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The crystal structure of (3Z,3'Z)-4,4'-((1,4-phenylenebis(methylene))bis(azanediy))bis(pent-3-en-2-one), C₁₈H₂₄N₂O₂

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Abstract

C₁₈H₂₄N₂O₂, triclinic, $P\bar{1}$ (no. 2), $a = 6.088(10)$ Å, $b = 8.911(14)$ Å, $c = 16.44(3)$ Å, $\alpha = 81.772(17)^\circ$, $\beta = 84.683(18)^\circ$, $\gamma = 76.045(17)^\circ$, $V = 855(2)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0511$, $wR_{\text{ref}}(F^2) = 0.1858$, $T = 293$ K.

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The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

To the solution of pentane-2,4-dione (1.00 g, 10 mmol) in ethanol (20 ml) was added the solution of 1,4-phenylenedimethanamine (0.681 g, 5 mmol) in ethanol (20 ml) dropwisely. The mixture was refluxed for 6 h, then it

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.22 × 0.21 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.08 mm ⁻¹
Diffractometer, scan mode:	Bruker Apex-II, φ and ω
θ_{max} , completeness:	24.5°, 98%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5544, 2782, 0.027
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2032
$N(\text{param})_{\text{refined}}$:	200
Programs:	Bruker [1], SHELX [2], Olex2 [3]

was distilled under reduced pressure to get a yellow solid. The solid was recrystallized by acetone to get the title compound (3Z,3'Z)-4,4'-((1,4-phenylenebis(methylene))bis(azanediy))bis(pent-3) (0.95 g, yield 63%) as a white single crystal which was suitable for X-ray diffraction.

Experimental details

The data were scaled and corrected for absorption using SADABS-2016/2 (Bruker). The hydrogen atoms were placed at calculated positions and refined as riding atoms.

Comment

Some α,β -unsaturated ketones have drawn attentions for the reason that they have shown remarkable biological activities such as antimicrobial [4], antibacterial [5], anti-tumor [6], antioxidant [7] and anti-inflammatory [8, 9], anti-parasitic [10] activities and so on. In addition, these α,β -unsaturated ketones can also be used to prepare many complexes [11]. In this paper, we report the synthesis of the title compound by condensation reaction using 1,4-phenylenedimethanamine as the starting material. Its molecular structure was determined by X-ray single crystal diffraction. There is one independent molecule in the asymmetric unit. The angles of C4–N2–C6, C8–C9–C10 and C3–C2–O2 are 126.2 (2)°, 119.1° and 123.5 (2)° respectively, which are in normal range (*cf.* the Figure). Intramolecular hydrogen bonds [12] were observed as

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} */ <i>U</i> _{eq}
C1	0.7099 (5)	0.0529 (3)	0.5594 (2)	0.0882 (9)
H1A	0.6603	−0.0299	0.5944	0.132*
H1B	0.7361	0.0275	0.5039	0.132*
H1C	0.8480	0.0661	0.5782	0.132*
C2	0.5277 (4)	0.2039 (3)	0.56188 (14)	0.0577 (6)
C3	0.3297 (4)	0.2001 (3)	0.61227 (14)	0.0581 (7)
H3	0.3151	0.1039	0.6397	0.070*
C4	0.1538 (4)	0.3282 (3)	0.62465 (12)	0.0511 (6)
C5	−0.0497 (5)	0.3076 (3)	0.68101 (16)	0.0783 (8)
H5A	−0.0311	0.1998	0.7029	0.118*
H5B	−0.0636	0.3692	0.7254	0.118*
H5C	−0.1839	0.3407	0.6506	0.118*
C6	−0.0208 (4)	0.6130 (3)	0.59672 (13)	0.0534 (6)
H6A	−0.1669	0.5872	0.5973	0.064*
H6B	−0.0085	0.6882	0.5487	0.064*
C7	−0.0144 (4)	0.6889 (2)	0.67341 (12)	0.0446 (5)
C8	0.1673 (4)	0.7519 (2)	0.68402 (13)	0.0490 (6)
H8	0.2896	0.7431	0.6453	0.059*
C9	0.1673 (4)	0.8279 (2)	0.75227 (12)	0.0497 (6)
H9	0.2910	0.8685	0.7584	0.060*
C10	−0.0123 (3)	0.8450 (2)	0.81146 (12)	0.0443 (5)
C11	−0.1926 (4)	0.7807 (3)	0.80170 (14)	0.0564 (6)
H11	−0.3141	0.7891	0.8408	0.068*
C12	−0.1932 (4)	0.7036 (3)	0.73365 (13)	0.0566 (6)
H12	−0.3155	0.6610	0.7283	0.068*
C13	−0.0135 (4)	0.9361 (2)	0.88354 (12)	0.0517 (6)
H13A	0.1410	0.9407	0.8907	0.062*
H13B	−0.0999	1.0421	0.8698	0.062*
C14	0.2359 (4)	0.6665 (3)	0.98366 (16)	0.0720 (8)
H14A	0.2905	0.5777	1.0230	0.108*
H14B	0.2393	0.6334	0.9304	0.108*
H14C	0.3305	0.7392	0.9812	0.108*
C15	−0.0047 (4)	0.7440 (3)	1.00949 (13)	0.0495 (6)
C16	−0.1120 (4)	0.6860 (3)	1.08084 (14)	0.0563 (6)
H16	−0.0310	0.5973	1.1114	0.068*
C17	−0.3334 (4)	0.7504 (3)	1.11074 (14)	0.0568 (6)
C18	−0.4356 (5)	0.6654 (4)	1.18587 (16)	0.0870 (9)
H18A	−0.3269	0.5726	1.2051	0.130*
H18B	−0.4760	0.7319	1.2286	0.130*
H18C	−0.5687	0.6377	1.1714	0.130*
N1	−0.1082 (3)	0.8709 (2)	0.96121 (10)	0.0491 (5)
H1	−0.2422	0.9178	0.9776	0.059*
N2	0.1572 (3)	0.47195 (19)	0.58836 (10)	0.0479 (5)
H2	0.2753	0.4824	0.5571	0.058*
O1	−0.4543 (3)	0.8756 (2)	1.07739 (10)	0.0700 (5)
O2	0.5639 (3)	0.32473 (19)	0.51952 (11)	0.0745 (6)

following: N1–H1⋯O1 hydrogen bond (*d*(H1⋯O1)=2.04 Å, N2–H2⋯O2 hydrogen bond (*d*(H2⋯O2)=2.07 Å.

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