Reversible Laser-Induced Bending of Pseudorotaxane Crystals


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Synthesis of \( p \)-phenylazobenzonitrile:

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\text{Aniline (9.48 g, 0.10 mol) was dissolved in 120 mL of DCM. Oxone (31.3 g, 0.10 mol) in water (150 mL) was added and the mixture was stirred vigorously at 25 °C for 2 h. The solution was extracted with DCM and organic layer was washed with HCl (1 M), sat. NaHCO}_3(aq) \text{ and sat. NaCl(aq), separately. The filtrate was evaporated to yield 1-nitrosobenzene (4.02 g, 37.5 mmol, 38%) as green oil. The product was used immediately for next step without purification. The above product (4.02 g, 37.5 mmol) and 4-aminobenzonitrile (4.46 g, 37.7 mmol) were dissolved in acetic acid (55 mL) and the mixture was stirred for 2 d at 25 °C. After that, acetic acid was evaporated and DCM (50 mL) was added to dissolve the solid. The solution was washed with sat. NaHCO}_3(aq) \text{ and sat. NaCl(aq), separately. The removal of the solvent by evaporation gave crude product which was then purified by silica gel column chromatography to yield } p \text{-phenylazobenzonitrile (4.97 g, 24.0 mmol, 64%) as orange solid.} \text{ } ^1\text{H NMR (500 MHz, CDCl}_3, \text{ r. t.): } \delta_{\text{H}} 3.95 (s, 2H, NCH}_2, 7.45 (d, 2H, C_6H}_4, J = 9 \text{ Hz}, 7.50 (m, 3H, C}_6\text{H}_5, 7.89 (d, 2H, C}_6\text{H}_4, J = 8 \text{ Hz).}
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The highlighted information is incorrect. This should be: \( \delta_{\text{H}} 7.52-7.54 \text{ (m, 3H, C}_6\text{H}_5), 7.80 \text{ (d, 2H, C}_6\text{H}_4, J = 2 \text{ Hz), 7.93-7.98 (m, 4H, Ar).} \)

![Fig. \( ^1\text{H NMR spectrum (500 MHz, CDCl}_3) \text{ of } p \text{-phenylazobenzonitrile.} \)](image)