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1,2-Bis(3,5-dimethylphenyl)ethane

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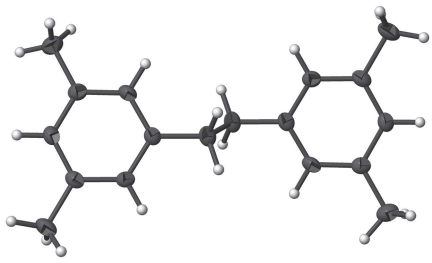
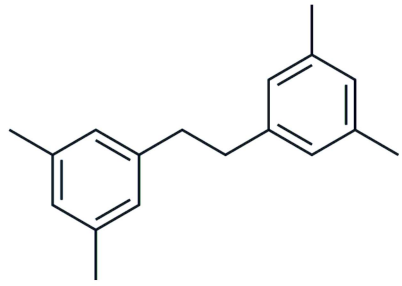
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{18}H_{22}$, is a coupling product of two metallated mesitylene molecules. The dihedral angle between the aromatic rings is 11.10 (5)° and the $C_{ar}—C_m—C_m—C_{ar}$ (ar = aromatic and m = methylene) torsion angle is 179.60 (14)°. No directional interactions beyond normal van der Waals contacts could be identified in the crystal. To our best knowledge, it is the first known coupling product of metallated mesitylene.

3D view



Chemical scheme



Structure description

In the presence of Lochmann–Schlosser's base, a metallation of the methyl group of mesitylene (1,3,5-trimethyl benzene) takes place (Schlosser, 1988). Trapping the reaction mixture with dibromoethane leads to the title compound (Fig. 1) after distillation. The dihedral angle between the aromatic rings is 11.10 (5)° and the $C_{ar}—C_{methyl}$ distances lie between 1.505 (2)-1.5100 (2) Å. For the benzene rings, normal bond length between 1.387 (2)–1.395 (2) Å are observed and the C—C—C angles range from 118.33 (14)–121.74 (14)°. No directional interactions beyond normal van der Waals' contacts could be observed in the crystal (Fig. 2).

The basic building unit, mesitylene, has been known for a long time and has been well characterized (Ladenburg 1874; Hewlett 1922). Derivatives of it have been crystallized and their structures determined (*e.g.* Trotter, 1959). The related compound nitromesitylene (Powell & Johnson, 1934) shows typical $C_{ar}—C_{ar}$ and $C_{ar}—C_{methyl}$ distances of 1.383 and 1.509 Å, respectively.

Synthesis and crystallization

The title compound was obtained by treating 11.60 ml (87.05 mmol, 1.0 eq.) mesitylene (1,3,5-trimethyl benzene) with 38.30 ml (95.76 mmol, 1.1 eq.) *n*-butyllithium and 10.75 g (95.76 mmol, 1.1 eq.) potassium-*tert*-butoxide at −78°C in 200 ml THF. Then, 8.25 ml

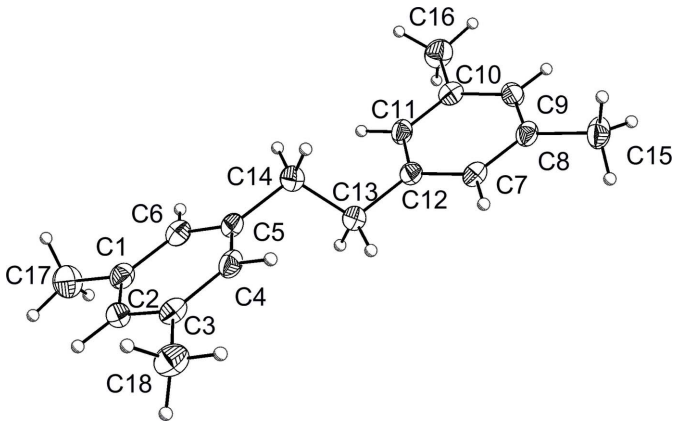


Figure 1
Molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

(95.76 mmol, 1.1 eq.) dibromoethane were added after stirring the solution for 1 h. The solution was quenched with water, extracted with diethyl ether and the organic phase was dried with sodium sulfate and distilled. The title compound crystallized as colourless plates. The yield was not determined.

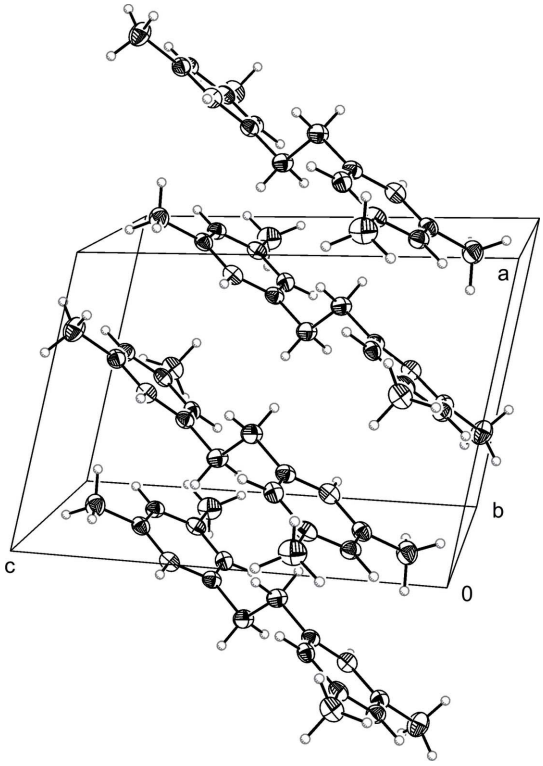


Figure 2
Unit-cell packing viewed along the *b*-axis direction.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{22}$
M_r	238.35
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	150
a, b, c (Å)	7.9315 (5), 8.2047 (5), 12.3474 (8)
α, β, γ (°)	108.513 (6), 98.090 (5), 103.805 (5)
V (Å^3)	719.18 (8)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.06
Crystal size (mm)	0.3 × 0.3 × 0.1
Data collection	
Diffractometer	Agilent Xcalibur, Sapphire3
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2014)
T_{min}, T_{max}	0.984, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	22466, 3471, 2598
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.660
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.057, 0.161, 1.05
No. of reflections	3471
No. of parameters	167
H-atom treatment	All H-atom parameters refined
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.28, −0.23

Computer programs: *CrysAlis PRO* (Agilent, 2014), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

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full crystallographic data

IUCrData (2018). **3**, x180636 [https://doi.org/10.1107/S2414314618006363]

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Crystal data

$C_{18}H_{22}$
M_r = 238.35
Triclinic, $P\overline{1}$
a = 7.9315 (5) Å
b = 8.2047 (5) Å
c = 12.3474 (8) Å
α = 108.513 (6)°
β = 98.090 (5)°
γ = 103.805 (5)°
V = 719.18 (8) Å^3
Z = 2
$F(000)$ = 260
D_x = 1.101 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4159 reflections
θ = 2.7–29.5°
μ = 0.06 mm^{-1}
T = 150 K
Plate, colourless
0.3 × 0.3 × 0.1 mm

Data collection

Agilent Xcalibur, Sapphire3 diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 16.0560 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2014)
T_{min} = 0.984, T_{max} = 1.000
22466 measured reflections
3471 independent reflections
2598 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 28.0°, θ_{min} = 2.7°
h = −10→10
k = −10→10
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.161
S = 1.05
3471 reflections
167 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.069P)^2 + 0.3054P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Special details

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

Refinement. The C-bound H atoms were included in calculated positions and treated as riding atoms, with C—H = 0.98 A° , U_{iso}(H) = 1.5U_{eq}(C) for methyl hydrogen atoms and C—H = 0.99 A° , U_{iso}(H) = 1.2U_{eq}(C) for methylene hydrogen atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.8890 (2)	0.0999 (2)	0.66953 (14)	0.0257 (3)
C2	0.9788 (2)	0.2551 (2)	0.76669 (14)	0.0251 (3)
H2	1.0541	0.2470	0.8305	0.030*
C3	0.9614 (2)	0.4226 (2)	0.77288 (13)	0.0248 (3)
C4	0.8487 (2)	0.4313 (2)	0.67960 (14)	0.0249 (3)
H4	0.8344	0.5445	0.6832	0.030*
C5	0.7558 (2)	0.2784 (2)	0.58078 (13)	0.0238 (3)
C6	0.7782 (2)	0.1138 (2)	0.57672 (13)	0.0254 (3)
H6	0.7168	0.0086	0.5094	0.030*
C7	0.6240 (2)	0.55118 (19)	0.29547 (13)	0.0225 (3)
H7	0.7017	0.6579	0.3560	0.027*
C8	0.5160 (2)	0.5651 (2)	0.20159 (13)	0.0238 (3)
C9	0.4034 (2)	0.4081 (2)	0.11365 (13)	0.0252 (3)
H9	0.3307	0.4159	0.0486	0.030*
C10	0.3946 (2)	0.2390 (2)	0.11882 (13)	0.0255 (3)
C11	0.5039 (2)	0.2301 (2)	0.21364 (13)	0.0245 (3)
H11	0.4989	0.1156	0.2180	0.029*
C12	0.62052 (19)	0.3845 (2)	0.30250 (13)	0.0225 (3)
C13	0.7396 (2)	0.3707 (2)	0.40370 (14)	0.0273 (4)
H13A	0.8214	0.4922	0.4531	0.033*
H13B	0.8134	0.2934	0.3723	0.033*
C14	0.6356 (2)	0.2929 (2)	0.48025 (14)	0.0288 (4)
H14A	0.5625	0.3707	0.5123	0.035*
H14B	0.5533	0.1717	0.4309	0.035*
C15	0.5176 (2)	0.7475 (2)	0.19759 (15)	0.0321 (4)
H15A	0.4313	0.7907	0.2395	0.048*
H15B	0.4851	0.7373	0.1156	0.048*
H15C	0.6373	0.8330	0.2351	0.048*
C16	0.2699 (2)	0.0688 (2)	0.02401 (15)	0.0371 (4)
H16A	0.3388	0.0067	−0.0255	0.056*
H16B	0.1826	0.0994	−0.0243	0.056*
H16C	0.2073	−0.0103	0.0602	0.056*
C17	0.9095 (3)	−0.0809 (2)	0.66367 (17)	0.0386 (4)
H17A	0.9959	−0.0651	0.7341	0.058*
H17B	0.9521	−0.1329	0.5937	0.058*
H17C	0.7937	−0.1619	0.6595	0.058*
C18	1.0642 (2)	0.5910 (2)	0.87696 (15)	0.0340 (4)
H18A	1.0235	0.5841	0.9472	0.051*
H18B	1.0443	0.6963	0.8623	0.051*
H18C	1.1919	0.6022	0.8892	0.051*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0263 (8)	0.0231 (8)	0.0334 (8)	0.0104 (6)	0.0123 (7)	0.0135 (7)
C2	0.0226 (7)	0.0296 (8)	0.0269 (8)	0.0088 (6)	0.0067 (6)	0.0144 (7)
C3	0.0238 (7)	0.0231 (8)	0.0276 (8)	0.0048 (6)	0.0107 (6)	0.0090 (6)
C4	0.0283 (8)	0.0197 (7)	0.0320 (8)	0.0085 (6)	0.0124 (7)	0.0134 (6)
C5	0.0223 (7)	0.0275 (8)	0.0271 (8)	0.0075 (6)	0.0099 (6)	0.0154 (6)
C6	0.0271 (8)	0.0217 (7)	0.0263 (8)	0.0049 (6)	0.0088 (6)	0.0083 (6)
C7	0.0243 (7)	0.0197 (7)	0.0228 (7)	0.0044 (6)	0.0092 (6)	0.0067 (6)
C8	0.0267 (8)	0.0232 (8)	0.0281 (8)	0.0103 (6)	0.0136 (6)	0.0130 (6)
C9	0.0256 (8)	0.0287 (8)	0.0259 (8)	0.0115 (6)	0.0074 (6)	0.0131 (6)
C10	0.0250 (8)	0.0234 (8)	0.0260 (8)	0.0050 (6)	0.0080 (6)	0.0072 (6)
C11	0.0278 (8)	0.0196 (7)	0.0295 (8)	0.0081 (6)	0.0092 (6)	0.0119 (6)
C12	0.0216 (7)	0.0253 (8)	0.0244 (7)	0.0080 (6)	0.0088 (6)	0.0118 (6)
C13	0.0242 (8)	0.0317 (8)	0.0298 (8)	0.0075 (6)	0.0067 (6)	0.0166 (7)
C14	0.0260 (8)	0.0357 (9)	0.0298 (8)	0.0096 (7)	0.0077 (7)	0.0180 (7)
C15	0.0411 (10)	0.0251 (8)	0.0388 (9)	0.0141 (7)	0.0157 (8)	0.0173 (7)
C16	0.0384 (10)	0.0289 (9)	0.0332 (9)	0.0009 (8)	0.0015 (8)	0.0068 (7)
C17	0.0438 (10)	0.0268 (9)	0.0504 (11)	0.0167 (8)	0.0107 (9)	0.0166 (8)
C18	0.0350 (9)	0.0278 (9)	0.0316 (9)	0.0037 (7)	0.0087 (7)	0.0048 (7)

Geometric parameters (Å, º)

C1—C2	1.388 (2)	C11—H11	0.9500
C1—C6	1.395 (2)	C11—C12	1.392 (2)
C1—C17	1.510 (2)	C12—C13	1.508 (2)
C2—H2	0.9500	C13—H13A	0.9900
C2—C3	1.393 (2)	C13—H13B	0.9900
C3—C4	1.387 (2)	C13—C14	1.527 (2)
C3—C18	1.505 (2)	C14—H14A	0.9900
C4—H4	0.9500	C14—H14B	0.9900
C4—C5	1.393 (2)	C15—H15A	0.9800
C5—C6	1.390 (2)	C15—H15B	0.9800
C5—C14	1.510 (2)	C15—H15C	0.9800
C6—H6	0.9500	C16—H16A	0.9800
C7—H7	0.9500	C16—H16B	0.9800
C7—C8	1.393 (2)	C16—H16C	0.9800
C7—C12	1.391 (2)	C17—H17A	0.9800
C8—C9	1.388 (2)	C17—H17B	0.9800
C8—C15	1.510 (2)	C17—H17C	0.9800
C9—H9	0.9500	C18—H18A	0.9800
C9—C10	1.395 (2)	C18—H18B	0.9800
C10—C11	1.388 (2)	C18—H18C	0.9800
C10—C16	1.508 (2)		
C2—C1—C6	118.55 (13)	C12—C13—H13A	109.0
C2—C1—C17	121.12 (15)	C12—C13—H13B	109.0

C6—C1—C17	120.33 (15)	C12—C13—C14	113.08 (13)
C1—C2—H2	119.2	H13A—C13—H13B	107.8
C1—C2—C3	121.61 (14)	C14—C13—H13A	109.0
C3—C2—H2	119.2	C14—C13—H13B	109.0
C2—C3—C18	121.13 (15)	C5—C14—C13	112.70 (13)
C4—C3—C2	118.33 (14)	C5—C14—H14A	109.1
C4—C3—C18	120.53 (14)	C5—C14—H14B	109.1
C3—C4—H4	119.1	C13—C14—H14A	109.1
C3—C4—C5	121.72 (14)	C13—C14—H14B	109.1
C5—C4—H4	119.1	H14A—C14—H14B	107.8
C4—C5—C14	120.34 (14)	C8—C15—H15A	109.5
C6—C5—C4	118.48 (14)	C8—C15—H15B	109.5
C6—C5—C14	121.18 (14)	C8—C15—H15C	109.5
C1—C6—H6	119.3	H15A—C15—H15B	109.5
C5—C6—C1	121.30 (14)	H15A—C15—H15C	109.5
C5—C6—H6	119.3	H15B—C15—H15C	109.5
C8—C7—H7	119.3	C10—C16—H16A	109.5
C12—C7—H7	119.3	C10—C16—H16B	109.5
C12—C7—C8	121.48 (14)	C10—C16—H16C	109.5
C7—C8—C15	120.62 (14)	H16A—C16—H16B	109.5
C9—C8—C7	118.62 (13)	H16A—C16—H16C	109.5
C9—C8—C15	120.74 (14)	H16B—C16—H16C	109.5
C8—C9—H9	119.3	C1—C17—H17A	109.5
C8—C9—C10	121.40 (14)	C1—C17—H17B	109.5
C10—C9—H9	119.3	C1—C17—H17C	109.5
C9—C10—C16	121.23 (15)	H17A—C17—H17B	109.5
C11—C10—C9	118.45 (14)	H17A—C17—H17C	109.5
C11—C10—C16	120.32 (14)	H17B—C17—H17C	109.5
C10—C11—H11	119.1	C3—C18—H18A	109.5
C10—C11—C12	121.74 (14)	C3—C18—H18B	109.5
C12—C11—H11	119.1	C3—C18—H18C	109.5
C7—C12—C11	118.31 (13)	H18A—C18—H18B	109.5
C7—C12—C13	121.10 (14)	H18A—C18—H18C	109.5
C11—C12—C13	120.59 (13)	H18B—C18—H18C	109.5