First mechanosynthesis of Piperlotines A, C and derivatives through solvent-free Horner-Wadsworth-Emmons reaction

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Fig. 1 1H NMR spectra of *N-(bromoacetyl)pyrrolidine* **1.**

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Fig. 2 13C NMR spectra of *N-(bromoacetyl)pyrrolidine* **1.**

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Fig. 3 HRMS spectra of *N-(bromoacetyl)pyrrolidine* **1.**

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Fig. 4 1H NMR spectra of *diethyl (2-oxo-2-(pyrrolidin-1-yl)ethyl)phosphonate* **2.**

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Fig. 5 13C NMR spectra of *diethyl (2-oxo-2-(pyrrolidin-1-yl)ethyl)phosphonate* **2.**

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Fig. 6 31P NMR spectra *diethyl (2-oxo-2-(pyrrolidin-1-yl)ethyl)phosphonate* **2.**



Fig. 7 HRMS spectra of *diethyl (2-oxo-2-(pyrrolidin-1-yl)ethyl)phosphonate* **2.**

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Fig. 8 1H NMR spectra of *(E)-3-(4-methoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one*  **3a.**



Fig. 9 13C NMR spectra of *(E)-3-(4-methoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one*  **3a.**

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Fig. 10 HRMS spectra of *(E)-3-(4-methoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one*  **3a.**

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Fig. 11 1H NMR spectra of *(E)-3-(3,5-dimethoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3b.**

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Fig. 12 13C NMR spectra of *(E)-3-(3,5-dimethoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3b.**

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Fig. 13 HRMS spectra of *(E)-3-(3,5-dimethoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3b.**

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Fig.14 1H NMR spectra of *(E)-1-(pyrrolidin-1-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one* **3c.**

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Fig. 15 13C NMR spectra of *(E)-1-(pyrrolidin-1-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one* **3c.**

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Fig. 16 HRMS spectra of *(E)-1-(pyrrolidin-1-yl)-3-(3,4,5-trimethoxyphenyl)prop-2-en-1-one* **3c.**



Fig. 17 1H NMR spectra of *(E)-3-(4-(dimethylamino)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3d.**



Fig. 18 13C NMR spectra of *(E)-3-(4-(dimethylamino)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3d.**

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Fig. 19 HRMS spectra of *(E)-3-(4-(dimethylamino)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3d.**



Fig. 20 1H NMR spectra of *(E)-3-(4-(methylthio)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3e.**

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Fig. 21 13C NMR spectra of *(E)-3-(4-(methylthio)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3e.**

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Fig. 22 HRMS spectra of *(E)-3-(4-(methylthio)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3e.**



Fig. 23 1H NMR spectra of *(2E,4E)-5-phenyl-1-(pyrrolidin-1-yl)penta-2,4-dien-1-one* **3f.**



Fig. 24 13C NMR spectra of *(2E,4E)-5-phenyl-1-(pyrrolidin-1-yl)penta-2,4-dien-1-one* **3f.**

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Fig. 25 HRMS spectra of *(2E,4E)-5-phenyl-1-(pyrrolidin-1-yl)penta-2,4-dien-1-one* **3f.**



Fig. 26 1H NMR spectra of *(E)-3-(4-chlorophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3g.**



Fig. 27 13C NMR spectra of *(E)-3-(4-chlorophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3g.**

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Fig. 28 HRMS spectra of *(E)-3-(4-chlorophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3g.**

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Fig. 29 1H NMR spectra of *(E)-3-(4-nitrophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3h.**



Fig. 30 13C NMR spectra of *(E)-3-(4-nitrophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3h.**



Fig. 31 HRMS spectra of *(E)-3-(4-nitrophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3h.**



Fig. 32 1H NMR spectra of *(E)-1-(pyrrolidin-1-yl)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one* **3i.**



Fig. 33 13C NMR spectra of *(E)-1-(pyrrolidin-1-yl)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one* **3i.**



Fig. 34 HRMS spectra of *(E)-1-(pyrrolidin-1-yl)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one* **3i.**