with isotropic temperature factors. The absolute structure was determined with final refinements of the structure with Rogers' η value (Rogers, 1981) which gave $\eta = 1{\cdot}76\,(1)$ for the final positions that appear in Table 1.* At convergence $R = 3{\cdot}10\%$, $wR = 3{\cdot}20\%$, $w = [\sigma^2(F) + 0{\cdot}00025F^2]^{-1}$, $\sigma^2(F)$ based on counting statistics, $(\Delta/\sigma)_{\max} = 0{\cdot}033$. GOF = 1·53, $(\Delta\rho)_{\max} = 0{\cdot}15$, $(\Delta\rho)_{\min} = -0{\cdot}14$ e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a MicroVAX II computer system using the *SHELXTL-PLUS* programs.

Atomic positions and thermal parameters are listed in Table 1, bond lengths and angles in Table 2. A stereoscopic view of the molecular structure of $C_{19}H_{24}O_2S$ is depicted in Fig. 1.

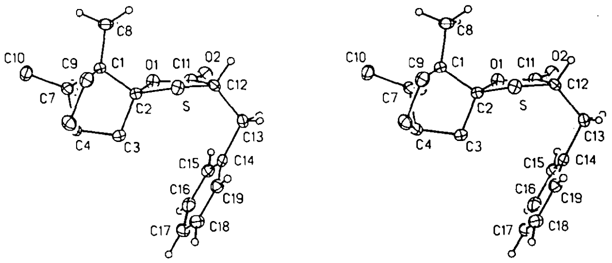

Fig. 1. A stereoscopic view of the molecular structure of $C_{19}H_{24}O_2S$.

Related literature. The observed configuration of the 1′,3′-oxathiolan-5′-one ring (the lactone ring with the S atom at the tip of the envelope form) is in agreement with what had been suggested from NMR studies by Pihlaja, Nikkila, Neuvonen & Keskinen (1976).

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52568 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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1,3,5-Triallyl-4,6-diphenyl-1,3,5-triazacyclohexan-2-one

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Abstract. $C_{24}H_{27}N_3O$, $M_r = 373{\cdot}5$, monoclinic, $P2_1/n$, $a = 9{\cdot}315\,(3)$, $b = 14{\cdot}674\,(6)$, $c = 15{\cdot}855\,(6)$ Å, $\beta = 98{\cdot}44\,(3)°$, $V = 2144\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}16$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1{\cdot}54178$ Å, $\mu = 5{\cdot}7$ cm^{-1}, $F(000) = 800$, $T = 291$ K, $R = 0{\cdot}062$ for 2900 observed reflections. X-ray analysis was undertaken to establish the exact nature of cycloaddition reaction product and its unambiguous stereochemical configuration. The presence of an exocyclic double bond at C2 forces the triazacyclohexane ring to adopt an envelope conformation, with N5 on the flap and a mirror plane through C2 and N5. The symmetry of the central ring is not retained by the phenyl substituents: C14—C19 in equatorial position and C23—C28 in axial position. Two of the N atoms (N1 and N3) are slightly pyramidal, as shown by the distances from the planes defined by the three covalently bonded C atoms: 0·05 and 0·19 Å respectively. The pyramidal character of N5 is well established, with a corresponding distance of 0·40 Å.

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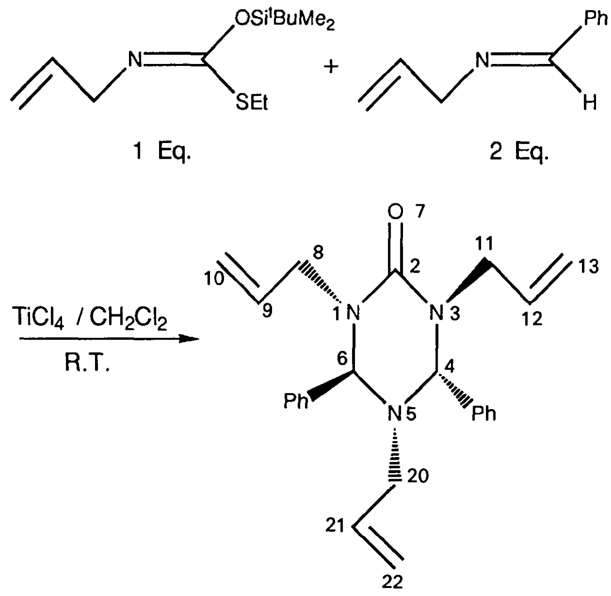
Experimental. The title compound was obtained in the course of studies on cycloaddition reactions. A new one-step sequence that provides a triazacyclohexane with a good overall yield is illustrated in the scheme.



Parallelepiped crystal with dimensions 0·2 × 0·3 × 0·4 mm. Lattice parameters refined using 16 reflections in the range $11 \leq 2\theta \leq 23°$. Huber diffractometer, graphite-monochromated Cu $K\alpha$ radiation. 3847 $hk \pm l$ independent reflections with $\sin\theta/\lambda \leq 0{\cdot}6$ Å^{-1}; $-11 \leq h \leq 11$, $0 \leq k \leq 17$, $0 \leq l \leq 18$, 2900 with $I \geq 2{\cdot}5\sigma(I)$. Standard reflection (012) checked every 50 reflections: no significant deviation. Structure solved by direct methods using *SHELXS*86 (Sheldrick, 1985). H atoms in computed positions. Anisotropic least-squares refinement (*SHELX*76; Sheldrick, 1976) using F; H isotropic with common refined temperature factor ($B = 9{\cdot}7$ Å^2). $w = 1/(\sigma^2 + 0{\cdot}015F^2)$, $R = 0{\cdot}062$, $wR = 0{\cdot}082$, $S = 0{\cdot}89$ for 2900 observed reflections. Final maximum shift/e.s.d. = 0·13. Maximum and minimum heights in final difference Fourier synthesis = 0·26 and −0·24 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The atomic parameters are given in Table 1. Fig. 1 is a stereoscopic view of the molecule, showing the numbering of the atoms (*PLUTO*; Motherwell & Clegg, 1978). Bond distances and angles are given in Table 2.*

Table 1. *Fractional atomic coordinates and equivalent isotropic temperature factors*

$$B_{eq} = (8\pi^2/3)\textstyle\sum_i\sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}(Å^2)
N1	0·3130 (2)	0·4853 (1)	0·6738 (1)	5·80
C2	0·2547 (3)	0·4002 (2)	0·6668 (1)	5·14
N3	0·1607 (2)	0·3777 (1)	0·7225 (1)	4·82
C4	0·1158 (2)	0·4448 (1)	0·7834 (1)	4·44
N5	0·2269 (2)	0·5131 (1)	0·8062 (1)	4·38
C6	0·2644 (2)	0·5543 (1)	0·7292 (1)	4·71
O7	0·2878 (2)	0·3447 (1)	0·6144 (1)	6·71
C8	0·4169 (4)	0·5129 (3)	0·6168 (2)	7·98
C9	0·5682 (5)	0·5179 (4)	0·6639 (4)	11·08
C10	0·6559 (6)	0·5839 (5)	0·6598 (5)	13·80
C11	0·0603 (3)	0·3028 (2)	0·6968 (2)	5·83
C12	−0·0874 (4)	0·3342 (2)	0·6585 (2)	7·33
C13	−0·2084 (5)	0·3050 (4)	0·6786 (4)	11·01
C14	0·0703 (2)	0·3977 (1)	0·8606 (1)	4·78
C15	−0·0458 (3)	0·4332 (2)	0·8949 (2)	6·41
C16	−0·0897 (4)	0·3931 (3)	0·9656 (2)	8·47
C17	−0·0192 (5)	0·3166 (3)	1·0012 (2)	8·36
C18	0·0966 (4)	0·2808 (2)	0·9682 (2)	7·29
C19	0·1415 (3)	0·3210 (2)	0·8974 (2)	5·76
C20	0·3566 (3)	0·4844 (2)	0·8648 (2)	5·55
C21	0·4389 (3)	0·5658 (2)	0·9020 (2)	6·79
C22	0·5762 (4)	0·5815 (3)	0·9051 (3)	9·87
C23	0·1415 (2)	0·6132 (2)	0·6863 (1)	4·87
C24	0·0759 (4)	0·5992 (2)	0·6026 (2)	6·96
C25	−0·0331 (4)	0·6598 (3)	0·5674 (2)	9·23
C26	−0·0738 (4)	0·7313 (3)	0·6123 (3)	9·09
C27	−0·0081 (3)	0·7445 (2)	0·6944 (3)	7·75
C28	0·0983 (3)	0·6865 (2)	0·7308 (2)	5·88

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

C2—N1	1·359 (3)	C6—N1	1·456 (3)
C8—N1	1·474 (3)	N3—C2	1·372 (3)
O7—C2	1·234 (3)	C4—N3	1·481 (2)
C11—N3	1·462 (3)	N5—C4	1·449 (2)
C14—C4	1·519 (3)	C6—N5	1·451 (2)
C20—N5	1·473 (3)	C23—C6	1·514 (3)
C9—C8	1·497 (6)	C10—C9	1·274 (7)
C12—C11	1·493 (4)	C13—C12	1·289 (6)
C15—C14	1·381 (3)	C19—C14	1·391 (3)
C16—C15	1·380 (4)	C17—C16	1·379 (6)
C18—C17	1·370 (6)	C19—C18	1·386 (4)
C21—C20	1·493 (3)	C22—C21	1·293 (5)
C24—C23	1·392 (3)	C28—C23	1·379 (3)
C25—C24	1·402 (5)	C26—C25	1·353 (6)
C27—C26	1·368 (6)	C28—C27	1·368 (4)

C6—N1—C2	122·2 (2)	C8—N1—C2	119·5 (2)
C8—N1—C6	117·9 (2)	N3—C2—N1	117·0 (2)
O7—C2—N1	121·6 (2)	O7—C2—N3	121·4 (2)
C4—N3—C2	121·9 (2)	C11—N3—C2	116·4 (2)
C11—N3—C4	116·8 (2)	N5—C4—N3	111·3 (2)
C14—C4—N3	111·2 (2)	C14—C4—N5	112·8 (2)
C6—N5—C4	109·3 (2)	C20—N5—C4	116·7 (2)
C20—N5—C6	111·9 (2)	N5—C6—N1	110·6 (2)
C23—C6—N1	114·1 (2)	C23—C6—N5	110·9 (2)
C9—C8—N1	111·3 (3)	C10—C9—C8	125·1 (5)
C12—C11—N3	113·2 (2)	C13—C12—C11	125·6 (4)
C15—C14—C4	118·2 (2)	C19—C14—C4	122·3 (2)
C19—C14—C15	119·5 (2)	C16—C15—C14	120·1 (3)
C17—C16—C15	120·0 (3)	C18—C17—C16	120·6 (3)
C19—C18—C17	119·6 (3)	C18—C19—C14	120·1 (3)
C21—C20—N5	110·3 (2)	C22—C21—C20	127·2 (4)
C24—C23—C6	122·8 (2)	C28—C23—C6	118·3 (2)
C28—C23—C24	118·9 (2)	C25—C24—C23	118·4 (3)
C26—C25—C24	121·7 (3)	C27—C26—C25	119·3 (3)
C28—C27—C26	120·6 (3)	C27—C28—C23	121·1 (3)

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bonds and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52620 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

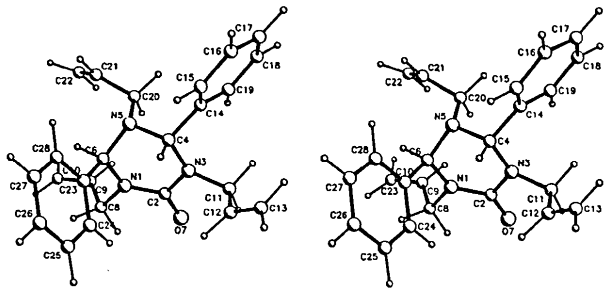

Fig. 1. Stereoscopic view of the molecule with atomic numbering scheme (*PLUTO*; Motherwell & Clegg, 1978).

Related literature. The crystal structures of 1,3,5-triphenyl-1,3,5-perhydrotriazine-2,4,6-trione (Usanmaz, 1979) and 1,3,5-tribenzamido-1,3,5-hexahydrotriazine monohydrate (George & Gilardi, 1987) are closely related to the title compound.

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Acta Cryst. (1990). C46, 1170–1172

Structure of a Neoclerodane Dilactone from *Baccharis rhomboidalis*

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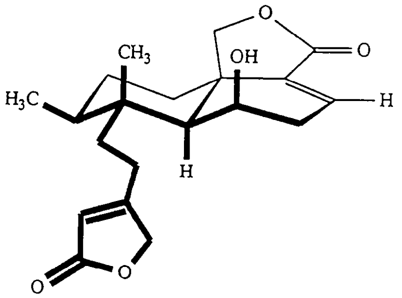
Abstract. 1-Hydroxyneoclerodane-3,13-diene-15,16;-19,20-diolide, $C_{20}H_{26}O_5$, $M_r = 346{\cdot}42$, orthorhombic, $P2_12_12_1$, $a = 15{\cdot}601$ (4), $b = 15{\cdot}962$ (4), $c = 7{\cdot}093$ (2) Å, $V = 1766{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}303$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}09$ mm^{-1}, $F(000) = 744$, $T = 293$ K, final $R = 0{\cdot}064$ for 1430 unique observed reflections with $F > 3\sigma(F)$. The molecular structure of the title compound consists of two *trans*-fused six-membered rings with a butenolide ring fused to both, and possesses as substituents a secondary and tertiary methyl group at C(8) and C(9), respectively, a secondary hydroxyl group at C(1), and a side chain with an α,β-unsaturated γ-lactone group at C(9). The methyl and hydroxyl groups and C(19) are in *cis*-axial configuration in agreement with the molecular structure proposed previously on the basis of chemical and spectroscopic methods [San-Martin, Rovirosa, Labbé, Givovich, Mahú & Castillo (1986). *Phytochemistry*, **25**, 1393–1395]. All bond lengths and angles are within the expected ranges.

Experimental. In the course of our chemical investigations of the genus *Baccharis*, the title neoclerodane dilactone was isolated from *Baccharis rhomboidalis* Remy from Central Chile. Colourless crystals, 0·5 × 0·5 × 1·0 mm, were used for X-ray analysis; Huber computer-controlled four-circle diffractometer, graphite-monochromated Mo $K\alpha$ radiation, ω-scan mode; cell parameters by least squares from 26 centred reflections, $2\theta \le 50°$; 1539 unique reflections



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