Crystal structure of songorine chloride trihydrate, $[C_{22}H_{32}NO_3]Cl \cdot 3\ H_2O$

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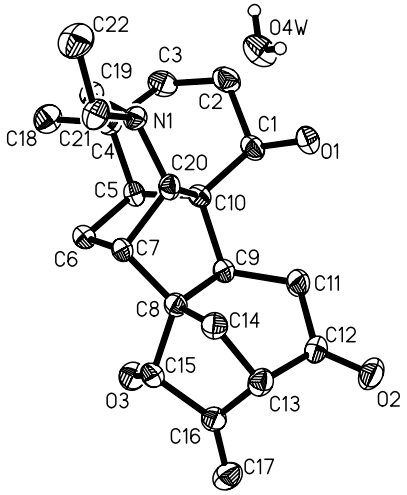

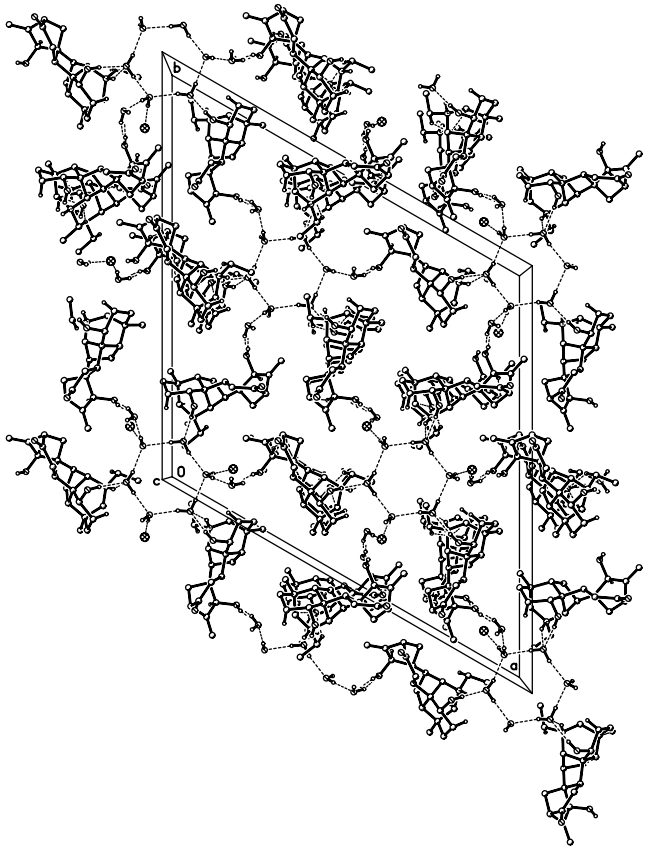

Abstract

$C_{22}H_{38}ClNO_6$, trigonal, *R*3 (no. 146), a = 28.293(2) Å, c = 7.4204(7) Å, V = 5144.3 Å^3, Z = 9, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.137, T = 296 K.

Source of material

Songorine, a C_{20}-diterpenoid alkaloid is isolated from the chloroform extract of the roots of *Aconitum szechenyianum Gay*, wetted by 10% ammonium hydroxide solution before. The purified compound is dissolved in a 1% hydrochloric acid solution at first. Then the solution is adjusted to pH 6.5 with a 10% NaOH solution. Colorless single crystals were obtained at room temperature for days by slowly volatilizing the solvent.

Experimental details

Water H atoms were located in a difference Fourier map and refined with restrained O—H bond lengths (0.85(1) Å) and fixed isotropic displacement parameters (0.08 Å^2). Other H atoms were placed at calculated positions and refined using a riding model with O—H distances restrained to 0.82 Å, C—H distances restrained to 0.93 Å and N—H distance restrained to 0.91 Å.

Discussion

Songorine belongs to the class of C_{20}-diterpenoid alkaloids, a large group of biologically active compounds of naturally occurrence. As a diterpenoid alkaloid, songorine has been found to enhance both the orthodromic population spike and field excitatory postsynaptic potential (EPSP) in CA1 region of hippocampal slices [1]. Recently, it was indicated that songorine can enhance the excitatory synaptic transmission in rat hippocampus, and was proved that songorine is a novel non-competitive antagonist at the $GABA_A$ receptor in rat brain [2].

In the title crystal structure, there are one songorine cation, one chloride ion and 3 crystal water molecules (figure top). The bond length of C12—O2 is 1.202 Å, and the angles of O2–C12–C11, O2–C12–C13 and C11–C12–C13 are 121.8(4)°, 122.8(4)° and 115.4(3)°, respectively, which indicates that the C12 and O2 are sp^2 hybridized, and C11, C12, C13 and O2 atoms are in a plane. The bond length of C17—C16 is 1.322(6) Å. The angles of C13–C16–C15, C17–C16–C15 and C17–C16–C13 are 108.0(3)°, 126.2(4)° and 125.5(4)°, respectively, which show an exo-methylene attached to C16.

The songorine cations are interlinked by hydrogen bonds (figure bottom). Intra-molecular hydrogen bonds O6W–H6WA···Cl1 and O5W–H5WA···Cl1 link the Cl^-, crystal water O5W and O6W together. The hydrogen bonds C2–H2A···O4W, O1–H1···O4W, N1–H1N···O4W fix the crystal water O4W to songorine. The intermolecular hydrogen bonds O6W–H6WB···O5W (symmetric code: $x,y,z-1$) and C21–H21A···O1, C19–H19B···O1, C15–H15···O2 (symmetric code: $x,y,z+1$), link the songorine molecules along [001]. The hydrogen bonds O5W–

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H5WB···O3 (symmetric code: *x, y*+1,*z*) and O3–H3···O5W (symmetric code: *x*,*y*–1,*z*) connect the molecules along [010]. The hydrogen bond O4W–H4WA···O6W (symmetric code: –*y*+2/3, *x*–*y*+1/3, *z*–2/3) links the molecules into triangles at the *ab* plane. Thus the hydrogen bonds form a network structure (figure, bottom). Based on the parameter Flack [3], x = 0.00(9), the absolute configuration of the ten chiral carbons in the title crystal structure are identified as 1*R*, 4*S*, 5*S*, 7*S*, 8*S*, 9*S*, 10*R*, 13*S*, 15*R* and 20*S* .

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.14 × 0.21 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.05 cm^{-1}
Diffractometer, scan mode:	Bruker SMART, φ/ω
$2\theta_{max}$:	50.18°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8777, 4031
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2777
$N(param)_{refined}$:	321
Programs:	SHELXS-97 [4], SHELXL-97 [5], SHELXTL [6]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1N)	9*b*	0.3836	0.8233	0.0177	0.051
H(1)	9*b*	0.4050	0.8492	0.3662	0.082
H(3)	9*b*	0.5564	1.0917	0.0285	0.078
H(1A)	9*b*	0.4001	0.9386	0.3521	0.044
H(2A)	9*b*	0.3313	0.8353	0.2019	0.064
H(2B)	9*b*	0.3152	0.8667	0.3401	0.064
H(3A)	9*b*	0.3196	0.9261	0.1231	0.056
H(3B)	9*b*	0.2827	0.8670	0.0482	0.078
H(5)	9*b*	0.4138	0.9857	0.0073	0.046
H(6A)	9*b*	0.4360	0.9465	–0.2981	0.050
H(6B)	9*b*	0.4769	1.0035	–0.2100	0.039
H(7)	9*b*	0.5051	0.9286	–0.2143	0.057
H(9)	9*b*	0.4942	1.0142	0.1701	0.044
H(11A)	9*b*	0.5039	0.9931	0.4497	0.052
H(11B)	9*b*	0.5013	0.9386	0.3939	0.105
H(13)	9*b*	0.6433	1.0091	0.2291	0.074
H(14A)	9*b*	0.5912	0.9574	–0.0169	0.084
H(14B)	9*b*	0.5520	0.9269	0.1474	0.059
H(15)	9*b*	0.5899	1.0371	–0.1566	0.053

Table 2. Continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(17A)	9*b*	0.6734	1.1170	0.2481	0.080
H(17B)	9*b*	0.6528	1.1373	0.0779	0.091
H(18A)	9*b*	0.3295	0.9579	–0.1888	0.089
H(18B)	9*b*	0.2957	0.8979	–0.2603	0.110
H(18C)	9*b*	0.3544	0.9370	–0.3362	0.074
H(19A)	9*b*	0.3229	0.8265	–0.1419	0.049
H(19B)	9*b*	0.3654	0.8639	–0.2861	0.038
H(20)	9*b*	0.4708	0.8827	0.0840	0.040
H(21A)	9*b*	0.4321	0.8393	–0.3130	0.080
H(21B)	9*b*	0.4471	0.8137	–0.1512	0.082
H(22A)	9*b*	0.3845	0.7451	–0.3367	0.075
H(22B)	9*b*	0.3431	0.7660	–0.3087	0.100
H(22C)	9*b*	0.3582	0.7404	–0.1470	0.081
H(4WA)	9*b*	0.395(2)	0.760(2)	0.266(8)	0.080
H(4WB)	9*b*	0.3455(4)	0.759(2)	0.24(1)	0.080
H(5WA)	9*b*	0.575(2)	0.147(2)	0.319(5)	0.080
H(5WB)	9*b*	0.549(2)	0.118(1)	0.163(5)	0.080
H(6WA)	9*b*	0.613(2)	0.205(2)	0.861(3)	0.080
H(6WB)	9*b*	0.604(2)	0.203(2)	1.048(4)	0.080

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}
N(1)	9*b*	0.4010(1)	0.8472(1)	–0.0739(4)	0.039(2)	0.035(2)	0.036(2)	0.017(1)	–0.001(1)	–0.005(1)
O(1)	9*b*	0.4136(1)	0.8794(1)	0.4073(3)	0.063(2)	0.052(2)	0.039(2)	0.022(1)	0.002(1)	0.010(1)
O(3)	9*b*	0.5509(1)	1.0736(1)	–0.0633(4)	0.061(2)	0.044(2)	0.053(2)	0.027(1)	–0.007(1)	–0.001(1)
O(2)	9*b*	0.6007(1)	1.0189(1)	0.5226(4)	0.060(2)	0.090(2)	0.043(2)	0.031(2)	–0.012(2)	–0.013(2)
C(1)	9*b*	0.3978(2)	0.9076(2)	0.2854(5)	0.047(2)	0.045(2)	0.036(2)	0.021(2)	0.011(2)	0.005(2)
C(2)	9*b*	0.3375(2)	0.8711(2)	0.2353(6)	0.043(2)	0.060(3)	0.053(3)	0.022(2)	0.014(2)	0.010(2)
C(3)	9*b*	0.3198(2)	0.8937(2)	0.0821(6)	0.041(2)	0.057(3)	0.060(3)	0.026(2)	0.010(2)	0.004(2)
C(4)	9*b*	0.3562(2)	0.90780(2)	–0.0828(5)	0.038(2)	0.043(2)	0.043(2)	0.021(2)	–0.001(2)	–0.000(2)
C(5)	9*b*	0.4137(1)	0.9523(1)	–0.0295(5)	0.044(2)	0.041(2)	0.033(2)	0.024(2)	0.002(2)	0.005(2)
C(6)	9*b*	0.4549(1)	0.9646(2)	–0.1880(5)	0.039(2)	0.042(2)	0.036(2)	0.017(2)	0.003(2)	0.005(2)
C(7)	9*b*	0.4900(1)	0.9411(1)	–0.1190(5)	0.036(2)	0.040(2)	0.029(2)	0.018(2)	0.005(2)	–0.000(2)
C(8)	9*b*	0.5320(1)	0.9796(2)	0.0173(5)	0.034(2)	0.044(2)	0.034(2)	0.020(2)	0.004(2)	0.000(2)
C(9)	9*b*	0.4948(1)	0.9800(1)	0.1764(5)	0.037(2)	0.041(2)	0.031(2)	0.018(2)	0.003(2)	0.000(2)
C(10)	9*b*	0.4363(1)	0.9318(1)	0.1235(5)	0.035(2)	0.037(2)	0.035(2)	0.018(2)	0.006(2)	0.003(2)
C(11)	9*b*	0.5169(2)	0.9767(2)	0.3621(5)	0.044(2)	0.070(3)	0.032(2)	0.024(2)	0.006(2)	–0.001(2)
C(12)	9*b*	0.5782(2)	1.0039(2)	0.3796(5)	0.048(2)	0.053(2)	0.036(2)	0.025(2)	–0.007(2)	–0.002(2)
C(13)	9*b*	0.6086(2)	1.0080(2)	0.2069(5)	0.041(2)	0.063(3)	0.043(2)	0.029(2)	–0.002(2)	–0.005(2)
C(14)	9*b*	0.5711(2)	0.9612(2)	0.0825(5)	0.040(2)	0.052(2)	0.044(2)	0.025(2)	0.002(2)	0.001(2)
C(15)	9*b*	0.5733(2)	1.0385(2)	–0.0424(5)	0.042(2)	0.047(2)	0.035(2)	0.019(2)	0.008(2)	0.003(2)
C(16)	9*b*	0.6161(1)	1.0578(2)	0.1053(5)	0.036(2)	0.049(2)	0.040(2)	0.020(2)	0.002(2)	–0.001(2)
C(17)	9*b*	0.6509(2)	1.1091(2)	0.1480(6)	0.046(2)	0.064(3)	0.063(3)	0.025(2)	–0.010(2)	–0.006(2)
C(18)	9*b*	0.3317(2)	0.9270(2)	–0.2309(6)	0.053(3)	0.060(3)	0.060(3)	0.035(2)	–0.007(2)	0.000(2)
C(19)	9*b*	0.3585(2)	0.8586(2)	–0.1576(5)	0.038(2)	0.048(2)	0.039(2)	0.019(2)	–0.004(2)	–0.001(2)
C(20)	9*b*	0.4506(2)	0.8955(1)	0.0108(5)	0.041(2)	0.047(2)	0.030(3)	0.024(2)	0.001(2)	–0.002(2)
C(21)	9*b*	0.4180(2)	0.8176(2)	–0.2044(6)	0.056(2)	0.053(2)	0.046(3)	0.034(2)	–0.005(2)	–0.010(2)
C(22)	9*b*	0.3718(2)	0.7623(2)	–0.2536(8)	0.090(4)	0.056(3)	0.078(4)	0.039(3)	–0.008(3)	–0.021(3)
O(4W)	9*b*	0.3800(2)	0.7789(1)	0.2364(5)	0.072(2)	0.063(2)	0.074(2)	0.036(2)	0.014(2)	0.019(2)
O(5W)	9*b*	0.5592(2)	0.1469(2)	0.2216(6)	0.116(3)	0.124(4)	0.076(3)	0.087(3)	–0.014(2)	–0.024(2)
Cl(1)	9*b*	0.59713(6)	0.15293(6)	0.6269(2)	0.089(1)	0.098(1)	0.0760(9)	0.0408(8)	0.0127(8)	0.0247(8)
O(6W)	9*b*	0.629(1)	0.2233(1)	0.9592(6)	0.060(2)	0.073(2)	0.114(3)	0.032(2)	0.000(2)	0.013(2)

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