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THE CRYSTAL AND MOLECULAR STRUCTURES OF TWO SUBSTITUTED PENTALENES

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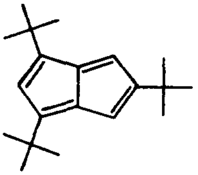
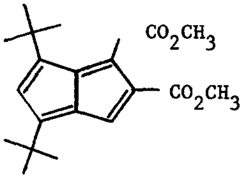
In addition to the first stable alkyl pentalene, 1,3,5-tri-t-butylpentalene(1)[1], Hafner and Suda synthesized dimethyl-4,6-di-t-butylpentalene-1,2-dicarboxylate (2)[2] and a series of other substituted pentalenes[3]. To obtain information about the geometry of the theoretically interesting pentalene system[4] we have performed X-ray crystal structure analyses of 1 and 2[5].



1



2

The results of the analyses are shown in figures 1 to 4. In both cases the pentalene system is planar with localized double bonds. In 2 the ester group in 1-position is nearly orthogonal to the plane of the bicyclic system, whereas the ester group in 2-position is approximately coplanar with the bicyclic system.

X-ray structure analysis of 1 :

Crystal data: dark blue orthorhombic plates, m.p. 59-60°C, recrystallized from n-hexane, $C_{20}H_{30}$, M = 270.5, a = 9.77 ± 0.01, b = 9.40 ± 0.01, c = 19.98 ± 0.01 Å, V = 1835 $Å^3$, Z = 4, d_{exp} = 1.00 gcm^{-3}, d_X = 0.979 gcm^{-3}, space group $P2_12_12_1$.

1219 reflections hk0 - hk12 with $\theta \leq 60^{\circ}$ were measured with $Cu_{K\alpha}$ radiation (λ = 1.5418 Å) on a two-circle diffractometer. 893 reflections with $|F| \geq 2\sigma_F$ were used for structure determination and refinement. The structure was determined by direct methods and refined to R = 0.128[6]. The positions of the hydrogen atoms were calculated from the position of the carbon atoms and not refined.

X-ray structure analysis of **2** :

Crystal data: blue monoclinic plates, crystallized from n-hexane, m.p. 120-121°C, $C_{20}O_4H_{26}$, M = 330.4, a = 11.13 ± 0.01, b = 12.56 ± 0.01, c = 13.81 ± 0.01 Å, β = 101.4 ± 0.1°, V = 1892 Å³, d_X = 1.160 gcm^{-3}, Z = 4, space group $P2_1/c$. 2284 independent reflections with $|F| \geq 3\sigma_F$ measured with $Cu_{K\alpha}$ radiation on a two-circle diffractometer were used for the analysis. The structure was solved by direct methods and refined to R = 0.103[6].

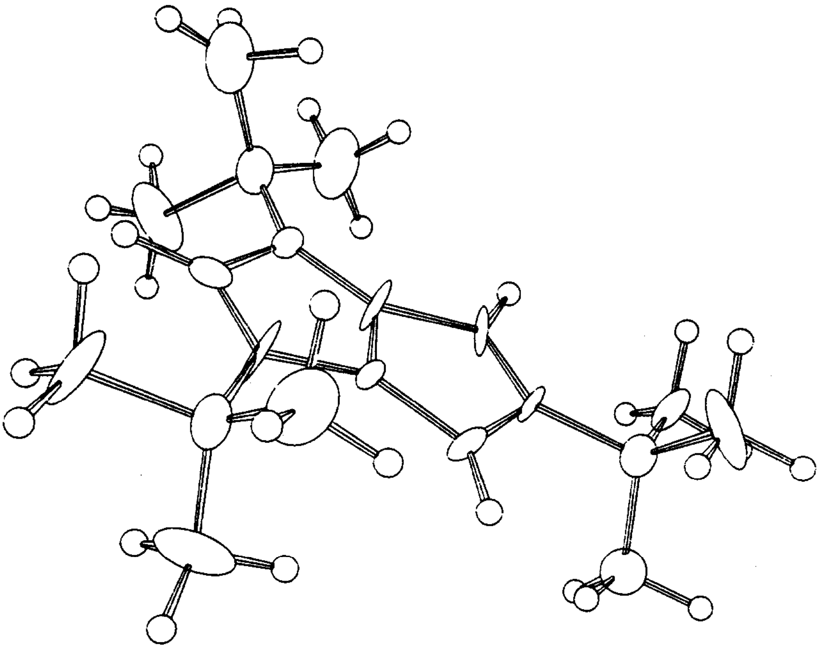


fig. 1: The molecular geometry of **1**

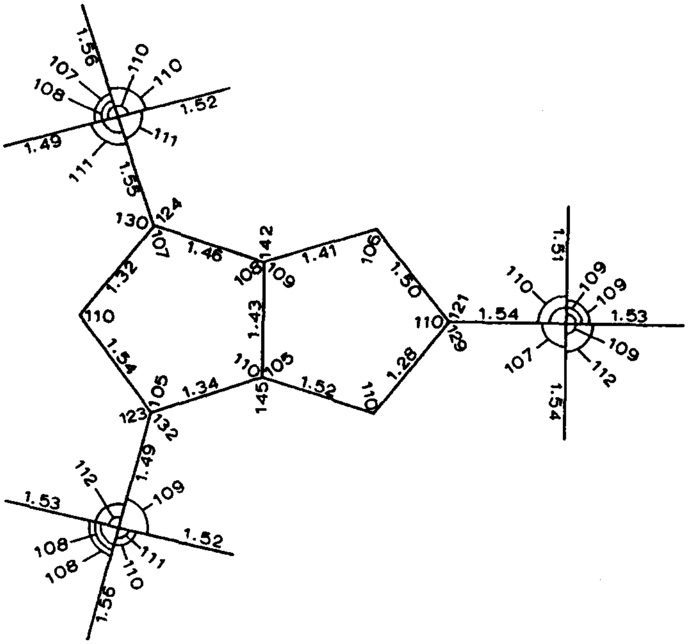


fig. 2: Bond lengths in Å and bond angles in 1; standard deviations $\sigma_r = 0.01$ Å, $\sigma_\alpha = 1°$

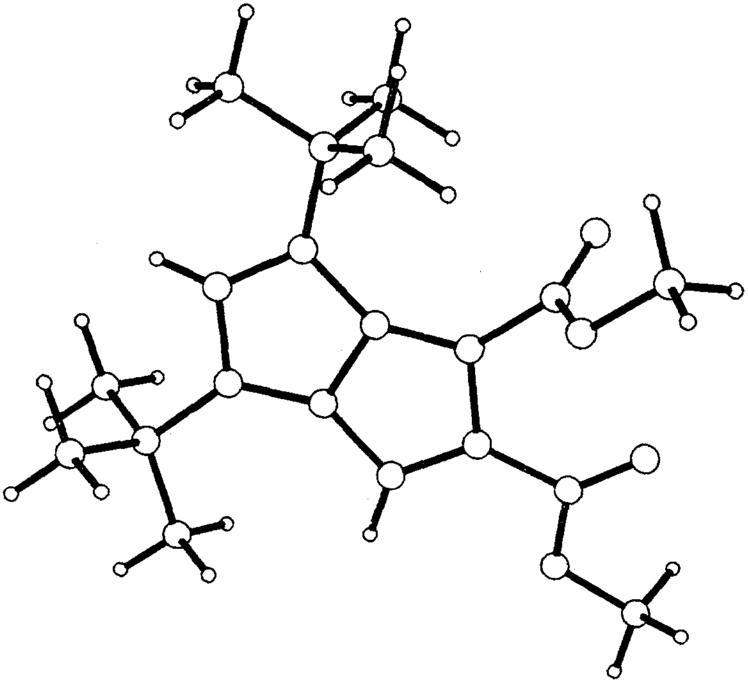


fig. 3: The molecular geometry of 2

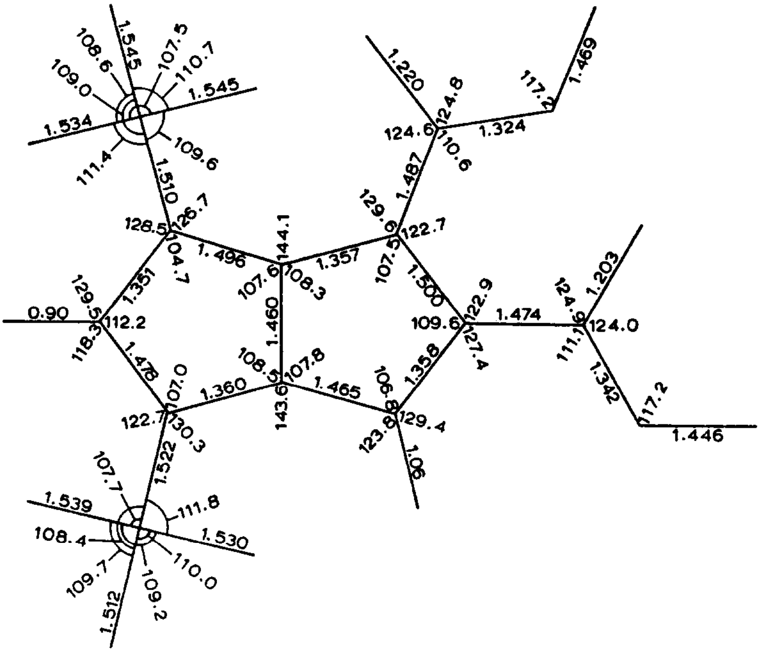


fig. 4: Bond lengths in Å and bond angles in **2**; standard deviations $\sigma_r = 0.006$ Å, $\sigma_\alpha = 0.4^\circ$

LITERATURE

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