Crystal structure of (2*E*,6*E*)-2,6-bis(3-nitrobenzylidene)cyclohexanone, C₂₀H₁₆N₂O₅

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Abstract

C₂₀H₁₆N₂O₅, monoclinic, *P*12₁/c1 (no. 14), *a* = 7.659(2) Å, *b* = 12.168(3) Å, *c* = 18.305(4) Å, β = 99.652(5)°, *V* = 1681.7 Å³, *Z* = 4, *R*_{gt}(*F*) = 0.081, *wR*_{ref}(*F*²) = 0.155, *T* = 298 K.

Source of material

A mixture of cyclohexanone (5 mmol), 3-nitrobenzaldehyde (10 mmol), and copper trifluoromethanesulfonate (0.05 mmol) was added in a round-bottomed flask. The mixture was stirred at 353 K for 8 h to complete the reaction. After cooling to room temperature, the reaction mixture was quenched by water (5 mL) and stirred for 10 min. The title compound was isolated with high purity by filtration. Single crystals suitable for X-ray crystal structure determination were obtained by slow evaporation of an ethanol solution (yield 96 %, m.p. 461–463 K). ¹H NMR and IR data are available in the CIF.

Experimental details

The H atoms were positioned geometrically and allowed to ride on their parent atoms, with *d*(C—H) = 0.93 Å and *U*_{iso} = 1.2*U*_{eq} for aromatic H atoms, and *d*(C—H) = 0.97 Å and *U*_{iso} = 1.5*U*_{eq} for methylene atoms. With respect to the value of the *R*_{gt}, it may be because that the obtained crystals were not good. So we had tried our best to get a better one, but we failed.

Discussion

2,6-Bis-substituted benzylidene-cycloalkanones are an interesting class of compounds because of their use as precursors to potentially bioactive pyrimidine compounds [1]. Recently, some of

them were designed as thiol alkylators for evaluation as candidate cytotoxic and anticancer agents [2]. The synthesis of this kind of cycloalkanones has gained great importance.

The X-ray crystal structure analysis indicates the configuration of the title molecule in *E*-form. The molecule contains two essentially planar phenyl rings with the dihedral angle between them being 13.8(1)°. The central alicyclic ring adopts the sofa conformation. Atoms C8, C9, C10, C11 and C13 are coplanar, with an r.m.s. deviation of 0.008(1) Å, atom C12 is 0.674(2) Å out of the plane. Steric repulsions occur between the atom pairs H2/H13B and H20/H11A (the interatomic distances are 2.241 Å and 2.181 Å, respectively) causing the two aryl rings to twist out of the plane of the adjacent alicyclic linkages and create torsion angles for C2–C3–C7–C8 and C10–C14–C15–C20 of –28.0(5)° and 25.8(6)°, respectively.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.13 × 0.14 × 0.17 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	1.05 cm ^{−1}
Diffractometer, scan mode:	Bruker SMART APEX CCD, φ/ω
2θ _{max} :	50.5°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	8851, 3030
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2225
<i>N</i> (<i>param</i>) _{refined} :	244
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL [5]

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(2)	4e	0.3270	0.1180	0.4300	0.050
H(4)	4e	0.0354	0.2500	0.2491	0.059
H(5)	4e	−0.0193	0.0785	0.1988	0.067
H(6)	4e	0.1021	−0.0748	0.2628	0.064
H(7)	4e	0.1969	0.3708	0.3392	0.051
H(11A)	4e	0.2524	0.4828	0.6363	0.062
H(11B)	4e	0.4167	0.4243	0.6127	0.062
H(12A)	4e	0.0607	0.3629	0.5618	0.060

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(12B)	4e	0.1943	0.2949	0.6190	0.060
H(13A)	4e	0.3672	0.2575	0.5283	0.059
H(13B)	4e	0.1730	0.2170	0.5006	0.059
H(14)	4e	0.2836	0.6678	0.4900	0.055
H(16)	4e	0.2084	0.8372	0.5417	0.054
H(18)	4e	0.3892	0.9110	0.7539	0.069
H(19)	4e	0.4860	0.7324	0.7747	0.068
H(20)	4e	0.4482	0.6069	0.6796	0.062

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
O(1)	4e	0.4256(4)	−0.0751(2)	0.4414(2)	0.117(3)	0.064(2)	0.081(2)	0.023(2)	−0.018(2)	0.006(2)
O(2)	4e	0.2389(5)	−0.1770(2)	0.3735(2)	0.170(3)	0.035(2)	0.093(2)	−0.011(2)	0.005(2)	−0.000(1)
O(3)	4e	0.2611(3)	0.5341(2)	0.4008(1)	0.097(2)	0.037(1)	0.041(1)	0.000(1)	−0.007(1)	0.004(1)
O(4)	4e	0.1410(4)	1.0249(2)	0.5727(2)	0.104(2)	0.048(2)	0.088(2)	0.009(2)	−0.002(2)	0.001(2)
O(5)	4e	0.2802(4)	1.0716(2)	0.6796(2)	0.129(3)	0.045(2)	0.101(2)	−0.014(2)	0.011(2)	−0.029(2)
N(1)	4e	0.3030(5)	−0.0880(2)	0.3918(2)	0.096(3)	0.037(2)	0.051(2)	0.005(2)	0.019(2)	−0.002(2)
N(2)	4e	0.2341(4)	1.0035(2)	0.6313(2)	0.073(2)	0.044(2)	0.074(2)	−0.010(2)	0.018(2)	−0.008(2)
C(1)	4e	0.2237(4)	0.0095(2)	0.3515(2)	0.056(2)	0.036(2)	0.039(2)	−0.002(2)	0.012(2)	−0.001(1)
C(2)	4e	0.2560(4)	0.1112(2)	0.3837(2)	0.049(2)	0.038(2)	0.037(2)	−0.001(1)	0.004(1)	−0.006(1)
C(3)	4e	0.1811(4)	0.2037(2)	0.3464(2)	0.045(2)	0.039(2)	0.037(2)	0.003(1)	0.008(1)	−0.002(1)
C(4)	4e	0.0818(4)	0.1887(3)	0.2760(2)	0.056(2)	0.049(2)	0.040(2)	0.003(2)	0.003(2)	−0.002(2)
C(5)	4e	0.0505(4)	0.0861(3)	0.2453(2)	0.063(2)	0.061(2)	0.039(2)	−0.007(2)	−0.002(2)	−0.010(2)
C(6)	4e	0.1221(5)	−0.0050(3)	0.2831(2)	0.071(2)	0.042(2)	0.047(2)	−0.012(2)	0.011(2)	−0.010(2)
C(7)	4e	0.2050(4)	0.3163(2)	0.3752(2)	0.050(2)	0.035(2)	0.042(2)	0.006(1)	0.003(2)	0.004(1)
C(8)	4e	0.2364(4)	0.3526(2)	0.4449(2)	0.044(2)	0.036(2)	0.038(2)	0.007(1)	−0.002(1)	−0.001(1)
C(9)	4e	0.2603(4)	0.4746(2)	0.4547(2)	0.043(2)	0.035(2)	0.044(2)	0.006(1)	−0.005(2)	0.003(2)
C(10)	4e	0.2852(4)	0.5223(2)	0.5312(2)	0.043(2)	0.038(2)	0.041(2)	0.002(1)	0.001(1)	−0.002(1)
C(11)	4e	0.2943(5)	0.4452(2)	0.5958(2)	0.069(2)	0.044(2)	0.042(2)	−0.004(2)	0.005(2)	−0.002(2)
C(12)	4e	0.1842(4)	0.3427(2)	0.5761(2)	0.064(2)	0.043(2)	0.044(2)	−0.003(2)	0.011(2)	0.000(2)
C(13)	4e	0.2461(4)	0.2817(2)	0.5127(2)	0.062(2)	0.040(2)	0.045(2)	0.001(2)	0.007(2)	0.000(2)
C(14)	4e	0.2953(4)	0.6323(3)	0.5356(2)	0.050(2)	0.044(2)	0.039(2)	−0.005(2)	−0.002(2)	−0.002(2)
C(15)	4e	0.3214(4)	0.7062(2)	0.5997(2)	0.048(2)	0.038(2)	0.042(2)	−0.006(1)	0.004(2)	−0.002(2)
C(16)	4e	0.2649(4)	0.8149(2)	0.5883(2)	0.050(2)	0.042(2)	0.043(2)	−0.007(2)	0.005(2)	−0.002(2)
C(17)	4e	0.2926(4)	0.8891(3)	0.6457(2)	0.052(2)	0.037(2)	0.055(2)	−0.011(2)	0.011(2)	−0.005(2)
C(18)	4e	0.3735(4)	0.8599(3)	0.7156(2)	0.064(2)	0.054(2)	0.054(2)	−0.013(2)	0.010(2)	−0.015(2)
C(19)	4e	0.4307(5)	0.7537(3)	0.7277(2)	0.065(2)	0.060(2)	0.042(2)	−0.011(2)	0.001(2)	−0.003(2)
C(20)	4e	0.4066(4)	0.6782(3)	0.6706(2)	0.059(2)	0.041(2)	0.053(2)	−0.005(2)	0.000(2)	−0.003(2)

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