$V = 1252.8$ (3) Å^3
$Z = 4$
$D_x = 1.375$ Mg m^{-3}

0.30 × 0.30 × 0.20 mm
Light yellow

Data collection

Stoe Stadi-4 four-circle diffractometer
ω scans
Absorption correction: none
5956 measured reflections
2879 independent reflections
2014 observed reflections [$I > 2.0\sigma(I)$]

$R_{\rm int} = 0.012$
$\theta_{\rm max} = 27.5°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = 0 \rightarrow 32$
3 standard reflections
frequency: 60 min
intensity variation: 3.0%

Refinement

Refinement on F
$R = 0.0361$
$wR = 0.0577$
$S = 0.970$
2014 reflections
224 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.003F^2]$

$(\Delta/\sigma)_{\rm max} = 0.003$
$\Delta\rho_{\rm max} = 0.18$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.19$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *CRYSRULER* (Rizzoli, Sangermano, Calestani & Andreetti, 1989)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.4152 (2)	0.1911 (2)	0.0450 (1)	0.0327 (3)
C11	0.2481 (2)	0.1100 (2)	0.0676 (1)	0.0405 (5)
C12	0.1434 (2)	0.2555 (3)	0.0977 (1)	0.0473 (5)
O13	0.0892 (2)	0.4119 (2)	0.0656 (1)	0.0563 (4)
C2	0.5749 (2)	0.2478 (2)	0.0748 (1)	0.0336 (4)
C21	0.6021 (2)	0.2243 (2)	0.1328 (1)	0.0383 (4)
C22	0.7256 (2)	0.3236 (2)	0.1644 (1)	0.0391 (4)
C221	0.7616 (2)	0.3009 (2)	0.2229 (1)	0.0398 (4)
C222	0.9276 (3)	0.3713 (2)	0.2490 (1)	0.0515 (6)
C223	0.9714 (3)	0.3451 (3)	0.3036 (1)	0.0618 (7)
C224	0.8482 (3)	0.2507 (3)	0.3334 (1)	0.0602 (6)
C225	0.6807 (3)	0.1840 (3)	0.3089 (1)	0.0631 (7)
C226	0.6366 (3)	0.2078 (3)	0.2541 (1)	0.0557 (6)
N3	0.7010 (2)	0.3226 (2)	0.0438 (1)	0.0375 (3)
C4	0.6206 (2)	0.3132 (2)	−0.0072 (1)	0.0378 (4)
C5	0.4451 (2)	0.2342 (2)	−0.0075 (1)	0.0342 (4)
N50	0.3158 (2)	0.2043 (2)	−0.0534 (1)	0.0438 (4)
O501	0.1575 (2)	0.1436 (3)	−0.0486 (1)	0.0761 (6)
O502	0.3727 (2)	0.2415 (2)	−0.0972 (1)	0.0678 (5)

Table 2. *Selected geometric parameters* (Å, °)

N1—C11	1.473 (2)	C221—C226	1.396 (3)
N1—C2	1.360 (2)	C222—C223	1.382 (3)
N1—C5	1.379 (2)	C223—C224	1.371 (3)
C11—C12	1.511 (2)	C224—C225	1.371 (3)
C12—O13	1.405 (2)	C225—C226	1.385 (3)
O13—H13	0.86 (2)	N3—C4	1.349 (2)
C2—C21	1.452 (3)	C4—C5	1.365 (2)
C2—N3	1.342 (2)	C5—N50	1.418 (2)
C21—C22	1.329 (2)	N50—O501	1.218 (2)
C22—C221	1.468 (3)	N50—O502	1.226 (2)
C221—C222	1.387 (2)		
C2—N1—C5	105.2 (1)	C221—C222—C223	121.4 (2)
C11—N1—C5	130.2 (1)	C222—C223—C224	120.1 (2)
C11—N1—C2	124.5 (1)	C223—C224—C225	119.7 (2)
N1—C11—C12	111.6 (1)	C224—C225—C226	120.6 (2)
C11—C12—O13	112.4 (2)	C221—C226—C225	120.6 (2)
C12—O13—H13	107 (2)	C2—N3—C4	106.0 (1)
N1—C2—N3	111.6 (1)	N3—C4—C5	109.6 (2)
N1—C2—C21	122.8 (1)	N1—C5—C4	107.6 (2)
C21—C2—N3	125.6 (2)	C4—C5—N50	126.2 (2)
C2—C21—C22	123.5 (2)	N1—C5—N50	126.2 (1)
C21—C22—C221	125.4 (2)	C5—N50—O502	116.5 (2)
C22—C221—C226	122.8 (2)	C5—N50—O501	120.6 (2)
C22—C221—C222	119.5 (2)	O501—N50—O502	122.8 (2)
C222—C221—C226	117.6 (2)		

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Molecular graphics: *PLUTON*93 (Spek, 1993). Software used to prepare material for publication: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1069). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 922–924

Isopropyl Hydrogen 9,10-Dihydro-9,10-ethenoanthracene-11,12-dicarboxylate

Phani Raj Pokkuluri, John R. Scheffer
and James Trotter

Department of Chemistry, University of British Columbia, Vancouver, BC, Canada V6T 1Z1

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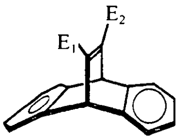
Abstract

The title compound, $C_{21}H_{18}O_4$, exists in the crystal as hydrogen-bonded dimers, O—H···O = 2.669 (3) Å. The molecule has normal dimensions. The C=O bond

of the CO_2H group is conjugated with the C11=C12 double bond [C=C—C=O torsion angle −164.4 (3)°, $\cos^2$(angle) 0.93], but the C=O bond of the $CO_2C_3H_7$ group is not conjugated [C=C—C=O 65.6 (3)°, $\cos^2$(angle) 0.17].

Comment

IR, UV and photochemical studies of solutions of the title compound (I) indicate an equilibrium between dimeric and intramolecularly hydrogen-bonded structures (Garcia-Garibay, Scheffer & Watson, 1992). Solid-state data show the dimer as the only species, and this has now been confirmed by an X-ray crystal structure study. The molecules are linked into dimers by pairs of hydrogen bonds [O3—H···O4^i: O···O = 2.669 (3), O—H = 1.21 (6), H···O = 1.48 (6) Å, O—H···O = 165°, symmetry code (i) $2-x$, $1-y$, $1-z$].



$E_1 = CO_2{}^iPr$
$E_2 = CO_2H$

(I)

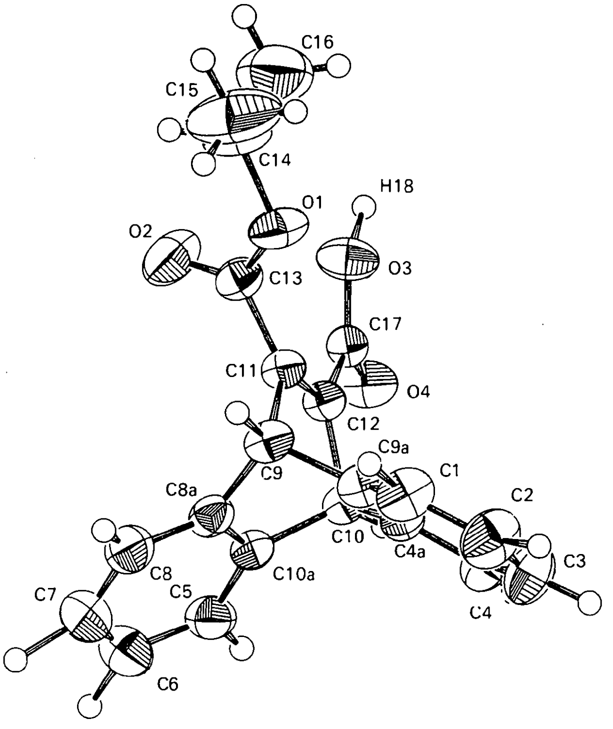


Fig. 1. View of the molecule showing 50% probability displacement ellipsoids for the non-H atoms.

Experimental

The title compound was prepared according to the method described by Garcia-Garibay, Scheffer & Watson (1992).

Crystal data

$C_{21}H_{18}O_4$
$M_r = 334.37$
Orthorhombic
Pbca
$a = 16.786$ (1) Å
$b = 15.099$ (1) Å
$c = 13.291$ (2) Å
$V = 3369$ (1) Å^3
$Z = 8$
$D_x = 1.32$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 24 reflections
$\theta = 23$–$38°$
$\mu = 0.70$ mm^{-1}
$T = 294$ K
Prism
0.25 × 0.20 × 0.15 mm
Colourless

Data collection

Rigaku AFC-6 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans
$T_{min} = 0.97$, $T_{max} = 1.00$
3578 measured reflections
3578 independent reflections
2067 observed reflections [$I > 3\sigma(I)$]

$\theta_{max} = 77.7°$
$h = 0 \rightarrow 21$
$k = 0 \rightarrow 19$
$l = -17 \rightarrow 0$
3 standard reflections monitored every 150 reflections intensity variation: 0.4%

Refinement

Refinement on F
$R = 0.055$
$wR = 0.077$
$S = 2.05$
2067 reflections
279 parameters (including 48 H atom parameters)
12 H atoms refined isotropically, six methyl H atoms fixed
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{max} = 0.01$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: *TEXSAN* (Molecular Structure Corporation, 1990)
Extinction coefficient: 1.4 (2) × 10^{-6}
Atomic scattering factors from *TEXSAN*

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.8906 (2)	0.6614 (1)	0.8143 (2)	0.058
O2	0.8320 (2)	0.6892 (2)	0.6664 (2)	0.077
O3	0.9614 (1)	0.5540 (2)	0.6062 (2)	0.058
O4	0.9159 (1)	0.4379 (2)	0.5212 (2)	0.056
C1	0.7857 (2)	0.4116 (3)	0.9527 (3)	0.059
C2	0.8092 (3)	0.3273 (3)	0.9841 (3)	0.074
C3	0.8281 (3)	0.2629 (3)	0.9148 (3)	0.069
C4	0.8241 (2)	0.2813 (2)	0.8123 (3)	0.054
C4a	0.8034 (2)	0.3649 (2)	0.7810 (2)	0.039
C5	0.6557 (2)	0.3975 (3)	0.5928 (3)	0.055
C6	0.5791 (3)	0.4336 (3)	0.5925 (4)	0.077
C7	0.5583 (3)	0.4979 (3)	0.6635 (4)	0.078
C8	0.6137 (2)	0.5265 (3)	0.7323 (4)	0.060
C8a	0.6887 (2)	0.4923 (2)	0.7320 (2)	0.044
C9	0.7573 (2)	0.5165 (2)	0.8029 (2)	0.043
C9a	0.7831 (2)	0.4298 (2)	0.8509 (2)	0.044
C10	0.7968 (2)	0.3973 (2)	0.6736 (2)	0.037
C10a	0.7100 (2)	0.4278 (2)	0.6618 (2)	0.041

C11	0.8255 (2)	0.5446 (2)	0.7326 (2)	0.039
C12	0.8468 (2)	0.4821 (2)	0.6675 (2)	0.038
C13	0.8506 (2)	0.6388 (2)	0.7325 (2)	0.044
C14	0.9174 (3)	0.7543 (3)	0.8191 (3)	0.068
C15	0.9078 (4)	0.7881 (4)	0.9175 (5)	0.125
C16	0.9912 (3)	0.7626 (4)	0.7684 (5)	0.118
C17	0.9111 (2)	0.4910 (2)	0.5927 (2)	0.038

Table 2. *Selected geometric parameters* (Å, °)

C1—C2	1.396 (6)	C9—C9a	1.519 (5)
C1—C9a	1.381 (5)	C9—C11	1.537 (4)
C2—C3	1.376 (6)	C10—C10a	1.536 (4)
C3—C4	1.392 (5)	C10—C12	1.533 (4)
C4—C4a	1.374 (5)	C11—C12	1.329 (4)
C4a—C9a	1.393 (4)	C11—C13	1.483 (4)
C4a—C10	1.513 (4)	C12—C17	1.474 (4)
C5—C6	1.396 (6)	C14—C15	1.413 (8)
C5—C10a	1.371 (5)	C14—C16	1.416 (7)
C6—C7	1.398 (7)	C13—O1	1.323 (4)
C7—C8	1.374 (7)	C13—O2	1.203 (4)
C8—C8a	1.361 (5)	C14—O1	1.474 (3)
C8a—C9	1.532 (5)	C17—O3	1.285 (3)
C8a—C10a	1.395 (4)	C17—O4	1.246 (3)
C2—C1—C9a	118.9 (4)	C4a—C10—C12	106.3 (2)
C1—C2—C3	120.6 (4)	C10a—C10—C12	105.3 (2)
C2—C3—C4	120.2 (4)	C5—C10a—C8a	120.6 (3)
C3—C4—C4a	119.5 (4)	C5—C10a—C10	126.8 (3)
C4—C4a—C9a	120.4 (3)	C8a—C10a—C10	112.6 (3)
C4—C4a—C10	127.0 (3)	C9—C11—C12	113.6 (3)
C9a—C4a—C10	112.6 (3)	C9—C11—C13	118.5 (2)
C6—C5—C10a	119.0 (4)	C12—C11—C13	127.1 (3)
C5—C6—C7	119.9 (4)	C10—C12—C11	114.3 (3)
C6—C7—C8	119.9 (4)	C10—C12—C17	120.9 (2)
C7—C8—C8a	120.3 (4)	C11—C12—C17	124.9 (3)
C8—C8a—C9	127.1 (3)	C11—C13—O1	113.0 (3)
C8—C8a—C10a	120.3 (3)	C11—C13—O2	122.2 (3)
C9—C8a—C10a	112.7 (3)	O1—C13—O2	124.7 (3)
C8a—C9—C9a	105.5 (2)	O1—C14—C15	110.4 (4)
C8a—C9—C11	104.6 (2)	O1—C14—C16	109.3 (4)
C9a—C9—C11	106.3 (2)	C15—C14—C16	120.6 (5)
C1—C9a—C4a	120.4 (3)	C12—C17—O3	117.1 (3)
C1—C9a—C9	126.3 (3)	C12—C17—O4	120.2 (3)
C4a—C9a—C9	113.3 (3)	O3—C17—O4	122.7 (3)
C4a—C10—C10a	105.2 (2)	C13—O1—C14	115.8 (3)

The structure was determined by direct methods. There is minor disorder in the isopropyl group; this was modelled by a split C16 site [15% occupancy (not refined) for C16′ at 1.014 (1), 0.725 (1), 0.897(2), isotropic], but the geometry of the group as a whole is rather unsatisfactory. Data collection, cell refinement, data reduction, structure solution, structure refinement and molecular graphics: *TEXSAN* (Molecular Structure Corporation, 1990).

We thank the Natural Sciences and Engineering Research Council of Canada for financial support.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry and stereo molecular and packing diagrams have been deposited with the IUCr (Reference: BK1006). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Molecules Isoelectronic with Triphenylmethanol: Diphenyl(4-pyridyl)methanol and Triphenylmethylamine

CHRISTOPHER GLIDEWELL

School of Chemistry, University of St Andrews,
St Andrews, Fife KY16 9ST, Scotland

GEORGE FERGUSON

Department of Chemistry and Biochemistry,
University of Guelph, Guelph, Ontario,
Canada N1G 2W1

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Abstract

Diphenyl(4-pyridyl)methanol, $C_{18}H_{15}NO$, forms chains in the solid state in which the molecules are linked by O—H···N hydrogen bonds; the O atoms, although potential hydrogen-bond acceptors, are not utilized as such in the intermolecular hydrogen bonding. Triphenylmethylamine, $C_{19}H_{17}N$, crystallizes as isolated molecules with perfectly ordered pyramidal NH_2 groups; there is no intermolecular hydrogen bonding despite the availability of potential hydrogen-bond donors and acceptors.

Comment

Triphenylmethanol crystallizes as almost perfectly tetrahedral tetramers, with the four hydroxyl H atoms presumably disordered over the six O···O edges (Ferguson, Gallagher, Glidewell, Low & Scrimgeour, 1992). Despite the exact match of the number of hydrogen-bond donors and hydrogen-bond acceptors in this tetrameric system, a structure is adopted which cannot have one O—H···O hydrogen bond per O···O atom pair. With only modest changes in the steric demands at the unique central C atom, while keeping the number of hydrogen-bond donors and acceptors unchanged, the patterns of hydrogen bonding can be altered drastically. Thus in 1,1,2-triphenylethanol there is O—H···π(arene), rather than O—H···O, intermolecular hydrogen bonding (Ferguson, Gallagher, Glidewell & Zakaria, 1994), while the crystal structures of 1,2,3-triphenyl-2-propanol and tribenzylmethanol contain no hydrogen bonds at all (Ferguson, Gallagher, Glidewell, Liles & Zakaria, 1993).

Etter has formulated some general principles for hydrogen-bond formation in systems where the numbers of hydrogen-bond donors and acceptors are identical (Etter, 1990; Etter, McDonald & Bernstein, 1990; Etter & Reutzel, 1991). Where there is an excess of hydrogen-bond donors over acceptors, the numerical mis-