Crystal structure of (*Z*)-*N*-(17-(1-(dimethylamino)ethyl)-10,13-dimethyl-4-oxo-4,5,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1*H*-cyclopenta-[*a*]phenanthren-3-yl)-3-methylbut-2-enimidic acid dichloromethane monosolvate, $C_{29}H_{45}Cl_2N_2O_2$

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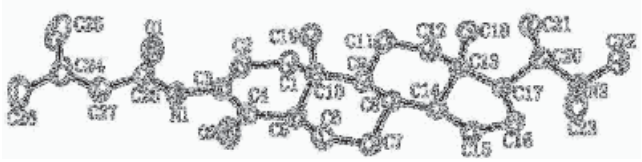

Abstract

$C_{29}H_{45}Cl_2N_2O_2$, monoclinic, $P2_1$ (no. 4), a = 8.4429(4) Å, b = 19.6901(9) Å, c = 9.0259(4) Å, β = 100.700(2)°, V = 1474.4 Å^3, Z = 2, $R_{gt}(F)$ = 0.0492, $wR_{ref}(F^2)$ = 0.1437, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.20×0.21×0.23 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.47 cm^{-1}
Diffractometer, scan mode:	Bruker Smart APEX II, φ and ω
$2\theta_{max}$:	51.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	17025, 5528
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5037
$N(param)_{refined}$:	316
Programs:	SHELX [3]

Source of material

The air-dried and powdered roots of Sarcococca ruscifolia were extracted with 90% ethanol under reflux two times. The combined extracts were concentrated, and suspended in water. The suspension was partitioned with chloroform to obtain the crude alkaloidal fraction [1]. The title compound pachysamine M was obtained from the crude alkaloidal fraction by silica gel column chromatography using petroleum ether/ethyl acetate/diethylamine (100:2:1) as the eluent and recrystallization with dichloromethane [2].

Experimental details

The hydrogen atoms were assigned with common isotropic displacement factors U_{iso}(H) = 1.2U_{eq}(C) and 1.5U_{eq}(O), respectively, and included in the final refinement with d(C–H) = 0.96 - 0.98 Å. The Flack parameter is –0.04(8).

Discussion

The title compound crystallizes with one molecule of the title compound and one dichlormethane molecule in the asymmetric unit of the space group $P2_1$ (figure). Geometric parameters of the title structure are in the usual ranges. The compound contains one carbonyl, one amide, two double bonds and one dimethylamino group. The C4–O2 distance is 1.217(3) Å. The amide was confirmed by the distance of 1.225(4) Å, 1.368(4) Å C28–O1 and C28–N1, respectively. The C2–C3 and C24–C27 distance is 1.329(4) Å and 1.334(4) Å. The dimethylamino group was confirmed by the distance of 1.225(4) Å, 1.368(4) Å, C28–O1 and C28–N1, respectively. The compound contains three six-membered rings and one five-membered ring. The intermolecular connection was achieved by van der Waals interactions only.

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1A)	2*a*	0.4705	0.8642	–0.3573	0.043
H(1B)	2*a*	0.3858	0.7944	–0.4003	0.043
H(2)	2*a*	0.3110	0.8871	–0.5908	0.045
H(5)	2*a*	0.1222	0.7738	–0.3172	0.039
H(6A)	2*a*	0.0261	0.8545	–0.0921	0.048
H(6B)	2*a*	–0.0745	0.7937	–0.1742	0.048
H(7A)	2*a*	0.1228	0.7169	–0.0710	0.047
H(7B)	2*a*	0.0753	0.7594	0.0610	0.047
H(8)	2*a*	0.3063	0.8259	0.0789	0.035
H(9)	2*a*	0.3821	0.7484	–0.1636	0.035
H(11A)	2*a*	0.5761	0.8543	–0.0182	0.045
H(11B)	2*a*	0.6167	0.8102	–0.1508	0.045
H(12A)	2*a*	0.6607	0.7146	0.0030	0.043
H(12B)	2*a*	0.7661	0.7738	0.0856	0.043
H(14)	2*a*	0.4005	0.6894	0.0661	0.035
H(15A)	2*a*	0.2424	0.6770	0.2410	0.040
H(15B)	2*a*	0.2997	0.7465	0.3207	0.040
H(16A)	2*a*	0.4587	0.6229	0.3652	0.041
H(16B)	2*a*	0.4945	0.6880	0.4673	0.041
H(17)	2*a*	0.6415	0.6486	0.2275	0.036
H(18A)	2*a*	0.5386	0.8080	0.3689	0.053
H(18B)	2*a*	0.5383	0.8512	0.2229	0.053
H(18C)	2*a*	0.7016	0.8250	0.3175	0.053
H(19A)	2*a*	0.4136	0.9303	–0.1429	0.059
H(19B)	2*a*	0.2544	0.9177	–0.0811	0.059
H(19C)	2*a*	0.2460	0.9461	–0.2446	0.059
H(20)	2*a*	0.7653	0.7300	0.4789	0.038
H(21A)	2*a*	1.0234	0.7101	0.4303	0.065
H(21B)	2*a*	0.9186	0.7502	0.2970	0.065
H(21C)	2*a*	0.9491	0.6723	0.2807	0.065
H(22A)	2*a*	0.8659	0.6796	0.7007	0.084
H(22B)	2*a*	1.0085	0.6449	0.6403	0.084
H(22C)	2*a*	0.8911	0.6009	0.7163	0.084
H(23A)	2*a*	0.7758	0.5639	0.3400	0.090
H(23B)	2*a*	0.8371	0.5310	0.4984	0.090
H(23C)	2*a*	0.9542	0.5753	0.4225	0.090
H(25A)	2*a*	–0.0147	1.0644	–1.1310	0.111
H(25B)	2*a*	0.0946	1.0038	–1.0613	0.111
H(25C)	2*a*	0.0735	1.0673	–0.9621	0.111
H(26A)	2*a*	–0.2670	1.0520	–1.1700	0.121
H(26B)	2*a*	–0.3465	1.0455	–1.0269	0.121

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Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(26C)	2*a*	–0.3347	0.9818	–1.1285	0.121
H(27)	2*a*	–0.2118	0.9509	–0.8538	0.059
H(30A)	2*a*	0.5944	0.5129	–0.2644	0.067
H(30B)	2*a*	0.5917	0.5659	–0.3947	0.067

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	2*a*	0.3669(3)	0.8414(1)	–0.3763(3)	0.037(1)	0.042(1)	0.033(1)	0.007(1)	0.015(1)	0.007(1)
C(2)	2*a*	0.2629(3)	0.8737(1)	–0.5108(3)	0.047(1)	0.037(1)	0.030(1)	0.004(1)	0.013(1)	0.004(1)
C(3)	2*a*	0.1056(3)	0.8840(1)	–0.5215(3)	0.040(1)	0.030(1)	0.028(1)	0.001(1)	0.0039(9)	0.0010(9)
C(4)	2*a*	0.0225(3)	0.8628(1)	–0.3975(3)	0.035(1)	0.041(1)	0.033(1)	–0.002(1)	0.0006(9)	0.005(1)
C(5)	2*a*	0.1181(3)	0.8193(1)	–0.2737(3)	0.033(1)	0.034(1)	0.033(1)	–0.000(1)	0.0074(9)	0.005(1)
C(6)	2*a*	0.0339(3)	0.8108(2)	–0.1397(3)	0.028(1)	0.053(2)	0.039(1)	0.001(1)	0.008(1)	0.009(1)
C(7)	2*a*	0.1268(3)	0.7618(2)	–0.0264(3)	0.029(1)	0.052(2)	0.038(1)	–0.004(1)	0.011(1)	0.013(1)
C(8)	2*a*	0.3030(3)	0.7828(1)	0.0243(3)	0.027(1)	0.031(1)	0.030(1)	0.0005(9)	0.0068(9)	0.0016(9)
C(9)	2*a*	0.3866(3)	0.7926(1)	–0.1129(3)	0.030(1)	0.030(1)	0.029(1)	0.0019(9)	0.0076(9)	0.0018(9)
C(10)	2*a*	0.2956(3)	0.8433(1)	–0.2308(2)	0.030(1)	0.028(1)	0.028(1)	0.0021(9)	0.0066(8)	0.0007(9)
C(11)	2*a*	0.5660(3)	0.8093(2)	–0.0628(3)	0.032(1)	0.049(2)	0.035(1)	–0.001(1)	0.011(1)	0.015(1)
C(12)	2*a*	0.6563(3)	0.7582(2)	0.0520(3)	0.028(1)	0.046(2)	0.035(1)	0.005(1)	0.0109(9)	0.008(1)
C(13)	2*a*	0.5750(3)	0.7494(1)	0.1892(2)	0.029(1)	0.025(1)	0.029(1)	0.0009(9)	0.0091(8)	0.0017(9)
C(14)	2*a*	0.3975(3)	0.7302(1)	0.1275(3)	0.031(1)	0.031(1)	0.027(1)	–0.0036(9)	0.0099(9)	0.0005(9)
C(15)	2*a*	0.3330(3)	0.7077(1)	0.2678(3)	0.030(1)	0.039(1)	0.034(1)	–0.003(1)	0.0097(9)	0.005(1)
C(16)	2*a*	0.4778(3)	0.6715(1)	0.3644(3)	0.034(1)	0.037(1)	0.033(1)	–0.005(1)	0.0076(9)	0.008(1)
C(17)	2*a*	0.6276(3)	0.6869(1)	0.2931(2)	0.035(1)	0.027(1)	0.028(1)	–0.0014(9)	0.0085(9)	0.0005(9)
C(18)	2*a*	0.5897(3)	0.8144(1)	0.2834(3)	0.037(1)	0.031(1)	0.038(1)	–0.000(1)	0.005(1)	0.001(1)
C(19)	2*a*	0.3031(3)	0.9162(1)	–0.1690(3)	0.048(1)	0.030(1)	0.040(1)	0.000(1)	0.005(1)	0.003(1)
C(20)	2*a*	0.7808(3)	0.6918(1)	0.4134(3)	0.034(1)	0.029(1)	0.032(1)	0.0019(9)	0.0096(9)	0.0012(9)
C(21)	2*a*	0.9320(3)	0.7076(2)	0.3494(3)	0.032(1)	0.057(2)	0.042(1)	–0.000(1)	0.008(1)	0.008(1)
C(22)	2*a*	0.8990(4)	0.6396(2)	0.6536(4)	0.060(2)	0.056(2)	0.046(2)	–0.004(2)	–0.005(1)	0.012(1)
C(23)	2*a*	0.8449(5)	0.5700(2)	0.4363(4)	0.082(2)	0.043(2)	0.054(2)	0.023(2)	0.008(2)	0.006(1)
C(24)	2*a*	–0.1201(4)	1.0056(2)	–0.9946(3)	0.079(2)	0.039(2)	0.033(1)	0.010(1)	0.003(1)	–0.000(1)
C(25)	2*a*	0.0207(5)	1.0381(2)	–1.0414(4)	0.095(3)	0.072(2)	0.065(2)	0.027(2)	0.041(2)	0.032(2)
C(26)	2*a*	–0.2817(6)	1.0228(2)	–1.0885(4)	0.102(3)	0.071(2)	0.057(2)	0.007(2)	–0.019(2)	0.014(2)
C(27)	2*a*	–0.1134(4)	0.9653(2)	–0.8749(3)	0.058(2)	0.045(2)	0.039(1)	–0.005(1)	–0.002(1)	0.006(1)
C(28)	2*a*	0.0327(4)	0.9406(1)	–0.7715(3)	0.059(2)	0.029(1)	0.031(1)	–0.001(1)	0.004(1)	0.001(1)
O(2)	2*a*	–0.1180(2)	0.8777(1)	–0.4011(2)	0.037(1)	0.089(2)	0.048(1)	0.010(1)	0.0064(8)	0.026(1)
C(30)	2*a*	0.5252(4)	0.5425(2)	–0.3340(4)	0.061(2)	0.053(2)	0.056(2)	–0.006(2)	0.014(2)	0.002(1)
O(1)	2*a*	0.1701(3)	0.9425(1)	–0.7970(2)	0.057(1)	0.067(1)	0.040(1)	0.009(1)	0.0149(9)	0.016(1)
N(1)	2*a*	–0.0024(3)	0.9144(1)	–0.6410(2)	0.046(1)	0.037(1)	0.033(1)	0.000(1)	0.0018(9)	0.0049(9)
N(2)	2*a*	0.7958(3)	0.6300(1)	0.5086(2)	0.041(1)	0.032(1)	0.036(1)	0.0018(9)	0.0064(9)	0.0061(8)
Cl(1)	2*a*	0.4358(3)	0.60225(8)	–0.2320(2)	0.192(2)	0.095(1)	0.134(1)	–0.028(1)	0.095(1)	–0.0494(9)
Cl(2)	2*a*	0.3799(1)	0.49350(5)	–0.4520(1)	0.0783(6)	0.0680(6)	0.0685(5)	–0.0207(5)	0.0097(4)	–0.0006(4)

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