Secondary aromatic amides from anilines:

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

John H. MacMillan (john.macmillan@temple.edu)
Department of Chemistry
Temple University, Philadelphia, Pa. 19122

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 temperature 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 triethyl 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. 144-45⁰.

N-[4-(trans-4-Peptylcyclohexyl)phenyl]butanamide, m.p. 142⁰.

**Data:**

m.p. 135-36⁰. I.r. (nujol mull) 3350, 2950, 1660, 1600 cm⁻¹.

Analysis: Calculated for C₂₆H₄₃NO:  C, 80.98  H,11.24  N,3.63  
   Found:  C, 81.20  H,11.42  N,3.43

**Lead reference:**


DOI: dx.doi.org/10.1080/00268947908069791

**Other references:**

DOI: Link: http://dx.doi.org/10.1080/01406567908071966


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

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