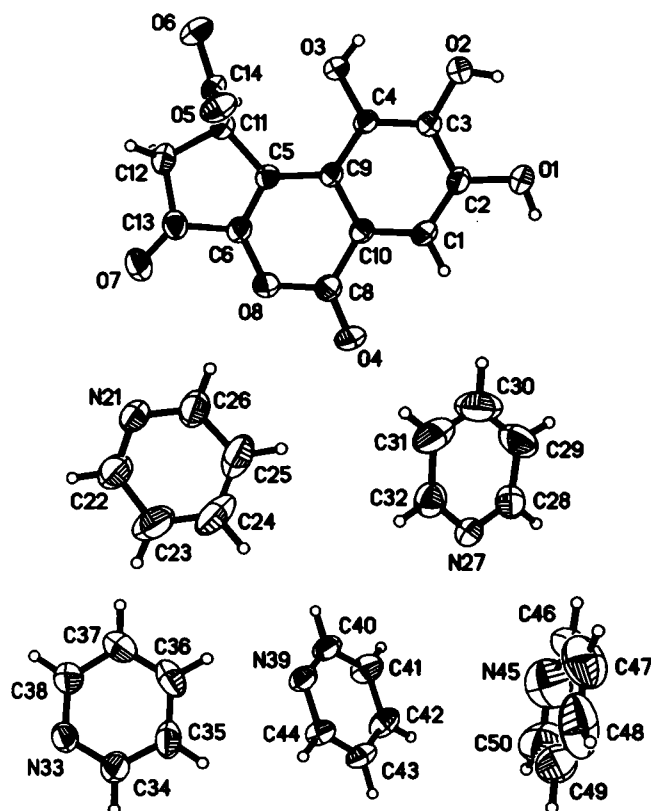


Crystal structure of brevifolincarboxylic acid pyridine pentasolvate, $C_{13}H_8O_8 \cdot 5 C_5H_5N$

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Abstract

$C_{38}H_{33}N_5O_8$, triclinic, $P\bar{1}$ (no. 2), $a = 11.127(2)$ Å, $b = 12.310(3)$ Å, $c = 14.266(6)$ Å, $\alpha = 80.58(3)^\circ$, $\beta = 69.80(3)^\circ$, $\gamma = 73.53(2)^\circ$, $V = 1753.9$ Å³, $Z = 2$, $R_{gt}(F) = 0.047$, $wR_{ref}(F^2) = 0.157$, $T = 295$ K.

Source of material

The title compound was isolated from *Duchesnea indica Focke* collected from the JiangXi Province, China. The leaves and stems (50 kg dry weight) were powdered and exhaustively extracted with 70 % ethanol. The ethanol extract was filtered and concentrated in vacuo to give a dark brown aqueous suspension, which was diluted with water to 5 L and partitioned sequentially with petroleum ether, ethyl acetate and *n*-butanol (3 × 5 L). *n*-Butanol solution (900 g) was subjected to silica gel column chromatography using gradient elution (dichloromethane/methanol, 20:1 to 1:2). The fraction eluted by dichloromethane/methanol (5:1) was purified by HP20 column chromatography and eluted with water/ethanol (100:0 to 5:95). The fraction of water/ethanol (20:80) was

further purified by ODS column chromatography using methanol/water (2:3) as eluent to afford brevifolincarboxylic acid. The compound was crystallized from methanol to give pale yellow crystals (50 mg). Single crystals suitable for X-ray analysis were obtained by slow evaporation from pyridine at room temperature.

Discussion

Brevifolincarboxylic acid was first isolated from *Duchesnea indica Focke* and showed a strong cytotoxic activity [1]. The antiviral agents containing brevifolincarboxylic acid as an active ingredient are claimed [2]. The main molecule is composed of two six-membered rings and one five-membered ring. Three attached hydroxyl groups form intramolecular hydrogen bonds with *ortho* oxygen atoms with $d(O2-H2\cdots O1) = 2.733$ Å, $\angle O2-H2\cdots O1 = 113.1^\circ$ and $d(O3-H3\cdots O2) = 2.707$ Å, $\angle O3-H3\cdots O2 = 111.2^\circ$. There is also an intermolecular hydrogen bond between two main molecules, with $d(C12\cdots O1) = 3.518$ Å and $\angle C12-H12B\cdots O1 = 117.0^\circ$. The acid molecule is a planar with an average deviation of 0.027 Å. The five-membered ring C5–C6/C11–C13 adopt an *envelope* conformation with C12 displaced by $-0.100(5)$ Å from the least-squares plane.

Table 1. Data collection and handling.

Crystal:	pale yellow, prismatic, size $0.25 \times 0.40 \times 0.50$ mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ :	0.93 cm ⁻¹
Diffractometer, scan mode:	Enraf-Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50.34°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7373, 6310
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 2473
$N(param)_{refined}$:	465
Programs:	SHELXS-97 [3], SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U_{iso}
H(1)	2i	0.2264	1.1307	-0.0542	0.066
H(11)	2i	-0.1549	0.8561	0.2325	0.060
H(12A)	2i	-0.0237	0.7113	0.3499	0.079
H(12B)	2i	-0.0537	0.6758	0.2606	0.079
H(1A)	2i	0.1077	1.3139	-0.0834	0.110
H(2)	2i	-0.1822	1.3360	0.0577	0.117
H(3)	2i	-0.2543	1.1402	0.2179	0.100
H(6)	2i	-0.2825	0.8834	0.3786	0.109
H(22)	2i	0.3909	1.2071	-0.1882	0.144
H(23)	2i	0.5678	1.2622	-0.3082	0.184
H(24)	2i	0.5362	1.4460	-0.3781	0.170

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Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(25)	2i	0.3285	1.5633	-0.3328	0.152
H(26)	2i	0.1622	1.4999	-0.2111	0.120
H(28)	2i	-0.4812	1.3000	0.1877	0.109
H(29)	2i	-0.6856	1.3956	0.2742	0.152
H(30)	2i	-0.7508	1.3781	0.4472	0.179
H(31)	2i	-0.6024	1.2727	0.5260	0.144
H(32)	2i	-0.3937	1.1853	0.4297	0.106
H(34)	2i	0.9032	0.5304	-0.0894	0.104
H(35)	2i	0.7995	0.7118	-0.1265	0.114
H(36)	2i	0.5762	0.7762	-0.0528	0.123
H(37)	2i	0.4621	0.6540	0.0598	0.126

Table 2. Continued.

Atom	Site	x	y	z	U _{iso}
H(38)	2i	0.5748	0.4737	0.0928	0.108
H(40)	2i	0.1393	0.0153	0.4196	0.091
H(41)	2i	0.1829	0.0122	0.2511	0.104
H(42)	2i	0.3943	0.0131	0.1449	0.100
H(43)	2i	0.5553	0.0099	0.2123	0.086
H(44)	2i	0.5022	0.0083	0.3815	0.081
H(46)	2i	-0.0490	0.5782	0.7007	0.175
H(47)	2i	-0.0068	0.7280	0.7406	0.200
H(48)	2i	0.1151	0.8359	0.6153	0.223
H(49)	2i	0.2005	0.7712	0.4591	0.208
H(50)	2i	0.1679	0.6155	0.4294	0.183

Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
C(1)	2i	0.1474(3)	1.1215(3)	-0.0066(2)	0.054(2)	0.064(2)	0.037(2)	-0.016(2)	-0.005(1)	0.000(2)
C(2)	2i	0.0369(3)	1.2102(3)	0.0074(2)	0.062(2)	0.050(2)	0.046(2)	-0.020(2)	-0.013(2)	0.007(2)
C(3)	2i	-0.0824(3)	1.1969(3)	0.0783(2)	0.052(2)	0.052(2)	0.049(2)	-0.009(2)	-0.011(2)	0.000(2)
C(4)	2i	-0.0884(3)	1.0942(2)	0.1356(2)	0.049(2)	0.049(2)	0.041(2)	-0.014(2)	-0.005(1)	-0.003(1)
C(5)	2i	0.0270(3)	0.8943(2)	0.1746(2)	0.053(2)	0.049(2)	0.035(2)	-0.012(2)	-0.010(1)	-0.005(1)
C(6)	2i	0.1398(3)	0.8142(3)	0.1559(2)	0.062(2)	0.057(2)	0.042(2)	-0.010(2)	-0.008(2)	-0.001(2)
O(8)	2i	0.2586(2)	0.8262(2)	0.0878(2)	0.062(2)	0.065(2)	0.055(1)	-0.000(1)	-0.003(1)	-0.001(1)
C(8)	2i	0.2641(3)	0.9289(3)	0.0315(2)	0.062(2)	0.070(2)	0.041(2)	-0.008(2)	-0.009(2)	-0.001(2)
C(9)	2i	0.0231(3)	1.0038(2)	0.1211(2)	0.050(2)	0.049(2)	0.035(2)	-0.012(2)	-0.010(1)	0.001(1)
C(10)	2i	0.1427(3)	1.0190(2)	0.0489(2)	0.051(2)	0.053(2)	0.035(2)	-0.011(2)	-0.010(2)	-0.002(1)
C(11)	2i	-0.0808(3)	0.8516(2)	0.2564(2)	0.056(2)	0.052(2)	0.042(2)	-0.019(2)	-0.013(2)	-0.001(1)
C(12)	2i	-0.0140(3)	0.7263(3)	0.2791(2)	0.083(2)	0.058(2)	0.056(2)	-0.024(2)	-0.020(2)	0.006(2)
C(13)	2i	0.1304(4)	0.7088(3)	0.2176(2)	0.082(3)	0.054(2)	0.054(2)	-0.005(2)	-0.017(2)	-0.004(2)
O(1)	2i	0.0331(2)	1.3121(2)	-0.0461(2)	0.071(2)	0.060(1)	0.073(2)	-0.020(1)	-0.008(1)	0.016(1)
O(2)	2i	-0.1951(2)	1.2800(2)	0.0961(2)	0.067(2)	0.057(1)	0.082(2)	-0.004(1)	-0.005(1)	0.013(1)
O(3)	2i	-0.2021(2)	1.0784(2)	0.2045(2)	0.051(1)	0.058(1)	0.066(1)	-0.008(1)	0.002(1)	0.008(1)
O(4)	2i	0.3709(2)	0.9359(2)	-0.0262(2)	0.057(2)	0.105(2)	0.066(2)	-0.005(1)	0.011(1)	0.010(1)
C(14)	2i	-0.1280(3)	0.9152(3)	0.3498(2)	0.054(2)	0.061(2)	0.042(2)	-0.021(2)	-0.007(2)	0.006(2)
O(5)	2i	-0.0579(2)	0.9554(2)	0.3738(2)	0.072(2)	0.099(2)	0.056(1)	-0.040(1)	-0.006(1)	-0.020(1)
O(6)	2i	-0.2500(2)	0.9161(2)	0.4058(2)	0.060(1)	0.104(2)	0.056(1)	-0.038(1)	-0.007(1)	-0.002(1)
O(7)	2i	0.2176(3)	0.6246(2)	0.2208(2)	0.103(2)	0.068(2)	0.092(2)	0.011(2)	-0.018(2)	0.013(1)
N(21)	2i	0.2598(3)	1.3464(3)	-0.1902(2)	0.085(2)	0.076(2)	0.073(2)	-0.036(2)	-0.010(2)	0.000(2)
C(22)	2i	0.3775(5)	1.2807(4)	-0.2180(4)	0.113(4)	0.086(3)	0.122(4)	-0.032(3)	0.017(3)	-0.009(3)
C(23)	2i	0.4853(5)	1.3130(5)	-0.2895(4)	0.112(4)	0.143(5)	0.152(5)	-0.046(4)	0.036(4)	-0.009(4)
C(24)	2i	0.4662(6)	1.4203(6)	-0.3311(4)	0.140(5)	0.155(6)	0.117(4)	-0.093(5)	0.013(4)	0.009(4)
C(25)	2i	0.3448(6)	1.4896(5)	-0.3036(4)	0.143(5)	0.122(4)	0.120(4)	-0.076(4)	-0.035(4)	0.039(3)
C(26)	2i	0.2451(4)	1.4500(4)	-0.2316(3)	0.104(3)	0.095(3)	0.103(3)	-0.043(3)	-0.033(3)	0.026(3)
N(27)	2i	-0.4181(3)	1.2338(2)	0.2987(2)	0.066(2)	0.067(2)	0.077(2)	-0.021(2)	-0.011(2)	0.007(2)
C(28)	2i	-0.5053(4)	1.2950(3)	0.2573(3)	0.084(3)	0.082(3)	0.099(3)	-0.021(2)	-0.030(3)	0.017(2)
C(29)	2i	-0.6281(5)	1.3514(4)	0.3079(5)	0.075(4)	0.108(4)	0.179(6)	0.000(3)	-0.042(4)	0.001(4)
C(30)	2i	-0.6659(6)	1.3420(5)	0.4100(6)	0.094(4)	0.124(5)	0.181(7)	-0.008(4)	0.017(5)	-0.050(5)
C(31)	2i	-0.5790(6)	1.2799(5)	0.4565(4)	0.128(5)	0.130(5)	0.086(3)	-0.057(4)	0.015(4)	-0.028(3)
C(32)	2i	-0.4547(4)	1.2272(3)	0.3981(3)	0.091(3)	0.087(3)	0.093(3)	-0.034(2)	-0.031(3)	0.006(2)
N(33)	2i	0.7498(3)	0.4846(2)	0.0068(2)	0.078(2)	0.061(2)	0.070(2)	-0.006(2)	-0.022(2)	0.001(2)
C(34)	2i	0.8129(4)	0.5556(3)	-0.0581(3)	0.088(3)	0.074(3)	0.075(3)	-0.011(2)	-0.007(2)	0.003(2)
C(35)	2i	0.7513(5)	0.6642(3)	-0.0811(3)	0.125(4)	0.061(3)	0.082(3)	-0.017(3)	-0.023(3)	0.010(2)
C(36)	2i	0.6198(5)	0.7024(4)	-0.0377(4)	0.118(4)	0.071(3)	0.108(4)	0.012(3)	-0.053(3)	-0.003(3)
C(37)	2i	0.5527(4)	0.6305(4)	0.0285(4)	0.078(3)	0.094(3)	0.129(4)	0.000(3)	-0.037(3)	0.003(3)
C(38)	2i	0.6214(4)	0.5228(3)	0.0479(3)	0.082(3)	0.081(3)	0.102(3)	-0.021(2)	-0.030(3)	0.013(2)
N(39)	2i	0.3160(3)	0.0131(2)	0.4169(2)	0.062(2)	0.090(2)	0.049(2)	-0.024(2)	-0.007(2)	-0.003(1)
C(40)	2i	0.2246(4)	0.0139(3)	0.3771(3)	0.059(2)	0.107(3)	0.055(2)	-0.029(2)	-0.006(2)	0.002(2)
C(41)	2i	0.2493(4)	0.0127(3)	0.2764(3)	0.079(3)	0.132(3)	0.061(2)	-0.040(2)	-0.023(2)	-0.011(2)
C(42)	2i	0.3747(4)	0.0123(3)	0.2138(3)	0.091(3)	0.105(3)	0.052(2)	-0.041(2)	-0.006(2)	-0.010(2)
C(43)	2i	0.4695(4)	0.0107(3)	0.2533(3)	0.064(2)	0.079(2)	0.061(2)	-0.027(2)	0.008(2)	-0.016(2)
C(44)	2i	0.4364(3)	0.0105(3)	0.3546(2)	0.059(2)	0.084(2)	0.059(2)	-0.024(2)	-0.009(2)	-0.013(2)
N(45)	2i	0.0566(8)	0.5816(5)	0.5619(9)	0.163(6)	0.182(6)	0.191(7)	-0.053(5)	-0.065(5)	-0.023(6)
C(46)	2i	0.0056(5)	0.6178(8)	0.6512(8)	0.095(4)	0.147(6)	0.143(6)	-0.031(4)	0.010(4)	0.028(5)
C(47)	2i	0.0275(9)	0.708(1)	0.6749(6)	0.148(7)	0.184(9)	0.144(6)	0.001(6)	-0.018(5)	-0.082(6)
C(48)	2i	0.1004(9)	0.7700(6)	0.602(1)	0.128(6)	0.105(5)	0.34(1)	-0.008(4)	-0.068(7)	-0.097(8)
C(49)	2i	0.1497(7)	0.7312(8)	0.5113(9)	0.140(5)	0.089(5)	0.23(1)	-0.029(5)	-0.011(6)	0.049(5)
C(50)	2i	0.1292(9)	0.6403(9)	0.4937(5)	0.194(8)	0.172(8)	0.080(4)	-0.023(6)	-0.052(5)	0.006(4)

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