REFERENCE NUMBER: 02110b

CRYSTAL STRUCTURE REPORT

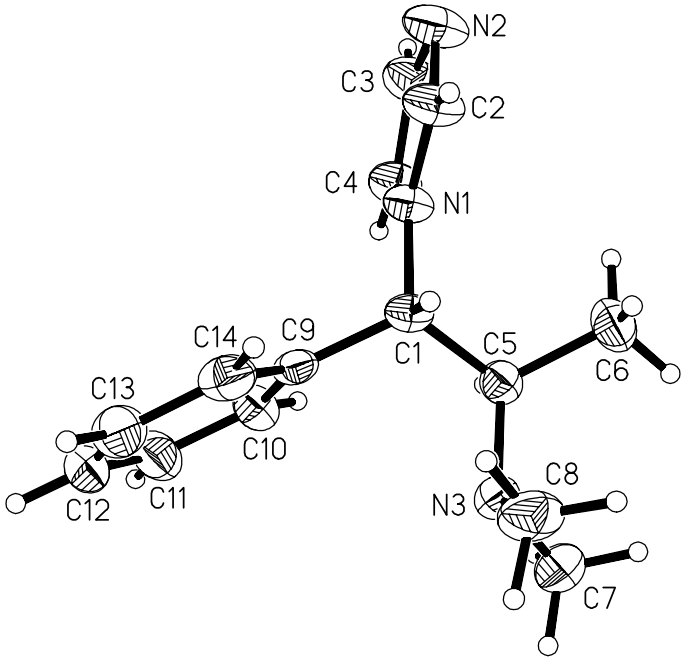
$C_{14}\ H_{19}\ N_3$

3642-30-1

Report prepared for:

Dr. Y. Che - OSI Pharmaceuticals, Inc.

23 April 2002



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Data collection

A crystal (approximate dimensions 0.32 x 0.29 x 0.12 mm^3) was placed onto the tip of a 0.1 mm diameter glass capillary and mounted on a Bruker SMART system for a data collection at 173(2) K. A preliminary set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames were oriented such that orthogonal wedges of reciprocal space were surveyed. This produced initial orientation matrices determined from 118 reflections. The data collection was carried out using MoKα radiation (graphite monochromator) with a frame time of 30 seconds and a detector distance of 4.9 cm. A randomly oriented region of reciprocal space was surveyed to the extent of 2.0 hemispheres and to a resolution of 0.84 Å. Three major sections of frames were collected with 0.30º steps in ω at 3 different ϕsettings and a detector position of -28° in 2θ. The intensity data were corrected for absorption and decay (SADABS).[1] Final cell constants were calculated from 3496 strong reflections from the actual data collection after integration (SAINT 6.01, 1999).[2] Please refer to Table 1 for additional crystal and refinement information.

Structure solution and refinement

The structure was solved using SHELXS-86[3] and refined using SHELXL-97.[3] The space group $P2_12_12_1$ was determined based on systematic absences and intensity statistics. A direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Full-matrix least squares / difference Fourier cycles were performed which located the remaining non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters unless stated otherwise. All hydrogen atoms were placed in ideal positions and refined as riding atoms with relative isotropic displacement parameters. The final full matrix least squares refinement converged to R1 = 0.0326 and wR2 = 0.0801 (F^2, all data).

Structure description

The structure was found as expected. C5 is R as known from the synthesis. C1 is S. The absolute configuration cannot be determined, but based on chemical knowledge it is correct as presented. The Flack parameter is indeterminate.

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 160 Kolthoff Hall, Department of Chemistry, University of Minnesota. All calculations were performed using SGI INDY R4400-SC or Pentium computers using the current SHELXTL suite of programs. All publications arising from this report MUST either 1)include Victor G. Young, Jr. as a coauthor or 2)acknowledge Victor G. Young, Jr. and the X-Ray Crystallographic Laboratory. Drafts must be supplied to the coauthor prior to journal submission. Additional drawings will be supplied upon request.

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[1] An empirical correction for absorption anisotropy, R. Blessing, Acta Cryst. A51, 33 - 38 (1995).

[2] SAINT V6.1, Bruker Analytical X-Ray Systems, Madison, WI.

[3] SHELXTL-Plus V5.10, Bruker Analytical X-Ray Systems, Madison, WI.

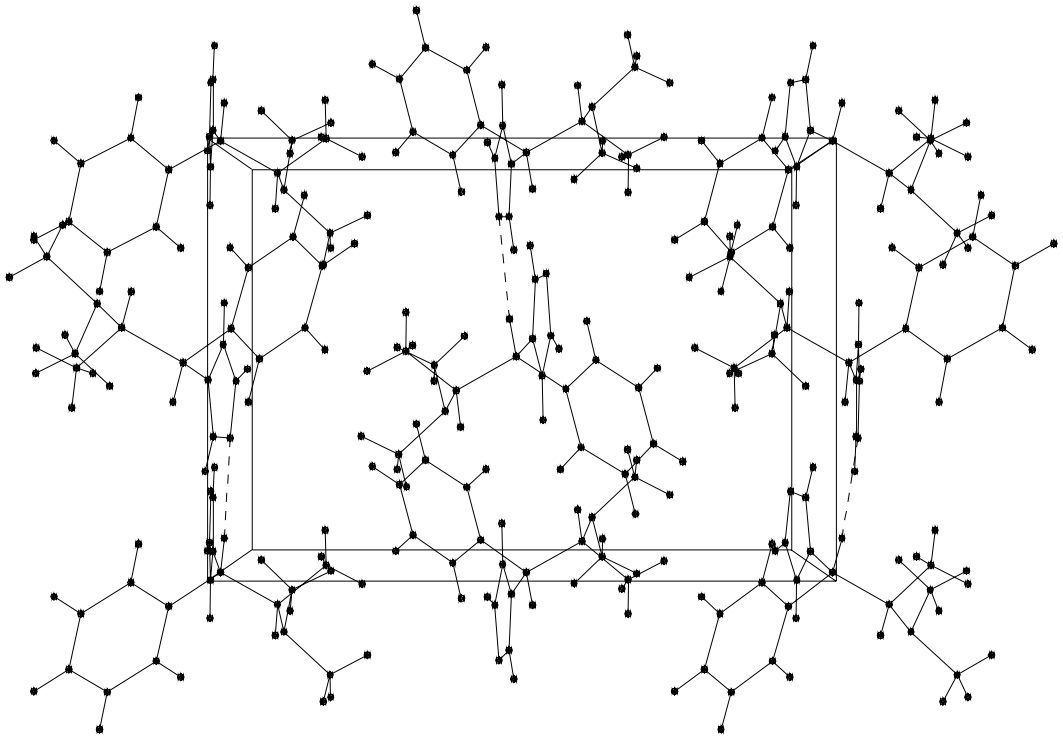

Table 1. Crystal data and structure refinement for 02110b.

Identification code	02110b	
Empirical formula	C14 H19 N3	
Formula weight	229.32	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 9.051(3) Å	α= 90°.
	b = 10.214(3) Å	β= 90°.
	c = 14.481(4) Å	γ = 90°.
Volume	1338.8(6) $Å^3$	
Z	4	
Density (calculated)	1.138 Mg/m^3	
Absorption coefficient	0.069 mm^{-1}	
F(000)	496	
Crystal habit and color	Colorless, Plate	
Crystal size	0.32 x 0.29 x 0.12 mm^3	
Theta range for data collection	2.44 to 25.05°.	
Index ranges	$-10 \le h \le 10$, $0 \le k \le 12$, $0 \le l \le 17$	
Reflections collected	13270	
Independent reflections	2371 [R(int) = 0.0224]	
Observed Reflections	2275	
Completeness to theta = 25.05°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.000000 and 0.934883	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2371 / 0 / 157	
Goodness-of-fit on F^2	1.073	
Final R indices [I>2sigma(I)]	R1 = 0.0326, wR2 = 0.0790	
R indices (all data)	R1 = 0.0344, wR2 = 0.0801	
Absolute structure parameter	0(2)	
Largest diff. peak and hole	0.115 and -0.200 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 02110b. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C1	1652(1)	4933(1)	4918(1)	27(1)
N1	3177(1)	4552(1)	5164(1)	31(1)
C2	3666(2)	3300(1)	5205(1)	42(1)
N2	5096(2)	3223(1)	5360(1)	52(1)
C3	5538(2)	4508(2)	5421(1)	46(1)
C4	4388(1)	5336(1)	5304(1)	37(1)
C5	1668(2)	5678(1)	3989(1)	29(1)
C6	2362(2)	4820(2)	3232(1)	45(1)
N3	171(1)	6160(1)	3789(1)	32(1)
C7	166(2)	7135(2)	3051(1)	50(1)
C8	-891(2)	5123(2)	3593(1)	49(1)
C9	886(1)	5651(1)	5695(1)	28(1)
C10	1213(2)	6941(1)	5928(1)	35(1)
C11	467(2)	7564(2)	6638(1)	47(1)
C12	-622(2)	6919(2)	7124(1)	50(1)
C13	-964(2)	5646(2)	6895(1)	51(1)
C14	-209(2)	5012(2)	6190(1)	39(1)

Table 3. Bond lengths [Å] and angles [°] for 02110b.

C1-N1	1.4775(16)	N3-C7	1.4603(18)
C1-C9	1.5122(18)	C7-H7A	0.9800
C1-C5	1.5459(18)	C7-H7B	0.9800
C1-H1A	1.0000	C7-H7C	0.9800
N1-C2	1.3547(18)	C8-H8A	0.9800
N1-C4	1.3725(17)	C8-H8B	0.9800
C2-N2	1.316(2)	C8-H8C	0.9800
C2-H2B	0.9500	C9-C14	1.3857(19)
N2-C3	1.375(2)	C9-C10	1.392(2)
C3-C4	1.351(2)	C10-C11	1.385(2)
C3-H3A	0.9500	C10-H10A	0.9500
C4-H4A	0.9500	C11-C12	1.378(2)
C5-N3	1.4704(17)	C11-H11A	0.9500
C5-C6	1.5378(19)	C12-C13	1.377(3)
C5-H5A	1.0000	C12-H12A	0.9500
C6-H6A	0.9800	C13-C14	1.389(2)
C6-H6B	0.9800	C13-H13A	0.9500
C6-H6C	0.9800	C14-H14A	0.9500
N3-C8	1.458(2)		

N1-C1-C9	112.11(10)	C4-C3-H3A	124.3
N1-C1-C5	109.33(10)	N2-C3-H3A	124.3
C9-C1-C5	114.44(10)	C3-C4-N1	105.60(13)
N1-C1-H1A	106.8	C3-C4-H4A	127.2
C9-C1-H1A	106.8	N1-C4-H4A	127.2
C5-C1-H1A	106.8	N3-C5-C6	115.25(11)
C2-N1-C4	106.43(12)	N3-C5-C1	109.12(10)
C2-N1-C1	124.39(11)	C6-C5-C1	110.12(11)
C4-N1-C1	128.89(11)	N3-C5-H5A	107.3
N2-C2-N1	112.67(13)	C6-C5-H5A	107.3
N2-C2-H2B	123.7	C1-C5-H5A	107.3
N1-C2-H2B	123.7	C5-C6-H6A	109.5
C2-N2-C3	103.90(12)	C5-C6-H6B	109.5
C4-C3-N2	111.41(14)	H6A-C6-H6B	109.5

C5-C6-H6C	109.5	C14-C9-C10	118.23(13)
H6A-C6-H6C	109.5	C14-C9-C1	118.95(12)
H6B-C6-H6C	109.5	C10-C9-C1	122.81(12)
C8-N3-C7	110.55(13)	C11-C10-C9	120.75(15)
C8-N3-C5	113.75(11)	C11-C10-H10A	119.6
C7-N3-C5	112.02(12)	C9-C10-H10A	119.6
N3-C7-H7A	109.5	C12-C11-C10	120.48(16)
N3-C7-H7B	109.5	C12-C11-H11A	119.8
H7A-C7-H7B	109.5	C10-C11-H11A	119.8
N3-C7-H7C	109.5	C13-C12-C11	119.30(15)
H7A-C7-H7C	109.5	C13-C12-H12A	120.4
H7B-C7-H7C	109.5	C11-C12-H12A	120.4
N3-C8-H8A	109.5	C12-C13-C14	120.46(15)
N3-C8-H8B	109.5	C12-C13-H13A	119.8
H8A-C8-H8B	109.5	C14-C13-H13A	119.8
N3-C8-H8C	109.5	C9-C14-C13	120.77(15)
H8A-C8-H8C	109.5	C9-C14-H14A	119.6
H8B-C8-H8C	109.5	C13-C14-H14A	119.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 02110b. 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}
C1	21(1)	23(1)	36(1)	0(1)	-4(1)	-1(1)
N1	24(1)	26(1)	42(1)	-1(1)	-3(1)	3(1)
C2	38(1)	29(1)	60(1)	-2(1)	-4(1)	7(1)
N2	40(1)	41(1)	74(1)	-5(1)	-7(1)	15(1)
C3	27(1)	50(1)	60(1)	-7(1)	-5(1)	5(1)
C4	29(1)	31(1)	51(1)	-4(1)	-5(1)	-1(1)
C5	28(1)	30(1)	31(1)	-3(1)	-1(1)	-1(1)
C6	40(1)	54(1)	40(1)	-13(1)	3(1)	7(1)
N3	33(1)	33(1)	30(1)	0(1)	-5(1)	3(1)
C7	59(1)	56(1)	36(1)	10(1)	-1(1)	16(1)
C8	35(1)	51(1)	62(1)	-8(1)	-19(1)	2(1)
C9	24(1)	31(1)	28(1)	6(1)	-5(1)	5(1)
C10	31(1)	36(1)	39(1)	-4(1)	3(1)	1(1)
C11	41(1)	52(1)	46(1)	-14(1)	-1(1)	10(1)
C12	40(1)	78(1)	34(1)	-2(1)	3(1)	23(1)
C13	34(1)	75(1)	44(1)	21(1)	11(1)	10(1)
C14	30(1)	42(1)	44(1)	14(1)	0(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 02110b.

	x	y	z	U(eq)
H1A	1089	4104	4813	32
H2B	3045	2558	5129	51
H3A	6526	4780	5530	55
H4A	4412	6265	5317	44
H5A	2319	6461	4069	35
H6A	2247	5251	2631	67
H6B	1866	3967	3218	67
H6C	3415	4695	3363	67
H7A	-824	7513	2994	75
H7B	444	6716	2467	75
H7C	876	7829	3197	75
H8A	-1888	5495	3567	74
H8B	-846	4461	4082	74
H8C	-652	4717	2999	74
H10A	1956	7399	5596	42
H11A	707	8443	6791	56
H12A	-1131	7348	7611	61
H13A	-1722	5199	7221	61
H14A	-446	4129	6044	47

Table 6. Torsion angles [°] for 02110b.

C9-C1-N1-C2	118.36(15)	C1-C5-N3-C8	-67.81(14)
C5-C1-N1-C2	-113.63(15)	C6-C5-N3-C7	-69.62(15)
C9-C1-N1-C4	-68.60(17)	C1-C5-N3-C7	165.91(11)
C5-C1-N1-C4	59.41(17)	N1-C1-C9-C14	-106.98(13)
C4-N1-C2-N2	0.02(19)	C5-C1-C9-C14	127.77(12)
C1-N1-C2-N2	174.38(13)	N1-C1-C9-C10	74.24(15)
N1-C2-N2-C3	-0.1(2)	C5-C1-C9-C10	-51.01(16)
C2-N2-C3-C4	0.1(2)	C14-C9-C10-C11	0.3(2)
N2-C3-C4-N1	-0.10(19)	C1-C9-C10-C11	179.10(13)
C2-N1-C4-C3	0.04(17)	C9-C10-C11-C12	-0.4(2)
C1-N1-C4-C3	-173.97(13)	C10-C11-C12-C13	-0.1(2)
N1-C1-C5-N3	-174.86(10)	C11-C12-C13-C14	0.8(2)
C9-C1-C5-N3	-48.16(14)	C10-C9-C14-C13	0.4(2)
N1-C1-C5-C6	57.71(13)	C1-C9-C14-C13	-178.48(12)
C9-C1-C5-C6	-175.59(11)	C12-C13-C14-C9	-0.9(2)
C6-C5-N3-C8	56.66(16)		

Symmetry transformations used to generate equivalent atoms: