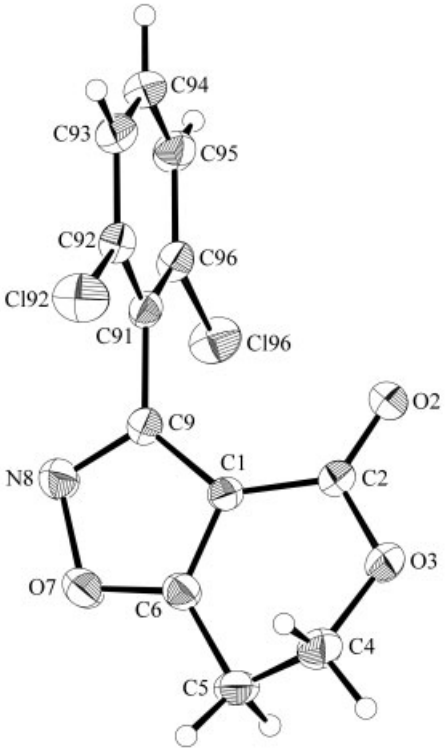
Crystal structure of 3-(2,6-dichlorophenyl)-6,7-dihydro-4*H*-pyrano[3,4-*d*]isoxazol-4-one, $C_{12}H_7Cl_2NO_3$

C. J. Easton[I], C. M. M. Hughes[II] and E. R. T. Tiekink*[,II,1]

[I] Australian National University, Research School of Chemistry, Canberra 0200, Australia
[II] The University of Adelaide, Department of Chemistry, Australia 5005

Received June 5, 2003, accepted and available on-line August 29, 2003; CCDC-No. 1267/1103



Abstract

$C_{12}H_7Cl_2NO_3$, monoclinic, $P12_1/c1$ (No. 14), a = 13.518(1) Å, b = 5.987(1) Å, c = 14.3838(8) Å, β = 99.736(5)°, V = 1147.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.039, $wR_{ref}(F^2)$ = 0.110, T = 293 K.

Source of material

The compound was prepared by reacting 2,6-dichlorobenzonitrile oxide with 5,6-dihydro-2*H*-pyran-2-one followed by treatment of the product isoxazoline with nickel peroxide [1] (mp 448 K – 450 K).

Discussion

The analysis shows the carbonyl to lie on the same side of the molecule as the aryl substituent. The product, along with the isomeric 3-(2,6-dichlorophenyl)-4,5-dihydro-7*H*-pyrano[4,3-*d*]isoxazol-7-one [2], are of interest owing to the use of bromine as a steric auxiliary to reverse the regioselectivity of nitrile oxide cycloaddition [3,4].

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.13 × 0.32 × 0.39 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	5.62 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC6R, $\omega/2\theta$
$2\theta_{max}$:	55.8°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2854, 2742
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1999
$N(param)_{refined}$:	163
Programs:	teXsan [5], SHELXS-86 [6], SHELXL-97 [7], DIFABS [8], ORTEPII [9]

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

Atom	Site	x	y	z	U_{iso}
H(4a)	4*e*	0.6155	–0.0232	0.9051	0.068
H(4b)	4*e*	0.5052	0.0454	0.9127	0.068
H(5a)	4*e*	0.5468	0.4182	0.9194	0.061
H(5b)	4*e*	0.6286	0.2959	0.9932	0.061
H(93)	4*e*	0.9702	0.0698	0.5890	0.059
H(94)	4*e*	0.9313	0.3153	0.4663	0.062
H(95)	4*e*	0.8213	0.6020	0.4736	0.059

* Correspondence author (e-mail: chmtert@nus.edu.sg)
[1] Current address: The National University of Singapore, Department of Chemistry, Singapore 117543

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}
Cl(92)	4*e*	0.91051(5)	0.0274(1)	0.76186(4)	0.0767(4)	0.0593(4)	0.0596(4)	0.0233(3)	0.0119(3)	0.0163(3)
Cl(96)	4*e*	0.70748(5)	0.7348(1)	0.60699(4)	0.0573(3)	0.0580(4)	0.0627(4)	0.0176(3)	0.0138(3)	0.0152(3)
O(2)	4*e*	0.5899(1)	0.1473(3)	0.6508(1)	0.0467(8)	0.065(1)	0.0403(7)	–0.0097(7)	0.0093(6)	–0.0111(7)
O(3)	4*e*	0.5383(1)	0.0908(3)	0.7844(1)	0.0410(8)	0.071(1)	0.0478(8)	–0.0142(7)	0.0148(6)	–0.0011(7)
O(7)	4*e*	0.7528(1)	0.5161(3)	0.9028(1)	0.0580(9)	0.0557(9)	0.0400(8)	–0.0086(8)	0.0084(6)	–0.0103(7)
N(8)	4*e*	0.8130(1)	0.5330(3)	0.8310(1)	0.052(1)	0.055(1)	0.0449(9)	–0.0131(9)	0.0106(8)	–0.0050(8)
C(1)	4*e*	0.6832(1)	0.3053(3)	0.7861(1)	0.0327(9)	0.039(1)	0.0337(9)	0.0016(8)	0.0076(7)	–0.0002(7)
C(2)	4*e*	0.6038(1)	0.1746(4)	0.7339(1)	0.0333(9)	0.044(1)	0.042(1)	0.0000(8)	0.0095(8)	–0.0026(8)
C(4)	4*e*	0.5640(2)	0.0885(5)	0.8864(2)	0.053(1)	0.076(2)	0.046(1)	–0.011(1)	0.018(1)	0.009(1)
C(5)	4*e*	0.6009(2)	0.3098(4)	0.9267(1)	0.046(1)	0.072(2)	0.036(1)	0.010(1)	0.0134(9)	0.001(1)
C(6)	4*e*	0.6785(2)	0.3770(4)	0.8730(1)	0.040(1)	0.045(1)	0.0357(9)	0.0051(9)	0.0043(8)	–0.0021(8)
C(9)	4*e*	0.7696(1)	0.4076(3)	0.7633(1)	0.0342(9)	0.038(1)	0.0378(9)	–0.0011(8)	0.0054(7)	0.0014(8)
C(91)	4*e*	0.8146(1)	0.3821(3)	0.6783(1)	0.0306(9)	0.039(1)	0.0382(9)	–0.0050(8)	0.0061(7)	0.0012(8)
C(92)	4*e*	0.8810(1)	0.2113(3)	0.6708(1)	0.036(1)	0.042(1)	0.045(1)	–0.0009(8)	0.0042(8)	0.0015(8)
C(93)	4*e*	0.9251(2)	0.1856(4)	0.5924(2)	0.041(1)	0.055(1)	0.053(1)	0.004(1)	0.0122(9)	–0.005(1)
C(94)	4*e*	0.9018(2)	0.3321(4)	0.5198(2)	0.046(1)	0.064(1)	0.047(1)	–0.006(1)	0.0173(9)	–0.004(1)
C(95)	4*e*	0.8362(2)	0.5024(4)	0.5236(2)	0.046(1)	0.058(1)	0.044(1)	–0.005(1)	0.0120(9)	0.011(1)
C(96)	4*e*	0.7924(1)	0.5251(3)	0.6025(1)	0.0341(9)	0.044(1)	0.045(1)	–0.0017(8)	0.0062(8)	0.0060(8)

Acknowledgment. The Australian Research Council is thanked for support.

References

1. Hughes, C. M. M.: Ph.D. Thesis, The University of Adelaide, 1995.
2. Easton, C. J.; Hughes, C. M. M.; Tiekink, E. R. T.: Crystal structure of 3-(2,6-dichlorophenyl)-4,5-dihydro-7*H*-pyrano[4,3-*d*]isoxazol-7-one, $C_{12}H_7Cl_2NO_3$. Z. Kristallogr. **218** (2003) 365-366.
3. Easton, C. J.; Hughes, C. M. M.; Tiekink, E. R. T.; Lubin, C. E.; Savage, G. P.; Simpson, G. W.: Reversal of regiochemistry in the synthesis of isozazoles by nitrile oxide cycloadditions. Tetrahedron Lett. **35** (1994) 3589-3592.
4. Easton, C. J.; Hughes, C. M. M.; Savage, G. P.; Simpson, G. W.: Cycloaddition reactions of nitrile oxides with alkenes. Adv. Het. Chem. **60** (1994) 261-327.
5. teXsan: Single Crystal Structure Analysis Software. Version 1.04. Molecular Structure Corporation. The Woodlands, TX, USA 1997.
6. Sheldrick, G. M.: SHELXS-86. Program for the solution of crystal structure. University of Göttingen, Germany 1986.
7. Sheldrick, G. M.: SHELXL-97. Program for crystal structure refinement. University of Göttingen, Germany 1997.
8. Walker, N.; Stuart, D.: An empirical method for corrrecting diffractometer data for absorption effects. Acta Crystallogr. A**39** (1983) 158-166.
9. Johnson, C. K.: ORTEPII. Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, 1976.