Data collection

Nicolet *R*3*m*/*E* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4336 measured reflections
4246 independent reflections
3397 observed reflections [$F > 3\sigma(F)$]

$R_{\rm int} = 0.0150$
$\theta_{\max} = 25.0°$
$h = 0 \rightarrow 11$
$k = -12 \rightarrow 12$
$l = -18 \rightarrow 18$
2 standard reflections monitored every 100 reflections intensity decay: <5%

Refinement

Refinement on *F*
$R = 0.0466$
$wR = 0.0466$
$S = 0.973$
3397 reflections
325 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\max} = 0.032$
$\Delta\rho_{\max} = 0.63$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	*y*	*z*	$U_{\rm eq}$
S1	1.2680 (1)	0.6734 (1)	0.2733 (1)	0.043 (1)
S2	0.9181 (1)	0.5441 (1)	0.3207 (1)	0.041 (1)
S3	1.3871 (1)	0.4694 (1)	0.3790 (1)	0.038 (1)
S4	0.9908 (1)	0.3274 (1)	0.4354 (1)	0.041 (1)
C1	1.0622 (5)	0.6797 (4)	0.2616 (3)	0.043 (2)
C2	1.2196 (4)	0.5230 (4)	0.3386 (2)	0.034 (1)
C3	1.0600 (4)	0.4650 (4)	0.3604 (2)	0.034 (1)
C4	0.8867 (4)	0.1567 (4)	0.3584 (2)	0.035 (1)
C5	0.8700 (5)	0.1600 (4)	0.2641 (2)	0.041 (1)
C6	0.7973 (5)	0.0267 (4)	0.2026 (3)	0.044 (2)
C7	0.7398 (4)	−0.1116 (4)	0.2364 (3)	0.041 (1)
C8	0.7450 (4)	−0.1236 (4)	0.3273 (3)	0.040 (1)
C9	0.8178 (4)	0.0123 (4)	0.3873 (2)	0.036 (1)
C10	1.3539 (4)	0.3219 (4)	0.2919 (2)	0.033 (1)
C11	1.2583 (5)	0.3057 (4)	0.2051 (2)	0.041 (2)
C12	1.2275 (5)	0.1844 (4)	0.1384 (2)	0.044 (2)
C13	1.2960 (5)	0.0823 (4)	0.1605 (2)	0.039 (1)
C14	1.3930 (5)	0.0932 (4)	0.2449 (2)	0.039 (1)
C15	1.4202 (4)	0.2147 (4)	0.3086 (2)	0.033 (1)
N1	0.8181 (4)	−0.0031 (4)	0.4839 (2)	0.044 (1)
N2	0.6684 (4)	−0.2525 (4)	0.1707 (3)	0.054 (1)
N3	1.5241 (4)	0.2237 (4)	0.3968 (2)	0.041 (1)
N4	1.2634 (5)	−0.0471 (4)	0.0919 (2)	0.054 (2)
O9	1.0252 (4)	0.7661 (3)	0.2191 (2)	0.059 (1)
O1	0.8801 (4)	0.1129 (3)	0.5381 (2)	0.059 (1)
O2	0.7596 (4)	−0.1309 (3)	0.5063 (2)	0.070 (2)
O3	0.6739 (5)	−0.2387 (4)	0.0910 (2)	0.075 (2)
O4	0.6090 (4)	−0.3751 (3)	0.2004 (3)	0.076 (2)
O5	1.5351 (4)	0.1077 (3)	0.4184 (2)	0.056 (1)
O6	1.5977 (4)	0.3498 (3)	0.4440 (2)	0.054 (1)
O7	1.3438 (5)	−0.1244 (4)	0.1083 (2)	0.074 (2)
O8	1.1591 (5)	−0.0694 (4)	0.0226 (2)	0.086 (2)
C1′	0.7423 (7)	0.4637 (6)	0.0102 (3)	0.073 (3)
N1′	0.7771 (5)	0.3696 (4)	0.0782 (2)	0.059 (2)
O1′	0.9283 (4)	0.3944 (4)	0.1051 (2)	0.074 (2)
O2′	0.6552 (5)	0.2721 (5)	0.1025 (3)	0.110 (2)

Table 2. *Selected geometric parameters* (Å, °)

S1—C1	1.777 (5)	S4—C4	1.766 (3)
S2—C3	1.747 (4)	C1′—N1′	1.492 (7)
S4—C3	1.770 (4)	S2—C1	1.778 (4)
C2—C3	1.346 (5)	S3—C10	1.776 (4)
N1′—O2′	1.206 (6)	C1—O9	1.201 (6)
S1—C2	1.749 (4)	N1′—O1′	1.220 (6)
S3—C2	1.767 (4)		
C1—S1—C2	96.9 (2)	C1—S2—C3	96.5 (2)
C2—S3—C10	101.6 (2)	C3—S4—C4	101.2 (2)
S1—C1—S2	112.1 (2)	S1—C1—O9	123.9 (3)
S2—C1—O9	124.0 (3)	S1—C2—S3	118.2 (2)
S1—C2—C3	116.8 (3)	S3—C2—C3	124.7 (3)
S2—C3—S4	117.4 (2)	S2—C3—C2	117.8 (3)
S4—C3—C2	124.6 (3)	S4—C4—C5	120.9 (3)
S4—C4—C9	122.4 (3)	S3—C10—C11	122.1 (3)
S3—C10—C15	120.6 (2)	C1′—N1′—O1′	117.7 (4)
C1′—N1′—O2′	118.7 (5)	O1′—N1′—O2′	123.7 (5)

Data collection: *P*3 *Data Collection Program* (Nicolet, 1985). Cell refinement: *P*3 *Data Collection Program*. Data reduction, structure solution, structure refinement, molecular graphics and preparation of material for publication: *SHELXTL* (Sheldrick, 1984).

This work has been supported by a grant for the Key Research Project in Climbing Program from the state Science and Technology Commission of China.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1133). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Cassoux, P. & Valade, L. (1991). *Inorganic Materials*, edited by D. W. Bruce & D. O. Hare, pp. 1–57. London: John Wiley.

Fang, Q., Min-Hua, J., Zheng, Q., Hong, L., Wen-Tao, Y. & Zhuang, Z. (1994). *J. Mater. Chem.* **4**, 1041–1045.

Fang, Q., Zheng, Q., Yao, Y. & Xiao-Zeng, Y. (1992). *Chin. Chem. Lett.* pp. 795–796.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Nicolet (1985). *P3 Data Collection Program*. Version 81.3. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Sheldrick, G. M. (1984). *SHELXTL User's Manual*. Revision 4.1. Nicolet XRD Corporation, Madison, Wisconsin, USA.

Williams, J. M., Wang, H. H., Emge, T. J., Geiser, U., Beno, M. A., Leung, P. C. W., Carlson, K. D., Thorn, R. J. & Schultz, A. J. (1987). *Prog. Inorg. Chem.* **35**, 51–218.

Acta Cryst. (1995). C**51**, 1604–1606

An Epoxyamide

William Clegg,* Massimo Ranelli and
Richard F. W. Jackson

*Department of Chemistry, University of Newcastle,
Newcastle upon Tyne NE1 7RU, England*

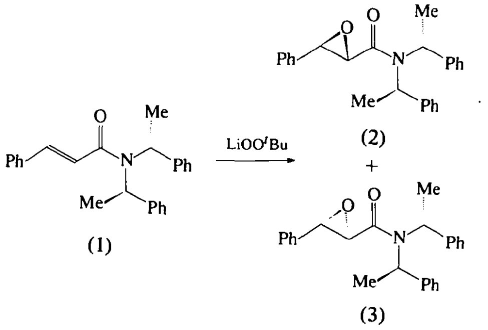
(*Received* 18 *November* 1994; *accepted* 25 *January* 1995)

Abstract

The title compound, (2*S*,3*R*)-3-phenyl-*N*,*N*-bis[(*R*)-1-phenylethyl]oxirane-2-carboxamide, $C_{25}H_{25}NO_2$, contains an oxirane ring with phenyl and amide substituents. These have no observable influence on the geometry of the ring which has two insignificantly different C—O bond lengths of 1.423 (3) and 1.425 (3) Å. A survey of previously published oxirane structures indicates that the lack of electronic influence on the ring geometry by carbonyl substituents is common.

Comment

Nucleophilic epoxidation of unsaturated amides can be carried out using lithium *tert*-butyl peroxide (Meth-Cohn, Moore & Taljaard, 1988). In an effort to prepare enantiomerically enriched epoxy amides, an investigation of the nucleophilic epoxidation of the chiral enantiomerically pure crotonamide derivative (1) was carried out.



Epoxidation of (1) gave the diastereoisomeric epoxides (2) and (3) (73% yield) in a ratio of 66:34 as judged by ^{1}H NMR (200 MHz). Crystallization of the diastereoisomeric mixture by vapour diffusion of petrol (333–353 K boiling range) into an isopropyl acetate solution yielded pure crystals of the major diastereoisomer (2) (confirmed by NMR). Because of the relatively low diastereoselectivity of the epoxidation process, no further synthetic work has been undertaken.

Although the enantiopole refinement (Flack, 1983) indicates a preference for the opposite absolute configuration, this is of marginal significance, with only very weak anomalous scattering effects. The correct configuration was assumed from the known configuration of the amine starting material.

The geometry of the cyclopropane ring shows considerable variation which can be correlated with the electronic properties of the substituents on the ring (Allen, 1980). For the ring atom with the most strongly electron-accepting substituents, the distal ring bond is shortened and the vicinal bonds are lengthened. We were both surprised and disappointed that in a survey of oxirane structures in the Cambridge Structural Database (Allen, Davies, Galloy, Johnson, Kennard, Macrae, Mitchell, Mitchell, Smith & Watson, 1991) there was a much less marked correlation in the case of the ring C—C and C—O bonds of these molecules (Clegg & Kelly, 1994). In particular, for around 150 well determined structures in which the oxirane is not part of a polycyclic system, there is no clear pattern for the C—O bond lengths, except in the case of a small number of structures containing very strongly electron-withdrawing substituents such as cyano, nitro or sulfonyl, for which the near C—O bond is shortened and the far C—O and C—C bonds are lengthened slightly. The title compound shows no significant difference between the two ring C—O bonds, which is in common with most of the 25 carbonyl-substituted oxirane structures located in the database.

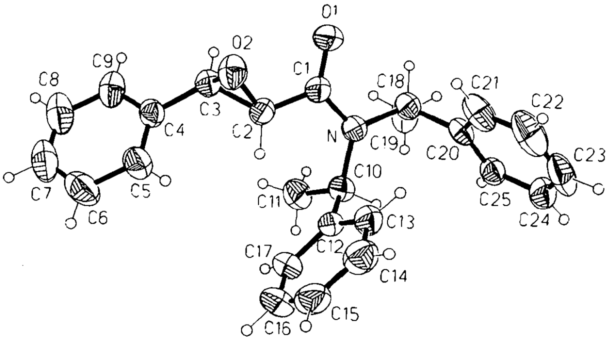


Fig. 1. The structure of the title compound with atom labels and 40% probability ellipsoids.

Experimental

The crotonamide was prepared by treatment of cinnamoyl chloride with (R,R')-α,α'-dimethyldibenzylamine (Eleveld, Hogeveen & Schudde, 1986), in the presence of sodium hydroxide, in a two-phase water/dichloromethane mixture. The epoxidation reaction was carried out at room temperature according to the general literature method of Meth-Cohn, Moore & Taljaard (1988).

Crystal data

$C_{25}H_{25}NO_2$
$M_r = 371.46$
Orthorhombic
$P2_12_12_1$
$a = 9.8537$ (10) Å
$b = 14.2586$ (13) Å
$c = 14.7465$ (14) Å
$V = 2071.9$ (3) Å^3
$Z = 4$
$D_x = 1.191$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 32 reflections
$\theta = 15.94$–$19.75°$
$\mu = 0.588$ mm^{-1}
$T = 295$ (2) K
Block
0.68 × 0.56 × 0.36 mm
Colourless

Data collection

Stoe Siemens diffractometer
ω/θ scans with on-line profile fitting (Clegg, 1981)
Absorption correction: none
5505 measured reflections
3366 independent reflections
3150 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0374$
$\theta_{\rm max} = 65.08°$
$h = -11 \rightarrow 11$
$k = -16 \rightarrow 16$
$l = -15 \rightarrow 17$
3 standard reflections
frequency: 60 min
intensity decay: 2%

Refinement

Refinement on F^2
$R(F) = 0.0480$
$wR(F^2) = 0.1510$
$S = 1.040$
3365 reflections
256 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0891P)^2 + 0.3409P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.169$ e Å^{-3}
$\Delta\rho_{\min} = -0.159$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0035 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	0.0588 (2)	0.73081 (12)	0.24290 (15)	0.0761 (6)
O2	0.3111 (2)	0.65183 (12)	0.25508 (12)	0.0654 (5)
N	0.1321 (2)	0.87578 (12)	0.28131 (13)	0.0520 (4)
C1	0.1518 (2)	0.7852 (2)	0.2578 (2)	0.0527 (5)
C2	0.2958 (2)	0.7509 (2)	0.2492 (2)	0.0537 (5)
C3	0.3387 (2)	0.6964 (2)	0.1706 (2)	0.0500 (5)
C4	0.4798 (2)	0.6937 (2)	0.1363 (2)	0.0546 (5)
C5	0.5896 (3)	0.7126 (2)	0.1905 (2)	0.0744 (8)
C6	0.7222 (3)	0.7060 (2)	0.1542 (3)	0.0961 (11)
C7	0.7368 (4)	0.6800 (2)	0.0641 (3)	0.0981 (12)
C8	0.6305 (4)	0.6613 (3)	0.0120 (3)	0.0928 (10)
C9	0.5017 (3)	0.6679 (2)	0.0469 (2)	0.0720 (7)
C10	0.2402 (2)	0.9477 (2)	0.2907 (2)	0.0541 (5)
C11	0.3036 (3)	0.9709 (2)	0.1992 (2)	0.0727 (7)
C12	0.3428 (2)	0.92557 (15)	0.3654 (2)	0.0532 (5)
C13	0.3002 (3)	0.8939 (2)	0.4490 (2)	0.0629 (6)
C14	0.3918 (3)	0.8761 (2)	0.5180 (2)	0.0760 (8)
C15	0.5280 (3)	0.8885 (2)	0.5047 (2)	0.0818 (9)
C16	0.5716 (3)	0.9205 (3)	0.4230 (3)	0.0892 (10)
C17	0.4812 (3)	0.9396 (2)	0.3537 (2)	0.0720 (7)
C18	−0.0110 (2)	0.9093 (2)	0.2873 (2)	0.0593 (6)
C19	−0.0443 (3)	0.9798 (3)	0.2122 (2)	0.0896 (10)
C20	−0.0425 (2)	0.9432 (2)	0.3824 (2)	0.0501 (5)
C21	−0.0590 (3)	0.8770 (2)	0.4495 (2)	0.0775 (8)
C22	−0.0886 (3)	0.8999 (4)	0.5355 (3)	0.0999 (13)
C23	−0.1042 (3)	0.9910 (4)	0.5587 (2)	0.1002 (14)
C24	−0.0886 (3)	1.0617 (3)	0.4937 (2)	0.0808 (9)
C25	−0.0584 (2)	1.0366 (2)	0.4038 (2)	0.0563 (5)

Table 2. *Selected geometric parameters* (Å, °)

O1—C1	1.220 (3)	C10—C12	1.527 (3)
O2—C2	1.423 (3)	C12—C13	1.378 (4)
O2—C3	1.425 (3)	C12—C17	1.390 (4)
N—C1	1.351 (3)	C13—C14	1.383 (4)
N—C10	1.486 (3)	C14—C15	1.367 (5)
N—C18	1.491 (3)	C15—C16	1.359 (5)
C1—C2	1.507 (3)	C16—C17	1.383 (5)
C2—C3	1.458 (3)	C18—C20	1.516 (3)
C3—C4	1.479 (3)	C18—C19	1.531 (4)
C4—C5	1.372 (4)	C20—C21	1.377 (3)
C4—C9	1.386 (4)	C20—C25	1.377 (3)
C5—C6	1.415 (4)	C21—C22	1.343 (6)
C6—C7	1.387 (6)	C22—C23	1.352 (6)
C7—C8	1.326 (6)	C23—C24	1.400 (6)
C8—C9	1.374 (5)	C24—C25	1.405 (4)
C10—C11	1.524 (4)		
C2—O2—C3	61.60 (15)	N—C10—C12	113.5 (2)
C1—N—C10	125.5 (2)	C11—C10—C12	114.4 (2)
C1—N—C18	117.2 (2)	C13—C12—C17	117.2 (2)
C10—N—C18	116.9 (2)	C13—C12—C10	120.8 (2)
O1—C1—N	123.0 (2)	C17—C12—C10	122.0 (2)
O1—C1—C2	119.1 (2)	C12—C13—C14	121.3 (3)
N—C1—C2	117.9 (2)	C15—C14—C13	120.8 (3)
O2—C2—C3	59.26 (14)	C16—C15—C14	118.7 (3)
O2—C2—C1	114.6 (2)	C15—C16—C17	121.2 (3)
C3—C2—C1	120.9 (2)	C16—C17—C12	120.8 (3)
O2—C3—C2	59.14 (15)	N—C18—C20	110.5 (2)
O2—C3—C4	117.8 (2)	N—C18—C19	111.7 (2)
C2—C3—C4	123.9 (2)	C20—C18—C19	114.5 (2)
C5—C4—C9	118.9 (3)	C21—C20—C25	119.0 (3)
C5—C4—C3	122.5 (2)	C21—C20—C18	118.0 (2)
C9—C4—C3	118.6 (2)	C25—C20—C18	123.0 (2)
C4—C5—C6	119.7 (3)	C22—C21—C20	122.6 (4)
C7—C6—C5	118.4 (3)	C21—C22—C23	119.8 (3)
C8—C7—C6	121.8 (3)	C22—C23—C24	120.5 (3)
C7—C8—C9	119.9 (4)	C23—C24—C25	119.0 (3)
C8—C9—C4	121.3 (3)	C20—C25—C24	119.2 (3)
N—C10—C11	111.2 (2)		

Fixed C—H bond lengths and ideal tetrahedral angles were imposed on the methyl groups, which were free to rotate about the C—C bonds during the refinement, contributing one extra parameter per methyl group. Isotropic H atoms were refined with a riding model and $U(\text{H}) = 1.2U_{eq}(\text{C})$.

Data collection: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: local programs. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL*93 and local programs.

We thank EPSRC for financial support, Mr David Waldram (Sterling Organics, Fawdon) for supplying the sample of (R,R')-α,α'-dimethyldibenzylamine and for some preliminary experiments, and Professor O. Meth-Cohn (Univ. of Sunderland) for helpful discussions.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1114). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H. (1980). *Acta Cryst.* B**36**, 81–96.

Allen, F. H., Davies, J. E., Galloy, J. J., Johnson, O., Kennard, O., Macrae, C. F., Mitchell, E. M., Mitchell, G. F., Smith, J. M. & Watson, D. G. (1991). *J. Chem. Inf. Comput. Sci.* **31**, 187–204.

Clegg, W. (1981). *Acta Cryst.* A**37**, 22–28.

Clegg, W. & Kelly, J. (1994). Unpublished results.

Eleveld, M. B., Hogeveen, H. & Schudde, E. P. (1986). *J. Org. Chem.* **51**, 3635–3642.

Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.

Meth-Cohn, O., Moore, C. & Taljaard, H. C. (1988). *J. Chem. Soc. Perkin Trans.* 1, pp. 2663–2674.

Sheldrick, G. M. (1990). *SHELXTL/PC User's Manual.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* Univ. of Göttingen, Germany.

Stoe & Cie (1988). *DIF4. Diffractometer Control Program.* Version 7.04. Stoe & Cie, Darmstadt, Germany.