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The Structures of Tanacetols A, $C_{17}H_{26}O_4$, and B, $C_{19}H_{30}O_5$, Two New Sesquiterpene Alcohols from *Tanacetum vulgare L.**

BY M. CALLERI AND G. CHIARI

Istituto di Mineralogia, Cristallografia e Geochimica dell'Università, Via San Massimo 22, 10123 *Torino, Italy*

AND D. VITERBO

Istituto di Chimica Fisica dell'Università, Corso M. d'Azeglio 48, 10125 *Torino, Italy*

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Abstract. Tanacetol A, m.p. 371 K, $[\alpha]_D^{25°C} = -99°$ ($CHCl_3$, 1·0 g dm^{-3}), is a ketol sesquiterpene isolated from a rare chemotype of *Tanacetum vulgare L.*: $M_r = 294{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}132$ (4), $b = 11{\cdot}255$ (3), $c = 13{\cdot}782$ (4) Å, $U = 1726{\cdot}8$ (8) Å^3, $Z = 4$, $D_x = 1{\cdot}13$ Mg m^{-3}, $F(000) = 640$, μ(Mo $K\alpha$) = 0·09 mm^{-1}, room temperature. Tanacetol B is the main constituent of the above chemotype and its monoacetyl derivative, m.p. 413 K, $[\alpha]_D^{25°C} = -205°$ ($CHCl_3$, 0·4 g dm^{-3}), was investigated: $M_r = 338{\cdot}4$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}589$ (3), $b = 10{\cdot}997$ (5), $c = 23{\cdot}416$ (9) Å, $U = 1954$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}14$ Mg m^{-3}, $F(000) = 736$, μ(Mo $K\alpha$) = 0·09 mm^{-1}, room temperature. The structures were solved by direct methods and refined to $R = 0{\cdot}041$ for tanacetol A and to $R = 0{\cdot}046$ for tanacetol B acetate using, respectively, 1088 and 1594 unique reflexions. Both ten-membered rings show a C(1)–C(10) *trans* double bond and present the energetically less favoured boat–chair conformation. The C(14) methyl and the C(15) methene are *syn* and on the α face of the rings; the oxygen of the keto group in tanacetol A is on the β face whereas the C(5) acetyl group in tanacetol B acetate is on the α face.

Introduction. In spite of the frequent occurrence of germacranes bearing the C(7) side chain oxidized at the lactone level (Fischer, Olivier & Fischer, 1979), only a few compounds are known with this side chain unoxidized or oxidized at levels lower than the lactone (Geissman, 1973). A recent finding and investigation of a rare chemotype of *Tanacetum vulgare L.*, from the South of Piedmont (Italy), revealed that this tansy produces C(12) unoxidized analogues of germacranolides (Appendino, Gariboldi & Nano, 1983). These constituents have been called tanacetols and they have in common a 1′-hydroxyisopropyl side chain. The structural elucidation of the two most abundant and less polar of them by spectroscopic methods was made difficult by the absence of substituents on the carbons C(6) or C(8) next to that bearing the side chain with the consequent impossibility of relating the orientation of the ring substituents to that of the hydroxyisopropyl function (Appendino *et al.*, 1983). It was then impossible to distinguish between the configurationally pseudo-enantiomeric alternatives (*A*) or (*A′*) and (*B*) or (*B′*), which are equally likely because of the conformational flexibility of the ten-membered ring. The present diffraction study showed that the correct stereostructures are (*A*) and (*B*) for tanacetol A and tanacetol B acetate, respectively. With these configurations, in order to have the 1′-hydroxyisopropyl side chain in the less hindered equatorial position, the ten-membered rings must adopt the boat–chair instead of the thermodynamically more stable chair–chair conformation (Guy, Sim & White, 1976) that would be compatible with the pseudo-enantiomers (*A′*) and (*B′*).

* Tanacetol is 5-(2-hydroxyisopropyl)-2-methyl-8-methylene-7-oxo-1-cyclodecen-10-yl acetate.

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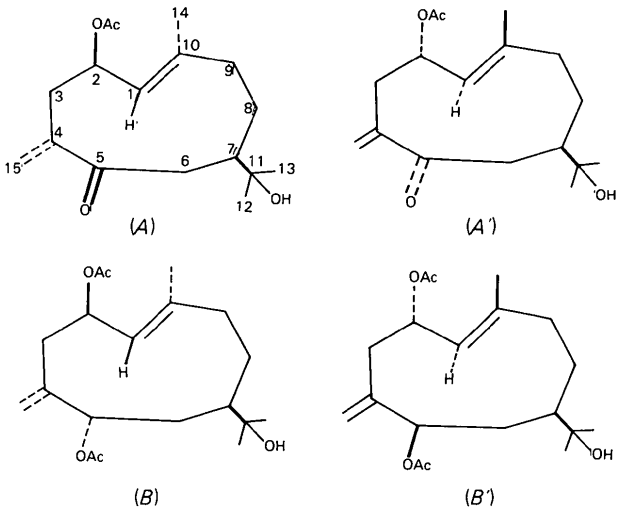

Experimental. The compounds purified by Appendino *et al.* (1983) as shining, transparent and fairly large prismatic crystals proved suitable for the X-ray analysis. The monoacetyl derivative of tanacetol B was preferred simply because it yielded much better crystals than the original compound.

Nicolet R3 four-circle diffractometer (graphite-monochromatized Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å); cell parameters refined from 22 reflexions for each crystal, $10° \le 2\theta \le 33°$; ω-scan technique, variable scan speed; direct methods.

Tanacetol A: 1317 independent reflexions, $3° \le 2\theta \le 45°$, scan speed range 1° min^{-1} for the weakest to 10° min^{-1} for the strongest reflexions, scan ranges 0·50° before and after the peak; 1090 intensities with $I \ge 2\,\sigma(I)$ placed on absolute scale by statistical methods using the Syntex (1976) suite of programs; for all subsequent computations the *SHELXTL* system (Sheldrick, 1981) was employed; program *SOLV*, using 259 phases with $|E| \ge 1{\cdot}40$ and 9 reflexions in the starting set, gave the structure; least-squares refinement, $\sum w(\Delta F)^2$ minimized, unit weights; all H atoms found on a difference Fourier map at an advanced stage of the anisotropic refinement; however, the H atoms of the tertiary CH, secondary CH_2 and primary CH_3 groups were forced to ride on the bonded C atom and the coordinates of the H atoms of the O(1)–H, =C(15)–$(H)_2$ and =C(1)–H groups (Fig. 1) were refined with the constraint C–H = 0·98 ± 0·04 Å; the thermal parameters were set equal to 1·2–1·4 times the equivalent U of the C atoms; two low-angle reflexions were discarded because of probable secondary extinction effects; $R = 0{\cdot}041$ for 1088 unique reflexions; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Tanacetol B acetate: The two octants hkl and $\bar{h}kl$ were explored within $2\theta \le 50°$, scan speed from 1 to 15° min^{-1}, scan ranges 0·4°; equivalent reflexions averaged to yield a total of 1984 reflexions, 1597 of which had $I \ge 2\,\sigma(I)$; crystal 0·20 × 0·24 × 0·70 mm, so an empirical absorption correction based on the ψ-scan method (North, Phillips & Mathews, 1968) was applied using six reflexions distributed over the 2θ range 9 to 39°; structure solved by the program *RANT* (Yao Jia-Xing, 1981); with 199 $|E|$'s > 1·60 and 1899 triple-phase relations, the best solution produced an E map showing all non-hydrogen atoms; the refinement followed mainly the procedure used for tanacetol A; the H of the O(1)–H group was refined isotropically and then fixed for the last cycles; the H atoms bonded to C(1), C(2), C(5) and C(7) (Fig. 2) were refined with the constraint C–H = 0·98 ± 0·03 Å, but with a common isotropic parameter that converged to U = 0·057 (4) Å^2; a separate thermal parameter was assigned to the two hydrogens of C(15), similarly constrained, common as well to the H atoms bonded to C(3), C(6), C(8) and C(9) which were forced to ride on the respective C atoms as those of the methyl groups; this latter thermal parameter converged to U = 0·074 (3) Å^2; weighting scheme used in the final stages: $w = 1/[\sigma^2(F_o) + GF_o^2]$ where σ is the standard deviation of the observed amplitudes, based on counting statistics, and G a variable to be adjusted after each cycle; convergence attained at: $R = 0{\cdot}046$, wR = 0·049, $G = 0{\cdot}00058$ for 1594 unique reflexions; three reflexions were discarded because of probable secondary extinction effects.

Discussion. The final parameters of the non-hydrogen atoms are given in Tables 1 and 2 for both structures.*

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters and figures showing perpendicular views of both molecules have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38372 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (Å^2 × 10^3) *of tanacetol A*

U_{eq} = ⅓ the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	7424 (4)	3197 (4)	8561 (3)	58 (1)
C(2)	8135 (4)	2099 (4)	8353 (3)	65 (2)
C(3)	8911 (4)	1748 (4)	9235 (3)	75 (2)
C(4)	9604 (4)	2810 (4)	9611 (3)	58 (2)
C(5)	9038 (4)	3507 (4)	10418 (3)	56 (1)
C(6)	9366 (4)	4796 (3)	10580 (3)	56 (1)
C(7)	8915 (3)	5620 (3)	9768 (3)	46 (1)
C(8)	7515 (4)	5584 (4)	9678 (3)	51 (1)
C(9)	7044 (4)	5350 (4)	8640 (3)	66 (2)
C(10)	7635 (4)	4256 (4)	8188 (3)	56 (1)
C(11)	9436 (4)	6891 (4)	9875 (4)	68 (2)
C(12)	9021 (5)	7684 (4)	9043 (4)	100 (2)
C(13)	9128 (5)	7460 (4)	10865 (4)	90 (2)
C(14)	8512 (4)	4492 (4)	7369 (3)	73 (2)
C(15)	10662 (4)	3104 (4)	9258 (4)	71 (2)
C(16)	7527 (4)	421 (3)	7408 (3)	60 (1)
C(17)	6608 (5)	−549 (4)	7323 (4)	78 (2)
O(1)	10732 (3)	6809 (3)	9886 (3)	90 (1)
O(2)	8290 (3)	3028 (3)	10944 (2)	78 (1)
O(3)	7304 (3)	1116 (3)	8161 (2)	84 (1)
O(4)	8351 (3)	578 (3)	6876 (2)	101 (2)

Table 2. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($Å^2 \times 10^3$) *of tanacetol B acetate*

$U_{eq} = \frac{1}{3}$ the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
O(1)	5446 (3)	6258 (2)	8359 (1)	67 (1)
O(2)	5434 (3)	10509 (2)	9282 (1)	69 (1)
O(3)	11647 (4)	11815 (2)	8554 (1)	92 (1)
O(4)	12782 (4)	11855 (3)	7697 (1)	110 (1)
O(5)	6675 (4)	10464 (3)	10144 (1)	112 (1)
C(1)	10888 (4)	9744 (3)	8773 (1)	63 (1)
C(2)	10474 (5)	10802 (3)	8402 (1)	69 (1)
C(3)	8582 (5)	11271 (3)	8484 (1)	74 (1)
C(4)	7241 (4)	10255 (3)	8470 (1)	60 (1)
C(5)	6813 (4)	9703 (3)	9045 (1)	53 (1)
C(6)	6197 (4)	8399 (3)	9067 (1)	54 (1)
C(7)	7599 (4)	7459 (3)	8888 (1)	49 (1)
C(8)	9190 (4)	7462 (3)	9311 (1)	58 (1)
C(9)	11020 (4)	7574 (3)	9037 (1)	70 (1)
C(10)	11113 (4)	8602 (3)	8608 (1)	64 (1)
C(11)	6788 (4)	6180 (3)	8793 (1)	57 (1)
C(12)	8197 (5)	5282 (3)	8592 (1)	70 (1)
C(13)	5828 (5)	5700 (3)	9321 (1)	73 (1)
C(14)	11354 (5)	8235 (3)	7987 (1)	81 (1)
C(15)	6468 (5)	9911 (3)	7993 (1)	79 (1)
C(16)	12749 (5)	12231 (3)	8168 (1)	71 (1)
C(17)	13926 (6)	13187 (4)	8402 (2)	108 (2)
C(18)	5571 (5)	10843 (3)	9825 (1)	69 (1)
C(19)	4127 (6)	11700 (3)	9992 (2)	94 (2)

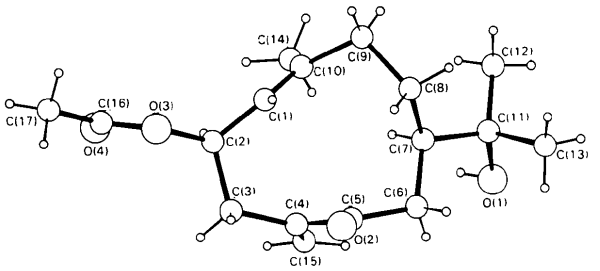

Fig. 1. Projection of the molecule of tanacetol A onto the mean plane through the ten-membered ring.

Figs. 1 and 2 show the projection of the two molecules on the mean plane through the ten-membered ring (the corresponding figures giving the perpendicular view have been deposited). Following the convention

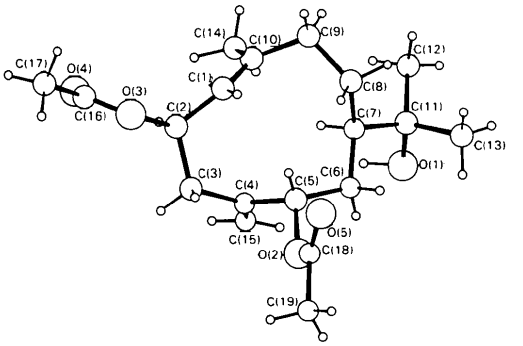

Fig. 2. Projection of the molecule of tanacetol B acetate onto the mean plane through the ten-membered ring.

proposed by Kupchan, Kelsey & Sim (1967) and by Rogers, Moss & Neidle (1972), the substituent at C(7) is on the β face. The interatomic distances and angles are given in Table 3. The corresponding bonds of the two molecules compare well.

The relevent mean values are, for tanacetol A and tanacetol B acetate respectively: $\langle C(sp^3) - C(sp^3)\rangle = 1{\cdot}544$ (4), 1·537 (3) Å; $\langle C(sp^3) - C(sp^2)\rangle = 1{\cdot}513$ (4), 1·508 (3) Å; $\langle C{=}O\rangle = 1{\cdot}208$ (5), 1·188 (4) Å; $\langle C(sp^3){-}O\rangle = 1{\cdot}461$ (5), 1·464 (4) Å; $\langle C(sp^2){-}O\rangle = 1{\cdot}322$ (5), 1·320 (3) Å. All these values appear standard even though some of the individual distances are somewhat longer than expected. It may be noted that the C(4)–C(5) bond in tanacetol A is not affected by conjugation since the substituents at the two C atoms are opposite with respect to the ring. The mean C–C(sp^3)–C angle is 112° in both rings which therefore do not reveal the angular strain found for instance in dihydromikanolide (Cox & Sim, 1974) and glaucolide A (Cox & Sim, 1975) showing the boat–chair conformation of the present compounds. This conformation is here attained without unduly short transannular separations; the C(10)···C(5) distances are > 3·30 Å in both tanacetols. The H···H separations shorter than 2·40 Å are: H(7)···H(14) = 2·25 (5) Å in tanacetol A; H(8)···H(5) = 2·17 (3); H(1)···H(5) = 2·28 (3),

Table 3. *Bond lengths* (Å) *and angles* (°) *for tanacetol A and tanacetol B acetate with e.s.d.'s in parentheses*

	A	B		A	B
C(1)–C(2)	1·496 (6)	1·486 (4)	C(2)–C(3)	1·543 (7)	1·537 (5)
C(3)–C(4)	1·514 (6)	1·512 (5)	C(4)–C(5)	1·500 (6)	1·511 (4)
C(5)–C(6)	1·513 (5)	1·510 (4)	C(6)–C(7)	1·538 (5)	1·541 (4)
C(7)–C(8)	1·563 (5)	1·561 (4)	C(8)–C(9)	1·545 (6)	1·535 (4)
C(9)–C(10)	1·529 (6)	1·514 (5)	C(10)–C(1)	1·320 (6)	1·326 (5)
C(2)–O(3)	1·466 (5)	1·469 (4)	O(3)–C(16)	1·322 (5)	1·313 (4)
C(16)–O(4)	1·187 (6)	1·180 (4)	C(16)–C(17)	1·501 (6)	1·484 (5)
C(4)–C(15)	1·317 (6)	1·317 (4)	C(5)–O(2)	1·229 (5)	1·480 (4)
O(2)–C(18)		1·326 (3)	C(18)–O(5)		1·197 (4)
C(18)–C(19)		1·498 (5)	C(7)–C(11)	1·550 (5)	1·552 (4)
C(11)–O(1)	1·445 (5)	1·442 (3)	C(11)–C(12)	1·525 (7)	1·530 (4)
C(11)–C(13)	1·546 (7)	1·530 (4)	C(10)–C(14)	1·516 (6)	1·520 (3)

	A	B		A	B
C(1)–C(2)–C(3)	110·9 (4)	112·7 (3)	C(2)–C(3)–C(4)	110·6 (3)	112·2 (3)
C(3)–C(4)–C(5)	116·9 (4)	115·0 (2)	C(4)–C(5)–C(6)	120·7 (3)	118·7 (2)
C(5)–C(6)–C(7)	113·1 (3)	114·4 (2)	C(6)–C(7)–C(8)	111·5 (3)	111·1 (2)
C(7)–C(8)–C(9)	114·6 (3)	115·8 (2)	C(8)–C(9)–C(10)	111·6 (3)	112·3 (2)
C(9)–C(10)–C(1)	119·5 (4)	120·5 (4)	C(10)–C(1)–C(2)	125·3 (4)	126·8 (3)
C(1)–C(2)–O(3)	108·9 (4)	108·8 (3)	C(3)–C(2)–O(3)	107·6 (3)	106·3 (2)
C(2)–O(3)–C(16)	118·0 (3)	118·9 (2)	O(3)–C(16)–O(4)	122·8 (4)	122·4 (3)
C(17)–C(16)–O(3)	111·4 (4)	112·2 (3)	C(17)–C(16)–O(4)	125·8 (4)	125·5 (3)
C(3)–C(4)–C(15)	121·9 (4)	122·0 (3)	C(5)–C(4)–C(15)	121·2 (4)	123·0 (3)
C(4)–C(5)–O(2)	119·5 (4)	104·2 (2)	C(6)–C(5)–O(2)	119·8 (4)	109·7 (4)
C(5)–O(2)–C(18)		118·1 (2)	O(2)–C(18)–O(5)		123·7 (3)
O(5)–C(18)–C(19)		124·6 (3)	O(2)–C(18)–C(19)		111·6 (3)
C(6)–C(7)–C(11)	111·4 (3)	111·9 (2)	C(8)–C(7)–C(11)	113·9 (3)	113·6 (2)
C(7)–C(11)–O(1)	108·4 (3)	109·2 (2)	C(7)–C(11)–C(12)	110·7 (4)	110·7 (2)
C(7)–C(11)–C(13)	112·6 (4)	112·6 (2)	C(12)–C(11)–C(13)	110·7 (4)	111·0 (2)
C(12)–C(11)–O(1)	110·4 (4)	108·4 (2)	C(13)–C(11)–O(1)	103·8 (4)	104·7 (2)
C(1)–C(10)–C(14)	124·2 (4)	123·1 (3)	C(9)–C(10)–C(14)	116·1 (4)	116·3 (3)

Table 4. *Endocyclic torsion angles (°) for some ten-membered rings with the boat–chair conformation*

	Calculated (1)	Tanacetol A (2)	Tanacetol B acetate (2)	Dihydromikanolide (3)	Shiromodiol (3)	Pregeijerene (3)	Ursiniolide A (4)
ω(1–2)	106	108	109	66	112	107	91
ω(2–3)	−40	−49	−48	−3	−49	−59	−54
ω(3–4)	92	94	91	55	86	82	80
ω(4–5)	−172	−156	−153	−163	−151	−150	−163
ω(5–6)	83	69	66	116	79	129	122
ω(6–7)	50	61	64	−2	57	−2	14
ω(7–8)	−118	−129	−129	−90	−129	−86	−79
ω(8–9)	57	51	49	73	53	59	43
ω(9–10)	61	66	64	57	64	66	91
ω(10–1)	−172	−165	−163	−153	−166	−165	−166

References: (1) Guy, Sim & White (1976); (2) present work (e.s.d.'s are ~0·3°); (3) Cox & Sim (1974); (4) Rychlewska (1981).

H(7)···H(14) = 2·31 (4) Å in tanacetol B acetate. An intermolecular hydrogen bond links, in A, O(1) to O(4)′ at $(\bar{x},\ 0{\cdot}5 + y,\ 1{\cdot}5 - z)$ [O(1)···O(4)′ = 2·977 (5), O(1) − H(1) = 0·88 (4), O(4)′···H(1) = 2·11 (3) Å] while in B the interaction is between O(1) and O(4)″ at $(2 - x,\ y - 0{\cdot}5,\ 1{\cdot}5 - z)$ [O(1)···O(4)″ = 2·889 (3), O(1) − H(1) = 1·02 (2), O(4)″···H(1) = 1·95 (2) Å]. Otherwise the packing is provided by van der Waals forces only, as revealed by the low densities.

Table 4 shows the relevant endocyclic angles (e.s.d.'s 0·2–0·4°) of tanacetols with those of a few other derivatives, with the boat–chair conformation, whose pattern is comparable to that of tanacetols. Among these germacrane derivatives, shiromodiol and tanacetols approach more closely the theoretical conformation of dimethylcyclodeca-1,5-diene according to the calculations of Guy, Sim & White (1976). The C(2)–C(1)–C(10)–C(9) torsion angles are, however, notably different from the ideal value of 180° in all three compounds (Table 4).

Two other natural compounds, related to tanacetols, with a 1′-hydroxyisopropyl chain at C(7) are known: hedycaryol (Wharton, Yui-Cheong Poon & Kluender, 1973) and the C(11) prenilogue of 6β-hydroxyhedycaryol (hydroxydiplophol, Sun & Fenical, 1979). Three conformations have been proposed for the former compound studied only in solution whilst hydroxydiplophol, according to the stereochemistry of its cyclization products, should assume the conformation $[{}_{15}D^{5},\ {}^{1}D_{14}]$ [after the notations of Samek & Harmatha (1978)].

The two-dimensional representation of the stereochemistry of tanacetols has been derived here and by Appendino *et al.* (1983) extending well established rules of germa-1(10),4-dienes (Rogers *et al.*, 1972; Samek & Harmatha, 1978; Fischer *et al.*, 1979) to the case of their 1(10),4(15) isomers, it seems therefore reasonable to extend in a similar way the Samek & Harmatha (1978) conventions to describe the conformation of the latter isomers. In the case of an exocyclic double bond we propose the symbols $^{x}_{D}$ and $^{D}_{x}$ to indicate the orientation of the *exo*-methylene group with respect to the mean plane through the ten-membered ring. The conformation of tanacetols can then be represented as $[{}^{D}_{15},\ {}^{1}D_{14}]$.

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