$V = 754.7\ (2)\ \text{Å}^3$
$Z = 4$
$D_x = 1.920\ \text{Mg m}^{-3}$

0.20 × 0.15 × 0.12 mm
Colorless

Data collection

Siemens *P*3/PC diffractometer
$2\theta/\theta$ scans
Absorption correction: refined from ΔF
$T_{\min} = 0.25,\ T_{\max} = 0.60$
1411 measured reflections
1305 independent reflections
776 observed reflections $[I > 2\sigma(I)]$

$\theta_{\max} = 25°$
$h = -8 \rightarrow 8$
$k = 0 \rightarrow 12$
$l = 0 \rightarrow 11$
3 standard reflections monitored every 50 reflections intensity decay: <3%

Refinement

Refinement on F
$R = 0.05$
$wR = 0.06$
$S = 1.61$
776 reflections
101 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.001600F^2]$
$(\Delta/\sigma)_{\max} = 0.0363$

$\Delta\rho_{\max} = 0.599\ \text{e Å}^{-3}$
$\Delta\rho_{\min} = -0.779\ \text{e Å}^{-3}$
Extinction correction: Zachariasen (1963)
Extinction coefficient: $3\ (2) \times 10^{-7}$
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)

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

	x	y	z	U_{eq}
Br(1)	0.8976 (2)	0.3368 (1)	0.0503 (1)	0.048 (1)
O(1)	1.1555 (11)	0.3081 (7)	0.3156 (8)	0.036 (3)
C(2)	1.0458 (15)	0.3953 (12)	0.2292 (12)	0.038 (4)
C(3)	1.0637 (20)	0.5048 (15)	0.2852 (13)	0.054 (5)
C(4)	1.1973 (19)	0.4903 (11)	0.4237 (14)	0.048 (5)
C(5)	1.2476 (17)	0.3711 (11)	0.4402 (11)	0.035 (4)
C(6)	1.3718 (17)	0.3032 (13)	0.5598 (12)	0.041 (5)
C(7)	1.4091 (18)	0.1860 (10)	0.5643 (12)	0.037 (4)
N(1)	1.5281 (16)	0.1284 (12)	0.6882 (12)	0.050 (4)
O(2)	1.6078 (18)	0.1950 (12)	0.7934 (10)	0.077 (5)
O(3)	1.5475 (17)	0.0175 (11)	0.6897 (10)	0.074 (5)

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

Br(1)—C(2)	1.87 (1)	O(1)—C(2)	1.36 (1)
O(1)—C(5)	1.39 (1)	C(2)—C(3)	1.30 (2)
C(3)—C(4)	1.42 (2)	C(4)—C(5)	1.35 (2)
C(5)—C(6)	1.45 (2)	C(6)—C(7)	1.30 (2)
C(7)—N(1)	1.41 (2)	N(1)—O(2)	1.25 (2)
N(1)—O(3)	1.22 (2)		
H(4)···O(2)	2.56 (1)	H(6)···O(3)	2.46 (1)
C(2)—O(1)—C(5)	103.7 (9)	Br(1)—C(2)—O(1)	114.1 (9)
Br(1)—C(2)—C(3)	131.5 (9)	O(1)—C(2)—C(3)	114.4 (9)
C(2)—C(3)—C(4)	104.7 (12)	C(3)—C(4)—C(5)	107.7 (11)
O(1)—C(5)—C(4)	109.4 (9)	O(1)—C(5)—C(6)	118.5 (10)
C(4)—C(5)—C(6)	132.1 (11)	C(5)—C(6)—C(7)	126.9 (11)
C(6)—C(7)—N(1)	122.2 (10)	C(7)—N(1)—O(2)	117.5 (11)
C(7)—N(1)—O(3)	119.5 (10)	O(2)—N(1)—O(3)	123.0 (11)

Data collection: *XSCANS* (Siemens, 1992). Cell refinement: *SHELXTL-Plus* (Sheldrick, 1991) and *SHELXL*93 (Sheldrick, 1993). Data reduction, structure solution (direct methods) and software used to prepare material for publication: *SHELXTL-Plus*. All computations were carried out with an IBM 486 DX computer.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1140). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Adducts from the Pyrolysis of Cellulose: (1α,2β,6β,7α)-3,8-Dioxatricyclo[5.3.1.1^{2,6}]-dodeca-4,9-diene-11,12-dione and (1*R*)-(1α,2β,3α,6α,8β,9α)-5,13,14-Trioxatetracyclo[7.3.1.1^{3,6}.0^{2,8}]tetradec-10-ene-7,12-dione

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Abstract

The compounds (1α,2β,6β,7α)-3,8-dioxatricyclo-[5.3.1.1^{2,6}]dodeca-4,9-diene-11,12-dione, $C_{10}H_8O_4$, (9), and (1*R*)-(1α,2β,3α,6α,8β,9α)-5,13,14-trioxatetracyclo-[7.3.1.1^{3,6}.0^{2,8}]tetradeca-10-ene-7,12-dione, $C_{11}H_{10}O_5$, (17), were obtained during studies of the products of

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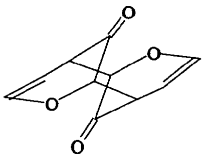
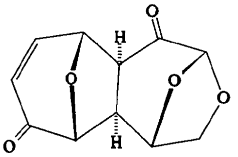
phosphoric acid-catalysed pyrolysis of cellulose. Compound (9) consists of centrosymmetric molecules, each half consisting of a 3,4-dihydro-2*H*-pyran-3-one ring in a flattened boat conformation, the halves being linked together at the 2,4 positions; the central fused ring has a chair conformation, consistent with it being crystallographically centrosymmetric. The second molecule, (17), is tetracyclic with a 6,5,6,5-membered fused ring pattern; the five-membered rings adopt envelope and twist conformations, while the six-membered rings are constrained through bonding requirements to flattened conformations. The 5,6-dihydro-2*H*-pyran-5-one ring can be described as a half boat.

Comment

The study of the pyrolysis products of cellulose has been motivated by their potential as a chemical feedstock. In the presence of inorganic acids, pyrolytic dehydration, rearrangement and charring reactions are substantially accelerated. The two title compounds, (9) and (17), were isolated from the high boiling tar fractions, in order to identify them and to determine the nature of their condensation reactions (Furneaux, Mason & Miller, 1984).



(9)



(17)

The structure of ($1\alpha,2\beta,6\beta,7\alpha$)-3,8-dioxatricyclo-[$5.3.1.1^{2,6}$]dodeca-4,9-diene-11,12-dione, (9), consists of the independent centrosymmetric tricyclic molecules shown in Fig. 1. The 3,4-dihydro-2*H*-pyran-3-one rings each adopt the same flattened half-boat conformation, as indicated in Table 5, with atoms C(2) and C(1) lying 0.04 (1) and −0.67 (1) Å from the plane of O(3), C(4), C(5) and C(6), respectively. Through the imposed crystallographic centre of symmetry, the central cyclohexadione adopts a chair conformation with C(1) and C(1a) 0.70 (1) Å from the plane of C(2), C(2a), C(6) and C(6a).

The structure of (1*R*)-($1\alpha,2\beta,3\alpha,6\alpha,8\beta,9\alpha$)-5,13,14-trioxatetracyclo[$7.3.1.1^{3,6}.0^{2,8}$]tetradeca-10-ene-7,12-dione, (17), consists of independent tetracyclic molecules, as shown in Fig. 2. The diagram illustrates the conformations adopted by each of the fused rings. Considering those in order from the left: the dioxolan has close to a pure envelope conformation (puckering θ 188°; Cremer & Pople, 1975) with the O(14) flap atom 0.59 (2) Å out of the plane; the tetrahydropyran-3-one is in a partially flattened chair conformation, consistent with the position of the C(7)=O(15) double bond and cyclic constraints, with a mean out-of-plane distance for C(2), C(3), C(6) and C(7) of 0.02 (2) Å, C(8) and O(14) being 0.19 (2) and −0.84 (2) Å, respectively, out of this plane; the tetrahydrofuran [C(1), C(2), C(8), C(9) and O(13)] is in a twist conformation (θ 164.6°; Table 5); and finally the 5,6-dihydro-2*H*-pyran-5-one is in a half-boat conformation, resulting from the coplanarity of atoms C(9), C(10), C(11), C(12), O(16) and C(1) [mean out-of-plane deviation 0.03 (2) Å], with O(13) being 0.81 (2) Å out of this plane. For this last ring, the θ,φ puckering angles are 54.3 and 6.6°, respectively (Cremer & Pople, 1975).

Only van der Waals interactions between the molecules are noted in both structures with closest contacts being H(4)···H(5) 2.62 (4) and O(16)···H(3) 2.50 (8) Å in (9) and (17), respectively. The pyranoid and dioxolan rings are similar to those reported for isoxazolines derived from levoglucosenone (Blake, Cook, Forsyth, Gould & Paton, 1992). No anomalies are found in the bond lengths and angles.

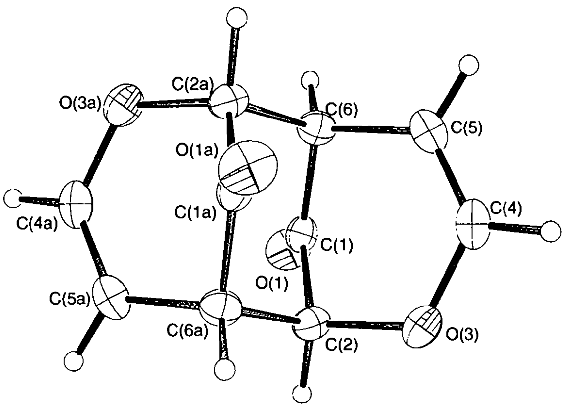


Fig. 1. An *ORTEP*II (Johnson, 1971) drawing of (9) with 30% probability ellipsoids for non-H atoms; atoms related by the centre of symmetry at (0,1,0) are labelled with an 'a'.

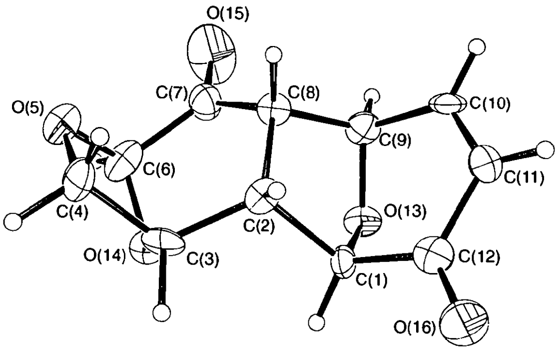


Fig. 2. An *ORTEP*II (Johnson, 1971) drawing of (17) with 30% probability ellipsoids for non-H atoms.

Experimental

Both compounds (9) and (17) were obtained by the methods of Furneaux, Mason & Miller (1984).

Compound (9)

Crystal data

$C_{10}H_8O_4$
$M_r = 192.2$
Monoclinic
$P2_1/c$
$a = 6.4574\,(12)$ Å
$b = 11.1198\,(24)$ Å
$c = 6.2652\,(11)$ Å
$\beta = 114.382\,(14)°$
$V = 409.75\,(14)$ Å^3
$Z = 2$
$D_x = 1.560$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 3.5$–$15.0°$
$\mu = 0.132$ mm^{-1}
$T = 291\,(2)$ K
Needle
0.235 × 0.175 × 0.075 mm
Colourless

Data collection

Nicolet *R*3*m* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
870 measured reflections
725 independent reflections
509 observed reflections $[F > 4\sigma(F)]$
$R_{\rm int} = 0.021$

$\theta_{\rm max} = 25.02°$
$h = -7 \rightarrow 7$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 7$
3 standard reflections monitored every 97 reflections intensity decay: 2%

Refinement

Refinement on F^2
$R(F) = 0.041$
$wR(F^2) = 0.100$
$S = 1.038$
725 reflections
81 parameters
All H-atom parameters refined isotropically
$w = 1/[\sigma^2(F_o^2) + 0.0423P^2 + 0.11P]$ where $P = [\max(F_o^2,0) + 2F_c^2]$

$(\Delta/\sigma)_{\rm max} = 0.012$
$\Delta\rho_{\rm max} = 0.177$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.145$ e Å^{-3}
Extinction correction: pseudo-secondary (*SHELXL*93; Sheldrick, 1993)
Extinction coefficient: 0.080 (13)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (9)

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

	x	y	z	$U_{\rm eq}$
C(1)	0.1131 (3)	0.8844 (2)	0.0364 (4)	0.033 (1)
O(1)	0.2691 (3)	0.8151 (2)	0.1216 (3)	0.051 (1)
C(2)	0.0680 (4)	0.9563 (2)	−0.1849 (4)	0.036 (1)
O(3)	−0.1474 (3)	0.9217 (2)	−0.3698 (2)	0.047 (1)
C(4)	−0.3121 (4)	0.8865 (2)	−0.2975 (5)	0.044 (1)
C(5)	−0.2880 (4)	0.8783 (2)	−0.0801 (4)	0.043 (1)
C(6)	−0.0660 (4)	0.9091 (2)	0.1255 (4)	0.037 (1)

Table 2. *Selected geometric parameters* (Å, °) *for* (9)

C(1)—O(1)	1.204 (2)	O(3)—C(4)	1.374 (3)
C(1)—C(6)	1.503 (3)	C(4)—C(5)	1.309 (3)
C(1)—C(2)	1.520 (3)	C(5)—C(6)	1.519 (3)
C(2)—O(3)	1.447 (3)	C(6)—C(2$^{\rm i}$)	1.544 (3)
C(2)—C(6$^{\rm i}$)	1.544 (3)		
O(1)—C(1)—C(6)	125.7 (2)	C(4)—O(3)—C(2)	115.5 (2)
O(1)—C(1)—C(2)	123.4 (2)	C(5)—C(4)—O(3)	126.0 (2)
C(6)—C(1)—C(2)	110.8 (2)	C(4)—C(5)—C(6)	122.1 (2)
O(3)—C(2)—C(1)	110.5 (2)	C(1)—C(6)—C(5)	104.2 (2)
O(3)—C(2)—C(6$^{\rm i}$)	110.5 (2)	C(1)—C(6)—C(2$^{\rm i}$)	110.5 (2)
C(1)—C(2)—C(6$^{\rm i}$)	107.9 (2)	C(5)—C(6)—C(2$^{\rm i}$)	108.9 (2)

Symmetry code: (i) $-x, 2-y, -z$.

Compound (17)

Crystal data

$C_{11}H_{10}O_5$
$M_r = 222.2$
Orthorhombic
$P2_12_12_1$
$a = 5.154\,(2)$ Å
$b = 12.257\,(8)$ Å
$c = 16.034\,(8)$ Å
$V = 1013.1\,(9)$ Å^3
$Z = 4$
$D_x = 1.457$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 19 reflections
$\theta = 14.6$–$16.4°$
$\mu = 0.125$ mm^{-1}
$T = 291\,(2)$ K
Needle
0.30 × 0.09 × 0.08 mm
Colourless

Data collection

Nicolet *R*3*m* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
509 measured reflections
478 independent reflections
333 observed reflections $[F > 4\sigma(F)]$

$\theta_{\rm max} = 18.5°$
$h = 0 \rightarrow 4$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 14$
3 standard reflections monitored every 97 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R(F) = 0.0461$
$wR(F^2) = 0.0621$
$S = 1.17$
478 reflections
167 parameters
$w = 1/[\sigma^2(F_o^2) + 0.089P^2]$ where $P = [\max(F_o^2,0) + 2F_c^2]/3$

$(\Delta/\sigma)_{\rm max} = 0.036$
$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.15$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 3. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2) *for* (17)

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

	x	y	z	$U_{\rm eq}$
C(1)	0.719 (2)	0.0706 (12)	0.0086 (6)	0.039 (3)
C(2)	0.908 (2)	0.0597 (8)	−0.0689 (8)	0.035 (3)
C(3)	0.768 (3)	0.0784 (10)	−0.1524 (8)	0.044 (4)
C(4)	0.943 (3)	0.0697 (16)	−0.2278 (9)	0.071 (5)
O(5)	0.945 (2)	−0.0446 (7)	−0.2478 (5)	0.066 (3)
C(6)	0.763 (3)	−0.0913 (11)	−0.1917 (7)	0.058 (4)
C(7)	0.912 (3)	−0.1394 (10)	−0.1178 (7)	0.052 (4)
C(8)	1.017 (2)	−0.0579 (9)	−0.0587 (7)	0.034 (3)
C(9)	0.928 (3)	−0.0872 (10)	0.0290 (8)	0.042 (3)
C(10)	1.099 (2)	−0.0357 (12)	0.0944 (7)	0.045 (3)
C(11)	1.061 (2)	0.0662 (12)	0.1182 (6)	0.055 (4)
C(12)	0.858 (2)	0.1289 (11)	0.0772 (8)	0.045 (3)
O(13)	0.677 (2)	−0.0402 (7)	0.0359 (4)	0.043 (2)
O(14)	0.592 (1)	−0.0116 (8)	−0.1648 (4)	0.056 (2)
O(15)	0.948 (2)	−0.2351 (7)	−0.1119 (5)	0.113 (4)
O(16)	0.816 (2)	0.2260 (6)	0.0949 (5)	0.067 (3)

Table 4. *Selected geometric parameters* (Å, °) *for* (17)

C(1)—O(13)	1.44 (1)	C(6)—C(7)	1.53 (1)
C(1)—C(12)	1.50 (2)	C(7)—O(15)	1.19 (1)
C(1)—C(2)	1.59 (1)	C(7)—C(8)	1.48 (1)
C(2)—C(3)	1.54 (2)	C(8)—C(9)	1.52 (1)
C(2)—C(8)	1.56 (1)	C(9)—O(13)	1.42 (1)
C(3)—O(14)	1.44 (1)	C(9)—C(10)	1.51 (1)
C(3)—C(4)	1.51 (2)	C(10)—C(11)	1.32 (1)
C(4)—O(5)	1.44 (2)	C(11)—C(12)	1.46 (1)
O(5)—C(6)	1.42 (1)	C(12)—O(16)	1.24 (1)
C(6)—O(14)	1.38 (1)		
O(13)—C(1)—C(12)	107.4 (9)	O(15)—C(7)—C(6)	121.3 (12)
O(13)—C(1)—C(2)	104.4 (9)	C(8)—C(7)—C(6)	114.7 (10)
C(12)—C(1)—C(2)	108.8 (10)	C(7)—C(8)—C(9)	108.8 (9)
C(3)—C(2)—C(8)	113.5 (9)	C(7)—C(8)—C(2)	115.2 (9)
C(3)—C(2)—C(1)	112.5 (9)	C(9)—C(8)—C(2)	102.0 (9)
C(8)—C(2)—C(1)	102.6 (9)	O(13)—C(9)—C(10)	108.0 (9)
O(14)—C(3)—C(4)	102.2 (10)	O(13)—C(9)—C(8)	104.4 (9)
O(14)—C(3)—C(2)	107.4 (9)	C(10)—C(9)—C(8)	111.6 (10)
C(4)—C(3)—C(2)	113.9 (10)	C(11)—C(10)—C(9)	120.8 (12)
O(5)—C(4)—C(3)	104.4 (12)	C(10)—C(11)—C(12)	118.3 (12)
C(6)—O(5)—C(4)	104.4 (10)	O(16)—C(12)—C(11)	121.9 (13)
O(14)—C(6)—O(5)	109.5 (10)	O(16)—C(12)—C(1)	122.8 (12)
O(14)—C(6)—C(7)	110.5 (9)	C(11)—C(12)—C(1)	115.2 (10)
O(5)—C(6)—C(7)	108.3 (11)	C(9)—O(13)—C(1)	102.9 (8)
O(15)—C(7)—C(8)	123.8 (12)	C(6)—O(14)—C(3)	100.6 (8)

Table 5. *Puckering parameters*

Compound	Ring Atoms*	Q (Å)	θ (°)	φ (°)
(9)	1,2,3,4,5,6	0.513	51.9	1.8
(9)	1,2,6a,1a,2a,6	0.610	0.0	0.0
(17)	14,3,4,5,6	0.397	–	188.0
(17)	14,6,7,8,2,3	0.623	136.1	175.9
(17)	13,1,2,8,9	0.447	–	164.6
(17)	13,1,12,11,10,9	0.610	54.3	6.6

* In the order of calculation (Cremer & Pople, 1975).

Preliminary photographs and the last collected shell of data indicated no significant data observable beyond $\theta = 18.5°$ for compound (17).

All H atoms were refined with isotropic displacement parameters; in (17), one common factor [final value 0.031 (11) $Å^2$] was used. All other atoms were refined with anisotropic displacement parameters. In (17), H(6), H(10) and H(11) were treated as riding atoms on C(6), C(10) and C(11), respectively, with C—H = 0.98 Å.

Programs used were *SHELXS*86 (Sheldrick, 1985) for direct-methods structure solution, *SHELXL*93 (Sheldrick, 1993) for refinement and *ORTEP*II (Johnson, 1971) for diagrams.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KH1010). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(2*S*,4*R*,5*R*)-(−)-3-Isopropyl-4-methyl-5-phenyl-1,2,3-oxathiazolidine 2-Oxide

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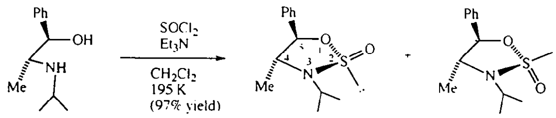
Abstract

The present structure determination, coupled with the known configuration of the starting amino alcohol, confirms the absolute configuration at the S atom of the title compound, $C_{12}H_{17}NO_2S$, and its 2*R* diastereoisomer. The five-membered oxathiazolidine 2-oxide ring has an envelope conformation with atom N3 deviating by 0.598 (3) Å from the least-squares plane formed by atoms O1, S2, C4 and C5. The bond lengths S2—O6 and S2—N3 are respectively shorter than the bond lengths S═O and S—N of sulfinamides.

Comment

In studies of the synthesis of chiral sulfoxides utilizing the displacement reactions of 1,2,3-oxathiazolidine 2-oxides with organometallic reagents (Benson & Snyder, 1991; Wudl & Lee, 1973), the title compound (1) and its 2*R* diastereoisomer (2) were prepared from the coupling reaction of (1*R*,2*R*)-(−)-2-(*N*-isopropylamino)-1-phenylpropanol, (3), thionyl chloride and triethylamine in methylene chloride.



(3) (1) (2)

The reaction provided a 97% yield of (1) and (2) in a ratio of 3:1. Fortunately, compound (1) is a white solid while (2) is an oil, so that (1) was easily isolated

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