ture was investigated by NMR and could not be established unambiguously.

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Structure of Kostanecki's Triketone

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Abstract. 2,4-Dibenzoyl-1,3,5-triphenylcyclohexanol, $C_{38}H_{32}O_3$, $M_r = 536{\cdot}7$, monoclinic, $C2/c$, $a = 41{\cdot}651\ (13)$, $b = 6{\cdot}299\ (2)$, $c = 23{\cdot}638\ (7)$ Å, $\beta = 114{\cdot}17\ (2)°$, $V = 5658{\cdot}0$ Å^3, $Z = 8$, $D_x = 1{\cdot}260$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71069$ Å, $\mu = 0{\cdot}851$ cm^{-1}, $F(000) = 2272$, $T = 153$ K. Final $R = 0{\cdot}046$, $wR = 0{\cdot}055$ for 2185 unique observed reflections. All the bulky side groups are in equatorial positions and the single hydroxyl group forms an intramolecular hydrogen bond to the O atom of a neighbouring benzoyl group with an O···O distance of 2·717 (7) Å.

Introduction. The title compound is one of the satellite products of the Mannich reaction to synthesize α-aminomethyl, α'-methylene-1,5-diketones (Pavel & Tilichenko, 1973). It was identified as the low-temperature fusion form of Kostanecki's triketone (Kostanecki & Rossbach, 1896). The present X-ray diffractometric investigation was undertaken to determine the molecular structure, and to define its stereochemistry.

Experimental. Colourless crystals were obtained by slow evaporation of a solution in ethylmethyl ketone. A crystal approximately $0{\cdot}2 \times 0{\cdot}2 \times 0{\cdot}3$ mm was used for the measurements. D_m not measured. Mo $K\alpha$ radiation was used with a graphite-crystal monochromator on a Syntex $P2_1$ single-crystal diffractometer. The unit-cell dimensions were determined from the angular settings of 24 reflections ($15 \le \theta \le 40°$) at 153 K. The space group was determined to be $C2/c$ from systematic absences.

The intensity data reflections (half of the sphere up to $2\theta = 56°$, $0 \le h \le 51$, $0 \le k \le 9$, $-29 \le l \le 29$) were measured using the $\omega/2\theta$-scan technique and a variable scan rate with a maximum of 30° min^{-1}. The intensity of the primary beam was checked throughout the data collection by monitoring three reference reflections after every 200. Space-group symmetry-equivalent reflections were averaged, resulting in 3931 unique reflections of which 2184 were observed with $I > 6\sigma(I)$. Lorentz and polarization corrections were applied, and the data were reduced to $|F_o|$ values.

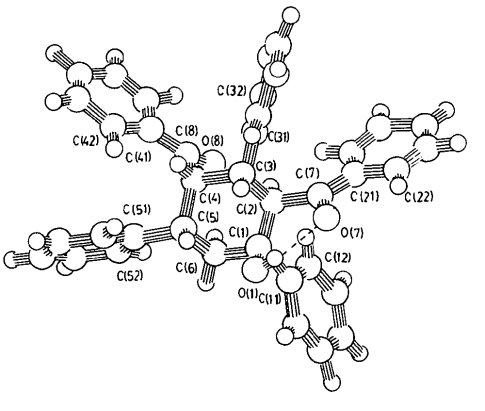

Fig. 1. Molecular structure and atom-numbering scheme.

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Table 1. *Atom coordinates* ($\times 10^5$; $\times 10^4$ *for* H *atoms*) *and isotropic temperature factors* (Å^2) *with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ac(\cos\beta)B(1,3)].$$

	x	y	z	B_{eq}/B_{iso}
O(1)	13526 (8)	81042 (43)	13086 (12)	3·7 (1)
C(1)	13155 (11)	59018 (66)	14252 (18)	3·0 (1)
C(2)	10412 (10)	55965 (66)	17100 (17)	2·8 (1)
C(3)	11651 (10)	68621 (62)	23337 (18)	2·6 (1)
C(4)	15383 (10)	62336 (65)	27962 (17)	2·7 (1)
C(5)	17968 (10)	65563 (62)	24822 (17)	2·6 (1)
C(6)	16769 (10)	52134 (65)	18911 (17)	2·7 (1)
C(7)	6793 (11)	63610 (70)	12685 (18)	3·4 (2)
O(7)	6528 (8)	81530 (50)	10392 (13)	4·6 (1)
C(8)	15640 (10)	40148 (67)	30620 (18)	2·7 (1)
O(8)	14460 (7)	24720 (43)	27250 (12)	3·33 (9)
C(11)	12183 (11)	46694 (67)	8197 (18)	3·0 (1)
C(12)	10704 (17)	26899 (78)	7271 (19)	6·2 (2)
C(13)	10055 (18)	15467 (77)	1916 (21)	6·9 (2)
C(14)	10919 (12)	23866 (83)	−2668 (19)	4·7 (2)
C(15)	12375 (12)	43244 (89)	−1824 (20)	4·9 (2)
C(16)	13031 (12)	54913 (83)	3564 (20)	4·3 (2)
C(21)	3640 (11)	50246 (73)	11120 (20)	3·9 (2)
C(22)	376 (12)	58178 (89)	7179 (19)	4·7 (2)
C(23)	−2620 (12)	46595 (112)	5630 (21)	6·6 (2)
C(24)	−2442 (12)	26576 (105)	7973 (25)	6·8 (2)
C(25)	727 (14)	18122 (92)	11902 (35)	8·6 (3)
C(26)	3751 (12)	29959 (85)	13406 (30)	6·9 (2)
C(31)	8967 (10)	69062 (62)	26159 (17)	2·6 (1)
C(32)	7818 (14)	51323 (72)	28200 (27)	5·6 (2)
C(33)	5367 (13)	52795 (81)	30769 (26)	5·5 (2)
C(34)	4009 (12)	71821 (77)	31323 (20)	4·0 (2)
C(35)	5156 (15)	89166 (79)	29451 (25)	5·7 (2)
C(36)	7615 (13)	87985 (70)	26907 (22)	4·2 (2)
C(41)	17435 (10)	36702 (66)	37495 (18)	2·7 (1)
C(42)	19599 (11)	51585 (74)	41575 (18)	3·5 (2)
C(43)	21304 (11)	46790 (83)	47919 (19)	4·2 (2)
C(44)	20733 (12)	27646 (83)	50097 (19)	4·4 (2)
C(45)	18544 (13)	12974 (76)	46087 (21)	4·7 (2)
C(46)	16881 (11)	17383 (69)	39787 (19)	3·6 (2)
C(51)	21771 (10)	62682 (65)	29190 (17)	2·5 (1)
C(52)	23636 (11)	44356 (68)	29528 (18)	3·2 (1)
C(53)	27179 (12)	43030 (83)	33556 (20)	4·3 (2)
C(54)	28913 (11)	59893 (88)	37262 (19)	4·5 (2)
C(55)	27048 (11)	78175 (80)	36952 (18)	4·1 (2)
C(56)	23530 (11)	79871 (70)	32984 (18)	3·3 (1)
H(2)	1041 (9)	4054 (60)	1818 (15)	4·0
H(3)	1181 (9)	8358 (55)	2209 (16)	4·0
H(4)	1588 (9)	7248 (58)	3130 (15)	4·0
H(5)	1771 (9)	8107 (56)	2360 (16)	4·0
H(6·1)	1846 (9)	5377 (57)	1737 (16)	4·0
H(6·2)	1667 (9)	3637 (58)	1975 (16)	4·0
H(12)	1016 (9)	2178 (58)	1008 (15)	4·0
H(13)	914 (9)	205 (58)	181 (16)	4·0
H(14)	1043 (9)	1506 (57)	−639 (16)	4·0
H(15)	1268 (9)	5161 (58)	−537 (16)	4·0
H(16)	1412 (9)	7005 (58)	390 (16)	4·0
H(22)	38 (9)	7211 (59)	554 (16)	4·0
H(23)	−506 (9)	5160 (58)	270 (15)	4·0
H(24)	−449 (9)	1874 (57)	697 (15)	4·0
H(25)	70 (9)	400 (58)	1364 (15)	4·0
H(26)	594 (9)	2404 (57)	1613 (15)	4·0
H(32)	864 (9)	3843 (58)	2766 (16)	4·0
H(33)	459 (9)	3933 (58)	3216 (16)	4·0
H(34)	213 (9)	7075 (57)	3298 (15)	4·0
H(35)	429 (9)	10206 (56)	2976 (15)	4·0
H(36)	857 (9)	10152 (56)	2586 (16)	4·0
H(42)	1997 (9)	6592 (57)	3994 (16)	4·0
H(43)	2297 (9)	5724 (56)	5059 (15)	4·0
H(44)	2194 (9)	2355 (59)	5447 (15)	4·0
H(45)	1815 (9)	−143 (58)	4735 (15)	4·0
H(46)	1514 (9)	680 (58)	3702 (16)	4·0
H(52)	2242 (9)	3272 (55)	2687 (16)	4·0
H(53)	2848 (9)	3103 (58)	3371 (16)	4·0
H(54)	3157 (9)	6011 (59)	4037 (15)	4·0
H(55)	2830 (9)	9108 (59)	3973 (16)	4·0
H(56)	2208 (9)	9255 (59)	3252 (15)	4·0
H(O)	1181 (9)	8615 (57)	1244 (16)	4·0

Table 2. *Bond lengths* (Å) *and selected bond angles and torsion angles* (°)

O(1)—C(1)	1·435 (5)	C(21)—C(26)	1·381 (7)
O(1)—H(O)	0·74 (4)	C(22)—C(23)	1·381 (7)
C(1)—C(2)	1·558 (6)	C(23)—C(24)	1·367 (9)
C(1)—C(6)	1·520 (6)	C(24)—C(25)	1·373 (9)
C(1)—C(11)	1·530 (6)	C(25)—C(26)	1·379 (9)
C(2)—C(3)	1·567 (6)	C(31)—C(32)	1·378 (7)
C(2)—C(7)	1·518 (6)	C(31)—C(36)	1·360 (6)
C(3)—C(4)	1·542 (6)	C(32)—C(33)	1·389 (9)
C(3)—C(31)	1·518 (6)	C(33)—C(34)	1·354 (7)
C(4)—C(5)	1·551 (6)	C(34)—C(35)	1·338 (7)
C(4)—C(8)	1·518 (6)	C(35)—C(36)	1·386 (9)
C(5)—C(6)	1·532 (5)	C(41)—C(42)	1·382 (6)
C(5)—C(51)	1·506 (6)	C(41)—C(46)	1·389 (6)
C(7)—O(7)	1·237 (5)	C(42)—C(43)	1·404 (6)
C(7)—C(21)	1·474 (7)	C(43)—C(44)	1·370 (7)
C(8)—O(8)	1·225 (5)	C(44)—C(45)	1·370 (7)
C(8)—C(41)	1·501 (5)	C(45)—C(46)	1·389 (6)
C(11)—C(12)	1·368 (7)	C(51)—C(52)	1·375 (6)
C(11)—C(16)	1·382 (6)	C(51)—C(56)	1·405 (6)
C(12)—C(13)	1·383 (7)	C(52)—C(53)	1·393 (7)
C(13)—C(14)	1·379 (7)	C(53)—C(54)	1·378 (7)
C(14)—C(15)	1·341 (8)	C(54)—C(55)	1·374 (7)
C(15)—C(16)	1·397 (7)	C(55)—C(56)	1·383 (7)
C(21)—C(22)	1·389 (7)		

C(1)—O(1)—H(O)	107· (3)	C(3)—C(4)—C(5)	108·9 (3)
O(1)—C(1)—C(2)	111·0 (3)	C(3)—C(4)—C(8)	114·2 (3)
O(1)—C(1)—C(6)	105·2 (5)	C(5)—C(4)—C(8)	111·9 (3)
O(1)—C(1)—C(11)	108·6 (3)	C(4)—C(5)—C(6)	109·5 (3)
C(2)—C(1)—C(6)	109·6 (3)	C(4)—C(5)—C(51)	113·4 (3)
C(2)—C(1)—C(11)	112·4 (3)	C(6)—C(5)—C(51)	114·7 (3)
C(6)—C(1)—C(11)	109·7 (3)	C(1)—C(6)—C(5)	111·1 (3)
C(1)—C(2)—C(3)	109·1 (3)	C(2)—C(7)—O(7)	118·4 (4)
C(1)—C(2)—C(7)	111·2 (3)	C(2)—C(7)—C(21)	121·6 (4)
C(3)—C(2)—C(7)	109·7 (3)	O(7)—C(7)—C(21)	120·0 (4)
C(2)—C(3)—C(4)	112·6 (3)	C(4)—C(8)—O(8)	121·3 (4)
C(2)—C(3)—C(31)	113·9 (3)	C(4)—C(8)—C(41)	120·1 (4)
C(4)—C(3)—C(31)	113·8 (3)	O(8)—C(8)—C(41)	118·6 (4)

C(1)—C(2)—C(3)—C(4)	−55·6 (4)	C(3)—C(2)—C(7)—O(7)	70·5 (4)
C(2)—C(3)—C(4)—C(5)	56·5 (4)	C(2)—C(7)—C(21)—C(22)	178·4 (6)
C(3)—C(4)—C(5)—C(6)	−58·2 (4)	O(7)—C(7)—C(21)—C(22)	−1·5 (4)
C(4)—C(5)—C(6)—C(1)	62·1 (4)	C(2)—C(3)—C(31)—C(32)	63·4 (5)
C(5)—C(6)—C(1)—C(2)	−61·1 (4)	C(4)—C(3)—C(31)—C(32)	−67·5 (5)
C(6)—C(1)—C(2)—C(3)	56·3 (4)	C(3)—C(4)—C(8)—O(8)	51·1 (4)
O(1)—C(1)—C(11)—C(12)	−161·3 (6)	C(5)—C(4)—C(8)—O(8)	−73·2 (4)
C(2)—C(1)—C(11)—C(12)	−38·0 (5)	C(4)—C(8)—C(41)—C(42)	−15·4 (4)
C(6)—C(1)—C(11)—C(12)	84·3 (5)	O(8)—C(8)—C(41)—C(42)	163·3 (6)
C(2)—C(1)—O(1)—H(O)	−30· (4)	C(4)—C(5)—C(51)—C(52)	99·7 (5)
C(1)—C(2)—C(7)—O(7)	−50·3 (4)	C(6)—C(5)—C(51)—C(52)	−27·0 (4)

Table 3. *Angles* (°) *between normals to mean planes*

Planes	Angles	
*P*1: C(1)—C(6)—C(5)—C(4)—C(3)—C(2)	*P*1–*P*2	66·4 (2)
*P*2: C(11)—C(12)—C(13)—C(14)—C(15)—C(16)	*P*1–*P*3	56·0 (2)
*P*3: C(51)—C(52)—C(53)—C(54)—C(55)—C(56)	*P*1–*P*4	103·9 (2)
*P*4: C(41)—C(42)—C(43)—C(44)—C(45)—C(46)	*P*1–*P*5	87·0 (2)
*P*5: C(31)—C(32)—C(33)—C(34)—C(35)—C(36)	*P*1–*P*6	97·8 (2)
*P*6: C(21)—C(22)—C(23)—C(24)—C(25)—C(26)	*P*4–*P*7	16·0 (2)
*P*7: C(4)—C(8)—O(8)—C(41)	*P*6–*P*8	1·5 (2)
*P*8: C(7)—O(7)—C(2)—C(21)		

The structure was determined using *MULTAN* and refined by the Gauss–Seidel block-matrix least-squares method using *BLOCK* with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for all H atoms, which were placed initially in fixed idealized positions, 1·1 Å from the atom to which they are bonded; all routines are from the *XTL/XTLE* software package (Syntex, 1976), calculations were performed on an Eclipse S/200 computer. Final $R = 0{\cdot}046$, $wR = 0{\cdot}055$, $w = 1/\sigma^2(F)$. $(\Delta/\sigma)_{max} = 0{\cdot}02$. $\Delta\rho < 0{\cdot}23$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The plot was made with *PLUTO* (Motherwell & Clegg, 1978), adapted to a PDP-11 compatible 128 kW

SM-4 computer and using the X-Y recorder as a plotter.

Discussion. Table 1 presents the atomic coordinates and isotropic temperature factors.* Table 2 lists bond lengths and angles. Fig. 1 shows a plot of the molecule. All the bulky side substituents are situated in equatorial positions. Most are nearly perpendicular to the mean plane of the cyclohexane ring (Table 3).

The aromatic rings are essentially planar but some atoms still deviate significantly from planarity, the maximal deviations being 0·011 (5) C(42), 0·009 (3) C(44), 0·09 (4) C(51), 0·09 C(54), 0·08 (4) C(41), 0·07 (5) Å C(43). Bond lengths and angles in the molecule under investigation are in good agreement with standard values for organic compounds.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52697 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *N'*,*N'*-Dibenzylbenzohydrazide

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Abstract. $C_{21}H_{20}N_2O$, $M_r = 316{\cdot}4$, monoclinic, $P2_1/c$, $a = 9{\cdot}985$ (2), $b = 17{\cdot}070$ (3), $c = 10{\cdot}119$ (2) Å, $\beta = 95{\cdot}99$ (1)°, $V = 1715{\cdot}3$ Å^3, $Z = 4$, $D_x = 1{\cdot}225$ g cm^{-3}, Mo $K\alpha$ ($\lambda = 0{\cdot}71069$ Å), $\mu = 0{\cdot}82$ cm^{-1}, $F(000) = 672$, $T = 295$ K. Final $R = 0{\cdot}043$, $wR = 0{\cdot}042$ for 1139 reflections with $I > 3\sigma(I)$. There is one hydrogen bond in the structure with N—H···O = 2·05 (4) and N···O = 2·923 (5) Å.

Experimental. Colorless needles from dilute ethanol, CAD-4 diffractometer, graphite monochromator, 0·5 × 0·3 × 0·25 mm crystal, cell parameters from 25 reflections automatically centered in the range $8{\cdot}3 < \theta < 15{\cdot}6°$, θ–2θ scan at variable θ speed of 1·03 to 8·24° min^{-1}; 5 standard reflections measured every 2 h, each scan recorded in 96 steps over the θ range of $1{\cdot}5 \times (1{\cdot}2 + 0{\cdot}35\tan\theta)°$, $\theta_{max} = 25°$, 3129 unique reflections measured, 2462 with $F_o > 0{\cdot}0$, 1647 reflections with $I > \sigma(I)$, $R_{int} = 0{\cdot}008$ for 134 reflections; index range for h, k, $l = -10$ to 11; 0 to 20, 0 to 12. All crystallographic calculations performed with the *TEXSAN* program system (Molecular Structure Corporation, 1985) on DEC MicroVAX II computer; structure solved by *MITHRIL* (Gilmore, 1983) incorporated in *TEXSAN*. Full-matrix least-squares refinement; atomic scattering factors from *International Tables for X-ray Crystallography* (1974), anisotropic temperature factors for C, N and O, individual isotropic terms for H; $\sum w(F_o - F_c)^2$ minimized, $w = 1/\sigma^2(F_o)$, reflections with $I < \sigma(I)$ excluded from refinement; maximum Δ/σ of 0·66 in the final least-squares cycle; $\Delta/\rho_{min,max} = -0{\cdot}31$, 0·28 e Å^{-3}. Final R, wR and S are 0·078, 0·053 and 1·19 for 1647 reflections with $I > \sigma(I)$. Atomic

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