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Original Research Article

Preparation of Tris (Diphenyl Methanol) Binding by Hydrogen Bonds through the Application of Microwave Techniques

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Abstract

The reduction of a biologically important compound, Benzophenone has been done by applying a microwave technique to improve the final product of Benzhydrol crystals. The crystal has been characterized by single crystallography and its physical properties have been studied.

Keywords

Benzophenone derivatives
Diphenylmethanol
Microwaves
Single-crystal X-ray study

Introduction

Diphenylmathanol, [$(C_6H_5)_2CHOH$] and its derivatives are important in many aspects such as a pharmaceutical industry (Meltzer et al., 1996), for instance, antihypertensive, antihistamines agents and antiallergenic agents. Other applications of Diphenyl methanolare in the synthesis of agrochemicals such as a dichlorophen and hexachlorophene, alsothe substance plays a key role in a fixative in perfumery and as a terminating group in polymerizations reactions (http://chemical and 21.com/ lifescience/ phar/ BENZHYDROL.htm, accessed 25/10/2009).

The essential route to produce benzhydrol is the catalytic reduction of benzophenone, catalysts such as sodium borohydride, LiAlH4, Al(OiPr)3/iPrOH and sodium or aluminium amalgams, have been tested for reduction of benzophenone (Peng et al., 2005). But electrochemical (Sopher and Utley, 1984), catalytic hydrogenation (Hattori et al., 2001), electrocatalytic hydrogenation (Cirtiu et al., 2007), ultrasound irradiation reduction and photocatalytic reduction (Shiragami et al., 1989) as well as possible photochemical (Bachmann, 1933) represent substitute routes to the synthesis of Diphenylmethanol. Our study includes the applying of microwaves reduction to prepare three molecules bound by hydrogen bonds as depicted in the figure.

Materials and methods

Di-phenyl Benzophenon (1 g, 5.51 mmol) and sodium Borohydrate (1.0417g, 27.55 mmol) were stirred in methanol (50 ml) for (1 h.).The mixture was treated with microwave for 3 minutes. After concentration, the product was isolated by (silica gel) uni plate chromatography (9.5:0.5 dichloromethane:methanol) at Rf=(0.8), m.p. (64°C).

Diffractometer

Rigaku AFC12 goniometer equipped with an enhanced sensitivity (HG) *Saturn724+* detector mounted at the window of an *FR-E+ SuperBright* molybdenum rotating anode generator with VHF *Varimax* optics (70µm focus).

Cell determination and data collection: *Crystal Clear-SM Expert 2.0 r11* (Rigaku, 2011).

Data reduction, cell refinement and absorption correction: *CrystalClear-SM Expert 2.0 r13* (Rigaku, 2011).

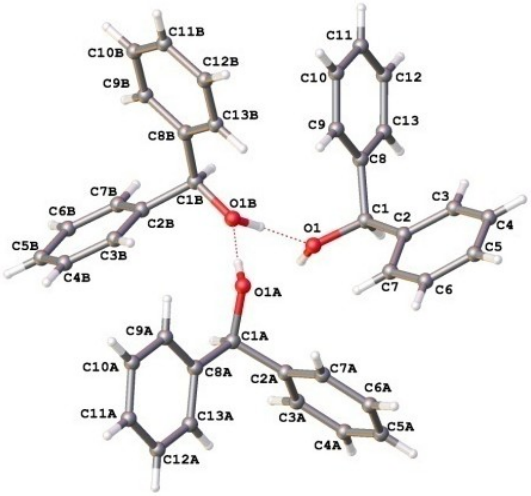
Structure solution: *SHELXS97* (Sheldrick, 2008) within OLEX2.

Graphics: *OLEX2* (Dolomanov et al., 2009).

Results and discussion

Hydrogen bond formed between an electropositive atom (actually hydrogen) and a strongly electronegative (oxygen atom). The force of the bond is really a special case of dipole forces, to recognize the possibility of hydrogen bonding, the electronegative oxygen atom have two unshared pairs of electron, and has a negative partial charge. While hydrogen atom has a partial positive charge, tries to find another atom as oxygen with excess electrons to share. The attraction has been obtained between the two atoms. As a consequence of this speech it was fixatives by single crystallography which appears below in Fig. 1 showing the structure and atom labeling scheme, and the other conclusions are summarized in Tables given as supplementary materials (Table 1-Crystal data and structure refinement details; Table 2-Atomic coordinates [× 10^4], equivalent isotropic displacement parameters [$Å^2$× 10^3] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor; Table 3-Bond lengths [Å] and angles [°]; Table 4-Anisotropic displacement parameters [$Å^2$× 10^3]. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ...+ 2\ h\ k\ a^*\ b^*\ U^{12}]$; Table 5-Hydrogen coordinates [× 10^4] and isotropic displacement parameters [$Å^2$× 10^3]; Table 6-Torsion angles [°]). The measurements of the crystal have been done with the Diffractometer.

Fig. 1: Sengel crystal of diphenyl methanol with crystal data.



$C_{13}H_{12}O$	v= 1497.02(18) $Å^3$
M_r=184.23	z=6
Triclinic p_1	µ = 1.72 mm^{-1}
a = 5.8589(4) Å	α = 88.231(6)° b = 12.4155(9) Å
β = 84.356(6)°	
c= 20.7737(15) Å	γ= 84.714(6)°
T=293(12)K	0.08 × 0.04 × 0.01 mm^3

Supplementary materials

Di-phenyl methanol

Improvement

H-atoms were placed in calculated positions [O1–C1=1.4348(16),O1–H1=0.91(2), O1A–C1A= 1.4362(15), O1A–H1A=1.4348(16), O1B–C1B= 1.4352(15), O1B–H1B=0.923(19)] and were included in the refinement in the riding model approximation.

Acknowledgement

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References

Bachmann, W. E., 1933. The photochemical reduction of ketones to hydrols. J. Am. Chem. Soc. 55, 391-395.

Cirtiu, C. M., Brisach-Wittmeyer, A., Menard, H., 2007. Comparative study of catalytic and electrocatalytic hydrogenation of benzophenone. Catal. Commun. 8, 751-754.

Dolomanov, O.V., Bourhis, L.J., Gildea, R.J., Howard, J.A.K., Puschmann, H., 2009. OLEX2: a complete structure solution, refinement and analysis program. J. Appl. Cryst. 42, 339-341.

Hattori, K., Sajiki, H., Hirota, K., 2001. Chemoselective control of hydrogenation among aromatic carbonyl and benzyl alcohol derivatives using Pd/C(en) catalyst. Tetrahedron 57, 4817-4824.

http://chemicalland21.com/lifescience/phar/BENZHYDROL.htm, accessed 25/10/2009.

Meltzer, P.C., Liang, A.Y., Madras, B.K., 1996. 2-Carbomethoxy-3-(diarylmethoxy)-1 alpha H, 5 alpha H-tropane analogs: synthesis and inhibition of binding at the dopamine transporter and comparison with piperazines of the GBR series. J. Med. Chem. 39, 371-379.

Peng, Y., Zhong, W., Song, G., 2005. Efficient and mild room temperature reduction of benzophenones under ultrasound irradiation. Ultrason. Sonochem. 12, 169-172.

Rigaku Corporation, CrystalClear-SM Expert 2.0 r13, Tokyo, Japan, 2011

Sheldrick, G. M., 2008. A short history of SHELX. Acta Cryst. A64, 112–122.

Shiragami, T., Pac, C., Yanagida, S., 1989. Nonmetallised CdS-catalysed photoreduction of aromatic ketones to alcohols and/or pinacols. J. Chem. Soc. Chem. Commun. 13, 831-832.

Sopher, D.W., Utley, J.H.P., 1984. Electro-organic reactions. Part 25. The role of chromium (III) in the modification of cathodic pinacolisation. J. Chem. Soc. Perkin Trans. 2, 1361-1367.

Supplementary Materials

Table 1. Details of information for Unit cell dimensions.

Identification code	2012ncs0885
Empirical formula	$C_{13}H_{12}O$
Formula weight	184.23
Temperature	293(2) K
Wavelength	0.71075 Å
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions	
a = 5.8589(4) Å	α = 88.231(6)°
b = 12.4155(9) Å	β = 84.356(6)°
c = 20.7737(15) Å	γ= 84.714(6)°
Volume	1497.02(18) Å^3
Z	6
Density (calculated)	1.226 Mg / m^3
Absorption coefficient	0.076 mm^{-1}
F(000)	588
Crystal	Tablet colourless
Crystal size	0.08 × 0.04 × 0.01 mm^3
θ range for data collection	3.30 – 27.48°
Index ranges	$-7 \le h \le 7$, $-16 \le k \le 15$, $-26 \le l \le 24$
Reflections collected	26336
Independent reflections	6854 [R_{int} = 0.0411]
Completeness to θ = 27.48°	99.6 %
Absorption correction	Semi–empirical from equivalents
Max. and min. transmission	1.000 and 0.899
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6854 / 0 / 391
Goodness-of-fit on F^2	1.026
Final R indices [$F^2 > 2\sigma(F^2)$]	$R1$ = 0.0432, $wR2$ = 0.0948
R indices (all data)	$R1$ = 0.0673, $wR2$ = 0.1051
Largest diff. peak and hole	0.216 and –0.209 e Å^{-3}

Table 2. Atomic coordinates [x 10^4], equivalent isotropic displacement parameters [$Å^2$x 10^3] and site occupancy factors. U_{eq} is defined as one third of the trace of the orthogonalized U^{ij} tensor.

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
O1	1797(2)	2542(1)	1968(1)	26(1)	1
O1A	8367(2)	1483(1)	2572(1)	26(1)	1
O1B	4578(2)	2816(1)	2880(1)	24(1)	1
C1	1311(2)	3182(1)	1403(1)	22(1)	1
C1A	7875(2)	371(1)	2632(1)	22(1)	1
C1B	3428(2)	3453(1)	3400(1)	20(1)	1
C2	3307(2)	2993(1)	877(1)	22(1)	1
C2A	7702(2)	–100(1)	1975(1)	22(1)	1
C2B	2982(2)	2758(1)	4003(1)	20(1)	1
C3	3367(3)	3651(1)	320(1)	27(1)	1
C3A	5785(2)	–617(1)	1855(1)	26(1)	1
C3B	4758(2)	2083(1)	4237(1)	23(1)	1
C4	5213(3)	3546(1)	–150(1)	29(1)	1
C4A	5680(3)	–1082(1)	1260(1)	32(1)	1
C4B	4377(3)	1479(1)	4802(1)	28(1)	1
C5	7036(3)	2772(1)	–75(1)	28(1)	1
C5A	7484(3)	–1032(1)	779(1)	32(1)	1
C5B	2201(3)	1538(1)	5140(1)	31(1)	1
C6	6966(3)	2102(1)	468(1)	29(1)	1
C6A	9406(3)	–521(1)	897(1)	29(1)	1
C6B	431(3)	2205(1)	4912(1)	32(1)	1
C7	5124(2)	2206(1)	943(1)	25(1)	1
C7A	9516(2)	–58(1)	1491(1)	24(1)	1
C7B	811(2)	2814(1)	4346(1)	27(1)	1
C8	931(2)	4378(1)	1548(1)	22(1)	1
C8A	9709(2)	–278(1)	2992(1)	20(1)	1
C8B	4853(2)	4373(1)	3539(1)	20(1)	1
C9	2613(2)	4884(1)	1828(1)	24(1)	1
C9A	11186(2)	226(1)	3346(1)	24(1)	1
C9B	4185(2)	5004(1)	4078(1)	27(1)	1
C10	2389(3)	5994(1)	1909(1)	30(1)	1
C10A	12878(2)	–385(1)	3658(1)	26(1)	1
C10B	5474(3)	5825(1)	4230(1)	31(1)	1
C11	475(3)	6614(1)	1706(1)	34(1)	1
C11A	13142(2)	–1499(1)	3617(1)	25(1)	1
C11B	7436(3)	6041(1)	3837(1)	29(1)	1
C12	–1209(3)	6120(1)	1438(1)	35(1)	1
C12A	11644(2)	–2011(1)	3277(1)	24(1)	1
C12B	8089(2)	5431(1)	3294(1)	29(1)	1
C13	–986(2)	5006(1)	1360(1)	29(1)	1
C13A	9931(2)	–1405(1)	2974(1)	23(1)	1
C13B	6811(2)	4598(1)	3145(1)	23(1)	1

Table 3. Bond lengths [Å] and angles [°].

Bond lengths [Å] and angles [°]		Bond lengths [Å] and angles [°].	
O1–C1	1.4348(16)	C3A–C2A–C7A	119.05(13)
O1–H1	0.91(2)	C3A–C2A–C1A	120.75(12)
O1A–C1A	1.4362(15)	C7A–C2A–C1A	120.14(12)
O1A–H1A	0.936(19)	C3B–C2B–C7B	118.85(13)
O1B–C1B	1.4352(15)	C3B–C2B–C1B	120.49(12)
O1B–H1B	0.923(19)	C7B–C2B–C1B	120.61(12)
C1–C8	1.5159(19)	C4–C3–C2	121.07(13)
C1–C2	1.5260(19)	C4–C3–H3	119.5
C1–H1C	0.9800	C2–C3–H3	119.5
C1A–C2A	1.5177(19)	C4A–C3A–C2A	120.35(13)
C1A–C8A	1.5235(19)	C4A–C3A–H3A	119.8
C1A–H1AA	0.9800	C2A–C3A–H3A	119.8
C1B–C2B	1.5139(18)	C4B–C3B–C2B	120.64(13)
C1B–C8B	1.5252(18)	C4B–C3B–H3B	119.7
C1B–H1BA	0.9800	C2B–C3B–H3B	119.7
C2–C7	1.3910(19)	C3–C4–C5	120.10(13)
C2–C3	1.3956(19)	C3–C4–H4	120.0
C2A–C3A	1.3908(19)	C5–C4–H4	120.0
C2A–C7A	1.3937(19)	C5A–C4A–C3A	120.28(14)
C2B–C3B	1.3914(19)	C5A–C4A–H4A	119.9
C2B–C7B	1.3921(19)	C3A–C4A–H4A	119.9
C3–C4	1.383(2)	C3B–C4B–C5B	120.06(14)
C3–H3	0.9300	C3B–C4B–H4B	120.0
C3A–C4A	1.391(2)	C5B–C4B–H4B	120.0
C3A–H3A	0.9300	C6–C5–C4	119.20(13)
C3B–C4B	1.3842(19)	C6–C5–H5	120.4
C3B–H3B	0.9300	C4–C5–H5	120.4
C4–C5	1.387(2)	C4A–C5A–C6A	119.60(14)
C4–H4	0.9300	C4A–C5A–H5A	120.2
C4A–C5A	1.385(2)	C6A–C5A–H5A	120.2
C4A–H4A	0.9300	C6B–C5B–C4B	119.71(13)
C4B–C5B	1.390(2)	C6B–C5B–H5B	120.1
C4B–H4B	0.9300	C4B–C5B–H5B	120.1
C5–C6	1.380(2)	C5–C6–C7	120.99(14)
C5–H5	0.9300	C5–C6–H6	119.5
C5A–C6A	1.388(2)	C7–C6–H6	119.5
C5A–H5A	0.9300	C7A–C6A–C5A	120.27(14)
C5B–C6B	1.379(2)	C7A–C6A–H6A	119.9
C5B–H5B	0.9300	C5A–C6A–H6A	119.9
C6–C7	1.388(2)	C5B–C6B–C7B	120.31(14)
C6–H6	0.9300	C5B–C6B–H6B	119.8
C6A–C7A	1.386(2)	C7B–C6B–H6B	119.8
C6A–H6A	0.9300	C6–C7–C2	120.23(13)
C6B–C7B	1.389(2)	C6–C7–H7	119.9
C6B–H6B	0.9300	C2–C7–H7	119.9
C7–H7	0.9300	C6A–C7A–C2A	120.44(14)
C7A–H7A	0.9300	C6A–C7A–H7A	119.8
C7B–H7B	0.9300	C2A–C7A–H7A	119.8
C8–C13	1.3887(19)	C6B–C7B–C2B	120.43(14)
C8–C9	1.3975(19)	C6B–C7B–H7B	119.8

Bond lengths [Å] and angles [°]		Bond lengths [Å] and angles [°].	
C8A–C9A	1.3906(18)	C2B–C7B–H7B	119.8
C8A–C13A	1.3954(18)	C13–C8–C9	118.73(13)
C8B–C13B	1.3868(18)	C13–C8–C1	121.03(12)
C8B–C9B	1.3913(18)	C9–C8–C1	120.04(12)
C9–C10	1.3856(19)	C9A–C8A–C13A	118.28(13)
C9–H9	0.9300	C9A–C8A–C1A	121.64(12)
C9A–C10A	1.391(2)	C13A–C8A–C1A	120.08(12)
C9A–H9A	0.9300	C13B–C8B–C9B	118.65(12)
C9B–C10B	1.3848(19)	C13B–C8B–C1B	122.06(11)
C9B–H9B	0.9300	C9B–C8B–C1B	119.29(12)
C10–C11	1.393(2)	C10–C9–C8	120.68(13)
C10–H10	0.9300	C10–C9–H9	119.7
C10A–C11A	1.3822(19)	C8–C9–H9	119.7
C10A–H10A	0.9300	C10A–C9A–C8A	120.46(12)
C10B–C11B	1.385(2)	C10A–C9A–H9A	119.8
C10B–H10B	0.9300	C8A–C9A–H9A	119.8
C11–C12	1.379(2)	C10B–C9B–C8B	120.87(13)
C11–H11	0.9300	C10B–C9B–H9B	119.6
C11A–C12A	1.3880(19)	C8B–C9B–H9B	119.6
C11A–H11A	0.9300	C9–C10–C11	119.79(14)
C11B–C12B	1.380(2)	C9–C10–H10	120.1
C11B–H11B	0.9300	C11–C10–H10	120.1
C12–C13	1.389(2)	C11A–C10A–C9A	120.83(13)
C12–H12	0.9300	C11A–C10A–H10A	119.6
C12A–C13A	1.3870(19)	C9A–C10A–H10A	119.6
C12A–H12A	0.9300	C9B–C10B–C11B	120.12(13)
C12B–C13B	1.3910(19)	C9B–C10B–H10B	119.9
C12B–H12B	0.9300	C11B–C10B–H10B	119.9
C13–H13	0.9300	C12–C11–C10	119.95(14)
C13A–H13A	0.9300	C12–C11–H11	120.0
C13B–H13B	0.9300	C10–C11–H11	120.0
C1–O1–H1	109.4(12)	C10A–C11A–C12A	119.15(13)
C1A–O1A–H1A	108.4(12)	C10A–C11A–H11A	120.4
C1B–O1B–H1B	109.3(11)	C12A–C11A–H11A	120.4
O1–C1–C8	111.69(11)	C12B–C11B–C10B	119.42(13)
O1–C1–C2	109.49(11)	C12B–C11B–H11B	120.3
C8–C1–C2	109.37(10)	C10B–C11B–H11B	120.3
O1–C1–H1C	108.7	C11–C12–C13	120.14(15)
C8–C1–H1C	108.7	C11–C12–H12	119.9
C2–C1–H1C	108.7	C13–C12–H12	119.9
O1A–C1A–C2A	111.11(11)	C13A–C12A–C11A	120.11(12)
O1A–C1A–C8A	109.71(11)	C13A–C12A–H12A	119.9
C2A–C1A–C8A	111.28(11)	C11A–C12A–H12A	119.9
O1A–C1A–H1AA	108.2	C11B–C12B–C13B	120.54(13)
C2A–C1A–H1AA	108.2	C11B–C12B–H12B	119.7
C8A–C1A–H1AA	108.2	C13B–C12B–H12B	119.7
O1B–C1B–C2B	110.81(10)	C12–C13–C8	120.70(14)
O1B–C1B–C8B	110.26(10)	C12–C13–H13	119.7
C2B–C1B–C8B	110.39(10)	C8–C13–H13	119.7
O1B–C1B–H1BA	108.4	C12A–C13A–C8A	121.09(12)
C2B–C1B–H1BA	108.4	C12A–C13A–H13A	119.5

Bond lengths [Å] and angles [°]		Bond lengths [Å] and angles [°].	
C8B–C1B–H1BA	108.4	C8A–C13A–H13A	119.5
C7–C2–C3	118.39(13)	C8B–C13B–C12B	120.39(12)
C7–C2–C1	122.22(12)	C8B–C13B–H13B	119.8
C3-C2-C1	119.36(12)	C12B-C13B-H13B	119.8

Table 4. Anisotropic displacement parameters [Å^{2}x 10^3]. The anisotropic displacement factor exponent takes the form: -2□^{2}[h^{2}a*^{2}U^{11} + ... + 2 h k a* b* U^{12}].

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O1	29(1)	29(1)	22(1)	8(1)	–5(1)	–13(1)
O1A	24(1)	17(1)	36(1)	1(1)	5(1)	–3(1)
O1B	24(1)	26(1)	20(1)	–6(1)	–4(1)	1(1)
C1	24(1)	25(1)	20(1)	4(1)	–6(1)	–8(1)
C1A	21(1)	19(1)	26(1)	0(1)	4(1)	–6(1)
C1B	19(1)	21(1)	19(1)	–3(1)	–1(1)	0(1)
C2	26(1)	20(1)	20(1)	–2(1)	–5(1)	–7(1)
C2A	22(1)	18(1)	25(1)	3(1)	–4(1)	1(1)
C2B	24(1)	18(1)	21(1)	–3(1)	–4(1)	–7(1)
C3	32(1)	26(1)	22(1)	2(1)	–3(1)	2(1)
C3A	22(1)	25(1)	32(1)	2(1)	–4(1)	0(1)
C3B	25(1)	21(1)	24(1)	–2(1)	–3(1)	–7(1)
C4	41(1)	25(1)	22(1)	3(1)	–1(1)	–2(1)
C4A	28(1)	30(1)	39(1)	–2(1)	–12(1)	–2(1)
C4B	39(1)	19(1)	27(1)	0(1)	–10(1)	–8(1)
C5	32(1)	28(1)	23(1)	–5(1)	2(1)	–4(1)
C5A	38(1)	29(1)	29(1)	–3(1)	–12(1)	5(1)
C5B	44(1)	29(1)	23(1)	3(1)	–4(1)	–17(1)
C6	32(1)	26(1)	29(1)	–4(1)	–4(1)	3(1)
C6A	33(1)	28(1)	24(1)	2(1)	–1(1)	5(1)
C6B	32(1)	41(1)	26(1)	–1(1)	3(1)	–16(1)
C7	34(1)	20(1)	23(1)	3(1)	–6(1)	–4(1)
C7A	24(1)	22(1)	27(1)	2(1)	–2(1)	–1(1)
C7B	23(1)	31(1)	28(1)	–3(1)	–3(1)	–8(1)
C8	22(1)	26(1)	18(1)	2(1)	2(1)	–4(1)
C8A	22(1)	21(1)	17(1)	0(1)	5(1)	–6(1)
C8B	22(1)	18(1)	19(1)	3(1)	–2(1)	–2(1)
C9	25(1)	26(1)	21(1)	0(1)	2(1)	–4(1)
C9A	30(1)	21(1)	21(1)	–3(1)	2(1)	–9(1)
C9B	30(1)	23(1)	26(1)	0(1)	6(1)	–6(1)
C10	37(1)	29(1)	23(1)	–3(1)	4(1)	–10(1)
C10A	29(1)	30(1)	20(1)	–1(1)	–2(1)	–14(1)
C10B	44(1)	22(1)	26(1)	–5(1)	2(1)	–8(1)
C11	47(1)	24(1)	27(1)	–1(1)	8(1)	1(1)

Atom	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C11A	28(1)	28(1)	20(1)	3(1)	–3(1)	–7(1)
C11B	36(1)	19(1)	34(1)	1(1)	–5(1)	–10(1)
C12	35(1)	36(1)	30(1)	4(1)	5(1)	7(1)
C12A	32(1)	19(1)	23(1)	1(1)	–2(1)	–6(1)
C12B	26(1)	27(1)	32(1)	4(1)	2(1)	–7(1)
C13	25(1)	36(1)	25(1)	2(1)	0(1)	–3(1)
C13A	27(1)	22(1)	21(1)	–2(1)	–3(1)	–10(1)
C13B	24(1)	24(1)	22(1)	–1(1)	–1(1)	–2(1)

Table 5. Hydrogen coordinates [x 10^4] and isotropic displacement parameters [Å^{2}x 10^3].

Atom	*x*	*y*	*z*	U_{eq}	*S.o.f.*
H1	530(30)	2215(15)	2123(9)	55(6)	1
H1A	7010(30)	1918(15)	2692(9)	54(6)	1
H1B	3570(30)	2742(14)	2574(9)	48(5)	1
H1C	–86	2955	1243	27	1
H1AA	6386	337	2889	27	1
H1BA	1944	3768	3269	24	1
H3	2147	4169	264	32	1
H3A	4566	–653	2176	32	1
H3B	6215	2038	4012	28	1
H4	5230	3994	–517	35	1
H4A	4394	–1428	1183	38	1
H4B	5578	1033	4956	33	1
H5	8292	2705	–387	33	1
H5A	7409	–1340	380	38	1
H5B	1940	1128	5518	37	1
H6	8171	1572	516	35	1
H6A	10625	–489	577	35	1
H6B	–1025	2247	5138	39	1
H7	5105	1748	1305	30	1
H7A	10810	284	1566	29	1
H7B	–392	3262	4195	32	1
H9	3896	4472	1961	29	1
H9A	11041	975	3375	29	1
H9B	2856	4872	4341	32	1
H10	3512	6323	2097	36	1
H10A	13843	–39	3897	31	1
H10B	5022	6233	4596	37	1
H11	333	7360	1752	41	1
H11A	14310	–1901	3814	30	1
H11B	8306	6591	3938	35	1
H12	–2497	6533	1308	42	1
H12A	11788	–2761	3252	29	1
H12B	9395	5579	3026	34	1
H13	–2133	4679	1181	34	1
H13A	8915	–1757	2755	27	1
H13B	7272	4190	2780	28	1

Table 6. Symmetry transformations used to generate equivalent atoms-Torsion angles [°]

Torsion angles [°]		Torsion angles [°]	
O1–C1–C2–C7	7.31(17)	O1–C1–C8–C13	–129.94(13)
C8–C1–C2–C7	130.00(13)	C2–C1–C8–C13	108.69(14)
O1–C1–C2–C3	–170.52(11)	O1–C1–C8–C9	55.32(16)
C8–C1–C2–C3	–47.83(16)	C2–C1–C8–C9	–66.05(15)
O1A–C1A–C2A–C3A	–127.44(13)	O1A–C1A–C8A–C9A	16.02(16)
C8A–C1A–C2A–C3A	109.99(13)	C2A–C1A–C8A–C9A	139.40(12)
O1A–C1A–C2A–C7A	55.29(16)	O1A–C1A–C8A–C13A	–164.39(11)
C8A–C1A–C2A–C7A	–67.29(15)	C2A–C1A–C8A–C13A	–41.01(16)
O1B–C1B–C2B–C3B	51.23(16)	O1B–C1B–C8B–C13B	7.59(17)
C8B–C1B–C2B–C3B	–71.22(15)	C2B–C1B–C8B–C13B	130.36(13)
O1B–C1B–C2B–C7B	–131.26(12)	O1B–C1B–C8B–C9B	–171.98(12)
C8B–C1B–C2B–C7B	106.30(14)	C2B–C1B–C8B–C9B	–49.21(16)
C7–C2–C3–C4	–1.6(2)	C13–C8–C9–C10	–0.85(19)
C1–C2–C3–C4	176.28(13)	C1–C8–C9–C10	174.00(12)
C7A–C2A–C3A–C4A	–0.2(2)	C13A–C8A–C9A–C10A	1.78(18)
C1A–C2A–C3A–C4A	–177.53(13)	C1A–C8A–C9A–C10A	–178.62(12)
C7B–C2B–C3B–C4B	–0.12(19)	C13B–C8B–C9B–C10B	–1.5(2)
C1B–C2B–C3B–C4B	177.43(12)	C1B–C8B–C9B–C10B	178.12(13)
C2–C3–C4–C5	0.5(2)	C8–C9–C10–C11	–0.4(2)
C2A–C3A–C4A–C5A	–0.1(2)	C8A–C9A–C10A–C11A	0.7(2)
C2B–C3B–C4B–C5B	0.3(2)	C8B–C9B–C10B–C11B	1.1(2)
C3–C4–C5–C6	1.0(2)	C9–C10–C11–C12	1.2(2)
C3A–C4A–C5A–C6A	0.4(2)	C9A–C10A–C11A–C12A	–2.2(2)
C3B–C4B–C5B–C6B	–0.4(2)	C9B–C10B–C11B–C12B	0.1(2)
C4–C5–C6–C7	–1.3(2)	C10–C11–C12–C13	–0.9(2)
C4A–C5A–C6A–C7A	–0.3(2)	C10A–C11A–C12A–C13A	1.1(2)
C4B–C5B–C6B–C7B	0.2(2)	C10B–C11B–C12B–C13B	–0.8(2)
C5–C6–C7–C2	0.1(2)	C11–C12–C13–C8	–0.3(2)
C3–C2–C7–C6	1.4(2)	C9–C8–C13–C12	1.2(2)
C1–C2–C7–C6	–176.48(13)	C1–C8–C13–C12	–173.60(12)
C5A–C6A–C7A–C2A	0.0(2)	C11A–C12A–C13A–C8A	1.4(2)
C3A–C2A–C7A–C6A	0.3(2)	C9A–C8A–C13A–C12A	–2.82(19)
C1A–C2A–C7A–C6A	177.60(12)	C1A–C8A–C13A–C12A	177.57(12)
C5B–C6B–C7B–C2B	0.0(2)	C9B–C8B–C13B–C12B	0.8(2)
C3B–C2B–C7B–C6B	0.0(2)	C1B–C8B–C13B–C12B	–178.82(13)
C1B–C2B–C7B–C6B	–177.59(12)	C11B–C12B–C13B–C8B	0.4(2)