

A new methodology for the synthesis of *N*-acylbenzotriazoles

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Dedicated to the late Prof. Alan R Katritzky for his excellent contributions to benzotriazole chemistry

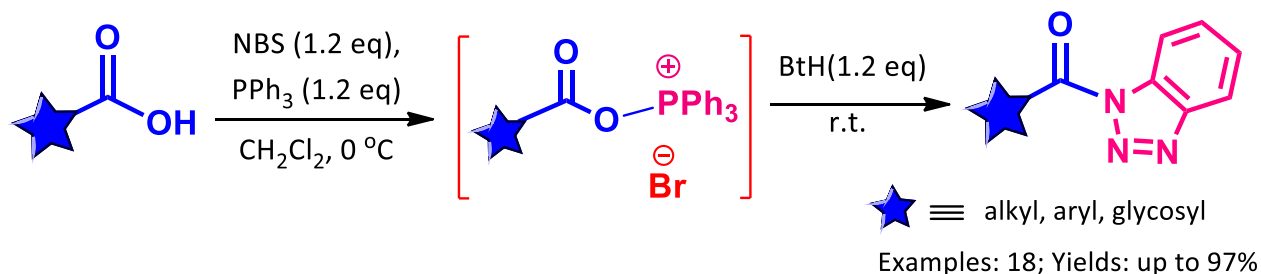
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Abstract

A facile and economic path for an easy access of diverse *N*-acylbenzotriazoles from carboxylic acid has been devised using NBS/PPh₃ in anhydrous dichloromethane. High yield of product was obtained at room temperature in one hour reaction time under mild reaction conditions.



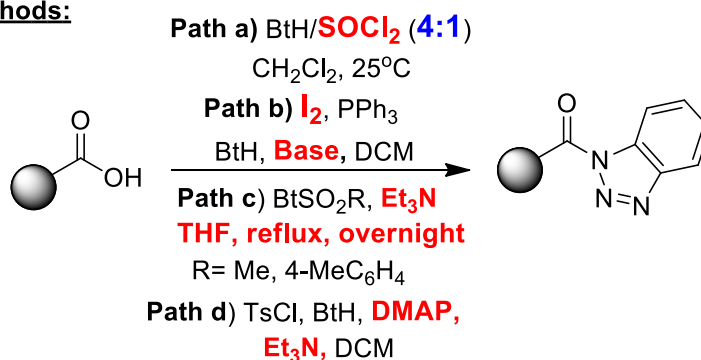
Keywords: *N*-acylbenzotriazole, benzotriazole, *N*-halosuccinimide, triphenylphosphine

Introduction

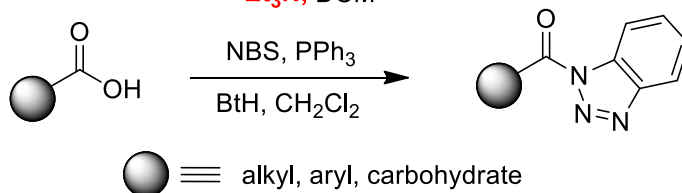
The interesting features of benzotriazole – its high solubility in organic solvents, stability in acidic as well as basic media, considerable compatibility with various organic reactions and easy introduction under standard reaction conditions, activation of numerous useful transformations and final elimination after the completion of a reaction sequence – make this moiety a favorable auxiliary for the synthesis of a wide range of compounds of great pharmacological and other interests.¹⁻⁹ *N*-acylbenzotriazoles are one of the most useful benzotriazole-derivatives which have been successfully utilized to prepare numerous biologically relevant compounds under neutral and mild conditions by *N*-, *C*-, *S*-, and *O*-acylations.¹⁰⁻²⁴ In recent years our group has been involved in exploration of the synthetic utility of *N*-acylbenzotriazoles to make sugar amides by *N*-acylation,²⁵ benzoxazoles and *N*-phenylamides by benzotriazole ring cleavage^{26,27} and ureas, carbamates and thiocarbamates by the Curtius rearrangement.²⁸

The earlier synthetic methods for conversion of carboxylic acids to *N*-acyl benzotriazole i.e. **path a, b, c** and **d** (Scheme 1) comprise some drawbacks such as, bulk amount of BtH with toxic thionyl chloride is required in Path a, in Path b iodine is used which is highly toxic with base, in Path c requires much longer time to complete the reaction, where as in path d activation of carboxylic acids with tosyl chloride was utilized to afford good yields of *N*-acyl benzotriazoles under one-pot condition using a base.^{11,29-31}

Previous methods:



This Work:

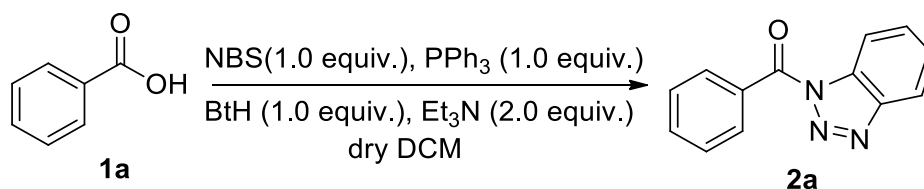


Scheme 1. Comparative illustration of previous and present work.

Therefore, an improved method is considered necessary to make this transformation more facile, economical and of low toxicity. As we know that the reaction of an alcohol with BtH in the presence of NBS/PPh₃ gives an alkylbenzotriazole, this idea can be applied to modify path b by using NBS in place of iodine. This leads to the introduction of a less toxic and cost-effective path to prepare *N*-acylbenzotriazoles, which we report in this manuscript.

Results and Discussion

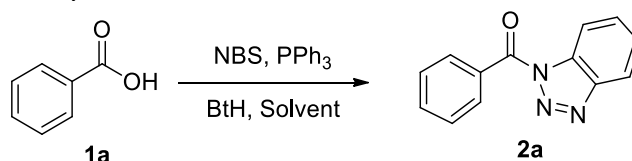
A prototype reaction for the synthesis of *N*-acylbenzotriazole **2a** was initiated with 1 equiv. PPh₃ and 1 equiv. of NBS in dry dichloromethane. After a few minutes, 1.0 equiv. of benzoic acid was added and the mixture was stirred continuously at 0°C for 30 min followed by addition of 1*H*-benzotriazole and Et₃N at an interval of 10 minutes. The mixture was stirred for 1hr to afford *N*-benzoylbenzotriazole in 71% yield (Scheme 2).



Scheme 2. Prototype reaction for synthesis of *N*-acylbenzotriazoles.

To optimize the reaction conditions for the better yield of resultant product, first of all we employed different types of *N*-halosuccinimide with 1.0 equiv. of PPh₃ and 2.0 equiv. of triethylamine in dry dichloromethane (entries 1-3) and found best results with *N*-bromosuccinimide. After establishing the reagent of choice, we started varying the molar ratios of *N*-bromosuccinimide, PPh₃ and triethylamine in dry dichloromethane (entries 3-8). It was found that reaction proceeds very smoothly even in the absence of base when we take 1.2 equiv. of both *N*-bromosuccinimide and PPh₃ in dry dichloromethane at 0°C. Optimization results for solvent revealed that dry dichloromethane is the most suitable solvent for the reaction (entries 8-12, Table 1).

Table 1. Reaction optimization study



Entry ^a	Reagent (equiv)	PPh ₃ (equiv.)	Base (equiv.)	Solvent ^b	Yield (%) ^c
1	NCS (1.2)	1.2	Et ₃ N (2.0)	DCM	59
2	NIS (1.2)	1.2	Et ₃ N (2.0)	DCM	53
3	NBS (1.0)	1.0	Et ₃ N (2.0)	DCM	71
4	NBS (.5)	0.5	Et ₃ N (1.0)	DCM	26
5	NBS (1.2)	1.2	Et ₃ N (2.0)	DCM	87
6	NBS (2.0)	2.0	Et ₃ N (4.0)	DCM	86
7	NBS (1.2)	1.2	Et ₃ N (1.0)	DCM	87
8	NBS (1.2)	1.2	Et ₃ N (0.0)	DCM	89
9	NBS (1.2)	1.2	Et ₃ N (0.0)	Toluene	52
10	NBS (1.2)	1.2	Et ₃ N (0.0)	Chloroform	57
11	NBS (1.2)	1.2	Et ₃ N (0.0)	THF	49
12	NBS (1.2)	1.2	Et ₃ N (0.0)	DMF	22

^a Molar ratio: carboxylic acid (1.0 mmol). ^b dry solvents. ^c Yields reported after purification by column chromatography (SiO₂).

After optimization of the reaction conditions, we attempted to generalize the reaction by applying it to different class of carboxylic acids including aryl, substituted aryl, fused aryl, aliphatic and glycosylated carboxylic acids where in all cases the reaction was found to be smooth and the corresponding *N*-acylbenzotriazole products were obtained in good to excellent yields. It was observed that variation of functional groups around the aromatic ring of the acid slightly affected the yield of product and a small decrease in yield was obtained with electron withdrawing groups. In the case of aliphatic acids, variation in length of carbon chain did not show any notable change in product yield. This reaction protocol also goes well with sugar acids and gives excellent product yields. The crude reaction product was purified by flash column chromatography (SiO₂) using a gradient of ethyl acetate/*n*-hexane, and the corresponding *N*-acylbenzotriazoles **2a-o** were obtained in good yield (Figure 1). All the synthesized compounds have been characterized by standard spectroscopic techniques including mass, IR, ¹H and ¹³C NMR studies.

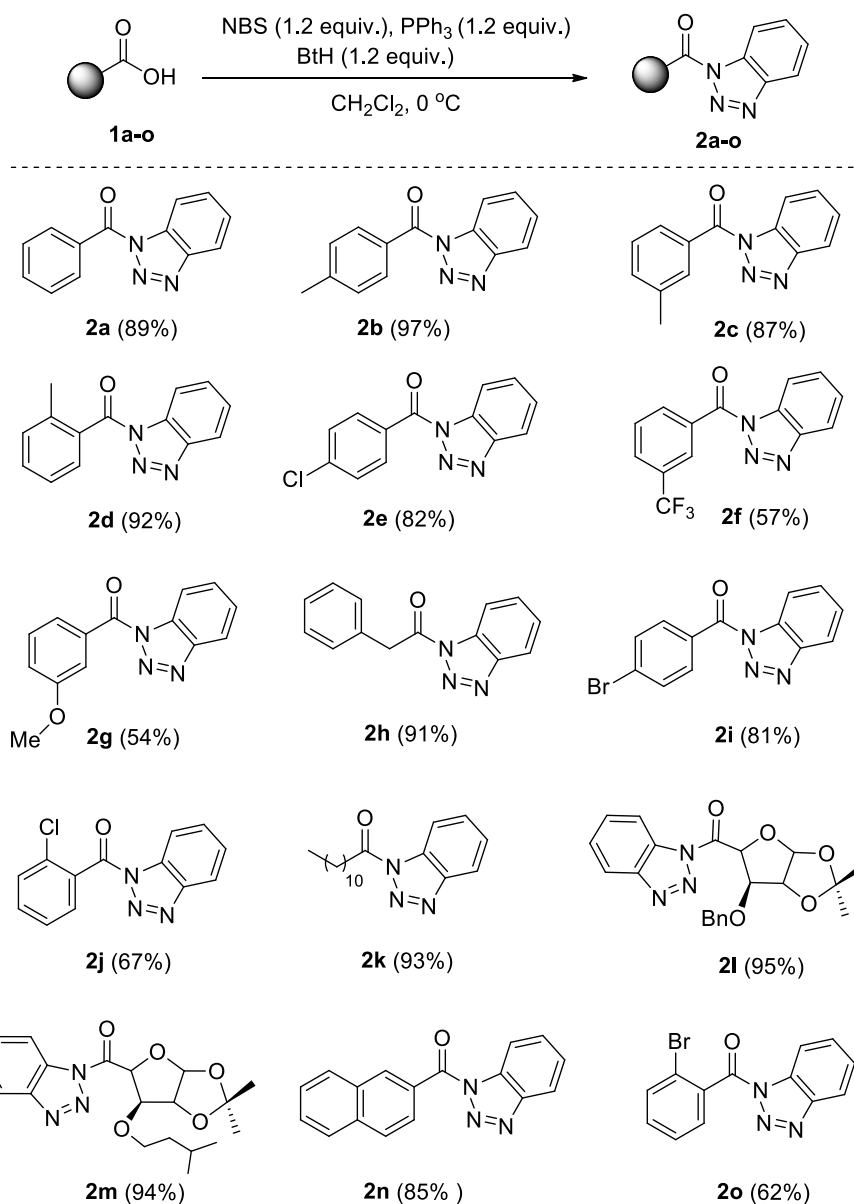
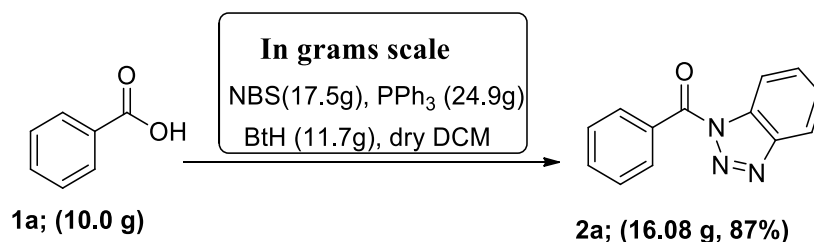


Figure 1. Synthesis of *N*-acylbenzotriazoles **2a-o** from acids using NBS/PPh₃. Molar ratios: carboxylic acids (**1a-o**) (1.0 equiv.), NBS (1.2 equiv), PPh₃ (1.2 equiv), benzotriazole (1.2 equiv). Yields reported after purification by column chromatography (SiO₂).

Synthesis of *N*-Acylbenzotriazole: reaction in gram scale

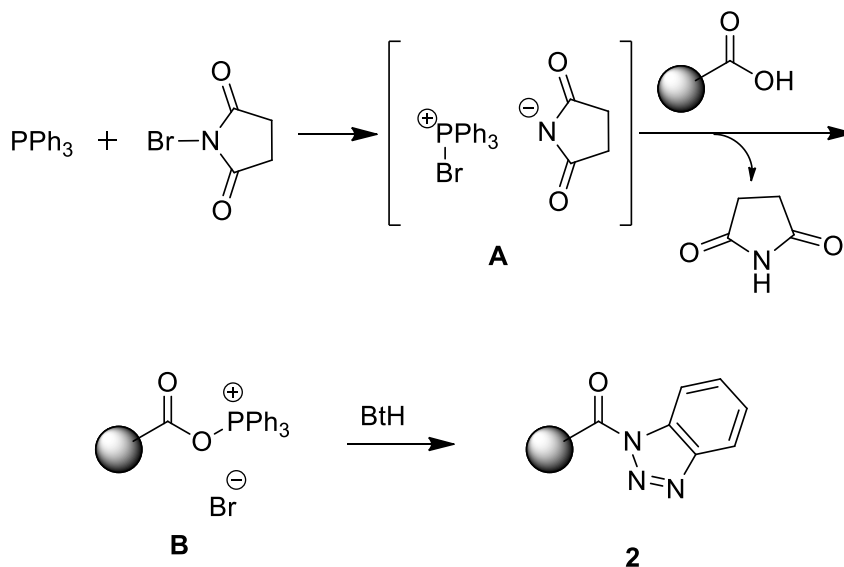
A scale-up reaction was carried out for quantitative generalization of the method starting with 10 g of benzoic acid. The reaction was found to go well in gram scale also and produced *N*-Benzoyl benzotriazole **2a** in 87% yield. This suggested that the method is equally applicable to milligram and gram scales and yield is not much affected by quantitative scale of reaction.



Scheme 3. Synthesis of *N*-acylbenzotriazole **2a** reaction in gram scale.

Mechanism

The reaction possibly begins with the formation of intermediate (**A**) generated by the reaction of PPh₃ and NBS.³² The intermediate (**A**) reacts with carboxylic acid to form acyloxyphosphonium ion³³ with the loss of succinimide. This acyloxyphosphonium ion (**B**) further reacts with BtH to form the final product *N*-acylbenzotriazole **2**.



Scheme 4. Possible mechanism for synthesis of *N*-acylbenzotriazole **2**.

Conclusions

In summary, we have developed a low-toxic, economical and efficient method for synthesis of *N*-acylbenzotriazole from carboxylic acid. The developed synthetic path was successfully employed for a variety of substrates and furnished good yields at 0 °C with simple purification methods. The method was found equally facile for milligram to gram scales.

Experimental Section

General. All reagents and solvents were of pure analytical grade. Thin-layer chromatography (TLC) was performed on 60 F254 silica gel, pre-coated on aluminium plates and revealed with either a UV lamp ($\lambda_{\text{max}} = 254 \text{ nm}$) or a specific colour reagent (Dragendorff's reagent or iodine vapour) or by spraying with methanolic H_2SO_4 solution and subsequent charring by heating at $100 \text{ }^\circ\text{C}$ (for a carbohydrate derivative only). Solvents were evaporated under reduced pressure at temperature $< 50 \text{ }^\circ\text{C}$. Column chromatography was carried out on silica gel (230-400 mesh, Merck). Distilled *n*-hexane and ethyl acetate were used for the column chromatography. ^1H and ^{13}C NMR were recorded at 500 and 125 MHz, respectively. Chemical shifts given in ppm downfield from internal TMS; *J* values in Hz. Infrared spectra recorded as Nujol mulls in KBr pellets.

Typical experimental procedure for synthesis of *N*-acylbenzotriazoles

Compound **1a** (1.0 g, 8.19 mmol) was added to a RB flask containing stirring solution of NBS (1.7g, 9.83 mmol) and PPh_3 (2.5g, 9.83 mmol) in Dry dichloromethane (30.0 mL) and temperature was maintained at $0 \text{ }^\circ\text{C}$. *1H*-Benzotriazole (1.2g, 9.83 mmol) was added portion-wise. After complete addition, the reaction mixture was stirred for 1 hour at room temperature. After completion of reaction (monitored by TLC), the reaction mass was concentrated under reduced pressure until dry. Purification using flash column chromatography using gradient mixtures of ethyl acetate and *n*-hexane afforded product **2a** (1.62g, 7.29 mmol) in pure form.

Physical data of developed compounds (2a-o)

(1*H*-1,2,3-Benzotriazol-1-yl)(phenyl)methanone (2a).²⁸ White crystalline solid, 1.62g, yield 89%; $R_f = 0.6$ (10% ethyl acetate/*n*-hexane); m.p. $110\text{-}114 \text{ }^\circ\text{C}$ (lit. m.p. $112 \text{ }^\circ\text{C}$); IR (KBr): ν_{max} 3109, 3069, 2925, 1712, 1599, 1488, 1451, 1379, 1379, 1047, 942, 838, 751 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.27 (d, $J = 8.5 \text{ Hz}$, 1H), 8.11 (d, $J = 7.5 \text{ Hz}$, 2H), 8.05 (d, $J = 8.5 \text{ Hz}$, 1H), 7.59-7.56 (m, 2H), 7.48-7.41 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.6, 145.6, 133.5, 132.2, 131.6, 131.3, 130.2, 128.3, 126.2, 120.0 and 114.7 ppm.

(1*H*-1,2,3-Benzotriazol-1-yl)(*p*-tolyl)methanone(2b).²⁸ white crystalline solid, 1.7g, yield 97%; $R_f = 0.5$ (5% ethyl acetate/*n*-hexane); m.p. $122\text{-}124 \text{ }^\circ\text{C}$ (lit. m.p. $123 \text{ }^\circ\text{C}$); ^1H NMR (500 MHz, CDCl_3): δ 8.27 (d, $J = 8.5\text{Hz}$, 1H), 8.11-8.09 (m, 3H), 7.63 (t, $J = 7.5\text{Hz}$, 1H), 7.48 (t, $J = 7.5\text{Hz}$, 1H), 7.32 (d, $J = 7.5\text{Hz}$, 2H), 2.43 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.5, 145.7, 144.8, 132.4, 132.0 (2C), 130.3, 129.2 (2C), 128.6, 126.2, 120.1, 114.8 and 21.8 ppm.

(1*H*-1,2,3-Benzotriazol-1-yl)(*m*-tolyl)methanone (2c).²⁸ White crystalline solid, 1.5g, yield 87%; $R_f = 0.6$ (5% ethyl acetate/*n*-hexane); m.p. $206\text{-}210 \text{ }^\circ\text{C}$ (lit. m.p. $205 \text{ }^\circ\text{C}$); ^1H NMR (500 MHz, CDCl_3): δ 8.37 (d, $J = 8.1\text{Hz}$, 1H), 8.35 (d, $J = 8.5\text{Hz}$, 1H), 8.13 (d, $J = 8.5\text{Hz}$, 1H), 8.99 (d, $J = 1.4\text{Hz}$, 1H), 7.66 (t, $J = 7.5\text{Hz}$, 1H), 7.52-7.41 (m, 3H), 2.44 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 166.9, 145.8, 138.3, 134.5, 132.4, 132.1, 131.5, 130.2, 129.0, 128.3, 126.3, 120.2, 114.8 and 21.4 ppm.

(1*H*-1,2,3-Benzotriazol-1-yl)(*o*-tolyl)methanone (2d).²⁸ White crystalline solid, 1.6g, yield 92%; $R_f = 0.5$ (5% ethyl acetate/*n*-hexane); m.p. $86\text{-}90^\circ\text{C}$; ^1H NMR (500 MHz, CDCl_3): δ 8.28 (d, $J = 8.5\text{Hz}$, 1H), 8.03 (d, $J = 8.5\text{Hz}$, 1H), 7.58 (t, $J = 7.5\text{Hz}$, 1H), 7.52 (d, $J = 7.5\text{Hz}$, 1H), 7.43-7.37 (m, 2H), 7.25-7.21(m, 2H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 168.1, 146.0, 137.8, 132.1, 131.7, 131.6, 130.9, 130.3, 129.9, 126.2, 125.3, 120.1, 114.4 and 19.9 ppm.

(1*H*-1,2,3-Benzotriazol-1-yl)(4-chlorophenyl)methanone(2e).²⁸ White crystalline solid, 1.3g, yield 82%; $R_f = 0.5$ (10% ethyl acetate/*n*-hexane); m.p. $136\text{-}140^\circ\text{C}$ (lit. m.p. $138 \text{ }^\circ\text{C}$); IR (KBr): ν_{max} 3118, 3094, 2925, 1707, 1591, 1481, 1450, 1380, 1047, 942, 838, 751 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3): δ 8.25 (d, $J = 7.5\text{Hz}$, 1H), 8.10-8.04 (m, 3H), 7.61-7.58 (m, 1H), 7.45-7.43 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 165.6, 145.7, 140.4, 133.2, 132.3, 130.6, 129.8, 128.9, 126.5, 120.3 and 114.8 ppm.

(1H-1,2,3-Benzotriazol-1-yl)(3-(trifluoromethyl) phenyl)methanone (2f).²⁷ White crystalline solid, 0.87g, yield 57%; $R_f = 0.7$ (10% ethyl acetate/n-hexane); m.p. 51-52°C; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 8.41 (s, 1H), 8.37 (d, $J = 7.5\text{Hz}$, 1H), 8.32 (d, $J = 8.5\text{Hz}$, 1H), 8.11 (d, $J = 8.5\text{Hz}$, 1H), 7.87 (d, $J = 7.5\text{Hz}$, 1H), 7.68-7.64 (m, 2H), 7.50 (t, $J = 7.5\text{Hz}$, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 165.4, 145.8, 134.9, 132.4, 132.2, 130.8, 130.2, 130.1, 129.1, 128.7, 128.6, 126.8, 120.4 and 114.8 ppm.

(1H-1,2,3-Benzotriazol-1-yl)(3-methoxyphenyl)methanone (2g).²⁷ White crystalline solid, 0.89g, yield 54%; $R_f = 0.7$ (10% ethyl acetate/n-hexane); m.p. 80-84°C; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 8.29 (d, $J = 8.5\text{Hz}$, 1H), 8.08 (d, $J = 8.5\text{Hz}$, 1H), 7.73 (d, $J = 8.0\text{Hz}$, 1H), 7.63-7.60 (m, 2H), 7.46 (t, $J = 7.5\text{Hz}$, 1H), 7.39 (t, $J = 7.5\text{Hz}$, 1H), 7.15-7.13 (m, 1H), 3.81 (s, 3H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 166.6, 159.5, 145.8, 132.6, 132.4, 130.4, 129.5, 126.4, 124.3, 120.3, 120.2, 116.2, 114.8 and 55.4 ppm.

1-(1H-1,2,3-Benzotriazol-1-yl)-2-phenylethanone (2h).²⁷ White solid, 1.6g, yield 91%; $R_f = 0.7$ (5% ethyl acetate/n-hexane); m.p. 67-68°C (lit. m.p. 65-66 °C); $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 8.23 (d, $J = 8.5\text{Hz}$, 1H), 8.09 (d, $J = 7.5\text{Hz}$, 1H), 7.59 (t, $J = 7.5\text{Hz}$, 1H), 7.46-7.44 (m, 3H), 7.36-7.28 (m, 3H), 4.70 (s, 2H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 170.3, 146.3, 132.6, 131.3, 130.5(2C), 129.9, 128.9, 127.7, 126.3, 120.2, 114.5 and 42.1 ppm.

(1H-1,2,3-Benzotriazol-1-yl)(4-bromophenyl)methanone. (2i).²⁸ Pale yellow solid, 1.2g, yield 81%; $R_f = 0.5$ (10% ethyl acetate/n-hexane); m.p. 138-140°C (lit. m.p. 142-143 °C); $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 8.27 (d, $J = 8.5\text{Hz}$, 1H), 8.07 (d, $J = 7.5\text{Hz}$, 1H), 8.03-8.01 (m, 2H), 7.63-7.59 (m, 3H), 7.45 (t, $J = 7.5\text{ Hz}$, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 165.8, 145.8, 133.3, 132.3, 131.9, 130.6, 130.2, 129.2, 126.5, 120.3 and 114.8 ppm.

(1H-1,2,3-Benzotriazol-1-yl)(2-chlorophenyl)methanone (2j).²⁸ White solid, 1.1g, yield 67%; $R_f = 0.5$ (10% ethyl acetate/n-hexane); m.p. 81-83°C; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 8.27 (d, $J = 8.5\text{Hz}$, 1H), 8.02 (d, $J = 7.5\text{Hz}$, 1H), 7.60-7.57 (m, 2H), 7.43-7.41 (m, 3H), 7.34-7.31 (m, 1H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 165.8, 146.1, 132.9, 132.7, 132.2, 131.2, 130.7, 130.3, 130.1, 126.8, 126.7, 120.3 and 114.3 ppm.

1-(1H-Benzo[d][1,2,3]triazol-1-yl)dodecan-1-one (2k).²⁷ White crystalline solid, 1.4g, yield 93%; $R_f = 0.6$ (5% ethyl acetate/n-hexane); m.p. = 46-47 °C; $^1\text{H NMR}$ (500 MHz, CDCl_3): δ 8.12 (d, $J = 8.5\text{Hz}$, 1H), 7.95 (d, $J = 8.5\text{Hz}$, 1H), 7.48 (t, $J = 8.5\text{Hz}$, 1H), 7.34 (t, $J = 7.5\text{Hz}$, 1H), 3.28 (t, $J = 6.5\text{Hz}$, 2H), 1.81-1.75 (m, 2H), 1.39-1.33 (m, 2H), 1.28-1.23 (m, 1H), 1.20-1.14 (m, 13H), .76 (t, $J = 6.5\text{Hz}$, 3H); $^{13}\text{C NMR}$ (125 MHz, CDCl_3): δ 172.5, 146.1, 131.0, 130.2, 125.9, 120.0, 114.4, 35.5, 31.9, 29.6, 29.5, 29.4, 29.3, 29.1, 24.4, 22.7 and 14.1 ppm.

(1H-1,2,3-Benzotriazol-1-yl)[6-(benzyloxy)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-5-yl]methanone (2l)²⁶. White solid, yield 95%; 1.19g, $R_f = 0.6$ (20% ethyl acetate/n-hexane); m.p. 158-160°C; $^1\text{H NMR}$ (300 MHz, CDCl_3): δ 8.29 (d, $J = 8.1\text{ Hz}$, 1H), 8.09 (d, $J = 7.8\text{ Hz}$, 1H), 7.68 (t, $J = 7.2\text{ Hz}$, 1H), 7.53 (t, $J = 7.5\text{ Hz}$, 1H), 6.89 (t, $J = 7.2\text{ Hz}$, 1H), 6.80 (t, $J = 7.5\text{ Hz}$, 2H), 6.71 (d, $J = 7.2\text{ Hz}$, 2H), 6.27 (d, $J = 3.0\text{ Hz}$, 1H), 6.06 (d, $J = 3.9\text{ Hz}$, 1H), 4.79 (d, $J = 3.9\text{ Hz}$, 1H), 4.73 (d, $J = 3.3\text{ Hz}$, 1H), 4.53 (d, $J = 11.7\text{ Hz}$, 1H), 4.17 (d, $J = 12.0\text{ Hz}$, 1H), 1.57 (s, 3H), 1.39 (s, 3H); $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ 165.7, 145.6, 135.8, 130.8, 130.5, 127.9, 127.6, 127.3, 126.2, 120.0, 114.1, 112.8, 105.9, 83.1, 82.3, 81.2, 71.8, 27.1 and 26.3 ppm.

(1H-1,2,3-Benzotriazol-1-yl)[6-(isopentyloxy)-2,2-dimethyltetrahydrofuro[2,3-d][1,3]dioxol-5-yl]methanone (2m). Oil, yield 94%; 1.2g, $R_f = 0.5$ (20% ethyl acetate/n-hexane); $^1\text{H NMR}$ (300 MHz, CDCl_3): δ 8.32 (d, $J = 8.1\text{ Hz}$, 1H), 8.15 (d, $J = 8.4\text{ Hz}$, 1H), 7.70 (t, $J = 7.5\text{ Hz}$, 1H), 7.55 (t, $J = 7.5\text{ Hz}$, 1H), 6.25 (s, 1H), 6.11 (d, $J = 4.2\text{ Hz}$, 1H), 4.71 (t, $J = 4.2\text{ Hz}$, 2H), 3.53 (t, $J = 4.5\text{ Hz}$, 1H), 3.08 (d, $J = 7.5\text{ Hz}$, 1H), 1.59 (s, 3H), 1.40 (s, 3H), 1.11-0.89 (m, 3H), 0.51 (d, $J = 6.0\text{ Hz}$, 3H), 0.21 (d, $J = 6.0\text{Hz}$, 3H) ; $^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ 165.9, 145.7, 131.0, 130.6, 126.4, 114.1, 112.8, 106.0, 84.7, 84.6, 82.3, 82.2, 81.5 (d), 68.6, 37.7, 27.2, 26.5, 24.0, 22.2 and 21.0 ppm.

(1H-1,2,3-Benzotriazol-1-yl)(naphthalen-2-yl)methanone (2n).²⁸ White solid, yield 85%; 1.1g, $R_f = 0.6$ (15% ethyl acetate/n-hexane); m.p. 118-120°C; $^1\text{H NMR}$ (300 MHz, CDCl_3): δ 8.73 (s, 1H), 8.31 (d, $J = 7.5\text{ Hz}$, 1H), 8.10-8.06 (m, 2H), 7.90-7.88 (m, 2H), 7.86-7.81(m, 1H), 7.79-7.59 (m, 1H), 7.57-7.53 (m, 1H), 7.51-7.42 (m, 1H); ^{13}C

NMR (125 MHz, CDCl₃): δ 166.7, 145.8, 135.7, 134.4, 132.5, 132.2, 130.4, 129.9, 129.2, 128.5, 128.3, 127.8, 127.1, 126.6, 126.4, 120.2 and 114.9 ppm.

(1H-1,2,3-Benzotriazol-1-yl)(2-Bromophenyl)methanone (2o).²⁸ Solid, R_f = 0.7 (5% ethyl acetate/n-hexane); ¹H NMR (300 MHz, CDCl₃): δ 8.28(d, J = 8.0 Hz, 1H), 8.03 (d, J = 8.5 Hz, 1H), 7.61-7.47 (m, 3H), 7.43- 7.32 (m, 3H); ¹³C NMR (75 MHz, CDCl₃): δ 166.5, 146.3, 135.1, 133.3, 132.6, 131.3, 130.8, 130.2, 127.3, 126.8, 120.6, 120.4 and 114.5 ppm.

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Supplementary Material

Characterization data (for all the developed acyl benzotriazoles) including copies of ¹H and ¹³C NMR spectra associated with this paper can be found in the online version.

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