C(2)-O(1)-C(5)	106.23(13)
O(1)-C(5)-C(6)	105.41(14)
O(1)-C(5)-C(4)	107.33(11)
C(6)-C(5)-C(4)	115.02(13)
C(7)-C(4)-C(3)	112.11(16)
C(7)-C(4)-C(5)	113.26(14)
C(3)-C(4)-C(5)	103.29(13)
O(6)-C(3)-C(2)	106.67(13)
O(6)-C(3)-C(4)	110.69(12)
C(2)-C(3)-C(4)	103.88(12)
O(1)-C(2)-C(8)	110.22(16)
O(1)-C(2)-C(3)	103.97(12)
C(8)-C(2)-C(3)	117.16(12)
O(2)-C(6)-C(5)	105.99(15)
C(9)-O(2)-C(6)	115.27(14)
O(3)-C(9)-O(2)	123.95(16)
O(3)-C(9)-C(10)	123.55(16)
O(2)-C(9)-C(10)	112.48(15)
C(11)-C(10)-C(15)	119.69(16)
C(11)-C(10)-C(9)	117.97(16)
C(15)-C(10)-C(9)	122.33(15)
C(12)-C(11)-C(10)	120.74(17)
C(11)-C(12)-C(13)	118.31(15)
C(12)-C(13)-C(14)	122.91(16)
C(12)-C(13)-N(1)	118.60(15)
C(14)-C(13)-N(1)	118.48(17)
C(13)-C(14)-C(15)	118.30(17)
C(14)-C(15)-C(10)	120.04(15)
O(4)-N(1)-O(5)	123.62(17)
O(4)-N(1)-C(13)	118.16(17)
O(5)-N(1)-C(13)	118.22(15)
C(16)-O(6)-C(3)	116.50(12)
O(7)-C(16)-O(6)	123.86(13)
O(7)-C(16)-C(17)	124.46(13)
O(6)-C(16)-C(17)	111.68(12)

C(22)-C(17)-C(18)	119.99(13)
C(22)-C(17)-C(16)	122.04(12)
C(18)-C(17)-C(16)	117.98(13)
C(19)-C(18)-C(17)	120.34(14)
C(20)-C(19)-C(18)	118.10(12)
C(19)-C(20)-C(21)	123.19(13)
C(19)-C(20)-N(2)	118.65(12)
C(21)-C(20)-N(2)	118.15(13)
C(22)-C(21)-C(20)	117.93(13)
C(21)-C(22)-C(17)	120.43(12)
O(8)-N(2)-O(9)	124.00(12)
O(8)-N(2)-C(20)	117.58(13)
O(9)-N(2)-C(20)	118.42(12)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $C_{21}H_{20}N_2O_9$. 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}
O(1)	68(1)	54(1)	34(1)	-4(1)	1(1)	-20(1)
C(5)	55(1)	44(1)	36(1)	-7(1)	8(1)	-8(1)
C(4)	50(1)	49(1)	42(1)	-8(1)	8(1)	-3(1)
C(3)	44(1)	46(1)	38(1)	-13(1)	4(1)	8(1)
C(2)	69(1)	42(1)	35(1)	-9(1)	5(1)	-1(1)
C(6)	52(1)	55(1)	48(1)	0(1)	5(1)	-10(1)
C(7)	53(1)	119(2)	62(1)	-8(1)	14(1)	-9(1)
C(8)	98(2)	54(1)	39(1)	-5(1)	4(1)	-22(1)
O(2)	48(1)	49(1)	47(1)	4(1)	2(1)	-3(1)
O(3)	56(1)	61(1)	66(1)	1(1)	12(1)	10(1)
C(9)	53(1)	42(1)	42(1)	-9(1)	0(1)	3(1)
C(10)	53(1)	36(1)	37(1)	-11(1)	-1(1)	-2(1)
C(11)	52(1)	44(1)	45(1)	-8(1)	-4(1)	6(1)
C(12)	62(1)	42(1)	40(1)	-4(1)	-5(1)	5(1)
C(13)	60(1)	39(1)	36(1)	-6(1)	1(1)	-6(1)
C(14)	47(1)	46(1)	48(1)	-7(1)	-4(1)	-6(1)
C(15)	52(1)	39(1)	43(1)	-3(1)	-6(1)	-2(1)
N(1)	71(1)	54(1)	42(1)	-5(1)	5(1)	-7(1)
O(4)	93(1)	92(1)	61(1)	31(1)	14(1)	13(1)
O(5)	65(1)	90(1)	61(1)	7(1)	1(1)	-24(1)
O(6)	37(1)	46(1)	37(1)	-15(1)	1(1)	1(1)
O(7)	35(1)	62(1)	59(1)	-23(1)	3(1)	-3(1)
C(16)	37(1)	36(1)	31(1)	-3(1)	0(1)	-4(1)
C(17)	33(1)	30(1)	28(1)	0(1)	1(1)	-4(1)
C(18)	30(1)	37(1)	35(1)	-3(1)	-2(1)	-6(1)
C(19)	38(1)	32(1)	28(1)	-5(1)	-2(1)	-7(1)
C(20)	36(1)	28(1)	28(1)	-1(1)	0(1)	0(1)
C(21)	31(1)	35(1)	33(1)	-1(1)	-4(1)	0(1)
C(22)	37(1)	35(1)	28(1)	-6(1)	-5(1)	-4(1)
N(2)	41(1)	40(1)	34(1)	-5(1)	-1(1)	5(1)
O(8)	54(1)	55(1)	61(1)	-31(1)	-1(1)	3(1)

O(9)	38(1)	61(1)	57(1)	-15(1)	7(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $C_{21}H_{20}N_2O_9$.

	x	y	z	U(eq)
H(5)	5512	6964	4016	54
H(4)	4917	5474	3196	56
H(3)	4045	6958	2470	51
H(2)	5524	8111	3048	58
H(6A)	7999	5390	3686	62
H(6B)	8422	6115	4225	62
H(7A)	2374	7058	3436	117
H(7B)	2468	6115	3847	117
H(7C)	1798	6017	3178	117
H(8A)	8291	8646	2621	95
H(8B)	7009	8242	2096	95
H(8C)	8714	7599	2344	95
H(11)	7710	3191	5474	56
H(12)	5722	2343	6116	58
H(14)	1170	3731	5415	56
H(15)	3172	4586	4772	54
H(18)	4927	4365	1035	41
H(19)	7005	3423	462	39
H(21)	11469	4535	1377	40
H(22)	9369	5433	1968	40

Table 6. Torsion angles [°] for $C_{21}H_{20}N_2O_9$.

C(2)-O(1)-C(5)-C(6)	151.38(12)
C(2)-O(1)-C(5)-C(4)	28.31(15)
O(1)-C(5)-C(4)-C(7)	-126.91(17)
C(6)-C(5)-C(4)-C(7)	116.16(18)
O(1)-C(5)-C(4)-C(3)	-5.41(16)
C(6)-C(5)-C(4)-C(3)	-122.34(14)
C(7)-C(4)-C(3)-O(6)	-141.41(15)
C(5)-C(4)-C(3)-O(6)	96.32(14)
C(7)-C(4)-C(3)-C(2)	104.43(17)
C(5)-C(4)-C(3)-C(2)	-17.83(16)
C(5)-O(1)-C(2)-C(8)	-166.16(11)
C(5)-O(1)-C(2)-C(3)	-39.79(14)
O(6)-C(3)-C(2)-O(1)	-81.65(13)
C(4)-C(3)-C(2)-O(1)	35.34(16)
O(6)-C(3)-C(2)-C(8)	40.22(19)
C(4)-C(3)-C(2)-C(8)	157.21(16)
O(1)-C(5)-C(6)-O(2)	173.85(11)
C(4)-C(5)-C(6)-O(2)	-68.14(16)
C(5)-C(6)-O(2)-C(9)	-178.32(12)
C(6)-O(2)-C(9)-O(3)	-0.1(2)
C(6)-O(2)-C(9)-C(10)	178.52(12)
O(3)-C(9)-C(10)-C(11)	6.8(2)
O(2)-C(9)-C(10)-C(11)	-171.77(12)
O(3)-C(9)-C(10)-C(15)	-174.01(16)
O(2)-C(9)-C(10)-C(15)	7.4(2)
C(15)-C(10)-C(11)-C(12)	-0.9(2)
C(9)-C(10)-C(11)-C(12)	178.31(13)
C(10)-C(11)-C(12)-C(13)	0.0(2)
C(11)-C(12)-C(13)-C(14)	0.9(2)
C(11)-C(12)-C(13)-N(1)	179.74(13)
C(12)-C(13)-C(14)-C(15)	-1.1(2)
N(1)-C(13)-C(14)-C(15)	-179.85(12)
C(13)-C(14)-C(15)-C(10)	0.2(2)
C(11)-C(10)-C(15)-C(14)	0.7(2)

C(9)-C(10)-C(15)-C(14)	-178.40(12)
C(12)-C(13)-N(1)-O(4)	9.8(2)
C(14)-C(13)-N(1)-O(4)	-171.33(14)
C(12)-C(13)-N(1)-O(5)	-169.92(16)
C(14)-C(13)-N(1)-O(5)	8.9(2)
C(2)-C(3)-O(6)-C(16)	-159.59(12)
C(4)-C(3)-O(6)-C(16)	88.03(15)
C(3)-O(6)-C(16)-O(7)	-0.4(2)
C(3)-O(6)-C(16)-C(17)	179.29(11)
O(7)-C(16)-C(17)-C(22)	-174.05(14)
O(6)-C(16)-C(17)-C(22)	6.27(18)
O(7)-C(16)-C(17)-C(18)	5.9(2)
O(6)-C(16)-C(17)-C(18)	-173.73(12)
C(22)-C(17)-C(18)-C(19)	1.5(2)
C(16)-C(17)-C(18)-C(19)	-178.48(12)
C(17)-C(18)-C(19)-C(20)	-1.61(19)
C(18)-C(19)-C(20)-C(21)	0.6(2)
C(18)-C(19)-C(20)-N(2)	-178.26(11)
C(19)-C(20)-C(21)-C(22)	0.5(2)
N(2)-C(20)-C(21)-C(22)	179.38(12)
C(20)-C(21)-C(22)-C(17)	-0.62(19)
C(18)-C(17)-C(22)-C(21)	-0.4(2)
C(16)-C(17)-C(22)-C(21)	179.63(12)
C(19)-C(20)-N(2)-O(8)	-20.80(18)
C(21)-C(20)-N(2)-O(8)	160.29(13)
C(19)-C(20)-N(2)-O(9)	158.89(13)
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Symmetry transformations used to generate equivalent atoms:

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N2 C20 C21 C22 179.38(12) . . . . ?
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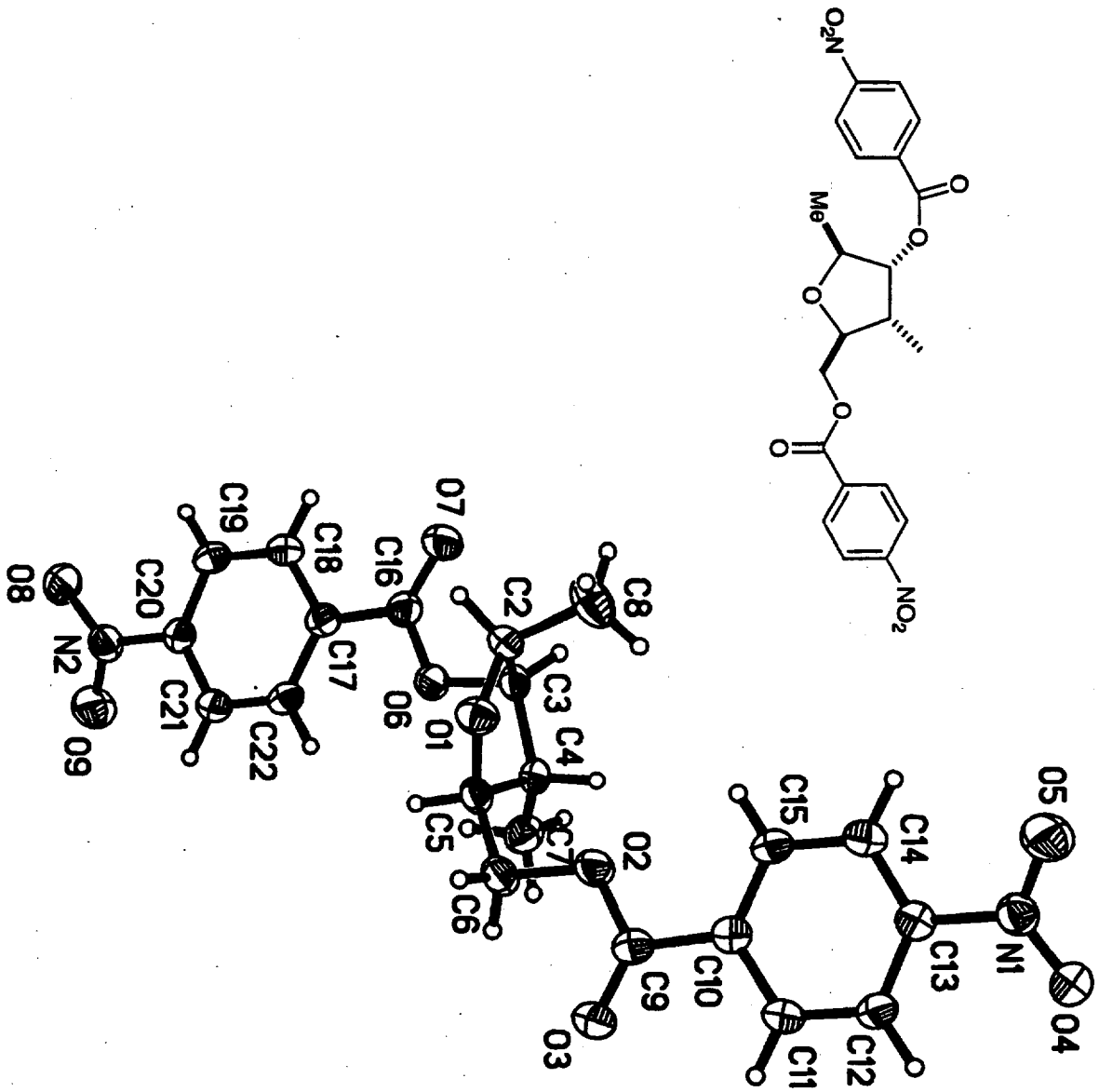

X-Ray Structure Determination For 50

A colorless fragment of a needle (0.40 x 0.12 x 0.10 mm^3) was used for the single crystal x-ray diffraction study of sa111m ($C_{21}H_{20}N_2O_9$, NE-III-39-3). The crystal was mounted on to a glass fiber with epoxy resin. X-ray intensity data were collected at 213(2)K on a Bruker SMART 1000 (**ref. 1**) platform-CCD x-ray diffractometer system (Mo-radiation, λ = 0.71073 Å, 50KV/45mA power). The CCD detector was placed at a distance of 4.8450 cm from the crystal.

A total of 1321 frames were collected for a hemisphere of reflections (with scan width of 0.3° in ω and ϕ angles of 0°, 90°, 180°, and 0° for every 606, 435, 230 and 50 frames, respectively, 60sec/frame exposure time). The frames were integrated using the Bruker SAINTPLUS software package (**ref. 2**) and using a narrow-frame integration algorithm. Based on a monoclinic crystal system, the integrated frames yielded a total of 6183 reflections at a maximum 2θ angle of 52.74° (0.80 Å resolution), of which 3680 were independent reflections (R_{int} = 0.0348, R_{sig} = 0.0456, redundancy = 1.7, completeness = 100%) and 3095 (84.1%) reflections were greater than 2σ(I). The unit cell parameters were, **a** = 6.9620(10) Å, **b** = 20.910(3) Å, **c** = 7.2754(11) Å, α = 90°, β = 106.319(3)°, γ = 90°, V = 1016.4(3) $Å^3$, Z = 2, calculated density D_c = 1.452 g/cm^3. No absorption corrections were applied (absorption coefficient μ = 0.115 mm^{-1}) to the raw intensity data.

The Bruker SHELXTL (Version 5.1) software package (**ref. 3**) was used for phase determination and structure refinement. The distribution of intensities (E^2-1 = 0.832) and systematic absent reflections indicated two possible space groups; P2(1), P2(1)/m. The space group P2(1) was later determined to be correct. Direct methods of phase determination followed by two Fourier cycles of refinement led to an electron density map from which all of the non-hydrogen atoms were identified in the asymmetry unit of the unit cell. There was one molecule of $C_{21}H_{20}N_2O_9$ present in the asymmetry unit of the unit cell. The relative configurations at C2, C3, C4, and C5 were S, R, R, and S, respectively.

Atomic coordinates, isotropic and anisotropic displacement parameters of all the non-hydrogen atoms were refined by means of a full matrix least-squares procedure on F^2. All the H-atoms were included in the refinement in calculated positions riding on the atoms to which they were attached. The refinement converged at R1 = 0.0356, wR2 = 0.0793, with intensity, I>2σ(I). The largest peak/hole in the final difference map were 0.164/-0.180 $Å^3$.

REFERENCES

1. SMART Software Reference Manual, Version 5.054, Bruker Analytical X-Ray System, Inc., Madison, WI 1997-1998.

2. SAINTPLUS Software Reference Manual, Version 5.02, Bruker Analytical X-Ray System, Inc., Madison, WI 1997-1998.

3. SHELXTL Software Reference Manual, Version 5.1, Bruker Analytical X-Ray System, Inc., Madison, WI 1997-1998.

Table 1. Crystal data and structure refinement for $C_{21}H_{20}N_2O_9$.

Identification code	sa111m	
Empirical formula	C21 H20 N2 O9	
Formula weight	444.39	
Temperature	213(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 6.9620(10) Å	α= 90°.
	b = 20.910(3) Å	β= 106.319(3)°.
	c = 7.2754(11) Å	γ = 90°.
Volume	1016.4(3) $Å^3$	
Z	2	
Density (calculated)	1.452 Mg/m^3	
Absorption coefficient	0.115 mm^{-1}	
F(000)	464	
Crystal size	.10 x .12 x .40 mm^3	
Theta range for data collection	1.95 to 26.37°.	
Index ranges	-8<=h<=8, -21<=k<=26, -8<=l<=9	
Reflections collected	6183	
Independent reflections	3680 [R(int) = 0.0348]	
Completeness to theta = 26.37°	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3680 / 1 / 291	
Goodness-of-fit on F^2	1.013	
Final R indices [I>2sigma(I)]	R1 = 0.0356, wR2 = 0.0793	
R indices (all data)	R1 = 0.0457, wR2 = 0.0840	
Absolute structure parameter	-1.0(8)	
Largest diff. peak and hole	0.164 and -0.180 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for $C_{21}H_{20}N_2O_9$. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	5317(2)	9690(1)	424(2)	35(1)
C(2)	6631(3)	10030(1)	2012(3)	33(1)
C(3)	8599(3)	10100(1)	1504(3)	29(1)
C(4)	8591(3)	9530(1)	220(3)	28(1)
C(5)	6394(3)	9506(1)	-902(3)	30(1)
C(6)	5601(3)	8882(1)	-1827(3)	36(1)
C(7)	10081(3)	9553(1)	-961(3)	38(1)
C(8)	6836(4)	9663(1)	3859(3)	53(1)
O(2)	6048(2)	8379(1)	-386(2)	36(1)
O(3)	5521(3)	7662(1)	-2757(2)	54(1)
C(9)	5920(3)	7786(1)	-1078(3)	32(1)
C(10)	6273(3)	7285(1)	461(3)	29(1)
C(11)	6354(3)	6650(1)	-102(3)	32(1)
C(12)	6623(3)	6164(1)	1226(3)	31(1)
C(13)	6769(3)	6326(1)	3104(3)	29(1)
C(14)	6676(3)	6947(1)	3707(3)	33(1)
C(15)	6456(3)	7433(1)	2366(3)	31(1)
N(1)	6984(3)	5802(1)	4502(2)	36(1)
O(4)	7598(3)	5288(1)	4133(2)	51(1)
O(5)	6519(3)	5906(1)	5974(2)	57(1)
O(6)	8526(2)	10664(1)	292(2)	32(1)
O(7)	9016(2)	11321(1)	2825(2)	44(1)
C(16)	8749(3)	11236(1)	1127(3)	31(1)
C(17)	8629(3)	11759(1)	-293(3)	28(1)
C(18)	8701(3)	12390(1)	345(3)	31(1)
C(19)	8591(3)	12893(1)	-920(3)	33(1)
C(20)	8475(3)	12749(1)	-2798(3)	29(1)
C(21)	8420(3)	12129(1)	-3476(3)	31(1)
C(22)	8473(3)	11636(1)	-2215(3)	31(1)
N(2)	8449(3)	13282(1)	-4132(3)	37(1)
O(8)	7884(3)	13803(1)	-3750(2)	54(1)

O(9)	9043(3)	13178(1)	-5535(2)	55(1)

Table 3. Bond lengths [Å] and angles [°] for $C_{21}H_{20}N_2O_9$.

O(1)-C(5)	1.430(2)
O(1)-C(2)	1.444(2)
C(2)-C(8)	1.520(3)
C(2)-C(3)	1.523(3)
C(3)-O(6)	1.465(2)
C(3)-C(4)	1.513(3)
C(4)-C(5)	1.518(3)
C(4)-C(7)	1.523(3)
C(5)-C(6)	1.500(3)
C(6)-O(2)	1.456(2)
O(2)-C(9)	1.331(2)
O(3)-C(9)	1.202(3)
C(9)-C(10)	1.503(3)
C(10)-C(15)	1.391(3)
C(10)-C(11)	1.395(3)
C(11)-C(12)	1.378(3)
C(12)-C(13)	1.384(3)
C(13)-C(14)	1.376(3)
C(13)-N(1)	1.474(3)
C(14)-C(15)	1.388(3)
N(1)-O(4)	1.215(2)
N(1)-O(5)	1.222(2)
O(6)-C(16)	1.331(2)
O(7)-C(16)	1.210(2)
C(16)-C(17)	1.491(3)
C(17)-C(18)	1.394(3)
C(17)-C(22)	1.396(3)
C(18)-C(19)	1.386(3)
C(19)-C(20)	1.380(3)
C(20)-C(21)	1.384(3)
C(20)-N(2)	1.476(3)
C(21)-C(22)	1.373(3)
N(2)-O(8)	1.216(2)
N(2)-O(9)	1.224(2)

C(5)-O(1)-C(2)	109.74(14)
O(1)-C(2)-C(8)	109.84(19)
O(1)-C(2)-C(3)	105.31(15)
C(8)-C(2)-C(3)	113.30(17)
O(6)-C(3)-C(4)	105.57(14)
O(6)-C(3)-C(2)	109.99(16)
C(4)-C(3)-C(2)	103.09(16)
C(3)-C(4)-C(5)	100.75(15)
C(3)-C(4)-C(7)	116.07(17)
C(5)-C(4)-C(7)	116.15(17)
O(1)-C(5)-C(6)	109.82(17)
O(1)-C(5)-C(4)	105.55(15)
C(6)-C(5)-C(4)	117.24(17)
O(2)-C(6)-C(5)	109.19(16)
C(9)-O(2)-C(6)	114.98(16)
O(3)-C(9)-O(2)	123.9(2)
O(3)-C(9)-C(10)	123.3(2)
O(2)-C(9)-C(10)	112.81(17)
C(15)-C(10)-C(11)	120.3(2)
C(15)-C(10)-C(9)	122.5(2)
C(11)-C(10)-C(9)	117.16(18)
C(12)-C(11)-C(10)	120.29(18)
C(11)-C(12)-C(13)	118.0(2)
C(14)-C(13)-C(12)	123.2(2)
C(14)-C(13)-N(1)	119.08(18)
C(12)-C(13)-N(1)	117.67(19)
C(13)-C(14)-C(15)	118.21(19)
C(14)-C(15)-C(10)	119.9(2)
O(4)-N(1)-O(5)	123.19(19)
O(4)-N(1)-C(13)	118.69(18)
O(5)-N(1)-C(13)	118.11(18)
C(16)-O(6)-C(3)	117.96(15)
O(7)-C(16)-O(6)	124.2(2)
O(7)-C(16)-C(17)	124.31(19)
O(6)-C(16)-C(17)	111.49(17)

C(18)-C(17)-C(22)	119.61(19)
C(18)-C(17)-C(16)	118.24(18)
C(22)-C(17)-C(16)	122.15(19)
C(19)-C(18)-C(17)	120.47(19)
C(20)-C(19)-C(18)	117.9(2)
C(19)-C(20)-C(21)	123.1(2)
C(19)-C(20)-N(2)	118.20(19)
C(21)-C(20)-N(2)	118.66(18)
C(22)-C(21)-C(20)	118.15(19)
C(21)-C(22)-C(17)	120.7(2)
O(8)-N(2)-O(9)	123.84(19)
O(8)-N(2)-C(20)	118.13(19)
O(9)-N(2)-C(20)	118.01(19)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for $C_{21}H_{20}N_2O_9$. 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}
O(1)	32(1)	41(1)	33(1)	-5(1)	9(1)	-3(1)
C(2)	42(1)	33(1)	23(1)	1(1)	9(1)	3(1)
C(3)	36(1)	24(1)	25(1)	3(1)	5(1)	1(1)
C(4)	33(1)	23(1)	28(1)	4(1)	8(1)	1(1)
C(5)	33(1)	29(1)	25(1)	5(1)	6(1)	0(1)
C(6)	45(1)	34(1)	26(1)	3(1)	5(1)	-4(1)
C(7)	42(1)	34(1)	42(1)	1(1)	17(1)	3(1)
C(8)	61(2)	67(2)	34(1)	13(1)	18(1)	0(1)
O(2)	50(1)	29(1)	28(1)	-2(1)	9(1)	-6(1)
O(3)	91(1)	39(1)	28(1)	-5(1)	11(1)	7(1)
C(9)	32(1)	30(1)	33(1)	-6(1)	9(1)	-3(1)
C(10)	25(1)	31(1)	31(1)	-2(1)	8(1)	-4(1)
C(11)	35(1)	34(1)	26(1)	-7(1)	8(1)	-2(1)
C(12)	31(1)	28(1)	33(1)	-6(1)	8(1)	0(1)
C(13)	27(1)	32(1)	30(1)	0(1)	9(1)	-3(1)
C(14)	35(1)	35(1)	30(1)	-6(1)	12(1)	-4(1)
C(15)	32(1)	28(1)	34(1)	-8(1)	9(1)	-2(1)
N(1)	41(1)	35(1)	33(1)	-1(1)	8(1)	-5(1)
O(4)	76(1)	35(1)	39(1)	1(1)	12(1)	12(1)
O(5)	88(1)	50(1)	47(1)	2(1)	39(1)	-3(1)
O(6)	43(1)	23(1)	28(1)	1(1)	9(1)	-2(1)
O(7)	66(1)	34(1)	30(1)	-5(1)	10(1)	1(1)
C(16)	32(1)	29(1)	31(1)	-3(1)	6(1)	0(1)
C(17)	26(1)	29(1)	30(1)	-3(1)	7(1)	-1(1)
C(18)	33(1)	30(1)	29(1)	-5(1)	10(1)	-2(1)
C(19)	36(1)	26(1)	37(1)	-7(1)	12(1)	-3(1)
C(20)	26(1)	29(1)	32(1)	1(1)	6(1)	-1(1)
C(21)	32(1)	34(1)	29(1)	-2(1)	10(1)	-1(1)
C(22)	31(1)	26(1)	35(1)	-5(1)	9(1)	-1(1)
N(2)	43(1)	31(1)	34(1)	2(1)	3(1)	-4(1)
O(8)	80(1)	32(1)	45(1)	1(1)	8(1)	7(1)

O(9)	78(1)	50(1)	43(1)	9(1)	28(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for $C_{21}H_{20}N_2O_9$.

	x	y	z	U(eq)
H(2A)	6070	10459	2114	39
H(3A)	9774	10103	2650	35
H(4A)	8887	9144	1040	34
H(5A)	6159	9836	-1917	35
H(6A)	4151	8913	-2400	43
H(6B)	6224	8782	-2845	43
H(7A)	11433	9542	-113	57
H(7B)	9887	9943	-1710	57
H(7C)	9873	9186	-1813	57
H(8A)	5519	9586	4021	79
H(8B)	7628	9910	4933	79
H(8C)	7492	9257	3803	79
H(11A)	6224	6553	-1393	38
H(12A)	6705	5736	866	37
H(14A)	6759	7038	4992	39
H(15A)	6431	7862	2745	38
H(18A)	8825	12474	1642	37
H(19A)	8595	13320	-510	39
H(21A)	8348	12047	-4765	38
H(22A)	8403	11211	-2651	37

Table 6. Torsion angles [°] for $C_{21}H_{20}N_2O_9$.

C(5)-O(1)-C(2)-C(8)	-117.55(18)
C(5)-O(1)-C(2)-C(3)	4.8(2)
O(1)-C(2)-C(3)-O(6)	84.79(19)
C(8)-C(2)-C(3)-O(6)	-155.14(18)
O(1)-C(2)-C(3)-C(4)	-27.41(19)
C(8)-C(2)-C(3)-C(4)	92.7(2)
O(6)-C(3)-C(4)-C(5)	-77.40(17)
C(2)-C(3)-C(4)-C(5)	38.01(17)
O(6)-C(3)-C(4)-C(7)	48.9(2)
C(2)-C(3)-C(4)-C(7)	164.33(17)
C(2)-O(1)-C(5)-C(6)	147.12(16)
C(2)-O(1)-C(5)-C(4)	19.9(2)
C(3)-C(4)-C(5)-O(1)	-35.99(18)
C(7)-C(4)-C(5)-O(1)	-162.25(17)
C(3)-C(4)-C(5)-C(6)	-158.62(17)
C(7)-C(4)-C(5)-C(6)	75.1(2)
O(1)-C(5)-C(6)-O(2)	-66.3(2)
C(4)-C(5)-C(6)-O(2)	54.1(2)
C(5)-C(6)-O(2)-C(9)	-162.72(17)
C(6)-O(2)-C(9)-O(3)	1.9(3)
C(6)-O(2)-C(9)-C(10)	-176.53(16)
O(3)-C(9)-C(10)-C(15)	-170.8(2)
O(2)-C(9)-C(10)-C(15)	7.6(3)
O(3)-C(9)-C(10)-C(11)	7.3(3)
O(2)-C(9)-C(10)-C(11)	-174.26(17)
C(15)-C(10)-C(11)-C(12)	-0.2(3)
C(9)-C(10)-C(11)-C(12)	-178.40(18)
C(10)-C(11)-C(12)-C(13)	1.2(3)
C(11)-C(12)-C(13)-C(14)	-0.6(3)
C(11)-C(12)-C(13)-N(1)	177.84(17)
C(12)-C(13)-C(14)-C(15)	-1.0(3)
N(1)-C(13)-C(14)-C(15)	-179.44(17)
C(13)-C(14)-C(15)-C(10)	2.0(3)
C(11)-C(10)-C(15)-C(14)	-1.4(3)

C(9)-C(10)-C(15)-C(14)	176.64(18)
C(14)-C(13)-N(1)-O(4)	-160.40(19)
C(12)-C(13)-N(1)-O(4)	21.1(3)
C(14)-C(13)-N(1)-O(5)	20.1(3)
C(12)-C(13)-N(1)-O(5)	-158.38(19)
C(4)-C(3)-O(6)-C(16)	-172.46(16)
C(2)-C(3)-O(6)-C(16)	77.0(2)
C(3)-O(6)-C(16)-O(7)	0.3(3)
C(3)-O(6)-C(16)-C(17)	-179.71(15)
O(7)-C(16)-C(17)-C(18)	-4.2(3)
O(6)-C(16)-C(17)-C(18)	175.78(17)
O(7)-C(16)-C(17)-C(22)	174.9(2)
O(6)-C(16)-C(17)-C(22)	-5.0(3)
C(22)-C(17)-C(18)-C(19)	0.9(3)
C(16)-C(17)-C(18)-C(19)	-179.88(18)
C(17)-C(18)-C(19)-C(20)	-2.1(3)
C(18)-C(19)-C(20)-C(21)	1.5(3)
C(18)-C(19)-C(20)-N(2)	-177.32(17)
C(19)-C(20)-C(21)-C(22)	0.3(3)
N(2)-C(20)-C(21)-C(22)	179.12(17)
C(20)-C(21)-C(22)-C(17)	-1.6(3)
C(18)-C(17)-C(22)-C(21)	1.0(3)
C(16)-C(17)-C(22)-C(21)	-178.21(18)
C(19)-C(20)-N(2)-O(8)	-23.5(3)
C(21)-C(20)-N(2)-O(8)	157.67(19)
C(19)-C(20)-N(2)-O(9)	154.76(19)
C(21)-C(20)-N(2)-O(9)	-24.1(3)

Symmetry transformations used to generate equivalent atoms:

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C19 C 0.8591(3) 1.28930(10) -0.0920(3) 0.0327(5) Uani 1 1 d . . .
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C20 C 0.8475(3) 1.27486(10) -0.2798(3) 0.0292(5) Uani 1 1 d . . .
C21 C 0.8420(3) 1.21285(10) -0.3476(3) 0.0313(5) Uani 1 1 d . . .
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C8 0.0609(16) 0.0667(19) 0.0337(12) 0.0133(12) 0.0180(12) -0.0004(13)
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O3 0.0907(13) 0.0393(11) 0.0277(9) -0.0047(7) 0.0110(9) 0.0066(9)
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C11 0.0346(11) 0.0339(13) 0.0262(11) -0.0067(9) 0.0084(9) -0.0019(9)
C12 0.0309(10) 0.0277(12) 0.0333(11) -0.0057(9) 0.0080(9) 0.0002(8)
C13 0.0266(10) 0.0315(12) 0.0301(11) -0.0002(9) 0.0094(9) -0.0026(8)
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C15 0.0317(11) 0.0281(12) 0.0339(12) -0.0078(9) 0.0090(9) -0.0017(9)
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 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.
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O6 C3 C4 105.57(14) . . ?
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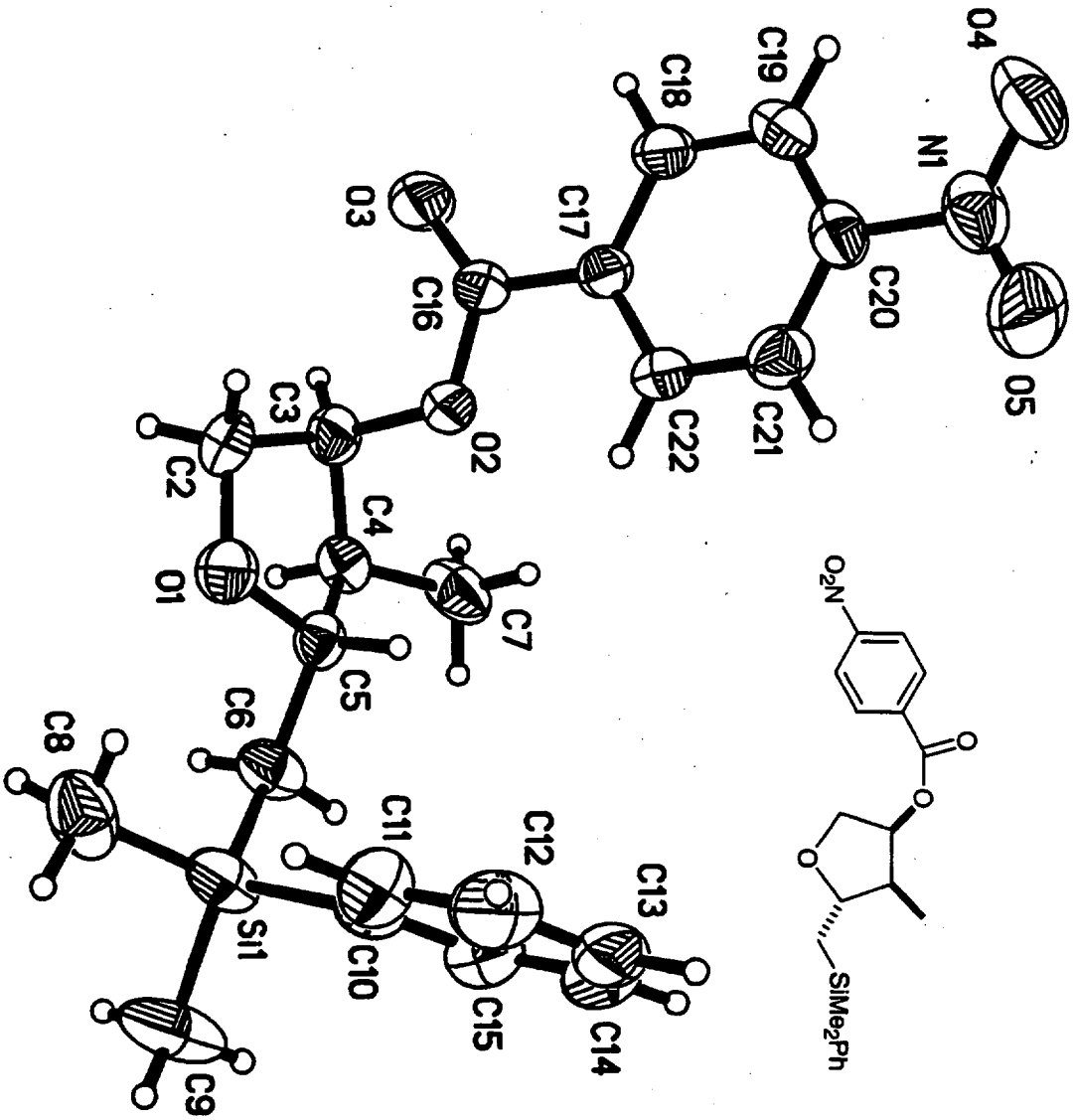

X-Ray Structure Determination For 52

A colorless fragment of a needle (0.57 x 0.22 x 0.09 mm^3) was used for the single crystal x-ray diffraction study of sa14twin ($C_{21}H_{25}NO_5Si$, NE-III-42-1-1). The crystal was mounted on to a glass fiber with epoxy resin. X-ray intensity data were collected at 223(2) K on a Bruker SMART 1000 (**ref. 1**) platform-CCD x-ray diffractometer system (Mo-radiation, λ = 0.71073 Å, 50KV/45mA power). The CCD detector was placed at a distance of 4.8450 cm from the crystal.

The crystal was a twin. The Bruker GEMINI (**ref. 2**) and RLATT (**ref. 3**) software programs were used to resolve the rotational twin law [Twin law: -180° rotation about the axis (–1, 0, 0) in reciprocal space]. Two different twinned-crystal orientation-matrices were obtained for the two twin components. These matrices were later refined using the Bruker SMART (**ref. 1**) program and were used in the frame intensity integration procedure.

A total of 3080 frames were collected for a sphere of reflections (with scan width of 0.3° in ω and ϕ angles of 0°, 120°, 240° and 0° for every 606, 606, 606, and 50 frames, respectively; 606 frames with scan width of 0.3° in ϕ and 0° starting ϕ angle; 30sec/frame exposure time). The frames were integrated using the Bruker SAINTPLUS software package (**ref. 4**) and using a narrow-frame integration algorithm. The two refined twinned-crystal orientation-matrices were used to generate two HKL data sets that were then combined to create one HKLF5 type intensity data set that was used in the final structure refinement. Based on a triclinic crystal system, the integrated frames yielded a total of 8652 reflections. There were 521 exact-overlaps, 5736 partial overlaps, 2395 non-overlaps reflections where only exact-overlaps and non-overlaps reflections were used in the structure refinement. There were 2751 independent reflections [maximum 2θ angle was 46.50° (0.90 Å resolution), R_{int} = 0.0525, R_{sig} = 0.0297, redundancy = 1.06, completeness = 31.8%] and 1874 (68.1%) reflections were greater than 2σ(I). The unit cell parameters were, **a** = 7.348(4) Å, **b** = 12.026(6) Å, **c** = 12.432(6) Å, α = 107.506(8)°, β = 90.370(9)°, γ = 95.405(9)°, V = 1042.4(9) $Å^3$, Z = 2, calculated density D_c = 1.273 g/cm^3. Absorption corrections were applied (absorption coefficient μ = 0.144 mm^{-1}) to the two HKL data sets (before the HKLF5 intensity data set was created) using the SADABS program in the SAINTPLUS software (**Ref 4**). The maximum/minimum transmission factors were 0.9872/0.9225.

The Bruker SHELXTL (Version 5.1) software package (**ref. 5**) was used for phase determination and structure refinement. The distribution of intensities (E^2-1 = 0.984) indicated two possible space groups; P-1 and P1. The space group P-1 was later determined to be correct. Direct methods of phase determination followed by two Fourier cycles of refinement led to an electron density map from which most of the non-hydrogen atoms were identified in the asymmetry unit of the unit cell. With subsequent isotropic refinement all of the non-hydrogen atoms were identified. There was one molecule of $C_{21}H_{25}NO_5Si$ present in the asymmetry unit of the unit cell. The relative configurations at

C3, C4, and C5 were S, S, and R, respectively (R, R, and S for it's racemic counterpart). The ratio of the major/minor components of the twinned-crystal was 71%/29%.

Atomic coordinates, isotropic and anisotropic displacement parameters of all the non-hydrogen atoms were refined by means of a full matrix least-squares procedure on F^2. All the H-atoms were included in the refinement in calculated positions riding on the atoms to which they were attached. The refinement converged at R1 = 0.0756, wR2 = 0.1947, with intensity, $I>2\sigma(I)$. The largest peak/hole in the final difference map were 0.195/-0.261 e/Å^3.