# Aromatic secondary amines from aromatic amides:

## N-Butyl-4-(trans-4-pentylcyclohexyl)aniline

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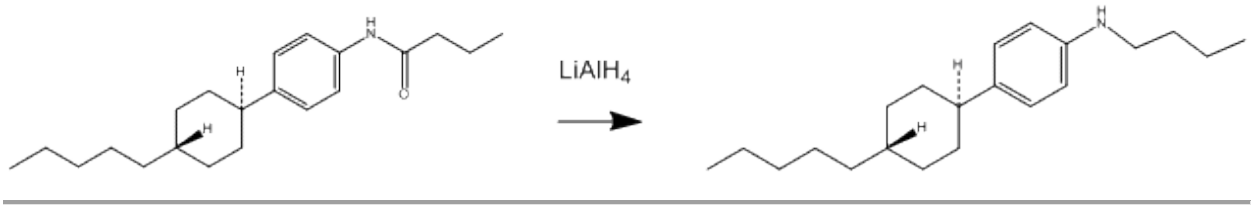


# Chemicals Used:

N-[4-(trans-4-Pentylcyclohexyl)phenyl]butanamide, prepared in high yield from the corresponding substituted aniline by reaction with butanoyl chloride, see primary reference 1.

Lithium aluminum hydride, Sigma Aldrich, powder, 95%, 19,987-7, freshly opened can.

Ether, Sigma Aldrich, anhydrous, 99+%, A.C.S. reagent, 17,926-4, freshly opened can.

Sodium sulfate, anhydrous, powder, 99+%, A.C.S. reagent, 23,859-7.

# Procedure:

These compounds were easily prepared in essentially quantitative yield by reduction of the previously described (reference 1) amides with lithium aluminum hydride in dry ether. A representative synthesis follows.

To a 50ml round bottom three neck flask, equipped with magnetic stirrer, water condenser, calcium chloride drying tube on top of the condenser, and nitrogen inlet was charged 25ml ether and 0.127 g (0.4 mmol) of N-(p-trans-pentylcyclohexyl)butanamide. The mixture was stirred until homogeneous. Lithium aluminum hydride (1.0 g, 26 mmol) was added and the mixture further stirred under nitrogen for 12 hours (overnight). Water was then slowly added dropwise to the solution via a 10 ml pressure equalized dropping funnel until a copious white precipitate separated with mild exotherm. The solution was then cooled to room temperature with an ice bath. The precipitate was suction filtered, washed with an additional 5 ml of ether, and the combined filtrate dried over ~2 g of anhydrous sodium sulfate for 2 hours. The dried ether solution was decanted to a 25 ml beaker, and the sulfate residue washed with an additional 2-3 ml of ether which was combined with the decanted ether solution. A boiling stone was added and the ether removed on a steam bath or warm water bath giving an essentially quantitative yield of N-butyl-(4-pentylcyclohexyl)aniline, a nematic liquid crystal.

An analytically pure sample of material was obtained by micro distillation in a Kugelrohr apparatus at reduced pressure under pump vacuum. The material slowly solidified on standing at room temperature.

## Author's Comments:

CAUTION! Lithium aluminum hydride is irritating and moisture sensitive. It reacts violently if exposed to large quantities of water or hydroxylic solvents. Weigh quickly in a glove box if possible. Wear latex gloves. The procedure is applicable for preparing a wide variety of secondary aromatic amines from aromatic amides.

By entirely analogous reactions other amines were synthesized in high yield by this procedure. See primary and secondary references. Examples are:

**N-Heptyl-(4-pentylcyclohexyl)aniline**, m.p. 46.4°.

I.r. (thin film, melted material), 3430, 3050, 2950, 1620, 1580 $cm^{-1}$.

| | | | |
|---|---|---|---|
| Analysis: Calculated for $C_{24}H_{41}N$: | C, 83.89 | H, 12.03 | N, 4.08 |
| Found: | C, 83.91 | H, 12.26 | N, 3.96 |

**N-Heptyl-(4-heptylcyclohexyl)aniline**, b.p. ~145°(0.1mm), m.p. 51.0°

I.r. (thin film, melted material) ,3430, 3050, 2950, 1620, 1590 $cm^{-1}$.

| | | | |
|---|---|---|---|
| Analysis: Calculated for $C_{26}H_{45}N$: | C, 84.02 | H, 12.21 | N, 3.77 |
| Found: | C, 83.91 | H, 12.42 | N, 3.72 |

# Data:

## N-Butyl-(4-pentylcyclohexyl)aniline

b.p. ~140°(0.1mm). K-N, (crystal-nematic), 28°, N-I (nematic-isotropic), 33.5°.

I.r. (thin film, melted material), 3350, 3050, 2950, 1620, $cm^{-1}$.

| | | | |
|---|---|---|---|
| Analysis: Calculated for $C_{21}H_{35}N$: | C, 83.65 | H, 11.70 | N, 4.65 |
| Found: | C, 83.73 | H, 11.75 | N, 4.77 |

# 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**

**http://jhm2.homestead.com/files/13.pdf**

# 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**

**http://jhm3.homestead.com/files/15.pdf**

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

**http://jhm3.homestead.com/files/patent1.pdf**

Chemspider deposition:

**http://www.chemspider.com/Chemical-Structure.29354029.html**

**Keywords: carbamate**, **amide, amine, aniline, acid chloride, secondary amine, reduction, lithium aluminum hydride**