



Following the methodology developed<sup>27</sup> using oxime ether mediated ring closure, Meldrum's acid could be very useful to synthesise spiro-heterobicyclic rings. Indeed, the spiro-heterobicycle **3** could be helpful to construct non natural amino acid by solvolysis of Meldrum's acid with alcohols would give a diastereomeric mixture of a half acid ester; and there could be further interconverted. The half carboxylic acid can be converted into amine or carbamate, through acid azide formation, and followed by Curtius rearrangement, would give an isocyanate, which could be easily converted into amine or carbamate by hydrolysis or alcohol treatment<sup>31</sup>.

The required spiro-heterobicyclic ring **3** could be synthesised by following the sequence of reactions. The reaction of Meldrum's acid with methyl vinyl ketone in presence of 1.1 eq. of anhydrous K<sub>2</sub>CO<sub>3</sub> and 1 eq. of benzyltriethylammonium chloride in acetonitrile as the solvent, at 50-60°C for 8-10 h<sup>32</sup> afforded the Michael adduct **1** in 81% yield with some double Michael addition product as by-product which was minimised by using 1.1eq. base. The Michael adduct **1** was readily converted to the corresponding oxime using standard conditions (NH<sub>2</sub>OH.HCl and Et<sub>3</sub>N in EtOH stirred at 50°C for 1 hr) and afforded **2** in 77% yield. The crude oxime **2** was used directly and treatment with *p*-toluenesulfonyl chloride and base (excess Et<sub>3</sub>N in dry CH<sub>2</sub>Cl<sub>2</sub> at RT) gave the cyclization product Spiro-3,4-dihydro-2*H*-pyrrole **3** directly in 78% of yield (Scheme I) instead the expected tosyl oxime (Scheme I).

The resultant Spiro-3,4-dihydro-2*H*-pyrrole **3** is crystalline and this product was fully characterised by single-crystal X-ray crystallographic analysis (Figure 1), clearly demonstrating the fully orthogonal nature of the two heterocyclic rings. The spiro-heterobicycle **3** was previously reported by Danheiser<sup>33</sup> and he obtained as oil but NMR data agree.

Similarly the Michael addition reaction with Meldrum's acid and  $\alpha,\beta$ -unsaturated ketones such as 1-phenylbut-2-en-1-one, 1-phenylprop-2-en-1-one, pent-3-en-2-one and chalcone was carried out in presence of anhydrous K<sub>2</sub>CO<sub>3</sub> and 1 eq. of benzyltriethylammonium chloride in acetonitrile as the solvent, at 50-60°C for 8-10 hr except chalcone other ketones gave good yield as Michael adduct as shown in Scheme II. The same Michael adduct on oximation using standard condition and followed by treatment with *p*-toluenesulfonyl chloride and base (excess Et<sub>3</sub>N in dry CH<sub>2</sub>Cl<sub>2</sub> at RT) gave the cyclization product Spiro-3,4-dihydro-2*H*-pyrrole as **6**, **9** and **12** in good yield as shown in Scheme II but as the compounds are solid but the formation of single crystal was so difficult and all compounds are characterised by Mass and NMR spectra.

## Experimental Section

### General procedure for the preparation of Michael adduct<sup>9-12</sup>

To a stirring solution 7.63 mmol of Meldrumic acid (1.1g) in acetonitrile (20 mL) was added 8.39 mmol

of anhydrous  $K_2CO_3$  (1.15g) and 7.63 mmol of benzyltriethylammonium chloride (1.73g). The mixture was stirred for 15 min at RT and 7.63 mmol of methyl vinyl ketone (0.64mL) was then added and the resultant mixture was stirred for 8 hr at  $50^\circ C$ . After TLC monitored, the mixture was cooled to RT, the reaction was quenched with water and mixture was washed with diethyl ether and then acidified by 6 N HCl. The adduct was extracted by diethyl ether and dried over  $Na_2SO_4$ , to give crude product, which was purified by recrystallization using EtOAc and petroleum ether to afford Michael adduct **1**.

### General procedure for the preparation of oxime of Michael adduct<sup>27,28</sup>

To the solution of 2.33 mmol Michael adduct ketone **1** (500mg) in 10 mL EtOH was added 4.66 mmol of  $NH_2OH \cdot HCl$  (183mg) and 4.66 mmol of  $Et_3N$  (470mg) and the reaction mixture was heated to  $50^\circ C$  for 1 hr. The progress of the reaction was monitored by TLC and on completion of the reaction mixture was allowed to cool to RT. EtOH was

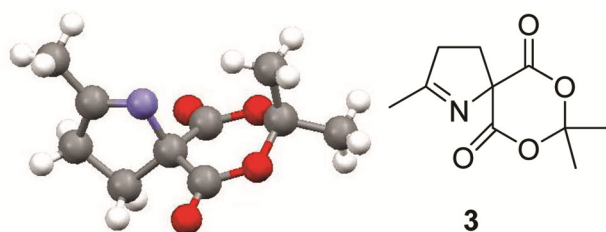


Figure 1 — X-ray crystal structure for spiro-3,4-dihydro-2H-pyrrole **3**

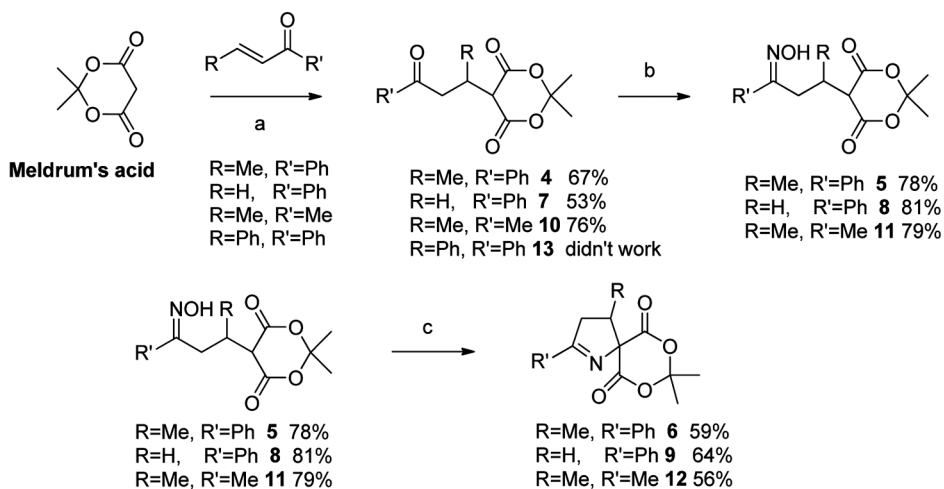
removed under reduced pressure and the residue was dissolved in EtOAc and  $H_2O$  added. The organic layer was separated, washed with brine and dried over  $Na_2SO_4$ . Concentration of the organic layer gave the crude product, which was purified by flash column chromatography (eluting with EtOAc : petrol) to afford the oxime product **2**.

### General procedure for cyclization to spiro-3,4-dihydro-2H-pyrrole<sup>29</sup>

The stirring solution 2.18 mmol of **2** oxime (500mg) in dry 10 mL  $CH_2Cl_2$  at  $0^\circ C$  was treated with 6.54 mmol of  $Et_3N$  or pyridine (660mg or 516mg) followed by slow addition 2.61 mmol of *p*-toluenesulphonyl chloride (494 mg) and reaction was stirred for 1 hrs and it was found in mass spectra showed presence of cyclized product. The reaction mixture was further stirred for 2 hrs and the reaction progress was monitored by TLC and by mass spectra and it was found that instead of tosyloxime the cyclized product was formed and after completion of reaction (1 M) HCl was added and product was extracted by  $CH_2Cl_2$  and the extracts were washed with saturated  $NaHCO_3$  and dried over  $Na_2SO_4$ . Concentration of  $CH_2Cl_2$  gave crude product which was purified by flash column chromatography (eluting with EtOAc : petrol) to afford the Spiro-3,4-dihydro-2H-pyrrole.

### (±) Dimethyl-5-(3-oxobutyl)-1,3-dioxane-4,6-dione, **1**<sup>32</sup>

Following the general procedure **1**, to the stirring solution 15.3 mmol of Meldrumic acid (2.21g) in



**Reagents and conditions:** (a) anhydrous  $K_2CO_3$ , benzyltriethylammonium chloride, acetonitrile, heated  $50-60^\circ C$ ; (b)  $NH_2OH \cdot HCl$ ,  $Et_3N$ , EtOH, heated  $50^\circ C$ ; (c) *p*-TsCl,  $Et_3N$ ,  $CH_2Cl_2$ , RT

acetonitrile (40 mL) was added 16.8 mmol of anhydrous  $K_2CO_3$  (2.31g) and 15.3 mmol of benzyltriethylammonium chloride (3.48g). The mixture was stirred for 15 min at RT and 15.3 mmol of methyl vinyl ketone (1.3mL) was then added and the resultant mixture was stirred for 8 hr at 50°C to give crude product, which was purified by recrystallization using EtOAc and petroleum ether to afford adduct **1** (2.67 g, 81%) as pale yellow solid (m.p.116-118°C, *lit* 119-120°C);  $R_f$  = 0.16 (EtOAc : petrol, 1:1); IR (film): 2892, 1785, 1747, 1710, 1382, 1303, 1249, 1165, 1010, 986, 730  $cm^{-1}$ ;  $^1H$  NMR:  $\delta_H$ (400 MHz;  $CDCl_3$ ;  $Me_4Si$ ) 1.74, 1.78 (6H, s,  $C(CH_3)_2$ ), 2.13 (3H, s,  $COCH_3$ ), 2.78 (2H, m,  $CH_2CH_2CH$ ), 2.75 (2H, t,  $J$  7.1 Hz,  $CH_2CH_2CH$ ), 3.86 (1H, t,  $J$  5.4 Hz,  $CH_2CH_2CH$ );  $^{13}C$  NMR:  $\delta_C$ (100 MHz;  $CDCl_3$ ;  $Me_4Si$ ) 19.5 ( $CH_2CH_2CH$ ), 25.8, 27.9 ( $C(CH_3)_2$ ), 29.5 ( $COCH_3$ ), 38.7 ( $CH_2CH_2CH$ ), 44.0 ( $CH_2CH_2CH$ ), 104.5 ( $C(CH_3)_2$ ), 164.7 ( $2 \times COO$ ), 207.5 (CO); MS (ESI $^-$ ):  $m/z$  213, ( $[M-H]^-$ , 100%); HRMS (ESI $^-$ )  $C_{10}H_{13}O_5^-$  ( $[M-H]^-$ ) requires: 213.0768. Found: 213.0762.

**(±) 5-(3-(Hydroxyimino)butyl)-2,2-dimethyl-1,3-dioxane-4,6-dione, 2**<sup>27,28</sup>

Following the general procedure **2**, to the solution 6.44 mmol of Michael adduct **1** (1.38 g) in EtOH (30mL), 12.88 mmol of  $NH_2OH.HCl$  (888mg) and 12.88 mmol of  $Et_3N$  (1.30g) was added and the mixture was gently heated (50°C) for 1 hrs, to give crude product which was purified by flash column chromatography to afford oxime **2** (1.14 g, 77%) as pale yellow semi-solid;  $R_f$  = 0.23 (EtOAc : petrol, 1:1); IR (film): 3319, 2982, 1786, 1743, 1715, 1372, 1307, 1240, 1176, 1012, 982, 731  $cm^{-1}$ ;  $^1H$  NMR: (major isomer)  $\delta_H$ (400 MHz;  $CDCl_3$ ;  $Me_4Si$ ) 1.74, 1.78 (6H, s,  $C(CH_3)_2$ ), 1.94, 1.91 (3H, s,  $CNOHCH_3$ ), 2.31 (2H, m,  $CH_2CH_2CH$ ), 2.44, 2.63 (2H, t,  $J$  7.4 Hz,  $CH_2CH_2CH$ ), 3.68 (1H, t,  $J$  6.8 Hz,  $CH_2CH_2CH$ ), 9.33 (1H, bs,  $OH$ );  $^{13}C$  NMR:  $\delta_C$ (100 MHz;  $CDCl_3$ ;  $Me_4Si$ ) 13.9, 15.1 ( $CNOHCH_3$ ), 21.3, 22.1 ( $CH_2CH_2CH$ ), 26.2, 27.3 ( $C(CH_3)_2$ ), 32.2, 35.9 ( $CH_2CH_2CH$ ), 44.8 ( $CH_2CH_2CH$ ), 104.8, 106.1 ( $C(CH_3)_2$ ), 157.6, 158.1 ( $C=NOH$ ), 162.7, 164.9 ( $2 \times COO$ ); MS (ESI $^-$ ):  $m/z$  228 ( $[M-H]^-$ , 100%); HRMS (ESI $^-$ )  $C_{10}H_{14}NO_5^-$  ( $[M-H]^-$ ) requires: 228.0881. Found: 228.0877.

**(±) 2,8,8-Trimethyl-7,9-dioxo-1-azaspiro[4.5]dec-1-ene-6,10-dione, 3**<sup>34,35</sup>

Following the general procedure **3**, to the solution 1.79 mmol of oxime **2** (412mg) in dry  $CH_2Cl_2$  (20mL)

was added 5.37 mmol of  $Et_3N$  (542mg) at 0°C and followed by 2.15 mol of *p*-toluenesulphonyl chloride (410 mg) and the mixture stirred for 30 min instead tosyl oxime, isolated cyclized product as spirobicyclic pyrrolidine which was purified by flash column chromatography to afford compound **3** (297 mg, 78%) as a crystalline solid; m.p. = 98°C;  $R_f$  = 0.63 (EtOAc : petrol, 1:1); IR (film): 2933, 1782, 1731, 1452, 1170, 914, 731  $cm^{-1}$ ;  $^1H$  NMR (400 MHz;  $CDCl_3$ ;  $Me_4Si$ ):  $\delta_H$  1.78, 2.06 ( $C(CH_3)_2$ ), 2.16 (3H, s,  $N=CCH_3$ ), 2.59 (2H, t,  $J$  7.6 Hz,  $C(4)HH$ ), 2.97 (2H, t,  $J$  7.6 Hz,  $C(3)HH$ );  $^{13}C$  NMR (100 MHz;  $CDCl_3$ ;  $Me_4Si$ ):  $\delta_C$  19.2 ( $COCH_3$ ), 28.3, 28.5 ( $C(CH_3)_2$ ), 31.4 (C4), 41.2 (C3), 80.7 (C2), 106.2 ( $C(CH_3)_2$ ), 167.4 ( $2 \times COO$ ), 182.9 (C5); MS (ESI $^-$ ):  $m/z$  445 ( $[2M+Na]^+$ , 100%), 234 ( $[M+Na]^+$ , 95%); HRMS (ESI $^+$ )  $C_{10}H_{13}NNaO_5^+$  ( $[M+Na]^+$ ) requires: 234.0742. Found: 234.0737.

**(±) 4,8,8-Trimethyl-2-phenyl-7,9-dioxo-1-azaspiro [4.5]dec-1-ene-6,10-dione, 6**

Following the general procedure **3**, to the solution 0.95 mmol of oxime **5** (289 mg) in dry  $CH_2Cl_2$  (15 mL) was added 2.85 mmol of  $Et_3N$  (288mg) at 0°C and followed by 1.14 mmol of *p*-toluenesulphonyl chloride (216 mg) and the mixture was stirred for 30 min instead tosyl oxime, isolated cyclized product as spirobicyclic pyrrolidine which was purified by flash column chromatography to afford compound **6** (160 mg, 59%) as a crystalline solid; m.p. = 103°C;  $R_f$  = 0.46 (EtOAc : petrol, 1:1.5); IR (film): 3078, 2956, 1779, 1737, 1170, 912, 740  $cm^{-1}$ ;  $^1H$  NMR (400 MHz;  $CDCl_3$ ;  $Me_4Si$ ):  $\delta_H$  0.96 (3H, d,  $CH_3$ ), 1.75, 2.00 ( $C(CH_3)_2$ ), 2.55 (2H, d,  $J$  7.5 Hz,  $C(4)HH$ ), 2.79 (1H, m,  $C(3)H$ ), 7.3-7.9 (ArH);  $^{13}C$  NMR (100 MHz;  $CDCl_3$ ;  $Me_4Si$ ):  $\delta_C$  28.9, 28.2 ( $C(CH_3)_2$ ), 31.9 (C4), 41.0 (C3), 89.7 (C5), 106.0 ( $C(CH_3)_2$ ), 112-139 (ArC), 167.0 ( $2 \times COO$ ), 180.9 (C2); MS (ESI $^-$ ):  $m/z$  310 ( $[M+Na]^+$ , 95%); HRMS (ESI $^+$ )  $C_{16}H_{17}NNaO_4^+$  ( $[M+Na]^+$ ) requires: 310.1055. Found: 234.1076.

**(±) 8,8-Dimethyl-2-phenyl-7,9-dioxo-1-azaspiro [4.5]dec-1-ene-6,10-dione, 9**

Following the general procedure **3**, to the solution 0.69 mmol of oxime **8** (200mg) in dry  $CH_2Cl_2$  (10mL) was added 2.07 mmol of  $Et_3N$  (209mg) at 0°C and followed by 0.83 mmol of *p*-toluenesulphonyl chloride (157 mg) and mixture was stirred for 30 min instead tosyl oxime, isolated cyclized product as spirobicyclic pyrrolidine which was purified by flash column chromatography to afford compound **9** (120 mg, 64%) as a crystalline solid; m.p. = 97°C;

$R_f = 0.53$  (EtOAc : petrol, 1:1); IR (film): 3093, 2931, 1778, 1735, 1177, 910  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  (400 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ):  $\delta_{\text{H}}$  1.70, 2.03 ( $\text{C}(\text{CH}_3)_2$ ), 2.52 (2H, t,  $J$  7.4 Hz,  $\text{C}(4)\text{HH}$ ), 2.90 (2H, t,  $J$  7.4 Hz,  $\text{C}(3)\text{HH}$ ), 7.3-7.9 (ArH);  $^{13}\text{C NMR}$  (100 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ):  $\delta_{\text{C}}$  28.9, 28.2 ( $\text{C}(\text{CH}_3)_2$ ), 31.9 (C4), 41.0 (C3), 89.7 (C5), 106.0 ( $\text{C}(\text{CH}_3)_2$ ), 114-138 (ArC), 167.0 ( $2 \times \text{COO}$ ), 180.9 (C2); MS (ESI $^-$ ):  $m/z$  296 ( $[\text{M}+\text{Na}]^+$ , 95%); HRMS (ESI $^+$ )  $\text{C}_{15}\text{H}_{15}\text{NNaO}_4^+$  ( $[\text{M}+\text{Na}]^+$ ) requires: 296.0899. Found: 234.0901.

### (±) 2,4,8,8-Tetramethyl-7,9-dioxo-1-azaspiro[4.5] dec-1-ene-6,10-dione, 12

Following the general procedure 3, to the solution 0.88 mmol of oxime 11 (215mg) in dry  $\text{CH}_2\text{Cl}_2$  (10mL) was added 2.64 mmol of  $\text{Et}_3\text{N}$  (267mg) at  $0^\circ\text{C}$  and followed by 1.06 mmol of *p*-toluenesulphonyl chloride (201 mg.) and the mixture stirred for 30 min instead tosyl oxime, isolated cyclized product as spirobicyclic pyrrolidine which was purified by flash column chromatography to afford compound 12 (111 mg, 56%) as a crystalline solid; m.p. =  $99^\circ\text{C}$ ;  $R_f = 0.51$  (EtOAc : petrol, 1:1); IR (film) 2934, 1780, 1732, 1456, 1172, 915, 735  $\text{cm}^{-1}$ ;  $^1\text{H NMR}$  (400 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ):  $\delta_{\text{H}}$  0.98 (3H, d,  $\text{CH}_3$ ), 1.79, 2.05 ( $\text{C}(\text{CH}_3)_2$ ), 2.16 (3H, s,  $\text{N}=\text{CCH}_3$ ), 2.64 (1H, m,  $\text{C}(4)\text{H}$ ), 2.94 (2H, d,  $J$  7.5 Hz,  $\text{C}(3)\text{HH}$ );  $^{13}\text{C NMR}$  (100 MHz;  $\text{CDCl}_3$ ;  $\text{Me}_4\text{Si}$ ):  $\delta_{\text{C}}$  18.2, ( $\text{CH}_3$ ), 21.5 ( $\text{N}=\text{CCH}_3$ ), 28.4, 28.7 ( $\text{C}(\text{CH}_3)_2$ ), 32.1 (C4), 43.2 (C3), 88.9 (C5), 106.7 ( $\text{C}(\text{CH}_3)_2$ ), 167.9 ( $2 \times \text{COO}$ ), 182.8 (C2); MS (ESI $^-$ ):  $m/z$  248 ( $[\text{M}+\text{Na}]^+$ , 95%); HRMS (ESI $^+$ )  $\text{C}_{11}\text{H}_{15}\text{NNaO}_4^+$  ( $[\text{M}+\text{Na}]^+$ ) requires: 248.0899. Found: 234.0839.

### Conclusion

In summary, we have developed a one-pot methodology for the synthesis of spiro-heterobicyclic compounds as spiro-3,4-dihydro-2*H*-pyrroles with alkyl and aryl substitutions on the five member heterocycle. It was also concluded that both aryl groups as substitution was not possible as we have started with chalcone as starting material and reaction did not work. The method is useful for the synthesis of the rigid spiro-heterocycle which can also be converted to the non-natural amino acids by hydrolysis to half acid ester and interconverted to amino group through Curtius rearrangement reaction. The present methodology developed will be very useful to get quick access to spiro-3,4-dihydro-2*H*-pyrroles which are part of many natural products of high importance.

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