Journal of Molecular Structure, 318 (1994) 113–121
0022-2860/94/$07.00

The structure of tricyclohexylmethane

D.G. Gillies, S. Luff, G.W. Smith, L.H. Sutcliffe*

Chemistry Department, University of Surrey, Guildford GU2 5XH, U.K.

(Received 23 August 1993)

Abstract

Tricyclohexylmethane has a melting point close to ambient temperature. Nevertheless the X-ray structure has been determined. The crystal is chiral but its chirality could not be established. The solid is unusual in that the atomic volume per carbon atom is 22.2 Å^3 which is substantially greater than is usual for a hydrocarbon. Since there is little data in the literature on the compound we have reported the solution-state proton and proton-decoupled NMR spectra.

Introduction

We are studying model traction fluids with the aim of linking molecular structure with engineering performance, thus eventually leading to new practical fluids [1–3]. There are three stages in our investigations, (i) molecular modelling, (ii) synthesis, and (iii) magnetic resonance measurements at ambient temperatures and high pressures. Generally, traction fluids are hydrocarbons containing cyclohexyl rings. It has been shown [4,5] that the highest traction is attained by the tricyclic derivatives and we have synthesized several such compounds for evaluation. Among the latter is tricyclohexylmethane (1, 1′, 1″-methylidyne-tris-cyclohexane). The compound has a melting point near to room temperature but nevertheless, we have obtained some good quality single crystals which were subjected to X-ray examination.

Experimental

Tricyclohexylmethane was prepared as follows. Triphenylmethane (10 g, 41 mmol; Aldrich) was dissolved in a mixture of cyclohexane (95 cm^3) plus toluene (5 cm^3). The solution was placed in a 500 cm^3 capacity Parr pressure vessel (S and M Products, Shirley Institute, Didsbury, Manchester) along with a slurry of activated Raney nickel catalyst (Sigma). Hydrogenation was then carried out at 400 psi and 100°C [6]. The crude product was obtained in 90% yield: it was recrystallized from ethanol (m.p. 57.5/58.5°C). Complete hydrogenation was proved by 300 MHz proton and 75 MHz carbon-13 NMR spectroscopy using a Bruker AC300 NMR spectrometer. As the spectra have not been reported before they are reproduced in Figs. 1 and 2. Without a more detailed examination, the proton spectra cannot be analysed fully; however, the group of lines centred at about 1.6 ppm can be assigned to the equatorial protons and the lines centred at about 1.2 ppm arise from the axial protons. The low intensity multiplet at 0.75 ppm is due to the proton attached to the central carbon atom, C1. No signals were observed in the aromatic region at about 7 ppm. The proton-decoupled carbon-13 spectrum is simple to assign, the carbon nuclei having the following chemical

* Corresponding author.

SSDI 0022-2860(93)07915-J

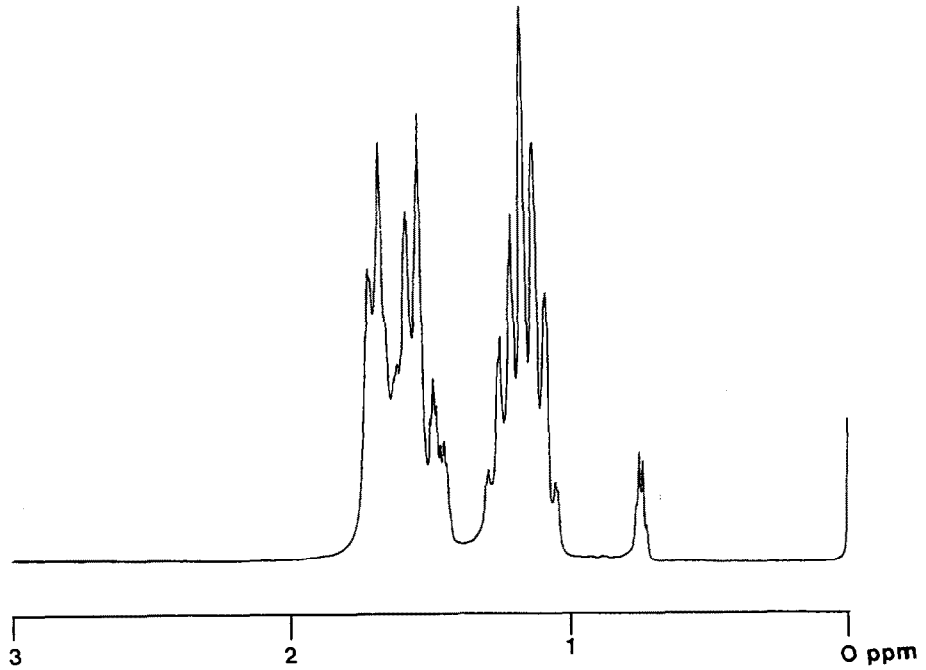

Fig. 1. The 300.1 MHz proton NMR spectrum of tricyclohexylmethane in deuteriochloroform with tetramethylsilane as internal reference.

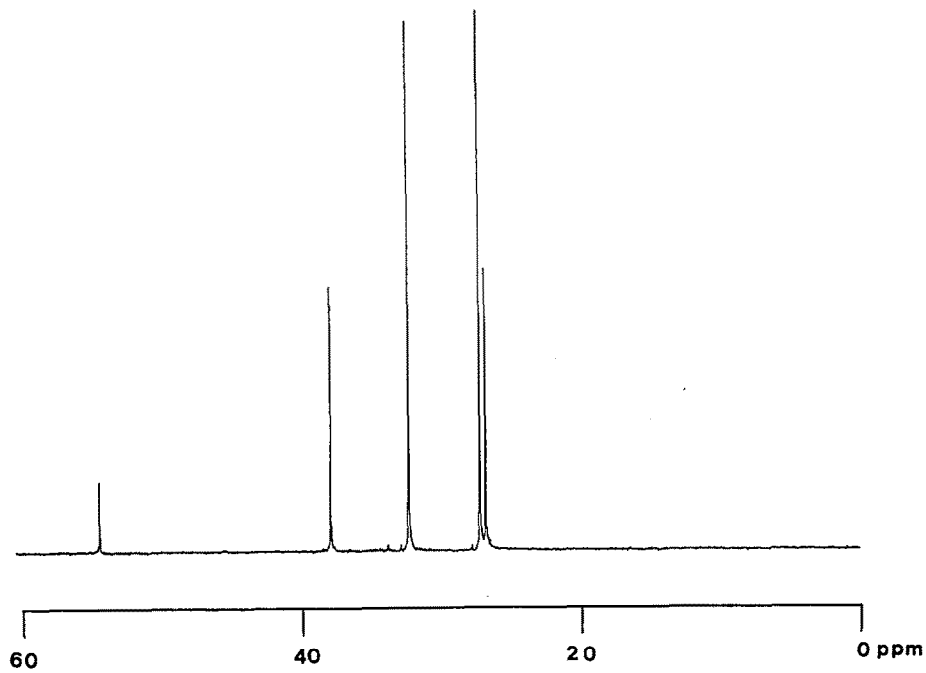

Fig. 2. The proton-decoupled 75.47 MHz carbon-13 NMR spectrum of tricyclohexylmethane in deuteriochloroform with tetramethylsilane as internal reference.

Table 1
Physical properties and parameters for data collection and refinement

Formula	$C_{19}H_{34}$
Mol. wt. (g)	262.48
Crystal system	Orthorhombic
Space group $P2_12_12_1$ (19)	
a (Å)	6.380 (8)
b (Å)	14.289 (4)
c (Å)	18.335 (4)
V (Å^3)	1687.9 (2.9)
Z	4
ρ (g cm^{-3}) calc.	1.033
μ (Mo Kα) (cm^{-1})	0.53
λ (Å)	0.71073
F (000)	592

Table 2
Positional parameters and their estimated standard deviations

Atom	x	y	z	B(A2)
C1	0.4339(3)	0.0683(1)	0.30393(9)	2.97(3)
C11	0.2370(3)	0.1231(1)	0.32881(9)	3.14(3)
C12	0.2509(4)	0.1473(1)	0.4103(1)	4.32(4)
C13	0.0554(4)	0.1975(2)	0.4374(1)	5.11(5)
C14	0.0150(4)	0.2852(2)	0.3944(1)	5.63(5)
C15	0.0032(4)	0.2642(1)	0.3139(1)	5.37(5)
C16	0.1973(4)	0.2124(1)	0.2863(1)	4.37(4)
C21	0.4641(3)	0.0675(1)	0.22027(9)	3.07(3)
C22	0.6840(3)	0.0356(1)	0.1971(1)	3.84(3)
C23	0.7144(3)	0.0433(2)	0.1152(1)	4.17(4)
C24	0.5442(3)	−0.0083(2)	0.0738(1)	4.38(4)
C25	0.3266(3)	0.0227(2)	0.0968(1)	4.10(4)
C26	0.2970(3)	0.0131(1)	0.17891(9)	3.47(3)
C31	0.4537(3)	−0.0316(1)	0.33583(9)	3.17(3)
C32	0.2503(3)	−0.0795(1)	0.3596(1)	3.88(3)
C33	0.2931(4)	−0.1802(1)	0.3809(1)	4.95(4)
C34	0.4604(4)	−0.1877(2)	0.4393(1)	5.27(5)
C35	0.6608(4)	−0.1392(2)	0.4171(1)	4.93(4)
C36	0.6196(3)	−0.0392(2)	0.3958(1)	4.39(4)
H1	0.5550	0.1038	0.3238	3*
H11	0.1126	0.0818	0.3218	3*
H12A	0.3745	0.1866	0.4192	5*
H12B	0.2701	0.0892	0.4390	5*
H13A	0.0713	0.2114	0.4899	5*
H13B	−0.0690	0.1545	0.4325	5*
H14A	0.1295	0.3304	0.4041	6*
H14B	−0.1216	0.3131	0.4109	6*
H15A	−0.0119	0.3238	0.2858	5*
H15B	−0.1255	0.2272	0.3029	5*
H16A	0.3225	0.2533	0.2896	4*
H16B	0.1793	0.1971	0.2333	4*
H21	0.4502	0.1336	0.2035	3*
H22A	0.7926	0.0730	0.2223	4*
H22B	0.7049	−0.0305	0.2118	4*
H23A	0.8548	0.0173	0.1011	4*
H23B	0.7121	0.1102	0.1003	4*
H24A	0.5614	0.0006	0.0204	5*
H24B	0.5586	−0.0766	0.0831	5*
H25A	0.3054	0.0883	0.0818	4*
H25B	0.2196	−0.0159	0.0703	4*
H26A	0.3048	−0.0543	0.1929	4*
H26B	0.1533	0.0363	0.1925	4*
H31	0.5100	−0.0701	0.2951	3*
H32A	0.1890	−0.0462	0.4025	4*
H32B	0.1475	−0.0780	0.3184	4*
H33A	0.3402	−0.2154	0.3363	5*
H33B	0.1629	−0.2101	0.3983	5*
H34A	0.4911	−0.2542	0.4487	5*
H34B	0.4074	−0.1603	0.4851	5*
H35A	0.7612	−0.1412	0.4588	5*
H35B	0.7258	−0.1727	0.3751	5*
H36A	0.7524	−0.0101	0.3791	5*
H36B	0.5715	−0.0040	0.4397	5*

Starred atoms were refined isotropically.
Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $(4/3)$-$[a2 \times B(1,1) + b2 \times B(2,2) + c2 \times B(3,3) + ab(\cos\gamma) \times B(1,2) + ac(\cos\beta) \times B(1,3) + bc(\cos\alpha) \times B(2,3)]$

shifts:

C1	54.39 p.p.m.
C11, C21, C31	37.84 p.p.m.
C12, C16, C22, C26, C32, C36	32.25 p.p.m.
C13, C15, C23, C25, C33, C35	27.19 p.p.m.
C14, C24, C34	26.76 p.p.m.

The compound crystallizes as long needles. A suitable specimen of approximate dimensions $0.4 \times 0.35 \times 0.6$ mm was mounted on a CADA diffractometer and its orthorhombic cell determined from 25 accurately centred reflections in the θ range 10–12°. The radiation used was monochromatized Mo Kα. Intensity data were collected from two octants of reciprocal space, $1° \leq \theta \leq 26°$ covering the index range $0 \leq h \leq 7$, $0 \leq k \leq 17$

Table 3
General temperature factor expressions

Name	$U(1,1)$	$U(2,2)$	$U(3,3)$	$U(1,2)$	$U(1,3)$	$U(2,3)$
C1	0.0293(7)	0.0405(7)	0.0428(8)	−0.0023(6)	−0.0021(7)	−0.0028(6)
C11	0.0357(8)	0.0386(7)	0.0450(8)	0.0002(7)	0.0003(7)	−0.0013(7)
C12	0.064(1)	0.0509(9)	0.0490(9)	0.0085(9)	0.0007(9)	−0.0064(8)
C13	0.077(1)	0.058(1)	0.060(1)	0.012(1)	0.017(1)	−0.0078(9)
C14	0.075(1)	0.050(1)	0.089(1)	0.015(1)	0.020(1)	−0.009(1)
C15	0.071(1)	0.0517(9)	0.081(1)	0.023(1)	0.010(1)	0.011(1)
C16	0.058(1)	0.0505(9)	0.057(1)	0.0112(8)	0.0106(9)	0.0071(8)
C21	0.0275(7)	0.0456(8)	0.0434(8)	−0.0007(7)	0.0013(6)	0.0006(7)
C22	0.0250(7)	0.067(1)	0.0534(9)	−0.0013(8)	0.0009(7)	−0.0039(8)
C23	0.0329(8)	0.068(1)	0.057(1)	−0.0027(8)	0.0110(8)	−0.0039(9)
C24	0.0471(9)	0.073(1)	0.0463(9)	−0.0008(9)	0.0070(8)	−0.0084(9)
C25	0.0380(8)	0.073(1)	0.0443(9)	−0.0036(9)	−0.0027(8)	−0.0018(8)
C26	0.0263(6)	0.0616(9)	0.0441(8)	−0.0020(7)	0.0003(7)	−0.0022(7)
C31	0.0324(7)	0.0442(8)	0.0440(7)	0.0035(7)	−0.0010(7)	0.0000(7)
C32	0.0378(8)	0.0455(8)	0.064(1)	−0.0017(8)	0.0017(8)	0.0059(8)
C33	0.058(1)	0.0462(9)	0.085(1)	−0.0010(9)	0.005(1)	0.008(1)
C34	0.085(1)	0.053(1)	0.062(1)	0.016(1)	0.006(1)	0.0128(9)
C35	0.062(1)	0.066(1)	0.059(1)	0.018(1)	−0.0159(9)	0.0033(9)
C36	0.0446(9)	0.062(1)	0.061(1)	0.0032(9)	−0.0136(8)	0.0051(9)

The form of the anisotropic thermal parameter is: $\exp\{-2PI2[h2a2U(1,1) + k2b2U(2,2) + l2c2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)]\}$ where a, b, and c are reciprocal lattice constants.

Table 4
Bond distances (Å)

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
C1	C11	1.553(2)	C16	H16A	0.996(2)	C31	C32	1.534(2)
C1	C21	1.546(2)	C16	H16B	1.002(2)	C31	C36	1.530(3)
C1	C31	1.560(2)	C21	C22	1.537(2)	C31	H31	0.998(2)
C1	H1	0.997(2)	C21	C26	1.526(2)	C32	C33	1.529(3)
C11	C12	1.536(2)	C21	H21	1.007(2)	C32	H32A	1.002(2)
C11	C16	1.527(2)	C22	C23	1.518(3)	C32	H32B	1.000(2)
C11	H11	1.000(2)	C22	H22A	0.994(2)	C33	C34	1.517(3)
C12	C13	1.526(3)	C22	H22B	1.001(2)	C33	H33A	1.008(2)
C12	H12A	0.986(2)	C23	C24	1.519(3)	C33	H33B	0.989(2)
C12	H12B	0.997(2)	C23	H23A	1.004(2)	C34	C35	1.513(3)
C13	C14	1.513(3)	C23	H23B	1.004(2)	C34	H34A	0.995(2)
C13	H13A	0.989(2)	C24	C25	1.518(3)	C34	H34B	0.987(2)
C13	H13B	1.012(2)	C24	H24A	0.993(2)	C35	C36	1.518(3)
C14	C15	1.508(4)	C24	H24B	1.004(2)	C35	H35A	0.998(2)
C14	H14A	0.996(2)	C25	C26	1.524(2)	C35	H35B	0.999(2)
C14	H14B	1.008(3)	C25	H25A	0.995(2)	C36	H36A	0.993(2)
C15	C16	1.533(3)	C25	H25B	1.006(2)	C36	H36B	1.001(2)
C15	H15A	1.008(2)	C26	H26A	1.007(2)			
C15	H15B	1.000(2)	C26	H26B	1.007(2)			

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table 5
Bond angles (deg)

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
C11	C1	C21	113.3(1)	C11	C16	H16B	109.2(2)	C25	C26	H26B	109.2(2)
C11	C1	C31	115.2(1)	C15	C16	H16A	109.7(2)	H26A	C26	H26B	107.6(2)
C11	C1	H1	104.9(1)	C15	C16	H16B	109.6(2)	C1	C31	C32	117.0(1)
C21	C1	C31	110.8(1)	H16A	C16	H16B	106.3(2)	C1	C31	C36	113.0(1)
C21	C1	H1	105.7(1)	C1	C21	C22	113.0(1)	C1	C31	H31	105.3(1)
C31	C1	H1	106.0(1)	C1	C21	C26	114.2(1)	C32	C31	C36	110.4(2)
C1	C11	C12	110.8(1)	C1	C21	H21	106.5(1)	C32	C31	H31	105.4(2)
C1	C11	C16	114.5(1)	C22	C21	C26	110.2(1)	C36	C31	H31	104.4(2)
C1	C11	H11	107.6(1)	C22	C21	H21	106.3(1)	C31	C32	C33	110.5(2)
C12	C11	C16	108.3(1)	C26	C21	H21	106.0(1)	C31	C32	H32A	109.7(2)
C12	C11	H11	107.8(2)	C21	C22	C23	111.6(1)	C31	C32	H32B	109.3(2)
C16	C11	H11	107.7(2)	C21	C22	H22A	110.2(2)	C33	C32	H32A	109.0(2)
C11	C12	C13	112.2(2)	C21	C22	H22B	109.4(2)	C33	C32	H32B	109.3(2)
C11	C12	H12A	109.9(2)	C23	C22	H22A	109.4(2)	H32A	C32	H32B	109.1(2)
C11	C12	H12B	109.3(2)	C23	C22	H22B	108.7(2)	C32	C33	C34	111.9(2)
C13	C12	H12A	109.0(2)	H22A	C22	H22B	107.4(2)	C32	C33	H33A	109.0(2)
C13	C12	H12B	109.1(2)	C22	C23	C24	111.5(2)	C32	C33	H33B	110.3(2)
H12A	C12	H12B	107.2(2)	C22	C23	H23A	109.8(2)	C34	C33	H33A	109.0(2)
C12	C13	C14	111.5(2)	C22	C23	H23B	109.6(2)	C34	C33	H33B	109.3(2)
C12	C13	H13A	109.3(2)	C24	C23	H23A	109.1(2)	H33A	C33	H33B	107.1(2)
C12	C13	H13B	108.7(2)	C24	C23	H23B	109.1(2)	C33	C34	C35	111.8(2)
C14	C13	H13A	110.8(2)	H23A	C23	H23B	107.6(2)	C33	C34	H34A	109.3(2)
C14	C13	H13B	109.5(2)	C23	C24	C25	111.7(2)	C33	C34	H34B	109.3(2)
H13A	C13	H13B	107.0(2)	C23	C24	H24A	110.4(2)	C35	C34	H34A	109.1(2)
C13	C14	C15	110.6(2)	C23	C24	H24B	109.3(2)	C35	C34	H34B	109.5(2)
C13	C14	H14A	109.2(2)	C25	C24	H24A	109.6(2)	H34A	C34	H34B	107.8(2)
C13	C14	H14B	108.9(2)	C25	C24	H24B	109.0(2)	C34	C35	C36	111.3(2)
C15	C14	H14A	110.1(2)	H24A	C24	H24B	106.6(2)	C34	C35	H35A	108.8(2)
C15	C14	H14B	109.4(2)	C24	C25	C26	111.2(2)	C34	C35	H35B	109.5(2)
C14	C13	H13A	110.8(2)	H23A	C23	H23B	107.6(2)	C33	C34	H34A	109.3(2)
C14	C13	H13B	109.5(2)	C23	C24	C25	111.7(2)	C33	C34	H34B	109.3(2)
H13A	C13	H13B	107.0(2)	C23	C24	H24A	110.4(2)	C35	C34	H34A	109.1(2)
C13	C14	C15	110.6(2)	C23	C24	H24B	109.3(2)	C35	C34	H34B	109.5(2)
C13	C14	H14A	109.2(2)	C25	C24	H24A	109.6(2)	H34A	C34	H34B	107.8(2)
C13	C14	H14B	108.9(2)	C25	C24	H24B	109.0(2)	C34	C35	C36	111.3(2)
C15	C14	H14A	110.1(2)	H24A	C24	H24B	106.6(2)	C34	C35	H35A	108.8(2)
C15	C14	H14B	109.4(2)	C24	C25	C26	111.2(2)	C34	C35	H35B	109.5(2)
H14A	C14	H14B	108.6(2)	C24	C25	H25A	109.2(2)	C36	C35	H35A	109.6(2)
C14	C15	C16	112.4(2)	C24	C25	H25B	108.9(2)	C36	C35	H35B	109.5(2)
C14	C15	H15A	109.5(2)	C26	C25	H25A	110.0(2)	H35A	C35	H35B	108.0(2)
C14	C15	H15B	110.2(2)	C26	C25	H25B	110.0(2)	C31	C36	C35	111.9(2)
C16	C15	H15A	109.0(2)	H25A	C25	H25B	107.6(2)	C31	C36	H36A	109.8(2)
C16	C15	H15B	109.7(2)	C21	C26	C25	111.0(1)	C31	C36	H36B	109.3(2)
H15A	C15	H15B	105.9(2)	C21	C26	H26A	109.6(1)	C35	C36	H36A	109.4(2)
C11	C16	C15	112.2(2)	C21	C26	H26B	109.9(2)	C35	C36	H36B	109.1(2)
C11	C16	H16A	109.7(2)	C25	C26	H26A	109.6(2)	H36A	C36	H36B	107.2(2)

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table 6
Torsional angles (deg)

Atom 1	Atom 2	Atom 3	Atom 4	Angle	Atom 1	Atom 2	Atom 3	Atom 4	Angle
C21	C1	C11	C12	−162.4	C22	C21	C26	H26B	−177.4
C21	C1	C11	C16	−39.7	H21	C21	C26	C25	58.1
C21	C1	C11	H11	80.0	H21	C21	C26	H26A	179.2
C31	C1	C11	C12	68.5	H21	C21	C26	H26B	−62.8
C31	C1	C11	C16	−168.7	C21	C22	C23	C24	−54.5
C31	C1	C11	H11	−49.1	C21	C22	C23	H23A	−175.6
H1	C1	C11	C12	−47.6	C21	C22	C23	H23B	66.4
H1	C1	C11	C16	75.1	H22A	C22	C23	C24	−176.7
H1	C1	C11	H11	−165.2	H22A	C22	C23	H23A	62.3
C11	C1	C21	C22	165.4	H22A	C22	C23	H23B	−55.8
C11	C1	C21	C26	−67.6	H22B	C22	C23	C24	66.3
C11	C1	C21	H21	49.1	H22B	C22	C23	H23A	−54.8
C31	C1	C21	C22	−63.4	H22B	C22	C23	H23B	−172.8
C31	C1	C21	C26	63.7	C22	C23	C24	C25	54.2
C31	C1	C21	H21	−179.6	C22	C23	C24	H24A	176.5
H1	C1	C21	C22	51.0	C22	C23	C24	H24B	−66.5
H1	C1	C21	C26	178.0	H23A	C23	C24	C25	175.7
H1	C1	C21	H21	−65.3	H23A	C23	C24	H24A	−62.0
C11	C1	C31	C32	24.8	H23A	C23	C24	H24B	55.0
C11	C1	C31	C36	−105.2	H23B	C23	C24	C25	−67.0
C11	C1	C31	H31	141.5	H23B	C23	C24	H24A	55.3
C21	C1	C31	C32	−105.5	H23B	C23	C24	H24B	172.3
C21	C1	C31	C36	124.5	C23	C24	C25	C26	−55.3
C21	C1	C31	H31	11.2	C23	C24	C25	H25A	66.2
H1	C1	C31	C32	140.3	C23	C24	C25	H25B	−176.6
H1	C1	C31	C36	10.3	H24A	C24	C25	C26	−178.0
H1	C1	C31	H31	−103.0	H24A	C24	C25	H25A	−56.5
C1	C11	C12	C13	−177.5	H24A	C24	C25	H25B	60.7
C1	C11	C12	H12A	61.1	H24B	C24	C25	C26	65.6
C1	C11	C12	H12B	−56.3	H24B	C24	C25	H25A	−172.9
C16	C11	C12	C13	56.2	H24B	C24	C25	H25B	−55.7
C16	C11	C12	H12A	−65.2	C24	C25	C26	C21	56.7
C16	C11	C12	H12B	177.4	C24	C25	C26	H26A	−64.4
H11	C11	C12	C13	−60.1	C24	C25	C26	H25B	178.0
H11	C11	C12	H12A	178.5	H25A	C25	C26	C21	−64.3
H11	C11	C12	H12B	61.1	H25A	C25	C26	H26A	174.6
C1	C11	C16	C15	−179.1	H25A	C25	C26	H26B	56.9
C1	C11	C16	H16A	−56.9	H25B	C25	C26	C21	177.3
C1	C11	C16	H16B	59.2	H25B	C25	C26	H26A	56.2
C12	C11	C16	C15	−55.0	H25B	C25	C26	H26B	−61.4
C12	C11	C16	H16A	67.2	C1	C31	C32	C33	173.3
C12	C11	C16	H16B	−176.7	C1	C31	C32	H32A	−66.6
H11	C11	C16	C15	61.3	C1	C31	C32	H32B	53.0
H11	C11	C16	H16A	−176.5	C36	C31	C32	C33	−55.5
H11	C11	C16	H16B	−60.4	C36	C31	C32	H32A	64.6
C11	C12	C13	C14	−57.3	C36	C31	C32	H32B	−175.8
C11	C12	C13	H13A	179.9	H31	C31	C32	C33	56.6
C11	C12	C13	H13B	63.4	H31	C31	C32	H32A	176.8
H12A	C12	C13	C14	64.6	H31	C31	C32	H32B	−63.7
H12A	C12	C13	H13A	−58.2	C1	C31	C36	C35	−170.7

Table 6 (continued)

Atom 1	Atom 2	Atom 3	Atom 4	Angle	Atom 1	Atom 2	Atom 3	Atom 4	Angle
H12A	C12	C13	H13B	−174.6	C1	C31	C36	H36A	−49.0
H12B	C12	C13	C14	−178.6	C1	C31	C36	H36B	68.3
H12B	C12	C13	H13A	58.6	C32	C31	C36	C35	56.1
H12B	C12	C13	H13B	−57.8	C32	C31	C36	H36A	177.7
C12	C13	C14	C15	54.8	C32	C31	C36	H36B	−64.9
C12	C13	C14	H14A	−66.6	H31	C31	C36	C35	−56.8
C12	C13	C14	H14B	175.0	H31	C31	C36	H36A	64.9
H13A	C13	C14	C15	176.7	H31	C31	C36	H36B	−177.7
H13A	C13	C14	H14A	55.4	C31	C32	C33	C34	55.4
H13A	C13	C14	H14B	−63.0	C31	C32	C33	H33A	−65.3
H13B	C13	C14	C15	−65.5	C31	C32	C33	H33B	177.3
H13B	C13	C14	H14A	173.2	H32A	C32	C33	C34	−65.2
H13B	C13	C14	H14B	54.8	H32A	C32	C33	H33A	174.1
C13	C14	C15	C16	−54.1	H32A	C32	C33	H33B	56.7
C13	C14	C15	H15A	−175.4	H32B	C32	C33	C34	175.7
C13	C14	C15	H15B	68.5	H32B	C32	C33	H33A	55.0
H14A	C14	C15	C16	66.7	H32B	C32	C33	H33B	−62.3
H14A	C14	C15	H15A	−54.6	C32	C33	C34	C35	−54.8
H14A	C14	C15	H15B	−170.7	C32	C33	C34	H34A	−175.7
H14B	C14	C15	C16	−174.1	C32	C33	C34	H34B	66.6
H14B	C14	C15	H15A	64.6	H33A	C33	C34	C35	65.9
H14B	C14	C15	H15B	−51.4	H33A	C33	C34	H34A	−55.0
C14	C15	C16	C11	55.8	H33A	C33	C34	H34B	−172.7
C14	C15	C16	H16A	−66.4	H33B	C33	C34	C35	−177.4
C14	C15	C16	H16B	177.2	H33B	C33	C34	H34A	61.8
H15A	C15	C16	C11	177.3	H33B	C33	C34	H34B	−55.9
H15A	C15	C16	H16A	55.1	C33	C34	C35	C36	54.2
H15A	C15	C16	H16B	−61.2	C33	C34	C35	H35A	175.1
H15B	C15	C16	C11	−67.2	C33	C34	C35	H35B	−67.0
H15B	C15	C16	H16A	170.7	H34A	C34	C35	C36	175.2
H15B	C15	C16	H16B	54.3	H34A	C34	C35	H35A	−64.0
C1	C21	C22	C23	−175.2	H34A	C34	C35	H35B	53.9
C1	C21	C22	H22A	−53.5	H34B	C34	C35	C36	−67.1
C1	C21	C22	H22B	64.4	H34B	C34	C35	H35A	53.8
C26	C21	C22	C23	55.7	H34B	C34	C35	H35B	171.6
C26	C21	C22	H22A	177.4	C34	C35	C36	C31	−55.3
C26	C21	C22	H22B	−64.7	C34	C35	C36	H36A	−177.2
H21	C21	C22	C23	−58.8	C34	C35	C36	H36B	65.8
H21	C21	C22	H22A	62.9	H35A	C35	C36	C31	−175.7
H21	C21	C22	H22B	−179.2	H35A	C35	C36	H36A	62.4
C1	C21	C26	C25	175.0	H35A	C35	C36	H36B	−54.6
C1	C21	C26	H26A	−63.9	H35B	C35	C36	C31	66.0
C1	C21	C26	H26B	54.2	H35B	C35	C36	H36A	−55.9
C22	C21	C26	C25	−56.5	H35B	C35	C36	H36B	−172.9
C22	C21	C26	H26A	64.6					

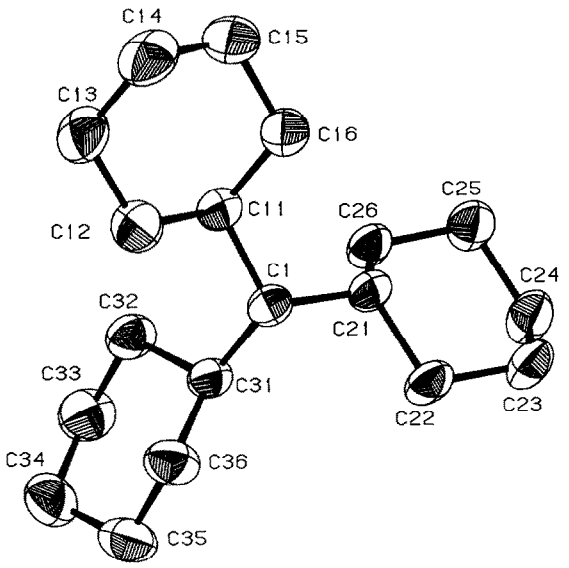

Fig. 3. A perspective view of tricyclohexylmethane.

and $-22 \leq l \leq 22$, with the 004 reflection to be the intensity standard, measured hourly.

After the usual Lp correction, from a total of 3750 reflections 3188 have $I > 3\alpha(I)$ with 421 having zero intensity. Analysis of the intensity of the standard reflection showed negligible decay. Inspection of the three principal zones showed the space group to be uniquely determined as $P2_12_12_1(19)$. The data were averaged, based upon the point group 222, which yielded 2890 unique data, 653 observed averaged data with agreement factors for the intensity of all reflections of 0.021. The relevant crystal data are given in Table 1.

Structure solution and refinement

Routine application of the Direct Methods program MULTAN produced 18 atoms of the molecule, and subsequent structure factor/Fourier calculation revealed the remaining carbon atom. Isotropic full-matrix refinement, with hydrogen atoms in fixed calculated position, $d = 1.0$ Å, converged at $R = 0.094$.

Anisotropic refinement of non-hydrogen atoms converged smoothly to $R = 0.049$, $wR = 0.064$

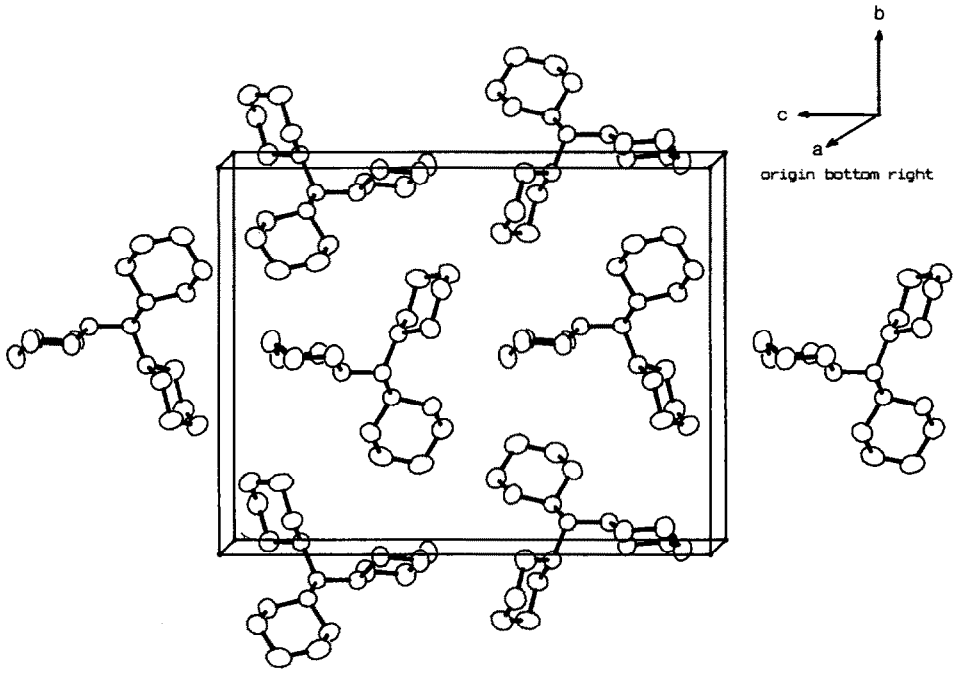

Fig. 4. The packing of tricyclohexylmethane in the unit cell.

and $S = 1.049$ using a weighting scheme:

$$w^{-1} = [\sigma(F^2) + (0.042)^2 + 4.2]$$

The highest residual peak in a difference Fourier map was 0.3 electrons.

A refinement based upon the enantiomorphic coordinates produced the same R, wR and S values, so that the chirality of the crystal could not be established.

Description of the structure

Tables 2 and 3 give the positional coordinates and thermal vibration parameters, and in Tables 4–6 are listed selected bond distances and angles. Figure 3 is an ORTEP diagram of the molecule. All bond and angle geometries are within normal limits for a hydrocarbon but it is worthy of note that the atomic volume per carbon atom is 22.2 Å^3 which is substantially greater than would be expected for a hydrocarbon. Thus the molecules pack quite loosely in the unit cell as may be seen from the packing diagram shown in Fig. 4, and as a result, all intermolecular contacts exceed 3.92 Å except for C13–C24 $(1/2 - x, \bar{y}, 1/2 + z)$ where the distance is 3.76 Å.

The angles around C1 show only a small increase from the tetrahedral value due to a slight strain caused by the bulky cyclohexane groups. It is interesting to note that these rings arrange themselves irregularly: the angles between their mean planes are:

(C11–C16)–(C21–C26) = 119°
(C11–C16)–(C31–C36) = 83°
(C21–C26)–(C31–C36) = 107°

From our data it can be seen that the crystal is chiral but, of course, the individual molecules are not chiral.

Acknowledgements

One of us (S.L.) wishes to thank S.E.R.C. and BP International for a CASE Studentship.

References

1 D.G. Gillies, S.J. Matthews and L.H. Sutcliffe, Magn. Reson. Chem., 29 (1991) 777.
2 D.G. Gillies, S.J. Matthews and L.H. Sutcliffe, Magn. Reson. Chem., 29 (1991) 823.
3 D.G. Gillies, S.J. Matthews and L.H. Sutcliffe, J. Mol. Struct., 54 (1992) 205.
4 K.-H. Hentschel, J. Synth. Lubr., 2 (1985) 143.
5 M. Muraki, Tribol. Int., 20 (1987) 347.
6 S. Luff, submitted for publication.