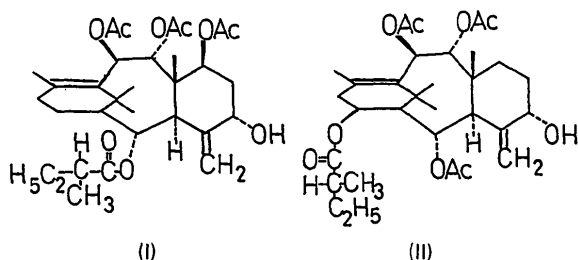


Discussion. Since the NMR spectrum of the isolated compound [taiwanxan, (II), m.p. = 500–503 K] indicates three acetyl groups (2.016, 2.028, 2.039) and an α -methylbutyrate group (0.88, *t*, $J = 7.4$ Hz; 1.11, *d*, $J = 7$ Hz), the structure is very similar to the known compound (I) (De Marciano & Halsall, 1969), tax-4(20),11-diene-2 α ,5 α ,7 β ,9 α ,10 β -pentol 7 β ,9 α ,10 β -triacetate 2 α - α -methylbutyrate, C₃₁H₄₆O₉, m.p. = 500–502 K. The structure of taiwanxan, (II), was revealed by X-ray single-crystal diffraction studies as tax-4(20),11-diene-2 α ,5 α ,9 α ,10 β ,14 β -pentol 2 α ,9 α ,10 β -triacetate 14 β - α -methylbutyrate.



Comparison of the NMR spectra of taiwanxan with that of compound (I) (see Table 1) confirmed that taiwanxan is a geometric isomer of compound (I).

An ORTEPB drawing is given in Fig. 1. The positional and isotropic thermal parameters are given in

Table 2.* Bond lengths and angles are given in Table 3. Compounds (I) and (II) have two differences: an α -methylbutyrate attaches to the 2 α position in compound (I) and 14 β in (II), while an acetate group attaches to 7 β in (I) and 2 α in (II). The geometry of the three acetyl groups in (II) is normal. Only C4–C15 and C11–C12 are double bonds. All the other C–C bonds in the skeleton are single.

The authors express their appreciation for support of this work to the National Science Council for a research grant and for the use of the CAD-4 diffractometer.

* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43802 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1987). C43, 1382–1384

Structure of Methyl *trans*-Communate

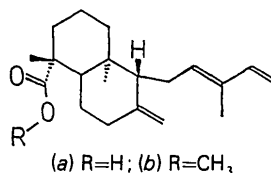
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(Received 16 December 1986; accepted 23 February 1987)

Abstract. C₂₁H₃₂O₂, $M_r = 316.47$, orthorhombic, $P2_12_12_1$, $a = 7.414$ (2), $b = 9.786$ (3), $c = 25.412$ (4) Å, $V = 1843.68$ Å³, $Z = 4$, $D_x = 1.14$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7093$ Å, $\mu(\text{Mo } K\alpha) = 0.72$ cm⁻¹, $F(000) = 696$, $T = 298$ K, $R = 0.051$ for 1494 reflections. *trans*-Communic acid was isolated from the fresh leaves of *Calocedrus formosana* (florin) Florin, then converted into the crystalline methyl ester with diazomethane. The structure is confirmed by X-ray diffraction study.

Introduction. *trans*-Communic acid (*a*) has been isolated from the heartwood of *Juniperus communis* L. and converted with diazomethane into methyl *trans*-communate (*b*) (Arya, Erdtman & Kubota, 1961). *trans*-Communic acid has also been separated by chromatography from the bled resin of New Zealand kauri (Thomas, 1966).



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The crystalline form of *trans*-communic acid was isolated from the fresh leaves of *Calocedrus formosana* (florin) Florin with the following properties: m.p. 402–404 K, $\lambda_{\text{max}}^{\text{EtOH}} = 232$ nm, $\alpha = +38.1^\circ$ ($c = 0.23$ in EtOH). It was reported (Arya, Erdtman & Kubota, 1961; Thomas, 1966) that *trans*-communic acid is difficult to purify and polymerizes very easily, and that it had not been obtained in a crystalline form. Thus, it was converted into its methyl ester. The NMR and IR spectra and m.p. of the ester are identical with those reported by the above authors. A single-crystal X-ray diffraction study was carried out to confirm the structure.

Experimental. *Calocedrus formosana* (florin) Florin was collected in Taiwan and was extracted with acetone and then partitioned with *n*-hexane. Communic acid was isolated from combined extracts with column chromatography (silica gel, Merck 5700, 70–230 mesh), eluting with *n*-hexane/ethyl acetate (1:9). Communic acid is converted with diazomethane into the methyl *trans*-commutate. Single crystals of methyl *trans*-commutate were grown from ethanol at room temperature.

A colorless crystal of dimensions $0.35 \times 0.45 \times 0.7$ mm was used for data collection. CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Unit-cell dimensions determined from a least-squares refinement of 25 reflections ($20.7 < 2\theta < 24.1^\circ$). Intensity data for $2\theta < 50^\circ$ were collected at variable scan speed $(20/16) - (20/3)^\circ \text{ min}^{-1}$ by use of the $\omega/2\theta$ scan technique, with a scan range $0.6^\circ + 0.35 \tan\theta$; $h 0 \rightarrow 8$, $k 0 \rightarrow 11$, $l 0 \rightarrow 30$. Three reflections monitored every 2 h, fluctuation within 1%. No correction for absorption, 1884 unique reflections, 1494 observed reflections with $I > 3\sigma(I)$.

The structure was solved by direct methods using *MULTAN*. All non-hydrogen atoms located from *E* map. The positions of H atoms were calculated from an idealized geometry and checked with the *D* map. All non-H atoms were refined anisotropically and all H atoms were fixed and not refined. Final $R(F) = 5.11\%$ ($wR = 6.29\%$), weighting scheme $w = 1/[\sigma^2(F) + (0.01F)^2]$, $\Delta/\sigma < 0.01$, peak in difference

map $< 0.25 \text{ e} \text{ \AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations were performed using the NRCC SDP PDP-11 package (Gabe & Lee, 1981) and *MULTAN* and *ORTEPB* from the Enraf–Nonius (1979) *Structure Determination Package* on a PDP-11/23 computer.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses

$$B_{\text{eq}} = (\frac{8}{3}\pi^2) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
C1	0.6758 (7)	0.8265 (5)	0.4625 (2)	5.6 (3)
C2	0.5990 (6)	0.7414 (4)	0.4292 (2)	4.3 (2)
C3	0.5942 (6)	0.5911 (4)	0.4332 (1)	3.7 (2)
C4	0.6834 (6)	0.5284 (5)	0.4825 (2)	4.8 (3)
C5	0.5190 (6)	0.5191 (4)	0.3956 (1)	3.4 (2)
C6	0.5089 (6)	0.3673 (4)	0.3896 (2)	3.6 (2)
C7	0.3203 (5)	0.3183 (4)	0.3725 (1)	3.0 (2)
C8	0.1818 (6)	0.3224 (4)	0.4163 (2)	3.5 (2)
C9	0.2180 (7)	0.3553 (5)	0.4659 (2)	4.8 (2)
C10	−0.0045 (6)	0.2842 (4)	0.3995 (2)	4.2 (2)
C11	−0.0039 (6)	0.1409 (4)	0.3748 (2)	4.0 (2)
C12	0.1273 (5)	0.1381 (4)	0.3286 (1)	3.1 (2)
C13	0.3235 (5)	0.1748 (4)	0.3453 (1)	2.8 (2)
C14	0.4055 (6)	0.0690 (4)	0.3827 (2)	3.4 (2)
C15	0.4383 (5)	0.1869 (4)	0.2949 (1)	3.5 (2)
C16	0.4302 (6)	0.0587 (4)	0.2601 (2)	4.2 (2)
C17	0.2359 (7)	0.0293 (5)	0.2443 (2)	4.6 (2)
C18	0.1116 (5)	0.0105 (4)	0.2918 (2)	3.6 (2)
C19	−0.0837 (6)	−0.0027 (5)	0.2714 (2)	4.9 (2)
C20	0.1523 (6)	−0.1281 (4)	0.3173 (2)	3.9 (2)
C21	0.0719 (8)	−0.2842 (5)	0.3835 (2)	5.8 (3)
O1	0.2605 (5)	−0.2088 (3)	0.3020 (1)	6.3 (2)
O2	0.0503 (4)	−0.1534 (3)	0.3590 (1)	5.2 (2)

Table 2. Bond lengths (Å) and angles (°) with e.s.d.'s in parentheses

C1–C2	1.316 (6)	C12–C18	1.563 (5)
C3–C5	1.311 (5)	C15–C16	1.536 (5)
C7–C8	1.515 (5)	C18–C19	1.544 (5)
C8–C10	1.493 (6)	C20–O2	1.325 (5)
C12–C13	1.557 (5)	C3–C4	1.544 (5)
C13–C15	1.543 (5)	C6–C7	1.540 (5)
C17–C18	1.530 (6)	C8–C9	1.328 (5)
C20–O1	1.191 (5)	C11–C12	1.526 (5)
C2–C3	1.475 (5)	C13–C14	1.531 (5)
C5–C6	1.495 (5)	C16–C17	1.523 (6)
C7–C13	1.566 (5)	C18–C20	1.533 (6)
C10–C11	1.537 (5)	C21–O2	1.433 (5)
C1–C2–C3	126.7 (4)	C2–C3–C4	116.2 (3)
C2–C3–C5	119.7 (3)	C4–C3–C5	124.1 (3)
C3–C5–C6	129.0 (3)	C5–C6–C7	112.6 (3)
C6–C7–C8	113.6 (3)	C6–C7–C13	113.0 (3)
C8–C7–C13	111.0 (2)	C7–C8–C9	124.5 (3)
C7–C8–C10	114.2 (3)	C9–C8–C10	121.3 (3)
C8–C10–C11	110.0 (3)	C10–C11–C12	109.5 (3)
C11–C12–C13	112.4 (3)	C11–C12–C18	115.3 (3)
C13–C12–C18	114.7 (3)	C7–C13–C12	108.3 (2)
C7–C13–C14	109.8 (2)	C7–C13–C15	107.8 (2)
C12–C13–C14	112.6 (3)	C12–C13–C15	107.8 (2)
C14–C13–C15	110.4 (3)	C13–C15–C16	113.2 (3)
C15–C16–C17	110.0 (3)	C16–C17–C18	112.6 (3)
C12–C18–C17	109.3 (3)	C12–C18–C19	109.7 (3)
C17–C18–C20	116.1 (3)	C17–C18–C19	108.1 (3)
C17–C18–C20	108.7 (3)	C19–C18–C20	104.6 (3)
C18–C20–O1	125.6 (3)	C18–C20–O2	113.0 (3)
O1–C20–O2	121.4 (4)	C20–O2–C21	116.8 (3)

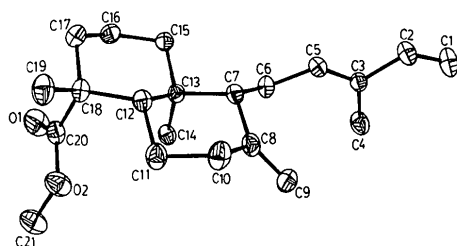


Fig. 1. The molecular structure and labeling scheme of $\text{C}_{21}\text{H}_{32}\text{O}_2$ (methyl *trans*-commutate).

Discussion. The *ORTEPB* drawing of the molecular structure is given in Fig. 1. The positional and isotropic thermal parameters are given in Table 1.* Bond lengths and angles are given in Table 2. Three bonds, C1—C2, C3—C5 and C8—C9, are double bonds, and all the other C—C bonds are single. The geometry of the methyl ester group is normal. The result confirmed the structure proposed by Arya, Erdtman & Kubota (1961) and Thomas (1966).

* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43803 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The authors express their appreciation for support of this work to the National Science Council for a research grant and for the use of the CAD-4 diffractometer.

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Acta Cryst. (1987). **C43**, 1384–1386

Structure of 2-*tert*-Butyl-6-methyl-1,6a λ^4 -dithia-6-azapentalene

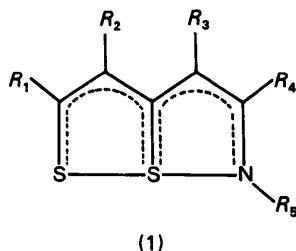
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(Received 17 July 1986; accepted 3 March 1987)

Abstract. C₁₀H₁₅NS₂, *M*_r = 213.4, triclinic, *P* $\bar{1}$, *a* = 5.891 (5), *b* = 8.857 (4), *c* = 11.449 (3) Å, α = 105.96 (3), β = 95.82 (4), γ = 74.15 (5)°, *V* = 552.23 (6) Å³, *Z* = 2, *D*_x = 1.28 g cm⁻³, λ (Mo *K*α) = 0.71073 Å, μ = 4.2 cm⁻¹, *F*(000) = 228, *T* = 293 K, final *R* = 0.0413 for 2089 observed unique reflections. A crystal-structure determination of the title compound showed the molecule to be bicyclic, almost planar, and to possess elongated S—S and compressed S—N bond lengths of 2.496 (1) and 1.791 (2) Å, respectively, and an N—S—S bond angle of 173.4 (1)°.

Introduction. A number of crystal-structure analyses of heterocyclic compounds of the type (1) have been carried out.



The compounds studied so far have all possessed *N*-aryl groups. A summary of the bond lengths of the S—S and S—N bonds is given:

	<i>R</i> ₁	<i>R</i> ₂	<i>R</i> ₃	<i>R</i> ₄	<i>R</i> ₅	S—S(Å)	S—N(Å)	Reference
1(a)	Ph	H	Ph	H	Ph	2.396 (12)	1.871 (10)	Leung & Nyburg (1972)
1(b)	Ph	H	Ph	H	3-Quinoliny	2.364 (7)	1.887 (2)	Leung & Nyburg (1971)
1(c)	Ph	H	H	Ph	Ph	2.440 (2)	1.860 (4)	Borel, Leclaire, Le Coustumer & Mollier (1978).

It can be seen that there are no large differences in the bond lengths under consideration. A structural feature also of interest in these compounds concerns the internal S—S—N bond angles, which for the three compounds listed are 174.5, 174.2, and 173.9°, respectively.

We have undertaken the crystal-structure analysis of the title compound, which is the first of this class of compounds in which all the substituents are alkyl, to determine what effect an alkyl group bonded to N has on the structural parameters of interest.

Experimental. The title compound was prepared by treatment of 5-*tert*-butyl-3-(2-dimethylaminovinyl)-1,2-dithiolium perchlorate (Vilsmeier salt) in dimethylformamide with aqueous methylamine (Dingwall, Ingram, Reid & Symon, 1973) at room temperature. The compound was recrystallized from hexane, to give

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† Deceased 23 March 1987.