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Crystal structure of 5-(*E*)-benzylidenebicyclo[4.2.2]deca-2,7-diene-9,9,10,10-tetracarbonitrile, $C_{21}H_{14}N_4$

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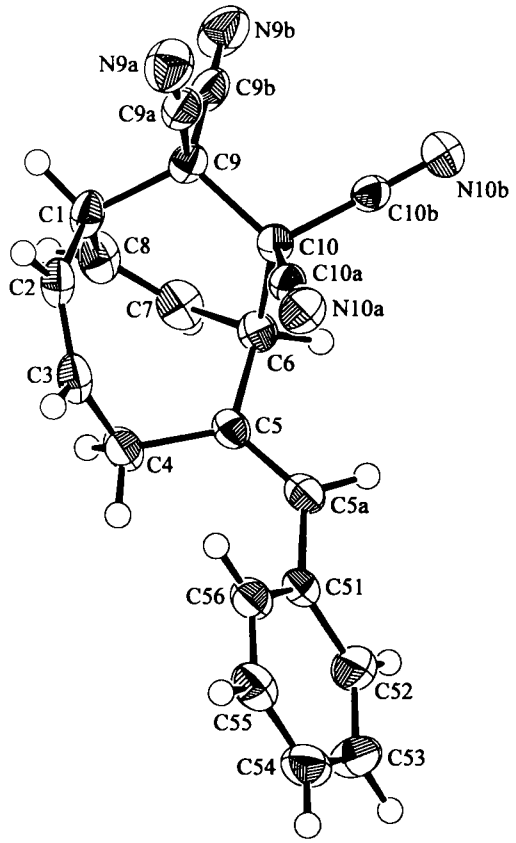

Abstract

$C_{21}H_{14}N_4$, triclinic, $P\bar{1}$ (No. 2), $a = 7.630(2)$ Å, $b = 9.497(2)$ Å, $c = 12.467(2)$ Å, $\alpha = 91.92(1)°$, $\beta = 99.16(1)°$, $\gamma = 101.20(2)°$, $V = 873.0$ Å^3, $Z = 2$, $R_{gt}(F) = 0.035$, $wR_{ref}(F^2) = 0.095$, $T = 293$ K.

Source of material

Treatment of a mixture of (*E*)- and (*Z*)-7-benzylidenecycloocta-1,3,5-triene with ethenetetracarbonitrile in ethyl acetate gave a mixture of nine compounds [1]. Chromatography on silica gel gave the title compound; mp 443.5 K – 445 K.

Experimental details

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation.

Discussion

The crystal structure determination confirms quantitative (NMR) and qualitative evidence (Dreiding models) that indicates that the preferred conformation is the one with the C4 atom directed away from the C9 and C10 atoms, i.e. those bearing the nitrile groups.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.10 × 0.40 × 0.40 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	0.75 cm^{-1}
Diffractometer, scan mode:	CAD4F, $\omega/2\theta$
$2\theta_{max}$:	45°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2288, 2288
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1568
$N(param)_{refined}$:	226
Programs:	teXsan [2], SHELXS-86 [3], SHELXL-97 [4], ORTEPII [5])

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*i*	–0.0267	0.4986	0.3414	0.041
H(2)	2*i*	0.1963	0.4562	0.4712	0.041
H(3)	2*i*	0.3248	0.2763	0.4751	0.044
H(4a)	2*i*	0.3015	0.0958	0.3539	0.044
H(4b)	2*i*	0.1121	0.1226	0.3005	0.044
H(5a)	2*i*	0.4708	0.1916	0.1179	0.037
H(6)	2*i*	0.2488	0.3069	0.0787	0.036
H(7)	2*i*	–0.0492	0.2307	0.0908	0.045
H(8)	2*i*	–0.1736	0.3048	0.2270	0.048
H(52)	2*i*	0.5582	–0.0548	0.1047	0.049
H(53)	2*i*	0.7476	–0.1983	0.1835	0.058
H(54)	2*i*	0.8755	–0.1569	0.3655	0.056
H(55)	2*i*	0.8089	0.0247	0.4684	0.050
H(56)	2*i*	0.6157	0.1659	0.3918	0.043

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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(9a)	1*i*	0.3437(2)	0.7777(2)	0.3883(2)	0.053(1)	0.051(1)	0.046(1)	0.008(1)	0.0108(9)	–0.006(1)
N(9b)	2*i*	–0.0399(2)	0.6633(2)	0.1025(1)	0.054(1)	0.077(1)	0.039(1)	0.038(1)	0.0071(9)	0.009(1)
N(10a)	2*i*	0.6256(2)	0.5107(2)	0.3228(1)	0.0270(9)	0.044(1)	0.042(1)	0.0082(8)	0.0018(8)	0.0016(8)
N(10b)	2*i*	0.3917(2)	0.6294(2)	0.0267(1)	0.051(1)	0.044(1)	0.040(1)	0.0133(9)	0.0147(9)	0.0115(9)
C(1)	2*i*	0.0581(2)	0.4493(2)	0.3115(1)	0.024(1)	0.051(1)	0.030(1)	0.0112(9)	0.0087(8)	0.0059(9)
C(2)	2*i*	0.1753(2)	0.3994(2)	0.4065(2)	0.028(1)	0.051(1)	0.026(1)	0.0074(9)	0.0087(8)	0.0076(9)
C(3)	2*i*	0.2519(2)	0.2852(2)	0.4094(2)	0.032(1)	0.052(1)	0.029(1)	0.007(1)	0.0093(9)	0.013(1)
C(4)	2*i*	0.2387(3)	0.1681(2)	0.3224(2)	0.035(1)	0.038(1)	0.040(1)	0.0056(9)	0.0101(9)	0.0140(9)
C(5)	2*i*	0.3148(2)	0.2152(2)	0.2217(1)	0.031(1)	0.026(1)	0.028(1)	0.0010(8)	0.0050(8)	0.0023(8)
C(5a)	2*i*	0.4477(2)	0.1641(2)	0.1860(2)	0.041(1)	0.025(1)	0.026(1)	0.0031(9)	0.0057(9)	0.0029(8)
C(6)	2*i*	0.2265(2)	0.3209(2)	0.1532(1)	0.031(1)	0.032(1)	0.026(1)	0.0050(8)	0.0032(8)	0.0025(8)
C(7)	2*i*	0.0242(2)	0.2880(2)	0.1491(2)	0.029(1)	0.043(1)	0.036(1)	–0.0009(9)	–0.0039(9)	0.002(1)
C(8)	2*i*	–0.0511(2)	0.3384(2)	0.2259(2)	0.022(1)	0.052(1)	0.044(1)	0.0003(9)	0.004(1)	0.009(1)
C(9)	2*i*	0.1778(2)	0.5614(2)	0.2503(1)	0.025(1)	0.038(1)	0.0246(9)	0.0124(8)	0.0018(8)	0.0027(8)
C(9a)	2*i*	0.2767(3)	0.6842(2)	0.3258(2)	0.032(1)	0.040(1)	0.030(1)	0.0121(9)	0.0073(9)	0.003(1)
C(9b)	2*i*	0.0559(3)	0.6177(2)	0.1655(2)	0.033(1)	0.050(1)	0.031(1)	0.020(1)	0.0084(9)	0.004(1)
C(10)	2*i*	0.3121(2)	0.4858(2)	0.1938(1)	0.022(1)	0.032(1)	0.0232(9)	0.0071(8)	0.0022(8)	0.0053(8)
C(10a)	2*i*	0.4868(2)	0.4974(2)	0.2692(1)	0.024(1)	0.028(1)	0.030(1)	0.0054(8)	0.0073(9)	0.0019(8)
C(10b)	2*i*	0.3581(2)	0.5667(2)	0.0996(2)	0.028(1)	0.031(1)	0.029(1)	0.0112(8)	0.0050(9)	0.0034(9)
C(51)	2*i*	0.5627(2)	0.0704(2)	0.2395(2)	0.033(1)	0.026(1)	0.032(1)	0.0036(8)	0.0091(9)	0.0059(8)
C(52)	2*i*	0.6068(3)	–0.0389(2)	0.1784(2)	0.046(1)	0.040(1)	0.037(1)	0.011(1)	0.010(1)	0.001(1)
C(53)	2*i*	0.7216(3)	–0.1244(2)	0.2252(2)	0.051(1)	0.039(1)	0.062(2)	0.018(1)	0.017(1)	0.002(1)
C(54)	2*i*	0.7975(3)	–0.1000(2)	0.3339(2)	0.038(1)	0.042(1)	0.066(2)	0.013(1)	0.012(1)	0.018(1)
C(55)	2*i*	0.7575(3)	0.0082(2)	0.3952(2)	0.039(1)	0.044(1)	0.040(1)	0.005(1)	0.004(1)	0.013(1)
C(56)	2*i*	0.6416(3)	0.0929(2)	0.3491(2)	0.041(1)	0.031(1)	0.037(1)	0.0041(9)	0.0092(9)	0.0060(9)

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