

Synthesis and Transformation of Mono - and Dicarboxylic Acids with Its Complimentary Anilides

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Abstract – The fascination of is thiocyanates as synthons and as cyclizing operators proceeds because of their diverse responses and because of their simple accessibility. It would not be strange to refer to that, in comparison to isocyanates (- N=C=O), their Sulfur analogs, is thiocyanates (- N=C=S), are less terrible and somewhat less hazardous. The utilization of isocyanates is radically restricted by the analysts [6] after December 3, 1984 which is the date of Bhopal Disaster held in Union carbide factory, Bhopal, Madhya Pradesh (India) because of the spillage of Methyl isocyanate (MIC) where a huge number of individuals were died because of the toxic impact of MIC (Me-N=C=O). In the present study, a blend of Phenyl is thiocyanate (2) and monocarboxylic acid (1), in the proportion of 1:1 and Phenyl is thiocyanate (2) and dicarboxylic acid (4) in the proportion of 2:1 are taken for buildup response by warming at 160°-170°C for 15 minutes under solvent free condition. Pyridine was utilized as an impetus/base in both the cases. The items acquired were monoanilides (3) and dianilides (5) of mono-and dicarboxylic acids individually, which were recrystallized from aqueous ethanol. Dicarboxylic acids gave startling outcomes in a portion of the cases. The response of a chloroformate or di-tert-butyl dicarbonate and sodium azide with an aromatic carboxylic acid delivers the corresponding acyl azide, apparently through the arrangement of an azidoformate. The acyl azide experiences a Curtius revision to shape an isocyanate derivative, which is caught either by an alkoxide or by an amine to frame the aromatic carbamate or urea. For instance, phthalic acid produces N-phenylphthalimide independent of the molar proportion of the acid and Phenyl is thiocyanate though maleic acid produces neither mono-nor dianilifisdes with Phenyl are thiocyanate under the current condition. An appropriate deliberate investigation was done towards the buildup of Phenyl is thiocyanate with mono-and dicarboxylic acids.

Keywords: Monocarboxylic Acids, Dicarboxylic Acids, Phenyl Is thiocyanate, Pyridine and Solvent Free

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1. INTRODUCTION

There are a few methods [1, 2, 10] accessible for the transformation of mono-and dicarboxylic acids into their N-substituted amides. With the end goal of accomplishing an advantageous and quick synthesis of N-phenyl alkanamides, the responses of a few mono-and dicarboxylic acids with phenyl is thiocyanate have been studied. We have shown that the utilization of different sorts of monocarboxylic and dicarboxylic acids changes OF in isolated RBCs in rats in vitro. Some monocarboxylic acids, having straight hydrocarbon chains of in excess of 4 carbon atoms long, increment OF in both a portion ward and carbon molecule number-subordinate way. Benzene-monocarboxylic acid (Benzoic acid) and a portion of its derivatives additionally increment OF in rodent RBCs. Then again, some dicarboxylic acids, including benzene dicarboxylic acids, abatement OF in rodent RBCs. A progression of investigations indicated that monocarboxylic and dicarboxylic acids directly affect the cell film, especially the

phospholipid layer, and change its resistance to osmotic pressure in rodent erythrocytes.

The condensation reaction was done by heating at 160°-170°C for 15 minutes, a blend of Phenyl is thiocyanate (2) and monocarboxylic acid (1), with the ratio of 1:1 and Phenyl is thiocyanate (2) and dicarboxylic acid (4) in the ratio of 2:1. Pyridine was utilized as a catalyst in both the cases. The items got were monoanilides (3) and dianilides (5) which were recrystallized from aqueous ethanol.

2. EXPERIMENTAL SETUP

All the prepared compounds are known in the writing. TLC (silica gel/benzene and ethyl acetate with ratio 9:1) and their melting focuses checked the virtue of the compounds. Melting point was recorded by metal block melting point apparatus and is uncorrected. The IR spectra of the compounds were

recorded on Nicolet is 5 Thermos Fisher Scientific, USA.

2.1 Procedure

✓ Synthesis of Anilides of Monocarboxylic Acids (3)

Phenyl is thiocyanate (2) and the monocarboxylic acid (1), taken in the ratio of 1:1 within the sight of pyridine as a catalyst, were warmed at 160°-170°C for around 15 minutes with consistent blending. The reaction blend was altogether blended, cooled and the buildup repeatedly washed with petroleum ether (40-60o) to evacuate unreacted phenyl is thiocyanate. The unreacted carboxylic acid was evacuated by treatment with aqueous sodium bicarbonate pursued by washing with distilled water. The separate monoanilide (3) was isolated and the rough item was recrystallized from aqueous ethanol. The applicable information is given in Table-1.

✓ Synthesis of Dianilides of Dicarboxylic Acids (5)

Phenyl is thiocyanate and the dicarboxylic acid taken in the ratio of 2:1 separately within the sight of pyridine as a catalyst, were warmed at 160°-170°C for around 15 minutes with consistent mixing. The reaction blend was completely blended, cooled and the residue more than once washed with petroleum ether (40-60o) to evacuate unreacted phenyl is thiocyanate. The blend was then consequently treated with benzene to expel anhydride, assuming any. The unreacted carboxylic acid was evacuated by treatment with aqueous NaHCO₃, trailed by washing with distilled water. The individual dianilide was isolated and the crude item was recrystallized from aqueous ethanol. The relevant data are given in Table-2.

3. RESULTS AND DISCUSSION

The condensation of carboxylic acids with is thiocyanates continued through addition-elimination reactions to yield amides. Phenyl is thiocyanate (2) gave mono- and dianilides with mono- and dicarboxylic acids individually. Pyridine as a catalyst/base facilitated the reaction. Obviously, this is inferable from the way that pyridine as a base upgrade the concentration of the carboxylate particle which is the reactive species towards phenyl is thiocyanate.

Monoanilides are shaped by condensation of monocarboxylic acid with aniline which can not arise by the decomposition of the phenyl is thiocyanate by water as phenyl is thiocyanate does not react with water. To rationalize the monoanilide arrangement by the interaction monocarboxylic acids with phenyl is thiocyanate within the sight of pyridine at 160°-170°C, the accompanying mechanism is proposed.

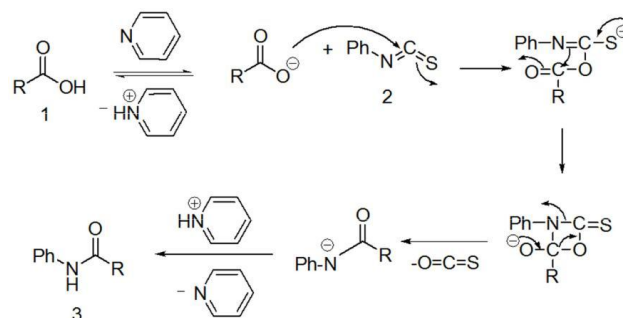


Figure 1. Synthesis of anilides of monocarboxylic acids.

Table 1. anilides of monocarboxylic acids.

Compound 3	R	Yield (%)	Melting Point (°C)	
			Found	Reported ^{3, 4, 5}
(a)	CH ₃	80	110-112	114
(b)	Ph-CH ₂	75	114-116	118
(c)	Ph	68	158-160	162
(d)	2-CH ₃ , C ₆ H ₄	57	121-123	125
(e)	2-OH, C ₆ H ₄	42	136-138	135
(f)	4-NO ₂ , C ₆ H ₄	71	212-214	211
(g)	PhCH=CH	80	150-152	153

Table 2. dianilides of dicarboxylic acids

Product	-(CH ₂) _n -	Yield (%)	Melting Point (°C)	
			Found	3, 4 Reported
5	(a) n = 0	43	146-148	148 (monoanilide)
5	(b) n = 2	50	223-225 (withoutpy)	225-227
5	(c) n = 4	74	238-240	239
5	(d)	63	>300	337

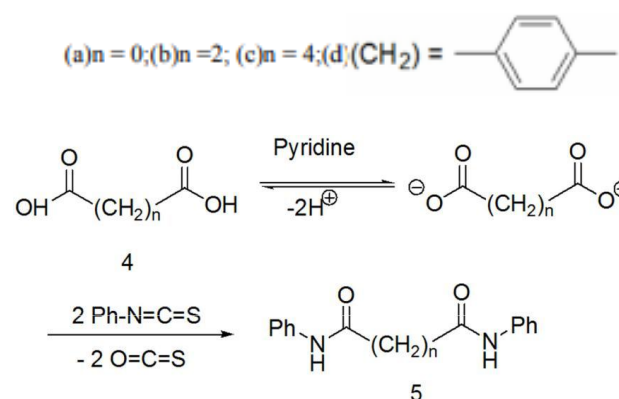


Figure 2. Synthesis of dianilides of dicarboxylic acids.

Dicarboxylic acids manage the cost of intriguing outcomes with phenyl is thiocyanate. All the dicarboxylic acids (4) bear the cost of the corresponding dianilides regardless of the molar ratio of the reactants. In any case, phthalic acid structures N-phenylphthalimide (Yield: 60%, m.p.200-202°C) where phenyl is thiocyanate goes about as a cyclizing specialist. The yields of the anilides increment when two moles of the phenyl is thiocyanate are utilized for one mole of dicarboxylic acid. It is seen that the nearness and nonattendance of pyridine assumes a significant role amid the

preparation of anilides with dicarboxylic acids. The reaction of phenyl is thiocyanate and succinic acid (5b) in 2:1 molar ratio without pyridine at 160°C-170°C manages the dianilide of succinic acid (Yield; half, m.p. 223-225°C) while addition of pyridine under comparative conditions furnishes a blend of mono- and dianilides of succinic acid which couldn't be isolated by partial crystallization. The reaction of phenyl is thiocyanate and oxalic acid dry out (5a) in 2:1 molar ratio either in the nearness or nonattendance of pyridine bears oxalic acid (monoanilide of oxalic acid) just (Yield: half, m.p. 146-148°C) and this technique neglects to create dianilide of oxalic acid. The arrangement of mono- and dianilides is distinguished by TLC (Silica gel/Benzene: Ethylacetate = 9: 1). Nevertheless, maleic acid neglects to give expected any sort of anilides by utilizing this technique. A strong profound darker mass is created that might be a polymeric substance because of the nearness of carbon-carbon twofold bond in the parent acid, which could not be dissected. Adipic acid dianilide (Yield: 74%, m.p. 248-250°C), Terephthalic acid dianilide (Yield: 63%, m.p. 337°C) were acquired conveniently by this strategy utilizing adipic acid (5c) and terephthalic acid (5d) individually with phenyl is thiocyanate with the ratio of 1:2.

All the compounds, reported [5, 7, 8, 9] were characterized by IR data and by comparison with reported melting point. The characteristic IR for anilides is observed at $\lambda_{\max}(\text{KBr})$: 3200-3300(NH), 1660-1700(CO, amide) cm^{-1} .

The direct conversion of mono- and dicarboxylic acids into their mono- and dianilides separately by utilizing phenyl is thiocyanate under solvent free condition is by all accounts profitable; it defeats a portion of the disadvantages of different methods. For instance, a substantial abundance of a portion of the reactants can be evaded and the reaction time is extremely short. Additionally, workup for the isolation of anilides is simple. The present strategy is even effective with phenolic acids like salicylic acids where free - OH group remains intact in anilide derivative [$\lambda_{\max}(\text{KBr})$: 3568(free OH)].

4. CONCLUSION

A straight forward and a quick technique for the synthesis of monoanilides and dianilides of mono- and dicarboxylic acids were created under solvent free conditions. It is noteworthy that the reaction is effective even because of phenolic acids, for example, salicylic acid, and the item can be isolated in an unadulterated state in the greater part of the cases. In perspective on the simplicity and speed of the reaction, the technique can be regarded as conceivably significant. We cleared up that OF reaction to monocarboxylic and dicarboxylic acids differs among rodent and guinea pig RBCs. He natural exercises of different carboxylic acids thought

to be needy on their chemical structure as well as on the characteristics of the lipid layer. He phospholipid organization of the cell membrane was accounted for to shift between tissues inside similar species and between similar tissues among different species. We, in this manner, need to elucidate whether the wonder saw in this study can be extrapolated to the erythrocytes of species other than rats and guinea pigs.

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