Supplementary Material

Solid phase synthesis of bicyclic pyrrolidines

Paolo Quadrelli

University of Pavia, Department of Chemistry, Viale Taramelli 12, 27100 – Pavia (Italy)
Email: paolo.quadrelli@unipv.it

Table of Contents

1. X-ray diffraction analysis of compound 21a

Table S1. Crystal data and structure refinement

Identification code	Ron85	
Empirical formula	$C_{27}H_{30}N_2O_8S$	
Formula weight	542.59	
Temperature	295(2) K	
Wavelength	1.54186 Å [Cu-K_α]	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 8.9760(3) Å	α = 90°
	b = 9.1571(2) Å	β = 94.768(3)°
	c = 33.3606(8) Å	γ = 90°
Volume	2732.56(12) Å^3	
Z	4	
Density (calculated)	1.319 Mg/m^3	
Absorption coefficient	1.492 mm^{-1}	
F(000)	1144	
Crystal size	0.62 x 0.55 x 0.46 mm	
Data collection range	$2.66 \le \theta \le 64.95°$	
Index ranges	$-10 \le h \le 10$, $-8 \le k \le 10$, $-31 \le l \le 36$	
Reflections collected	4628	
Independent reflections	4131 [R(int) = 0.0279]	
Absorption correction	Psi scans	
Max. and min. transmission	0.597 and 0.449	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4131 / 20 / 348	
Goodness-of-fit on F^2	1.118	
Final R indices [$I > 2\sigma(I)$]	R_1 = 0.0369, wR_2 = 0.1024	
R indices (all data)	R_1 = 0.0394, wR_2 = 0.1048	
Largest diff. peak and hole	0.171 and -0.251 e.Å^{-3}	
Extinction coefficient	0.0033(2)	

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å x 10^3) with estimated standard deviations (e.s.d.s.) in parentheses. U_{eq} is defined as 1/3 of the trace of the orthogonalized U_{ij} tensor

	x	y	z	U_{eq}
C(1)	11767(2)	10652(2)	1433.7(6)	43.3(5)
C(111)	12760(2)	9845(2)	1164.2(6)	50.3(5)
C(112)	12724(3)	10230(3)	763.9(7)	67.8(7)
C(113)	13665(4)	9551(5)	509.7(9)	96.1(11)
C(114)	14635(4)	8489(4)	654.6(12)	99.7(12)
C(115)	14675(3)	8092(3)	1049.7(11)	87.5(9)
C(116)	13741(2)	8755(3)	1302.0(8)	64.3(6)
C(2)	12569(2)	11751(2)	1737.0(6)	43.5(5)
C(3)	14184(2)	11369(2)	1849.7(6)	48.1(5)
O(31)	15252.0(16)	11601.1(19)	1661.7(5)	66.3(4)
N(4)	14263.5(17)	10646.3(18)	2214.1(5)	47.3(4)
C(41)	15646(2)	10071(3)	2406.4(8)	67.0(6)
C(5)	12944(2)	10692(2)	2396.9(6)	44.0(5)
O(51)	12776.7(16)	10145.8(18)	2719.4(4)	59.9(4)
C(6)	11804(2)	11532(2)	2125.9(6)	41.0(4)
C(7)	10387.8(19)	10634.4(19)	2002.4(6)	38.6(4)
C(71)	9094(2)	11630(2)	1839.4(7)	49.7(5)
C(72)	9900(2)	9766(2)	2356.5(6)	43.5(4)
O(73)	9862(2)	8463.2(16)	2383.1(5)	65.3(4)
O(74)	9471.3(16)	10674.8(16)	2638.3(4)	53.6(4)
C(75)	9157(3)	10029(3)	3015.7(7)	71.9(7)
N(8)	10974.7(16)	9674.8(16)	1697.7(4)	39.0(4)
C(9)	9895(2)	8710(2)	1474.0(6)	45.2(5)
C(10)	10433(2)	7129(2)	1505.2(6)	50.2(5)
S(11)	9415.9(6)	5912.0(5)	1176.55(16)	50.78(18)
O(111)	7851.0(17)	6233.1(19)	1180.1(5)	68.8(5)
O(112)	9934(2)	4458.4(16)	1270.1(5)	71.1(5)
C(12)	9995(2)	6362(2)	697.9(6)	45.6(5)
C(13)	11198(2)	5630(2)	563.4(7)	55.6(5)
C(14)	11734(2)	6022(2)	203.5(7)	56.4(6)
C(15)	11072(2)	7152(2)	-22.1(6)	48.8(5)
C(16)	9847(2)	7864(2)	112.7(6)	53.8(5)
C(17)	9291(2)	7472(2)	472.6(6)	52.9(5)
C(18)	11680(2)	7664(2)	-400.8(7)	55.0(5)
O(19)	11175(2)	8660(2)	-601.8(5)	83.1(6)
O(20)	12859.7(18)	6885.6(18)	-483.8(5)	65.5(4)
C(21)	13610(3)	7262(3)	-838.8(7)	63.6(6)
C(22)	14993(3)	6362(3)	-818.3(9)	81.0(8)

Table S3. Anisotropic displacement parameters (Å x 10^3). The anisotropic displacement factor exponent takes the form:

$$-2\pi^2[h^2a^{*2}U_{11}+...+2hka^*b^*U_{12}]$$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C(1)	39.4(9)	48.7(11)	41.7(11)	8.2(9)	1.8(8)	-0.1(8)
C(111)	40.7(10)	64.3(13)	46.2(12)	-5.1(10)	5.3(9)	-9.5(9)
C(112)	58.6(13)	97.8(19)	47.2(14)	-4.8(13)	5.6(11)	-15.4(13)
C(113)	86(2)	146(3)	59.8(17)	-32.6(19)	28.1(15)	-41(2)
C(114)	68.8(18)	120(3)	116(3)	-53(2)	42.3(19)	-18.9(18)
C(115)	61.1(15)	85(2)	118(3)	-32.8(19)	20.3(16)	3.6(14)
C(116)	53.3(12)	65.6(14)	74.4(16)	-9.9(13)	7.4(11)	4.0(11)
C(2)	43.8(10)	38.1(10)	48.5(12)	7.1(9)	3.9(8)	-2.9(8)
C(3)	42.6(10)	48.8(11)	53.0(13)	-2.0(10)	4.7(9)	-9.6(9)
O(31)	46.0(8)	84.0(11)	70.0(10)	4.3(9)	11.8(7)	-14.8(8)
N(4)	37.1(8)	49.1(9)	54.5(11)	2.6(8)	-2.2(7)	-1.9(7)
C(41)	42.4(11)	75.1(16)	81.6(17)	10.1(14)	-5.3(11)	5.1(11)
C(5)	44.4(10)	41.4(10)	45.7(12)	-3.4(9)	-0.2(9)	-5.8(8)
O(51)	57.4(9)	73.8(10)	47.4(9)	10.8(8)	-2.0(7)	-3.4(8)
C(6)	40.8(9)	34.4(9)	47.7(11)	-1.2(8)	3.2(8)	-1.0(7)
C(7)	36.9(9)	36.5(9)	42.6(11)	2.0(8)	4.3(8)	0.7(7)
C(71)	43.0(10)	48.9(11)	57.6(13)	9.5(10)	6.0(9)	6.4(9)
C(72)	42.1(10)	44.2(11)	44.2(11)	0.5(9)	3.4(8)	-0.7(8)
O(73)	99.5(12)	43.1(8)	55.5(9)	5.8(7)	19.7(8)	-8.3(8)
O(74)	58.6(8)	55.9(8)	48.4(8)	0(7)	16.8(7)	7.8(7)
C(75)	78.5(16)	92.2(19)	47.5(14)	6.7(13)	20.5(12)	7.6(14)
N(8)	38.5(8)	39.6(8)	38.8(9)	-0.5(7)	3.2(6)	-2.6(6)
C(9)	43.4(10)	46.0(11)	45.2(11)	-2.2(9)	-2.0(8)	-3.4(8)
C(10)	61.9(12)	44.6(11)	43.1(12)	4.5(9)	-2.4(9)	-5.9(9)
S(11)	59.1(3)	42.9(3)	50.9(3)	-0.2(2)	7.4(2)	-12.1(2)
O(111)	54.2(9)	78.9(11)	75.0(11)	-8.7(9)	15.1(8)	-18.7(8)
O(112)	105.1(13)	38.2(8)	70.9(11)	7.1(7)	13.3(9)	-10.3(8)
C(12)	49.9(11)	42.4(10)	43.5(11)	-4.3(9)	-1.1(9)	-5.2(9)
C(13)	60.6(13)	49.5(12)	56.8(14)	9.2(10)	5.2(11)	9.6(10)
C(14)	57.1(12)	55.2(12)	57.6(14)	4.0(11)	9.0(10)	10.8(10)
C(15)	53.3(11)	50.1(11)	42.0(12)	-2.0(9)	-1.9(9)	-1.8(9)
C(16)	59.5(12)	55.3(12)	44.8(13)	1.3(10)	-6.0(10)	10.9(10)
C(17)	53.5(11)	57.6(12)	46.7(12)	-4.7(10)	-1.4(9)	8.4(10)
C(18)	61.1(13)	54.5(13)	48.6(13)	0.4(11)	0.2(10)	1.9(11)
O(19)	97.1(13)	85.7(13)	68.6(11)	28.0(10)	19.2(10)	27.8(11)
O(20)	68.1(10)	71.8(10)	58.7(10)	14.7(8)	18.2(8)	11.3(8)
C(21)	64.5(14)	71.4(15)	56.1(14)	10.1(12)	12.8(11)	-4.6(12)
C(22)	72.8(16)	87.1(19)	86.4(19)	13.1(16)	26.0(14)	7.9(14)

Table S4. Hydrogen atom coordinates (x 10^4) and isotropic displacement parameters (Å x 10^3) with e.s.d.s. in parentheses

	x	y	z	U_{eq}
H(1)	11017	11192	1263	52
H(112)	12065	10950	663	81
H(113)	13633	9821	240	115
H(114)	15266	8039	485	120
H(115)	15335	7371	1149	105
H(116)	13770	8466	1570	77
H(2)	12463	12759	1640	52
H(41a)	15473	9703	2668	100
H(41b)	16381	10834	2433	100
H(41c)	16000	9295	2246	100
H(6)	11555	12468	2246	49
H(71a)	9425	12245	1631	75
H(71b)	8780	12226	2054	75
H(71c)	8272	11041	1732	75
H(75a)	8546	9178	2966	108
H(75b)	8637	10723	3169	108
H(75c)	10078	9758	3164	108
H(9a)	9780	9003	1194	54
H(9b)	8929	8793	1583	54
H(10a)	10372	6796	1779	60
H(10b)	11476	7098	1450	60
H(13)	11647	4873	715	67
H(14)	12544	5527	112	68
H(16)	9391	8614	-40	65
H(17)	8462	7945	561	63
H(21a)	13856	8294	-838	76
H(21b)	12978	7042	-1081	76
H(22a)	15634	6636	-586	122
H(22b)	15505	6519	-1056	122
H(22c)	14734	5348	-800	122

Table S5. Interatomic distances (Å) with e.s.d.s in parentheses

C(1)-N(8)	1.479(2)	C(1)-C(111)	1.510(3)
C(1)-C(2)	1.560(3)	C(111)-C(112)	1.379(3)
C(111)-C(116)	1.384(3)	C(112)-C(113)	1.392(4)
C(113)-C(114)	1.367(5)	C(114)-C(115)	1.365(5)
C(115)-C(116)	1.377(4)		
C(2)-C(3)	1.508(3)	C(2)-C(6)	1.531(3)
C(3)-O(31)	1.207(2)	C(3)-N(4)	1.381(3)
N(4)-C(5)	1.377(2)	N(4)-C(41)	1.448(3)
C(5)-O(51)	1.207(2)	C(5)-C(6)	1.517(3)
C(6)-C(7)	1.541(2)	C(7)-N(8)	1.474(2)
C(7)-C(72)	1.519(3)	C(7)-C(71)	1.540(3)
C(72)-O(73)	1.197(2)	C(72)-O(74)	1.336(2)
O(74)-C(75)	1.440(3)	N(8)-C(9)	1.469(2)
C(9)-C(10)	1.527(3)	C(10)-S(11)	1.764(2)
S(11)-O(112)	1.436(2)	S(11)-O(111)	1.436(2)
S(11)-C(12)	1.768(2)	C(12)-C(13)	1.378(3)
C(12)-C(17)	1.385(3)	C(13)-C(14)	1.378(3)
C(14)-C(15)	1.384(3)	C(15)-C(16)	1.385(3)
C(15)-C(18)	1.493(3)	C(16)-C(17)	1.385(3)
C(18)-O(19)	1.198(3)	C(18)-O(20)	1.325(3)
O(20)-C(21)	1.452(3)	C(21)-C(22)	1.487(3)

Table S6. Angles between interatomic vectors (°) with e.s.d.s in parentheses

N(8)-C(1)-C(111)	113.3(2)	N(8)-C(1)-C(2)	102.9(2)
C(111)-C(1)-C(2)	116.0(2)	C(112)-C(111)-C(116)	118.0(2)
C(112)-C(111)-C(1)	119.0(2)	C(116)-C(111)-C(1)	123.1(2)
C(111)-C(112)-C(113)	120.6(3)	C(114)-C(113)-C(112)	120.2(3)
C(115)-C(114)-C(113)	119.7(3)	C(114)-C(115)-C(116)	120.3(3)
C(115)-C(116)-C(111)	121.2(3)		
C(3)-C(2)-C(6)	104.4(2)	C(3)-C(2)-C(1)	113.2(2)
C(6)-C(2)-C(1)	104.45(14)	O(31)-C(3)-N(4)	123.8(2)
O(31)-C(3)-C(2)	128.2(2)	N(4)-C(3)-C(2)	108.0(2)
C(5)-N(4)-C(3)	113.1(2)	C(5)-N(4)-C(41)	123.5(2)
C(3)-N(4)-C(41)	123.0(2)	O(51)-C(5)-N(4)	123.9(2)
O(51)-C(5)-C(6)	127.8(2)	N(4)-C(5)-C(6)	108.3(2)
C(5)-C(6)-C(2)	104.1(2)	C(5)-C(6)-C(7)	113.0(2)
C(2)-C(6)-C(7)	105.2(2)	N(8)-C(7)-C(72)	111.5(2)
N(8)-C(7)-C(71)	114.3(2)		
C(72)-C(7)-C(71)	109.1(2)	N(8)-C(7)-C(6)	99.68(13)
C(72)-C(7)-C(6)	110.9(2)	C(71)-C(7)-C(6)	111.1(2)
O(73)-C(72)-O(74)	123.9(2)	O(73)-C(72)-C(7)	126.3(2)
O(74)-C(72)-C(7)	109.8(2)	C(72)-O(74)-C(75)	116.6(2)
C(9)-N(8)-C(7)	116.78(14)	C(9)-N(8)-C(1)	113.0(2)
C(7)-N(8)-C(1)	105.49(14)	N(8)-C(9)-C(10)	110.1(2)
C(9)-C(10)-S(11)	114.33(14)	O(112)-S(11)-O(111)	119.15(11)
O(112)-S(11)-C(10)	108.02(10)	O(111)-S(11)-C(10)	108.76(10)
O(112)-S(11)-C(12)	107.35(10)	O(111)-S(11)-C(12)	108.72(10)
C(10)-S(11)-C(12)	103.78(9)		
C(13)-C(12)-C(17)	121.0(2)	C(13)-C(12)-S(11)	118.6(2)
C(17)-C(12)-S(11)	120.4(2)	C(14)-C(13)-C(12)	119.8(2)
C(13)-C(14)-C(15)	120.2(2)	C(14)-C(15)-C(16)	119.5(2)
C(14)-C(15)-C(18)	121.6(2)	C(16)-C(15)-C(18)	118.9(2)
C(17)-C(16)-C(15)	120.8(2)	C(16)-C(17)-C(12)	118.7(2)
O(19)-C(18)-O(20)	124.3(2)	O(19)-C(18)-C(15)	124.4(2)
O(20)-C(18)-C(15)	111.3(2)	C(18)-O(20)-C(21)	118.4(2)
O(20)-C(21)-C(22)	105.8(2)		