Reduction of a benzonitrile; 1-[4-(trans-4-Pentylcyclohexyl)phenyl]methanamine

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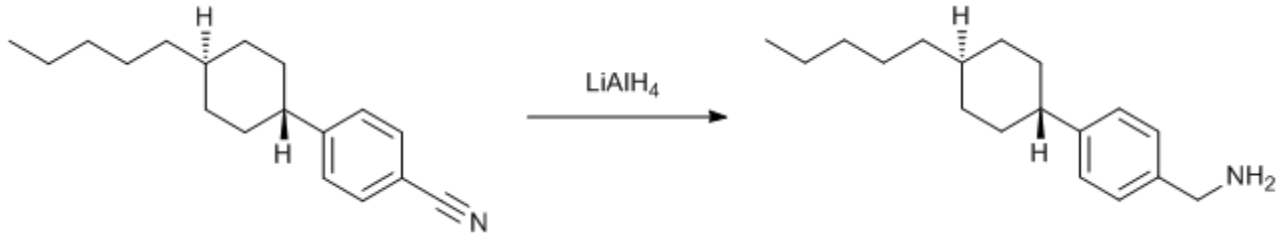

Chemicals Used

4-(Trans-4-pentylcyclohexyl)-benzonitrile (Fluorochem), 300840
Lithium aluminum hydride, Sigma Aldrich, powder, 95%, 19,987-7. freshly opened can
Ether, Sigma Aldrich, anhydrous, 99.8%, 29,608-2, freshly opened bottle
Sodium sulfate, Sigma Aldrich, anhydrous, 99+%, 23,931-3

Procedure

An oven dried 100ml three neck flask, equipped with magnetic stirrer, water condenser and nitrogen inlet was charged under nitrogen with 1.0 g (4 mmol) 4-(trans-4-pentylcyclohexyl)-benzonitrile (Fluorochem) and 50 ml of anhydrous ether. The mixture was stirred until all of the nitrile had dissolved. 1.0 g of freshly opened powdered lithium aluminum hydride was then added through a powder funnel and the mixture stirred overnight. A 10ml pressure equalized dropping funnel was then attached and the excess hydride decomposed by drop wise addition of 5 ml water over one hour. A copious white precipitate of aluminum hydroxide was then present. The precipitate was filtered off with a small Buchner funnel under water aspirator pressure and washed under vacuum with 5 ml ether. The filtrate of ether/amine was dried for one hour over 2 grams of anhydrous sodium sulfate and the ether solution decanted into a 100 ml round bottom flask. The sulfate residue was twice stirred, utilizing a glass rod, with two 5 ml ether portions which were each decanted and combined with the filtrate. Ether then was removed on a rotovap (water bath ~80^0C) and the residue pipetted into a 2 ml conical distilling flask equipped with a short path distilling head and an oil bath. Distillation under full rotary pump vacuum (bath temperature ~150^0), yielded 800 mg (80%) of the liquid crystalline amine, which solidified on standing at room temperature. Yields of all liquid crystalline amines in these references are in the 70-85% range, with losses mostly mechanical.

Author's Comments

Caution! Lithium aluminum hydride is extremely water sensitive. Use a freshley opened can if possible and weigh in glovebox if possible. On contact with excess water it decomposes violently with hydrogen evolution. It should always be decomposed in a reaction mixture when necessary by slow dropwise addition of water. This and other amines described in the primary and secondary references are hydroscopic and should be stored in dessicators.The methanamines may be converted to N-alkyl substituted liquid crystalline cyclohexylphenyl amines or biphenyl amines by further reaction with acid chlorides and lithium aluminum hydride reduction (see references).

Data

B.p. 135^0(0.1 mm),

m.p. crystal-isotropic (K-I), 27^0, cooling, isotropic-smectic-A, (I-S_A) , 9^0, Smectic-A-crystal, (S_A-K), 0^0.

I.r. (cm-1) 3300,3250,3010,2900,1600

Analysis: Calculated for $C_{18}H_{29}N$: C, 83.32, H, 11.27, N, 5.40. Found: C, 83.22, H, 11.40, N, 5.46

Lead Reference

John H. MacMillan and Mortimer M. Labes, "Low Transition Temperature Liquid Crystalline Amines Incorporating the Trans-1,4-Cyclohexane Ring System", Molecular Crystals and Liquid Crystals, Vol. 55, p 61, (1979). DOI: dx.doi.org/10.1080/00268947908069791

Other References

John H. MacMillan and Mortimer M. Labes, "Low Transition Temperature Liquid Crystalline Amines Incorporating the Biphenyl Ring System", Mol. Crystals and Liquid Crystals Letters, Vol. 56, p51, (1979). DOI: Link: http://dx.doi.org/10.1080/01406567908071966

Chemspider deposition: http://www.chemspider.com/Chemical-Structure.18536103.html

John H. MacMillan and Mortimer M. Labes, "Amine Substituted Liquid Crystal Compositions", U.S. Patent 4,293,193, Oct. 6, 1981.
Keywords: amines, **hydrogenation**