

PREPARATION OF BUTADIENE CYCLOTETRAMERS ON COMPLEX
NICKEL CATALYSTS

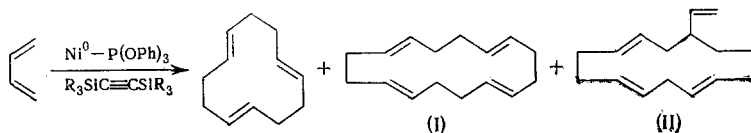
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We found that butadiene on the catalytic system: $\text{Ni}(\text{acac})_2\text{-P}(\text{OPh})_3\text{-Al}(\text{C}_2\text{H}_5)_3$, 1:1:3 (4) (60°C , 3 h), in the presence of bis(trimethylsilyl)acetylene, is converted in 90% yield to a mixture of cyclic oligomers that consists of trans,trans,trans-1,5,9-cyclododecatriene (I), trans-1,5,9,13-cyclohexadecatetraene (II), and vinyl-trans-1,7,10-cyclotetradecatriene (III) in an 80:15:5 ratio. The mixture of cyclotetramers is easily isolated by fractional distillation through a column. After separation by preparative GLC the hydrocarbons had the following constants:

Hydrocarbon (II), bp 103° (1 mm); n_D^{20} 1.5058. Infrared spectrum (ν , cm^{-1}): 975, 3030, (trans-CH=CH-). NMR spectrum (δ , ppm): 1.9 s (16H, $-\text{CH}_2-$), 5.1 s (8H, $-\text{CH}=\text{CH}-$), m/e 216.

Hydrocarbon (III), bp 126° (0.8 mm); n_D^{20} 1.5138. Infrared spectrum (ν , cm^{-1}): 920, 1000, 3085 ($-\text{C}=\text{CH}_2$), 975, 3030 (trans-CH=CH-). NMR spectrum (δ , ppm): 1.5 m (10H, $-\text{CH}_2-$), 1.92 (8H, $=\text{C}-\text{CH}_2-$), 2.3 (1H, $\text{C}_4\text{-H}$), 5.1 (2H, $-\text{C}=\text{CH}_2$), 6.0 (1H, $\text{H}-\text{C}=\text{C}$), m/e 216.



Only succinic acid is obtained by the ozonolysis of (II), while the ozonolysis of (III) gives succinic acid and 3-carboxypimelic acid.

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