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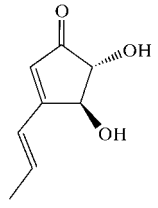
The fungal metabolite terrein (alternative name: *trans*-2,3-dihydroxy-4-propenylcyclopent-4-enone), $C_8H_{10}O_3$, forms monoclinic ($P2_1$) crystals. The molecules form hydrogen-bonded chains, with O···O distances of 2.7115 (16) and 2.8155 (15) Å.

Comment

The discovery of bioactive natural products is greatly facilitated by the opportunity to examine unusual organisms. One such group, which has proven to be a rich source of interesting compounds, is the endophytic fungi. Such fungi are commonly present within the living tissues of plants and often appear to act in a symbiotic fashion with them. Frequently, such endophytes develop host specificity and thus are found nowhere else in nature except with their respective plant host.

As part of our survey of endophytic fungi of the highlands of Papua New Guinea, the fungus *Pestalotiopsis microspora* was isolated and examined for bioactive components. Previous work has demonstrated that *P. microspora* from other plant



(I)

sources produces a variety of bioactive compounds including the anticancer drug taxol (Strobel *et al.*, 1996). Bioassay guided fractionation of a culture solution of *P. microspora* provided a crystalline solid. X-ray analysis of a suitable crystal established that the isolated material was terrein, (I). Previously terrein has been isolated from fungi of the *Aspergillus* (Raistrick & Smith, 1935; Misawa *et al.*, 1962; Qureshi *et al.*, 1968, 1976), *Penicillum* (Grove, 1954) and *Phoma* (Dunn *et al.*, 1975) genera, and exhibits both antibacterial and plant growth inhibitor activity (Kamata *et al.*, 1983; Qureshi *et al.*, 1976). However, no crystal structure of terrein has yet appeared. In order to characterize the hydrogen bonding modes, often crucial to bioactivity, the X-ray analysis was performed.

Crystalline terrein forms a hydrogen-bonding network composed entirely of intermolecular hydrogen bonds. The carbonyl at C1 acts as a hydrogen bond acceptor to the C2 OH in a neighbouring molecule. The C2 OH interacts both at C1 (as described above) and as a hydrogen-bond acceptor to the C3 OH of an adjacent molecule.

Experimental

Samples of *P. Microspora* were isolated from leaf and stem tissue of a Pandanus sp. using previously described methods (Strobel *et al.*, 1996). A culture (5 l) of *P. microspora* was grown on M1D media (Pinkerton & Strobel, 1976) enriched with 1 g l^{-1} of soytone. After a 15-day incubation (296 K), the fluid was filtered then twice extracted with equal volumes of methylene chloride. Evaporation of methylene chloride gave 250 mg of crude material. Preliminary separation was achieved on a silica gel column using $CHCl_3$–MeOH (20:1). Fractions containing common materials by TLC analysis (silica, $CHCl_3$–MeOH, 7:1) were combined and examined for antifungal activity utilizing the plant pathogenic fungus *Pythium ultimum* as a test organism. The active fraction [R_f = 0.45, silica TLC ($CHCl_3$–MeOH, 7:1)] was further purified on silica gel using ethyl acetate–hexane (4:1). Pooling of common fractions provided 55 mg of terrein. Plate-like crystals (m.p. 392–395 K) were obtained by slow evaporation of a methanol solution. A suitable crystal was obtained only after cutting a contact twinned crystal parallel to the plane of the plate. Although the lack of suitable anomolous scatterers did not allow determination of absolute stereochemistry, optical rotation, [α] = + 142° (c = 0.175 g per 100 ml, water), verified that the structure described herein is (+)-terrein (Barton & Miller, 1955).

Crystal data

$C_8H_{10}O_3$
M_r = 154.16
Monoclinic, $P2_1$
a = 4.5377 (2) Å
b = 6.9065 (2) Å
c = 12.3009 (5) Å
β = 90.372 (2)°
V = 385.50 (3) Å^3
Z = 2
D_x = 1.328 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7566 reflections
θ = 1.02–32.58°
μ = 0.102 mm^{-1}
T = 200.0 (1) K
Thin plate, colourless
0.32 × 0.30 × 0.12 mm

Data collection

Nonius KappaCCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
T_{min} = 0.968, T_{max} = 0.988
7566 measured reflections
1479 independent reflections
1344 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 32.58°
$h = -6 \rightarrow 6$
$k = -10 \rightarrow 10$
$l = -18 \rightarrow 18$
Intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.035
$wR(F^2)$ = 0.090
S = 1.049
1479 reflections
109 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.0370P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.5 (11)

Table 1
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.90 (3)	1.83 (3)	2.7115 (16)	165 (2)
O3—H3···O2^{ii}	0.80 (2)	2.03 (2)	2.8155 (15)	168.4 (18)

Symmetry codes: (i) $1-x, y-\frac{1}{2}, 1-z$; (ii) $x-1, y, z$.

Hydroxy H atoms were located and isotropically refined. Other H atoms were treated as riding (C—H = 0.95–1.00 Å). Friedel pairs (1025) were merged and averaged in the data set, since anomalous dispersion effects are negligible.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO–SMN*; program(s) used to solve structure: *SIR*97 (Altomare *et al.*, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997).

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