−0.0845	0.9345	0.0932	0.061
H(73b)	4e	0.3587	0.4695	0.7207	0.069
H(74a)	4e	−0.3431	0.9775	0.0338	0.064
H(74b)	4e	0.6425	0.5266	0.7473	0.072
H(75a)	4e	−0.3950	0.9342	−0.0697	0.067
H(75b)	4e	0.6961	0.6826	0.7444	0.072

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cl(72a)	4e	0.2154(2)	0.8374(1)	0.07118(7)	0.073(1)	0.073(1)	0.065(1)	0.013(1)	−0.0094(9)	0.0006(9)
Cl(72b)	4e	0.0295(2)	0.5489(1)	0.68107(7)	0.068(1)	0.071(1)	0.073(1)	−0.0182(9)	0.0017(9)	−0.001(1)
Cl(76a)	4e	−0.1898(2)	0.8495(1)	−0.15026(7)	0.089(1)	0.080(1)	0.049(1)	0.001(1)	−0.0023(9)	−0.0037(9)
Cl(76b)	4e	0.4771(2)	0.8356(1)	0.71713(8)	0.061(1)	0.063(1)	0.110(2)	−0.0139(9)	−0.001(1)	0.014(1)
O(2a)	4e	0.5053(6)	0.5801(3)	−0.1273(2)	0.079(3)	0.084(3)	0.061(3)	0.030(3)	0.023(3)	−0.008(2)
O(2b)	4e	−0.3049(6)	0.9231(3)	0.5712(2)	0.055(3)	0.078(3)	0.105(4)	0.028(3)	0.008(3)	0.006(3)
O(3a)	4e	0.2218(7)	0.5382(3)	−0.1391(2)	0.103(4)	0.046(3)	0.066(3)	0.006(3)	0.029(3)	−0.012(2)
O(3b)	4e	−0.0708(6)	0.8995(3)	0.5218(2)	0.066(3)	0.058(3)	0.062(3)	0.019(2)	0.006(2)	0.009(2)
O(9a)	4e	0.3919(5)	0.7630(3)	−0.0994(2)	0.045(3)	0.062(3)	0.098(3)	−0.005(2)	0.037(2)	−0.022(3)
O(9b)	4e	−0.1106(5)	0.8380(3)	0.6805(2)	0.054(3)	0.079(3)	0.060(3)	0.011(2)	0.028(2)	0.002(3)
N(8a)	4e	0.2912(6)	0.8306(3)	−0.0731(2)	0.049(3)	0.053(3)	0.098(4)	−0.005(3)	0.032(3)	−0.016(3)
N(8b)	4e	0.0041(7)	0.7761(4)	0.7154(2)	0.057(3)	0.080(4)	0.056(3)	0.011(3)	0.020(3)	0.009(3)
C(1a)	4e	0.2869(8)	0.6885(4)	−0.1086(3)	0.054(4)	0.046(4)	0.053(4)	0.005(3)	0.022(3)	−0.007(3)
C(1b)	4e	−0.0534(7)	0.8462(4)	0.6248(3)	0.041(4)	0.044(4)	0.053(4)	0.003(3)	0.006(3)	−0.003(3)
C(2a)	4e	0.351(1)	0.6006(5)	−0.1270(3)	0.082(5)	0.051(5)	0.044(4)	0.014(4)	0.019(4)	−0.003(3)
C(2b)	4e	−0.157(1)	0.8947(4)	0.5728(3)	0.060(5)	0.047(4)	0.068(5)	0.006(4)	0.011(4)	−0.003(4)
C(4a)	4e	0.0356(9)	0.5672(4)	−0.1461(3)	0.086(5)	0.050(4)	0.073(5)	−0.011(4)	0.022(4)	−0.018(4)
C(4b)	4e	0.1183(8)	0.8838(4)	0.5272(3)	0.076(5)	0.057(4)	0.054(4)	0.018(4)	0.021(3)	0.013(3)
C(5a)	4e	−0.0081(8)	0.6283(4)	−0.0942(3)	0.066(4)	0.050(4)	0.061(4)	−0.009(3)	0.024(3)	−0.014(3)
C(5b)	4e	0.1756(7)	0.7966(4)	0.5624(2)	0.060(4)	0.044(4)	0.050(4)	0.009(3)	0.012(3)	−0.002(3)
C(6a)	4e	0.1261(7)	0.7035(4)	−0.0901(2)	0.049(4)	0.035(3)	0.043(3)	0.000(3)	0.017(3)	−0.003(3)
C(6b)	4e	0.0923(7)	0.7966(4)	0.6210(2)	0.051(4)	0.042(4)	0.034(3)	0.002(3)	0.013(3)	0.001(3)
C(7a)	4e	0.1378(7)	0.7940(4)	−0.0665(3)	0.040(3)	0.043(4)	0.051(4)	−0.003(3)	0.011(3)	−0.001(3)
C(7b)	4e	0.1221(7)	0.7529(4)	0.6788(2)	0.042(3)	0.040(3)	0.036(3)	−0.005(3)	0.010(3)	−0.002(3)
C(71a)	4e	0.0020(7)	0.8464(3)	−0.0377(2)	0.040(3)	0.030(3)	0.044(3)	−0.004(3)	0.014(3)	−0.002(3)
C(71b)	4e	0.2667(7)	0.6873(4)	0.7007(2)	0.038(3)	0.056(4)	0.029(3)	0.003(3)	0.015(3)	0.006(3)
C(72a)	4e	0.0253(7)	0.8700(3)	0.0245(3)	0.044(4)	0.036(3)	0.049(4)	−0.001(3)	0.007(3)	0.002(3)
C(72b)	4e	0.2406(7)	0.5927(4)	0.7025(2)	0.053(4)	0.053(4)	0.035(3)	−0.007(3)	0.012(3)	0.005(3)
C(73a)	4e	−0.1032(8)	0.9187(4)	0.0511(3)	0.063(4)	0.047(4)	0.047(4)	−0.001(3)	0.021(3)	−0.001(3)
C(73b)	4e	0.3795(9)	0.5329(4)	0.7197(2)	0.079(5)	0.046(4)	0.049(4)	0.005(4)	0.016(3)	0.006(3)
C(74a)	4e	−0.2569(8)	0.9436(4)	0.0159(3)	0.048(4)	0.043(4)	0.076(5)	0.010(3)	0.032(3)	−0.011(4)
C(74b)	4e	0.5481(8)	0.5670(5)	0.7354(3)	0.055(4)	0.080(5)	0.045(4)	0.020(4)	0.012(3)	0.014(4)
C(75a)	4e	−0.2868(8)	0.9194(4)	−0.0460(3)	0.043(4)	0.050(4)	0.073(5)	0.001(3)	0.004(3)	0.002(3)
C(75b)	4e	0.5804(8)	0.6600(5)	0.7340(3)	0.050(4)	0.071(5)	0.060(4)	0.007(4)	0.007(3)	0.015(4)
C(76a)	4e	−0.1558(7)	0.8734(4)	−0.0726(2)	0.039(3)	0.046(4)	0.047(4)	−0.004(3)	0.008(3)	0.002(3)
C(76b)	4e	0.4422(8)	0.7191(4)	0.7172(2)	0.049(4)	0.057(4)	0.042(4)	−0.003(3)	0.014(3)	0.003(3)

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