

Also, the C(8)–C(9) and C(8)–C(10) distances [1.397 (9) and 1.403 (8) Å, respectively] are shorter than expected, while the C(8)–N(2) distance [1.514 (7) Å] is somewhat longer. These atoms were not as well behaved as the others during the refinement, due either to extreme thermal motion or to disorder. Thus the H associated with C(8) has been omitted.

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Structure of Terpin

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Abstract. *trans*-4-(1-Hydroxyisopropyl)-1-methylcyclohexan-1-ol monohydrate, C₁₀H₂₀O₂·H₂O, *M_r* = 190.28, monoclinic, *Cc*, *a* = 10.912 (3), *b* = 22.791 (4), *c* = 10.705 (2) Å, β = 120.64 (2)°, *V* = 2290.85 Å³, *Z* = 8, *D_m* = 1.10 (2), *D_x* = 1.11 Mg m⁻³, λ(Mo Kα) = 0.7093 Å, μ(Mo Kα) = 0.077 mm⁻¹, *F*(000) = 848, *T* = 298 K, final *R* = 0.047 for 1665 observed reflections. The title compound has the *trans* configuration with two molecules in the asymmetric unit. The water and *trans*-terpin molecules form a three-dimensional network connected by hydrogen bonding. C–C bonds range from 1.513 (6) to 1.554 (5) Å and C–O bonds from 1.427 (5) to 1.463 (6) Å; bond angles are normal.

Introduction. *Dendrocalamus latiflorus* Munro (Ma Bamboo) also called *Sinocalamus latiflorus* (Munro) or *Bambusa latiflorus* (Munro) is one of the major natural resources in Taiwan.

Tjon Sie Fat (1978), Chao, Ku, Huang, Lin, Lin & Pan (1963), Wu, Liu & Chen (1983), Chen & Juang (1982) and Lou (1983) have reported studies on the constituents of *D. latiflorus*. The chemical constituents were reported to be taxiphyllin and β-cellulose.

References

- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.22, p. 71. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- LAPP, R. L. & JACOBSON, R. S. (1979). *ALLS. A Generalized Crystallographic Least-squares Program*. US DOE Rep. IS-4708, Iowa State Univ., Ames, Iowa.
- MAIN, P., LESSINGER, L., WOOLFSON, M. M., GERMAIN, G. & DECLERCQ, J.-P. (1976). *MULTAN76. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England.
- MINE, A. & MATSUNAKI, S. (1975). *Pestic. Biochem. Physiol.* 5, 444–450.
- POWELL, D. R. & JACOBSON, R. A. (1980). *FOUR. A General Crystallographic Fourier Program*. US DOE Rep. IS-4737, Iowa State Univ., Ames, Iowa.

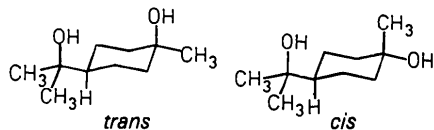
We have studied the methanol extract of the stem of *D. latiflorus* Munro collected at Chia-Yi, Taiwan. The following compounds were isolated and characterized: long-chain alkanes, allantoin, triclin, *p*-hydroxybenzaldehyde, long-chain carboxylic acids, β-sitosterol, stigmasterol, β-sitosteroyl-β-glycopyranoside, stigmasteroyl-β-glycopyranoside, acacetin, 2,2',4,4'-tetramethyl-*trans*-stilbene, and an unknown crystal *X*, C₁₀H₂₂O₃, m.p. 375.5 K. The ¹H NMR spectrum of compound *X* shows signals between 1.1 and 1.8 p.p.m. (in CDCl₃), its IR spectrum indicates the presence of O–H stretching (3240, 3480 cm⁻¹) and C–H stretching (2940 cm⁻¹), and its mass spectrum shows a base peak at 96 and a largest peak at 154 (2%). Since the spectroscopic data were insufficient for structural identification, a single crystal of compound *X* was prepared and an X-ray diffraction study was carried out. The unknown *X* was determined to be *trans*-terpin.

Experimental. IR spectra were recorded with a JASCO model IRA-1 spectrometer, NMR spectra were recorded at 90 MHz on the Varian EM-390 spectrometer, and mass spectra were obtained with a JEOL JMS-300 mass spectrometer. Separations by column chromatography were carried out on silica gel (Merck, Kieselgel 60, 70–230 mesh).

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Crystal dimensions: $0.2 \times 0.4 \times 0.6$ mm. CAD-4 diffractometer. Unit cell by least-squares refinement of 25 reflections ($18 < 2\theta < 37^\circ$). D_m by flotation in *n*-hexane/ CCl_4 . $2\theta_{\text{max}} = 50^\circ$. $-13 < h < 0$, $-27 < k < 0$, $-12 < l < 12$. Three standard reflections monitored every 2 h: variation $< 3\%$. 2220 unique reflections, 1665 observed with $I \geq 3\sigma(I)$. Absorption ignored. $R = 0.047$, $wR = 0.037$, $S = 4.04$ based on F . $w = 1/\sigma^2(F_o)$. Structure solved by direct methods using the *MULTAN* program with 185 highest E 's, 5 smallest E 's and 913 relationships. H atoms found on difference Fourier map. $(\Delta/\sigma)_{\text{max}} = 0.01$. Peaks on final difference map -0.19 to $0.16 \text{ e } \text{\AA}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computing programs: NRCC *SDP* PDP-11 Package (Gabe & Lee, 1981), *MULTAN* and *ORTEP* from Enraf-Nonius (1979) *Structure Determination Package*.

Discussion. There are two isomers of terpin, 4-(1-hydroxyisopropyl)-1-methylcyclohexan-1-ol, *i.e.* *trans*-terpin and *cis*-terpin. The *cis* form was originally assigned as the *trans* form (Barnes, 1958). The *trans* form, originally assigned as the *cis* form, was found in *Myrocapus silvestris*, *Protinium brasiliensis*, Chinese anise and sweet fennel oil.



There are two molecules in the asymmetric unit, both appear to be the *trans* form. The atomic coordinates and the equivalent isotropic temperature factors of the two molecules are listed in Table 1.* The molecular structure and labeling scheme of molecules *A* and *B* are shown in Fig. 1. The bond distances and angles are listed in Table 2. All the C—C bonds are single bonds ranging from 1.513 (6) to 1.554 (5) Å, the C—O bonds are also single ranging from 1.427 (5) to 1.463 (6) Å. The bond angles are quite normal.

The *trans*-terpin and water molecules are connected by three-dimensional hydrogen bonding. The $\text{O}\cdots\text{O}$ distances, listed in Table 3, are between 2.722 (4) and 2.872 (4) Å.

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* 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 43195 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and equivalent isotropic temperature factors (\AA^2)

$$B_{\text{eq}} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i a_j a_i a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
C(1A)	0.1117 (4)	0.0888 (2)	0.1557 (4)	3.2 (2)
C(2A)	0.0953 (5)	0.1558 (2)	0.1543 (4)	3.6 (2)
C(3A)	0.1600 (4)	0.1795 (2)	0.3084 (4)	3.6 (2)
C(4A)	0.3162 (5)	0.1635 (2)	0.4064 (4)	3.7 (3)
C(5A)	0.3324 (5)	0.0963 (2)	0.4052 (4)	3.9 (3)
C(6A)	0.2677 (5)	0.0722 (2)	0.2509 (5)	4.1 (3)
C(7A)	0.0426 (4)	0.0617 (2)	0.0012 (4)	3.8 (2)
C(8A)	0.0312 (5)	-0.0049 (2)	0.0050 (5)	4.8 (3)
C(9A)	0.1135 (5)	0.0798 (2)	-0.0820 (5)	5.1 (3)
O(10A)	-0.1037 (3)	0.0832 (1)	-0.0848 (3)	3.8 (2)
C(11A)	0.3742 (5)	0.1863 (2)	0.5587 (5)	5.1 (3)
O(12A)	0.3973 (3)	0.1878 (1)	0.3483 (3)	3.8 (2)
O(13A)	0.3280 (4)	0.3032 (1)	0.2831 (3)	5.8 (2)
C(1B)	0.6588 (4)	0.1613 (2)	0.2500 (4)	3.4 (3)
C(2B)	0.6431 (5)	0.0941 (2)	0.2507 (4)	3.5 (3)
C(3B)	0.5544 (5)	0.0707 (2)	0.0978 (5)	3.7 (3)
C(4B)	0.6118 (4)	0.0867 (2)	-0.0015 (4)	3.7 (3)
C(5B)	0.6291 (5)	0.1534 (2)	-0.0010 (4)	3.7 (3)
C(6B)	0.7189 (5)	0.1777 (2)	0.1535 (5)	4.0 (3)
C(7B)	0.7438 (5)	0.1880 (2)	0.4036 (4)	3.6 (3)
C(8B)	0.7309 (5)	0.2545 (2)	0.3988 (5)	5.2 (4)
C(9B)	0.8991 (5)	0.1701 (2)	0.4869 (5)	5.1 (3)
O(10B)	0.6828 (3)	0.1667 (1)	0.4886 (3)	3.6 (2)
C(11B)	0.5183 (5)	0.0639 (2)	-0.1543 (5)	5.2 (3)
O(12B)	0.7515 (3)	0.0619 (1)	0.0560 (3)	3.9 (2)
O(13B)	0.7503 (4)	0.9469 (1)	0.1238 (4)	5.8 (2)

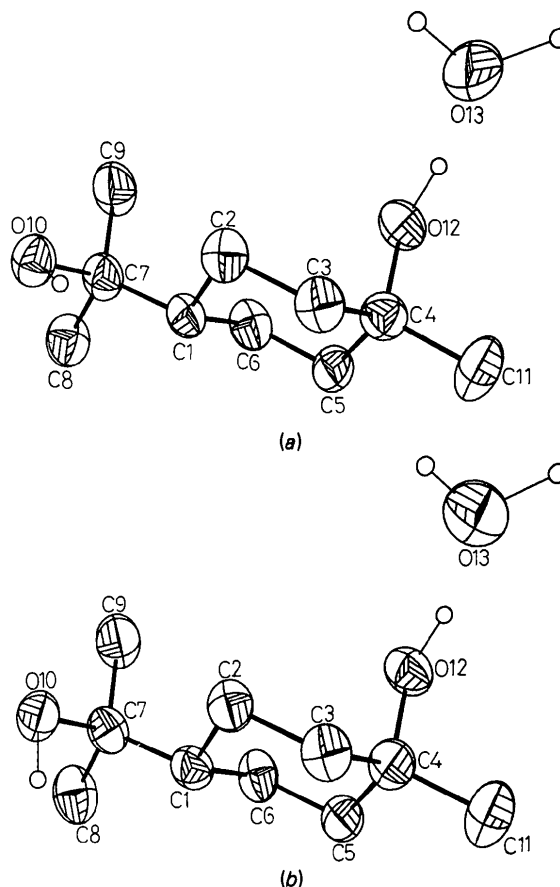


Fig. 1. Structure of (a) molecule *A* and (b) molecule *B*.

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

C(1A)—C(2A)	1.537 (5)	C(1B)—C(6B)	1.525 (5)
C(2A)—C(3A)	1.525 (5)	C(3B)—C(4B)	1.527 (5)
C(4A)—C(11A)	1.509 (5)	C(4B)—O(12B)	1.438 (5)
C(7A)—C(8A)	1.525 (6)	C(7B)—C(9B)	1.514 (6)
C(1B)—C(2B)	1.543 (5)	C(1A)—C(7A)	1.554 (5)
C(2B)—C(3B)	1.513 (5)	C(4A)—C(5A)	1.544 (6)
C(4B)—C(11B)	1.513 (6)	C(5A)—C(6A)	1.530 (6)
C(7B)—C(8B)	1.520 (6)	C(7A)—O(10A)	1.463 (5)
C(1A)—C(6A)	1.520 (6)	C(1B)—C(7B)	1.543 (5)
C(3A)—C(4A)	1.521 (5)	C(4B)—C(5B)	1.531 (6)
C(4A)—O(12A)	1.427 (5)	C(5B)—C(6B)	1.534 (6)
C(7A)—C(9A)	1.504 (6)	C(7B)—O(10B)	1.458 (4)
C(2A)—C(1A)—C(6A)	109.8 (3)	C(2A)—C(1A)—C(7A)	113.2 (2)
C(6A)—C(1A)—C(7A)	112.6 (3)	C(1A)—C(2A)—C(3A)	110.7 (3)
C(2A)—C(3A)—C(4A)	113.6 (3)	C(3A)—C(4A)—C(5A)	108.9 (3)
C(3A)—C(4A)—C(11A)	111.6 (3)	C(3A)—C(4A)—O(12A)	109.4 (3)
C(5A)—C(4A)—C(11A)	111.3 (3)	C(5A)—C(4A)—O(12A)	106.4 (3)
C(11A)—C(4A)—O(12A)	109.0 (3)	C(4A)—C(5A)—C(6A)	112.0 (3)
C(1A)—C(6A)—C(5A)	112.2 (3)	C(1A)—C(7A)—C(8A)	111.7 (3)
C(1A)—C(7A)—C(9A)	113.9 (3)	C(1A)—C(7A)—O(10A)	108.5 (3)
C(8A)—C(7A)—C(9A)	111.3 (3)	C(8A)—C(7A)—O(10A)	105.9 (3)
C(9A)—C(7A)—O(10A)	105.1 (3)	C(2B)—C(1B)—C(6B)	109.5 (3)
C(2B)—C(1B)—C(7B)	113.2 (3)	C(6B)—C(1B)—C(7B)	113.2 (3)
C(1B)—C(2B)—C(3B)	110.9 (3)	C(2B)—C(3B)—C(4B)	113.9 (3)
C(3B)—C(4B)—C(5B)	109.3 (3)	C(3B)—C(4B)—C(11B)	112.3 (3)
C(3B)—C(4B)—O(12B)	109.2 (3)	C(5B)—C(4B)—C(11B)	111.1 (3)
C(5B)—C(4B)—O(12B)	106.7 (3)	C(11B)—C(4B)—O(12B)	108.1 (3)
C(4B)—C(5B)—C(6B)	111.9 (3)	C(1B)—C(6B)—C(5B)	112.5 (3)
C(1B)—C(7B)—C(8B)	111.2 (3)	C(1B)—C(7B)—C(9B)	114.0 (3)
C(1B)—C(7B)—O(10B)	108.5 (3)	C(8B)—C(7B)—C(9B)	110.1 (3)
C(8B)—C(7B)—O(10B)	106.8 (3)	C(9B)—C(7B)—O(10B)	105.8 (3)

Table 3. The O...O distances (Å) in the unit cell

O(10A)—O(13A)	2.861 (4)	O(12A)—O(10B)	2.725 (4)
O(10A)—O(12A)	2.727 (4)	O(13A)—O(10A)	2.861 (4)
O(10A)—O(13B)	2.770 (4)	O(13A)—O(12A)	2.727 (4)
O(12A)—O(13A)	2.727 (4)	O(13A)—O(10B)	2.798 (4)
O(10B)—O(12A)	2.725 (4)	O(12B)—O(13B)	2.722 (4)
O(10B)—O(13A)	2.798 (4)	O(13B)—O(10A)	2.770 (4)
O(10B)—O(13B)	2.872 (4)	O(13B)—O(10B)	2.872 (4)
O(12B)—O(10A)	2.727 (4)	O(13B)—O(12B)	2.722 (4)

References

- BARNES, C. S. (1958). *Aust. J. Chem.* **11**, 134–146.
- CHAO, S. C., KU, Y. C., HUANG, L. Y., LIN, T. N., LIN, S. C. & PAN, T. T. (1963). *Taiwan Lin Yeh Shih Yen So Pao Kao*, No. 90, pp. 1–12.
- CHEN, T. L. & JUANG, T. J. (1982). *K'o Hsueh Fa Chan Yueh K'an*, **10**(3), 264–271.
- Enraf-Nonius (1979). *Structure Determination Package*. Enraf-Nonius, Delft.
- GABE, E. J. & LEE, F. L. (1981). *Acta Cryst.* **A37**, S339.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- LOU, K. C. (1983). MSc Thesis, Tamkang Univ.
- TJON SIE FAT, L. (1978). *Proc. K. Ned. Akad. Wet. Ser. C*, **81**(3), 347–354.
- WU, C. M., LIU, W. L. & CHEN, C. C. (1983). *Instrum. Anal. Foods: Recent Prog., Proc. Symp. 3rd Int. Flavor Conf.*, pp. 303–314.

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Structure of *p*-Chlorophenyl(phenylseleno)acetylene

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Abstract. C₁₄H₉ClSe, *M_r* = 291.6, monoclinic, *P*2₁, *a* = 7.270 (2), *b* = 5.835 (3); *c* = 14.550 (6) Å, β = 100.83 (3)°, *V* = 606.3 Å³, *Z* = 2, *D_x* = 1.597 Mg m⁻³, λ(Mo Kα) = 0.71073 Å, μ = 3.25 mm⁻¹, *F*(000) = 288, *T* = 296 K, final *R* = 0.048 for 1309 unique observed reflections. The structure consists of molecules of *p*-ClPhC≡CSePh possibly connected by very weak, secondary Se...Cl bonds of 3.704 (2) Å into zigzag chains parallel to the *ab* plane. In the nearly linear C—C≡C—Se— sequence, the bond lengths are 1.446 (9), 1.174 (10) and 1.836 (7) Å, respectively, while Se—C(Ph) = 1.915 (5) Å and angle C—Se—C = 98.4 (3)°.

Introduction. We recently determined the structure of a Wittig-reaction intermediate, phenacyl-α-(phenylseleno)triphenylphosphorane (Husebye, Meyers, Zingaro, Braga, Comasseto & Petragnani, 1986). Heating this compound under vacuum results in the formation of a phenylselenoacetylene and triphenylphosphine oxide (Braga, Comasseto & Petragnani, 1984). The present study was undertaken in order to learn something about the structure of molecules in which Se is bonded directly to an acetylenic C atom.

Experimental. The preparation of *p*-chlorophenyl(phenylseleno)acetylene has previously been reported