Crystal structure of 8-(2-methylphenyl)-11-[(*E*)-(2-methylphenyl)-methylidene]-14-hydroxy-3,13-diazaheptacyclo-[13.7.1.^{9,13}.0^{2,9}.0^{2,14}.0^{3,7}.0^{19,23}]tetracosa-1(22),15,17,19(23),20-pentaen-10-one methanol monosolvate, $C_{37}H_{34}N_2O_2{\cdot}CH_3OH$, $C_{38}H_{38}N_2O_3$

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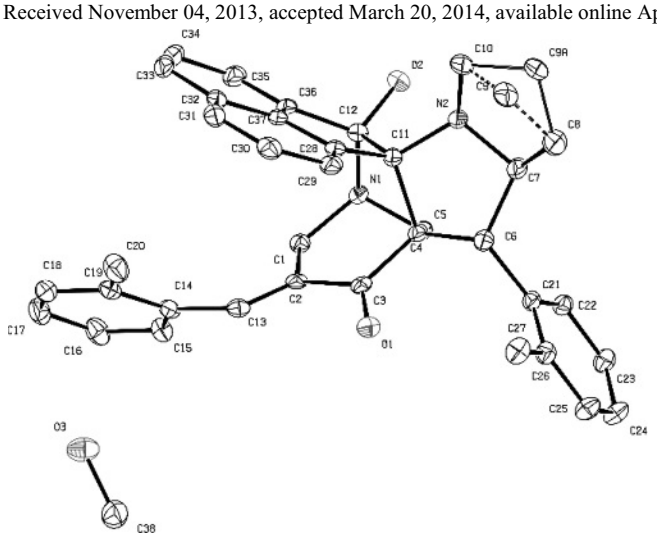

Abstract

$C_{38}H_{38}N_2O_3$, monoclinic, $P2_1/c$ (no. 14), a = 11.3562(2) Å, b = 17.0817(4) Å, c = 15.2368(3) Å, β = 93.539(1)°, V = 2950.1 Å^3, Z = 4, $R_{gt}(F)$ = 0.0498, $wR_{ref}(F^2)$ = 0.1133, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.21×0.1×0.18 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.81 cm^{-1}
Diffractometer, scan mode:	Bruker Kappa APEXII, φ and ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	29114, 6771
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 5042
$N(param)_{refined}$:	402
Programs:	SHELX [7], PLATON [8], SAINT [9]

Source of material

A mixture of 3,5-bis[(*E*)-(2-methylphenyl)methylidene]tetrahydro-4(1*H*)-pyridinone (0.10 g, 0.33 mmol), acenaphthenequinone (0.06 g, 0.33 mmol) and proline (0.038 g, 0.33 mmol) were dissolved in methanol (5 mL) and refluxed for 30 minutes. After completion of the reaction as evident from *TLC*, the mixture was poured into water (50 mL). The precipitated solid was filtered and washed with water to afford the product which was recrystallised from methanol to reveal the title compound as colourless crystals.

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Experimental details

The C atom of the pyrrolizine unit is disordered over two positions with site occupancies of C9 = 0.463 and C9A = 0.537. H atoms were placed in idealized positions and allowed to ride on their parent atoms, with C–H = 0.93, 0.96, 0.97 Å for aromatic, methyl and methylene respectively. H atoms attached to C8 and C10 atoms are located from difference fourier maps and refined isotropically.

Discussion

The synthesis and chemistry of heterocyclic compounds have been an interesting field in view of their structural diversity and remarkable ability to serve as biomimetics and active pharmacophores. Many of the most famous natural alkaloids or synthetic drugs consist of at least one heterocyclic ring. Chemistry of azomethine ylides have gained significance, as it serves as an expedient route for the construction of nitrogen-containing heterocycles [1]. Acenaphthenequinone is a versatile precursor for azomethine ylide cycloaddition as it reacts with various α-amino acids generating reactive 1,3-dipoles [2, 3]. The pyrrolizine substructure occurs in many natural products of potential use in medicine and agriculture [4]. In view of its medicinal importance, we report the crysal structure of the title compound. The molecule of the title compound is shown in the figure (30% ellipsoid). The crystal structure of title compound is build up by two discrete moieties, the 8-(2-methylphenyl)-11-[(*E*)-(2-methylphenyl)-methylidene]-14-hydroxy-3,13-diazaheptacyclo[13.7.1.^{9,13}.0^{2,9}-.0^{2,14}.0^{3,7}.0^{19,23}]tetracosa-1(22),15,17,19(23),20-pentaen-10-one molecule and one methanol molecule. According to puckering analysis [5] the central piperidine ring (C1-C5/N1) adopts a twisted half chair conformation which is comparable to the closely related structure [6]. The pyrrolidine ring (C4/C5/N1/C12/C11) shows an envelope conformation with the carbon atom C5 out of plane. In the pyrrolizine ring system, the pyrrolidine ring (C4/C6/C7/C11/N2) adopts a twisted conformation according to the puckering analysis. The disordered pyrrolidine ring adopts envelope conformations in both conformers with the carbon atoms C9 and C9A to be out-of-plane respectively. The two disordered atoms are asymmetrically distributed on both sides of the mean plane. The twist of the 2-methyl phenyl methylidine is indicated by the torsion angle C2–C13–C14–C15 = –47.1° which is due to the non-bonded interactions between one of the ortho H atoms in the aryl ring and the equatorial H atom at the 2-position of the piperidinone ring. The C14–H14B···π, C23–H23···π inter-

actions with the 2.92, 2.91 Å distances of C14–Cg1, C23–Cg2 exist in the compound which stabilizes the structure.

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{iso}
H(2)	4*e*		0.0624	0.0771	–0.1121	0.040
H(1A)	4*e*		0.2492	0.2482	0.0652	0.022
H(1B)	4*e*		0.1916	0.2183	0.1496	0.022
H(5A)	4*e*		0.0061	0.1479	0.1269	0.023
H(5B)	4*e*		–0.0100	0.0904	0.0458	0.023
H(6)	4*e*		–0.2278	0.2378	–0.0343	0.024
H(7)	4*e*		–0.1917	0.0761	–0.0581	0.032
H(8A)	4*e*	0.463	–0.3236	0.0845	–0.1719	0.044
H(8B)	4*e*	0.463	–0.3585	0.1670	–0.1344	0.044
H(8C)	4*e*	0.537	–0.3539	0.0965	–0.1457	0.044
H(8D)	4*e*	0.537	–0.3302	0.1871	–0.1529	0.044
H(9A)	4*e*	0.463	–0.2749	0.1571	–0.2819	0.032
H(9B)	4*e*	0.463	–0.2484	0.2305	–0.2218	0.032
H(9C)	4*e*	0.537	–0.2840	0.1509	–0.2858	0.027
H(9D)	4*e*	0.537	–0.2381	0.0680	–0.2530	0.027
H(10A)	4*e*	0.463	–0.0703	0.1997	–0.2466	0.033
H(10B)	4*e*	0.463	–0.1000	0.1097	–0.2568	0.033
H(10C)	4*e*	0.537	–0.0601	0.1290	–0.2590	0.033
H(10D)	4*e*	0.537	–0.1132	0.2118	–0.2383	0.033
H(13)	4*e*		0.0631	0.4127	0.0856	0.023
H(15)	4*e*		0.2800	0.3282	0.2170	0.029
H(16)	4*e*		0.4678	0.3731	0.2556	0.036
H(17)	4*e*		0.5458	0.4751	0.1783	0.038
H(18)	4*e*		0.4346	0.5340	0.0654	0.035
H(20A)	4*e*		0.1537	0.5363	0.0219	0.053
H(20B)	4*e*		0.1841	0.4683	–0.0420	0.053
H(20C)	4*e*		0.2660	0.5421	–0.0326	0.053
H(22)	4*e*		–0.1844	0.0665	0.0990	0.025
H(23)	4*e*		–0.2884	0.0343	0.2193	0.028
H(24)	4*e*		–0.4379	0.1156	0.2610	0.034
H(25)	4*e*		–0.4874	0.2255	0.1790	0.033
H(27A)	4*e*		–0.4711	0.3086	0.0620	0.042
H(27B)	4*e*		–0.4152	0.2746	–0.0215	0.042
H(27C)	4*e*		–0.3389	0.3263	0.0453	0.042
H(29)	4*e*		–0.1389	0.3372	–0.1445	0.027
H(30)	4*e*		–0.0582	0.4457	–0.2131	0.031
H(31)	4*e*		0.1378	0.4525	–0.2377	0.032
H(33)	4*e*		0.3482	0.3858	–0.2154	0.032
H(34)	4*e*		0.4565	0.2843	–0.1564	0.033
H(35)	4*e*		0.3692	0.1820	–0.0810	0.030
H(3)	4*e*		0.2527	0.0823	0.0947	0.054
H(38A)	4*e*		0.4328	0.0896	0.0465	0.055
H(38B)	4*e*		0.3571	0.0204	0.0054	0.055
H(38C)	4*e*		0.4495	0.0046	0.0841	0.055

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4*e*		–0.1150(1)	0.33004(6)	0.07119(7)	0.0197(7)	0.0270(6)	0.0189(6)	0.0034(5)	–0.0007(5)	–0.0002(5)
O(2)	4*e*		0.1277(1)	0.09062(6)	–0.09155(7)	0.0294(7)	0.0244(6)	0.0269(6)	–0.0037(5)	0.0051(5)	–0.0086(5)
N(1)	4*e*		0.1343(1)	0.16045(7)	0.04041(8)	0.0189(8)	0.0213(7)	0.0153(6)	–0.0019(6)	0.0034(5)	0.0000(5)
N(2)	4*e*		–0.0834(1)	0.14474(8)	–0.12856(8)	0.0248(8)	0.0330(8)	0.0129(6)	–0.0111(6)	0.0030(6)	–0.0033(6)
C(1)	4*e*		0.1755(1)	0.23161(9)	0.0881(1)	0.0188(9)	0.0225(8)	0.0140(7)	–0.0005(7)	0.0003(6)	–0.0002(6)
C(2)	4*e*		0.0903(1)	0.30005(9)	0.08261(9)	0.0198(9)	0.0248(8)	0.0084(7)	0.0003(7)	0.0002(6)	–0.0006(6)
C(3)	4*e*		–0.0368(1)	0.28342(9)	0.05745(9)	0.0198(9)	0.0239(8)	0.0082(7)	–0.0001(7)	0.0014(6)	0.0041(6)
C(4)	4*e*		–0.0630(1)	0.20528(9)	0.01293(9)	0.0175(9)	0.0231(8)	0.0118(7)	–0.0023(7)	0.0020(6)	0.0012(6)
C(5)	4*e*		0.0132(1)	0.14292(9)	0.0640(1)	0.0193(9)	0.0215(8)	0.0159(7)	–0.0023(7)	0.0029(6)	0.0003(6)
C(6)	4*e*		–0.1942(1)	0.18957(9)	–0.0085(1)	0.0188(9)	0.0253(8)	0.0145(7)	–0.0025(7)	–0.0010(6)	0.0024(6)
C(7)	4*e*		–0.1938(2)	0.1291(1)	–0.0828(1)	0.027(1)	0.037(1)	0.0149(8)	–0.0129(8)	0.0028(7)	–0.0021(7)
C(8)	4*e*		–0.2940(2)	0.1357(1)	–0.1543(1)	0.030(1)	0.059(1)	0.0205(9)	–0.015(1)	–0.0033(8)	–0.0057(9)
C(9)	4*e*	0.463	–0.2378(4)	0.1743(3)	–0.2262(2)	0.030(2)	0.032(3)	0.017(2)	–0.003(2)	–0.009(2)	0.002(2)
C(9A)	4*e*	0.537	–0.2404(3)	0.1233(3)	–0.2386(2)	0.025(2)	0.023(2)	0.020(2)	0.002(2)	–0.005(1)	–0.004(1)
C(10)	4*e*		–0.1147(2)	0.1566(1)	–0.2235(1)	0.034(1)	0.036(1)	0.0122(8)	–0.0103(8)	0.0006(7)	–0.0053(7)
C(11)	4*e*		–0.0084(1)	0.20199(9)	–0.0789(1)	0.0211(9)	0.0246(8)	0.0127(7)	–0.0053(7)	0.0021(6)	–0.0019(6)
C(12)	4*e*		0.1209(2)	0.16653(9)	–0.0572(1)	0.0216(9)	0.0222(8)	0.0157(7)	–0.0022(7)	0.0040(6)	–0.0033(6)
C(13)	4*e*		0.1223(2)	0.37529(9)	0.0936(1)	0.0215(9)	0.0237(8)	0.0129(7)	0.0027(7)	–0.0011(6)	–0.0006(6)
C(14)	4*e*		0.2421(2)	0.40420(9)	0.1171(1)	0.0219(9)	0.0202(8)	0.0179(8)	–0.0006(7)	0.0010(6)	–0.0047(6)
C(15)	4*e*		0.3107(2)	0.3697(1)	0.1862(1)	0.025(1)	0.0223(8)	0.0241(9)	–0.0015(7)	–0.0031(7)	–0.0009(7)
C(16)	4*e*		0.4235(2)	0.3964(1)	0.2094(1)	0.026(1)	0.0259(9)	0.036(1)	0.0024(8)	–0.0081(8)	–0.0043(8)
C(17)	4*e*		0.4696(2)	0.4575(1)	0.1636(1)	0.020(1)	0.0289(9)	0.046(1)	–0.0022(8)	0.0011(8)	–0.0134(9)
C(18)	4*e*		0.4027(2)	0.4927(1)	0.0957(1)	0.034(1)	0.0214(8)	0.033(1)	–0.0062(8)	0.0103(8)	–0.0066(7)
C(19)	4*e*		0.2887(2)	0.46764(9)	0.0718(1)	0.032(1)	0.0198(8)	0.0202(8)	–0.0010(7)	0.0039(7)	–0.0051(7)
C(20)	4*e*		0.2166(2)	0.5072(1)	–0.0019(1)	0.051(1)	0.0263(9)	0.028(1)	–0.0095(9)	–0.0040(9)	0.0063(8)
C(21)	4*e*		–0.2685(1)	0.16801(9)	0.0678(1)	0.0164(9)	0.0252(8)	0.0144(7)	–0.0055(7)	–0.0005(6)	0.0000(6)
C(22)	4*e*		–0.2441(1)	0.0995(1)	0.1158(1)	0.0165(9)	0.0252(8)	0.0206(8)	–0.0010(7)	0.0022(6)	–0.0008(7)
C(23)	4*e*		–0.3068(2)	0.0797(1)	0.1877(1)	0.021(1)	0.0263(9)	0.0231(8)	–0.0007(7)	0.0023(7)	0.0058(7)
C(24)	4*e*		–0.3966(2)	0.1277(1)	0.2120(1)	0.027(1)	0.034(1)	0.0233(9)	0.0005(8)	0.0099(7)	0.0064(7)
C(25)	4*e*		–0.4252(2)	0.1942(1)	0.1631(1)	0.024(1)	0.0309(9)	0.0289(9)	0.0049(8)	0.0082(7)	0.0028(8)
C(26)	4*e*		–0.3631(2)	0.2153(1)	0.0906(1)	0.0187(9)	0.0258(8)	0.0203(8)	–0.0017(7)	–0.0011(7)	0.0025(7)
C(27)	4*e*		–0.4005(2)	0.2878(1)	0.0394(1)	0.024(1)	0.0309(9)	0.0288(9)	0.0025(8)	0.0006(7)	0.0064(8)
C(28)	4*e*		0.0147(2)	0.27953(9)	–0.12363(9)	0.0226(9)	0.0263(8)	0.0094(7)	–0.0051(7)	0.0008(6)	–0.0026(6)
C(29)	4*e*		–0.0584(2)	0.3394(1)	–0.1524(1)	0.025(1)	0.0311(9)	0.0128(7)	–0.0015(8)	–0.0001(6)	–0.0014(7)
C(30)	4*e*		–0.0089(2)	0.4048(1)	–0.1943(1)	0.033(1)	0.0273(9)	0.0161(8)	–0.0016(8)	–0.0032(7)	0.0014(7)
C(31)	4*e*		0.1090(2)	0.4096(1)	–0.2081(1)	0.036(1)	0.0283(9)	0.0151(8)	–0.0101(8)	–0.0006(7)	0.0018(7)
C(32)	4*e*		0.1874(2)	0.3495(1)	–0.1777(1)	0.028(1)	0.0281(9)	0.0110(7)	–0.0093(7)	0.0003(6)	–0.0022(6)

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

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(33)	4*e*		0.3108(2)	0.3462(1)	–0.1858(1)	0.028(1)	0.036(1)	0.0167(8)	–0.0138(8)	0.0029(7)	0.0002(7)
C(34)	4*e*		0.3755(2)	0.2850(1)	–0.1505(1)	0.021(1)	0.043(1)	0.0185(8)	–0.0085(8)	0.0029(7)	–0.0012(8)
C(35)	4*e*		0.3231(2)	0.2225(1)	–0.1052(1)	0.024(1)	0.0335(9)	0.0168(8)	–0.0024(8)	0.0029(7)	–0.0009(7)
C(36)	4*e*		0.2040(2)	0.22314(9)	–0.0980(1)	0.021(1)	0.0275(9)	0.0121(7)	–0.0043(7)	0.0036(6)	–0.0049(6)
C(37)	4*e*		0.1368(2)	0.28604(9)	–0.13457(9)	0.024(1)	0.0262(8)	0.0086(7)	–0.0057(7)	0.0019(6)	–0.0033(6)
O(3)	4*e*		0.3057(1)	0.05602(8)	0.11885(8)	0.0337(8)	0.0433(8)	0.0301(7)	0.0139(6)	0.0021(6)	–0.0027(6)
C(38)	4*e*		0.3933(2)	0.0415(1)	0.0589(1)	0.031(1)	0.035(1)	0.045(1)	–0.0002(9)	0.0064(9)	–0.0035(9)

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