C14	−0.0681 (1)	0.4513 (1)	0.4815 (7)	0.0589
Cl	−0.11653 (2)	0.33708 (2)	−0.4435 (1)	0.0540
O(1*w*)	0.0472 (1)	0.2169 (1)	0.7526 (8)	0.1568
O(2*w*)	0	1/4	0.1250	0.2004

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

S—C7	1.672 (3)	N4—C7	1.330 (3)
N2—N3	1.363 (4)	N4—C8	1.429 (3)
N2—C6	1.284 (3)	C5—C6	1.474 (4)
N3—C7	1.376 (3)	C6—C14	1.497 (4)
N3—N2—C6	120.8 (2)	C5—C6—C14	118.4 (3)
N2—N3—C7	119.4 (2)	S—C7—N3	119.4 (2)
C7—N4—C8	127.7 (2)	S—C7—N4	125.5 (2)
N1—C5—C6	118.0 (2)	N3—C7—N4	115.1 (2)
C4—C5—C6	125.0 (3)	N4—C8—C9	121.9 (3)
N2—C6—C5	114.7 (2)	N4—C8—C13	117.3 (3)
N2—C6—C14	126.9 (3)		

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*···*A*	*D*—H···*A*
N4—H···Cl[i]	3.199 (3)	144 (3)
N1—H···Cl[i]	3.023 (3)	150 (3)
N3—H···S[ii]	3.584 (3)	175 (2)

Symmetry codes: (i) $y - \frac{1}{4}, \frac{1}{4} - x, \frac{1}{4} - z$; (ii) $-x, 1 - y, z$.

Intensities were corrected for Lorentz and polarization effects. The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990). The structure refinement by full-matrix least-squares on *F* was carried out using *SHELX*76 (Sheldrick, 1976). All H atoms, except those of water molecules, were located by difference Fourier maps and refined isotropically. Non-H atoms were refined with anisotropic displacement parameters.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1047). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2029–2032

(2*R*)-3-[(4*S*)-4-Benzyl-2-oxo-3-oxazolidinyl]-3-oxo-2-[(1*R*,2*S*)-2-vinylcyclohexyl]propionic Acid Methyl Ester and (2*R*)-3-[(4*S*)-4-Benzyl-2-oxo-3-oxazolidinyl]-3-oxo-2-[(1*R*,2*S*)-2-vinylcyclopentyl]propionic Acid Methyl Ester

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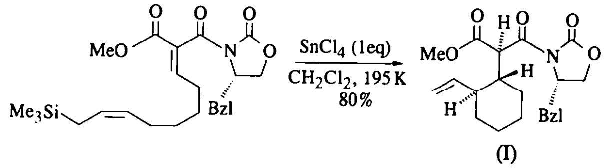
Abstract

Both the title structures, $C_{22}H_{27}NO_5$ and $C_{21}H_{25}NO_5$, exhibit similar conformations, as shown by a least-squares fit of the atoms common to both. The oxazolidine ring is intermediate between envelope and twist forms, with a slight dominance of the envelope in the former structure but the twist in the latter. Part of the oxazolidine ring of the former structure, however, shows high displacement parameters.

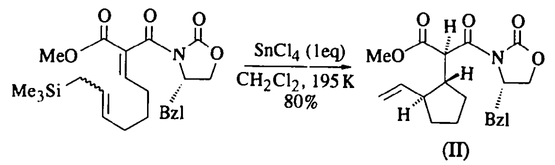
Comment

This work forms part of our studies on the synthesis of enantiopure *trans*-1,2-disubstituted cyclopentanes and cyclohexanes. These compounds are of special interest because of their frequent appearance as components of natural product molecules. They were easily obtained *via* an intramolecular allysilane addition of chiral alkylidene-1,3-dicarbonyl compounds. Further details of the reaction have been published elsewhere (Tietze & Schünke, 1995).

Both compounds (Figs. 1 and 2) have similar conformations. Fig. 3 shows a least-squares fit of both mol-



(I)



(II)

ecules in which the r.m.s deviation is 0.069 Å for the identical part of the structure. All bond lengths and angles are in the expected ranges. The five-membered oxazolidinone ring in (I) adopts a 63% envelope conformation with the flap at C5 and a 37% twist conformation with the axis through C2 and N3 pointing down. The ring puckering parameters are $q2$ = 0.121 Å and $\varphi2$ = 317.3°. In (II) the ring adopts a 60% twist conformation with its axis through C2 and N3 pointing down and a 40% envelope with the flap at C5 pointing down. The ring puckering parameters are $q2$ = 0.180 Å and $\varphi2$ = 313.2°. The hexane ring in (I) consists of a 98% chair conformation with C1″ pointing down. The puckering parameters are $q2$ = 0.018 Å, $\varphi2$ = 129.2° and $q3$ = −0.580 Å. The pentane ring in (II) adopts a 64% twist conformation with its axis through C4″ and C5″ pointing up and a 36% envelope conformation with flap C2″ pointing up. The ring parameters are $q2$ = 0.445 Å and $\varphi2$ = 204.4°.

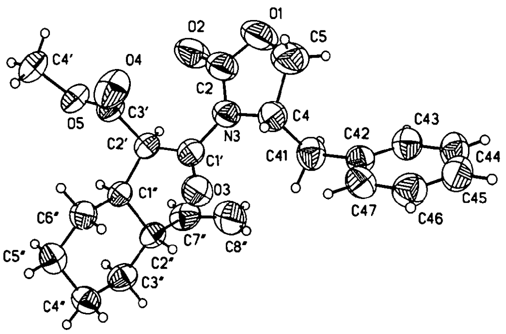


Fig. 1. Crystal structure of (I) showing 50% probability displacement ellipsoids.

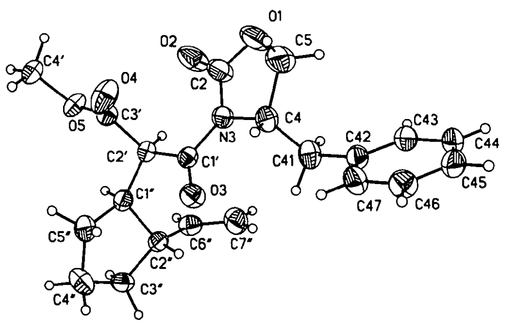


Fig. 2. Crystal structure of (II) showing 50% probability displacement ellipsoids.

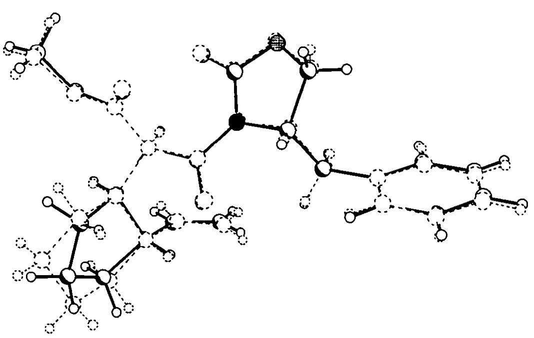


Fig. 3. Least-squares fit of (I) and (II). All non-H atoms are fitted except the substituted hexane or pentane rings at C2′.

Experimental

Both compounds were crystallized from a mixture of diethyl ether/petroleum ether at room temperature.

Compound (I)

Crystal data

$C_{22}H_{27}NO_5$	Cu $K\alpha$ radiation
M_r = 385.45	λ = 1.54178 Å
Orthorhombic	Cell parameters from 75 reflections
$P2_12_12_1$	θ = 10.5–28°
a = 9.3870 (4) Å	μ = 0.719 mm^{-1}
b = 10.0616 (4) Å	T = 293 (2) K
c = 21.7222 (12) Å	Block
V = 2051.6 (2) Å^3	0.5 × 0.4 × 0.4 mm
Z = 4	Colourless
D_x = 1.248 Mg m^{-3}	
D_m not measured	

Data collection

Siemens $P4$ four-circle diffractometer	R_{int} = 0.0504
Profile from $\theta/2\theta$ scans	θ_{max} = 56.74°
Absorption correction: none	h = −10 → 10
5345 measured reflections	k = −10 → 10
2732 independent reflections	l = −23 → 23
2428 observed reflections [$I > 2\sigma(I)$]	3 standard reflections monitored every 100 reflections intensity decay: none

Refinement

Refinement on F^2	Extinction correction: *SHELXL*93 (Sheldrick, 1993)
$R[F^2 > 2\sigma(F^2)]$ = 0.0429	Extinction coefficient: 0.0060 (6)
$wR(F^2)$ = 0.1122	Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
S = 1.044	Absolute configuration: Flack (1983)
2731 reflections	Flack parameter = −0.0 (3)
255 parameters	
H atoms: see text	
$w = 1/[\sigma^2(F_o^2) + (0.0715P)^2 + 0.0545P]$ where $P = (F_o^2 + 2F_c^2)/3$	
$(\Delta/\sigma)_{max} < 0.001$	
$\Delta\rho_{max}$ = 0.163 e Å^{-3}	
$\Delta\rho_{min}$ = −0.129 e Å^{-3}	

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

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

	x	y	z	U_{eq}
O1	0.1978 (3)	0.7417 (2)	−0.01621 (9)	0.1119 (10)
C2	0.1819 (4)	0.7641 (3)	0.04366 (13)	0.0783 (9)
O2	0.1849 (3)	0.8742 (2)	0.06487 (9)	0.0938 (8)
N3	0.1598 (3)	0.6447 (2)	0.07385 (9)	0.0609 (6)

C4	0.1575 (3)	0.5323 (3)	0.03013 (12)	0.0653 (7)
C5	0.2048 (5)	0.6018 (4)	−0.02872 (16)	0.1141 (15)
C1′	0.1648 (3)	0.6227 (3)	0.13689 (11)	0.0521 (6)
O3	0.1636 (2)	0.50857 (18)	0.15593 (7)	0.0674 (5)
C2′	0.1688 (2)	0.7435 (2)	0.17896 (10)	0.0472 (5)
C3′	0.3205 (3)	0.7971 (2)	0.17789 (11)	0.0548 (6)
O4	0.4202 (2)	0.7443 (3)	0.15422 (10)	0.0879 (7)
O5	0.32850 (18)	0.90879 (16)	0.21004 (8)	0.0636 (5)
C4′	0.4693 (3)	0.9637 (3)	0.21937 (15)	0.0805 (9)
C1″	0.1210 (2)	0.7199 (2)	0.24614 (10)	0.0456 (6)
C2″	−0.0241 (2)	0.6504 (2)	0.25277 (11)	0.0501 (6)
C3″	−0.0662 (3)	0.6465 (3)	0.32098 (12)	0.0625 (7)
C4″	0.0473 (3)	0.5819 (3)	0.36134 (12)	0.0658 (7)
C5″	0.1889 (3)	0.6514 (3)	0.35343 (12)	0.0676 (7)
C6″	0.2328 (3)	0.6518 (3)	0.28605 (11)	0.0565 (6)
C7″	−0.1377 (3)	0.7147 (3)	0.21544 (14)	0.0693 (8)
C8″	−0.2104 (3)	0.6592 (5)	0.17124 (15)	0.0978 (11)
C41	0.0107 (3)	0.4694 (3)	0.02552 (13)	0.0666 (7)
C42	0.0096 (3)	0.3510 (3)	−0.01727 (11)	0.0558 (6)
C43	−0.0728 (3)	0.3487 (3)	−0.07049 (12)	0.0635 (7)
C44	−0.0743 (3)	0.2382 (3)	−0.10800 (13)	0.0718 (8)
C45	0.0039 (3)	0.1287 (3)	−0.09330 (13)	0.0702 (8)
C46	0.0852 (3)	0.1287 (3)	−0.04138 (13)	0.0708 (8)
C47	0.0881 (3)	0.2395 (3)	−0.00370 (12)	0.0662 (7)

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

O1—C2	1.328 (3)	C1′—C2′	1.521 (3)
O1—C5	1.434 (4)	C2′—C3′	1.523 (3)
C2—O2	1.201 (4)	C2′—C1″	1.545 (3)
C2—N3	1.385 (4)	C3′—O4	1.192 (3)
N3—C1′	1.388 (3)	C3′—O5	1.325 (3)
N3—C4	1.477 (3)	O5—C4′	1.447 (3)
C4—C5	1.523 (4)	C7″—C8″	1.304 (4)
C1′—O3	1.221 (3)		
C2—O1—C5	110.9 (2)	O3—C1′—N3	118.9 (2)
O2—C2—O1	122.0 (3)	O3—C1′—C2′	123.3 (2)
O2—C2—N3	128.5 (2)	N3—C1′—C2′	117.8 (2)
O1—C2—N3	109.5 (3)	C1′—C2′—C3′	107.30 (19)
C2—N3—C1′	126.9 (2)	C1′—C2′—C1″	115.94 (19)
C2—N3—C4	111.2 (2)	C3′—C2′—C1″	109.91 (19)
C1′—N3—C4	120.9 (2)	O4—C3′—O5	124.1 (2)
N3—C4—C41	112.0 (2)	O4—C3′—C2′	125.6 (2)
N3—C4—C5	100.6 (2)	O5—C3′—C2′	110.2 (2)
C41—C4—C5	113.6 (3)	C3′—O5—C4′	116.7 (2)
O1—C5—C4	106.1 (2)		

Compound (II)

Crystal data

$C_{21}H_{25}NO_5$
M_r = 371.42
Orthorhombic
$P2_12_12_1$
a = 9.272 (5) Å
b = 9.699 (2) Å
c = 21.210 (6) Å
V = 1907.4 (12) Å^3
Z = 4
D_x = 1.293 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 52 reflections
θ = 10–12.5°
μ = 0.092 mm^{-1}
T = 153 (2) K
Block
0.60 × 0.40 × 0.30 mm
Colourless

Data collection

Stoe–Siemens AED four-circle diffractometer
Profile from $2\theta/\omega$ scans
Absorption correction: none
5584 measured reflections
3361 independent reflections
3029 observed reflections [$I > 2\sigma(I)$]

R_{int} = 0.0298
θ_{max} = 25.06°
$h = -11 \rightarrow 11$
$k = -11 \rightarrow 11$
$l = -25 \rightarrow 25$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0415$
$wR(F^2) = 0.1072$
S = 1.079
3361 reflections
246 parameters
H atoms: see text
$w = 1/[\sigma^2(F_o^2) + (0.0441P)^2 + 0.7871P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.385 e Å^{-3}
$\Delta\rho_{min}$ = −0.171 e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0082 (14)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983)
Flack parameter = 0.2 (13)

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

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

	x	y	z	U_{eq}
O1	0.7064 (3)	0.2677 (2)	0.51474 (8)	0.0710 (7)
C2	0.6852 (3)	0.2412 (3)	0.45347 (11)	0.0486 (7)
O2	0.6858 (3)	0.12669 (19)	0.43275 (8)	0.0695 (7)
N3	0.6630 (2)	0.36350 (19)	0.42199 (8)	0.0352 (4)
C4	0.6603 (3)	0.4814 (3)	0.46587 (10)	0.0392 (6)
C41	0.5091 (3)	0.5401 (3)	0.47352 (12)	0.0415 (6)
C42	0.5074 (3)	0.6602 (2)	0.51839 (11)	0.0351 (5)
C43	0.4302 (3)	0.6558 (3)	0.57450 (10)	0.0373 (5)
C44	0.4281 (3)	0.7668 (3)	0.61499 (11)	0.0413 (6)
C45	0.5041 (3)	0.8846 (3)	0.60090 (12)	0.0432 (6)
C46	0.5821 (3)	0.8910 (3)	0.54542 (12)	0.0446 (6)
C47	0.5830 (3)	0.7805 (3)	0.50484 (11)	0.0425 (6)
C5	0.7215 (4)	0.4134 (3)	0.52448 (14)	0.0665 (9)
C1′	0.6604 (2)	0.3849 (2)	0.35738 (10)	0.0295 (5)
O3	0.6553 (2)	0.50087 (16)	0.33758 (7)	0.0413 (4)
C2′	0.6655 (2)	0.2586 (2)	0.31526 (9)	0.0280 (4)
C3′	0.8193 (2)	0.2066 (2)	0.31444 (10)	0.0312 (5)
O4	0.9195 (2)	0.2662 (2)	0.33699 (10)	0.0573 (5)
O5	0.82959 (17)	0.08895 (15)	0.28349 (8)	0.0377 (4)
C4′	0.9733 (3)	0.0343 (3)	0.27628 (12)	0.0424 (6)
C1″	0.6083 (2)	0.2786 (2)	0.24813 (10)	0.0337 (5)
C2″	0.4607 (2)	0.3481 (2)	0.24220 (10)	0.0329 (5)
C3″	0.4460 (3)	0.3561 (3)	0.17033 (10)	0.0374 (5)
C4″	0.5963 (3)	0.3931 (3)	0.14740 (12)	0.0497 (7)
C5″	0.6998 (3)	0.3566 (3)	0.20136 (11)	0.0423 (6)
C6″	0.3439 (3)	0.2723 (3)	0.27517 (11)	0.0399 (6)
C7″	0.2742 (3)	0.3187 (3)	0.32447 (13)	0.0540 (7)

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

O1—C2	1.339 (3)	C1′—O3	1.202 (3)
O1—C5	1.435 (4)	C1′—C2′	1.516 (3)
C2—O2	1.195 (3)	C2′—C3′	1.513 (3)
C2—N3	1.376 (3)	C3′—O4	1.194 (3)
N3—C1′	1.386 (3)	C3′—O5	1.320 (3)
N3—C4	1.475 (3)	O5—C4′	1.442 (3)
C4—C5	1.518 (4)		
C2—O1—C5	110.0 (2)	O3—C1′—N3	119.08 (19)
O2—C2—O1	122.3 (2)	O3—C1′—C2′	123.45 (19)
O2—C2—N3	128.6 (2)	N3—C1′—C2′	117.47 (18)
O1—C2—N3	109.1 (2)	C3′—C2′—C1′	107.77 (17)
C2—N3—C1′	127.66 (19)	C3′—C2′—C1″	110.94 (17)
C2—N3—C4	111.38 (18)	C1′—C2′—C1″	115.79 (18)
C1′—N3—C4	120.51 (18)	O4—C3′—O5	124.2 (2)
N3—C4—C5	99.99 (19)	O4—C3′—C2′	124.6 (2)
N3—C4—C41	111.88 (19)	O5—C3′—C2′	111.22 (18)
C5—C4—C41	114.8 (2)	C3′—O5—C4′	115.91 (18)
O1—C5—C4	105.9 (2)		

The structures were solved by direct methods using *SHELXS*86 (Sheldrick, 1990). All non-H atoms were refined anisotropically using *SHELXL*93 (Sheldrick, 1993). H atoms were refined using a riding model. The isotropic displacement parameters were set to 1.2 times (1.5 times for methyl groups) the equivalent displacement parameter of the atom they are attached to. The absolute structure were known for both compounds. For (I) the absolute structure parameter was consistent with this information [−0.0(3): Flack, 1983; Bernardinelli & Flack, 1985]. Compound (II) was measured with Mo radiation, and therefore the absolute structure could not be determined.

Atoms O1 and C5 of the oxazolidinone ring in (I) seemed to be disordered because of relatively high displacement parameters. Similar observations were made in (II) for this part of the structure, but with much lower values for the anisotropic displacement parameters. In consideration of the different temperatures for both data collections, we decided to interpret the high values for the anisotropic displacement parameters in (I) as thermal vibration instead of disorder.

All calculations were performed using the program *PUCKER* (Gould & Taylor, 1994; Cremer & Pople, 1975).

Data collection: *XSCANS* (Siemens, 1994) for (I); *DIF*4 (Stoe & Cie, 1988*a*) for (II). Cell refinement: *XSCANS* for (I); *DIF*4 for (II). Data reduction: *XSCANS* for (I); *REDU*4 (Stoe & Cie, 1988*b*) for (II). For both compounds, program(s) used to solve structures: *SHELXS*86 (Sheldrick, 1990); program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1994); software used to prepare material for publication: *SHELXL*93.

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1097). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N-[1-(2-Benzo[*b*]thienyl)ethyl]-*N*′-carbamoylurea and 1-[1-(2-Benzo[*b*]-thienyl)ethyl][1,3,5]triazine-2,4,6-trione Methanol Solvate

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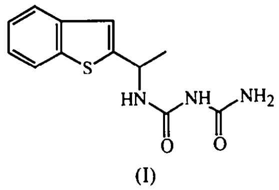
Abstract

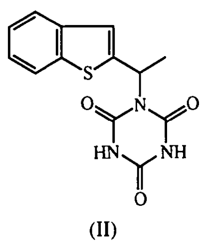
The title compounds, $C_{12}H_{13}N_3O_2S$ and $C_{13}H_{11}N_3O_3S.$-CH_4O, were prepared by treating the precursor urea, [1-(2-benzo[*b*]thienyl)ethyl]urea, with *N*-chlorocarbonyl isocyanate. The products were isolated by preparative thin-layer chromatography. Analytical samples were obtained by crystallization from methanol and the crystal structures of both compounds were determined.

Comment

Leukotrienes have been linked to inflammatory disease and the pivotal enzyme responsible for initiating leukotriene biosynthesis is 5-lipoxygenase (Samuelsson, 1983). Zileuton, *N*-[1-(2-benzo[*b*]thienyl)ethyl]-*N*-hydroxyurea, is a selective inhibitor of 5-lipoxygenase currently undergoing clinical trials (Carter *et al.*, 1991; Israel *et al.*, 1993). During the course of the zileuton development program, many syntheses were investigated, some of which are documented (Hsiao & Kolasa, 1992). These synthetic efforts provided a large number of novel methodology-specific intermediates and by-products. As a consequence of our need to identify and monitor zileuton process-related entities, we synthesized and fully characterized the biuret title compound, *N*-[1-(2-benzo-[*b*]thienyl)ethyl]-*N*′-carbamoylurea, (I). This report describes the preparation, isolation and X-ray structure determination of compound (I) as well as the triazinetrione



(I)



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