

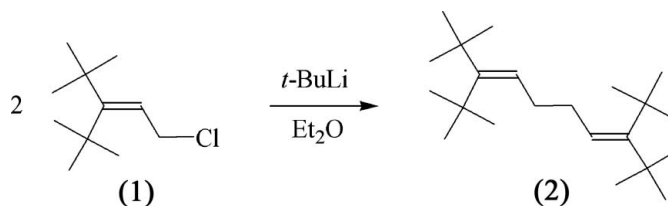
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## Key indicators

Single-crystal X-ray study  
 $T = 173$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å  
 $R$  factor = 0.056  
 $wR$  factor = 0.148  
Data-to-parameter ratio = 16.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.3,8-Di-*tert*-butyl-2,2,9,9-tetramethyldeca-  
3,7-dieneThe title compound,  $\text{C}_{22}\text{H}_{42}$ , displays crystallographic inversion symmetry. The conformation of the central chain is defined by the torsion angle  $\text{C}=\text{C}-\text{C}-\text{C}$  of  $124.3$  (3)°. The  $\text{C}=\text{C}-\text{C}$  angle in the chain is  $133.2$  (2)°. The central  $\text{C}-\text{C}$  bond is short at  $1.516$  (4) Å.

## Comment

During our studies on highly *tert*-butylated hydrocarbons (Hänel, 1996; Hopf *et al.*, 2001) we investigated the behaviour of the allyl chloride (1) towards various metals and metal-organic reagents. When (1) is reacted with *tert*-butyl lithium in diethyl ether at 203 K it dimerizes and yields the diene (2). Here we present the structure of (2).

The molecule of (2), which displays crystallographic inversion symmetry, is shown in Fig. 1. Some evidence of steric crowding is provided by short intramolecular  $\text{H}\cdots\text{H}$  contacts, such as  $\text{H}1\text{A}\cdots\text{H}11\text{C} = 1.82$  Å,  $\text{H}1\text{A}\cdots\text{H}9\text{A} = 1.81$  Å and  $\text{H}5\text{A}\cdots\text{H}7\text{A} = 1.85$  Å; there are no unusually short intermolecular contacts. The conformation of the central chain is largely defined by the  $\text{C}3=\text{C}4-\text{C}5-\text{C}5^i$  torsion angle (Table 1); the torsion angle around the central bond  $\text{C}5-\text{C}5^i$  is  $180^\circ$  by symmetry. Molecular dimensions (Table 1) may be regarded as normal, such as the  $\text{C}3=\text{C}4$  and  $\text{C}4-\text{C}5$  bond lengths and the  $\text{C}3=\text{C}4-\text{C}5$  angle. A similar  $\text{C}=\text{C}$  bond length,  $1.349$  (2) Å, and  $\text{C}=\text{C}-\text{C}$  angle,  $131.1$  (2)°, were observed in 1,1,4,4-tetra-*tert*-butyl-1,3-butadiene [(3); Hopf *et al.*, 1994, 1996). The  $\text{C}5-\text{C}5^i$  bond seems rather short at  $1.516$  (4) Å; no other *tert*-butyl-substituted chain of a similar nature is available for comparison. Some caution is indicated in view of problems with the structure of (3), in which contamination by small quantities of an impurity initially led to systematically incorrect dimensions, later corrected (Hopf *et al.*, 1994, 1996).

## Experimental

To a solution of (1) (1.0 g, 5.3 mmol) in diethyl ether (7 ml) was added over a period of 30 min at 203 K a 1.7 M *tert*-butyllithium solution (6.3 ml) in diethyl ether. The mixture was stirred for 30 min at room temperature and refluxed for 12 h to complete the

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dimerization. After work-up, solvent removal and recrystallization (ethanol), (2) (280 mg, 34%) was obtained. The spectroscopic data agree with the proposed structure (Hänel, 1996).

Crystal data

$C_{22}H_{42}$   
 $M_r = 306.56$   
 Triclinic,  $P\bar{1}$   
 $a = 6.350 (4) \text{ \AA}$   
 $b = 8.161 (4) \text{ \AA}$   
 $c = 10.655 (6) \text{ \AA}$   
 $\alpha = 87.47 (4)^\circ$   
 $\beta = 88.08 (4)^\circ$   
 $\gamma = 67.63 (4)^\circ$

$V = 510.0 (5) \text{ \AA}^3$   
 $Z = 1$   
 $D_x = 0.998 \text{ Mg m}^{-3}$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.06 \text{ mm}^{-1}$   
 $T = 173 (2) \text{ K}$   
 Tablet, colourless  
 $0.7 \times 0.5 \times 0.2 \text{ mm}$

Data collection

Stoe STADI-4 diffractometer  
 $\omega/\theta$  scans  
 Absorption correction: none  
 2714 measured reflections  
 1783 independent reflections  
 1201 reflections with  $I > 2\sigma(I)$

$R_{int} = 0.057$   
 $\theta_{max} = 24.9^\circ$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: none

Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.056$   
 $wR(F^2) = 0.148$   
 $S = 1.08$   
 1783 reflections  
 106 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0574P)^2 + 0.1511P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 0.16 \text{ e \AA}^{-3}$   
 $\Delta\rho_{min} = -0.17 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C3—C4	1.336 (3)	C5—C5 <sup>i</sup>	1.516 (4)
C4—C5	1.508 (3)		
C3—C4—C5	133.2 (2)	C4—C5—C5 <sup>i</sup>	112.0 (2)
C3—C4—C5—C5 <sup>i</sup>	124.3 (3)		

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

Methyl H atoms were located in difference syntheses, idealized (C—H =  $0.98 \text{ \AA}$  and H—C—H =  $110^\circ$ ) and refined on the basis of

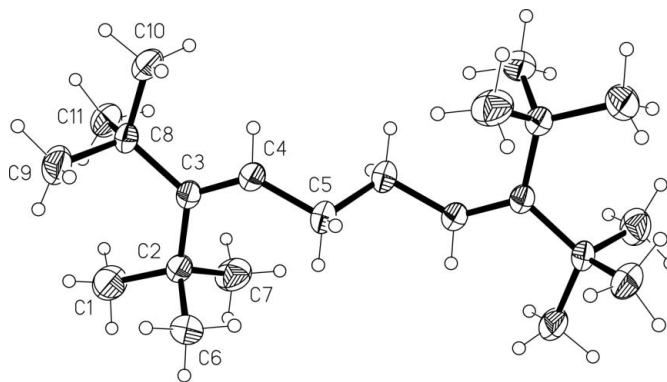


Figure 1

The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

rigid groups allowed to rotate but not tip. Other H atoms were placed in calculated positions (C—H =  $0.95 \text{ \AA}$  for  $sp^2$  CH and  $0.99 \text{ \AA}$  for  $CH_2$ ) and allowed to ride on their parent C atoms. All  $U_{iso}(H)$  values were fixed at 1.2 times  $U_{eq}$  of the parent atom.

Data collection: *DIF4* (Stoe & Cie, 1992); cell refinement: *DIF4*; data reduction: *REDU4* (Stoe & Cie, 1992); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* (Siemens, 1994); software used to prepare material for publication: *SHELXL97*.

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