Secondary aromatic amides from anilines:

N-[4-(trans-4-Heptylcyclohexyl)phenyl]heptanamide

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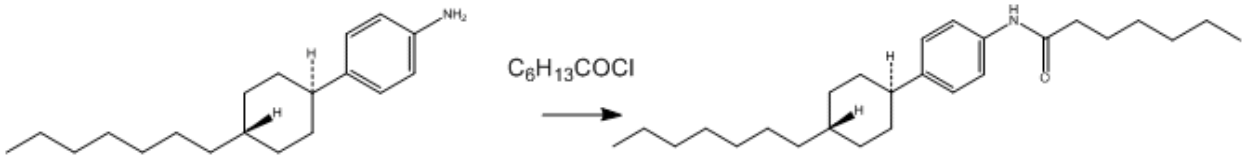

Chemicals Used:

(4-trans-4-Heptylcyclohexyl)aniline, prepared in high yield from the carbamate by basic hydrolysis of Methyl- (p-trans-heptylcyclohexyl)carbamate, see primary reference 1.

Chemspider deposition: http://www.chemspider.com/Chemical-Structure.29354034.html

Heptanoyl chloride Sigma Aldrich, 99%, 14,724-9

Toluene, Sigma Aldrich ,99.5+ %, A.C.S. Reagent, 17,941-8

Ethanol, 190 proof, Sigma Aldrich , 95+%, 49,351-1

Procedure:

To a 50ml round bottom three neck flask, equipped with magnetic stirrer, water condenser, calcium chloride drying tube on top of the condenser, septum cap and nitrogen inlet was charged 0.196 g (0.72 mmol) of (4-trans-4-heptylcyclohexyl)aniline and 10 ml toluene. The mixture was stirred until homogeneous. Then 1 ml heptanoyl chloride was syringed into the solution and a copious white precipitate immediately separated with mild exotherm. The solution was then cooled to room emperature with an ice bath. The nearly quantitative precipitate was suction filtered, air dried on small watch glass. and taken up in 10 ml of boiling 190 proof ethanol. Cooling gave 95 mg (34%) of analytically pure N-[4-(trans-4-Heptylcyclohexyl)phenyl]heptanamide.

Author's Comments:

CAUTION! Heptanoyl chloride is lachrimatory, irritating and moisture sensitive. Manipulate in a glove box if possible and syringe into a nitrogen blanketed solution. Wear latex gloves. The procedure is applicable for preparing a wide variety of secondary amides from aromatic amines.

The reactions should be run in an efficient fume hood. Small amounts of HCl evolve, which on larger scale can be trapped by addition of equimolar triethlyl amine to the mixture prior to acid chloride.

By entirely analogous reactions other amides were synthesized in high yield by this procedure. See primary and secondary references.

N-[4-(trans-4-Pentylcyclohexyl)phenyl]heptanamide, m.p. 144-45°.

N-[4-(trans-4-Peptylcyclohexyl)phenyl]butanamide, m.p. 142°.

Data:

m.p. 135-36°. I.r. (nujol mull) 3350, 2950, 1660, 1600 cm^{-1}.

Analysis: Calculated for $C_{26}H_{43}NO$:	C, 80.98	H,11.24	N,3.63
Found:	C, 81.20	H,11.42	N,3.43

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**

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

Chemspider deposition:

http://www.chemspider.com/Chemical-Structure.29354289.html

Keywords: carbamate, **amide, amine, aniline, acid chloride,**