Supplementary Material for Organic & Biomolecular Chemistry

Synthesis and crystal structure of a salen-type copper(II) complex derived from 3,5'-*O*-dimethyl-2',3'-diamino-2',3'-dideoxy-β-D-*xylo*-uridine

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Supplementary experimental data:

General methods.--Electronic spectra were recorded with a Varian Cary 1 or Cary 5E spectrophotometer at room temperature. IR spectra were measured on a Perkin-Elmer 2000 spectrometer; NMR spectra on a Bruker Avance 250, mass spectra were carried out on a Finnigan MAT SSQ 710 or a Finnigan MAT 95XL TRAP, elemental analyses on a Vario EL III.

3,5'-O-dimethyluridine-2',3'-bistrifluoromethane-sulfonate **2**. 3,5'-*O*-dimethyluridine **1** (5 g, 18.4 mmol) and pyridine (3.63 g, 46 mmol) were dissolved in dry CH_2Cl_2. trifluoromethanesulfonic anhydride (12.96 g, 46 mmol) was added drop wise to the vigorously stirred solution at -20°C under an argon atmosphere. The reaction mixture was allowed to warm up to room temperature and washed two times with water to remove the formed pyridinium salts. The organic layer was dried with Na_2SO_4 and evaporated. **2** was obtained as white powder (7.66g, 88%). δ_H (250 MHz; $CDCl_3$) 7.93 (1 H, d, *J* 7.9, H-5), 6.90 (1 H, d, *J* 6.0, H-2'), 6.39 (1 H, d, *J* 7.9, H-6), 6.18 (1 H, d, *J* 5.9, H-1'), 5.60 (1 H, s, H-3'), 4.76 (1 H, s, H-4'), 3.54 (2 H, dd, *J* 4.4 and 1.9, 2 × H-5'), 3.47 (3H, s, -OC*H*$_3$), 3.08 (3 H, s, -NC*H*$_3$); δ_C ($CDCl_3$) 28.5 (NCH_3), 58.9 (OCH_3), 70.6 (C-5'), 85.7 (C-4'), 85.9 (C-2'), 90.8 (C-3'), 92.8 (C-1'), 108.5 (C-5), 114.8, 116.9, 119.8, 121.9 (2 × -CF_3), 156.3 (C-2), 158.0 (C-4).

3,5'-O-dimethyl-2',3'-diazido-2',3'-dideoxy-β-D-xylo-uridine **3** and *3-methyl-1-(3-azido-5-methoxymethyl-2(R),5(S)-dihydro-furan-2-yl)-1H-pyrimidine-2,4-dione* **4**. 2 (1.45 g, 3 mmol) was dissolved in 30 mL dry DMF. Sodium azide (980 mg, 15 mmol) was added and the suspension was stirred at 50°C for 8 hours. The solvent was evaporated, the residue extracted with ethyl acetate and water. The organic phase was dried with sodium sulphate and the solvent was evaporated yielding 1.2 g of a mixture of **3** and **4** as yellow oil. **3** and **4** were separated by column chromatography (silica gel 60, ethyl acetate/hexane 2:1, R_f(**3**) 0.7, R_f(**4**) 0.5) yielding 310 mg (32%) of **3** and 420 mg (50%) of **4** as colourless oils.
3: υ_{max} (ATR, cm^{-1}) 2102, 1707, 1657, 1457, 1248, 1092, 805, 762; δ_H (250 MHz; $CDCl_3$) 7.52 (1 H, d, *J* 8.2, H-5), 5.84 (1 H, d, *J* 3.0, H-1'), 5.75 (1 H, d, *J* 8.2, H-6), 4.35 (1 H, dt, *J* 5.1 and 4.8, H-4'), 4.13 (1 H, dd, *J* 3.1 and 3.0, H-2'), 4.02 (1 H, dd, *J* 3.1 and 1.5, H-3'), 3.64 (2 H, d, *J* 5.2, 2 × H-5'), 3.38 (3H, s, -OC*H*$_3$), 3.27 (3 H, s, -NC*H*$_3$); δ_C ($CDCl_3$) 27.6 (NCH_3), 59.4 (OCH_3), 70.0 (C-5'), 79.9 (C-4'), 69.3 (C-2'), 65.0 (C-3'), 89.2 (C-1'), 101.8 (C-5), 136.7 (C-6), 151.1 (C-2), 162.6 (C-4). MS (DEI) *m/z* 323 ($[M+1]^+$).
4: υ_{max} (ATR, cm^{-1}) 2117, 1711, 1661, 1457, 1194, 1086, 1048, 807,761; δ_H ($CDCl_3$) 7.65 (d, J_{5-6} = 8.1 Hz, H-5), 6.80 (m, H-3'), 5.71 (m, H-1'), 5.50 (d, H-6), 4.93 (m, H-4'), 3.52 (m, 2xH-5') 3.38 (s, CH_3), 3.31 (s, CH_3); δ_C ($CDCl_3$) 27.8 (NCH_3), 59.2 (OCH_3), 73.2 (C-5'), 83.8 (C-4'), 135.9 (C-2'), 86.7 (C-3'), 102.3 (C-1'), 114.3 (C-5), 138.0 (C-6), 151.7 (C-2), 162.8 (C-4). MS (DEI) *m/z* 280 ($[M+1]^+$).

3,5'-O-dimethyl-2',3'-diamino-2',3'-dideoxy-β-D-xylo-uridine **5**. In 30 mL DMF containing 230 mg hydrazine monohydrate **3** (300 mg, 930 mmol) was dissolved. The solution was warmed up to 50°C and Raney-Ni (suspension in methanol) was successively added until no further gas generation was observed. The mixture was stirred for an additional hour, filtered

and the solvent evaporated. Chromatography over silica gel (water/methanol 1:2, R_f 0.4) gave **5** (200 mg, 80%) as yellow oil (Found: C, 47.64; H, 6.49; N, 19.33. $C_{11}H_{18}N_4O_4$ requires C, 48.88; H, 6.71; N, 20.73%). υ_{max} (ATR, cm^{-1}) 3363, 2896, 1704, 1653, 1461, 1294, 1054, 804, 761; δ_H (250 MHz; $CDCl_3$) 7.73 (1 H, d, *J* 8.1, H-5), 5.71 (1 H, d, *J* 6.0, H-1'), 5.72 (1 H, d, *J* 8.3, H-6), 4.22 (1 H, dt, *J* 7.54 and 2.88, H-4'), 3.61 (2 H, d, *J* 2.9, 2 × H-5'), 3.26 (1 H, dd, *J* 6.0 and 9.5, H-3'), 3.07 (1 H, dd, *J* 6.1 and 7.9, H-2'), 3.35 (3H, s, -OC*H*$_3$), 3.25 (3 H, s, -NC*H*$_3$); δ_C ($CDCl_3$) 27.6 (NCH_3), 59.0 (OCH_3), 72.1 (C-5'), 78.2 (C-4'), 64.5 (C-2'), 59.9 (C-3'), 90.1 (C-1'), 101.5 (C-5), 138.0 (C-6), 151.9 (C-2), 162.9 (C-4). ESI-HRMS *m/z* 293.12263 ($[M+Na]^+$, $C_{11}H_{18}NaN_4O_4^+$ calc. 293.12257).

3,5'-O-dimethyl-N,N'-bis(3,5-di-tert-butylsalicylidene)-2',3'-diamino-2',3'-dideoxy-β-D-xylo-uridine **6**. To a solution of **5** (530 mg, 1.96 mmol) in dry ethanol 119 mg (3.93 mmol) of 3,5-di-tert-butylsalicyl aldehyde were added and solution was refluxed for two hours. The solvent was evaporated and the raw product was cleaned by chromatography (silica gel 60, ethyl acetate/toluene 1:4, R_f 0.6) to yield 1.30 g (95%) of **6** as yellow amorphous powder (Found: C, 70.36; H, 8.07; N, 7.45. $C_{41}H_{58}N_4O_6$ requires C, 70.06; H, 8.32; N, 7.97%). υ_{max} (ATR, cm^{-1}) 2955, 1712, 1665, 1622, 1457, 1439, 1249, 1096, 803, 760; δ_H (250 MHz; $CDCl_3$) 12.84 (1 H, s, OH), 12.70 (1 H, s, OH), 8.35 (1 H, s, =N*H*-), 8.29 (1 H, s, =N*H*-), 7.92 (1 H, d, *J* 8.2, H-5), 7.18-6.99 (4 H, m, $H_{arom.}$), 6.16 (1 H, d, *J* 4.2, H-1'), 5.82 (1 H, d, *J* 8.2, H-6), 4.66 (1 H, m, H-4'), 4.13-4.16 (2 H, m, H-2' and H-3'), 3.60 (2 H, m, 2 × H-5'), 3.40 (3H, s, -OC*H*$_3$), 3.23 (3 H, s, -NC*H*$_3$), 1.36-1.19 (36H, 4s, -C*H*$_{3\ tert\text{-}butyl}$); ESI-HRMS *m/z* 725.42586 ($[M+Na]^+$, $C_{41}H_{58}NaN_4O_6^+$ calc. 725.42541).

3,5'-O-dimethyl-2',3'-bis(3,5-di-tert-butylsalicylaldiminato)-2',3'-dideoxy-β-D-xylo-uridine-copper(II) **7**. 74 mg of 6 (0.11 mmol) and 23.4 mg (0.23 mmol) triethylamine were dissolved in 20 mL methanol. 14.2 mg (0.11 mmol) copper(II) chloride were added and the resulting dark green solution was stirred at room temperature for one day, filtered and evaporated. The residue was recrystallised from THF/methanol yielding 61 mg (76%) green crystals suitable for single crystal X-ray structure analysis (Found: C, 64.00; H, 7.44; N, 7.16. $C_{41}H_{56}CuN_4O_6$ requires C, 64.42; H, 7.38; N, 7.33%). υ_{max} (ATR, cm^{-1}) 2951, 1706, 1665, 1630, 1525, 1462, 1252, 1157, 1112, 807, 604; λ_{max}(THF)/nm 240, 268sh, 277 sh and 383 (logε 4.714, 4.483, 4.466, 4.101); MS (FAB in NBA) m/z 764 $[M+1]^+$.

Table 1. Crystal data and structure refinement for **7**.

Identification code	FO1968	
Empirical formula	C41 H56 Cu N4 O6	
Formula weight	764.44	
Temperature	183(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 11.4800(3) Å	α= 90°.
	b = 12.8012(4) Å	β= 90°.
	c = 28.1994(8) Å	γ = 90°.
Volume	4144.1(2) $Å^3$	
Z	4	
Density (calculated)	1.225 Mg/m^3	
Absorption coefficient	0.575 mm^{-1}	
F(000)	1628	
Crystal size	0.03 x 0.02 x 0.02 mm^3	
Theta range for data collection	1.92 to 27.47°.	
Index ranges	-14<=h<=14, -16<=k<=16, -36<=l<=36	
Reflections collected	8961	
Independent reflections	8961 [R(int) = 0.0000]	
Completeness to theta = 27.47°	98.5 %	

Max. and min. transmission	0.9886 and 0.9829
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8961 / 0 / 469
Goodness-of-fit on F^2	1.065
Final R indices [I>2sigma(I)]	R1 = 0.0534, wR2 = 0.1039
R indices (all data)	R1 = 0.0775, wR2 = 0.1155
Absolute structure parameter	-0.012(12)
Largest diff. peak and hole	0.397 and -0.417 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **7**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Cu	-2900(1)	11786(1)	2681(1)	22(1)
O(1)	-2308(2)	12631(2)	3177(1)	26(1)
O(2)	-3240(2)	12999(2)	2318(1)	24(1)
O(3)	-3549(2)	8037(2)	2524(1)	26(1)
O(4)	-2604(2)	9024(2)	1671(1)	32(1)
O(5)	1333(2)	6378(2)	2893(1)	34(1)
O(6)	-1629(2)	7958(2)	3688(1)	32(1)
N(1)	-2419(2)	10517(2)	3009(1)	22(1)
N(2)	-3889(2)	10879(2)	2292(1)	21(1)
N(3)	-1698(2)	7939(2)	2878(1)	24(1)
N(4)	-118(3)	7211(2)	3284(1)	26(1)
C(1)	-1799(3)	12362(3)	3570(1)	24(1)
C(2)	-1467(3)	13163(3)	3898(1)	24(1)
C(3)	-874(3)	12873(3)	4305(1)	28(1)
C(4)	-588(3)	11835(3)	4428(1)	31(1)
C(5)	-939(3)	11075(3)	4118(1)	30(1)
C(6)	-1541(3)	11304(3)	3693(1)	27(1)
C(7)	-1868(3)	10432(3)	3401(1)	27(1)
C(8)	85(3)	11614(3)	4888(1)	32(1)
C(9)	264(5)	10442(4)	4964(2)	64(2)
C(10)	-577(4)	12079(5)	5305(1)	57(2)
C(11)	1291(4)	12126(4)	4860(2)	47(1)
C(12)	-1767(3)	14309(3)	3785(1)	27(1)
C(13)	-1133(4)	14653(3)	3329(1)	33(1)
C(14)	-1422(4)	15037(3)	4190(1)	34(1)
C(15)	-3094(3)	14433(3)	3707(1)	33(1)
C(16)	-3723(3)	13037(3)	1902(1)	21(1)
C(17)	-3596(3)	13966(3)	1614(1)	25(1)
C(18)	-4198(3)	14015(3)	1190(1)	29(1)
C(19)	-4920(3)	13218(3)	1012(1)	27(1)
C(20)	-4990(3)	12323(3)	1273(1)	26(1)
C(21)	-4396(3)	12198(3)	1710(1)	24(1)
C(22)	-4472(3)	11179(3)	1927(1)	22(1)
C(23)	-5517(4)	13357(3)	524(1)	36(1)
C(24)	-6262(5)	14350(4)	528(2)	66(2)
C(25)	-4565(5)	13437(5)	147(2)	70(2)
C(26)	-6312(4)	12440(3)	403(1)	38(1)
C(27)	-2777(4)	14853(3)	1776(1)	28(1)
C(28)	-1536(3)	14424(3)	1842(2)	32(1)
C(29)	-3177(3)	15350(3)	2246(1)	31(1)
C(30)	-2700(4)	15736(3)	1407(1)	40(1)
C(31)	-2808(3)	8523(2)	2872(1)	24(1)
C(32)	-2688(3)	9637(2)	2697(1)	22(1)
C(33)	-3904(3)	9831(2)	2496(1)	23(1)
C(34)	-4158(3)	8807(3)	2234(1)	25(1)
C(35)	-3784(3)	8708(3)	1725(1)	28(1)
C(36)	-2239(4)	8996(4)	1191(1)	51(1)
C(37)	-1178(3)	7686(3)	2453(1)	26(1)
C(38)	-171(3)	7159(3)	2433(1)	28(1)
C(39)	424(3)	6870(3)	2865(1)	27(1)
C(40)	-1185(3)	7708(3)	3310(1)	27(1)
C(41)	512(3)	7029(3)	3730(1)	34(1)

Table 3. Bond lengths [Å] and angles [°] for **7**.

Cu-O(1)	1.894(2)	O(1)-Cu-N(1)	91.34(11)	C(20)-C(21)-C(16)	120.2(3)
Cu-O(2)	1.900(2)	O(2)-Cu-N(1)	174.21(11)	C(20)-C(21)-C(22)	116.2(3)
Cu-N(1)	1.950(3)	O(1)-Cu-N(2)	163.65(11)	C(16)-C(21)-C(22)	123.5(3)
Cu-N(2)	1.960(3)	O(2)-Cu-N(2)	93.72(11)	N(2)-C(22)-C(21)	125.3(3)
O(1)-C(1)	1.301(4)	N(1)-Cu-N(2)	86.34(11)	C(25)-C(23)-C(26)	108.9(4)
O(2)-C(16)	1.300(4)	C(1)-O(1)-Cu	129.9(2)	C(25)-C(23)-C(24)	110.4(4)
O(3)-C(31)	1.439(4)	C(16)-O(2)-Cu	127.2(2)	C(26)-C(23)-C(24)	107.8(4)
O(3)-C(34)	1.459(4)	C(31)-O(3)-C(34)	111.9(2)	C(25)-C(23)-C(19)	108.0(3)
O(4)-C(36)	1.416(5)	C(36)-O(4)-C(35)	112.2(3)	C(26)-C(23)-C(19)	112.1(3)
O(4)-C(35)	1.421(4)	C(7)-N(1)-C(32)	124.0(3)	C(24)-C(23)-C(19)	109.7(3)
O(5)-C(39)	1.222(4)	C(7)-N(1)-Cu	128.4(2)	C(17)-C(27)-C(30)	112.1(3)
O(6)-C(40)	1.224(4)	C(32)-N(1)-Cu	107.2(2)	C(17)-C(27)-C(28)	109.7(3)
N(1)-C(7)	1.277(4)	C(22)-N(2)-C(33)	125.8(3)	C(30)-C(27)-C(28)	107.0(3)
N(1)-C(32)	1.462(4)	C(22)-N(2)-Cu	124.9(2)	C(17)-C(27)-C(29)	112.1(3)
N(2)-C(22)	1.285(4)	C(33)-N(2)-Cu	109.2(2)	C(30)-C(27)-C(29)	107.3(3)
N(2)-C(33)	1.460(4)	C(37)-N(3)-C(40)	122.1(3)	C(28)-C(27)-C(29)	108.5(3)
N(3)-C(37)	1.378(4)	C(37)-N(3)-C(31)	118.8(3)	O(3)-C(31)-N(3)	107.4(2)
N(3)-C(40)	1.386(5)	C(40)-N(3)-C(31)	119.0(3)	O(3)-C(31)-C(32)	103.9(3)
N(3)-C(31)	1.478(4)	C(40)-N(4)-C(39)	125.5(3)	N(3)-C(31)-C(32)	113.7(3)
N(4)-C(40)	1.382(5)	C(40)-N(4)-C(41)	117.6(3)	N(1)-C(32)-C(31)	123.2(3)
N(4)-C(39)	1.403(5)	C(39)-N(4)-C(41)	116.9(3)	N(1)-C(32)-C(33)	106.9(3)
N(4)-C(41)	1.470(5)	O(1)-C(1)-C(2)	118.8(3)	C(31)-C(32)-C(33)	101.0(3)
C(1)-C(2)	1.431(5)	O(1)-C(1)-C(6)	123.3(3)	N(2)-C(33)-C(32)	106.6(3)
C(1)-C(6)	1.429(5)	C(2)-C(1)-C(6)	117.9(3)	N(2)-C(33)-C(34)	126.7(3)
C(2)-C(3)	1.386(5)	C(3)-C(2)-C(1)	118.3(3)	C(32)-C(33)-C(34)	102.3(3)
C(2)-C(12)	1.541(5)	C(3)-C(2)-C(12)	122.4(3)	O(3)-C(34)-C(35)	110.0(3)
C(3)-C(4)	1.411(5)	C(1)-C(2)-C(12)	119.3(3)	O(3)-C(34)-C(33)	102.5(3)
C(4)-C(5)	1.369(5)	C(2)-C(3)-C(4)	124.7(3)	C(35)-C(34)-C(33)	118.5(3)
C(4)-C(8)	1.536(5)	C(5)-C(4)-C(3)	116.4(3)	O(4)-C(35)-C(34)	110.6(3)
C(5)-C(6)	1.414(5)	C(5)-C(4)-C(8)	123.8(4)	C(38)-C(37)-N(3)	121.8(3)
C(6)-C(7)	1.437(5)	C(3)-C(4)-C(8)	119.9(3)	C(37)-C(38)-C(39)	120.1(3)
C(8)-C(10)	1.521(6)	C(4)-C(5)-C(6)	122.5(3)	O(5)-C(39)-N(4)	119.1(3)
C(8)-C(9)	1.529(6)	C(5)-C(6)-C(1)	120.1(3)	O(5)-C(39)-C(38)	126.1(3)
C(8)-C(11)	1.534(6)	C(5)-C(6)-C(7)	116.9(3)	N(4)-C(39)-C(38)	114.8(3)
C(12)-C(14)	1.526(5)	C(1)-C(6)-C(7)	123.0(3)	O(6)-C(40)-N(4)	122.4(3)
C(12)-C(15)	1.547(5)	N(1)-C(7)-C(6)	124.0(3)	O(6)-C(40)-N(3)	122.2(3)
C(12)-C(13)	1.543(5)	C(10)-C(8)-C(9)	110.0(4)	N(4)-C(40)-N(3)	115.3(3)
C(16)-C(21)	1.430(5)	C(10)-C(8)-C(11)	108.9(4)		
C(16)-C(17)	1.447(5)	C(9)-C(8)-C(11)	107.7(4)		
C(17)-C(18)	1.382(5)	C(10)-C(8)-C(4)	109.2(3)		
C(17)-C(27)	1.542(5)	C(9)-C(8)-C(4)	111.6(3)		
C(18)-C(19)	1.408(5)	C(11)-C(8)-C(4)	109.4(3)		
C(19)-C(20)	1.364(5)	C(14)-C(12)-C(2)	111.6(3)		
C(19)-C(23)	1.547(5)	C(14)-C(12)-C(15)	107.4(3)		
C(20)-C(21)	1.418(5)	C(2)-C(12)-C(15)	110.3(3)		
C(21)-C(22)	1.442(5)	C(14)-C(12)-C(13)	109.1(3)		
C(23)-C(25)	1.529(6)	C(2)-C(12)-C(13)	109.8(3)		
C(23)-C(26)	1.526(6)	C(15)-C(12)-C(13)	108.5(3)		
C(23)-C(24)	1.532(6)	O(2)-C(16)-C(21)	122.9(3)		
C(27)-C(30)	1.538(5)	O(2)-C(16)-C(17)	119.6(3)		
C(27)-C(28)	1.539(5)	C(21)-C(16)-C(17)	117.4(3)		
C(27)-C(29)	1.541(5)	C(18)-C(17)-C(16)	118.1(3)		
C(31)-C(32)	1.516(4)	C(18)-C(17)-C(27)	121.8(3)		
C(32)-C(33)	1.526(5)	C(16)-C(17)-C(27)	120.1(3)		
C(33)-C(34)	1.533(5)	C(17)-C(18)-C(19)	124.8(3)		
C(34)-C(35)	1.504(5)	C(20)-C(19)-C(18)	116.8(3)		
C(37)-C(38)	1.340(5)	C(20)-C(19)-C(23)	123.4(3)		
C(38)-C(39)	1.445(5)	C(18)-C(19)-C(23)	119.7(3)		
O(1)-Cu-O(2)	90.20(10)	C(19)-C(20)-C(21)	122.4(3)		

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **7**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Cu	23(1)	19(1)	24(1)	0(1)	-2(1)	-1(1)
O(1)	35(2)	22(1)	23(1)	-1(1)	-6(1)	-2(1)
O(2)	27(1)	22(1)	23(1)	2(1)	-3(1)	-1(1)
O(3)	20(1)	22(1)	37(1)	0(1)	-1(1)	-3(1)
O(4)	27(2)	44(2)	26(1)	-4(1)	2(1)	-6(1)
O(5)	19(1)	36(2)	46(2)	6(1)	3(1)	6(1)
O(6)	37(2)	32(2)	27(1)	3(1)	3(1)	10(1)
N(1)	21(2)	21(1)	25(2)	-1(1)	1(1)	3(1)
N(2)	21(1)	18(1)	24(1)	-2(1)	-1(1)	-2(1)
N(3)	25(2)	22(2)	26(2)	-1(1)	2(1)	2(1)
N(4)	22(2)	26(2)	31(2)	1(1)	2(1)	-2(1)
C(1)	21(2)	29(2)	24(2)	0(2)	2(1)	-1(2)
C(2)	19(2)	26(2)	26(2)	2(2)	3(1)	-2(2)
C(3)	23(2)	36(2)	25(2)	-3(2)	0(1)	-4(2)
C(4)	26(2)	39(2)	27(2)	4(2)	-3(1)	-1(2)
C(5)	33(2)	28(2)	30(2)	5(2)	-4(2)	1(2)
C(6)	30(2)	24(2)	26(2)	-1(2)	2(2)	-2(2)
C(7)	28(2)	23(2)	30(2)	6(2)	-1(2)	2(2)
C(8)	29(2)	36(2)	31(2)	6(2)	-7(2)	-1(2)
C(9)	86(4)	47(3)	58(3)	12(2)	-39(3)	-6(3)
C(10)	47(3)	95(4)	28(2)	12(2)	-4(2)	10(3)
C(11)	37(3)	66(3)	38(2)	1(2)	-9(2)	1(2)
C(12)	25(2)	27(2)	28(2)	-6(2)	-3(1)	-1(2)
C(13)	37(2)	25(2)	37(2)	-1(2)	-3(2)	-5(2)
C(14)	40(2)	27(2)	36(2)	-10(2)	-5(2)	0(2)
C(15)	28(2)	32(2)	38(2)	-6(2)	-3(2)	5(2)
C(16)	16(2)	21(2)	25(2)	0(1)	0(1)	4(1)
C(17)	26(2)	22(2)	26(2)	-2(1)	3(2)	0(2)
C(18)	32(2)	26(2)	30(2)	4(2)	1(2)	3(2)
C(19)	30(2)	28(2)	24(2)	-1(2)	0(1)	-1(2)
C(20)	24(2)	27(2)	27(2)	-2(2)	-1(2)	-4(2)
C(21)	23(2)	20(2)	29(2)	-2(2)	3(2)	0(1)
C(22)	15(2)	20(2)	32(2)	-5(2)	1(2)	-5(1)
C(23)	44(2)	37(2)	28(2)	5(2)	-4(2)	-5(2)
C(24)	92(4)	40(3)	65(3)	9(2)	-42(3)	1(3)
C(25)	67(4)	115(5)	27(2)	11(3)	-3(2)	-37(4)
C(26)	37(2)	44(2)	34(2)	4(2)	-7(2)	-4(2)
C(27)	28(2)	23(2)	34(2)	-1(2)	1(2)	-7(2)
C(28)	26(2)	25(2)	46(2)	-3(2)	2(2)	0(2)
C(29)	27(2)	27(2)	41(2)	-5(2)	2(2)	-5(2)
C(30)	45(3)	29(2)	46(2)	4(2)	-3(2)	-13(2)
C(31)	20(2)	24(2)	29(2)	-1(1)	1(2)	3(2)
C(32)	21(2)	19(1)	27(2)	0(2)	3(2)	-4(1)
C(33)	24(2)	17(2)	27(2)	3(1)	0(1)	-1(1)
C(34)	17(2)	22(2)	35(2)	-2(2)	-1(1)	2(1)
C(35)	25(2)	28(2)	30(2)	-3(2)	-5(2)	-3(2)
C(36)	41(3)	85(3)	28(2)	-4(2)	5(2)	-13(3)
C(37)	25(2)	26(2)	28(2)	1(2)	2(1)	-3(2)
C(38)	25(2)	28(2)	30(2)	-3(2)	1(2)	-2(2)
C(39)	23(2)	22(2)	37(2)	0(2)	2(2)	-4(2)
C(40)	28(2)	20(2)	31(2)	1(2)	2(2)	-1(2)
C(41)	33(2)	35(2)	35(2)	5(2)	-7(2)	3(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **7**.

	x	y	z	U(eq)
H(3A)	-644	13411	4517	34
H(5A)	-771	10366	4191	36
H(7A)	-1662	9751	3505	32
H(9A)	693	10328	5260	95
H(9B)	710	10153	4698	95
H(9C)	-495	10095	4983	95
H(10A)	-686	12830	5253	85
H(10B)	-133	11967	5597	85
H(10C)	-1339	11739	5332	85
H(11A)	1202	12880	4814	71
H(11B)	1725	11830	4592	71
H(11C)	1718	11994	5155	71
H(13A)	-290	14579	3372	49
H(13B)	-1320	15385	3260	49
H(13C)	-1385	14213	3063	49
H(14A)	-583	14978	4248	51
H(14B)	-1848	14840	4477	51
H(14C)	-1614	15759	4104	51
H(15A)	-3510	14219	3995	49
H(15B)	-3341	13994	3441	49
H(15C)	-3274	15165	3637	49
H(18A)	-4118	14632	1006	35
H(20A)	-5454	11764	1158	31
H(22A)	-4995	10691	1789	27
H(24A)	-6637	14437	219	98
H(24B)	-5766	14955	594	98
H(24C)	-6860	14291	775	98
H(25A)	-4100	12795	147	104
H(25B)	-4061	14035	217	104
H(25C)	-4924	13532	-165	104
H(26A)	-5852	11795	392	57
H(26B)	-6676	12560	93	57
H(26C)	-6918	12375	646	57
H(28A)	-1550	13853	2074	48
H(28B)	-1026	14983	1957	48
H(28C)	-1243	14163	1538	48
H(29A)	-3232	14808	2491	47
H(29B)	-3941	15676	2202	47
H(29C)	-2612	15880	2345	47
H(30A)	-2443	15447	1102	60
H(30B)	-2140	16263	1514	60
H(30C)	-3468	16059	1368	60
H(31A)	-3184	8507	3191	29
H(32A)	-2120	9643	2428	27
H(33A)	-4447	9857	2773	27
H(34A)	-5013	8665	2253	30
H(35A)	-3871	7974	1621	33
H(35B)	-4289	9149	1523	33
H(36A)	-1425	9223	1170	77
H(36B)	-2730	9464	1002	77
H(36C)	-2309	8281	1069	77
H(37A)	-1544	7891	2165	31
H(38A)	153	6975	2135	33
H(41A)	50	7296	3996	51
H(41B)	1264	7391	3720	51
H(41C)	642	6278	3772	51