

1,7-Diethyl-4,10-diisopropyltetracene

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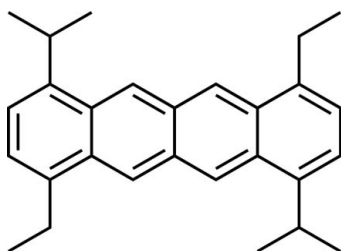
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Key indicators: single-crystal X-ray study; $T = 203$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.081; wR factor = 0.282; data-to-parameter ratio = 18.6.

The molecule of the title compound, $\text{C}_{28}\text{H}_{32}$, is located on a crystallographic inversion center. The ethyl groups are essentially coplanar with the tetracene ring, making a torsion angle of -0.4 (4)°. The isopropyl groups adopt an asymmetric conformation with their terminal methyl groups positioned on opposite sides of the tetracene plane [the $\text{Me}-\text{C}-\text{C}$ torsion angles are -22.5 (4) and 100.9 (3)°]. In the crystal, the molecules adopt an arrangement without significant $\pi-\pi$ interactions along the stacking direction (y axis).

Related literature

For applications of tetracene derivatives, see: Anthony (2008). For crystallochromy, see: Klebe *et al.* (1989). For the synthesis, see: Kitamura *et al.* (2011). For structures of related alkyl-substituted tetracene derivatives, see: Kitamura, Abe *et al.* (2010); Kitamura, Tsukuda *et al.* (2010).



Experimental

Crystal data

$\text{C}_{28}\text{H}_{32}$	$V = 1061.0$ (7) Å ³
$M_r = 368.54$	$Z = 2$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 12.901$ (4) Å	$\mu = 0.06$ mm ⁻¹
$b = 5.057$ (2) Å	$T = 203$ K
$c = 16.962$ (6) Å	$0.25 \times 0.13 \times 0.1$ mm
$\beta = 106.513$ (9)°	

Data collection

Rigaku R-AXIS RAPID IP diffractometer	2423 independent reflections
9469 measured reflections	1318 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.083$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.081$	130 parameters
$wR(F^2) = 0.282$	H-atom parameters constrained
$S = 1.10$	$\Delta\rho_{\text{max}} = 0.26$ e Å ⁻³
2423 reflections	$\Delta\rho_{\text{min}} = -0.42$ e Å ⁻³

Data collection: *RAPID-AUTO* (Rigaku, 1999); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LD2024).

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supplementary materials

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1,7-Diethyl-4,10-diisopropyltetracene

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Comment

Tetracene is a promising organic semiconducting molecule for OFETs, OLEDs, and solar cells (Anthony, 2008). In addition, we have recently found that alkyl-substituted tetracenes possess interesting chromophore properties. Depending on the length, shape, and the number of alkyl side chains, the solid-state color of the tetracenes varies through yellow, orange and red (Kitamura, Abe *et al.*, 2010; Kitamura, Tsukuda *et al.*, 2010). The difference in color can be attributed to crystallochromy (Klebe, *et al.*, 1989), *i.e.* to a color change caused by different molecular interactions based on different molecular arrangements induced by the substituents. Very recently, we have prepared *anti/syn*-regioisomeric mixtures of alkyl-substituted tetracenes and reported that the solid-state color of the mixtures changed before and after recrystallization from Et₂O (Kitamura, *et al.*, 2011). To further investigate the effects of alkyl side chains on the solid-state colorations, we have synthesized an *anti/syn* mixture of ethyl/isopropyl-substituted tetracene (*anti* isomer – the title compound; *syn* isomer – 1,10-diethyl-4,7-diisopropyltetracene). The molecular arrangement in the crystal of the *anti* isomer is shown on Fig. 2.

Experimental

The *anti/syn* ethyl/isopropyl-substituted tetracene mixture was prepared as an orange solid (329 mg) according to the method described by Kitamura *et al.* (2011), except that 2-ethyl-5-isopropyl furan was used. Recrystallization from Et₂O afforded a yellow solid (263 mg). ¹H-NMR: δ 1.51–1.56 (m, 18H), 3.28 (q, J = 7.5 Hz, 4H), 3.92–3.99 (m, 2H), 7.23–7.28 (m, 4H), 8.89 (s, 4H), 8.95 (s, 4H); ¹³C-NMR: δ 14.52, 23.59, 26.63, 28.65, 120.24, 122.75, 122.99, 123.27, 137.81, 142.57; EIMS: *m/z* (%) 368 (100); Elemental analysis for C₂₈H₃₂: C, 91.25; H, 8.75. Found: C, 91.17; H, 8.89. Single crystals suitable for X-ray analysis were obtained by slow evaporation from Et₂O.

Refinement

All the H atoms were positioned geometrically and refined using a riding model with C—H = 0.94 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic C—H, C—H = 0.99 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for CH, and C—H = 0.97 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for CH₃. The positions of methyl H atoms were optimized rotationally.

Figures

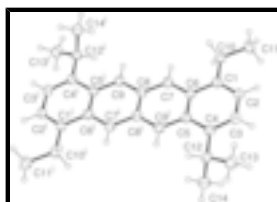


Fig. 1. The molecular structure of the title compound, showing the atomic numbering numbering and 30% probability displacement ellipsoids for non-H atoms. Symmetry code: (i) $-x, -y + 1, -z$.

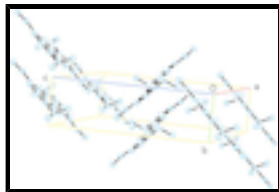


Fig. 2. The packing diagram of the title compound. Hydrogen atoms are omitted for clarity.

1,7-Diethyl-4,10-diisopropyltetracene

Crystal data

$C_{28}H_{32}$

$M_r = 368.54$

Monoclinic, $P2_1/n$

Hall symbol: $-P\ 2_1/n$

$a = 12.901\ (4)\ \text{\AA}$

$b = 5.057\ (2)\ \text{\AA}$

$c = 16.962\ (6)\ \text{\AA}$

$\beta = 106.513\ (9)^\circ$

$V = 1061.0\ (7)\ \text{\AA}^3$

$Z = 2$

$F(000) = 400$

$D_x = 1.154\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 3820 reflections

$\theta = 3.3\text{--}27.5^\circ$

$\mu = 0.06\ \text{mm}^{-1}$

$T = 203\ \text{K}$

Prism, yellow

$0.25 \times 0.13 \times 0.1\ \text{mm}$

Data collection

Rigaku R-Axis RAPID IP
diffractometer

Radiation source: fine-focus sealed tube
graphite

Detector resolution: $10\ \text{pixels mm}^{-1}$

ω scans

9469 measured reflections

2423 independent reflections

1318 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.083$

$\theta_{\text{max}} = 27.5^\circ$, $\theta_{\text{min}} = 3.3^\circ$

$h = -16 \rightarrow 16$

$k = -6 \rightarrow 6$

$l = -22 \rightarrow 22$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.081$

$wR(F^2) = 0.282$

$S = 1.10$

2423 reflections

130 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1483P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} < 0.001$

$\Delta\rho_{\text{max}} = 0.26\ \text{e \AA}^{-3}$

$\Delta\rho_{\text{min}} = -0.42\ \text{e \AA}^{-3}$

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is used for estimating s.u.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2359 (2)	0.9140 (5)	-0.04992 (16)	0.0445 (7)
C2	0.3434 (2)	0.8926 (5)	-0.01162 (17)	0.0475 (7)
H2	0.3918	1.0027	-0.0286	0.057*
C3	0.3853 (2)	0.7094 (5)	0.05323 (17)	0.0492 (7)
H3	0.4606	0.7005	0.0766	0.059*
C4	0.32137 (19)	0.5464 (5)	0.08311 (16)	0.0436 (7)
C5	0.20585 (18)	0.5603 (5)	0.04493 (15)	0.0418 (7)
C6	0.16283 (19)	0.7463 (4)	-0.02117 (16)	0.0428 (7)
C7	0.05225 (19)	0.7561 (5)	-0.05762 (16)	0.0457 (7)
H7	0.025	0.8765	-0.1008	0.055*
C8	-0.02107 (19)	0.5923 (5)	-0.03256 (16)	0.0429 (7)
C9	-0.13335 (19)	0.6016 (5)	-0.06961 (16)	0.0446 (7)
H9	-0.1606	0.722	-0.1128	0.054*
C10	0.1906 (2)	1.0987 (5)	-0.12112 (17)	0.0501 (7)
H10A	0.1395	1.2188	-0.1063	0.06*
H10B	0.1497	0.9937	-0.1683	0.06*
C11	0.2731 (2)	1.2644 (6)	-0.14795 (19)	0.0597 (8)
H11A	0.322	1.1486	-0.1658	0.09*
H11B	0.314	1.3712	-0.1021	0.09*
H11C	0.236	1.3789	-0.193	0.09*
C12	0.36829 (19)	0.3617 (5)	0.15477 (17)	0.0470 (7)
H12	0.3269	0.1943	0.1437	0.056*
C13	0.4870 (2)	0.2951 (6)	0.16758 (19)	0.0586 (8)
H13A	0.5306	0.452	0.1855	0.088*
H13B	0.4974	0.2324	0.1163	0.088*
H13C	0.5088	0.1583	0.2091	0.088*
C14	0.3535 (2)	0.4794 (6)	0.23422 (18)	0.0586 (8)
H14A	0.2772	0.509	0.2276	0.088*
H14B	0.392	0.6462	0.246	0.088*
H14C	0.3819	0.3576	0.2794	0.088*

supplementary materials

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0426 (14)	0.0452 (14)	0.0469 (16)	-0.0039 (11)	0.0144 (11)	-0.0033 (11)
C2	0.0424 (14)	0.0539 (15)	0.0479 (16)	-0.0092 (12)	0.0153 (12)	-0.0026 (12)
C3	0.0405 (14)	0.0496 (15)	0.0564 (18)	-0.0035 (11)	0.0120 (12)	-0.0034 (12)
C4	0.0399 (13)	0.0459 (14)	0.0436 (15)	-0.0020 (11)	0.0099 (11)	-0.0050 (11)
C5	0.0383 (13)	0.0444 (14)	0.0422 (15)	-0.0013 (10)	0.0108 (10)	-0.0027 (11)
C6	0.0423 (14)	0.0432 (14)	0.0435 (16)	-0.0015 (10)	0.0134 (11)	-0.0016 (11)
C7	0.0408 (14)	0.0498 (15)	0.0461 (16)	0.0001 (11)	0.0117 (11)	0.0043 (11)
C8	0.0389 (14)	0.0438 (14)	0.0465 (16)	-0.0009 (10)	0.0129 (11)	-0.0001 (10)
C9	0.0398 (13)	0.0459 (14)	0.0454 (16)	0.0013 (11)	0.0074 (11)	0.0048 (11)
C10	0.0486 (16)	0.0493 (15)	0.0515 (18)	-0.0053 (11)	0.0125 (13)	0.0029 (12)
C11	0.0577 (18)	0.0622 (18)	0.061 (2)	-0.0058 (14)	0.0193 (15)	0.0121 (14)
C12	0.0398 (14)	0.0497 (15)	0.0491 (17)	-0.0002 (11)	0.0087 (11)	0.0013 (12)
C13	0.0436 (16)	0.0650 (18)	0.063 (2)	0.0063 (13)	0.0088 (13)	0.0059 (14)
C14	0.0576 (18)	0.0711 (19)	0.0466 (17)	0.0016 (14)	0.0140 (13)	0.0007 (14)

Geometric parameters (\AA , $^\circ$)

C1—C2	1.359 (3)	C9—H9	0.94
C1—C6	1.451 (3)	C10—C11	1.522 (3)
C1—C10	1.507 (4)	C10—H10A	0.98
C2—C3	1.422 (4)	C10—H10B	0.98
C2—H2	0.94	C11—H11A	0.97
C3—C4	1.362 (3)	C11—H11B	0.97
C3—H3	0.94	C11—H11C	0.97
C4—C5	1.448 (3)	C12—C13	1.522 (3)
C4—C12	1.516 (3)	C12—C14	1.534 (4)
C5—C9 ⁱ	1.394 (3)	C12—H12	0.99
C5—C6	1.448 (3)	C13—H13A	0.97
C6—C7	1.385 (3)	C13—H13B	0.97
C7—C8	1.411 (3)	C13—H13C	0.97
C7—H7	0.94	C14—H14A	0.97
C8—C9	1.406 (3)	C14—H14B	0.97
C8—C8 ⁱ	1.430 (5)	C14—H14C	0.97
C9—C5 ⁱ	1.394 (3)		
C2—C1—C6	117.7 (2)	C11—C10—H10A	108.3
C2—C1—C10	122.9 (2)	C1—C10—H10B	108.3
C6—C1—C10	119.4 (2)	C11—C10—H10B	108.3
C1—C2—C3	122.2 (2)	H10A—C10—H10B	107.4
C1—C2—H2	118.9	C10—C11—H11A	109.5
C3—C2—H2	118.9	C10—C11—H11B	109.5
C4—C3—C2	123.0 (2)	H11A—C11—H11B	109.5
C4—C3—H3	118.5	C10—C11—H11C	109.5
C2—C3—H3	118.5	H11A—C11—H11C	109.5
C3—C4—C5	117.4 (2)	H11B—C11—H11C	109.5

C3—C4—C12	121.7 (2)	C4—C12—C13	114.0 (2)
C5—C4—C12	120.9 (2)	C4—C12—C14	110.1 (2)
C9 ⁱ —C5—C6	118.2 (2)	C13—C12—C14	109.4 (2)
C9 ⁱ —C5—C4	122.0 (2)	C4—C12—H12	107.7
C6—C5—C4	119.8 (2)	C13—C12—H12	107.7
C7—C6—C5	119.2 (2)	C14—C12—H12	107.7
C7—C6—C1	120.9 (2)	C12—C13—H13A	109.5
C5—C6—C1	119.8 (2)	C12—C13—H13B	109.5
C6—C7—C8	122.6 (2)	H13A—C13—H13B	109.5
C6—C7—H7	118.7	C12—C13—H13C	109.5
C8—C7—H7	118.7	H13A—C13—H13C	109.5
C9—C8—C7	122.6 (2)	H13B—C13—H13C	109.5
C9—C8—C8 ⁱ	119.0 (3)	C12—C14—H14A	109.5
C7—C8—C8 ⁱ	118.4 (3)	C12—C14—H14B	109.5
C5 ⁱ —C9—C8	122.7 (2)	H14A—C14—H14B	109.5
C5 ⁱ —C9—H9	118.7	C12—C14—H14C	109.5
C8—C9—H9	118.7	H14A—C14—H14C	109.5
C1—C10—C11	115.9 (2)	H14B—C14—H14C	109.5
C1—C10—H10A	108.3		
C6—C1—C2—C3	1.5 (4)	C2—C1—C6—C5	-1.4 (4)
C10—C1—C2—C3	-177.3 (2)	C10—C1—C6—C5	177.4 (2)
C1—C2—C3—C4	-1.5 (4)	C5—C6—C7—C8	0.4 (4)
C2—C3—C4—C5	1.3 (4)	C1—C6—C7—C8	178.6 (2)
C2—C3—C4—C12	-177.3 (2)	C6—C7—C8—C9	-179.9 (2)
C3—C4—C5—C9 ⁱ	178.6 (2)	C6—C7—C8—C8 ⁱ	-0.6 (5)
C12—C4—C5—C9 ⁱ	-2.7 (4)	C7—C8—C9—C5 ⁱ	179.9 (2)
C3—C4—C5—C6	-1.2 (4)	C8 ⁱ —C8—C9—C5 ⁱ	0.7 (5)
C12—C4—C5—C6	177.4 (2)	C2—C1—C10—C11	-0.4 (4)
C9 ⁱ —C5—C6—C7	-0.4 (4)	C6—C1—C10—C11	-179.2 (2)
C4—C5—C6—C7	179.5 (2)	C3—C4—C12—C13	-22.5 (4)
C9 ⁱ —C5—C6—C1	-178.6 (2)	C5—C4—C12—C13	158.9 (2)
C4—C5—C6—C1	1.3 (4)	C3—C4—C12—C14	100.9 (3)
C2—C1—C6—C7	-179.6 (2)	C5—C4—C12—C14	-77.7 (3)
C10—C1—C6—C7	-0.7 (4)		

Symmetry codes: (i) $-x, -y+1, -z$.

Fig. 1

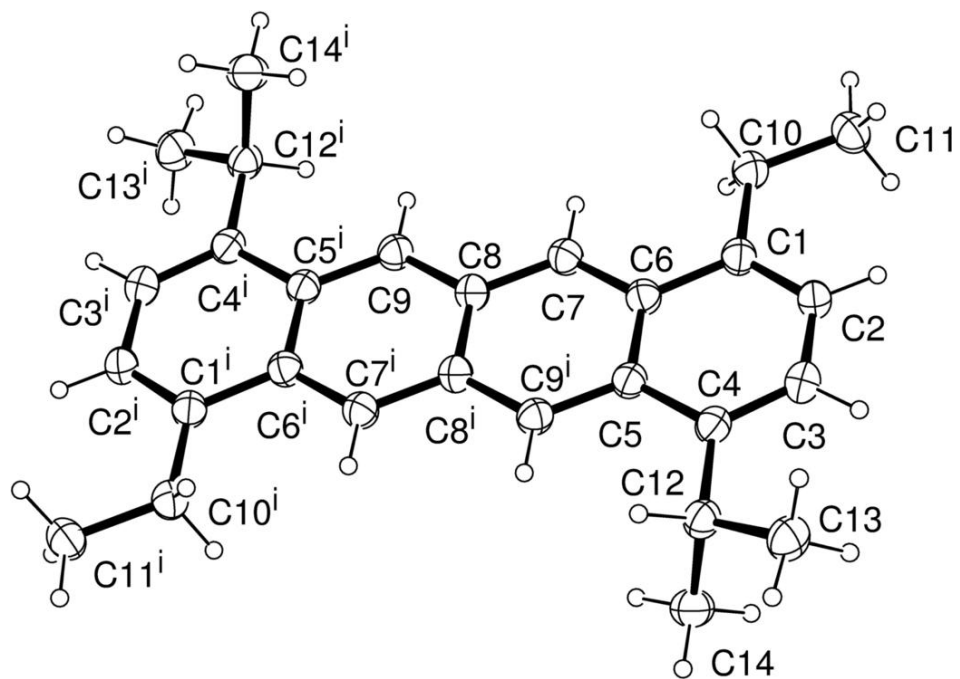


Fig. 2

