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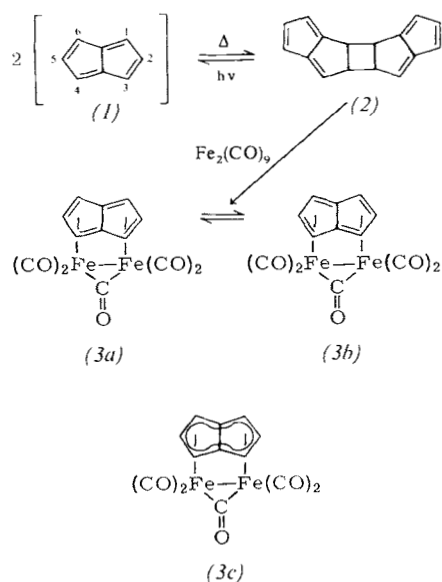
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## Synthesis of Carbonyliron Complexes of Pentalene and Its Derivatives

By Wolf Weidemüller and Klaus Hafner[\*]

Pentalene (1) and its methyl derivatives are characterized—like cyclobutadiene<sup>[1]</sup>—by a highly pronounced tendency to dimerization<sup>[2,3]</sup>. The only alkyl derivative hitherto isolated, 1,3,5-tri-*tert*-butylpentalene, is, for steric reasons, not capable of dimerization<sup>[4]</sup>. On photolysis in a matrix at  $-196^{\circ}\text{C}$  several dimeric pentalenes could be converted to the corresponding, UV-spectroscopically detectable monomers.



We have now observed that fission of pentalene dimers is also achieved by transition metals. Treating (2)<sup>[5]</sup> with  $\text{Fe}_2(\text{CO})_9$  at  $50^{\circ}\text{C}$  (in methylcyclohexane under carbon monoxide in a sealed vessel) yielded the pentalene-pentacarbonyldiiron complex (3) as yellow-brownish crystals, stable at room temperature under nitrogen<sup>[6]</sup> [yield 9%, m. p.  $140^{\circ}\text{C}$  dec., 100 MHz  $^1\text{H-NMR}$  spectrum (in  $\text{C}_6\text{D}_6$ ):  $\text{A}_2\text{B}$  system at  $\tau=4.54$  (t,  $J=2.5$  Hz, H-2, H-5) and  $\tau=6.77$  (d,  $J=2.5$ , H-1, H-3, H-4, H-6); IR spectrum (KBr pellet): 2030, 1985,  $1945\text{ cm}^{-1}$  (terminal CO),  $1750\text{ cm}^{-1}$  (bridging CO)]. Similarly the corresponding complex of 1,3-dimethylpentalene could be synthesized in 21% yield from its dimer<sup>[3]</sup> as green crystals, melting at  $136^{\circ}\text{C}$  [100 MHz  $^1\text{H-NMR}$  spectrum (in  $\text{C}_6\text{D}_6$ ):  $\text{A}_2\text{B}$  system at  $\tau=5.61$  (t,  $J=2.5$  Hz, H-5) and  $\tau=6.93$  (d,  $J=2.5$  Hz, H-4, H-6);

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$\tau=6.00$  (s, H-2) and  $\tau=8.87$  (s,  $2\text{CH}_3$ ); IR spectrum (KBr pellet): 2020, 1980,  $1940\text{ cm}^{-1}$  (terminal CO),  $1750\text{ cm}^{-1}$  (bridging CO)]. Like the IR spectra the mass spectra of these complexes too resemble those of the 1-dimethylamino and 1-phenyl derivatives<sup>[8]</sup> of (3) obtained from the corresponding 1,2-dihydropentalenes<sup>[7]</sup> and  $\text{Fe}(\text{CO})_5$ . Besides molecular ion peaks ( $\text{M}^+=354$  and 382) fragment peaks due to successive cleavage of five carbonyl groups and two iron atoms also appear.

The NMR spectra of these complexes do not allow distinction of either structures (3a)  $\rightleftharpoons$  (3b) with rapidly oscillating  $\pi$ -electron system or (3c) with a delocalized pentalene ligand. F. G. A. Stone *et al.*<sup>[9]</sup> recently reported the comparable bis(trimethylgermyldicarbonylruthenium) complex obtained by dehydrogenative transannular cyclization of cyclooctatetraene. Its bonding structure proposed on the basis of an X-ray analysis is not incompatible with a structure of type (3c).

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## Synthesis of 4-Mercapto-2-azetidiones having $\alpha$ -Isopropylideneacetate Groups on Nitrogen

By Rudolf Lattrell[\*]

Dedicated to Professor Werner Schultheis on the occasion of his 70th birthday

4-Mercapto-2-azetidiones (4-mercapto- $\beta$ -lactams) having  $\alpha$ -isopropylideneacetate groups attached to the nitrogen have not hitherto been described although they have long been discussed in connection with degradation reactions of penicillins and biosynthesis of penicillins and cephalosporins<sup>[1]</sup>. We have now found a general method for the synthesis of 4-mercapto-2-azetidiones<sup>[2]</sup>, and in particular for compounds of type (5) named in the title.

Starting materials were the *cis*- or *trans*-4-tritylthio-2-azetidiones (2) that have been obtained from the cysteine derivative (1) and substituted acetyl chlorides. Methanethiol can be split from the primary products of the  $\beta$ -lactam syntheses under mild conditions<sup>[2,3]</sup>.

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