1,4-Bis(*p*-pentazolylphenyl)butan, 1-*p*-Azidophenyl-4-*p*-pentazolylphenyl-butan und 1,4-Bis(*p*-azidophenyl)butan

1,4-Bis(*p*-pentazolylphenyl)butane, 1-*p*-Azidophenyl-4-*p*-pentazolylphenyl-butane and 1,4-Bis(*p*-azidophenyl)butane

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1,4-Bis(*p*-pentazolylphenyl)butane (**1**), 1-*p*-azidophenyl-4-*p*-pentazolylphenyl-butane (**2**) and 1,4-bis(*p*-azidophenyl)butane (**3**) were obtained by the reaction of 1,4-diphenylbutane-4',4"-bis(diazonium) ions with sodium azide in methanol at −50 °C. In the ^{1}H and ^{13}C NMR spectra the three compounds can be distinguished unequivocally. At −50 °C a mixture with a composition **1:2:3** of 10:30:60 was obtained. By recrystallization first from dichloromethane/methanol and then from dichloromethane/petroleum ether the pentazole components were enriched to a composition ratio of 21:62:17. The rate constants of the decompositions **1** → **2** and **2** → **3** at 0 °C were determined from the variation of the ^{1}H NMR intensities. At room temperature all of the material is converted to **3**. **3** crystallizes in two monoclinic modifications. At −70 °C a modification **3**-*LT* having space group $P2_1/c$ ($a = 950.8$, $b = 1192.6$, $c = 701.3$ pm, $\beta = 92.55°$, $Z = 2$; $R = 0.075$) was obtained. The modification crystallizing at room temperature (**3**-*HT*) has space group $I2/a$ ($a = 1514.5$, $b = 498.1$, $c = 2027.9$ pm, $\beta = 92.73°$, $Z = 4$; $R = 0.040$). Whereas both modifications consist of nearly identical molecules, their packings are quite different. When the low temperature modification is warmed to room temperature, its crystals jump like flees and are disrupted to a fine powder.

Key words: 1,4-Bis(*p*-pentazolylphenyl)butane, 1-*p*-Azidophenyl-4-*p*-pentazolylphenyl-butane, 1,4-Bis(*p*-azidophenyl)butane, Crystal Structure