$S = 1.05$	$(\Delta/\sigma)_{max} < 0.001$
3260 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
244 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	Extinction correction: none
	Scattering factors from *International Tables for Crystallography* (Vol. C)

Data collection: *SMART* (Bruker, 1996). Cell refinement: *SMART*. Data reduction: *SHELXTL* (Sheldrick, 1997). Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1433). Services for accessing these data are described at the back of the journal.

References

Bott, G., Field, L. D. & Sternhell, S. (1980). *J. Am. Chem. Soc.* **102**, 5618–5626.

Bruker (1996). *SMART Software Reference Manual*. Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Cosmo, R., Hambley, T. W. & Sternhell, S. (1990). *Acta Cryst.* B**46**, 557–562.

Krohn, K. (1990). *Tetrahedron*, **46**, 291–318.

Rohr, J. & Thiercke, R. (1992). *Nat. Prod. Rep.* **9**, 103–137.

Sheldrick, G. M. (1997). *SHELXTL*. Version 5.03. *Reference Manual*. Bruker Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1999). C**55**, 622–624

Spiro[1-azabicyclo[5.3.0]decane-6,2′(5′*H*)-furan]-5′,10-dione: an example of kryptoracemic crystallization

VINCENT M. LYNCH, SCOTT K. BUR AND STEPHEN F. MARTIN

Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712, USA. E-mail: vmlynch@mail.utexas.edu

(*Received 28 April 1998; accepted 2 October 1998*)

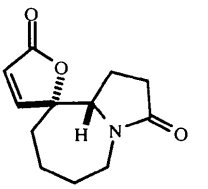
Abstract

A racemic mixture of the title compound, $C_{12}H_{15}NO_3$, crystallizes in the chiral, monoclinic space group $P2_1$, with one enantiomerically related pair of molecules per asymmetric unit. This mode of crystallization is extremely rare. The molecules pack to form several close C—H···O interactions. Interestingly, while the conformations of the individual rings in the two molecules are very similar, the overall molecular conformation is different.

Comment

Kryptoracemic crystallization, a term coined by Bernal *et al.* (1996), describes the phenomenon of a racemate crystallizing into a chiral space group where the contents of the asymmetric unit constitute a racemic mixture. Kryptoracemic crystallization is similar to conglomerate crystallization in that a racemic compound forms chiral crystals. However, in conglomerate crystallization, which has been estimated to occur with a frequency of 5–10% (Jacques *et al.*, 1981), the racemate spontaneously resolves into a mixture of crystals of the pure enantiomers. Judging by the number of known examples, kryptoracemic crystallization occurs much less frequently (Bernal *et al.*, 1996). However, many of the examples of organic molecules forming kryptoracemic crystals identified by Bernal *et al.* (1996) were incorrectly characterized as such. Those that are true kryptoracemates are reported by Furberg & Hassel (1950), Mostad *et al.* (1975), Schouwstra (1973), Baert *et al.* (1978), Bigoli *et al.* (1981) and Furusaki *et al.* (1982).

In the title compound, (I), molecules of alternating chirality stack in columns along the *c* axis. Within



(I)

a column, molecules are connected by a series of C—H···O hydrogen bonds. Adjacent columns related by an *a*-axis translation are also hydrogen-bonded resulting in a two-dimensional C—H···O hydrogen-bonding network perpendicular to the *b* axis. Three of

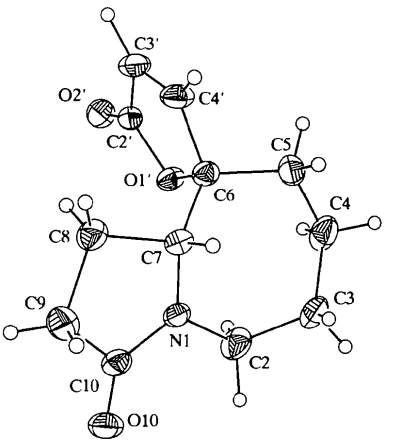


Fig. 1. View of molecule 1 showing the atom-labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. H atoms are drawn to an arbitrary scale.

the four carbonyl-O atoms are involved in this hydrogen-bonding scheme. The fourth carbonyl-O atom, O2′*A* of molecule 2, is 3.254 (2) Å from N1 of molecule 1. This O···N vector is approximately perpendicular to the plane defined by the three atoms bound to N1.

The seven-membered azepine rings each assume a conformation intermediate between a chair and a twist chair, while the lactam rings are in the envelope conformation. Fig. 2 shows the superposition of azepine ring atoms of molecule 2 onto the equivalent atoms of an inverted molecule 1. The major difference in conformation is in the orientation of the lactam-ring atoms.

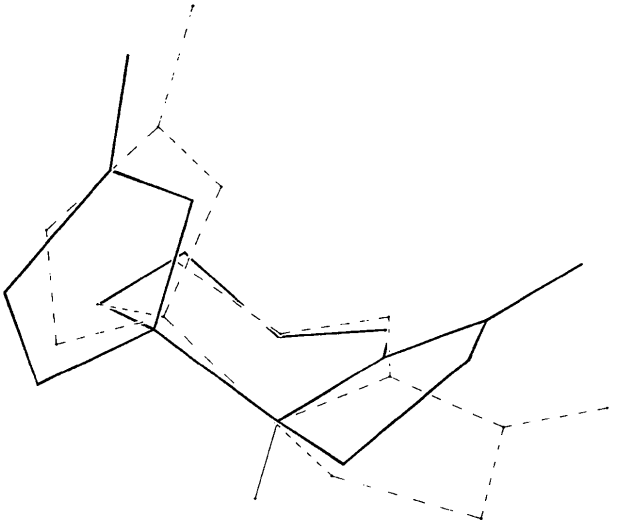


Fig. 2. Fit by least-squares of atoms of the azepine ring of molecule 1 (dashed lines) to the equivalent atoms of molecule 2 (solid lines) illustrating the conformational differences. For the purposes of this comparison, the configuration of molecule 1 was inverted.

Experimental

The structure of the title compound was undertaken as part of a project aimed at the synthesis of Stemona alkaloids, such as (+)-croomine (Martin & Barr, 1996), using the vinylogous Mannich reaction. Full details of the synthetic procedures have been reported elsewhere (Martin & Bur, 1997).

Crystal data

$C_{12}H_{15}NO_3$
M_r = 221.25
Monoclinic
$P2_1$
a = 9.153 (1) Å
b = 11.495 (1) Å
c = 10.277 (1) Å
β = 93.161 (4)°
V = 1079.6 (2) Å^3
Z = 4
D_x = 1.361 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 51 reflections
θ = 12.2–12.5°
μ = 0.098 mm^{-1}
T = 183 (2) K
Cut triangular prism
0.7 × 0.6 × 0.5 mm
Colorless

Data collection

Siemens $P4$ diffractometer
ω scans
Absorption correction: none
5988 measured reflections
3526 independent reflections
3263 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 30°
$h = -12 \rightarrow 8$
$k = -1 \rightarrow 16$
$l = -14 \rightarrow 14$
3 standard reflections every 75 reflections
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.082
S = 1.01
3526 reflections
406 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0418P)^2 + 0.2386P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = -0.012$
$\Delta\rho_{max}$ = 0.262 e Å^{-3}
$\Delta\rho_{min}$ = −0.168 e Å^{-3}
Extinction correction: *SHELXL*
Extinction coefficient: 0.042 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected torsion angles (°)*

	Molecule 1	Molecule 2†	
N1—C2—C3—C4	−74.7 (3)	58.3 (3)	[5.9 (13)]
C2—C3—C4—C5	76.9 (3)	−88.0 (3)	[−63.6 (7)]
C3—C4—C5—C6	−58.4 (3)	68.2 (2)	
C4—C5—C6—C7	64.9 (2)	−56.0 (2)	
C5—C6—C7—N1	−79.0 (2)	67.7 (2)	
C6—C7—N1—C2	50.3 (2)	−72.0 (3)	[−93.5 (7)]
C7—N1—C2—C3	18.8 (3)	15.3 (4)	[66.5 (11)]

† The values involving C2*B* are in square brackets.

The absolute configuration could not be determined from the X-ray results. One atom of the azepine ring of molecule 2 was found to be disordered about two positions representing different conformations of the seven-membered ring. The site occupancy factors for the two atoms, C2*A* and C2*B*, were refined while refining a common isotropic displacement parameter. The site occupancy factor for C2*A* refined to 76 (1)% and the site occupancy factors were subsequently fixed. C2*A* was then refined anisotropically. H atoms on C2*A* were calculated in idealized positions. No H atoms were calculated for C2*B*. All other H atoms were located in a difference electron-density map and refined isotropically. The C—H bond lengths ranged from 0.91 (3) to 1.11 (3) Å.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *XS* in *SHELXTL-Plus* (Sheldrick, 1994). Program(s) used to refine structure: *XL* in *SHELXTL-Plus*. Molecular graphics: *XP* in *SHELXTL-Plus*.

Funding for this work was provided by the Robert A. Welch Foundation and the National Institute of Health.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1145). Services for accessing these data are described at the back of the journal.

References

Baert, F., Fouret, R., Oonk, H. A. J. & Kroon, J. (1978). *Acta Cryst.* B**34**, 222–226.
Bernal, I., Cai, J., Massoud, S. S., Watkins, S. F. & Fronczek, F. R. (1996). *J. Coord. Chem.* **38**, 165–181.
Bigoli, F., Lanfranchi, M., Leporati, E., Nardelli, M. & Pellinghelli, M. A. (1981). *Acta Cryst.* B**37**, 1258–1265.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Furberg, S. & Hassel, O. (1950). *Acta Chem. Scand.* pp. 1020–1023.
Furusaki, A., Abe, K. & Matsumoto, T. (1982). *Bull. Chem. Soc. Jpn*, **55**, 611–612.
Jacques, J., Collet, A. & Wilen, S. H. (1981). *Enantiomers, Racemates, and Resolutions*, p. 81. New York: John Wiley.
Martin, S. F. & Barr, K. J. (1996). *J. Am. Chem. Soc.* **118**, 3299–3300.
Martin, S. F. & Bur, S. K. (1997). *Tetrahedron Lett.* **44**, 7641–7644.
Mostad, A., Romming, C. & Tressum, L. (1975). *Acta Chem. Scand. Ser. B*, **29**, 171–176.
Schouwstra, Y. (1973). *Acta Cryst.* B**29**, 1636–1641.
Sheldrick, G. M. (1994). *SHELXTL-Plus*. Release 5.03. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1994). *XSCANS User's Manual*. Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Acta Cryst. (1999). C**55**, 624–626

N,N-Bis(*p*-nitrophenylsulfonyl)cyclohexylamine

JOSEPH S. BRUNZELLE,[a] VERONICA A. CURTIS-PALMER[b] AND STEPHEN F. PAVKOVIC[a]

[a]*Department of Chemistry, Loyola University Chicago, Chicago, IL 60626, USA, and* [b]*Department of Chemistry, Northeastern Illinois University, Chicago, IL 60625, USA. E-mail: spavko1@luc.edu*

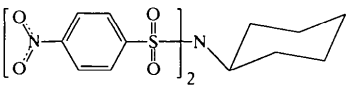
(*Received 20 August 1998; accepted 15 December 1998*)

Abstract

The title compound ($C_{18}H_{19}N_3O_8S_2$) undergoes pyrolytic reaction to yield cyclohexene and *N,N*-bis(*p*-nitrophenylsulfonyl)amine without rearrangement. Non-bonded contacts between oxygen and β-carbon H atoms were thought to be important in the reaction mechanism, but distances were found to be very similar to those present in a derivative which does not undergo pyrolysis.

Comment

N,N-Disulfonimide derivatives of certain alkyl amines stereoselectively pyrolyze to form alkenes in high yields and without skeletal rearrangement (Curtis *et al.*, 1981). The reaction only occurs when the α-C atom on the parent amine is tertiary. A mechanism was proposed that involved a β-H atom linked to an O atom in the transition state. This suggested that preferred orientations at α- and/or β-C atoms might determine whether or not a given compound underwent pyrolysis. Accordingly, structure determinations were planned for the *N*-alkyl-*N,N*-disulfonimide compounds tested. We have reported the structure of the non-reactive compound *N,N*-bis(*p*-nitrophenylsulfonyl)-phenethylamine (Curtis & Pavkovic, 1983). Now we wish to report the structure of the reactive cyclohexylamine derivative, (I), which forms cyclohexene upon pyrolysis.



(I)

Selected bond distances and angles are listed in Table 1. Fig. 1 shows a diagram of the molecule. The central feature in this structure is the nearly planar grouping of the amine-N atom and the three atoms bonded to it. The N is bonded to two S and a tertiary C atom from the cyclohexyl group. The N atom is 0.10 (4) Å out of the plane defined by its bonded atoms.

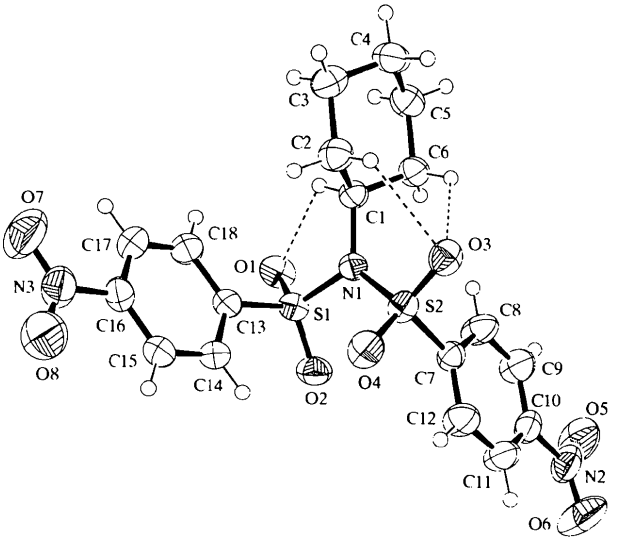


Fig. 1. An *ORTEP*III molecular structure diagram showing non-H atoms at 50% probability. Short non-bonded contacts between O and α- and β-C H atoms are shown as dashed lines.

Bond distances from each S to like target atoms are the same in both cases and comparable angles about S are very similar. However, orientations about S—N bonds for S-bonded *p*-nitrophenyl groups are decidedly different as shown by torsion angles of −82.9 (1)° for C1—N1—S1—C13 and −117.8 (1)° for C1—N1—S2—C7. Least-squares planes of the two phenyl rings