SI-OL0101436

Supplementary Material

Title:

A facile three-component coupling procedure for the synthesis of substituted tetrahydroisoindole-1,3-diones from α,β-unsaturated aldehydes

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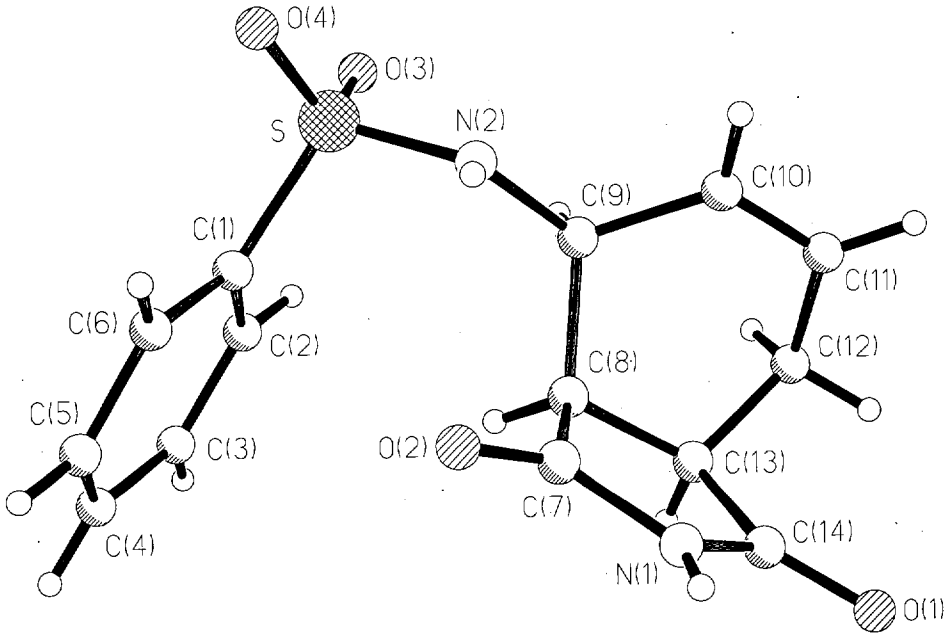
O(4)
O(3)
S
N(2)
C(9)
C(10)
C(1)
C(11)
C(6)
C(2)
C(8)
C(12)
C(5)
O(2)
C(3)
C(13)
C(7)
C(14)
C(4)
N(1)
O(1)

Table 1. Crystal data and structure refinement for 1d.

Identification code	852
Empirical formula	C14 H14 N2 O4 S
Formula weight	306.33
Crystal system	Monoclinic
Space group	P21/n
Unit cell dimensions	a= 10.707(2) A alpha= 90 deg. b= 6.9070(10) A beta= 98.39(3) deg. c= 19.518(4) A gamma= 90 deg.
Temperature for cell	293(2)
Number of reflecions for cell	5000
Theta for cell determination	4 to 20 deg
Volume	1428.0(4) A^3
Z	4
Density (calculated)	1.425 Mg/m^3
Absorption coefficient	0.244 mm^-1
F(000)	640
Crystal colour	colourless
Crystal description	prism
Crystal size	0.5 x 0.5 x 0.4 mm
Temperature	293(2) K
Wavelength	0.71073 A
Radiation type	MoK\a
Radiation source	fine-focus sealed tube
Monochromator	graphite
Measurment device	STOE-IPDS
Scan method	laser scanned imaging plate
Standards (intensity + orient.)	50-200
Intensity after	360 sec
Theta range for data collection	2.11 to 24.31 deg.
Index ranges	-12<=h<=12, -7<=k<=7, 0<=l<=22

Reflections collected	4084
Independent reflections	2161 [R(int) = 0.0363]
Reflections unique	2161
Reflections observed	1762
Criterion	>2sigma(I)
Absorption correction	Mean imaging plate intensity method
Structure solution primary	direct
Structure solution secondary	difmap
Hydrogen positions	geom
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2158 / 0 / 211
Final R indices [I>2sigma(I)]	R1 = 0.0401, wR2 = 0.0962
R indices (all data)	R1 = 0.0510, wR2 = 0.1113
Goodness-of-fit on F^2(all)	1.021
Goodness-of-fit on F^2(obs)	1.109
Shift/esd(max)	0.000
Shift/esd(mean)	0.000
Weighting scheme	
calc w=1/[\s^2^(Fo^2^)+(0.0548P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3	
Largest diff. peak and hole	0.226 and -0.268 e.A^-3
Diff. density rms	0.050
Data collection	STOE-EXPOSE
Cell refinement	STOE-CELL
Data reduction	STOE-INTEGRATE
Structure solution	SHELXS-86 (Sheldrick, 1990)
Structure refinement	SHELXL-93 (Sheldrick, 1993)
Molecular graphics	XP
Publication material	SHELXL-93

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 1d. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
C(1)	10233(2)	175(3)	3108(1)	42(1)
C(2)	9881(3)	1548(3)	2598(1)	55(1)
C(3)	10713(3)	3009(4)	2491(2)	72(1)
C(4)	11875(3)	3103(5)	2891(2)	80(1)
C(5)	12210(3)	1778(5)	3394(2)	79(1)
C(6)	11401(2)	287(4)	3510(1)	61(1)
C(7)	8984(2)	2810(3)	4646(1)	39(1)
C(8)	8074(2)	2744(3)	3980(1)	35(1)
C(9)	7518(2)	669(3)	3830(1)	34(1)
C(10)	6513(2)	271(3)	4267(1)	43(1)
C(11)	5636(2)	1592(3)	4318(1)	47(1)
C(12)	5695(2)	3477(3)	3953(1)	45(1)
C(13)	7050(2)	4228(3)	4084(1)	40(1)
C(14)	7373(2)	4858(3)	4829(1)	45(1)
N(1)	8492(2)	3982(3)	5101(1)	46(1)
N(2)	8444(2)	-900(2)	3902(1)	37(1)
O(1)	6775(2)	5930(2)	5144(1)	66(1)
O(2)	9990(2)	1979(2)	4771(1)	51(1)
O(3)	8230(2)	-1797(2)	2677(1)	55(1)
O(4)	9861(2)	-3293(2)	3539(1)	56(1)
S	9157(1)	-1621(1)	3279(1)	41(1)

Table 3. Bond lengths [A] and angles [deg] for 1d.

C(1)-C(6)	1.379(3)
C(1)-C(2)	1.386(3)
C(1)-S	1.758(2)
C(2)-C(3)	1.383(4)
C(3)-C(4)	1.370(5)
C(4)-C(5)	1.351(5)
C(5)-C(6)	1.385(4)
C(7)-O(2)	1.214(3)
C(7)-N(1)	1.364(3)
C(7)-C(8)	1.507(3)
C(8)-C(13)	1.536(3)
C(8)-C(9)	1.563(3)
C(9)-N(2)	1.462(3)
C(9)-C(10)	1.493(3)
C(10)-C(11)	1.323(3)
C(11)-C(12)	1.490(3)
C(12)-C(13)	1.527(3)
C(13)-C(14)	1.508(3)
C(14)-O(1)	1.204(3)
C(14)-N(1)	1.377(3)
N(2)-S	1.605(2)
O(3)-S	1.428(2)
O(4)-S	1.431(2)
C(6)-C(1)-C(2)	120.0(2)
C(6)-C(1)-S	119.7(2)
C(2)-C(1)-S	120.1(2)
C(3)-C(2)-C(1)	119.6(3)
C(4)-C(3)-C(2)	120.0(3)
C(5)-C(4)-C(3)	120.3(3)
C(4)-C(5)-C(6)	121.1(3)
C(1)-C(6)-C(5)	119.0(3)
O(2)-C(7)-N(1)	124.4(2)
O(2)-C(7)-C(8)	127.1(2)
N(1)-C(7)-C(8)	108.5(2)
C(7)-C(8)-C(13)	104.5(2)
C(7)-C(8)-C(9)	111.7(2)
C(13)-C(8)-C(9)	112.2(2)
N(2)-C(9)-C(10)	110.1(2)
N(2)-C(9)-C(8)	115.3(2)
C(10)-C(9)-C(8)	110.3(2)
C(11)-C(10)-C(9)	119.7(2)
C(10)-C(11)-C(12)	119.1(2)
C(11)-C(12)-C(13)	108.8(2)
C(14)-C(13)-C(12)	109.8(2)
C(14)-C(13)-C(8)	104.6(2)
C(12)-C(13)-C(8)	115.5(2)
O(1)-C(14)-N(1)	124.8(2)
O(1)-C(14)-C(13)	127.2(2)
N(1)-C(14)-C(13)	108.0(2)
C(7)-N(1)-C(14)	114.0(2)
C(9)-N(2)-S	123.2(2)
O(3)-S-O(4)	119.33(9)
O(3)-S-N(2)	107.38(11)
O(4)-S-N(2)	105.42(10)
O(3)-S-C(1)	107.46(11)
O(4)-S-C(1)	108.13(11)
N(2)-S-C(1)	108.77(10)

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 1d.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
C(1)	39(1)	53(1)	35(1)	-14(1)	8(1)	-1(1)
C(2)	52(2)	61(2)	53(2)	-5(1)	8(1)	0(1)
C(3)	81(2)	67(2)	73(2)	6(1)	26(2)	-7(2)
C(4)	80(3)	84(2)	81(2)	-12(2)	27(2)	-31(2)
C(5)	49(2)	122(3)	67(2)	-23(2)	11(2)	-29(2)
C(6)	42(2)	93(2)	46(1)	-1(1)	3(1)	-9(1)
C(7)	39(1)	32(1)	45(1)	-3(1)	7(1)	-2(1)
C(8)	37(1)	35(1)	36(1)	4(1)	11(1)	-2(1)
C(9)	34(1)	36(1)	32(1)	1(1)	4(1)	1(1)
C(10)	41(1)	39(1)	50(1)	8(1)	11(1)	-4(1)
C(11)	38(1)	52(1)	53(1)	8(1)	19(1)	0(1)
C(12)	41(1)	50(1)	46(1)	7(1)	8(1)	11(1)
C(13)	44(1)	33(1)	44(1)	10(1)	12(1)	5(1)
C(14)	50(2)	36(1)	50(1)	-2(1)	13(1)	4(1)
N(1)	48(1)	45(1)	42(1)	-8(1)	2(1)	3(1)
N(2)	38(1)	40(1)	34(1)	-3(1)	1(1)	4(1)
O(1)	71(1)	63(1)	67(1)	-17(1)	17(1)	18(1)
O(2)	42(1)	52(1)	56(1)	-7(1)	-2(1)	7(1)
O(3)	47(1)	73(1)	42(1)	-21(1)	-1(1)	-9(1)
O(4)	60(1)	44(1)	64(1)	-13(1)	9(1)	14(1)
S	38(1)	43(1)	40(1)	-13(1)	4(1)	0(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 1d.

	x	y	z	U(eq)
H(2)	9089(3)	1488(3)	2330(1)	66
H(3)	10485(3)	3928(4)	2147(2)	86
H(4)	12436(3)	4083(5)	2817(2)	96
H(5)	12996(3)	1868(5)	3667(2)	95
H(6)	11642(2)	-625(4)	3855(1)	73
H(8)	8498(20)	3098(26)	3636(11)	32(5)
H(9)	7154(18)	662(26)	3356(11)	30(5)
H(10)	6503(2)	-898(3)	4502(1)	51
H(11)	4992(2)	1347(3)	4579(1)	56
H(12A)	5132(2)	4403(3)	4124(1)	54
H(12B)	5430(2)	3305(3)	3460(1)	54
H(13)	7151(21)	5310(31)	3835(11)	44(6)
HN1	8842(27)	4077(34)	5499(15)	60(8)
HN2	8838(22)	-1013(30)	4236(12)	35(7)