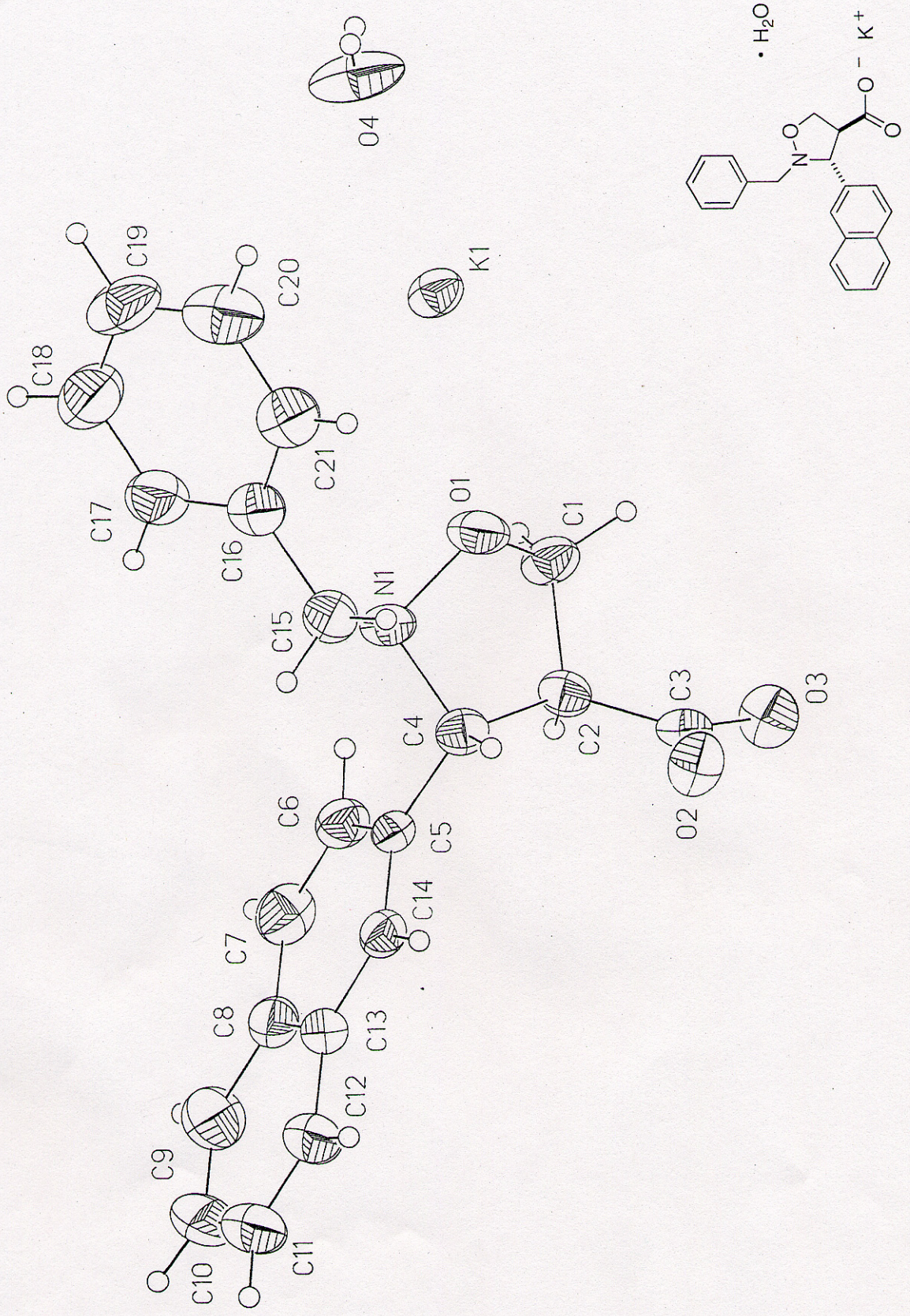
O4
K1
C19
C20
C18
C21
C17
C16
O1
C1
C15
N1
C4
C2
C3
O3
O2
C6
C5
C14
C7
C8
C13
C12
C9
C11
C10
• H2O
O
N
O
O
K+

Table 1. Crystal data and structure refinement for WSJ01.

Empirical formula	$[C_{21}H_{18}NO_3]^- K^+ H_2O$
Formula weight	389.48
Crystallization Solvent	Ethanol/THF
Crystal Habit	Blade
Crystal size	0.31 x 0.25 x 0.11 mm^3
Crystal color	Colorless

Data Collection

Preliminary Photos	Rotation
Type of diffractometer	CCD area detector
Wavelength	0.71073 Å MoKα
Data Collection Temperature	98(2) K
θ range for 7998 reflections used in lattice determination	2.50 to 25.50°
Unit cell dimensions	a = 6.7539(13) Å b = 8.2935(16) Å β= 91.678(3)° c = 34.251(7) Å
Volume	1917.7(6) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.349 Mg/m^3
F(000)	816
Data collection program	Bruker SMART
θ range for data collection	2.38 to 28.67°
Completeness to θ = 28.67°	92.4 %
Index ranges	$-8 \le h \le 9, -10 \le k \le 10, -45 \le l \le 44$
Data collection scan type	ω scans at 7 ϕ settings
Data reduction program	Bruker SAINT v6.1
Reflections collected	33077
Independent reflections	4566 [R_{int}= 0.1032]
Absorption coefficient	0.303 mm^{-1}
Absorption correction	None
Max. and min. transmission (theory)	0.9671 and 0.9105

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 1990)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 1997)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	4566 / 0 / 324
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.798
Final R indices [$I>2\sigma(I)$, 3026 reflections]	R1 = 0.0733, *w*R2 = 0.0929
R indices (all data)	R1 = 0.1131, *w*R2 = 0.0960
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	0.605 and -0.385 e.Å^{-3}

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for WSJ01. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
K(1)	9902(1)	7017(1)	9649(1)	39(1)
O(1)	6333(3)	5665(2)	9377(1)	46(1)
O(2)	1388(3)	4094(2)	9634(1)	45(1)
O(3)	3030(2)	1983(2)	9870(1)	46(1)
O(4)	13167(4)	8714(3)	9781(1)	72(1)
N(1)	5728(3)	5485(3)	8958(1)	36(1)
C(1)	6302(4)	4064(4)	9509(1)	42(1)
C(2)	4406(4)	3321(3)	9330(1)	34(1)
C(3)	2798(4)	3142(3)	9630(1)	33(1)
C(4)	3909(4)	4476(3)	8992(1)	34(1)
C(5)	3433(4)	3665(3)	8609(1)	30(1)
C(6)	4816(4)	2617(3)	8443(1)	39(1)
C(7)	4389(5)	1804(4)	8110(1)	48(1)
C(8)	2529(4)	1988(4)	7911(1)	43(1)
C(9)	2041(6)	1171(5)	7562(1)	63(1)
C(10)	240(6)	1381(5)	7379(1)	75(1)
C(11)	-1143(5)	2419(6)	7541(1)	70(1)
C(12)	-736(4)	3218(5)	7878(1)	52(1)
C(13)	1128(4)	3038(4)	8076(1)	37(1)
C(14)	1639(4)	3859(3)	8424(1)	33(1)
C(15)	5256(4)	7128(4)	8838(1)	43(1)
C(16)	7090(4)	8137(3)	8809(1)	40(1)
C(17)	8462(5)	7830(4)	8522(1)	52(1)
C(18)	10192(5)	8717(5)	8506(1)	64(1)
C(19)	10583(6)	9911(5)	8772(1)	65(1)
C(20)	9263(6)	10220(4)	9058(1)	64(1)
C(21)	7510(5)	9346(4)	9074(1)	49(1)

Table 4. Bond lengths [Å] and angles [°] for WSJ01.

K(1)-O(2)#1	2.625(2)	C(15)-H(15B)	0.96(3)
K(1)-O(4)	2.644(2)	C(16)-C(21)	1.377(4)
K(1)-O(3)#2	2.743(2)	C(16)-C(17)	1.392(4)
K(1)-O(2)#2	2.789(2)	C(17)-C(18)	1.383(5)
K(1)-O(1)	2.7932(18)	C(17)-H(17)	0.95(4)
K(1)-C(3)#2	3.117(3)	C(18)-C(19)	1.366(5)
K(1)-C(21)	3.167(3)	C(18)-H(18)	1.02(3)
K(1)-C(20)	3.359(4)	C(19)-C(20)	1.368(6)
K(1)-C(1)	3.475(3)	C(19)-H(19)	0.98(3)
K(1)-C(16)	3.525(3)	C(20)-C(21)	1.391(5)
K(1)-K(1)#3	4.1212(14)	C(20)-H(20)	0.90(4)
O(1)-C(1)	1.403(3)	C(21)-H(21)	0.83(2)
O(1)-N(1)	1.486(3)		
O(2)-C(3)	1.237(3)	O(2)#1-K(1)-O(4)	100.22(8)
O(2)-K(1)#4	2.625(2)	O(2)#1-K(1)-O(3)#2	125.05(6)
O(2)-K(1)#2	2.789(2)	O(4)-K(1)-O(3)#2	110.44(8)
O(3)-C(3)	1.271(3)	O(2)#1-K(1)-O(2)#2	80.89(7)
O(3)-K(1)#2	2.743(2)	O(4)-K(1)-O(2)#2	107.79(8)
O(4)-H(4AA)	0.92(4)	O(3)#2-K(1)-O(2)#2	47.15(6)
O(4)-H(44B)	0.73(3)	O(2)#1-K(1)-O(1)	87.11(6)
N(1)-C(15)	1.457(4)	O(4)-K(1)-O(1)	167.96(7)
N(1)-C(4)	1.493(3)	O(3)#2-K(1)-O(1)	71.99(6)
C(1)-C(2)	1.531(4)	O(2)#2-K(1)-O(1)	82.67(6)
C(1)-H(1A)	0.96(2)	O(2)#1-K(1)-C(3)#2	102.09(7)
C(1)-H(1B)	1.05(3)	O(4)-K(1)-C(3)#2	113.03(8)
C(2)-C(3)	1.523(4)	O(3)#2-K(1)-C(3)#2	23.98(6)
C(2)-C(4)	1.534(4)	O(2)#2-K(1)-C(3)#2	23.35(6)
C(2)-H(2A)	1.03(3)	O(1)-K(1)-C(3)#2	74.22(6)
C(3)-K(1)#2	3.117(3)	O(2)#1-K(1)-C(21)	137.69(9)
C(4)-C(5)	1.500(4)	O(4)-K(1)-C(21)	100.92(9)
C(4)-H(4A)	0.99(2)	O(3)#2-K(1)-C(21)	79.94(9)
C(5)-C(14)	1.360(3)	O(2)#2-K(1)-C(21)	125.63(9)
C(5)-C(6)	1.408(4)	O(1)-K(1)-C(21)	67.55(7)
C(6)-C(7)	1.348(4)	C(3)#2-K(1)-C(21)	102.65(10)
C(6)-H(6)	0.97(2)	O(2)#1-K(1)-C(20)	139.56(10)
C(7)-C(8)	1.420(4)	O(4)-K(1)-C(20)	76.92(9)
C(7)-H(7)	0.95(3)	O(3)#2-K(1)-C(20)	92.34(10)
C(8)-C(9)	1.405(4)	O(2)#2-K(1)-C(20)	138.95(10)
C(8)-C(13)	1.415(4)	O(1)-K(1)-C(20)	91.29(7)
C(9)-C(10)	1.364(5)	C(3)#2-K(1)-C(20)	116.30(11)
C(9)-H(9)	0.96(3)	C(21)-K(1)-C(20)	24.38(9)
C(10)-C(11)	1.397(5)	O(2)#1-K(1)-C(1)	67.22(7)
C(10)-H(10)	0.95(4)	O(4)-K(1)-C(1)	167.33(8)
C(11)-C(12)	1.352(5)	O(3)#2-K(1)-C(1)	77.39(8)
C(11)-H(11)	0.93(3)	O(2)#2-K(1)-C(1)	69.73(7)
C(12)-C(13)	1.420(4)	O(1)-K(1)-C(1)	22.71(6)
C(12)-H(12)	1.02(3)	C(3)#2-K(1)-C(1)	69.90(8)
C(13)-C(14)	1.409(4)	C(21)-K(1)-C(1)	90.11(8)
C(14)-H(14)	0.88(2)	C(20)-K(1)-C(1)	113.42(8)
C(15)-C(16)	1.501(4)	O(2)#1-K(1)-C(16)	115.12(7)
C(15)-H(15A)	0.90(3)	O(4)-K(1)-C(16)	115.23(8)

O(3)#2-K(1)-C(16)	91.73(7)	C(5)-C(4)-C(2)	114.6(2)
O(2)#2-K(1)-C(16)	129.18(7)	N(1)-C(4)-H(4A)	106.9(12)
O(1)-K(1)-C(16)	52.75(6)	C(5)-C(4)-H(4A)	112.2(12)
C(3)#2-K(1)-C(16)	110.15(7)	C(2)-C(4)-H(4A)	108.4(13)
C(21)-K(1)-C(16)	22.96(8)	C(14)-C(5)-C(6)	118.5(3)
C(20)-K(1)-C(16)	40.86(8)	C(14)-C(5)-C(4)	121.3(3)
C(1)-K(1)-C(16)	73.44(7)	C(6)-C(5)-C(4)	120.2(2)
O(2)#1-K(1)-K(1)#3	41.93(4)	C(7)-C(6)-C(5)	121.5(3)
O(4)-K(1)-K(1)#3	108.65(6)	C(7)-C(6)-H(6)	120.4(16)
O(3)#2-K(1)-K(1)#3	84.58(5)	C(5)-C(6)-H(6)	117.9(16)
O(2)#2-K(1)-K(1)#3	38.96(4)	C(6)-C(7)-C(8)	121.1(3)
O(1)-K(1)-K(1)#3	83.20(4)	C(6)-C(7)-H(7)	121.3(17)
C(3)#2-K(1)-K(1)#3	60.80(6)	C(8)-C(7)-H(7)	117.6(17)
C(21)-K(1)-K(1)#3	149.91(7)	C(7)-C(8)-C(9)	122.6(3)
C(20)-K(1)-K(1)#3	174.29(7)	C(7)-C(8)-C(13)	117.9(3)
C(1)-K(1)-K(1)#3	61.24(5)	C(9)-C(8)-C(13)	119.5(3)
C(16)-K(1)-K(1)#3	134.26(5)	C(10)-C(9)-C(8)	120.9(4)
C(1)-O(1)-N(1)	102.03(19)	C(10)-C(9)-H(9)	119.2(17)
C(1)-O(1)-K(1)	107.07(14)	C(8)-C(9)-H(9)	119.8(17)
N(1)-O(1)-K(1)	124.89(14)	C(9)-C(10)-C(11)	119.6(4)
C(3)-O(2)-K(1)#4	152.17(17)	C(9)-C(10)-H(10)	119(2)
C(3)-O(2)-K(1)#2	93.37(16)	C(11)-C(10)-H(10)	121(2)
K(1)#4-O(2)-K(1)#2	99.11(6)	C(12)-C(11)-C(10)	121.3(3)
C(3)-O(3)-K(1)#2	94.74(16)	C(12)-C(11)-H(11)	117(2)
K(1)-O(4)-H(4AA)	117(2)	C(10)-C(11)-H(11)	121.7(19)
K(1)-O(4)-H(44B)	135(3)	C(11)-C(12)-C(13)	120.7(4)
H(4AA)-O(4)-H(44B)	104(3)	C(11)-C(12)-H(12)	126.3(15)
C(15)-N(1)-O(1)	103.4(2)	C(13)-C(12)-H(12)	112.9(15)
C(15)-N(1)-C(4)	111.8(2)	C(14)-C(13)-C(8)	119.0(2)
O(1)-N(1)-C(4)	100.70(19)	C(14)-C(13)-C(12)	123.0(3)
O(1)-C(1)-C(2)	105.8(2)	C(8)-C(13)-C(12)	118.0(3)
O(1)-C(1)-K(1)	50.22(11)	C(5)-C(14)-C(13)	122.0(3)
C(2)-C(1)-K(1)	155.79(19)	C(5)-C(14)-H(14)	118.4(13)
O(1)-C(1)-H(1A)	111.1(15)	C(13)-C(14)-H(14)	119.4(13)
C(2)-C(1)-H(1A)	113.9(14)	N(1)-C(15)-C(16)	111.5(2)
K(1)-C(1)-H(1A)	77.7(14)	N(1)-C(15)-H(15A)	105(2)
O(1)-C(1)-H(1B)	105.2(17)	C(16)-C(15)-H(15A)	111.2(18)
C(2)-C(1)-H(1B)	116.2(16)	N(1)-C(15)-H(15B)	109.8(16)
K(1)-C(1)-H(1B)	78.6(17)	C(16)-C(15)-H(15B)	111.5(15)
H(1A)-C(1)-H(1B)	104(2)	H(15A)-C(15)-H(15B)	107(2)
C(1)-C(2)-C(3)	112.1(2)	C(21)-C(16)-C(17)	118.0(3)
C(1)-C(2)-C(4)	102.2(2)	C(21)-C(16)-C(15)	121.1(3)
C(3)-C(2)-C(4)	115.2(2)	C(17)-C(16)-C(15)	120.8(3)
C(1)-C(2)-H(2A)	109.9(13)	C(21)-C(16)-K(1)	63.76(15)
C(3)-C(2)-H(2A)	108.7(15)	C(17)-C(16)-K(1)	99.94(17)
C(4)-C(2)-H(2A)	108.5(14)	C(15)-C(16)-K(1)	103.01(17)
O(2)-C(3)-O(3)	123.8(3)	C(18)-C(17)-C(16)	120.8(4)
O(2)-C(3)-C(2)	120.6(2)	C(18)-C(17)-H(17)	120(2)
O(3)-C(3)-C(2)	115.6(2)	C(16)-C(17)-H(17)	119(2)
O(2)-C(3)-K(1)#2	63.28(14)	C(19)-C(18)-C(17)	120.3(4)
O(3)-C(3)-K(1)#2	61.28(14)	C(19)-C(18)-H(18)	124.6(18)
C(2)-C(3)-K(1)#2	169.60(15)	C(17)-C(18)-H(18)	115.0(18)
N(1)-C(4)-C(5)	109.9(2)	C(18)-C(19)-C(20)	119.8(4)
N(1)-C(4)-C(2)	104.23(19)	C(18)-C(19)-H(19)	120.3(19)

C(20)-C(19)-H(19)	119.9(19)	C(20)-C(21)-C(16)	120.9(4)
C(21)-C(20)-C(19)	120.2(4)	C(20)-C(21)-K(1)	85.54(18)
C(21)-C(20)-K(1)	70.07(18)	C(16)-C(21)-K(1)	93.29(18)
C(19)-C(20)-K(1)	102.0(2)	C(20)-C(21)-H(21)	119.8(18)
C(21)-C(20)-H(20)	119(2)	C(16)-C(21)-H(21)	119.3(18)
C(19)-C(20)-H(20)	121(2)	K(1)-C(21)-H(21)	91.0(17)
K(1)-C(20)-H(20)	98(2)		

Symmetry transformations used to generate equivalent atoms:
#1 x+1,y,z
#2 -x+1,-y+1,-z+2
#3 -x+2,-y+1,-z+2
#4 x-1,y,z

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for WSJ01. 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}
K(1)	298(3)	364(3)	490(4)	71(3)	-36(3)	-18(3)
O(1)	451(12)	431(13)	483(13)	54(10)	-191(10)	-98(10)
O(2)	347(10)	452(12)	548(13)	134(10)	66(9)	138(10)
O(3)	472(11)	368(11)	527(12)	73(11)	12(9)	88(10)
O(4)	414(14)	412(15)	1310(30)	64(15)	-340(16)	-46(12)
N(1)	313(12)	328(13)	429(15)	15(11)	-91(11)	-55(10)
C(1)	276(16)	442(19)	530(20)	80(16)	-59(15)	-18(14)
C(2)	315(15)	353(18)	344(16)	7(13)	-65(12)	32(12)
C(3)	290(14)	312(15)	382(16)	-44(14)	-77(12)	-45(13)
C(4)	271(14)	344(16)	397(18)	-27(14)	-8(13)	20(13)
C(5)	277(14)	313(15)	298(15)	30(12)	-12(12)	-73(12)
C(6)	296(15)	442(19)	434(18)	-20(14)	17(14)	-9(13)
C(7)	447(18)	454(19)	540(20)	-89(18)	122(16)	13(16)
C(8)	463(16)	481(18)	358(17)	-70(16)	66(14)	-147(16)
C(9)	650(20)	760(30)	490(20)	-200(20)	108(19)	-100(20)
C(10)	720(30)	1100(40)	420(20)	-260(20)	-20(20)	-320(20)
C(11)	480(20)	1130(40)	470(20)	-80(20)	-100(19)	-190(20)
C(12)	417(18)	700(20)	420(19)	-19(19)	-73(15)	-58(18)
C(13)	354(15)	438(17)	320(15)	3(15)	-10(12)	-94(15)
C(14)	273(15)	363(16)	347(17)	18(14)	30(13)	-12(13)
C(15)	350(16)	510(20)	421(19)	96(17)	-63(15)	23(16)
C(16)	415(16)	306(15)	457(18)	79(15)	-77(14)	41(14)
C(17)	500(20)	560(20)	510(20)	40(20)	-89(17)	-51(18)
C(18)	510(20)	680(30)	730(30)	180(20)	10(20)	-100(20)
C(19)	480(20)	530(20)	920(30)	320(20)	-170(20)	-170(20)
C(20)	740(30)	330(20)	820(30)	50(20)	-270(20)	-25(19)
C(21)	470(20)	430(20)	570(20)	77(18)	-46(19)	62(17)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for WSJ01.

	x	y	z	U_{iso}
H(1A)	7490(40)	3500(30)	9442(7)	31(7)
H(1B)	6380(40)	4140(40)	9815(10)	64(10)
H(2A)	4710(30)	2200(40)	9216(7)	48(8)
H(4A)	2830(30)	5210(30)	9073(6)	17(6)
H(6)	6130(40)	2550(30)	8565(7)	47(8)
H(7)	5300(40)	1070(40)	8005(8)	54(9)
H(9)	2960(40)	420(40)	7456(9)	53(10)
H(10)	-10(50)	870(50)	7134(12)	99(13)
H(11)	-2380(40)	2590(40)	7423(9)	58(9)
H(12)	-1630(40)	4020(30)	8009(8)	43(8)
H(14)	740(30)	4460(30)	8538(6)	10(6)
H(15A)	4620(40)	7040(40)	8604(8)	49(8)
H(15B)	4340(40)	7590(30)	9014(7)	36(8)
H(17)	8190(40)	7000(40)	8337(10)	79(12)
H(18)	11090(40)	8420(40)	8283(10)	65(10)
H(19)	11810(40)	10540(40)	8760(9)	65(10)
H(20)	9510(50)	11000(40)	9238(10)	73(12)
H(21)	6700(30)	9550(30)	9246(7)	20(8)
H(4AA)	13020(50)	9790(50)	9838(11)	95(15)
H(44B)	14180(50)	8520(40)	9846(10)	69(13)

Table 7. Hydrogen bonds for WSJ01 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(4)-H(4AA)...O(3)#5	0.92(4)	1.82(4)	2.730(3)	169(4)
O(4)-H(44B)...O(3)#3	0.73(3)	2.14(3)	2.861(3)	171(4)

Symmetry transformations used to generate equivalent atoms:
#1 x+1,y,z
#2 -x+1,-y+1,-z+2
#3 -x+2,-y+1,-z+2
#4 x-1,y,z
#5 x+1,y+1,z