Cvetka Turk, Jurij Svete*, Branko Stanovnik*, Ljubo Goli•, Amalija Golobi•, and Lovro Seli•.

Unusual Reactions of 5,5-Dimethyl-2-(indenyl-2)-3-pyrazolidinone with Acetylenedicarboxylates.

Supporting Information:

Experimental details (Procedures, Spectral and Analytical Data)

Experimental Details: TLC: *Merck, Alufolien Kieselgel 60 F 254, 0.2mm*. M.p.: *Kofler* micro hot stage. IR: *Perkin-Elmer 1310* spectophotometer. ^{1}H-NMR and ^{13}C-NMR: *Bruker Avance DPX 300* spectrometer. Elemental analyses: *Perkin-Elmer CHN Analyser 2400*. MS: *Autospeck Q (VG-Analytical)* spectrometer.

Preparation of compound **3**: A mixture of **1** (1 mmol), **2** (1 mmol), anhydrous ethanol (5 ml), and trifluoroacetic acid (3 drops) was stirred at 20° for 24 hours. The precipitate was collected by filtration to give **3** in 60% yield, mp 160–161° (from ethanol). ^{1}H-NMR (300 MHz, [D_6]-DMSO): δ 1.22 (6H, s, 5-Me), 2.47 (2H, s, 4-CH_2), 3.77 (2H, s, 1'- CH_2), 6.11 (1H, s, 2-H), 6.46 (1H, s, 3'-H), 7.02 (1H, ddd, *J* = 1.3, 7.3, 7.4 Hz, 5'-H), 7.17 (1H, dd, *J* = 7.3, 7.4 Hz, 6'-H), 7.24 (1H, d, *J* = 7.3, Hz, 7'-H), 7.36 (1H, d, *J* = 7.3 Hz, 4'-H). ^{13}C-NMR (75.5 MHz, $CDCl_3$): δ 26.0, 38.1, 47.2, 56.6, 110.9, 120.0, 123.4, 126.6, 138.8, 143.4, 143.8, 171.4. Anal. Calcd. for $C_{14}H_{16}N_2O$ (228.30): C, 73.66; H, 7.06; N, 12.27. Found: C, 73.40; H, 7.34; N, 12.28.

Preparation of compounds **7** and **8**: A mixture of **3** (1 mmol), **5** (1 mmol), and anhydrous ethanol (5 ml) was heated at reflux temperature for 4 hours, cooled, and the precipitate (a mixture of **7** and **8**) collected by filtration. The precipitate was suspended in chloroform (4 ml), stirred at 20° for 5 minutes, and the undissolved material collected by flitration to give **7**. The filtrate was evaporated *in vacuo* to give **8**.

Compound **7**: Yield: 41%, mp 245–247° (from ethanol). ^{1}H-NMR (300 MHz, [D_6]-DMSO): δ 1.23 (3H, s, 4-Me), 1.35 (3H, s, 4-Me), 2.29 (1H, dd, *J* = 15.5, 1.5 Hz, 12-Ha), 2.89 (1H, d, *J* = 15.5 Hz, 12-Hb), 3.43 (1H, d, *J* = 17.2 Hz, 3-Ha), 3.62 (3H, s, OMe),3.77 (1H, d, *J* = 17.1 Hz, 3-Hb), 3.79 (3H, s, OMe), 4.74 (1H, s, 7a-H), 7.17–7.24 (3H, m, 9-H, 10-H, and 11-H), 7.43–7.47 (1H, m, 8-H), 8.85 (1H, br s, 1-H). ^{13}C-NMR (75.5 MHz, [D_6]-DMSO): δ 27.8, 29.1, 46.3, 50.7, 51.6, 52.8, 55.4, 58.2, 87.8, 102.7, 124.4, 125.4, 127.0, 127.4, 139.2, 140.7, 148.2, 163.7, 163.9, 169.4. Anal. Calcd. for $C_{20}H_{22}N_2O_5$ (370.40): C, 64.85; H, 5.99; N, 7.56. Found: C, 64.92; H, 6.08; N, 7.57.

Compound **8**: Yield: 28%, mp 199–201° (from ethanol), ms 370 (M^+). ^{1}H-NMR (300 MHz, [D_6]-DMSO): δ 1.12 (6H, s, 7-Me), 2.78 (2H, br s, 6-CH_2), 3.59 (3H, s, OMe), 3.60 (3H, s, OMe), 3.64 (2H, br s, 1'-CH_2), 6.38 (1H, s, 1-H), 6.46 (1H, s, 3'-H), 7.13 (1H, ddd, *J* = 7.3, 7.2, 1.4 Hz, 5'-H), 7.22 (1H, dd, *J* = 7.1, 7.4 Hz, 6'-H), 7.28 (1H, d, *J* = 7.1 Hz, 7'-H), 7.39 (1H, d, *J* = 7.2 Hz, 4'-H). ^{13}C-NMR (75.5 MHz, [D_6]-DMSO): δ 26.6, 38.2, 52.3, 53.3, 57.0, 68.3, 113.1, 119.3, 121.0, 123.8, 125.3, 126.9, 139.7, 142.8, 150.0, 158.4, 164.3, 166.2, 194.1. Anal. Calcd. for $C_{20}H_{22}N_2O_5$ (370.40): C, 64.85; H, 5.99; N, 7.56. Found: C, 64.61; H, 5.94; N, 7.57.

Preparation of compounds **9** and **10**: A mixture of **3** (1 mmol), **6** (1 mmol), and anhydrous ethanol (5 ml) was heated at reflux temperature for 6 hours, cooled, and the precipitate collected by filtration to give **9**. The filtrate was evaporated *in vacuo*, the residue triturated with a mixture of ether and petroleum ether, and the precipitate collected by filtration to give **10**.

Compound **9**: Yield: 48%, mp 220–222° (from ethanol). ^{1}H-NMR (300 MHz, $CDCl_3$): δ 1.27 (3H, t, *J* = 7.2 Hz, C*H*$_3$CH$_2$), 1.36 (3H, t, *J* = 7.2 Hz, C*H*$_3$CH$_2$), 1.44 (3H, s, 4-Me), 1.46 (3H, s, 4-Me), 2.41 (1H, dd, *J* = 15.4, 1.6 Hz, 12-Ha), 2.80 (1H, d, *J* = 15.5 Hz, 12-Hb), 3.41 (1H, d, *J* = 16.6 Hz, 3-Ha), 3.80 (1H, d, *J* = 16.2 Hz, 3-Hb), 4.14 (1H, dq, *J* = 7.2, 14.3 Hz, CH$_3$C*H*$_2$–Ha), 4.18 (1H, dq, *J* = 7.2, 14.3 Hz, CH$_3$C*H*$_2$–Hb), 4.30 (1H, dq, *J* = 7.2, 10.9 Hz, CH$_3$C*H*$_2$–Ha), 4.37 (1H, dq, *J* = 7.2, 10.9 Hz, CH$_3$C*H*$_2$–Hb), 4.66 (1H, s, 7a-H), 6.57 (1H, br s, 1-H), 7.13–7.19 (3H, m, 9-H, 10-H, and 11-H), 7.63–7.67 (1H, m, 8-H). ^{13}C-NMR (75.5 MHz, [D_6]-DMSO): δ 14.1, 14.8, 28.3, 30.1, 47.5, 52.8, 56.2, 60.3, 61.2, 62.8, 88.9, 104.6, 124.6, 127.3, 127.7, 128.3, 138.9, 140.1, 148.3, 164.2, 164.5, 170.4. Anal. Calcd. for $C_{22}H_{26}N_2O_5$ (398.45): C, 66.32; H, 6.58; N, 7.03. Found: C, 66.67; H, 6.68; N, 7.03.

Compound **10**: Yield: 8%, mp 141–142° (petroleum ether). M^+ 398 (EI); MH^+ 399 (FAB). ^{1}H-NMR (300 MHz, $CDCl_3$): δ 1.06 (3H, t, *J* = 7.2 Hz, C*H*$_3$CH$_2$), 1.24 (6H, s,

7-Me), 1.28 (3H, t, J = 7.2 Hz, CH_3CH$_2$), 2.92 (2H, br s, 6-CH$_2$), 3.70 (2H, br s, 1'-CH$_2$), 4.12 (2H, q, J = 7.2 Hz, CH$_3$CH_2), 4.22 (2H, q, J = 7.2 Hz, CH$_3$CH_2), 4.22 (1H, s, 1-H), 6.44 (1H, s, 3'-H), 7.14-7.20 (2H, m, Ph), 7.21-7.25 (1H, m, Ph), 7.35-7.38 (1H, m, Ph). Anal. Calcd. for $C_{22}H_{26}N_2O_5$ (398.45): C, 66.32; H, 6.58; N, 7.03. Found: C, 66.20; H, 6.34; N, 6.92. HRMS Calcd. for $C_{22}H_{26}N_2O_5$: 398.184172. Found: 398.185270.

Crystal structure analysis.

Compound **3**: $C_{14}H_{16}N_2O$, M_r=228.3, tetragonal, space group $P4_2/n$, No. 86:2, a=20.197(1), c=6.068(1)Å, V=2475.3(5) Å^3, Z=8, ρ_{cal}=1.225 g cm^{-3}, Mo $K\alpha$ radiation, T=293(1) K, crystal dimensions: 0.60 x 0.17 x 0.16 mm, colourless, Enraf Nonius CAD4 diffractometer, 12242 measured, 2966 independent, 1394 observed reflections (I>2.5σ(I)), R_{int}=0.029, R=0.044, R_w=0.051, 2292 contributing refl., 203 parameters, $\Delta\rho_{max}$=0.268 e Å^{-3}, $\Delta\rho_{min}$=-0.259 e Å^{-3}.

Compound **7**: $C_{20}H_{22}N_2O_5$, M_r=370.4, monoclinic, space group $P2_1/n$, No. 14, a=6.137(1), b=16.013(1), c=19.412(1)Å, β=93.83(1)°, V=1903.4(1) Å^3, Z=4, ρ_{cal}=1.293 g cm^{-3}, Mo $K\alpha$ radiation, T=293(1) K, crystal dimensions: 0.76 x 0.72 x 0.61 mm, colourless, Enraf Nonius CAD4 diffractometer, 18362 measured, 4571 independent, 3298 observed reflections (I>2.5σ(I)), R_{int}=0.021, R=0.041, R_w=0.053, 3852 contributing refl., 311 parameters, $\Delta\rho_{max}$=0.260 e Å^{-3}, $\Delta\rho_{min}$=-0.258 e Å^{-3}.

Both structures were solved by direct methods using SIR92[1] program. The Xtal3.4[2] system of crystallographic programs was used for the correlation and reduction of the data, structure refinement and interpretation. OrtepII[3] was used to produce molecular graphics. Details of the structure analysis, coordinates of atoms with displacement parameters, bond lengths and angles are available free of charge via the Internet at http://pubs.acs.org as Supporting Information.

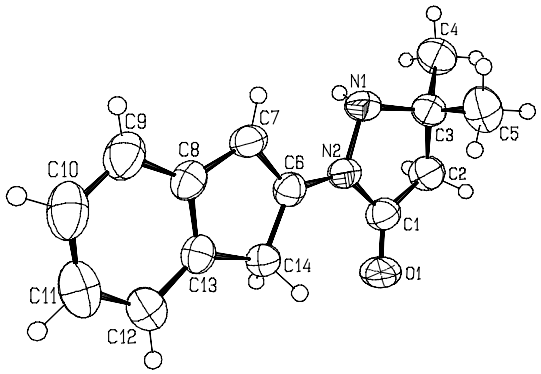

Figure 1. ORTEP view of **3**, showing the labeling of the non-hydrogen atoms. (Ellipsoids at 50% probability level.)

[1] Altomare, A.; Burla, M.C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Polidori, G. *J Appl Cryst*, **1994**, *27*, 435.
[2] Johnson, C. K. *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA **1976**.
[3] Hall, S. R.; King, G S D.; Stewart, J. M. *The Xtal3.4 User's Manual*, University of Western Australia: Lamb, Perth **1995**.

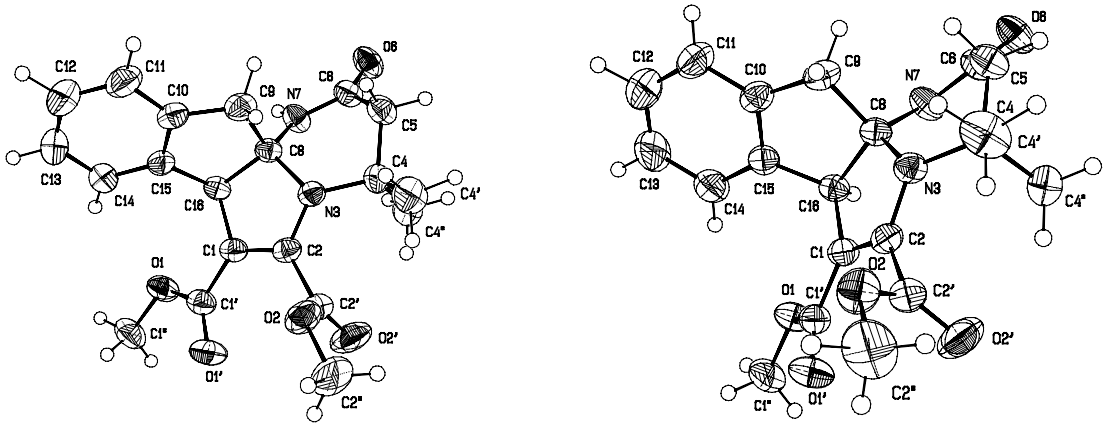

Figure 2. ORTEP views of compoud **7**, showing the labeling of the non-hydrogen atoms. (Ellipsoids at 50% probability level.)

X-ray structure determination of compounds 3 and 7

Contents

Experimental (Crystal data, Data collection, Structure refinement)

Positional and Isotropic Displacement Parameters

Bond lengths and angles

CIF files

Experimental

Compound	**3**	**7**
Crystal data		
Chemical formula	$C_{14}H_{16}N_2O$	$C_{20}H_{22}N_2O_5$
Chemical formula weight M_r	228.3	370.4
Crystal system	Tetragonal	Monoclinic
Space group	$P4_2/n$ No. 86 : 2	$P2_1/n$ No. 14
a (Å)	20.197(1)	6.137(1)
b (Å)	20.197(1)	16.013(1)
c (Å)	6.068(1)	19.412(1)
β (°)	90.00	93.83(1)
V (Å^3)	2475.3(5)	1903.4(1)
Z	8	4
Calculated density (Mg m^{-3})	1.225	1.293
Radiation type	Mo *K*α	Mo *K*α
Wavelength (Å)	0.71069	0.71069
No. of refl. for cell parameters	43	100
θ range (°)	9.0 – 15.5	8.0-14.9
μ (mm^{-1})	0.0732	0.0875
Temperature (K)	293(1)	293(1)
Crystal shape	Prism	Prism
Crystal size (mm)	0.60 x 0.17 x 0.16	0.76 x 0.72 x 0.61
Crystal colour	Colourless	Colourless
Data collection		
Diffractometer type	Enraf Nonius CAD4	Enraf Nonius CAD4
Data collection method	ω	ω/2θ
No. of measured reflections	12242	18362
No. of independent reflection	2966	4571
No. of observed reflections	1394	3298
Criterion of observed reflections	I>2.5σ(I)	I>2.5σ(I)
R_{int}	0.029	0.021
θ_{max} (°)	28	28
Range of *h, k, l*	$0 \leq h \leq 26$	$-8 \leq h \leq 8$

	$-26 \le k \le 26$	$-21 \le k \le 21$
	$-8 \le l \le 8$	$-25 \le l \le 25$
Intensity decay (%)	-0.31	0.81
Structure refinement		
Refinement on	F	F
R	0.044	0.041
R_w	0.051	0.053
S	0.735	0.448
No. of contributing reflections	2292	3852
No. of parameters	203	311
Max shift/error	0.00080	0.0488
Average shift/error	0.00008	0.0015
Extinction coeffi. (Zachariasen)	$5(1)\cdot10^3$	$6(1)\cdot10^3$
$\Delta\rho_{max}$ (e Å^{-3})	0.268	0.260
$\Delta\rho_{min}$ (e Å^{-3})	-0.259	-0.258

Data were corrected for a decay and for Lorentz and polarisation effects but not for absorption as linear absorption coefficient is small enough in both cases. Structures were solved by direct methods using SIR92[10] program. Hydrogen atoms were found from the difference Fourier maps and included in the further calculations. We employed full-matrix least-squares refinement on F magnitudes with anisotropic temperature factors for all non-hydrogen atoms, using the empirical weighting function. The parameters of hydrogen atoms were included in F_c calculations with isotropic temperature factors, only their positions were refined. The contributing reflections in the final least-square cycle were all observed reflections and those unobserved reflections for which F_c was larger than F_o. The Xtal3.4[11] system of crystallographic programs was used for the correlation and reduction of the data, structure refinement and interpretation. OrtepII[12] was used to produce molecular graphics.

[10]Altomare, A.; Burla, M.C.; Camalli, M.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Polidori, G. *J Appl Cryst*, **1994**, *27*, 435.

[11]Hall, S. R.; King, G S D.; Stewart, J. M. *The Xtal3.4 User's Manual*, University of Western Australia: Lamb, Perth **1995**.

[12]Johnson, C. K. *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA **1976**.

Compound 3

Non-Hydrogen Positional and Isotropic Displacement Parameters

	x/a	y/b	z/c	U
O(1)	0.6059(1)	0.4781(1)	0.5730(3)	* 0.0721(8)
N(1)	0.6394(1)	0.4166(1)	1.1014(4)	* 0.0540(8)
N(2)	0.5972(1)	0.4333(1)	0.9189(3)	* 0.0487(7)
C(1)	0.6300(1)	0.4641(1)	0.7506(5)	* 0.0528(9)
C(2)	0.6999(1)	0.4745(2)	0.8279(6)	* 0.060(1)
C(3)	0.7077(1)	0.4226(1)	1.0103(5)	* 0.0538(9)
C(4)	0.7541(2)	0.4427(2)	1.1944(7)	* 0.075(1)
C(5)	0.7269(2)	0.3562(2)	0.9135(7)	* 0.075(1)
C(6)	0.5312(1)	0.4144(1)	0.9277(4)	* 0.0449(7)
C(7)	0.5027(1)	0.3812(1)	1.1007(4)	* 0.0492(8)
C(8)	0.4331(1)	0.3703(1)	1.0445(4)	* 0.0491(8)
C(9)	0.3826(2)	0.3385(2)	1.1575(6)	* 0.065(1)
C(10)	0.3207(2)	0.3347(2)	1.0625(7)	* 0.071(1)
C(11)	0.3083(2)	0.3623(2)	0.8586(7)	* 0.068(1)
C(12)	0.3583(1)	0.3944(1)	0.7436(6)	* 0.0572(9)
C(13)	0.4208(1)	0.3984(1)	0.8380(4)	* 0.0466(8)
C(14)	0.4828(1)	0.4288(1)	0.7532(4)	* 0.0461(8)

Bond Distances (Angstroms)

O(1)-C(1)	1.216(4)
N(1)-N(2)	1.437(3)
N(1)-C(3)	1.491(3)
N(2)-C(1)	1.368(4)
N(2)-C(6)	1.388(3)
C(1)-C(2)	1.501(4)
C(2)-C(3)	1.532(5)
C(3)-C(4)	1.515(5)
C(3)-C(5)	1.515(5)
C(6)-C(7)	1.373(4)
C(6)-C(14)	1.469(4)
C(7)-C(8)	1.462(4)
C(8)-C(9)	1.387(4)
C(8)-C(13)	1.398(4)
C(9)-C(10)	1.379(5)
C(10)-C(11)	1.379(6)
C(11)-C(12)	1.389(5)
C(12)-C(13)	1.388(4)
C(13)-C(14)	1.487(4)

Bond Angles (degrees)

N(2)-N(1)-C(3)	104.1(2)
N(1)-N(2)-C(1)	113.2(2)
N(1)-N(2)-C(6)	118.4(2)
C(1)-N(2)-C(6)	128.3(2)
O(1)-C(1)-N(2)	124.9(3)
O(1)-C(1)-C(2)	128.4(3)
N(2)-C(1)-C(2)	106.6(2)
C(1)-C(2)-C(3)	103.1(2)
N(1)-C(3)-C(2)	103.2(2)
N(1)-C(3)-C(4)	108.8(2)
N(1)-C(3)-C(5)	108.0(3)
C(2)-C(3)-C(4)	114.4(3)
C(2)-C(3)-C(5)	110.6(3)
C(4)-C(3)-C(5)	111.4(3)

N(2)-C(6)-C(7)	124.5(2)
N(2)-C(6)-C(14)	123.8(2)
C(7)-C(6)-C(14)	111.6(2)
C(6)-C(7)-C(8)	107.4(2)
C(7)-C(8)-C(9)	131.4(3)
C(7)-C(8)-C(13)	108.6(2)
C(9)-C(8)-C(13)	120.0(3)
C(8)-C(9)-C(10)	119.1(3)
C(9)-C(10)-C(11)	121.2(3)
C(10)-C(11)-C(12)	120.4(3)
C(11)-C(12)-C(13)	118.7(3)
C(8)-C(13)-C(12)	120.6(2)
C(8)-C(13)-C(14)	109.1(2)
C(12)-C(13)-C(14)	130.3(3)
C(6)-C(14)-C(13)	103.2(2)

Compound 7

Non-Hydrogen Positional and Isotropic Displacement Parameters

	x/a	y/b	z/c		U
C(1)	0.0468(2)	0.25113(9)	0.65219(7)	*	0.0359(4)
C(1′)	-0.0933(2)	0.31538(9)	0.67848(8)	*	0.0404(4)
C(1")	-0.4122(3)	0.3958(1)	0.6499(1)	*	0.0582(6)
C(2)	0.1921(2)	0.20560(9)	0.69175(7)	*	0.0362(4)
C(2′)	0.2453(3)	0.2200(1)	0.76782(7)	*	0.0458(5)
C(2")	0.4876(6)	0.2927(2)	0.8461(1)	*	0.083(1)
C(4)	0.3960(2)	0.0675(1)	0.68353(7)	*	0.0409(4)
C(4′)	0.6029(3)	0.0841(1)	0.7298(1)	*	0.0605(6)
C(4")	0.2278(4)	0.0183(1)	0.7221(1)	*	0.0581(6)
C(5)	0.4569(3)	0.0156(1)	0.62097(9)	*	0.0455(5)
C(6)	0.2670(3)	0.00461(9)	0.56848(8)	*	0.0431(4)
C(8)	0.2317(2)	0.15456(8)	0.58179(6)	*	0.0329(4)
C(9)	0.4198(2)	0.1887(1)	0.54016(8)	*	0.0443(5)
C(10)	0.3309(2)	0.2653(1)	0.50522(7)	*	0.0415(4)
C(11)	0.4292(4)	0.3138(1)	0.45682(9)	*	0.0598(6)
C(12)	0.3245(5)	0.3842(1)	0.4316(1)	*	0.0790(9)
C(13)	0.1243(5)	0.4069(1)	0.4540(1)	*	0.0790(9)
C(14)	0.0222(4)	0.3582(1)	0.50195(9)	*	0.0572(6)
C(15)	0.1271(2)	0.28683(9)	0.52733(7)	*	0.0382(4)
C(16)	0.0490(2)	0.22299(8)	0.57778(7)	*	0.0330(4)
N(3)	0.3015(2)	0.14713(8)	0.65597(6)	*	0.0362(3)
N(7)	0.1506(2)	0.07452(8)	0.55589(7)	*	0.0418(4)
O(1)	-0.2566(2)	0.33422(7)	0.63113(6)	*	0.0498(4)
O(1′)	-0.0726(2)	0.34749(8)	0.73481(6)	*	0.0585(4)
O(2′)	0.1530(3)	0.1876(1)	0.81280(6)	*	0.0737(5)
O(2)	0.4111(2)	0.27229(8)	0.77597(6)	*	0.0553(4)
O(6)	0.2203(2)	-0.06285(7)	0.54089(7)	*	0.0556(4)

Bond Distances (Angstroms)

C(1)-C(1′)	1.455(2)
C(1)-C(2)	1.351(2)
C(1)-C(16)	1.514(2)
C(1′)-O(1)	1.348(2)
C(1′)-O(1′)	1.207(2)
C(1")-O(1)	1.436(2)
C(2)-C(2′)	1.509(2)
C(2)-N(3)	1.368(2)
C(2′)-O(2′)	1.191(2)

C(2′)-O(2)	1.319(2)
C(2")-O(2)	1.448(3)
C(4)-C(4′)	1.529(2)
C(4)-C(4")	1.533(3)
C(4)-C(5)	1.538(2)
C(4)-N(3)	1.486(2)
C(5)-C(6)	1.506(2)
C(6)-N(7)	1.342(2)
C(6)-O(6)	1.231(2)
C(8)-C(9)	1.552(2)
C(8)-C(16)	1.566(2)
C(8)-N(3)	1.480(2)
C(8)-N(7)	1.452(2)
C(9)-C(10)	1.487(2)
C(10)-C(11)	1.388(3)
C(10)-C(15)	1.392(2)
C(11)-C(12)	1.371(3)
C(12)-C(13)	1.379(4)
C(13)-C(14)	1.395(3)
C(14)-C(15)	1.386(2)
C(15)-C(16)	1.515(2)

Bond Angles (degrees)

C(1′)-C(1)-C(2)	124.4(1)
C(1′)-C(1)-C(16)	126.0(1)
C(2)-C(1)-C(16)	109.5(1)
C(1)-C(1′)-O(1)	110.4(1)
C(1)-C(1′)-O(1′)	126.2(1)
O(1)-C(1′)-O(1′)	123.4(1)
C(1)-C(2)-C(2′)	124.4(1)
C(1)-C(2)-N(3)	114.1(1)
C(2′)-C(2)-N(3)	121.4(1)
C(2)-C(2′)-O(2′)	124.6(2)
C(2)-C(2′)-O(2)	109.2(1)
O(2′)-C(2′)-O(2)	126.1(1)
C(4′)-C(4)-C(4")	111.2(1)
C(4′)-C(4)-C(5)	108.8(1)
C(4′)-C(4)-N(3)	110.5(1)
C(4")-C(4)-C(5)	108.4(1)
C(4")-C(4)-N(3)	111.0(1)
C(5)-C(4)-N(3)	106.8(1)
C(4)-C(5)-C(6)	112.3(1)
C(5)-C(6)-N(7)	113.8(1)
C(5)-C(6)-O(6)	123.0(1)
N(7)-C(6)-O(6)	123.2(1)
C(9)-C(8)-C(16)	106.4(1)
C(9)-C(8)-N(3)	111.0(1)
C(9)-C(8)-N(7)	112.4(1)
C(16)-C(8)-N(3)	105.3(1)
C(16)-C(8)-N(7)	111.9(1)
N(3)-C(8)-N(7)	109.6(1)
C(8)-C(9)-C(10)	105.3(1)
C(9)-C(10)-C(11)	127.4(2)
C(9)-C(10)-C(15)	111.9(1)
C(11)-C(10)-C(15)	120.7(2)
C(10)-C(11)-C(12)	119.1(2)
C(11)-C(12)-C(13)	120.7(2)
C(12)-C(13)-C(14)	120.8(2)
C(13)-C(14)-C(15)	118.6(2)
C(10)-C(15)-C(14)	120.0(2)
C(10)-C(15)-C(16)	111.2(1)
C(14)-C(15)-C(16)	128.8(1)
C(1)-C(16)-C(8)	102.3(1)
C(1)-C(16)-C(15)	116.1(1)
C(8)-C(16)-C(15)	104.3(1)

C(2)-N(3)-C(4)	126.7(1)
C(2)-N(3)-C(8)	108.6(1)
C(4)-N(3)-C(8)	120.0(1)
C(6)-N(7)-C(8)	120.5(1)
C(1')-O(1)-C(1")	117.2(1)
C(2')-O(2)-C(2")	117.1(2)

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_computing_molecular_graphics     xtal
_computing_publication_material   xtal_BONDLA_CIFIO

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_cell_angle_beta                  90.00000
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  C2  .69987(15)  .47451(17)  .8279(6)  .0596(17)  Uani  ?  ?
  C3  .70766(13)  .42262(15)  1.0103(5)  .0538(15)  Uani  ?  ?
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  C6  .53117(12)  .41438(12)  .9277(4)  .0449(13)  Uani  ?  ?
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  C10  .32071(16)  .33474(16)  1.0625(7)  .071(2)  Uani  ?  ?
  C11  .30827(16)  .36226(15)  .8586(7)  .068(2)  Uani  ?  ?
  C12  .35831(14)  .39444(14)  .7436(6)  .0572(16)  Uani  ?  ?
  C13  .42079(12)  .39835(12)  .8380(4)  .0466(13)  Uani  ?  ?
  C14  .48282(12)  .42878(14)  .7532(4)  .0461(14)  Uani  ?  ?
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  H7  .5247(13)  .3660(13)  1.216(5)  .04500  Uiso  ?  ?
  H9  .3900(15)  .3190(16)  1.286(5)  .06100  Uiso  ?  ?
  H10  .2854(16)  .3106(15)  1.132(5)  .06500  Uiso  ?  ?
  H11  .2640(16)  .3603(14)  .794(5)  .06200  Uiso  ?  ?
  H12  .3491(14)  .4144(14)  .601(5)  .05400  Uiso  ?  ?
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  H22  .7279(15)  .4653(14)  .712(5)  .05700  Uiso  ?  ?
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  C7  .0563(15)  .0529(14)  .0383(13)  .0014(11)  .0003(12)  .0054(12)
  C8  .0538(14)  .0449(13)  .0486(14)  .0022(11)  .0096(12)  -.0010(11)
  C9  .0641(18)  .0611(18)  .070(2)  .0013(14)  .0180(16)  .0144(16)
  C10  .0575(18)  .0568(17)  .099(3)  -.0044(14)  .0213(18)  .0098(18)
  C11  .0485(16)  .0566(17)  .100(3)  -.0023(13)  .0042(17)  -.0085(18)
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  C13  .0470(13)  .0436(13)  .0493(14)  .0020(10)  .0063(11)  -.0029(11)
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  C11  C12  C13  118.7(3)  .  .  .  ?
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_reflns_limit_k_min             0
_reflns_limit_k_max             26
_reflns_limit_l_min             0
_reflns_limit_l_max             8
_reflns_number_total            2966
_reflns_number_observed         1394
_reflns_observed_criterion
refl_observed_if_I____>2.500_sigma(I___)
_reflns_d_resolution_high          .758
_reflns_d_resolution_low         14.281

loop_
_reflns_scale_group_code
_reflns_scale_meas_F
  ?   2.1072

loop_
_refln_index_h
_refln_index_k
_refln_index_l
_refln_F_meas
_refln_F_calc
_refln_F_sigma
_refln_observed_status
_refln_scale_group_code
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#-end OF DATA FOR COMPOUND 3

data_COMP7 #  STRUCTURE  OF COMPOUND 7

_audit_creation_date            99-10-01
_audit_creation_method          from_xtal_archive_file_using_CIFIO
_chemical_formula_sum           'C20 H22 N2 O5'
_chemical_formula_weight        370.4
_chemical_melting_point         ?
_computing_data_reduction       xtal_DIFDAT_SORTRF_ADDREF
_computing_structure_solution   xtal
_computing_structure_refinement xtal_CRYLSQ
_computing_molecular_graphics   xtal
_computing_publication_material xtal_BONDLA_CIFIO

_cell_length_a                  6.137(1)
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_cell_length_b                     16.013(1)
_cell_length_c                     19.412(1)
_cell_angle_alpha                  90.00000
_cell_angle_beta                   93.83(1)
_cell_angle_gamma                  90.00000
_cell_volume                       1903.4(1)
_cell_formula_units_Z              4
_cell_measurement_temperature      293
_cell_measurement_reflns_used      100
_cell_measurement_theta_min        8.0
_cell_measurement_theta_max        14.9
_cell_special_details
;
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;

_symmetry_cell_setting             monoclinic
_symmetry_space_group_name_H-M     p_1_21/n_1
_symmetry_space_group_name_Hall    -p_2yn
loop_
_symmetry_equiv_pos_as_xyz
  +x,+y,+z  1/2-x,1/2+y,1/2-z  -x,-y,-z  1/2+x,1/2-y,1/2+z

_exptl_crystal_description         ?
_exptl_crystal_preparation         ?
_exptl_crystal_colour              colourless
_exptl_crystal_size_max            0.76
_exptl_crystal_size_mid            0.72
_exptl_crystal_size_min            0.61
_exptl_crystal_density_diffrn        1.293
_exptl_crystal_density_meas        ?
_exptl_crystal_density_method      ?
_exptl_crystal_F_000               784

_exptl_absorpt_process_details     ?
_exptl_absorpt_coefficient_mu      0.0875
_exptl_absorpt_correction_type     none
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_exptl_absorpt_correction_T_max    ?
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;
 ?
;

_diffrn_special_details
;
 ?
;
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_diffrn_radiation_wavelength       ?
_diffrn_radiation_type             xray_tube
_diffrn_radiation_source           ?

_diffrn_radiation_monochromator    graphite
_diffrn_radiation_detector         ?
_diffrn_measurement_device         CAD4
_diffrn_measurement_method         ?

_diffrn_standards_number           3
_diffrn_standards_interval_count   ?
_diffrn_standards_interval_time    ?
_diffrn_standards_decay_%          ?

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_diffrn_standard_refln_index_h
_diffrn_standard_refln_index_k
_diffrn_standard_refln_index_l
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_diffrn_reflns_number              18362
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_diffrn_reflns_av_sigmaI/netI         .014
_diffrn_reflns_limit_h_min         -8
_diffrn_reflns_limit_h_max         8
_diffrn_reflns_limit_k_min         -21
_diffrn_reflns_limit_k_max         21
_diffrn_reflns_limit_l_min         -25
_diffrn_reflns_limit_l_max         25
_diffrn_reflns_theta_min              1.65
_diffrn_reflns_theta_max             27.94
_diffrn_reflns_reduction_process   perpendicular_monochr_Lp

loop_
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_atom_type_oxidation_number
_atom_type_number_in_cell
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
  O  ?  0  20  .008  .006  Int._Tables_Vol_IV_Tables_2.2B_and_2.3.1
  N  ?  0  8  .004  .003  Int._Tables_Vol_IV_Tables_2.2B_and_2.3.1
  C  ?  0  80  .002  .002  Int._Tables_Vol_IV_Tables_2.2B_and_2.3.1
  H  ?  0  88  0  0  Int._Tables_Vol_IV_Tables_2.2B_and_2.3.1

loop_
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_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_thermal_displace_type
_atom_site_calc_flag
_atom_site_calc_attached_atom
  C1  .0468(2)  .25113(9)  .65219(7)  .0359(6)  Uani  ?  ?
  C1′  -.0933(2)  .31538(9)  .67848(8)  .0404(7)  Uani  ?  ?
  C1"  -.4122(3)  .39578(13)  .64988(13)  .0582(10)  Uani  ?  ?
  C2  .1921(2)  .20560(9)  .69175(7)  .0362(6)  Uani  ?  ?
  C2′  .2453(3)  .22005(10)  .76782(7)  .0458(8)  Uani  ?  ?
  C2"  .4876(6)  .29273(18)  .84614(12)  .0826(16)  Uani  ?  ?
  C4  .3960(2)  .06746(10)  .68353(7)  .0409(7)  Uani  ?  ?
  C4′  .6029(3)  .08408(15)  .72980(11)  .0605(11)  Uani  ?  ?
  C4"  .2278(4)  .01827(13)  .72215(11)  .0581(10)  Uani  ?  ?
  C5  .4569(3)  .01560(11)  .62097(9)  .0455(8)  Uani  ?  ?
  C6  .2670(3)  .00461(9)  .56848(8)  .0431(7)  Uani  ?  ?
  C8  .2317(2)  .15456(8)  .58179(6)  .0329(6)  Uani  ?  ?
  C9  .4198(2)  .18871(11)  .54016(8)  .0443(8)  Uani  ?  ?
  C10  .3309(2)  .26528(10)  .50522(7)  .0415(7)  Uani  ?  ?
  C11  .4292(4)  .31380(13)  .45682(9)  .0598(10)  Uani  ?  ?
  C12  .3245(5)  .38418(14)  .43160(12)  .0790(14)  Uani  ?  ?
  C13  .1243(5)  .40690(14)  .45401(12)  .0790(14)  Uani  ?  ?
  C14  .0222(4)  .35821(12)  .50195(9)  .0572(10)  Uani  ?  ?
  C15  .1271(2)  .28683(9)  .52733(7)  .0382(7)  Uani  ?  ?
  C16  .0490(2)  .22299(8)  .57778(7)  .0330(6)  Uani  ?  ?
  N3  .30153(19)  .14713(8)  .65597(6)  .0362(6)  Uani  ?  ?
  N7  .1506(2)  .07452(8)  .55589(7)  .0418(6)  Uani  ?  ?
  O1  -.25659(18)  .33422(7)  .63113(6)  .0498(6)  Uani  ?  ?
  O1′  -.0726(2)  .34749(8)  .73481(6)  .0585(7)  Uani  ?  ?
  O2′  .1530(3)  .18765(10)  .81280(6)  .0737(9)  Uani  ?  ?
```

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  O2  .4111(2)  .27229(8)  .77597(6)  .0553(6)  Uani  ?  ?
  O6  .2203(2)  -.06285(7)  .54089(7)  .0556(7)  Uani  ?  ?
  H7  .041(3)  .0726(10)  .5257(9)  .03810  Uiso  ?  ?
  H1"1  -.469(3)  .3820(13)  .6885(12)  .05562  Uiso  ?  ?
  H1"2  -.340(3)  .4462(14)  .6641(10)  .05562  Uiso  ?  ?
  H1"3  -.504(3)  .4089(13)  .6097(11)  .05562  Uiso  ?  ?
  H11  .573(4)  .2956(13)  .4412(11)  .05769  Uiso  ?  ?
  H12  .386(4)  .4185(15)  .3955(12)  .07314  Uiso  ?  ?
  H13  .051(4)  .4529(16)  .4365(12)  .07193  Uiso  ?  ?
  H14  -.118(4)  .3717(13)  .5155(10)  .05387  Uiso  ?  ?
  H16  -.092(3)  .1996(9)  .5609(8)  .03022  Uiso  ?  ?
  H2"1  .508(4)  .2421(17)  .8721(13)  .07450  Uiso  ?  ?
  H2"2  .363(4)  .3123(16)  .8644(14)  .07450  Uiso  ?  ?
  H2"3  .590(4)  .3335(17)  .8419(13)  .07450  Uiso  ?  ?
  H4"1  .188(3)  .0502(13)  .7614(11)  .05361  Uiso  ?  ?
  H4"2  .287(3)  -.0358(13)  .7366(10)  .05361  Uiso  ?  ?
  H4"3  .091(3)  .0066(12)  .6907(11)  .05361  Uiso  ?  ?
  H4'1  .702(3)  .1200(13)  .7048(11)  .05561  Uiso  ?  ?
  H4'2  .670(3)  .0340(14)  .7412(10)  .05561  Uiso  ?  ?
  H4'3  .573(3)  .1144(13)  .7717(11)  .05561  Uiso  ?  ?
  H51  .572(3)  .0453(11)  .5988(9)  .04336  Uiso  ?  ?
  H52  .501(3)  -.0401(12)  .6372(9)  .04336  Uiso  ?  ?
  H91  .547(3)  .2036(11)  .5717(9)  .04101  Uiso  ?  ?
  H92  .457(3)  .1487(11)  .5090(9)  .04101  Uiso  ?  ?

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_atom_site_aniso_U_22
_atom_site_aniso_U_33
_atom_site_aniso_U_12
_atom_site_aniso_U_13
_atom_site_aniso_U_23
  C1  .0366(7)  .0368(7)  .0351(6)  -.0026(5)  .0084(5)  -.0050(5)
  C1'  .0412(7)  .0364(7)  .0448(8)  -.0039(6)  .0121(6)  -.0063(6)
  C1"  .0563(10)  .0481(10)  .0714(12)  .0145(8)  .0128(9)  -.0041(9)
  C2  .0379(6)  .0383(7)  .0331(6)  -.0053(5)  .0078(5)  -.0026(5)
  C2'  .0604(9)  .0442(8)  .0336(7)  .0011(7)  .0082(6)  -.0029(6)
  C2"  .122(2)  .0752(15)  .0464(11)  -.0143(15)  -.0229(12)  -.0102(10)
  C4  .0393(7)  .0424(7)  .0406(7)  .0049(6)  -.0014(6)  .0004(6)
  C4'  .0542(10)  .0660(12)  .0584(11)  .0127(9)  -.0180(8)  -.0085(9)
  C4"  .0678(12)  .0481(10)  .0599(11)  .0027(8)  .0143(9)  .0100(8)
  C5  .0400(8)  .0446(8)  .0511(8)  .0087(6)  -.0020(6)  -.0055(7)
  C6  .0446(8)  .0402(8)  .0440(7)  .0056(6)  .0001(6)  -.0084(6)
  C8  .0307(6)  .0364(7)  .0315(6)  .0004(5)  .0013(5)  -.0043(5)
  C9  .0349(7)  .0564(9)  .0429(8)  .0028(6)  .0111(6)  .0004(7)
  C10  .0455(8)  .0470(8)  .0326(6)  -.0059(6)  .0064(6)  -.0047(6)
  C11  .0698(12)  .0655(11)  .0464(9)  -.0098(9)  .0209(8)  .0029(8)
  C12  .116(2)  .0618(12)  .0631(12)  -.0025(12)  .0355(13)  .0162(10)
  C13  .125(2)  .0506(11)  .0637(12)  .0191(12)  .0219(13)  .0189(9)
  C14  .0769(12)  .0481(9)  .0475(9)  .0148(9)  .0105(8)  .0032(7)
  C15  .0457(7)  .0386(7)  .0301(6)  -.0005(6)  .0018(5)  -.0038(5)
  C16  .0284(6)  .0349(6)  .0357(6)  -.0002(5)  .0025(5)  -.0057(5)
  N3  .0389(6)  .0391(6)  .0304(5)  .0024(5)  .0013(4)  -.0030(4)
  N7  .0408(6)  .0368(6)  .0462(7)  .0038(5)  -.0089(5)  -.0103(5)
  O1  .0448(6)  .0513(6)  .0537(6)  .0104(5)  .0062(5)  -.0113(5)
  O1'  .0640(7)  .0596(7)  .0525(7)  .0068(6)  .0073(5)  -.0230(6)
  O2'  .1020(11)  .0820(10)  .0400(6)  -.0176(8)  .0264(7)  -.0006(6)
  O2  .0709(8)  .0572(7)  .0364(5)  -.0119(6)  -.0073(5)  -.0041(5)
  O6  .0632(7)  .0397(6)  .0618(7)  .0097(5)  -.0110(6)  -.0152(5)

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_atom_sites_solution_secondary    ?
_atom_sites_solution_hydrogens    ?

_refine_special_details
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_refine_ls_extinction_method       Zachariasen
_refine_ls_extinction_coef         5993(1121)
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Eq22_p292_"Cryst._Comp."_Munksgaard_1970
_refine_ls_abs_structure_details   none
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_refine_ls_number_reflns           3852
_refine_ls_number_parameters       311
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_refine_ls_wR_factor_all              .059
_refine_ls_wR_factor_obs              .053
_refine_ls_goodness_of_fit_all        .425
_refine_ls_goodness_of_fit_obs        .448
_refine_ls_shift/esd_max              .049
_refine_ls_shift/esd_mean             .002
_refine_diff_density_min              -.258
_refine_diff_density_max              .260


_geom_special_details              ?

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_geom_bond_publ_flag
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  C1  C2  1.3511(19)  .  .  ?
  C1  C16  1.5139(19)  .  .  ?
  C1’  O1  1.3480(18)  .  .  ?
  C1’  O1’  1.207(2)  .  .  ?
  C1"  O1  1.436(2)  .  .  ?
  C1"  H1"1  .88(2)  .  .  ?
  C1"  H1"2  .95(2)  .  .  ?
  C1"  H1"3  .95(2)  .  .  ?
  C2  C2’  1.509(2)  .  .  ?
  C2  N3  1.3684(18)  .  .  ?
  C2’  O2’  1.191(2)  .  .  ?
  C2’  O2  1.319(2)  .  .  ?
  C2"  O2  1.448(3)  .  .  ?
  C2"  H2"1  .96(3)  .  .  ?
  C2"  H2"2  .92(3)  .  .  ?
  C2"  H2"3  .91(3)  .  .  ?
  C4  C4’  1.529(2)  .  .  ?
  C4  C4"  1.534(3)  .  .  ?
  C4  C5  1.538(2)  .  .  ?
  C4  N3  1.486(2)  .  .  ?
  C4’  H4’1  .99(2)  .  .  ?
  C4’  H4’2  .92(2)  .  .  ?
  C4’  H4’3  .98(2)  .  .  ?
  C4"  H4"1  .96(2)  .  .  ?
  C4"  H4"2  .97(2)  .  .  ?
  C4"  H4"3  1.02(2)  .  .  ?
  C5  C6  1.506(2)  .  .  ?
  C5  H51  .973(19)  .  .  ?
  C5  H52  .978(18)  .  .  ?
  C6  N7  1.342(2)  .  .  ?
```

```
  C6  O6  1.231(2)  .  .  ?
  C8  C9  1.553(2)  .  .  ?
  C8  C16  1.5659(18)  .  .  ?
  C8  N3  1.4794(16)  .  .  ?
  C8  N7  1.4525(18)  .  .  ?
  C9  C10  1.487(2)  .  .  ?
  C9  H91  .989(17)  .  .  ?
  C9  H92  .921(18)  .  .  ?
  C10  C11  1.388(3)  .  .  ?
  C10  C15  1.392(2)  .  .  ?
  C11  C12  1.371(3)  .  .  ?
  C11  H11  1.00(2)  .  .  ?
  C12  C13  1.379(4)  .  .  ?
  C12  H12  .98(2)  .  .  ?
  C13  C14  1.394(3)  .  .  ?
  C13  H13  .92(2)  .  .  ?
  C14  C15  1.386(2)  .  .  ?
  C14  H14  .94(2)  .  .  ?
  C15  C16  1.515(2)  .  .  ?
  C16  H16  .978(15)  .  .  ?
  N7  H7  .865(17)  .  .  ?

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_geom_angle_site_symmetry_3
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  C1'  C1  C2  124.42(12)  .  .  .  ?
  C1'  C1  C16  126.04(12)  .  .  .  ?
  C2  C1  C16  109.51(12)  .  .  .  ?
  C1  C1'  O1  110.42(12)  .  .  .  ?
  C1  C1'  O1'  126.16(14)  .  .  .  ?
  O1  C1'  O1'  123.40(14)  .  .  .  ?
  O1  C1"  H1"1  110.7(14)  .  .  .  ?
  O1  C1"  H1"2  110.5(13)  .  .  .  ?
  O1  C1"  H1"3  108.1(13)  .  .  .  ?
  H1"1  C1"  H1"2  99.8(18)  .  .  .  ?
  H1"1  C1"  H1"3  120.6(19)  .  .  .  ?
  H1"2  C1"  H1"3  106.7(17)  .  .  .  ?
  C1  C2  C2'  124.40(13)  .  .  .  ?
  C1  C2  N3  114.11(12)  .  .  .  ?
  C2'  C2  N3  121.36(12)  .  .  .  ?
  C2  C2'  O2'  124.65(15)  .  .  .  ?
  C2  C2'  O2  109.23(12)  .  .  .  ?
  O2'  C2'  O2  126.11(14)  .  .  .  ?
  O2  C2"  H2"1  109.0(15)  .  .  .  ?
  O2  C2"  H2"2  102.4(16)  .  .  .  ?
  O2  C2"  H2"3  105.0(15)  .  .  .  ?
  H2"1  C2"  H2"2  100(2)  .  .  .  ?
  H2"1  C2"  H2"3  126(2)  .  .  .  ?
  H2"2  C2"  H2"3  113(2)  .  .  .  ?
  C4'  C4  C4"  111.22(15)  .  .  .  ?
  C4'  C4  C5  108.76(14)  .  .  .  ?
  C4'  C4  N3  110.53(14)  .  .  .  ?
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C4"  C4  C5  108.41(14)  .  .  .  ?
C4"  C4  N3  110.97(13)  .  .  .  ?
C5  C4  N3  106.79(12)  .  .  .  ?
C4  C4'  H4'1  109.1(12)  .  .  .  ?
C4  C4'  H4'2  109.3(13)  .  .  .  ?
C4  C4'  H4'3  112.3(12)  .  .  .  ?
H4'1  C4'  H4'2  110.1(18)  .  .  .  ?
H4'1  C4'  H4'3  106.2(17)  .  .  .  ?
H4'2  C4'  H4'3  109.9(18)  .  .  .  ?
C4  C4"  H4"1  109.1(12)  .  .  .  ?
C4  C4"  H4"2  110.4(12)  .  .  .  ?
C4  C4"  H4"3  110.8(12)  .  .  .  ?
H4"1  C4"  H4"2  110.7(17)  .  .  .  ?
H4"1  C4"  H4"3  109.3(17)  .  .  .  ?
H4"2  C4"  H4"3  106.7(16)  .  .  .  ?
C4  C5  C6  112.29(13)  .  .  .  ?
C4  C5  H51  107.9(11)  .  .  .  ?
C4  C5  H52  108.4(11)  .  .  .  ?
C6  C5  H51  107.9(10)  .  .  .  ?
C6  C5  H52  107.2(10)  .  .  .  ?
H51  C5  H52  113.3(15)  .  .  .  ?
C5  C6  N7  113.85(13)  .  .  .  ?
C5  C6  O6  122.96(14)  .  .  .  ?
N7  C6  O6  123.17(14)  .  .  .  ?
C9  C8  C16  106.41(11)  .  .  .  ?
C9  C8  N3  110.96(11)  .  .  .  ?
C9  C8  N7  112.35(12)  .  .  .  ?
C16  C8  N3  105.35(10)  .  .  .  ?
C16  C8  N7  111.87(10)  .  .  .  ?
N3  C8  N7  109.65(11)  .  .  .  ?
C8  C9  C10  105.35(12)  .  .  .  ?
C8  C9  H91  110.3(11)  .  .  .  ?
C8  C9  H92  108.9(11)  .  .  .  ?
C10  C9  H91  109.5(10)  .  .  .  ?
C10  C9  H92  112.0(11)  .  .  .  ?
H91  C9  H92  110.7(15)  .  .  .  ?
C9  C10  C11  127.39(15)  .  .  .  ?
C9  C10  C15  111.94(13)  .  .  .  ?
C11  C10  C15  120.66(15)  .  .  .  ?
C10  C11  C12  119.1(2)  .  .  .  ?
C10  C11  H11  118.5(12)  .  .  .  ?
C12  C11  H11  122.3(12)  .  .  .  ?
C11  C12  C13  120.7(2)  .  .  .  ?
C11  C12  H12  121.4(14)  .  .  .  ?
C13  C12  H12  117.9(14)  .  .  .  ?
C12  C13  C14  120.8(2)  .  .  .  ?
C12  C13  H13  121.3(15)  .  .  .  ?
C14  C13  H13  117.8(15)  .  .  .  ?
C13  C14  C15  118.7(2)  .  .  .  ?
C13  C14  H14  121.2(13)  .  .  .  ?
C15  C14  H14  120.1(13)  .  .  .  ?
C10  C15  C14  120.01(15)  .  .  .  ?
C10  C15  C16  111.19(12)  .  .  .  ?
C14  C15  C16  128.79(15)  .  .  .  ?
C1  C16  C8  102.29(10)  .  .  .  ?
C1  C16  C15  116.04(11)  .  .  .  ?
C1  C16  H16  111.7(9)  .  .  .  ?
C8  C16  C15  104.35(11)  .  .  .  ?
C8  C16  H16  111.2(9)  .  .  .  ?
C15  C16  H16  110.6(9)  .  .  .  ?
C2  N3  C4  126.69(11)  .  .  .  ?
C2  N3  C8  108.59(11)  .  .  .  ?
C4  N3  C8  120.01(11)  .  .  .  ?
C6  N7  C8  120.47(12)  .  .  .  ?
C6  N7  H7  118.4(11)  .  .  .  ?
C8  N7  H7  120.0(11)  .  .  .  ?
C1'  O1  C1"  117.22(14)  .  .  .  ?
```

```
  C2′  O2  C2"  117.08(17)  .  .  .  ?

loop_
_geom_torsion_atom_site_label_1
_geom_torsion_atom_site_label_2
_geom_torsion_atom_site_label_3
_geom_torsion_atom_site_label_4
_geom_torsion
_geom_torsion_site_symmetry_1
_geom_torsion_site_symmetry_2
_geom_torsion_site_symmetry_3
_geom_torsion_site_symmetry_4
_geom_torsion_publ_flag
                                  ?  ?  ?  ?  ?  ?  ?  ?  ?  ?


_reflns_limit_h_min               0
_reflns_limit_h_max               8
_reflns_limit_k_min               0
_reflns_limit_k_max               21
_reflns_limit_l_min               -25
_reflns_limit_l_max               25
_reflns_number_total              4571
_reflns_number_observed           3298
_reflns_observed_criterion
refl_observed_if_I____>2.500_sigma(I___)
_reflns_d_resolution_high            .758
_reflns_d_resolution_low            12.341

loop_
_reflns_scale_group_code
_reflns_scale_meas_F
  ?  .448291

loop_
_refln_index_h
_refln_index_k
_refln_index_l
_refln_F_meas
_refln_F_calc
_refln_F_sigma
_refln_observed_status
_refln_scale_group_code
                                  ?  ?  ?  ?  ?  ?  ?  ?

#-end OF DATA FOR STRUCTURE OF COMPOUND 7
```