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Synthesis and characterization of two novel compounds: 2-Methyl-N-prop-2-ynylbenzamide and N-prop-2-ynylbenzamide

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Abstract: *This study presents the synthesis and characterization of 2-Methyl-N-prop-2-ynylbenzamide and N-prop-2-ynylbenzamide by Schotten-Baumann method. These compounds were synthesized by treating propargylamine with acyl chloride in the presence of potassium carbonate as the sacrificial base, a yield of 61% and 48% respectively were obtained. The products were characterized with 1-H and 13-CNMR, IR and mass spectral.*

Keywords: *acyl chloride, carboxamides, N-prop-2-ynylbenzamide, propargylamine, 2-Methyl-N-prop-2-ynylbenzamide.*

I. Introduction

The amide bond plays a major role in the elaboration and composition of biological systems, representing for example the main chemical bond that links amino acid building blocks together to give proteins. Amide bonds are not limited to biological systems, they are indeed present in a huge array of molecules, including the major marketed drugs [1,2]. The prevalence of the amide bond, particularly in peptides and proteins, sometimes gives the incorrect impression that there are no remaining synthetic challenges. This is surprising, as it is often the case that even simple amides resist formation, forcing chemists to resort to the ever more exotic and expensive reagents for their syntheses. In living systems, most amide bonds are formed by the complex factories of ribosomes. Synthetic chemists, by contrast, do not have the luxury of working with this single-molecule but, and instead deal with trillions of molecules that must be coaxed into precise reaction trajectories. This strategy necessitates that nearly every functional group be protected by a bulky hydrophobic appendage, leading to a reliable, but rather wasteful approach as in peptide synthesis, in which dozens of molecules are sacrificed to form just one amide bond. Improved methods for the synthesis of amide functionality, whether catalytic and waste-free or chemoselective are suitable for fragment coupling, are in great demand. [3,4,5].

II. Materials And Method

A solution of the appropriate amine in tetrahydrofuran was added to aqueous potassium carbonate in a 1litre three-necked round bottomed flask. The flask was immersed in an ice bath on a magnetic stirrer, equipped with a magnetic bar, an air condenser (carrying a guard tube with $CaCl_2$), an addition funnel with a stopper and a thermometer immersed in the solution. A solution of acid chloride 0.03M was added dropwise over 50 minutes. The mixture was allowed to stir overnight and warm to ambient temperature. The mixture was transferred to a separatory funnel and extracted with Chloroform. The extracts were combined, and washed with water, 10% aqueous HCl, saturated aqueous $NaHCO_3$, and water successively. The washed extract was dried over anhydrous magnesium sulphate and the solvent was removed under vacuum. The resulting residue was purified by recrystallization from methanol.

III. Results And Discussion

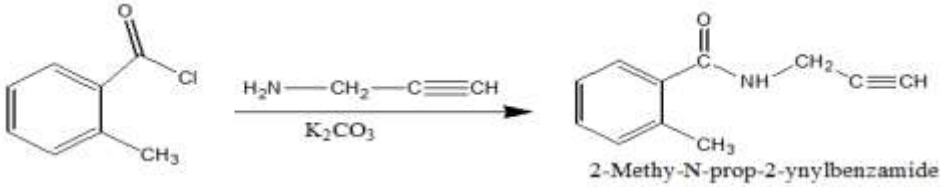
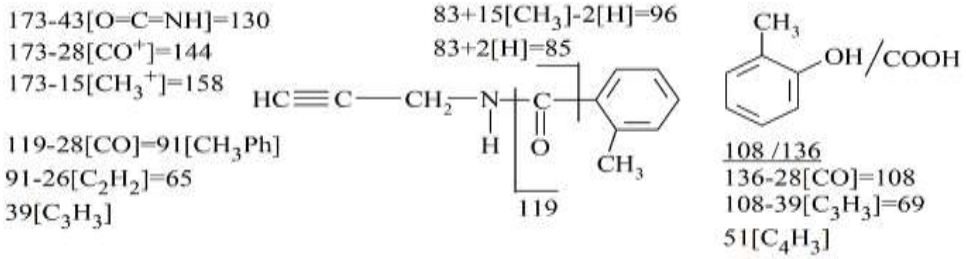


Figure 1. Synthesis of 2-methyl-N-prop-2-ynylbenzamide

Molecular Formular	$C_{11}H_{11}NO$ (FW = 173.21)
Yield (%)	61%
m.p. (^oC)	53-57

UV (λ_{max}) Dioxan soln. 205.2nm
IR (V_{max}) 3472, 2958, 2916, 2874, 2779, 2737, 2358, 1978, 1958, 1758, 1737, 1453, 1379, 1289, 1263, 1178, 1136, 1119, 1087, 1060, 926, 894, 862, 679cm^{-1}
^{1}H nmr ($CDCl_3$), δ 2.19(1HC≡, t, unconjugated), 2.2(3ArCH_3, s), 3.99(2H, t) 7.1(2ArH, q), 7.15(NH), 7.22(2ArH, q).
^{13}C nmr ($CDCl_3$), δ 20, 28, 72, 80, 126, 128, 130, 131, 136, 137, 170

m/z	[M]+/[M+1] M-15/28/43	CH3PhCO+/- [CO/C2H2/C3H3]	CH3PhCO2H/- [CO/C3H3/C4H3]	H2COCNCH2CCH HOCNHCH2CHCH2
EI (20eV) (% Int. Base)	173/158/144/130 (53.6/26.9/23.4/19.)	119/91 (100/52.2)	136/69/51 (13.3/1.8/)	96/85 (8.9/1.1)
EI (70eV) (% Int. Base)	173/158/144/130 (30.8/12.1/11.7/8.7)	119/91/65/39 (100/87.7/23/8)	136/69/51 (6.0/4.1/4.3)	85 (1.1)
CI – NH3 MS (% Int. Base)	174/158/144/130 (100/6.7/4.7/5.4)	119/91/65/39 (35.1/21.6/3.37)	136/108 (6.7/2.7)	96 (2.0)



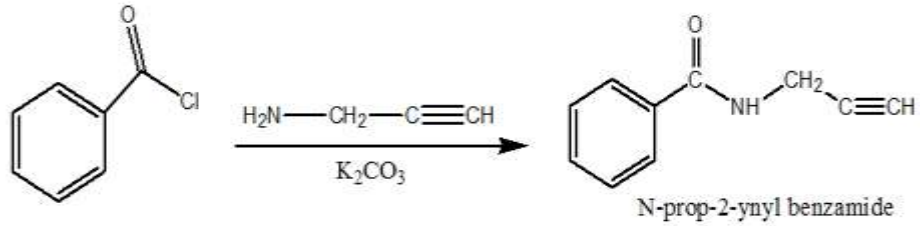
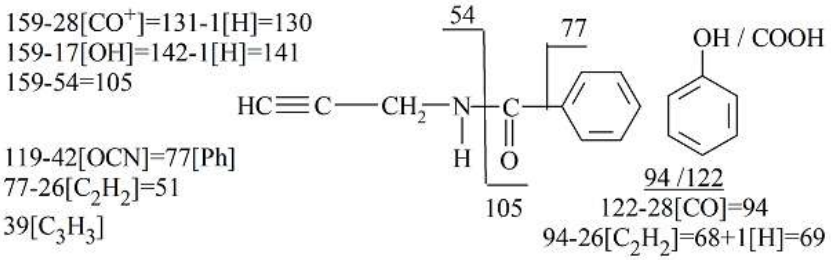


Figure 2. Synthesis of N-prop-2-ynyl benzamide

Molecular Formular $C_{10}H_9NO$ (FW = 159.19)
Yield (%) 49%
m.p. (^{o}C) 106-109
UV (λ_{max}) Dioxan soln. 204.6, 228.2nm
IR (V_{max}) 3489, 2916, 2847, 2776, 2706, 2325, 1963, 1915, 1757, 1721, 1442, 1394, 1357, 1321, 1260, 1224, 1175, 1078, 909, 679cm^{-1}
^{1}H nmr ($CDCl_3$), δ Not Determined
^{13}C nmr ($CDCl_3$), δ Not Determined

m/z	[M]+ /[M+1]+/M-17[OH]/28[CO]	PhOCN/ PhCO-[CO/C3H3]	HCCCH2NH HCCCH2	PhCOOH [CO/C2H2]
EI (20eV) (% Int. Base)	159/141/130 (55.5/9.6/70.1	119/105/77 (2.2/100/44)	54 (1.4)	69 (2.1)
EI (70eV) (% Int. Base)	159/141/130 (38/3.2/34.9)	105/77/51 (100/89.3/24.3)	39 (5.5)	69 (2.1)
CI – NH3 MS (% Int. Base)	160/130 (100/14)	105/77 (37.5/12.9)	39 (2.0)	122/94 (2.2/4.5)



IV. Conclusion

In this study, our interest was the synthesis and characterization of two carboxamides, 2-Methyl-N-prop-2-ynylbenzamide and N-prop-2-ynylbenzamide. The synthesis of these compounds in the presence of potassium carbonate as a sacrificial base, gave the yields of 61% and 49% respectively. The pharmacological activities of both carboxamides remain to be explored.

Acknowledgements

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