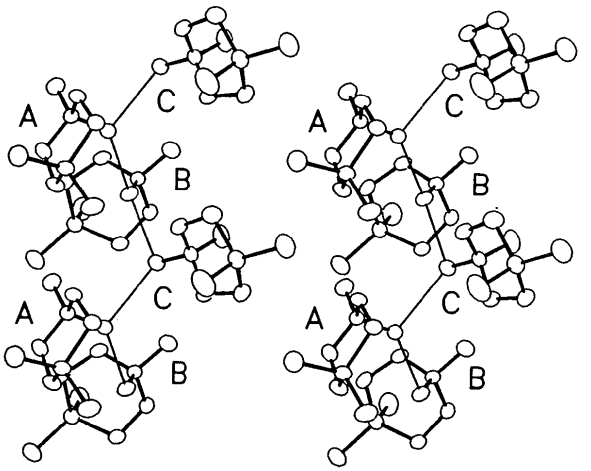

Fig. 2. Stereoview of the hydrogen-bonded structure viewed normal to the *b* axis.

were obtained on the product which were wholly consistent with the assigned structure.

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Structure of Galphimine B

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Abstract. (4*R*)-Trihydroxy-13α-methoxycarbonyl-30-nor-3,4-seco-7α,18β-fridela-1,20-dien-3,24-olide methylene chloride solvate, $C_{30}H_{44}O_7.0.898CH_2Cl_2$, $M_r = 601.6$, orthorhombic, $P2_12_12_1$, $a = 9.797$ (4), $b = 15.039$ (7), $c = 20.135$ (8) Å, $V = 2966.5$ (8) Å^3, $Z = 4$, $D_x = 1.347$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 23.66$ cm^{-1}, $F(000) = 1288$, $T = 298$ K, $R = 0.062$ for 2004 reflections with $F > 3\sigma(F)$. In the pentacyclic molecule, rings *C* and *D* adopt a chair conformation while ring *E* shows a half-chair conformation (ring junctions: *C*/*D*, *trans*; *D*/*E*, *cis*). The seven-membered ring *A* and six-membered ring *B* are considerably distorted (twist and monoplanar conformations, respectively) which is reflected in the pseudo-*trans* *A*/*B* ring junction [torsion angles −11.3 (3) and 1.3 (3)°] Intramolecular O(3)—H(3)···O(4) [*D*—H 1.04 (6), H···*A* 1.72 (6), *D*···*A* 2.720 (8) Å, *D*—H···*A* 160 (1)°] and intermolecular O(4)—H(4*a*)···O(3) ($x - 0.5$, $1.5 - y$, $2 - z$) [*D*—H 0.77 (7), H···*A* 2.26 (7), *D*···*A* 3.005 (8) Å, *D*—H···*A* 165 (1)°] hydrogen bonds stabilize the molecules in the crystal.

Experimental. The title compound was isolated from aerial parts of *Galphymia glauca* (*Cav.*) Kuntze. Crystallization from methanol–methylene chloride yielded a colorless single crystal of dimensions 0.26 × 0.30 × 0.38 mm. The unit-cell parameters were refined from least-squares analysis of 2θ values of 25 reflections from $7.35 < 2\theta < 23.93°$. Intensities for 2183 reflections (2152 unique, $R_{int} = 0.029$) having $3 < 2\theta < 110°$ and $0 < h < 10$, $0 < k < 15$, $0 < l < 21$, were measured on a Nicolet *P3F* diffractometer

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Table 1. *Atomic coordinates* ($\times 10^4$) *and isotropic or equivalent isotropic temperature factors* ($Å^2 \times 10^3$) *for galphimine B*

Atoms of the statistically disordered solvent molecules were refined isotropically with standard occupancy factors of 0.24 (1) for Cl(1)*a*, 0.737 (9) for Cl(1)*b* and 0.898 (9) for Cl(2) and C(32). Other non-H atoms were refined anisotropically, for which $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

	x	*y*	*z*	U_{eq}
C(1)	−2502 (6)	4192 (3)	9750 (3)	50 (2)
C(2)	−3233 (6)	3485 (4)	9907 (3)	58 (2)
C(3)	−4719 (7)	3451 (5)	10030 (4)	68 (2)
O(1)	−5319 (4)	4166 (3)	10298 (2)	65 (1)
O(2)	−5417 (6)	2826 (3)	9879 (3)	99 (2)
C(4)	−3127 (6)	6158 (4)	10691 (3)	51 (2)
O(3)	−2447 (4)	6875 (2)	10348 (2)	53 (1)
C(5)	−3912 (5)	5523 (3)	10206 (3)	44 (2)
C(6)	−5114 (6)	6024 (4)	9886 (3)	51 (2)
C(7)	−4778 (6)	6668 (3)	9320 (3)	47 (2)
O(4)	−4519 (5)	7535 (3)	9596 (2)	55 (1)
C(8)	−3555 (5)	6402 (4)	8905 (3)	43 (2)
C(9)	−3305 (5)	5374 (4)	8915 (3)	42 (2)
C(10)	−2908 (6)	5149 (3)	9660 (3)	43 (2)
C(11)	−2046 (6)	5146 (4)	8499 (2)	45 (2)
C(12)	−1976 (6)	5591 (4)	7808 (3)	50 (2)
C(13)	−2051 (5)	6608 (3)	7880 (3)	43 (2)
C(14)	−3493 (5)	6829 (4)	8190 (3)	45 (2)
C(15)	−3696 (6)	7852 (4)	8226 (3)	55 (2)
C(16)	−3515 (7)	8300 (4)	7561 (3)	66 (2)
C(17)	−2156 (6)	8125 (4)	7224 (3)	57 (2)
C(18)	−1800 (6)	7109 (4)	7194 (3)	46 (2)
O(5)	−2690 (5)	6670 (3)	6744 (2)	72 (2)
C(19)	−356 (6)	6999 (4)	6908 (3)	55 (2)
C(20)	703 (7)	7650 (4)	7137 (3)	56 (2)
C(21)	402 (7)	8392 (4)	7450 (3)	61 (2)
C(22)	−1038 (7)	8668 (4)	7611 (3)	60 (2)
C(23)	−1994 (7)	5717 (4)	11088 (3)	62 (2)
C(24)	−4500 (6)	4812 (4)	10641 (3)	54 (2)
C(25)	−4498 (6)	4793 (4)	8673 (3)	58 (2)
C(26)	−4701 (6)	6483 (5)	7764 (3)	64 (2)
C(27)	−868 (5)	6870 (3)	8344 (2)	38 (2)
C(28)	−2211 (9)	8527 (5)	6518 (3)	80 (3)
C(29)	2127 (7)	7403 (5)	7004 (4)	78 (3)
O(6)	249 (4)	6382 (2)	8215 (2)	47 (1)
O(7)	−869 (4)	7400 (2)	8792 (2)	49 (1)
C(31)	1425 (6)	6556 (5)	8628 (3)	64 (2)
Cl(1)*a*	5657 (23)	951 (13)	8920 (10)	185 (10)
Cl(1)*b*	6643 (4)	238 (3)	8692 (2)	118 (2)
Cl(2)	6169 (3)	83 (3)	10092 (1)	124 (1)
C(32)	6387 (11)	856 (7)	9475 (6)	110 (4)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1.320 (8)	C(1)—C(10)	1.503 (7)
C(2)—C(3)	1.478 (9)	C(3)—O(1)	1.338 (8)
C(3)—O(2)	1.202 (9)	O(1)—C(24)	1.437 (7)
C(4)—O(3)	1.443 (7)	C(4)—C(5)	1.568 (8)
C(4)—C(23)	1.520 (9)	C(5)—C(6)	1.539 (8)
C(5)—C(10)	1.578 (7)	C(5)—C(24)	1.498 (8)
C(6)—C(7)	1.530 (8)	C(7)—O(4)	1.440 (6)
C(7)—C(8)	1.515 (8)	C(8)—C(9)	1.565 (8)
C(8)—C(14)	1.577 (7)	C(9)—C(10)	1.587 (7)
C(9)—C(11)	1.530 (7)	C(9)—C(25)	1.538 (8)
C(11)—C(12)	1.545 (7)	C(12)—C(13)	1.538 (7)
C(13)—C(14)	1.580 (7)	C(13)—C(18)	1.593 (7)
C(13)—C(27)	1.540 (7)	C(14)—C(15)	1.553 (8)
C(14)—C(26)	1.552 (8)	C(15)—C(16)	1.509 (8)
C(16)—C(17)	1.517 (9)	C(17)—C(18)	1.568 (9)
C(17)—C(22)	1.572 (9)	C(17)—C(28)	1.546 (9)
C(18)—O(5)	1.420 (7)	C(18)—C(19)	1.537 (8)
C(19)—C(20)	1.500 (9)	C(20)—C(21)	1.315 (9)
C(20)—C(29)	1.468 (9)	C(21)—C(22)	1.506 (9)
C(27)—O(6)	1.342 (6)	C(27)—O(7)	1.203 (6)
O(6)—C(31)	1.444 (7)	Cl(1)*a*—Cl(1)*b*	1.51 (2)
Cl(1)*a*—C(32)	1.33 (2)	Cl(1)*b*—C(32)	1.85 (1)
Cl(2)—C(32)	1.71 (1)		

C(2)—C(1)—C(10)	131.0 (6)	C(1)—C(2)—C(3)	127.1 (6)
C(2)—C(3)—O(1)	118.2 (6)	C(2)—C(3)—O(2)	123.0 (6)
O(1)—C(3)—O(2)	118.7 (6)	C(3)—O(1)—C(24)	119.4 (5)
O(3)—C(4)—C(5)	112.5 (4)	O(3)—C(4)—C(23)	103.9 (5)
C(5)—C(4)—C(23)	114.9 (5)	C(4)—C(5)—C(6)	109.8 (4)
C(4)—C(5)—C(10)	110.2 (4)	C(6)—C(5)—C(10)	111.1 (4)
C(4)—C(5)—C(24)	105.0 (4)	C(6)—C(5)—C(24)	107.5 (4)
C(10)—C(5)—C(24)	113.1 (4)	C(5)—C(6)—C(7)	117.2 (5)
C(6)—C(7)—O(4)	108.9 (4)	C(6)—C(7)—C(8)	114.4 (4)
O(4)—C(7)—C(8)	108.2 (4)	C(7)—C(8)—C(9)	112.2 (4)
C(7)—C(8)—C(14)	115.3 (4)	C(9)—C(8)—C(14)	114.1 (4)
C(8)—C(9)—C(10)	105.1 (4)	C(8)—C(9)—C(11)	109.9 (4)
C(10)—C(9)—C(11)	105.8 (4)	C(8)—C(9)—C(25)	116.0 (4)
C(10)—C(9)—C(25)	111.3 (4)	C(11)—C(9)—C(25)	108.2 (4)
C(1)—C(10)—C(5)	115.0 (4)	C(1)—C(10)—C(9)	112.5 (4)
C(5)—C(10)—C(9)	115.4 (4)	C(9)—C(11)—C(12)	115.6 (4)
C(11)—C(12)—C(13)	110.0 (4)	C(12)—C(13)—C(14)	106.8 (4)
C(12)—C(13)—C(18)	112.4 (4)	C(14)—C(13)—C(18)	112.3 (4)
C(12)—C(13)—C(27)	106.1 (4)	C(14)—C(13)—C(27)	112.3 (4)
C(18)—C(13)—C(27)	106.8 (4)	C(8)—C(14)—C(13)	108.0 (4)
C(8)—C)14)—C(15)	110.9 (4)	C(13)—C(14)—C(15)	110.0 (4)
C(8)—C(14)—C(26)	109.8 (4)	C(13)—C(14)—C(26)	113.2 (4)
C(15)—C(14)—C(26)	105.1 (5)	C(14)—C(15)—C(16)	112.7 (5)
C(15)—C(16)—C(17)	115.0 (5)	C(16)—C(17)—C(18)	112.4 (5)
C(16)—C(17)—C(22)	107.4 (5)	C(18)—C(17)—C(22)	111.7 (5)
C(16)—C(17)—C(28)	108.2 (5)	C(18)—C(17)—C(28)	110.7 (5)
C(22)—C(17)—C(28)	106.0 (5)	C(13)—C(18)—C(17)	113.2 (4)
C(13)—C(18)—O(5)	103.9 (4)	C(17)—C(18)—O(5)	109.9 (5)
C(13)—C(18)—C(19)	114.6 (4)	C(17)—C(18)—C(19)	108.9 (5)
O(5)—C(18)—C(19)	106.0 (4)	C(18)—C(19)—C(20)	116.8 (5)
C(19)—C(20)—C(21)	123.1 (6)	C(19)—C(20)—C(29)	115.9 (5)
C(21)—C(20)—C(29)	121.0 (6)	C(20)—C(21)—C(22)	123.1 (6)
C(17)—C(22)—C(21)	113.8 (5)	O(1)—C(24)—C(5)	114.6 (5)
C(13)—C(27)—O(6)	110.9 (4)	C(13)—C(27)—O(7)	128.5 (5)
O(6)—C(27)—O(7)	120.5 (5)	C(27)—O(6)—C(31)	116.1 (4)
Cl(1)*b*—Cl(1)*a*—C(32)	80.6 (12)	Cl(1)*a*—Cl(1)*b*—C(32)	45.4 (9)
Cl(1)*a*—C(32)—Cl(1)*b*	53.9 (10)	Cl(1)*a*—C(32)—Cl(2)	127.8 (11)
Cl(1)*b*—C(32)—Cl(2)	107.1 (5)		

using θ–2θ scans of variable speed 3–30° min^{-1} in 2θ, with Ni-filtered Cu radiation. Two standard reflections (021, $\bar{1}\bar{1}0$) were measured every 50 reflections and varied in intensity by < 1% during data collection.

Direct methods using *SHELXTL* (Sheldrick, 1983) revealed positions of all non-H atoms. The positional and anisotropic displacement parameters of all non-H atoms were refined by block-matrix least squares on F using the 2004 reflections for which $F > 3\sigma(F)$. The H-atom positions were located in a difference map; those bonded to heteroatoms were refined, while those bonded to C atoms were forced to ride on them with fixed isotropic temperature parameters $U = 0.06$ Å^2. Occupancies of atoms on statistically disordered solvent molecules were refined. Complex atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol IV). Final $R = 0.062$, $wR = 0.072$ $\{w = 1/[\sigma^2(F) + 0.002F^2]\}$, $(\Delta/\sigma)_{max} = 0.18$. The final difference map showed maximum positive and negative peaks of $\Delta\rho = 0.40$ and -0.31 e Å^{-3}. An isotropic extinction parameter refined to $\chi = 0.003$ (1) $[F_c^* = F_c(1.0 + 0.002\chi F_c^2/\sin 2\theta)^{-0.25}]$, but an absorption correction was not applied. Parallel and independent calculations were carried out on the two stereoisomers of the molecule and anomalous-dispersion corrections were applied to both.

After four cycles of block-matrix refinement (381 parameters, 2004 reflections) convergence was reached with one configuration giving $R = 0.067$ and $wR = 0.078$, whereas the other gave $R = 0.062$ and $wR = 0.072$. A statistical test on the wR-factor ratio (Hamilton, 1965) indicated that the former stereo-

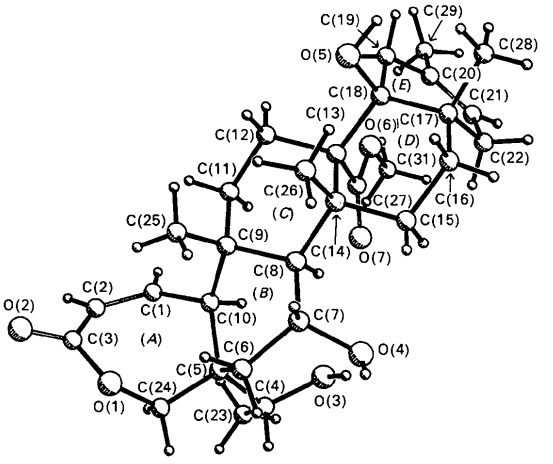

Fig. 1. Perspective view of galphimine B.

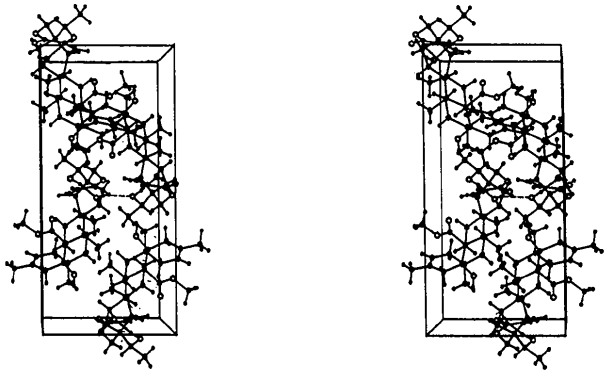

Fig. 2. Stereoscopic view of a unit cell of galphimine B viewed along the *b* axis.

isomer could be rejected at the 0.005 significance level [$\mathcal{R}_{(3,1623,0.005)} = 1.004$]. Accordingly, all coordinates reported herein refer to the statistically favored configuration: the 4*R*-enantiomer. In addition to the statistical disorder of the methylene chloride solvent molecule, an orientational disorder was also observed and modeled by splitting the position of one Cl atom [Cl(1)] into two major positions.

Atomic parameters are listed in Table 1. Distances and angles are listed in Table 2.* A perspective view of the molecule showing the atomic numbering and the stereochemistry is given in Fig. 1. Fig 2 shows the molecular packing.

Related literature. A galphimine B closely related *nor, seco*-triterpenoid has been isolated from *Lophanthera lactescens* (Dos S., Braz, Gottlieb & Shoolery, 1990).

* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55659 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0595]

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Structure of a Coupled Carbohydrate and Terpene

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Abstract. (1*S*-{1α,1[3*R**,5*R**(*R**)],2α,5β})-5-[1,2-Bis(benzyloxy)ethyl]-4,5-dihydro-3-[1-hydroxy-2-methyl-5-(1-methylvinyl)cyclohexyl]-2(3*H*)-furanone, $C_{30}H_{34}O_7$, $M_r = 506.3$, monoclinic, $P2_1$, $a =$ 12.780 (1), $b = 6.478$ (1), $c = 16.832$ (2) Å, $\beta =$ 93.00 (1)°, $V = 1391.6$ (3) Å^3, $Z = 2$, $D_x =$ 1.209 g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.9$ cm^{-1}, $F(000) = 540$, $T = 298$ K, $R = 0.0504$ and $wR =$ 0.0584 for 2177 reflections [$I \geq 2\sigma(I)$]. Crystal chirality was assigned on the basis of the two components coupled to form the title compound. The carbohydrate lactone, L-gulonic-γ-lactone, was utilized as a precursor to the tetrahydrofuran ring and the dibenzoate appendage. In a similar manner, the terpene ketone (+)-dihydrocarvone was the precursor to the cyclohexanol ring. The absolute stereoche-

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