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3,7,8-Trihydroxy-3-methyl-10-oxo-4,10-dihydro-1*H*,3*H*-pyrano[4,3-*b*]chromene-9-carboxylic acid (fulvic acid) methanol 0.75-solvate

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.007 Å
R factor = 0.075
wR factor = 0.196
Data-to-parameter ratio = 10.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $C_{14}H_{12}O_8\cdot0.75CH_4O$, crystallizes in a centrosymmetric triclinic unit cell, which contains four independent essentially planar molecules and three methanol solvent molecules in the asymmetric unit. The molecules in the crystal are linked by a hydrogen-bonding network.

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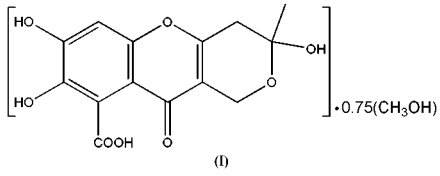
Comment

3,7,8-Trihydroxy-3-methyl-10-oxo-4,10-dihydro-1*H*,3*H*-pyrano[4,3-*b*]chromene-9-carboxylic acid (fulvic acid), (I) (Fig. 1), another yellow acidic metabolite, was isolated from *Paecilomyces sp*. Its formulation, having one more hydroxy group, differs from that of anhydrofulvic acid (Wang *et al.*, 2003).



(I)

The title compound crystallizes in a centrosymmetric triclinic unit cell, which contains four independent essentially planar molecules and three methanol solvent molecules in the asymmetric unit. The molecules in the crystal structure are linked by a hydrogen-bonding network (Table 1 and Fig. 2).

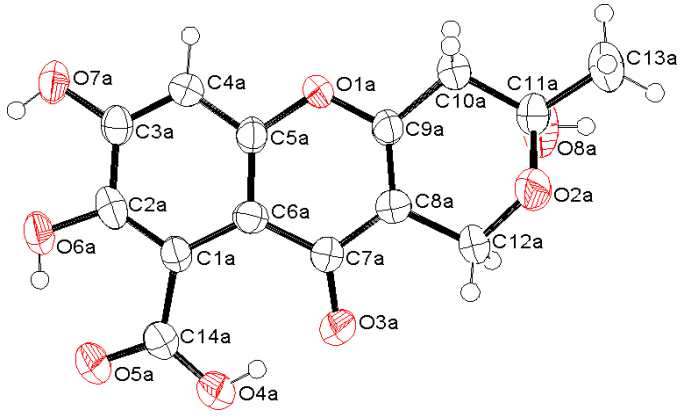


Figure 1
ORTEP-3 (Farrugia, 1997) view of one independent molecule of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms are drawn as spheres of arbitrary radii.

Experimental

The title compound was isolated from *Paecilomyces sp*., an endophytic fungus of *Cephalataxus fortunei*. Crystals were grown from methanol.

Crystal data

$C_{14}H_{12}O_8\cdot0.75CH_4O$
M_r = 332.27
Triclinic, $P\overline{1}$
a = 12.4830 (7) Å
b = 12.6558 (8) Å
c = 19.0259 (13) Å
α = 94.608 (3)°
β = 100.871 (3)°
γ = 98.399 (3)°
V = 2902.1 (3) Å^3
Z = 8
D_x = 1.521 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1065 reflections
θ = 2.2–19.4°
μ = 0.13 mm^{-1}
T = 298 (2) K
Chunk, yellow
0.23 × 0.15 × 0.13 mm

Data collection

Bruker SMART APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.953, T_{max} = 0.985
14 782 measured reflections
9473 independent reflections
4625 reflections with $I > 2\sigma(I)$
R_{int} = 0.052
θ_{max} = 25.0°
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 15$
$l = -22 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.075
$wR(F^2)$ = 0.196
S = 1.01
9473 reflections
873 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0668P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.002
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4*A*—H4*A*···O3*A*	0.82	1.57	2.389 (5)	173
O6*A*—H6*A*···O5*A*	0.82	1.73	2.443 (5)	144
O8*A*—H8*A*···O8*B*	0.82	2.06	2.860 (5)	166
O4*B*—H4*B*···O3*B*	0.82	1.56	2.373 (5)	168
O6*B*—H6*B*···O5*B*	0.82	1.73	2.449 (5)	146
O4*C*—H4*C*···O3*C*	0.82	1.59	2.405 (5)	176
O6*C*—H6*C*···O5*C*	0.82	1.73	2.456 (5)	147
O4*D*—H4*D*···O3*D*	0.82	1.58	2.386 (5)	168
O6*D*—H6*D*···O5*D*	0.82	1.70	2.435 (5)	148
O7*D*—H7*D*···O2	0.82	1.85	2.618 (4)	155
O8*D*—H8*D*···O7*B*	0.82	2.10	2.869 (5)	155
O7*A*—H7*A*···O8*C*^{i}	0.82	2.04	2.768 (5)	148
O7*B*—H7*B*···O1^{ii}	0.82	1.90	2.653 (5)	153
O8*B*—H8*B*···O7*D*^{iii}	0.82	2.07	2.852 (4)	159
O7*C*—H7*C*···O3^{iv}	0.82	1.90	2.655 (5)	152
O8*C*—H8*C*···O5*B*^{v}	0.82	2.23	3.031 (5)	166
O1—H1···O5*A*^{vi}	0.82	1.98	2.786 (5)	167
O2—H2···O5*C*^{vii}	0.82	1.97	2.789 (5)	175
O3—H3···O5*D*^{viii}	0.82	2.03	2.850 (5)	176

Symmetry codes: (i) $x, 1+y, z$; (ii) $1-x, 1-y, -z$; (iii) $x-1, y, z$; (iv) $x, y-1, z$; (v) $-x, 1-y, -z$; (vi) $1+x, y-1, z$; (vii) $1-x, -y, 1-z$; (viii) $1-x, 1-y, 1-z$.

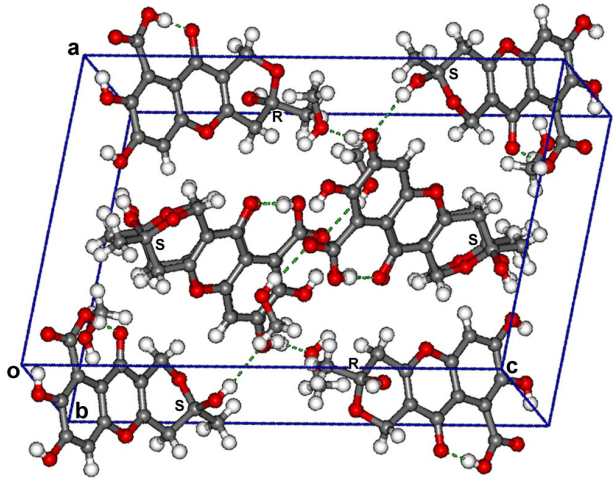


Figure 2
Packing diagram (Accelrys, 2001), showing the hydrogen bonding.

The H atoms were positioned geometrically (C—H = 0.93, 0.97 or 0.96 Å for phenyl, methylene or methyl H atoms, respectively, and O—H = 0.82 Å) and were included in the refinement in the riding-model approximation. The displacement parameters of phenyl and methylene H atoms were set to $1.2U_{eq}$ of their parent atoms, while those of methyl and O-bound H atoms were set to $1.5U_{eq}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997) and *ViewerPro* (Accelrys, 2001); software used to prepare material for publication: *SHELXL*97.

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