Table 1. *Fractional atomic coordinates and isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = \sum B_{ii}/3.$$

	x	y	z	B or B_{eq}
Rb	0·33047 (3)	0·03378 (2)	1·16264 (3)	1·71
O1	0·62081 (27)	−0·13513 (18)	1·04696 (24)	2·07
O2	0·70363 (23)	−0·32806 (15)	1·01999 (23)	2·11
O3	0·57468 (23)	−0·34298 (15)	0·69143 (23)	1·52
O4	0·86066 (21)	−0·17134 (19)	0·68467 (26)	1·67
O5	0·47342 (20)	−0·10666 (16)	0·46010 (21)	1·66
O6	0·72967 (22)	−0·15314 (17)	0·36071 (23)	1·97
C1	0·64073 (28)	−0·23754 (23)	0·96042 (29)	1·32
C2	0·5771 (3)	−0·22683 (22)	0·7736 (3)	1·26
C3	0·69001 (28)	−0·13951 (20)	0·6712 (3)	1·18
C4	0·62516 (27)	−0·13157 (20)	0·4824 (3)	1·20
H1	0·458 (3)	−0·1916 (21)	0·773 (3)	0·7 (5)
H2	0·679 (3)	−0·0728 (23)	0·7215 (29)	0·9 (5)
H3	0·476 (4)	−0·3522 (23)	0·662 (4)	1·5 (6)
H4	0·870 (3)	−0·2259 (21)	0·654 (3)	0·1 (6)
H5	0·646 (4)	−0·138 (3)	1·124 (4)	2·2 (8)

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

C1—O1	1·315 (3)	O1—C1—O2	124·5 (2)
C1—O2	1·202 (3)	O1—C1—C2	111·6 (2)
C1—C2	1·521 (3)	O2—C1—C2	123·9 (2)
C2—O3	1·423 (3)	C1—C2—C3	109·7 (2)
C2—C3	1·528 (3)	C1—C2—O3	110·5 (2)
C3—O4	1·400 (3)	O3—C2—C3	110·2 (2)
C3—C4	1·536 (3)	C2—C3—C4	108·8 (2)
C4—O5	1·245 (3)	C2—C3—O4	111·8 (2)
C4—O6	1·268 (3)	O4—C3—C4	114·0 (2)
O1—O6^{i}	2·559 (3)*	C3—C4—O5	117·7 (2)
O3—O6^{ii}	2·763 (2)*	C3—C4—O6	117·5 (2)
O4—O5^{iii}	2·824 (3)*	O5—C4—O6	124·8 (2)
Rb—O2	2·841 (2)	Rb—O5	2·974 (2)
Rb—O2	2·877 (2)	Rb—O1	3·085 (2)
Rb—O4	2·881 (2)	Rb—O3	3·121 (2)
Rb—O5	2·973 (2)	Rb—O3	3·122 (2)

* Hydrogen bond; symmetry codes: (i) $x, y, 1+z$; (ii) $-\frac{1}{2}+x, -\frac{1}{2}-y, 1-z$; (iii) $\frac{1}{2}+x, -\frac{1}{2}-y, 1-z$.

(Templeton, Templeton, Phillips & Hodgson, 1980). Covalent-bond distances in the caesium and rubidium compounds are nearly identical, but the average distances from rubidium and caesium to the eight nearest oxygen neighbors are 2·984 and 3·131 Å. The purpose of the present study was to obtain parameters needed for similar measurements of rubidium anomalous scattering near its K absorption edge, to be described elsewhere. All of this work is in agreement about the absolute configuration. The value $f'' = 2·973$ calculated by Cromer & Liberman (1970) for rubidium at Mo $K\alpha_1$ is close to that found here, 2·90 (4).

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N-3,4-Dichlorophenyl-3-methylbutanamide

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Abstract. $C_{11}H_{13}Cl_2NO$, $M_r = 246·1$, triclinic, $P\bar{1}$, $a = 4·818$ (1), $b = 10·933$ (2), $c = 12·309$ (3) Å, $\alpha = 105·99$ (2), $\beta = 97·86$ (2), $\gamma = 95·28$ (2)°, $V = 611·6$ (5) Å^3, $D_x = 1·34$ Mg m^{-3}, $Z = 2$, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 4·66$ mm^{-1}, $F(000) = 256$, $T = 293$ K. Final $R = 0·067$ for 1551 observed [$F > 3\sigma(F_o)$] reflections and 178 parameters. The phenyl and amido groups are planar and form a dihedral angle of 47·7 (5)°. This may be compared with values of 26·6 (6)° in *N*-3,4-dichlorophenyl-2-methylpropanamide [Precigoux, Busetta & Hospital (1976). *Acta Cryst.* B**32**, 943–945] and 17·1 (9)° in *N*-phenylacetamide [Brown (1966). *Acta Cryst.* **21**, 442–445]. The C_{ar}—N and N—C(=O) bond lengths are 1·420 (3) and 1·344 (4) Å respectively. The mean C—Cl bond length is 1·731 (3) Å. The crystal structure is stabilized

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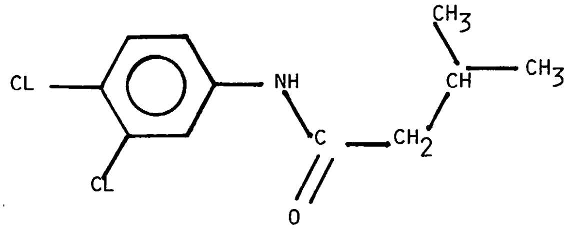
by an N(1)—H···O(1) [1+x, y, z] hydrogen bond: N···O = 2·841 (6) Å, H···O = 2·08 (4) Å, N—H···O = 169 (3)°.

Experimental. The compound (I) was synthesized by reaction of 3,4-dichlorobenzenamine with 3-methylbutanoyl chloride. White prismatic crystals (m.p. 363–364 K) were obtained by vapour diffusion from toluene/hexane. Crystal 0·37 × 0·15 × 0·28 mm, Nicolet $P2_1$ diffractometer, graphite-monochromatized Cu $K\alpha$ radiation. Cell parameters refined by least squares from the setting angles of 25 centred reflections with $45 \leq 2\theta \leq 55°$. Intensity data were collected by ω–2θ scans, scan range from 0·9° below α_1 to 0·9° above α_2, for 1941 reflections ($3 \leq 2\theta \leq 116°$: h 0/5, k −11/11, l −13/13). Three standard reflections (24$\bar{2}$, 01$\bar{7}$, 06$\bar{4}$) measured in every 100 showed no significant crystal decay. Of 1649 unique data ($R_{\rm int} = 0{\cdot}012$) 1551 with $F_o > 3\sigma(F_o)$ were used in the analysis. Structure solved by direct methods and refined by blocked cascade least squares based on F. Non-hydrogen atoms refined with anisotropic thermal parameters. Hydrogen atoms, partially located on a difference Fourier map, included at calculated positions; these positions were refined together with three overall U values for different H types. For 178 parameters, $R = 0{\cdot}067$, $wR = 0{\cdot}075$, $w^{-1} = \sigma^2(F_o) + 0{\cdot}00243F_o^2$, $S = 1{\cdot}79$, $\Delta/\sigma = 0{\cdot}11$ (max.) and 0·05 (mean), $\Delta\rho$ within ±0·54 e Å^{-3}. The *SHELXTL* program system (Sheldrick, 1983) was used throughout, with scattering factors taken from *International Tables for X-ray Crystallography* (1974).



(I)

Final atomic parameters are listed in Table 1* and molecular geometry in Table 2. The molecular structure and atomic numbering are illustrated in Fig. 1.

Table 1. *Atomic coordinates* (× 10^4, × 10^5 *for* Cl) *and isotropic thermal parameters* (Å^2 × 10^3)

	x	y	z	$U_{\rm eq}$*
Cl(1)	99311 (18)	60356 (7)	17527 (7)	80 (1)
Cl(2)	79656 (28)	61259 (8)	41174 (7)	103 (1)
C(1)	7386 (6)	4865 (2)	1857 (2)	53 (1)
C(2)	6485 (7)	4924 (3)	2877 (2)	61 (1)
C(3)	4323 (8)	4007 (3)	2921 (3)	70 (1)
C(4)	3186 (7)	3027 (3)	1941 (3)	62 (1)
C(5)	4152 (5)	2946 (2)	929 (2)	46 (1)
C(6)	6242 (5)	3883 (2)	878 (2)	49 (1)
N(1)	2929 (5)	1931 (2)	−67 (2)	50 (1)
C(7)	4376 (5)	1172 (3)	−774 (2)	55 (1)
C(8)	2594 (6)	94 (3)	−1734 (3)	62 (1)
C(9)	3741 (6)	−167 (3)	−2852 (2)	63 (1)
C(10)	3910 (14)	972 (4)	−3307 (4)	122 (2)
C(11)	1921 (10)	−1346 (4)	−3727 (4)	88 (2)
O(1)	6947 (4)	1327 (2)	−630 (2)	90 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

Cl(1)—C(1)	1·732 (3)	Cl(2)—C(2)	1·730 (2)
C(1)—C(2)	1·371 (4)	C(1)—C(6)	1·380 (3)
C(2)—C(3)	1·395 (5)	C(3)—C(4)	1·379 (4)
C(4)—C(5)	1·373 (4)	C(5)—C(6)	1·389 (4)
C(5)—N(1)	1·420 (3)	N(1)—C(7)	1·344 (4)
C(7)—C(8)	1·514 (3)	C(7)—O(1)	1·216 (3)
C(8)—C(9)	1·517 (4)	C(9)—C(10)	1·500 (6)
C(9)—C(11)	1·537 (4)		
Cl(1)—C(1)—C(2)	121·0 (2)	Cl(1)—C(1)—C(6)	118·2 (2)
C(2)—C(1)—C(6)	120·7 (3)	Cl(2)—C(2)—C(1)	121·2 (2)
Cl(2)—C(2)—C(3)	119·0 (2)	C(1)—C(2)—C(3)	119·8 (2)
C(2)—C(3)—C(4)	119·3 (3)	C(3)—C(4)—C(5)	120·9 (3)
C(4)—C(5)—C(6)	119·7 (2)	C(4)—C(5)—N(1)	119·6 (2)
C(6)—C(5)—N(1)	120·7 (2)	C(1)—C(6)—C(5)	119·6 (3)
C(5)—N(1)—C(7)	125·3 (2)	N(1)—C(7)—C(8)	115·5 (2)
N(1)—C(7)—O(1)	121·9 (2)	C(8)—C(7)—O(1)	122·6 (3)
C(7)—C(8)—C(9)	113·5 (2)	C(8)—C(9)—C(10)	112·9 (3)
C(8)—C(9)—C(11)	108·8 (3)	C(10)—C(9)—C(11)	111·6 (3)
C(4)—C(5)—N(1)—C(7)	−131·4 (3)	C(5)—N(1)—C(7)—C(8)	175·9 (3)
C(6)—C(5)—N(1)—C(7)	50·9 (4)	N(1)—C(7)—C(8)—C(9)	142·8 (3)
C(5)—N(1)—C(7)—O(1)	−2·3 (5)	O(1)—C(7)—C(8)—C(9)	−39·0 (5)

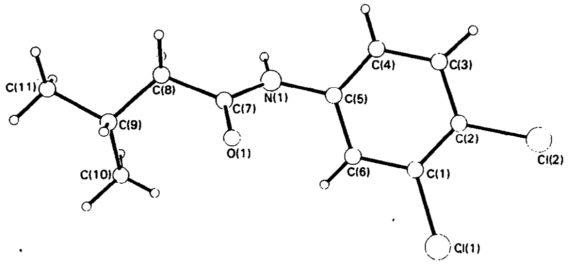


Fig. 1. A perspective view of the molecule showing atomic labelling.

Related literature. Geometrical data for this molecule are in good agreement with recent tabulations of mean bond lengths in organic molecules (Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The most relevant related crystal structure is that of N-3,4-dichlorophenyl-2-methylpropanamide (Precigoux, Busetta & Hospital, 1976). Structural characteristics of the carboxylic amide group are reviewed by Chakrabarti & Dunitz (1982).

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* Lists of structure factors, H-atom coordinates and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51520 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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8-α-Hydroxyachillin, a Guaianolide Sesquiterpene Lactone from *Dendroseris neriifolia*

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Abstract. *rel*-(5*S*,6*S*,7*R*,8*S*,11*S*)-8-Hydroxy-2-oxoguaia-1(10),3-dien-6,12-olide methanol solvate, $C_{15}H_{18}O_4.CH_3OH$, $M_r = 294{\cdot}35$, monoclinic, $P2_1$, $a = 10{\cdot}047\,(1)$, $b = 7{\cdot}717\,(1)$, $c = 10{\cdot}193\,(1)$ Å, $\beta = 112{\cdot}72\,(1)°$, $V = 728{\cdot}7\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}342$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}92$ cm^{-1}, $F(000) = 316$, $T = 293$ K, $R = 0{\cdot}0641$ for 1570 reflections. The cycloheptene ring is in a chair conformation, the five-membered cyclopentenone ring is planar while the five-membered lactone ring is in an envelope conformation. The structure contains a molecule of methanol solvent which is within hydrogen-bonded distance of the terpene, O(3)···O(5) $(x, y, 1+z) = 2{\cdot}814\,(4)$ Å, O(5)···O(4) $(1-x, y-0{\cdot}5, -z) = 2{\cdot}804\,(4)$ Å and O(5)···O(3′) $(1-x, y-0{\cdot}5, -z) = 2{\cdot}939\,(4)$ Å. The H atoms on the methanol could not be located. The achillin and matricarin series of sesquiterpenes differ only in the stereochemistry at C(11) in the lactone ring. Achillin is identified as the series with an α or *S* configuration at C(11).

Experimental. The plant *Dendroseris neriifolia* Hook & Arn. (Compositae-Lactuceae) was collected in the Juan Fernandez Islands (Masatierra) and the sesquiterpene was isolated by standard techniques. The pure sesquiterpene lactone (1) was recrystallized from methanol. A poor-quality colorless crystal of dimensions 0·32 × 0·40 × 0·50 mm was mounted on a Nicolet $R3m/\mu$ update of a $P2_1$ diffractometer; data collected in the ω scan mode ($3 \leq 2\theta \leq 55°$), variable scan rate 4 to 29·3° min^{-1}, graphite-monochromated Mo $K\alpha$ radiation; lattice parameters from a least-squares refinement of 25 reflections ($38{\cdot}14 \leq 2\theta \leq 43{\cdot}62°$); monitored reflections ($11\bar{5}$ and $\bar{1}40$) showed variations in intensities of less than $3\sigma(I)$; Laue symmetry $2/m$ and systematic absences ($0k0$, $k = 2n + 1$) consistent with space group $P2_1$; 1793 independent reflections measured ($-13 \leq h \leq 12$, $0 \leq k \leq 10$, $0 \leq l \leq 13$), equivalent reflections averaged, $1570 \geq 3\sigma(I)$; Lorentz–polarization corrections applied, ψ-scan-based empirical absorption correction (transmission factors 0·909–0·965); structure solved by direct methods and refined by anisotropic block-cascade least-squares techniques; the hydroxyl H atom was refined isotropically, all other terpene H atoms were located in a difference map but were constrained at a fixed distance from the attached atom; isotropic thermal parameters were refined for each H atom except for the methyl H atoms which were refined with a single isotropic thermal parameter; $R = 0{\cdot}0641$, $wR = 0{\cdot}0724$ for 204 parameters and 1570 reflections ($R = 0{\cdot}0945$ for all 1793 reflections), $S = 1{\cdot}527$, $(\Delta/\sigma)_{\max} = 0{\cdot}006$ with the largest peaks in the final difference map of $-0{\cdot}39$ and $+0{\cdot}28$ e Å^{-3}; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + 0{\cdot}00506F_o^2]^{-1}$; extinction correction $F = F_c/[1{\cdot}0 + 0{\cdot}002 \times 0{\cdot}009\,(3)\ F_c^2/\sin(2\theta)]^{0{\cdot}25}$ applied. Computer

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