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Synthesis of 1-(2-(4-Substitutedphenylamino)- Imidazo [2, 1-B] Benzothiazole-3-Yl) Propan-1- One

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Strictly as per the compliance and regulations of :

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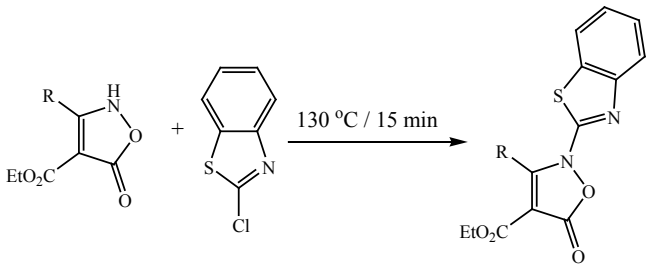
Chalak Azimi [α], Helal Tahazadeh [σ] & Rasol Kamari [ρ]

Abstract- 4-propionyl-3-(4-substitutedphenylamino) isoxazol-5(2H)-one, substituted on nitrogen with a 2-chlorobenzothiazole group, reacts with triethylamine (TEA) in ethanol under reflux conditions to provide a convenient synthesis of 1-(2-(4-substitutedphenyl amino) -imidazo [2,1-b] benzothiazole-3-yl) propan-1-one.

Keywords: isoxazolones; 2-chlorobenzothiazole; imidazobenzothiazoles; triethylamine (tea).

I. Introduction

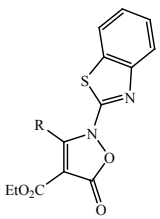
The synthesis of isoxazol-5(2H)-one with benzothiazole substituted on nitrogen **1a** has been reported by Prager and co-workers[1] as shown in Scheme I.



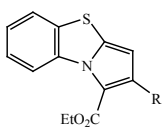
1a R=H

Scheme I

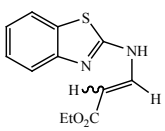
It has been reported[2] that the 2-benzothiazol-2-yl isoxazolones **1a** and **2b** gave the corresponding imidazobenzothiazoles **2a** and **2b** respectively on photolysis in ethyl acetate/ trifluoroacetic acid, and the acrylate **3** was obtained from the photolysis of **1a** in methanol.



1b R=Me

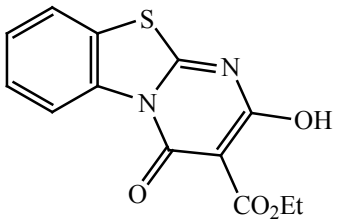


2a R=H
2b R=Me



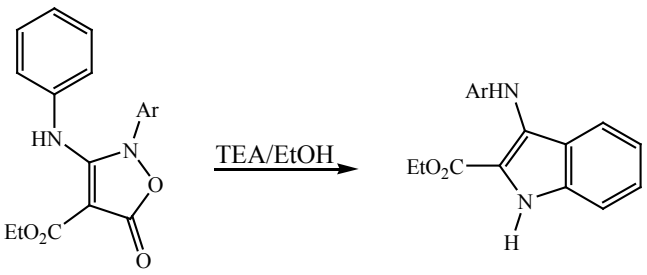
3

Base-catalysed rearrangement of isoxazolinyl heterocycle **1a** using a solution of sublimed potassium t-butoxide in dry tetrahydrofuran at 40 °C gave ethyl 2-hydroxy-4-oxo-4H-pyrimido [2, 1-b] benzothiazole-3-carboxylate **4**.[3]



4

Khalafy et al. recently reported[4] that the reaction of certain 2-aryl-3-arylaminoisoxazolones **5** with triethylamine (TEA) leads to the formation of indoles **6** and carbon dioxide, an outcome that is formally the same as that achieved by photolysis or pyrolysis[5] (Scheme II).



5 6

Scheme II

We have recently reported[6] rearrangement of 4-acetyl-3-(4-substituted phenylamino)-2- (5-nitropyridin-2-yl) isoxazol-5(2H)-ones (**7**, X: Br, Me, OMe) to Imidazo [1, 2-a] pyridines (**8**, X: Br, Me, OMe) under Flash-Vacuum-Pyrolysis (F.V.P) conditions (Scheme III).

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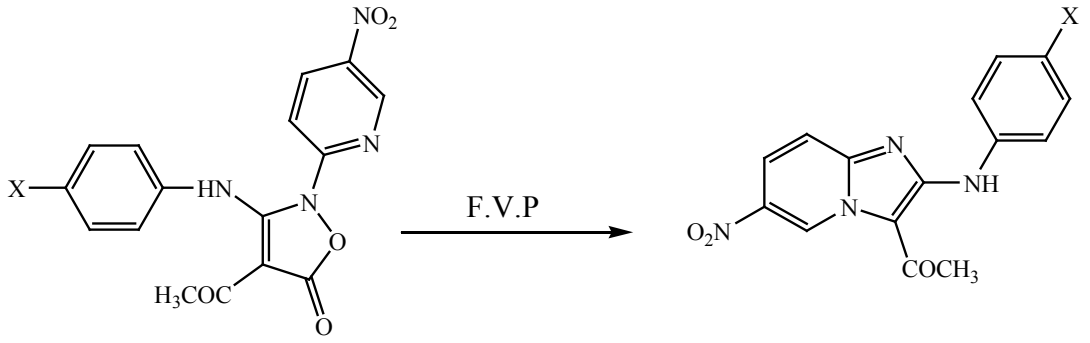
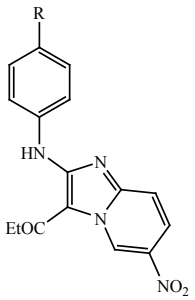


7, X: Br, Me, OMe

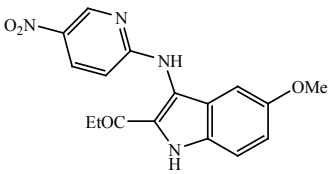
8, X: Br (90%), Me (93%), OMe (95%)

Scheme III

We have also reported[7] that 4-propionyl-3-(4-substituted phenylamino) isoxazole-5 (2H) -one, substituted on nitrogen with a nitropyridine group, react with triethylamine (TEA) to give imidazo [1,2- a]pyridines and indoles. With 4-bromophenyl and 4-methylphenyl group substituents only imidazopyridines **9a-b** are formed, but the 4-methoxyphenyl derivative gave a 3: 1 mixture of corresponding imidazo [2,1-a]pyridine **9c** and 2-pyridylaminoindole **10**, respectively.



9a R=Br
9b R=Me
9c R=OMe

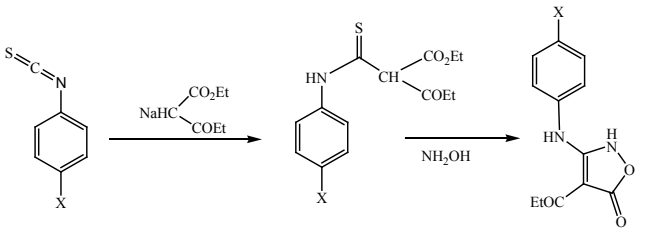


10

Here we describe the synthesis of new N-substituted derivatives of P-substituted 4-(phenylamino) isoxazol-5(2H)-ones **13** with a 2-chlorobenzothiazole group substituted on N-2 **14**, and their rearrangement in the presence of triethylamine to produce 1-(2-(4-substitutedphenylamino)-imidazo [2,1-b] benzothiazole-3-yl) propan-1-one **15**, as shown in (Scheme V).

II. Results and Discussion

The required isoxazolones **14** were synthesized by reaction of 2-chlorobenzothiazole with 2H-isoxazolones **13**, which in turn were made by a modification of the procedure of Worrall.[8,9] Thus, the reaction of the sodium salt of ethyl-3-oxopentanoate in ethanol with 4-phenylisothiocyanates **11** gave the thiocarbamates **12** in high yield, and these were converted to the corresponding isoxazolone **13** by reaction with 3 equiv of hydroxylamine (Scheme IV).

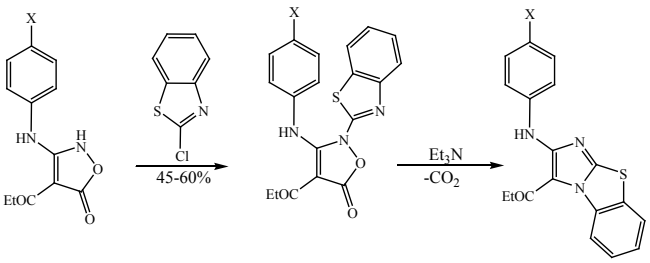


11 12 13

X: Br, Me, NO_2

Scheme IV

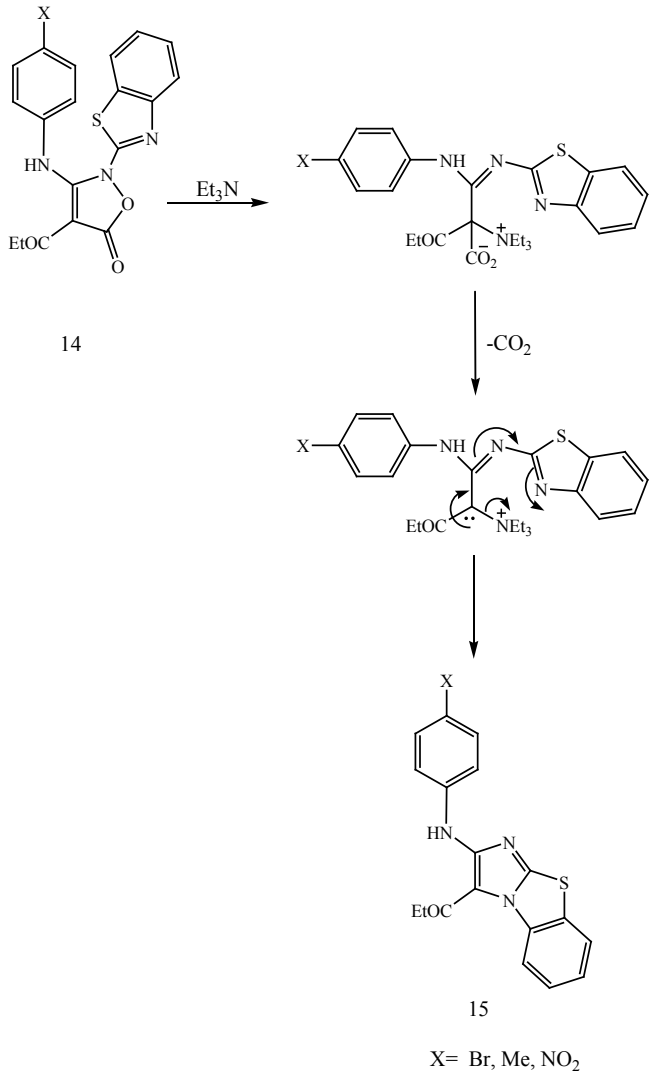
N-arylation of **13** with 2-chlorobenzothiazole in toluene under reflux conditions gave the corresponding N-substituted isoxazolones **14** in medium yield. The rearrangement of N-substituted isoxazolones **14**, as shown in (Scheme V), proceeded in 40-60% yield in refluxing ethanol for 48 h in the presence of triethylamine (TEA). The reaction pathway leading to the imidazobenzothiazole is consistent with our earlier suggestion for the formation of imidazopyridines, which is consistent with the electronic requirements of the reaction, as shown in (Scheme VI), or with the alternative pathway suggested by Prager and co-workers.[10]



13 14 15

X= Br, Me, NO_2

Scheme V



Scheme VI

III. Experimental

Freshly distilled solvents were used throughout, and anhydrous solvents were dried according to Perrin and Armarego.[11] Melting points were determined on a Philip Harris C4954718 apparatus and are uncorrected. Infrared spectra were recorded on a Thermonicolet (Nexus 670) FT-infrared spectrometer, using sodium chloride cells and measured as Nujol mulls or KBr. ^{1}H (300 MHz) and ^{13}C (300 MHz) NMR measurements were recorded on a Bruker 300 spectrometer in DMSO-d_6 or $CDCl_3$ using TMS as the internal reference. High resolution mass spectra were recorded on a Varian Matt 311 spectrometer. Mass spectra were registered in a HP 5973 MSD connected to HP 6890 GC interfaced by a Pentium PC and relative abundances of fragments are quoted in parentheses after the m/z values. Microanalyses were performed on a Leco Analyzer 932.

a) Ethyl-2-((4-bromophenyl)carbamothioyl)-3-xopentanoate (12, X: Br)

In a 100 mL round-bottomed flask, absolute ethanol (60 mL) was reacted with sodium (2.9 g, 0.126 mol) and after cooling to room temperature ethyl-3-oxopentanoate (20 g, 18.90 mL, 0.126 mol) was added. The reaction mixture was stirred at room temperature for 20 min; 4-bromophenyl isothiocyanate (26.82 g, 0.126 mol) was added and the stirring was continued for a further 6 h, during which a yellowish white precipitate of sodium ethyl-2- ((4-bromophenyl)carbamothioyl)-3-oxopentanoate salt was formed. The salt was collected and washed with light petroleum ether (b.p. 30-55 °C) (3 × 50 mL) to give yellow crystals m.p. 155-156 °C (31.18 g, 70%). The pure salt was dissolved in water (50 mL) and neutralized with dropwise addition of HCl (10%) to maintain the pH at 7. The product was extracted with dichloromethane (CH_2Cl_2) and the extract was washed with water (3 × 50 mL) and dried over anhydrous Na_2SO_4. Removal of solvent gave (10, X: Br) as a yellow oil (21.6g, 66%).

^{1}H-NMR($CDCl_3$)(δppm): 1.06 (t, J=7.1Hz, 3H), 1.3 (t, J=7.1Hz, 3H), 2.5 (q, J=7.1Hz, 2H), 4.32 (q, J=7.1Hz, 2H), 5.09 (s, 1H), 7.55 (d, J=8.6Hz, 2H), 7.73 (d, J=8.6Hz, 2H), 10.9 (bs, 1H, NH).

^{13}C-NMR($CDCl_3$)(δppm): 8.2, 14.34, 27.8, 63.63, 70.68, 120.37, 125.13, 132.37, 137.89, 166.08, 188.05.

FT-IR: 3285, 1759, 1723, 1548, 1431, 1285, 1146, 1023, 831 cm^{-1}.

b) Ethyl-2-((4-methylphenyl)carbamothioyl)-3-oxopentanoate (12, X: Me)

This compound was prepared as described above, using 4-methylphenyl isothiocyanate (1.34 g, 9 mmol) and stirring for a further 1.5 h after addition of 4-methylphenyl isothiocyanate to the ethyl-3-oxopentanoate salt to give ethyl-2-((4-methylphenyl) carbamothioyl)-3-oxopentanoate (1.92, 70.25%) as pale yellow solid, m.p. 52-53 °C.

^{1}H-NMR($CDCl_3$)(δppm): 1.06 (t, J=7.1Hz, 3H), 1.3 (t, J=7.1Hz, 3H), 2.35 (s, J=7.1Hz, 3H), 2.49 (q, J=7.1Hz, 2H), 4.32 (q, J=7.1Hz, 2H), 5.09 (s, 1H), 7.23 (d, J=8.3Hz, 2H), 7.66 (d, J=8.3Hz, 2H), 10.77 (bs, 1H, NH).

^{13}C-NMR($CDCl_3$)(δppm): 8.2, 14.35, 23.57, 34.3 63.47, 67.62, 123.64, 129.86, 136.41, 137.43, 166.16, 187.68.

FT-IR : 3284,1760, 1723, 1515, 1430, 1315, 1223, 1148, 1020, 831 cm^{-1}.

c) Ethyl-2-((4-nitrophenyl)carbamothioyl)-3 oxopentanoate (12, X: NO_2)

This compound was prepared as described above, using 4-nitrophenyl isothiocyanate (1.62 g, 9 mmol) and stirring for a further 1.5 h after addition of 4-nitrophenyl isothiocyanate to the ethyl-3-oxopentanoate salt to give ethyl-2-((4-nitrophenyl)carbamothioyl)-3-oxopentanoate (1.8, 68.20%) as pale yellow solid, m.p. 54-56 °C.

^{1}H-NMR($CDCl_3$) (δppm): 1.06 (t, J=7.1Hz, 3H), 1.3 (t, J=7.1Hz, 3H), 2.49 (q, J=7.1Hz, 2H), 4.32 (q,

J=7.1Hz, 2H), 5.09 (s, 1H), 6.8 (d, J=8.3Hz, 2H), 7.89 (d, J=8.3Hz, 2H), 10.77 (bs, 1H, NH).

^{13}C-NMR ($CDCl_3$) (δppm): 8.2, 14.35, 34.3 63.47, 67.62, 123.64, 129.86, 136.41, 137.43, 166.16, 187.68, 199.

FT-IR: 3284, 1760, 1723, 1515, 1430, 1350, 1315, 1223, 1148, 1020, 831 cm^{-1}.

d) 4-propionyl-3-(4-bromophenylamino)isoxazole-5(2H)-one (13, X: Br)

To a solution of hydroxylamine hydrochloride (7.06 g, 102 mmol) in water (30 mL), sodium bicarbonate (10.17 g, 102 mmol) was added slowly. Ethanol (80 mL) was added and the resulting potassium chloride was filtered off. Ethyl-2-(4-bromophenyl) carbamothioyl-3-oxopentanoate (**10**, 12.13g, 34 mmol) was added to the filtrate and the mixture was stirred at room temperature for 24 h. The reaction mixture was acidified with dilute HCl and the white precipitate was collected and recrystallized from acetone to give the title product (8.78 g, 79%) as colourless crystals, m.p.= 201 ^{o} C (dec.).

^{1}H-NMR (d_6-DMSO)(δppm): 1.11 (t, J=7.1Hz, 3H), 3.01 (q, J=7.1Hz, 2H), 7.37(d, J=8.4Hz, 2H), 7.57 (d, J=8.4Hz, 2H), 8.30 (bs, 1H, NH), 9.39 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO)(δppm): 7.9, 30.7, 59.96, 84, 118.02, 132.94, 137.10, 163.53, 164.74, 167.39.

FT-IR : 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1398, 1316, 1183, 1018, 818 cm^{-1}.

e) 4-propionyl-3-(4-methylphenylamino)isoxazole-5(2H)-one (13, X: Me)

The compound was prepared as described above using Ethyl-2-(4-methylphenyl)carbamothioyl-3-oxopentanoate (1.17 g, 4 mmol) and refluxing for 24 h to give the desired product as colourless crystals (0.7 g,73%), m.p. 165-167 ^{o}C.

^{1}H-NMR (d_6-DMSO + $CDCl_3$) (δppm): 1.11 (t, J=7.1Hz, 3H), 2.35 (s, Me, 3H), 3.01 (q, J=7.1Hz, 2H), 6.78 (d, J=9.2Hz, 2H), 6.79(bs, 1H, NH), 6.80(d, J=9.2Hz, 2H), 8.85 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO+ $CDCl_3$)(δppm): 7.9, 24.52, 30.85, 84.2, 121.53, 130.13, 133.29, 135.64, 163.59, 165.51, 166.74.

FT-IR : 3669, 2979, 2746, 1705, 1669, 1615, 1331, 1208, 1115, 1023, 800 cm^{-1}.

f) 4-propionyl-3-(4-nitrophenylamino)isoxazole-5(2H)-one (13, X: NO_2)

The compound was prepared as described above using Ethyl-2-(4-nitrophenyl)carbamothioyl-3-oxopentanoate (1.3 g, 4 mmol) and refluxing for 24 h to give the desired product as colourless crystals (0.5 g,65%), m.p. 162-164 ^{o}C.

^{1}H-NMR (d_6-DMSO + $CDCl_3$) (δppm): 1.11 (t, J=7.1Hz, 3H), 3.01 (q, J=7.1Hz, 2H), 6.78 (d, J=9.2Hz, 2H), 6.79(bs, 1H, NH), 7.8 (d, J=9.2Hz, 2H), 8.85 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO+ $CDCl_3$) (δppm): 7.9, 30.85, 84.2, 121.53, 130.13, 133.29, 135.64, 163.59, 165.51, 166.74.

FT-IR: 3669, 2979, 2746, 1705, 1669, 1615, 1350, 1331, 1208, 1115, 1023, 800 cm^{-1}.

g) 4-propionyl-3-(4-bromophenylamino)-2-(benzothiazol-2-yl)-isoxazol-5(2H)-one (14, X: Br)

4-propionyl-3-(4-bromophenylamino) isoxazole-5(2H)-one (93 mg, 0.3 mmol) and 2-chlorobenzothiazole (51 mg, 0.3 mmol) were refluxed in toluene (6 mL) for 48 h. The solvent was removed under reduced pressure. On addition of n-hexane (10 mL) to the residue (colourless oil) a white precipitate was formed. The precipitate was filtered and recrystallized from ethanol to give 4-propionyl-3-(4-bromophenylamino)-2-(benzothiazol-2-yl)-isoxazol-5(2H)-one as white prisms (84.4 mg, 60%) m.p. 153-155 ^{o}C.

^{1}H-NMR (d_6-DMSO)(δppm): 1.11 (t, J=7.1Hz, 3H), 3.01 (q, J=7.1Hz, 2H), 6.37(d, J=8.4Hz, 2H), 7.3 (d, J=8.4Hz, 2H), 7.6 (t, J=8.4Hz, 2H), 8.3 (d, J=8.4Hz, 2H), 8.30 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO)(δppm): 7.9, 30.7, 59.96, 84, 118.02, 121, 125.3, 132.94, 137.10, 163.53, 164.74, 167.39.

FT-IR: 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1402, 1398, 1301, 1183, 1018, 818 cm^{-1}.

MS *m/z*: (%) 442.9 (M+, 12%), 440 (M+, 11%), 417 (82), 415 (71), 371 (48), 369 (40), 334 (25), 294 (28), 291 (27), 290 (100), 262 (30), 224 (27), 177 (33), 161 (34), 150 (40), 135 (26), 134 (33), 108 (29), 44 (65).

h) 4-propionyl-3-(4-methylphenylamino)-2-(benzothiazol-2-yl)-isoxazol-5(2H)-one (14, X: Me)

This compound was prepared as described above, using the corresponding isoxazolone (11, X: Me) (67 mg, 0.27 mmol) and 2-chlorobenzothiazole (45.8 mg, 0.27 mmol) to give the desired product as white prisms (48 mg, 50%) after recrystalization from ethanol, m.p. 158-160 ^{o}C.

^{1}H-NMR (d_6-DMSO)(δppm): 1.11 (t, J=7.1Hz, 3H), 2.35 (s, J=7.1Hz, 3H), 3.01 (q, J=7.1Hz, 2H), 6.37(d, J=8.4Hz, 2H), 7.3 (d, J=8.4Hz, 2H), 7.6 (t, J=8.4Hz, 2H), 8.3 (d, J=8.4Hz, 2H), 8.33 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO) (δppm): 7.9, 24.3, 30.7, 59.96, 84, 118.02, 121, 125.3, 132.94, 137.10, 163.53, 164.74, 167.39.

FT-IR: 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1402, 1398, 1301, 1183, 1018, 818 cm^{-1}.

MS *m/z*: (%) 379.1 (M+, 12%), 371 (48), 369 (40), 334 (25), 294 (28), 291 (27), 290 (100), 262 (30), 224 (27), 177 (33), 161 (34), 150 (40), 135 (26), 134 (33), 108 (29), 44 (65).

i) 4-propionyl-3-(4-nitrophenylamino)-2-(benzothiazol-2-yl)-isoxazol-5(2H)-one (14, X: NO_2)

This compound was prepared as described above, using the corresponding isoxazolone (11, X: NO_2) (94 mg, 0.34 mmol) and 2-chlorobenzothiazole (57.1 mg, 0.34 mmol) to give the desired product as white prisms (44 mg, 47%) after recrystalization from ethanol, m.p. 168-170 °C.

^{1}H-NMR (d_6-DMSO)(δppm): 1.11 (t, J=7.1Hz, 3H), 3.01 (q, J=7.1Hz, 2H), 6.7(d, J=8.4Hz, 2H), 7.6 (t, J=8.4Hz, 2H), 7.9 (d, J=8.4Hz, 2H), 8.3 (d, J=8.4Hz, 2H), 8.33 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO) (δppm): 7.9, 30.7, 59.96, 84, 118.02, 121, 125.3, 132.94, 137.10, 163.53, 164.74, 167.39.

FT-IR : 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1450, 1402, 1398, 1301, 1183, 1018, 818 cm^{-1}.

MS *m/z*: (%) 410.1 (M+, 12%), 402 (48), 370 (40), 334 (25), 294 (28), 291 (27), 290 (100), 262 (30), 224 (27), 177 (33), 161 (34), 150 (40), 135 (26), 134 (33), 108 (29), 44 (65).

j) 1-(2-(4-bromophenylamino)-imidazo[2,1-b] benzothiazole-3-yl) propan-1-one (15, X: Br)

The isoxazolone (12, X: Br) (97.2 mg, 0.22 mmol) and triethylamine (0.3 mL) were refluxed in ethanol (10 mL) for 24 hours. The reaction mixture was left to cool to room tempreture and resulting precipitate was colleted to afford 1-(2-(4-bromophenylamino)-imidazo [2, 1-b] benzothiazole-3-yl) propan-1-one as white needles (49 mg, 55%), mp 177-182 °C.

^{1}H-NMR (d_6-DMSO)(δppm): 1.18 (t, J=7.1Hz, 3H), 2.5 (q, J=7.1Hz, 2H), 6.6(d, J=8.4Hz, 2H), 7.2 (t, J=8.4Hz, 1H), 7.3 (t, J=8.4Hz, 1H), 7.5 (d, J=8.4Hz, 2H), 8.2 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO) (δppm): 7.6, 32.7, 59.96, 84, 118.02, 121, 125.3, 132.94, 137.10, 143.53, 144.74, 147.39.

FT-IR: 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1450, 1402, 1398, 1301, 1183, 1018, 818 cm^{-1}.

MS *m/z*: (%) 399.1 (M+, 12%), 387 (48), 370 (40), 334 (25), 298 (28), 295 (27), 293 (100), 262 (30), 224 (27), 179 (33), 161 (34), 153(40), 145 (26), 134 (33), 108 (29), 44 (65).

k) 1-(2-(4-methylphenylamino)-imidazo[2,1-b] benzothiazole-3-yl) propan-1-one (15, X: Me)

The isoxazolone (12, X: Me) (83.3 mg, 0.22 mmol) and triethylamine (0.3 mL) were refluxed in ethanol (10 mL) for 24 hours. The reaction mixture was left to cool to room tempreture and resulting precipitate was colleted to afford 1-(2-(4-methylphenylamino)-imidazo [2, 1-b] benzothiazole-3-yl) propan-1-one as white needles (43 mg, 50%), mp 151-156 °C.

^{1}H-NMR (d_6-DMSO)(δppm): 1.18 (t, J=7.1Hz, 3H), 2.3 (s, J=7.1Hz, 3H), 2.5 (q, J=7.1Hz, 2H), 6.6 (d, J=8.4Hz, 2H), 6.9 (d, J=8.4Hz, 2H), 7.3 (t, J=8.4Hz, 1H), 7.5 (t, J=8.4Hz, 1H), 7.7 (d, J=8.4Hz, 2H), 8.2 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO) (δppm): 7.6, 24.3, 32.7, 59.96, 84, 118.02, 121, 125.3, 132.94, 137.10, 143.53, 144.74, 147.39.

FT-IR: 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1450, 1402, 1398, 1301, 1183, 1018, 818 cm^{-1}.

MS *m/z*: (%) 336.11 (M+, 12%), 330 (48), 310 (40), 304 (25), 298 (28), 295 (27), 293 (100), 262 (30), 224 (27), 179 (33), 161 (34), 153(40), 145 (26), 134 (33), 108 (29), 44 (65).

l) 1-(2-(4-nitrophenylamino)-imidazo[2,1-b] benzothiazole-3-yl) propan-1-one (15, X: NO_2)

The isoxazolone (12, X: NO_2) (90 mg, 0.22 mmol) and triethylamine (0.3 mL) were refluxed in ethanol (10 mL) for 24 hours. The reaction mixture was left to cool to room tempreture and resulting precipitate was colleted to afford 1-(2-(4-nitrophenylamino)-imidazo [2, 1-b] benzothiazole-3-yl) propan-1-one as white needles (41 mg, 49%), mp 199-200 °C.

^{1}H-NMR (d_6-DMSO)(δppm): 1.18 (t, J=7.1Hz, 3H), 2.5 (q, J=7.1Hz, 2H), 6.7 (d, J=8.4Hz, 2H), 7.3 (t, J=8.4Hz, 1H), 7.5 (t, J=8.4Hz, 1H), 7.7 (d, J=8.4Hz, 2H), 8.6 (bs, 1H, NH).

^{13}C-NMR (d_6-DMSO)(δppm): 7.6, 32.7, 70.96, 84, 118.02, 121, 125.3, 132.94, 137.10, 143.53, 144.74, 147.39.

FT-IR: 3250, 2950, 2740, 1723, 1696, 1666, 1607, 1563, 1456, 1450, 1402, 1398, 1353, 1301, 1183, 1018, 818 cm^{-1}.

MS *m/z*: (%) 366.11 (M+, 11%), 353 (48), 310 (40), 304 (25), 298 (28), 295 (27), 293 (100), 262 (30), 224 (27), 179 (33), 171 (34), 153(40), 149 (26), 134 (33), 108 (29), 44 (65).

IV. Conclusion

In conclusion we have shown that a variety of N-substituted isoxazolones **14**, rearranged with Triethylamine to give imidazo [2, 1-b] benzothiazole.

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