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

**Oscar Abelardo Ramírez-Marroquín1\* ● Flavio Manzano-Pérez1 ● Adolfo López-Torres1 ● Alejandro Hernández-López2 ● Abimelek Cortés-Pacheco1 ● Miguel Angel Reyes-González3**

1Instituto de Química Aplicada, Universidad del Papaloapan, Tuxtepec, México.

2Instituto de Biotecnología, Universidad del Papaloapan, Tuxtepec, México.

3Facultad de Ciencias Químicas, Universidad Autónoma de Nuevo León, San Nicolás de los Garza Nuevo León, México.

|  |  |
| --- | --- |
| **CONTENTS** | |
| Spectral information of compound **1** (NMR 1H, 13C, HMRS) | p. 2 |
| Spectral information of compound **2** (NMR 1H, 13C, 31P, HRMS) | p. 4 |
| Spectral information of compound **3a** (NMR 1H, 13C, HRMS) | p. 6 |
| Spectral information of compound **3b** (NMR 1H, 13C, HRMS) | p. 8 |
| Spectral information of compound **3c** (NMR 1H, 13C, HRMS) | p. 10 |
| Spectral information of compound **3d** (NMR 1H, 13C, HRMS) | p. 12 |
| Spectral information of compound **3e** (NMR 1H, 13C, HRMS) | p. 14 |
| Spectral information of compound **3f** (NMR 1H, 13C, HRMS)  Spectral information of compound **3g** (NMR 1H, 13C, HMRS)  Spectral information of compound **3h** (NMR 1H, 13C, HMRS)  Spectral information of compound **3h** (NMR 1H, 13C, HMRS) | p. 16  p. 18  p. 20  p. 22 |

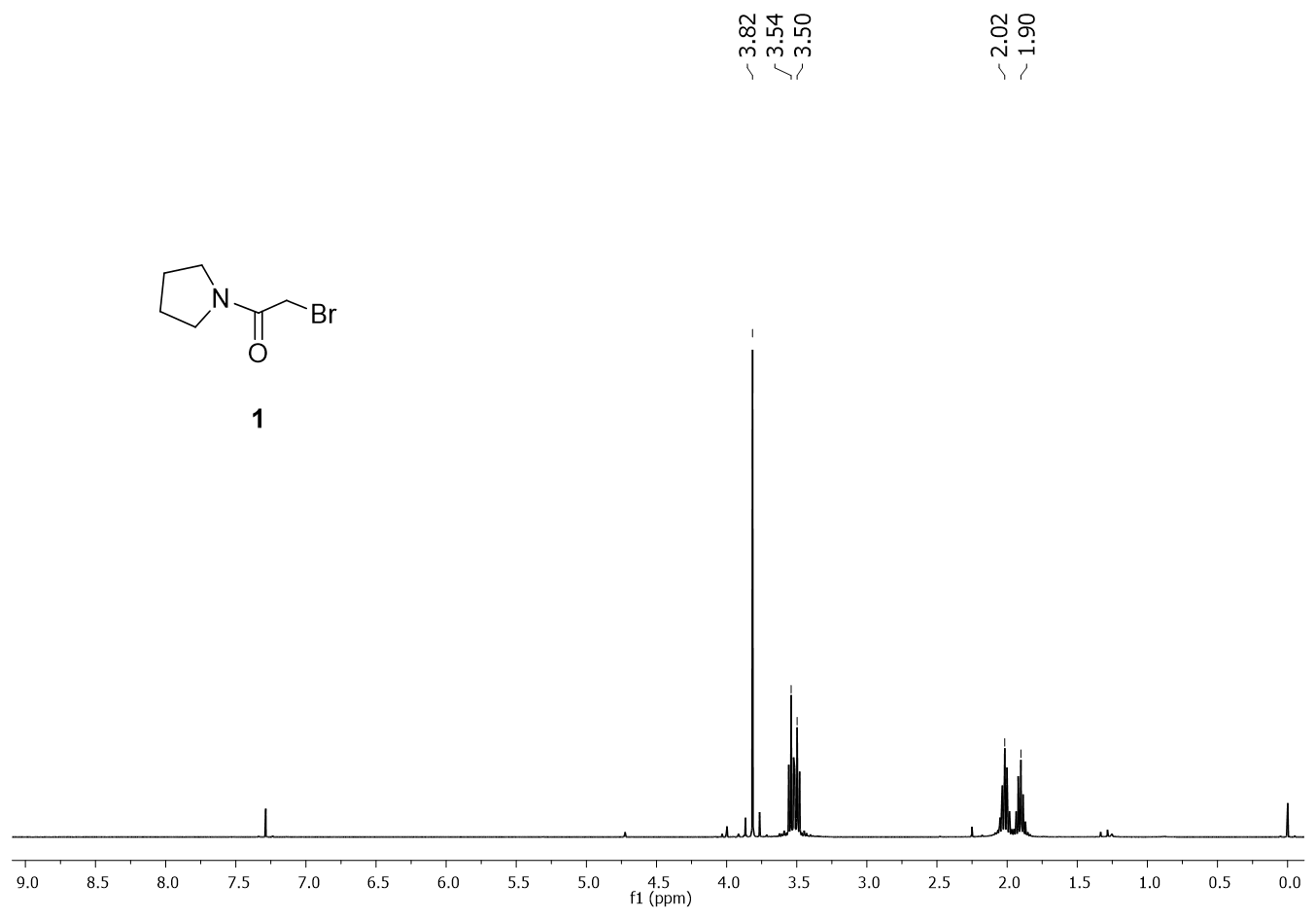
****

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

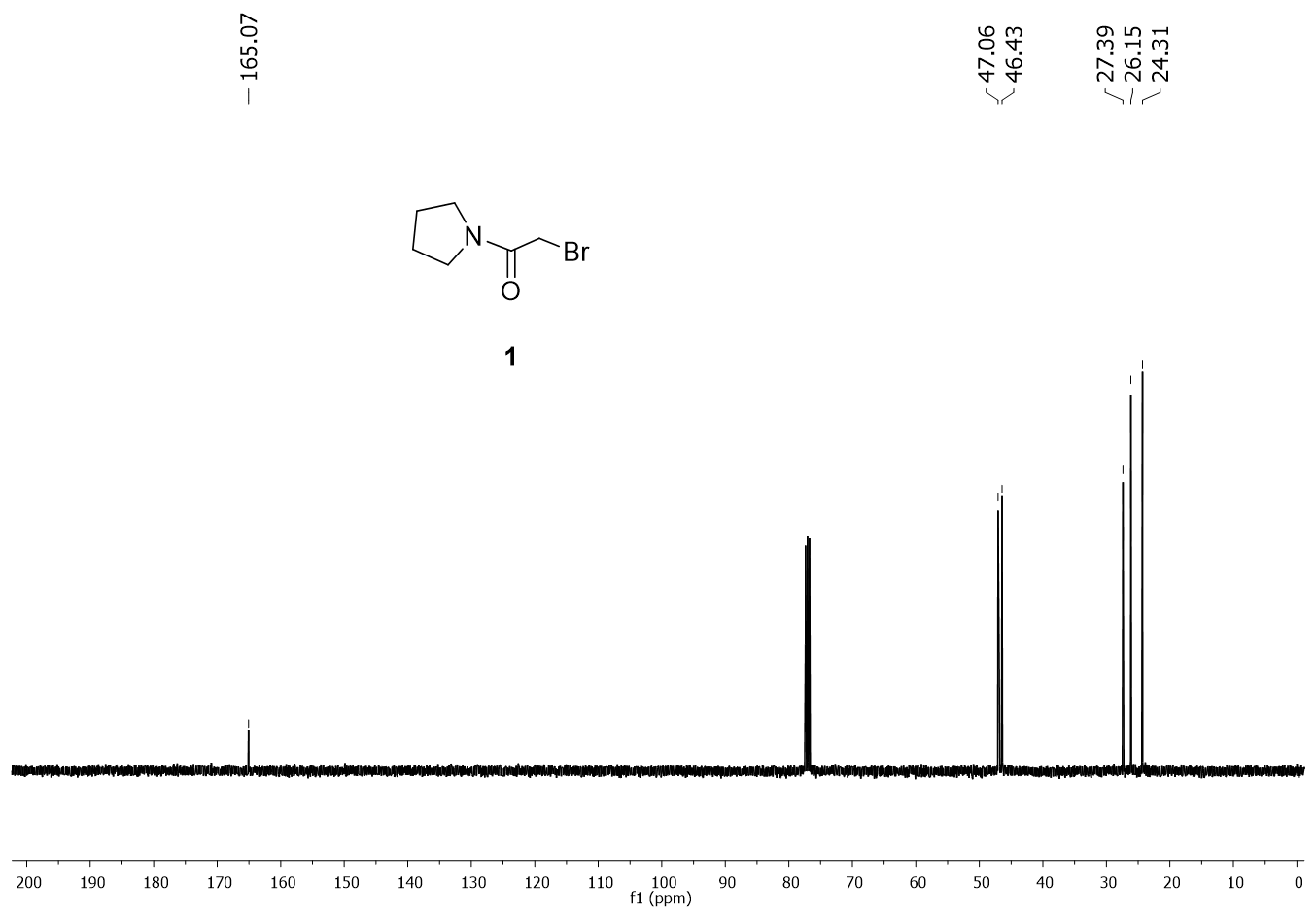
****

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

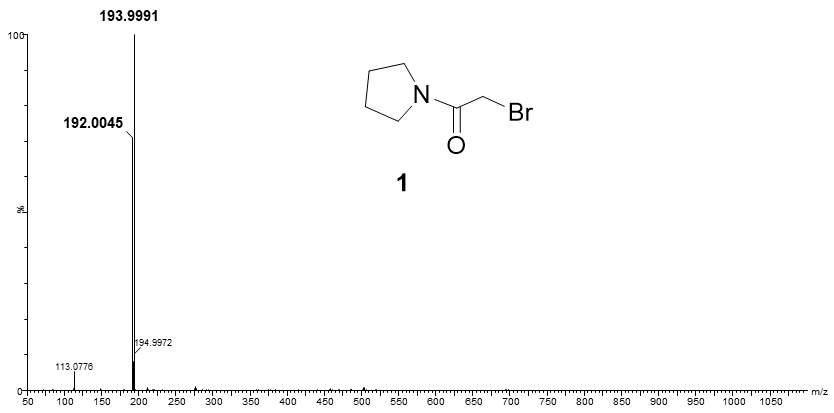
****

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

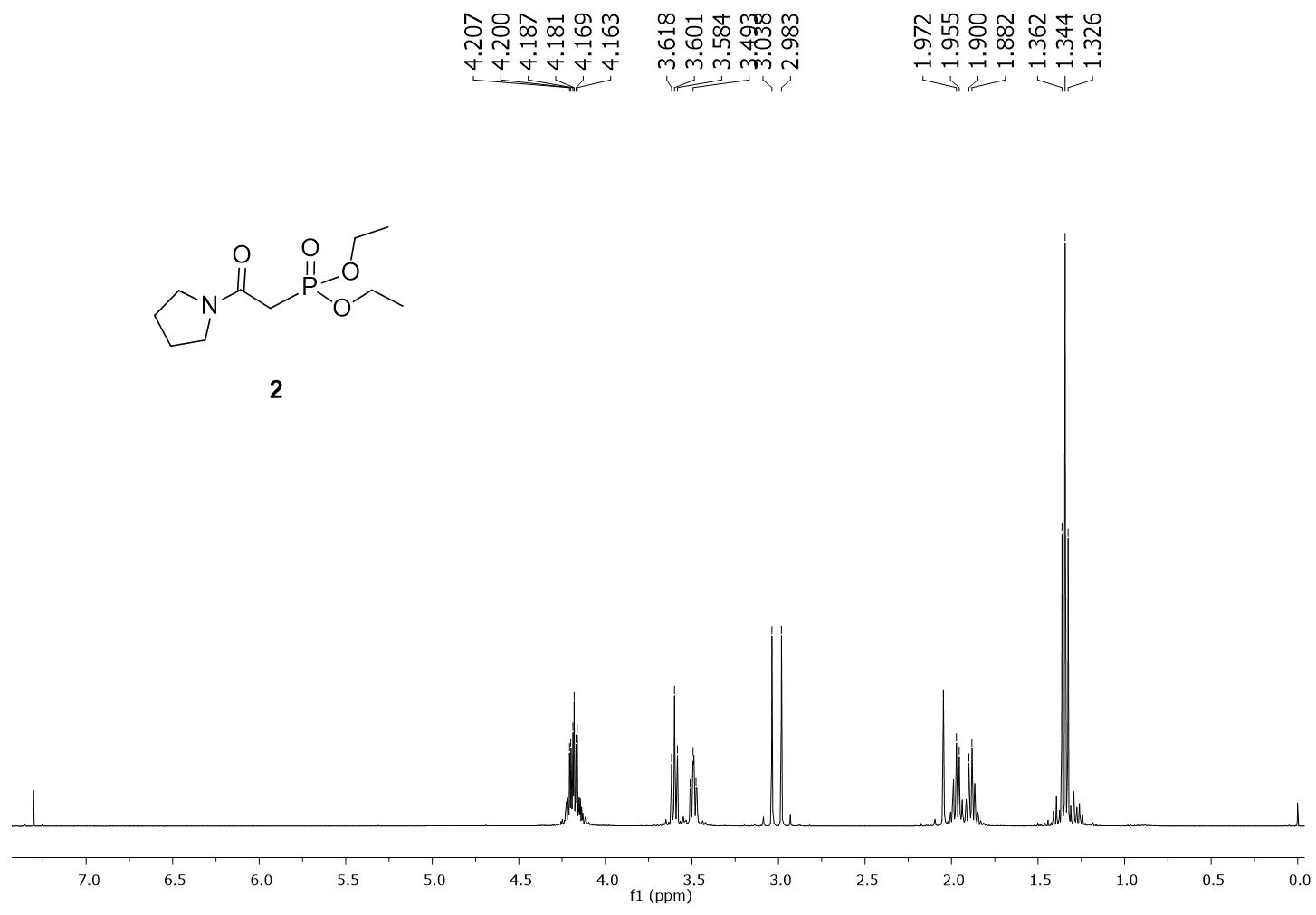
****

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

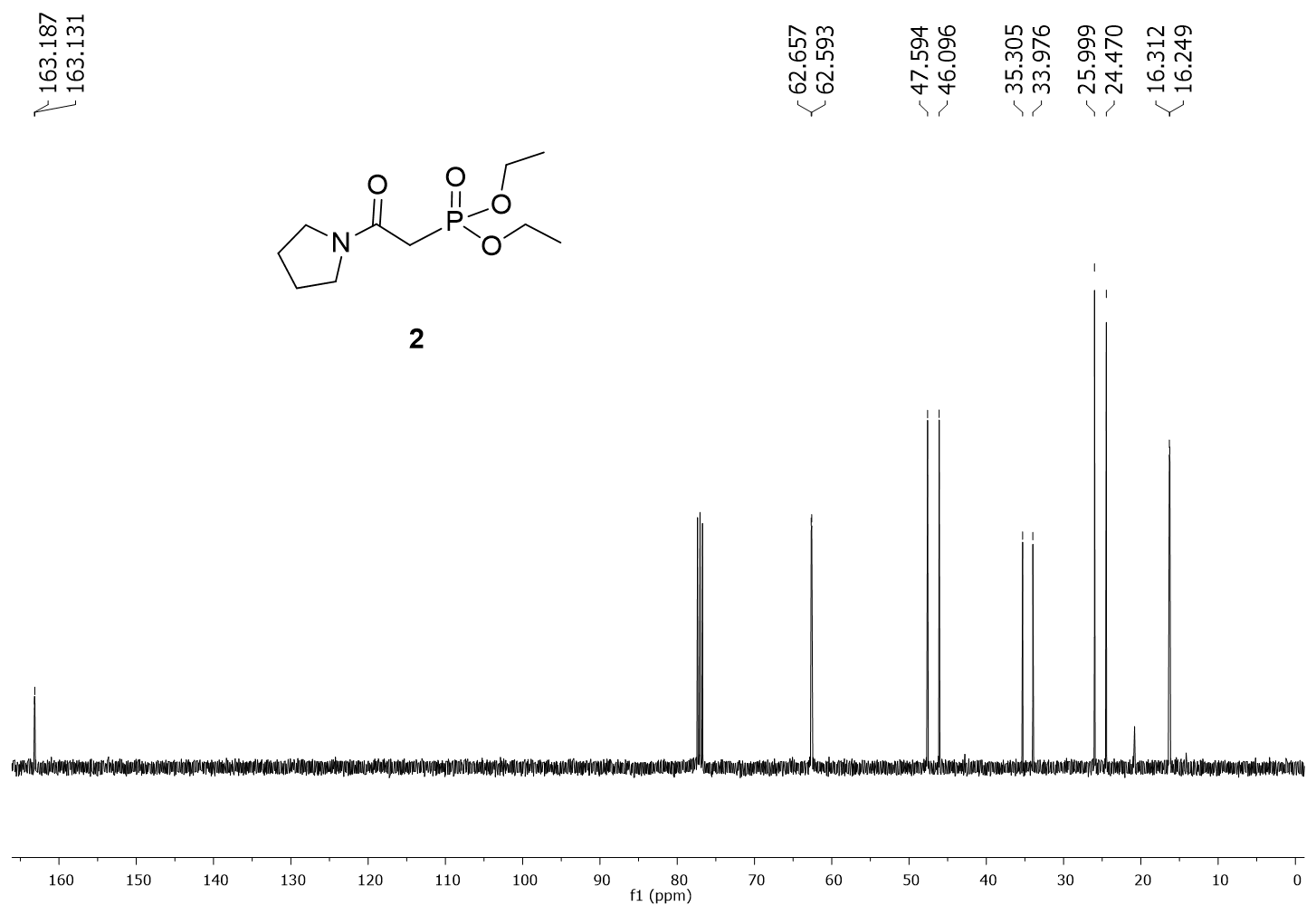
****

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

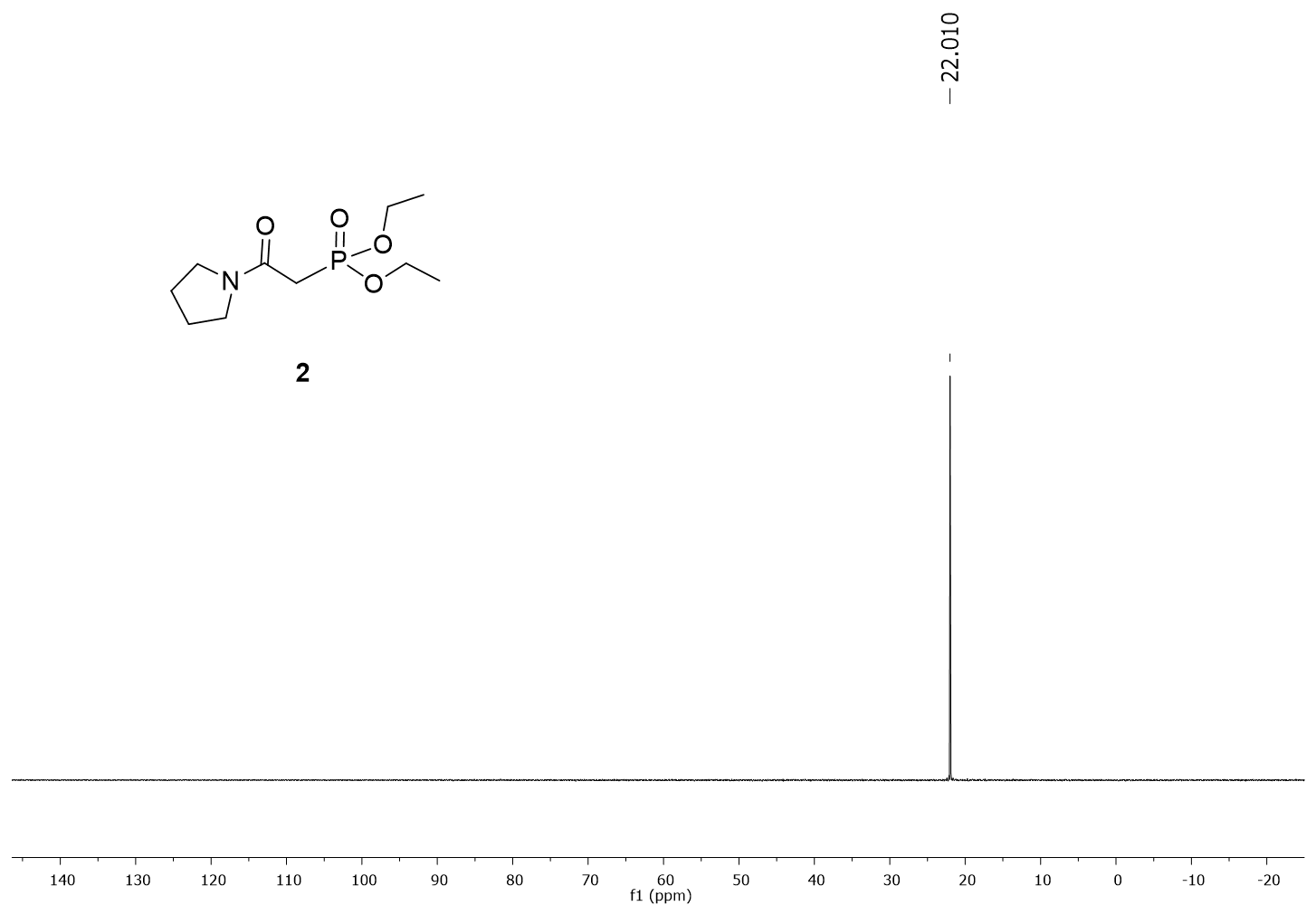
****

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

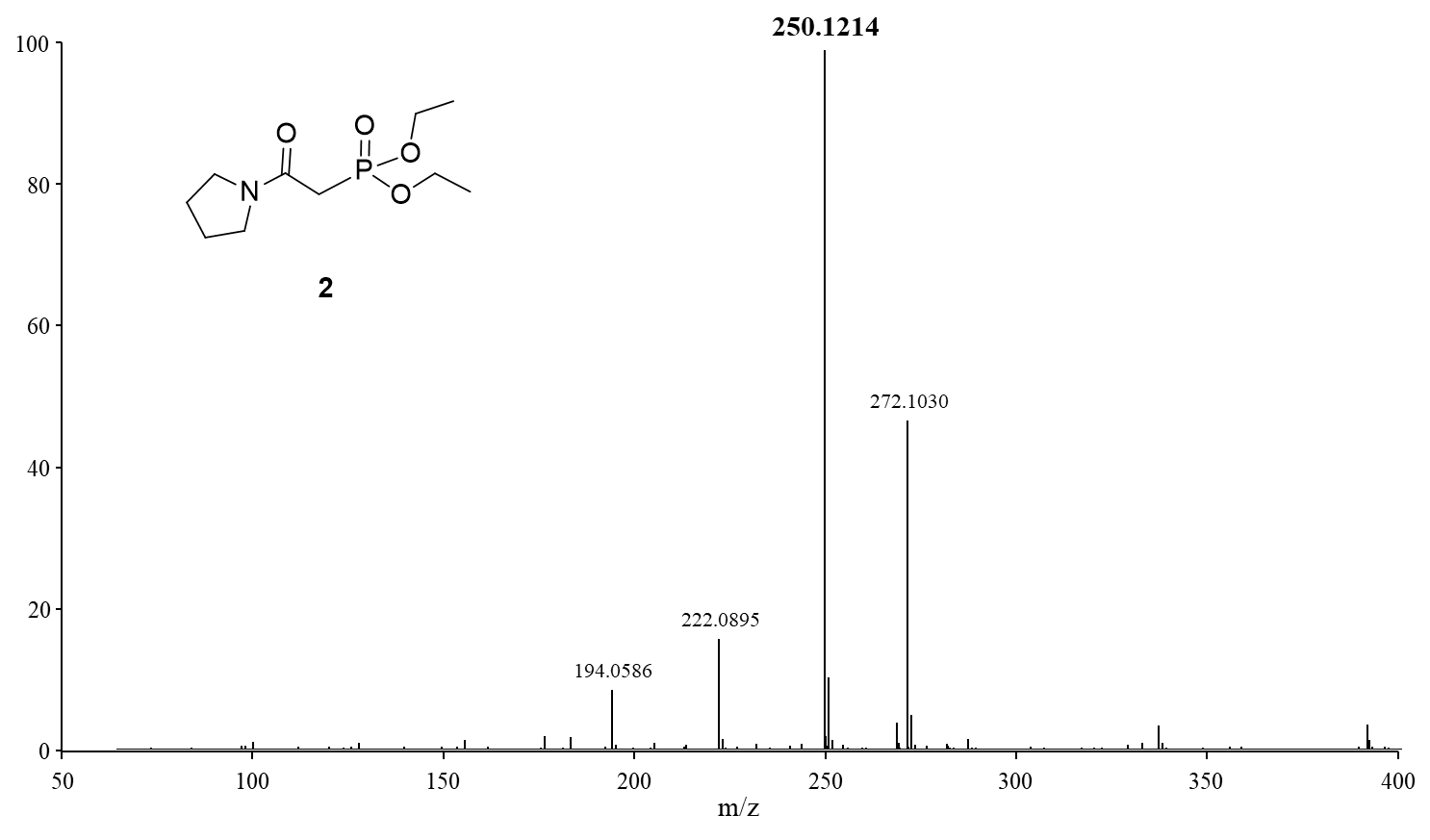


Fig. 7 HRMS spectra of *diethyl (2-oxo-2-(pyrrolidin-1-yl)ethyl)phosphonate* **2.**

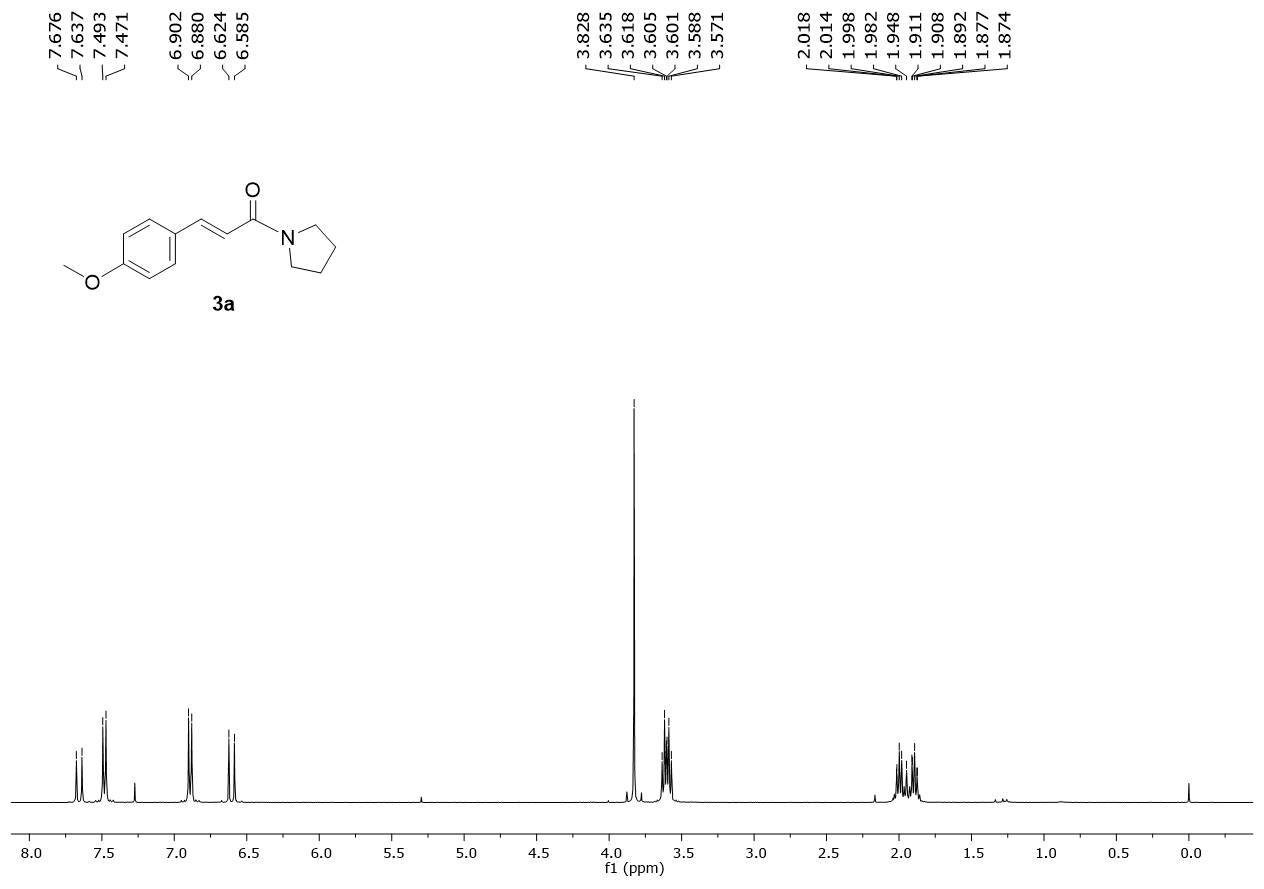
****

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

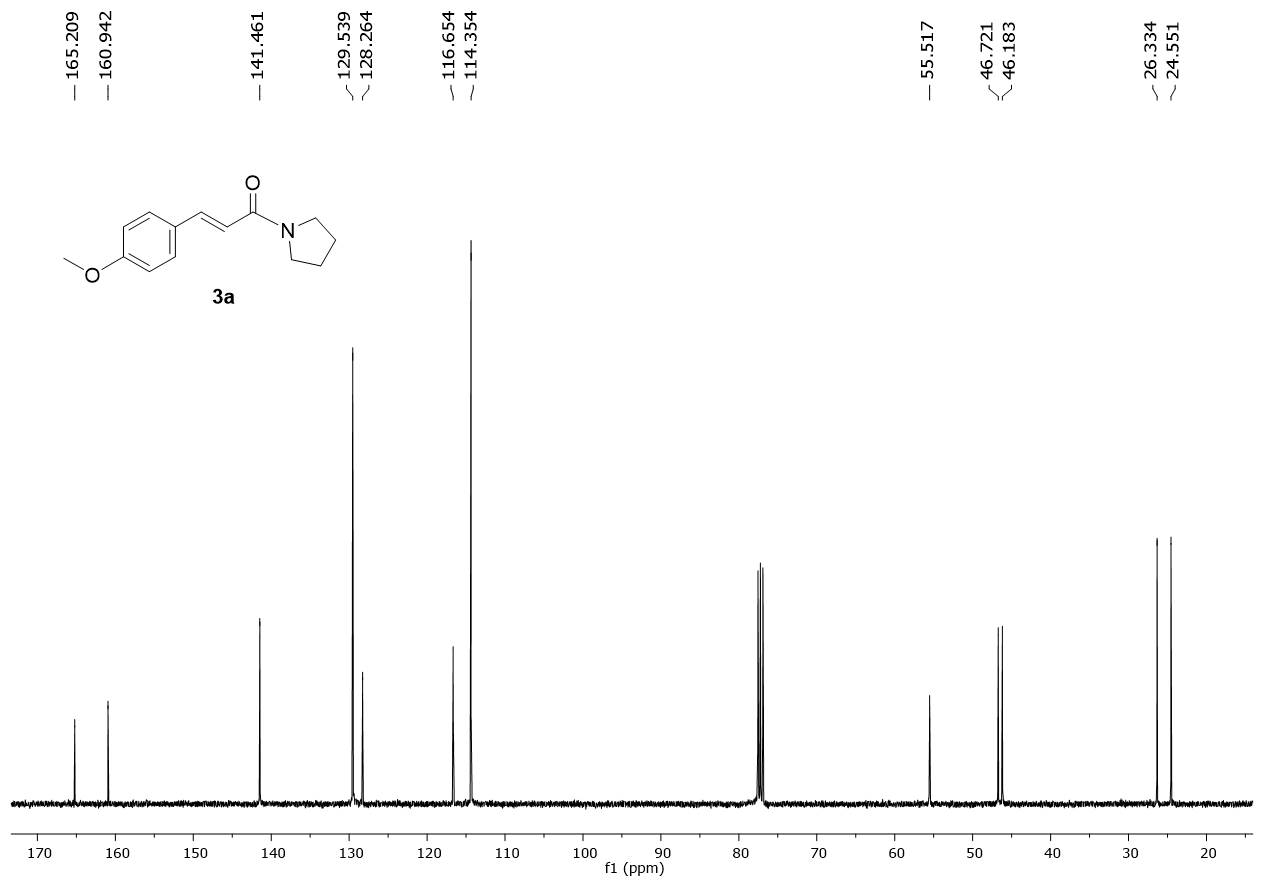


Fig. 9 13C NMR spectra of *(E)-3-(4-methoxyphenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one*  **3a.**

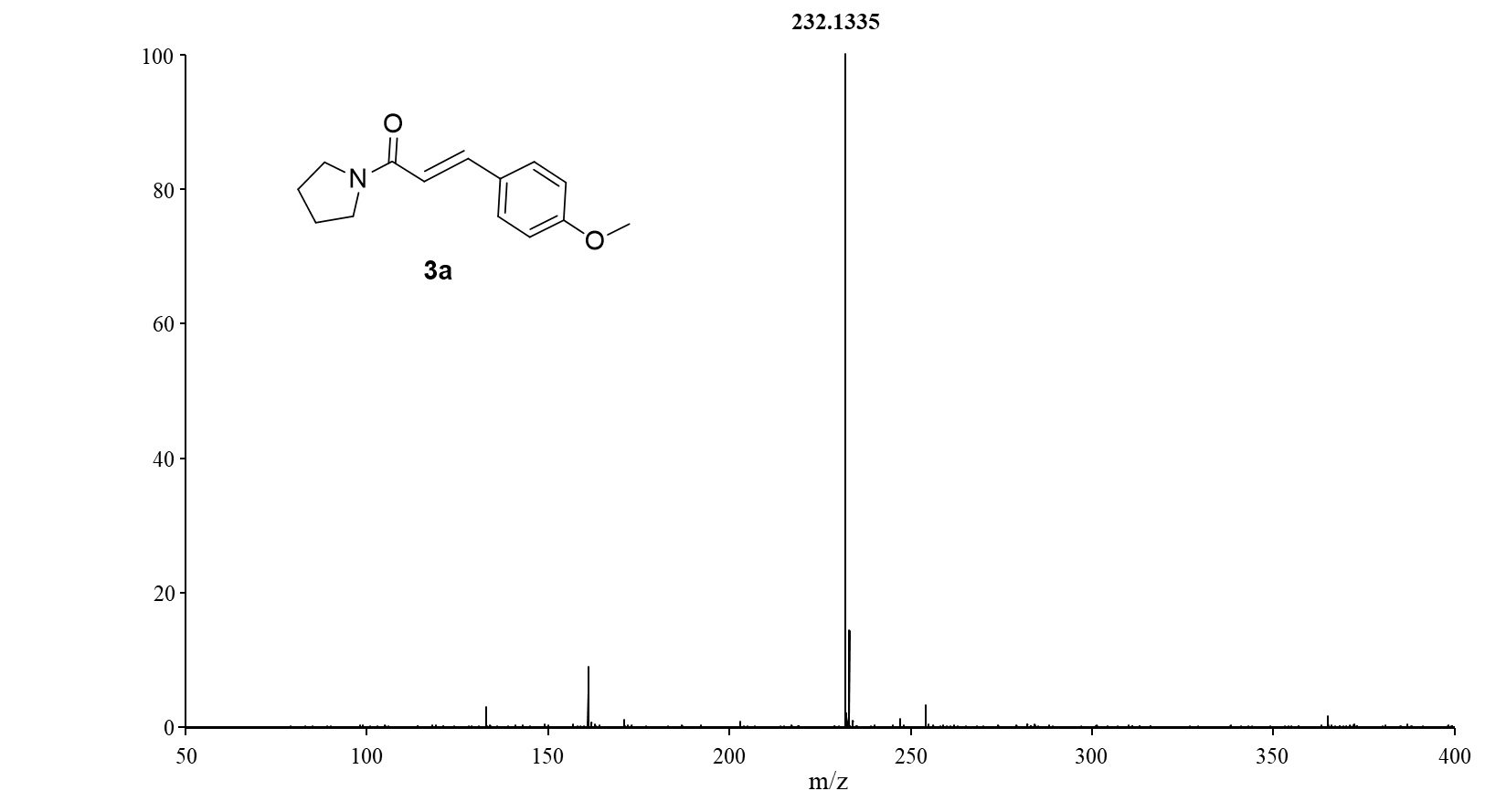
****

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

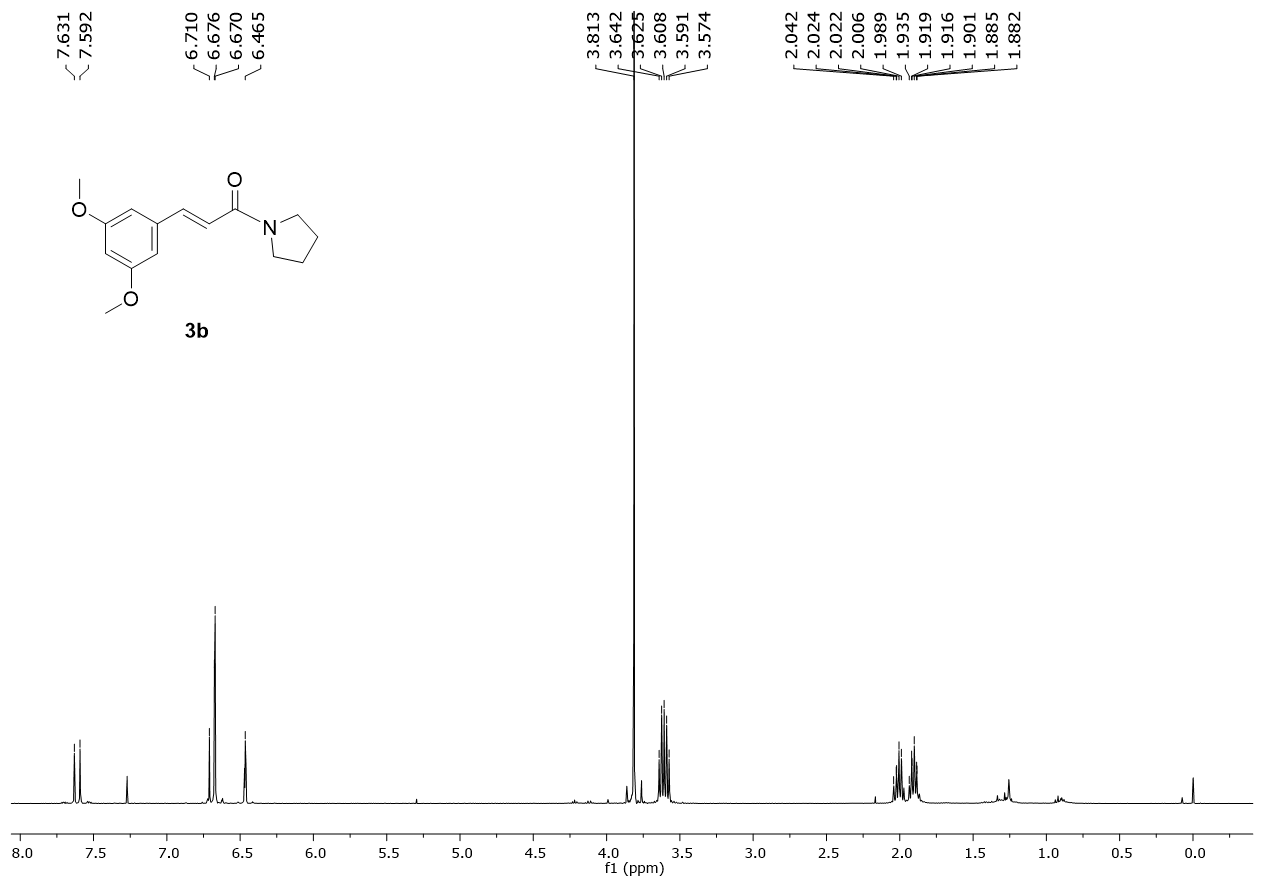
****

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

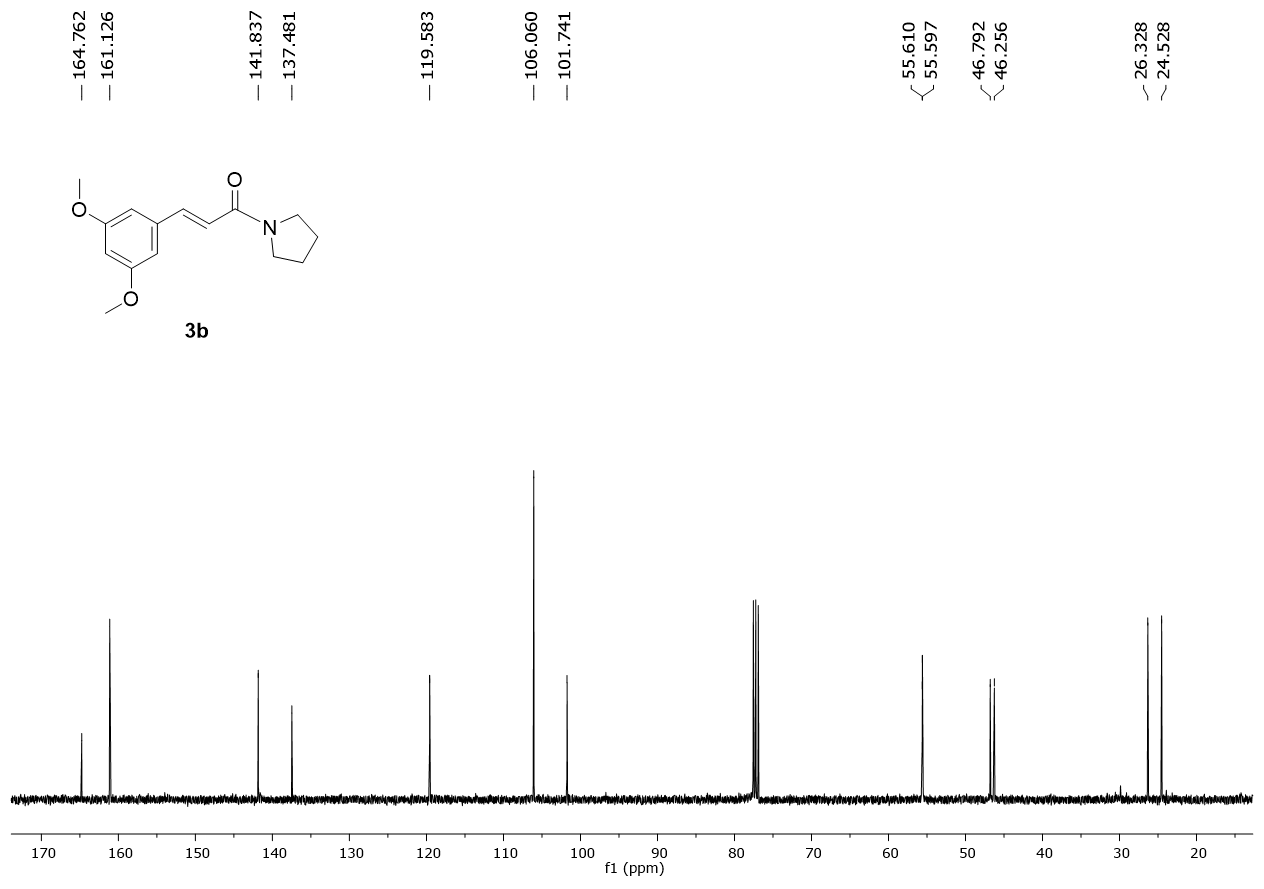
****

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

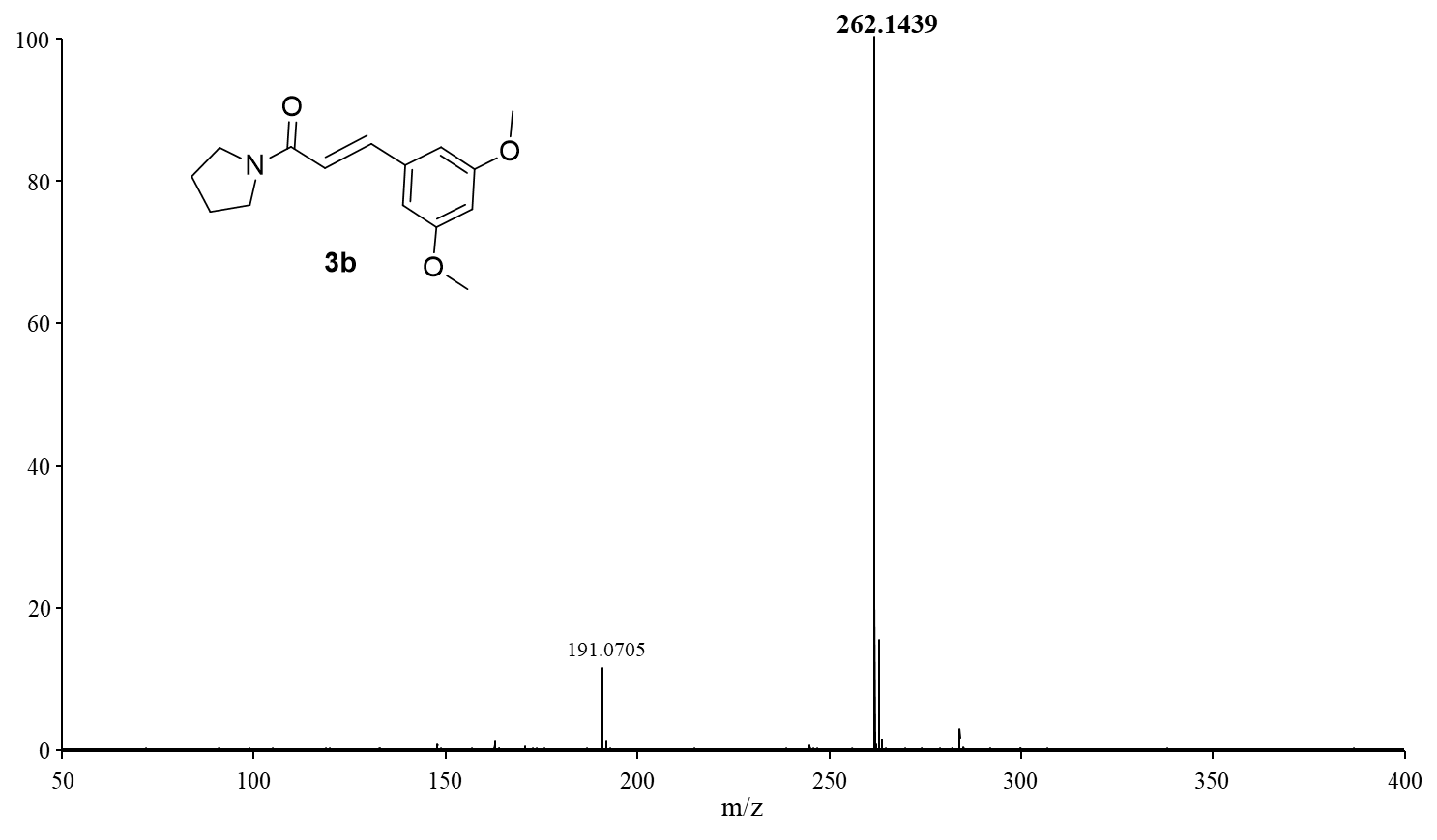
****

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

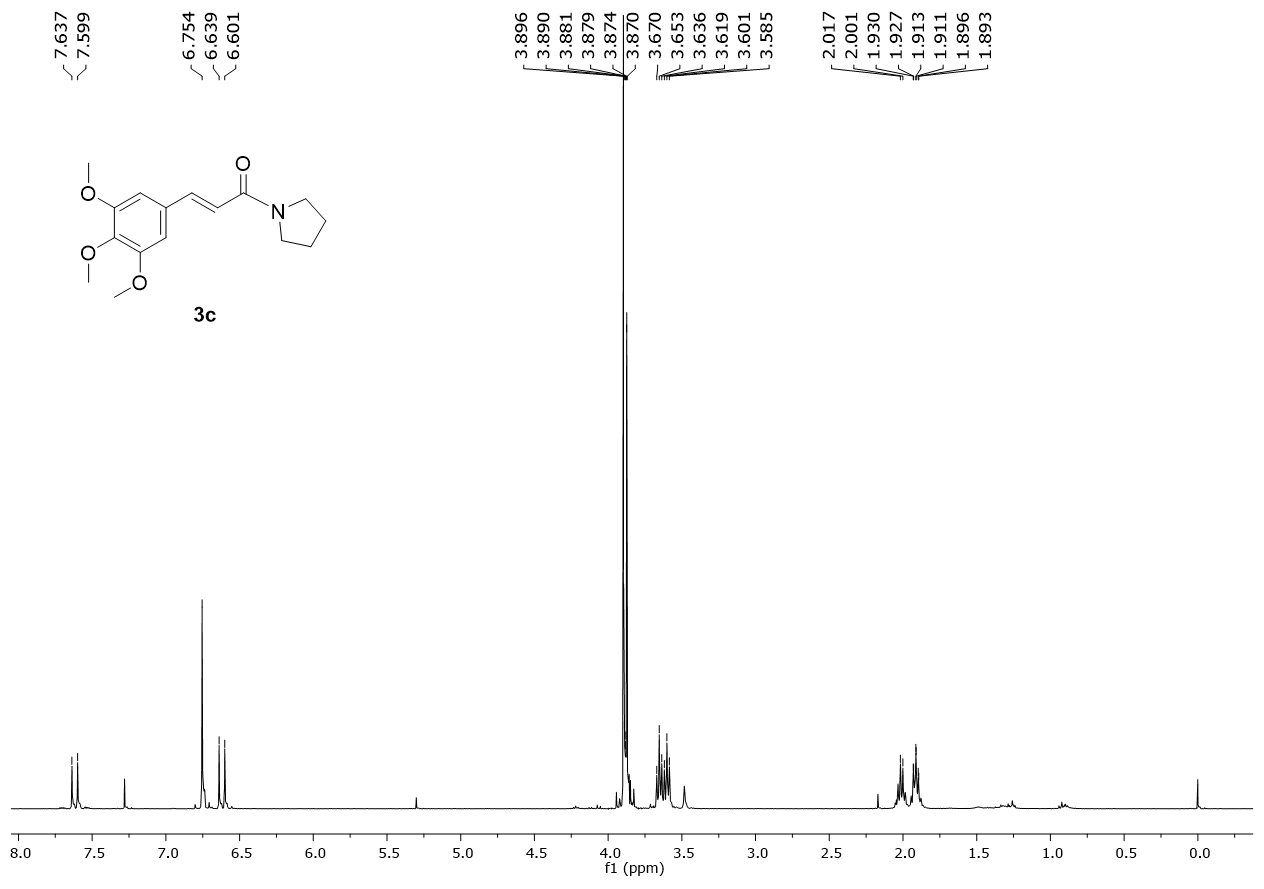
****

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

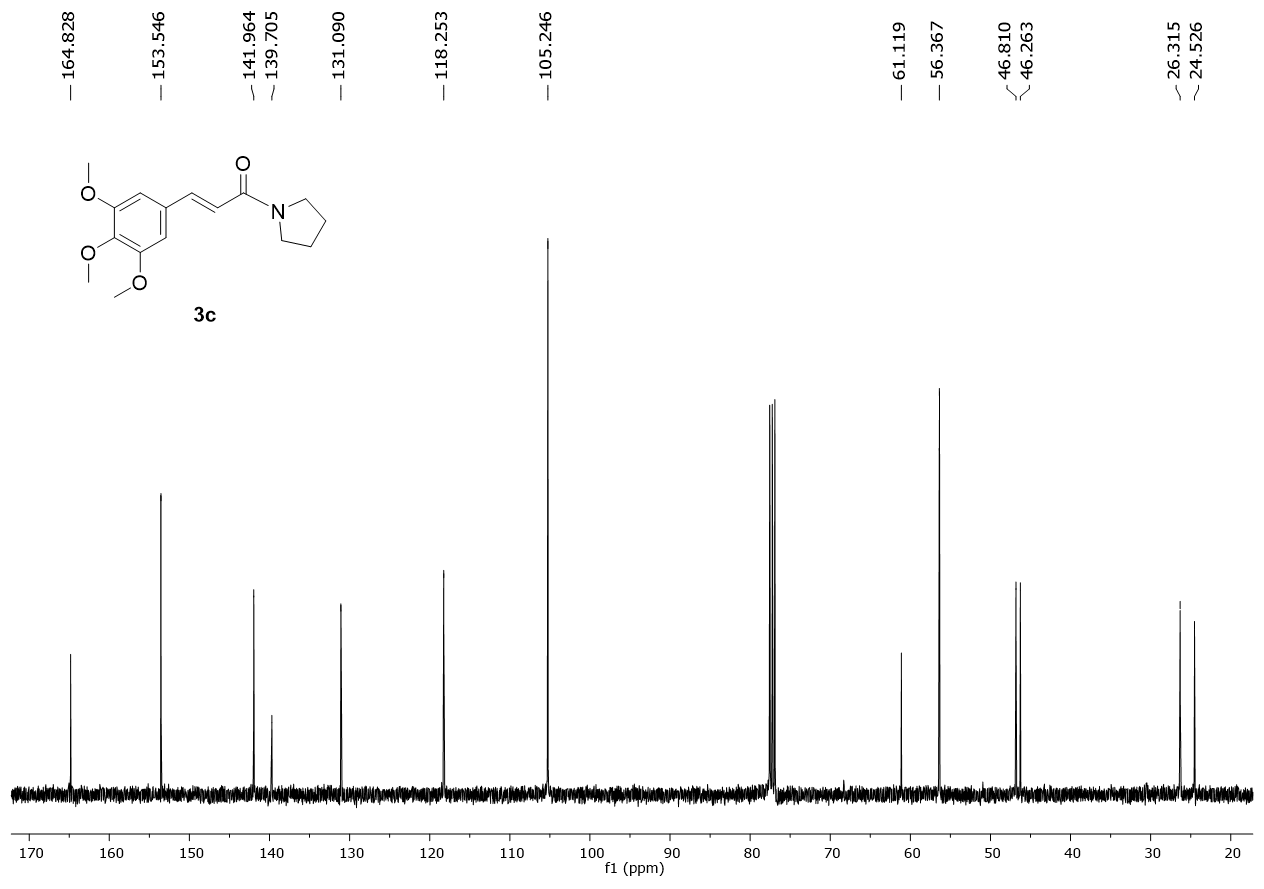
****

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

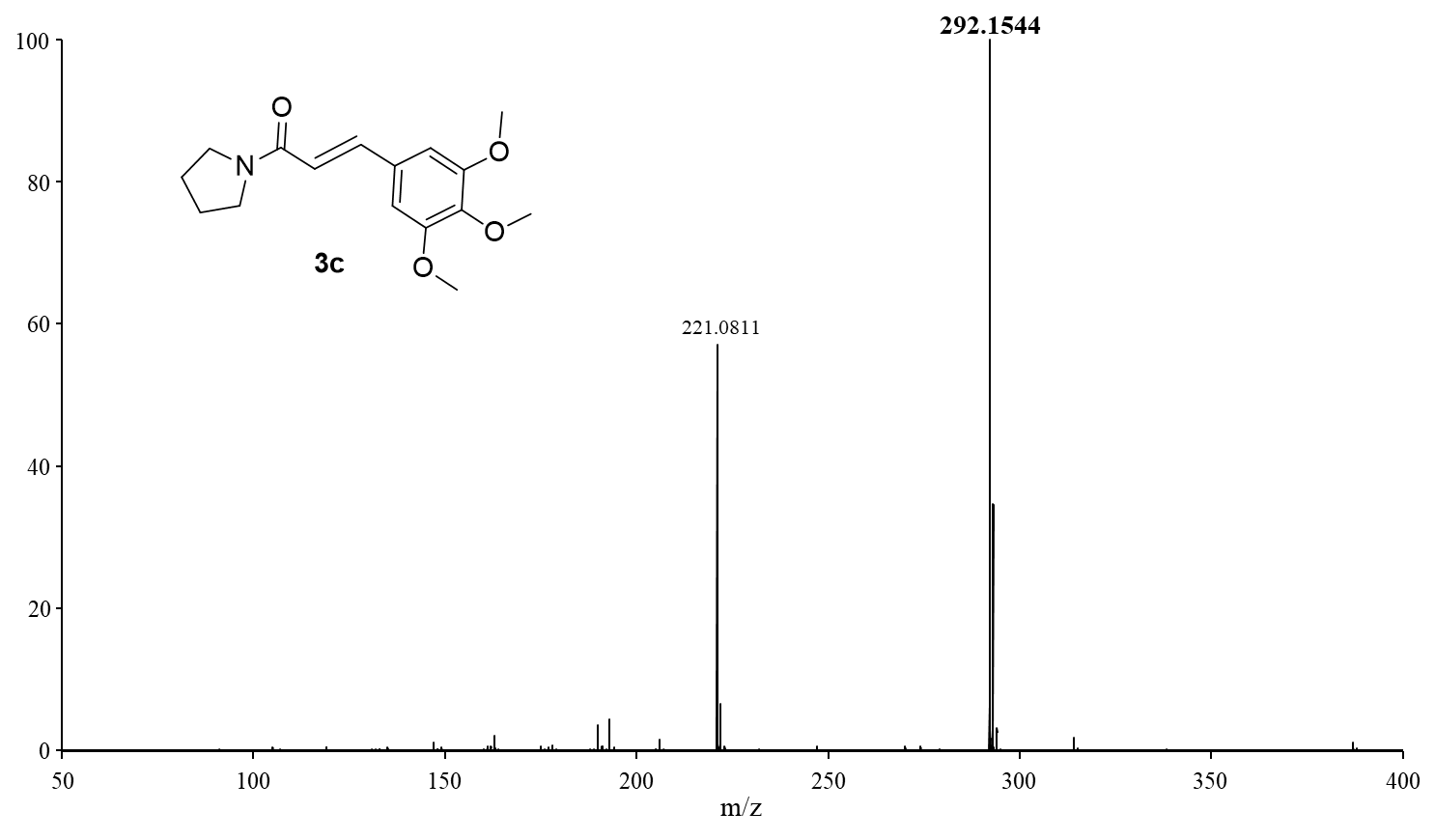
****

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

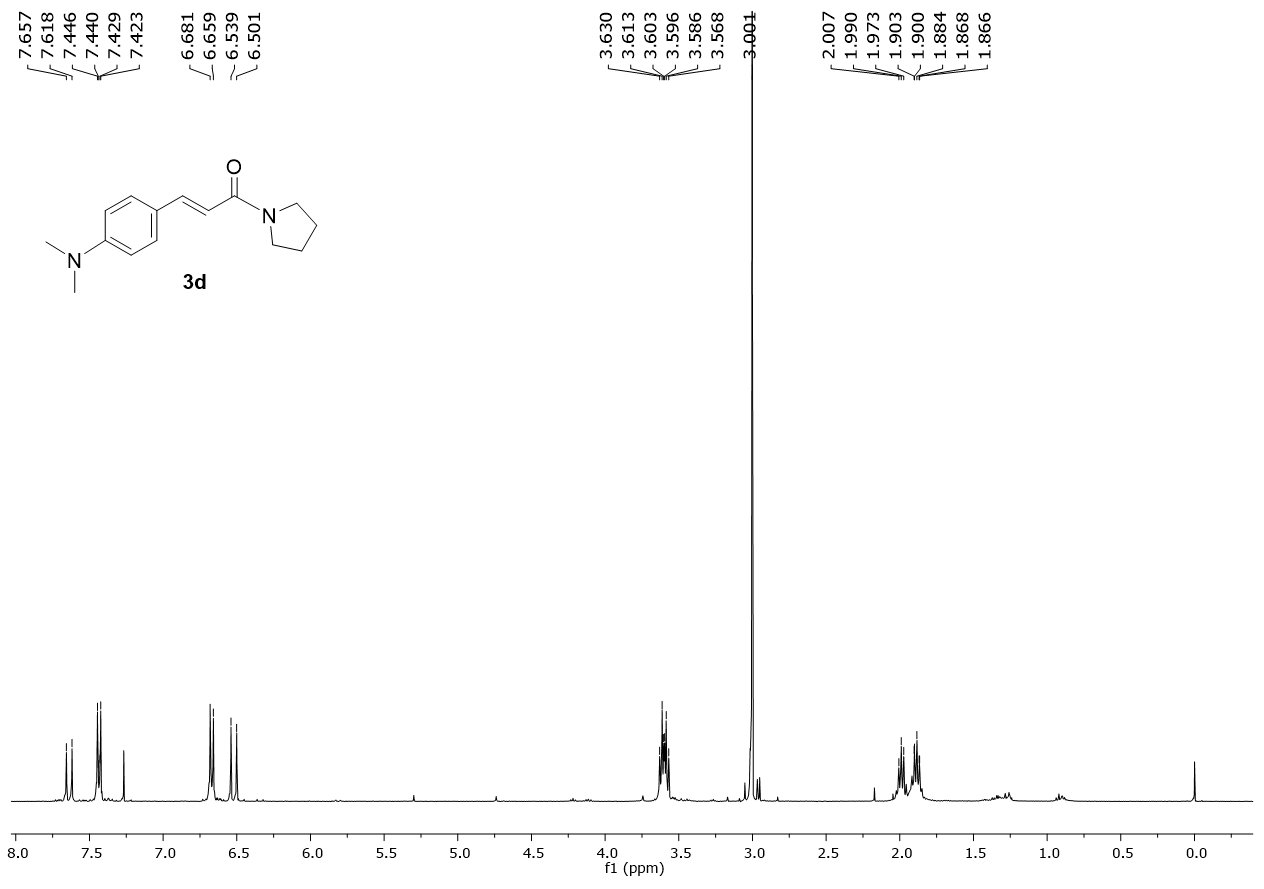


Fig. 17 1H NMR spectra of *(E)-3-(4-(dimethylamino)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3d.**

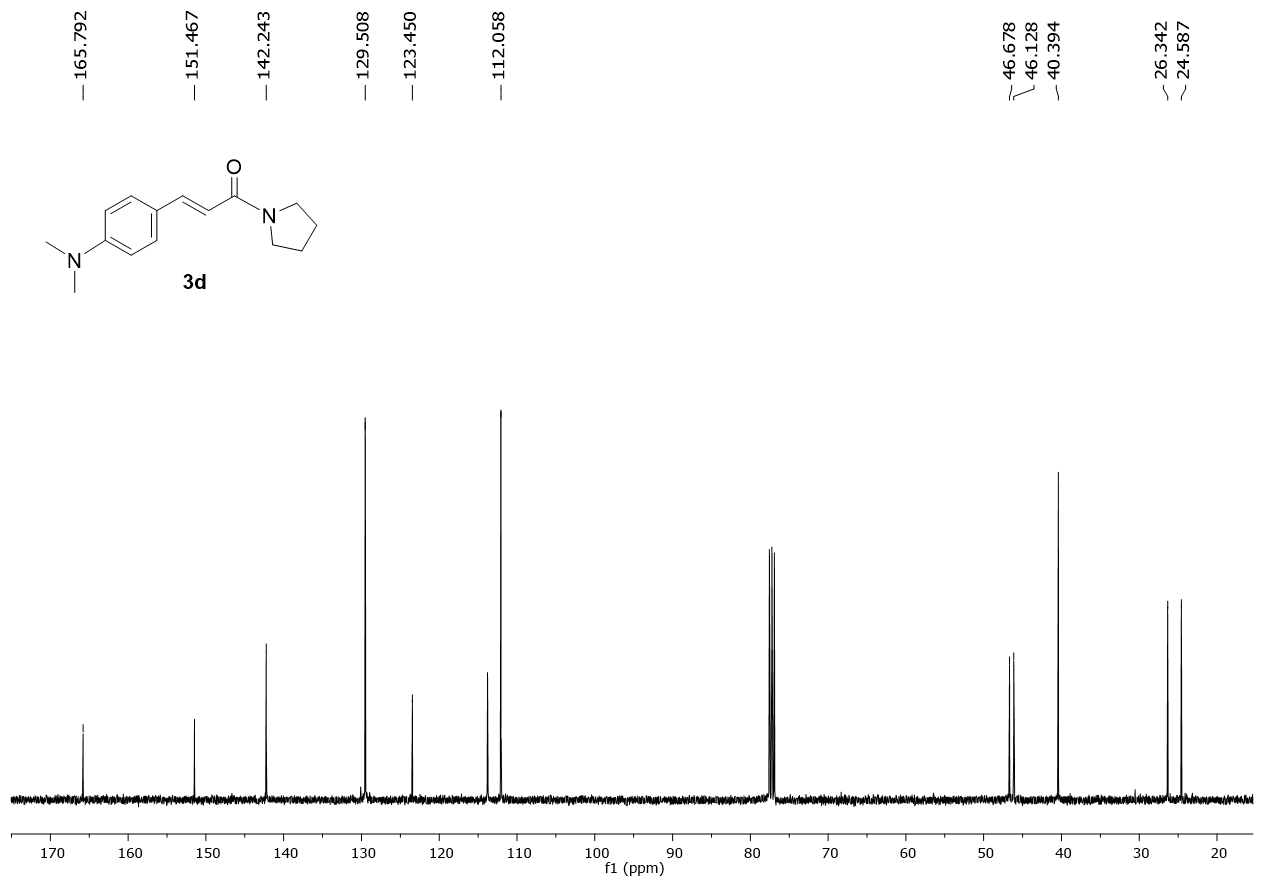


Fig. 18 13C NMR spectra of *(E)-3-(4-(dimethylamino)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3d.**

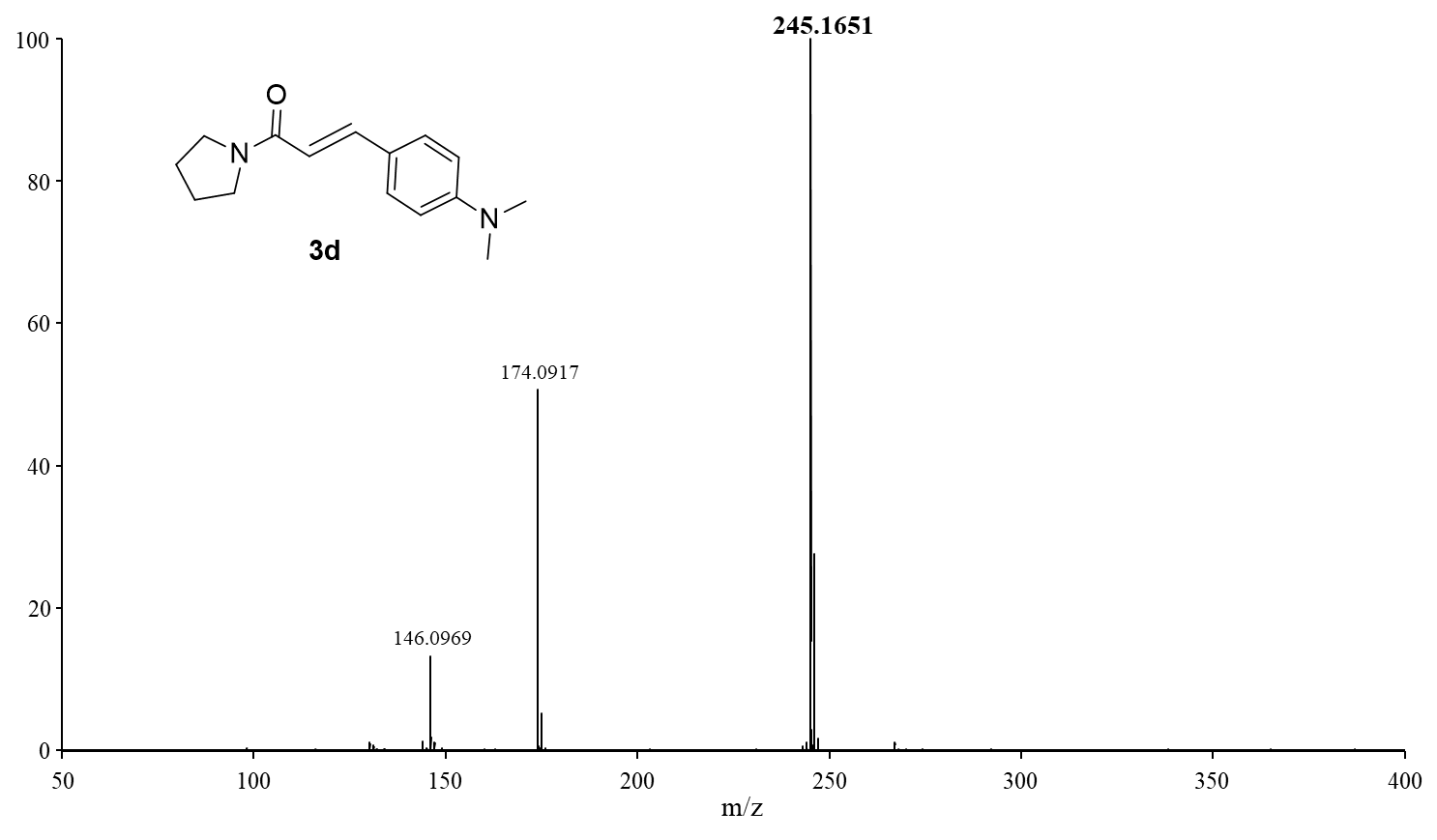
****

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

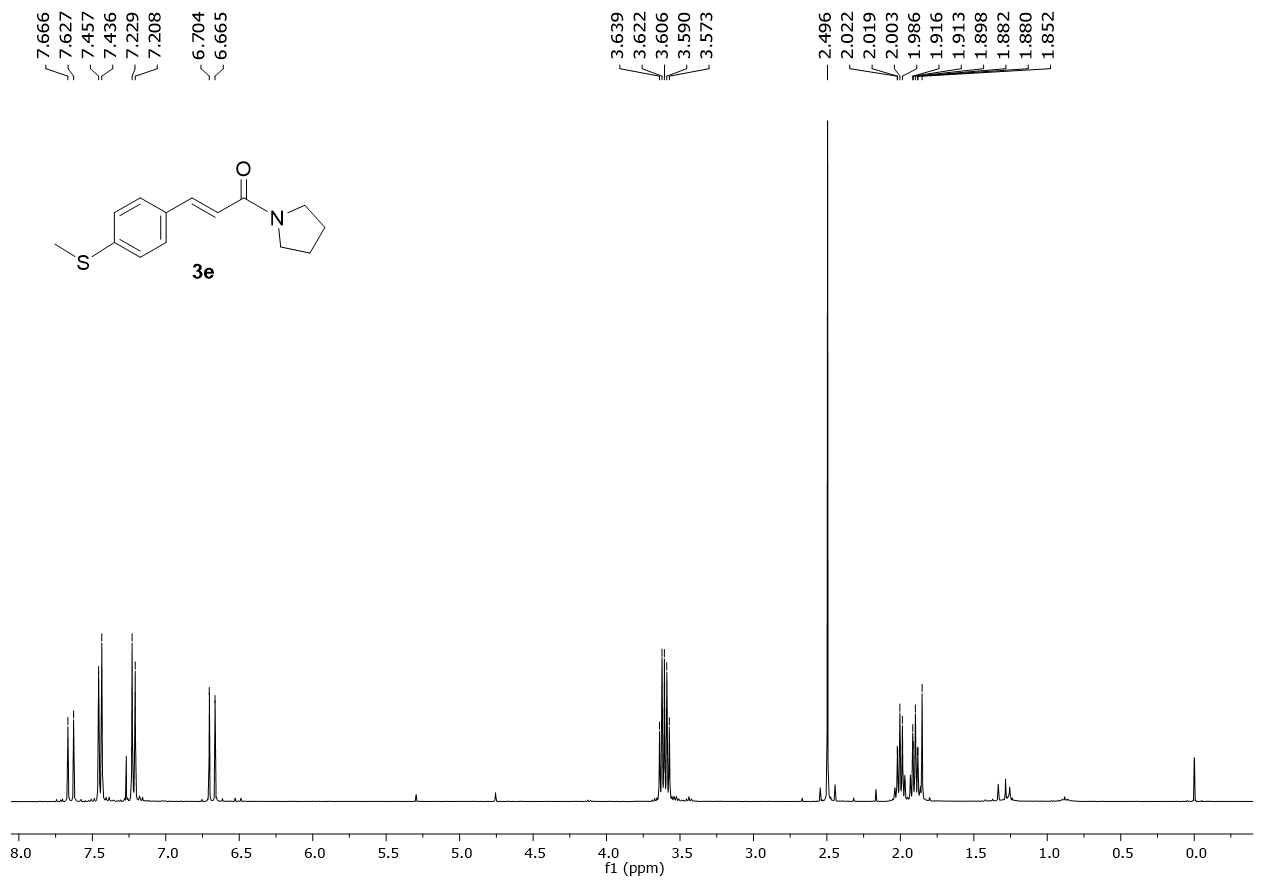


Fig. 20 1H NMR spectra of *(E)-3-(4-(methylthio)phenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3e.**

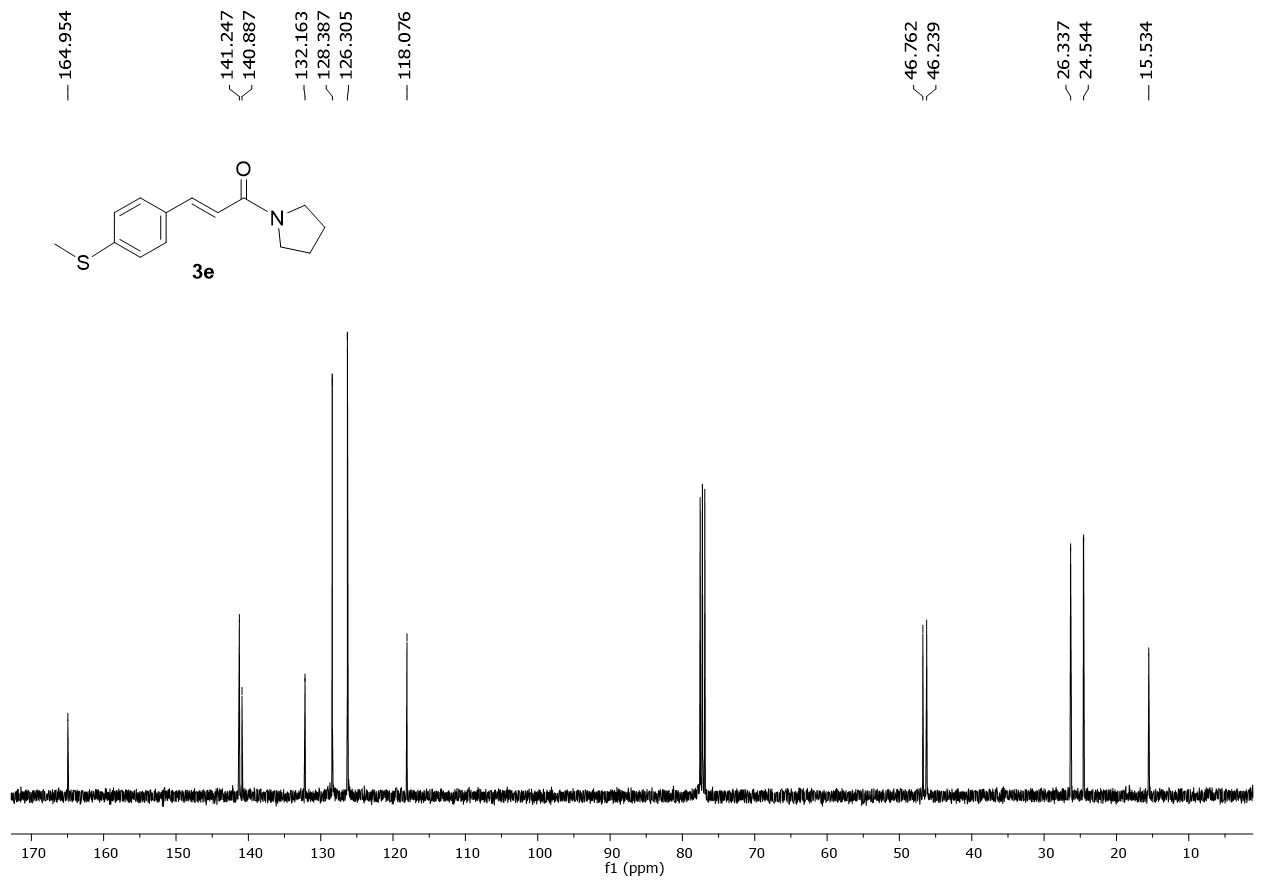
****

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

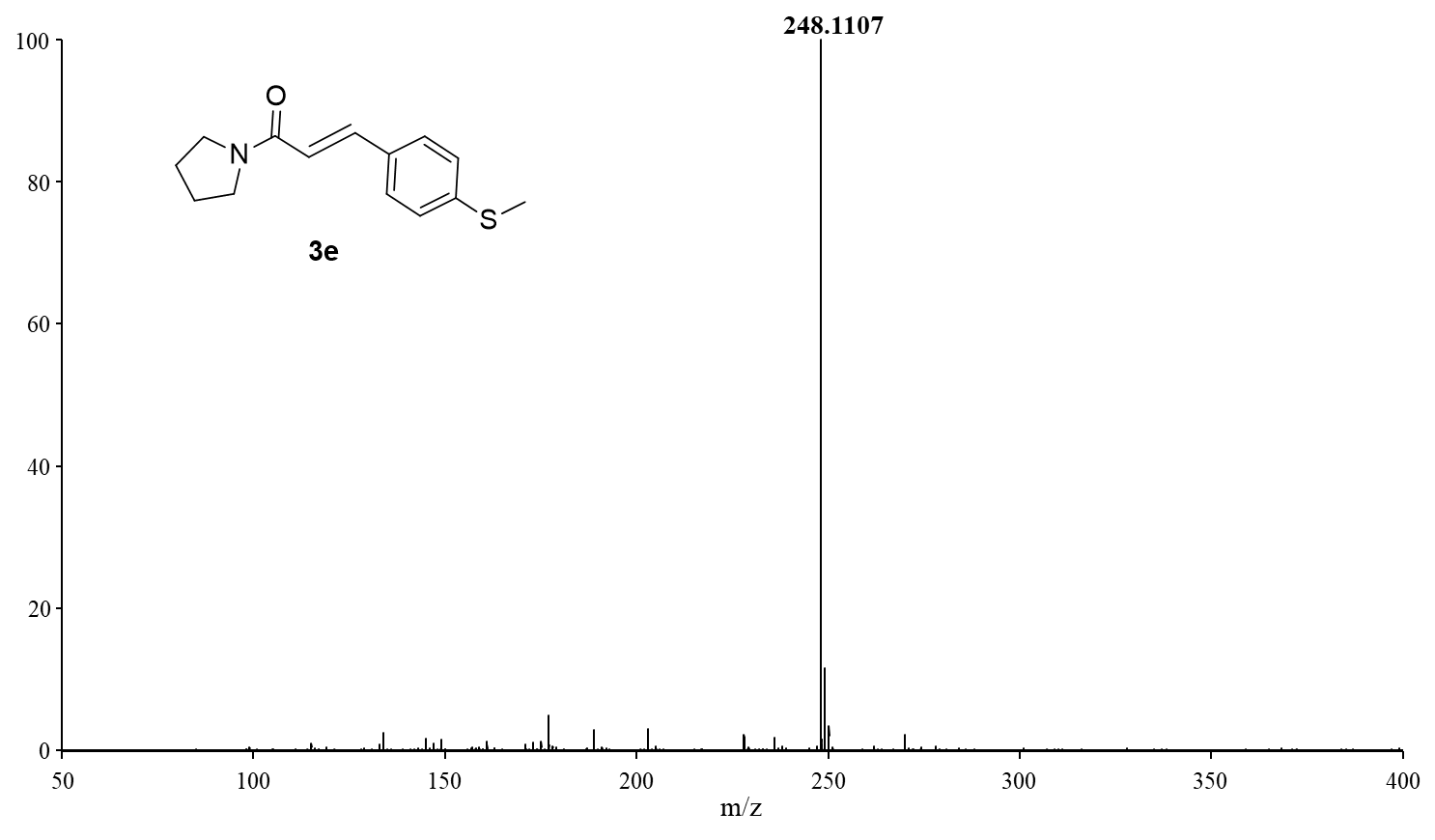
****

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

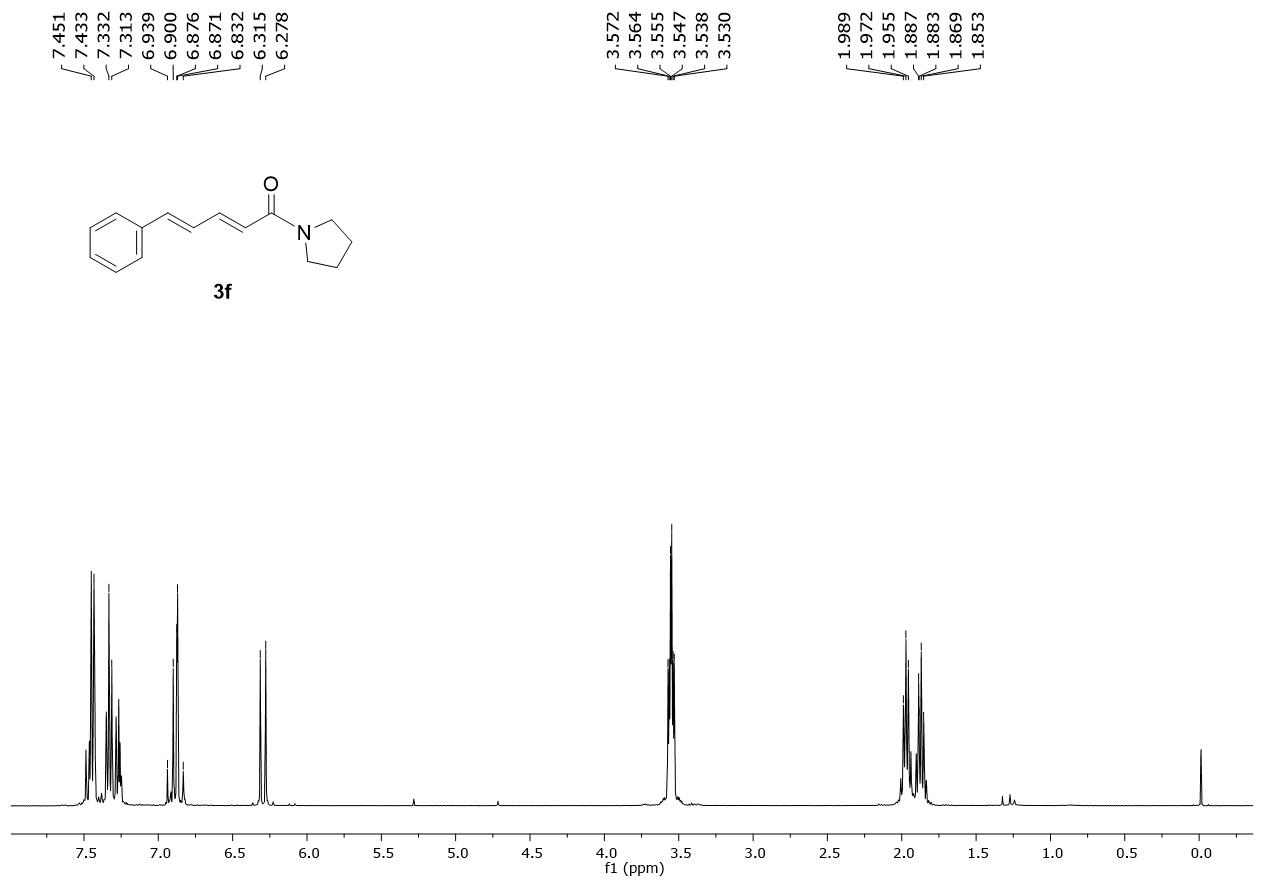


Fig. 23 1H NMR spectra of *(2E,4E)-5-phenyl-1-(pyrrolidin-1-yl)penta-2,4-dien-1-one* **3f.**

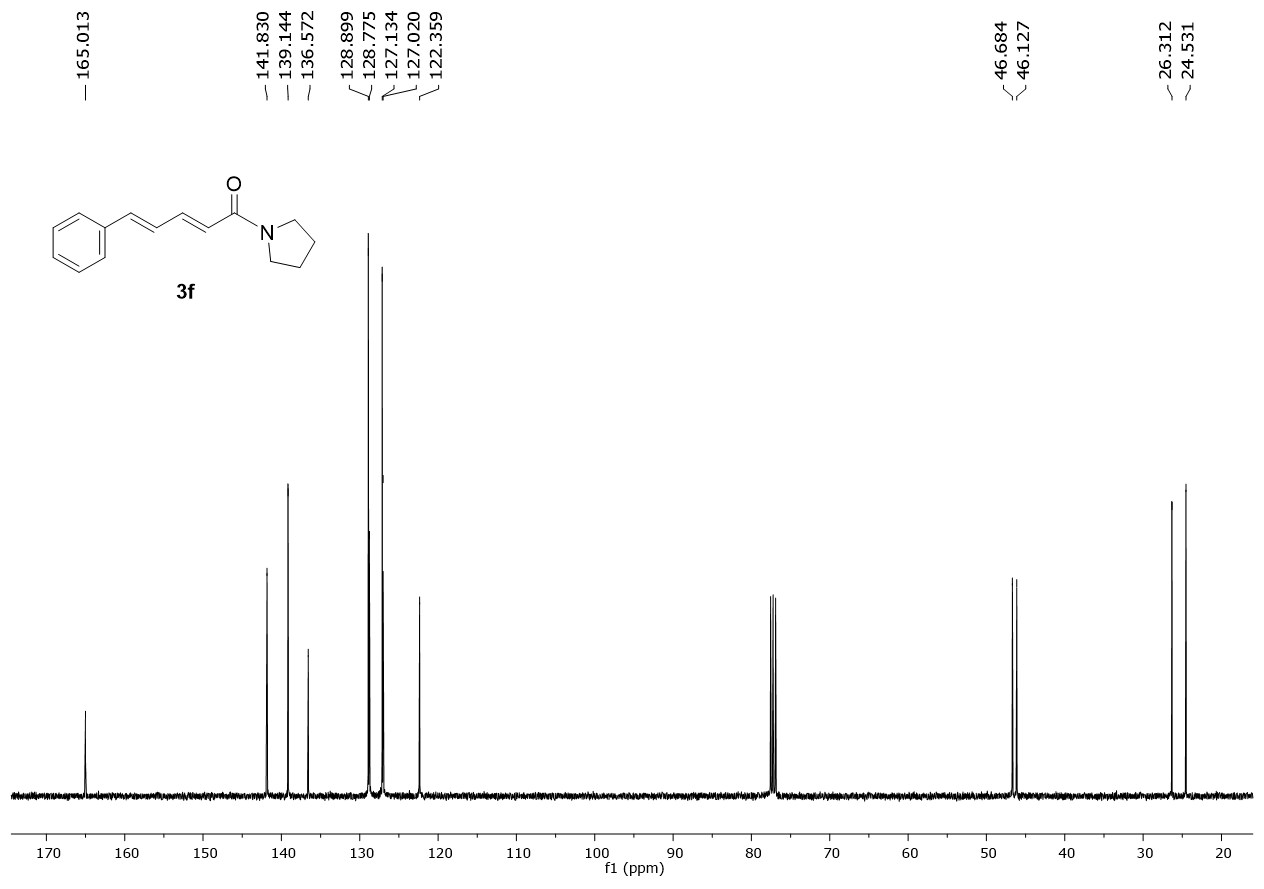


Fig. 24 13C NMR spectra of *(2E,4E)-5-phenyl-1-(pyrrolidin-1-yl)penta-2,4-dien-1-one* **3f.**

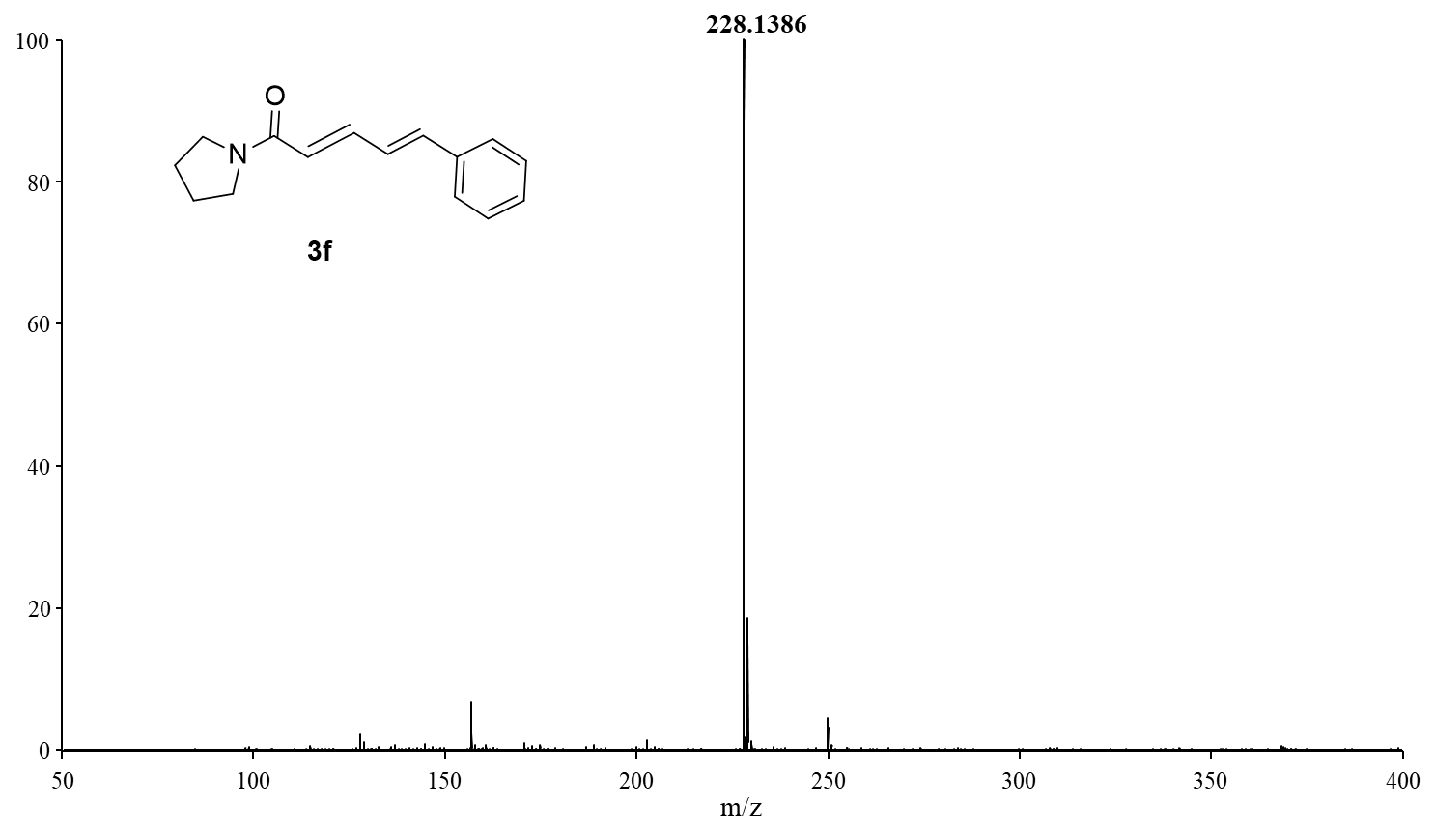
****

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

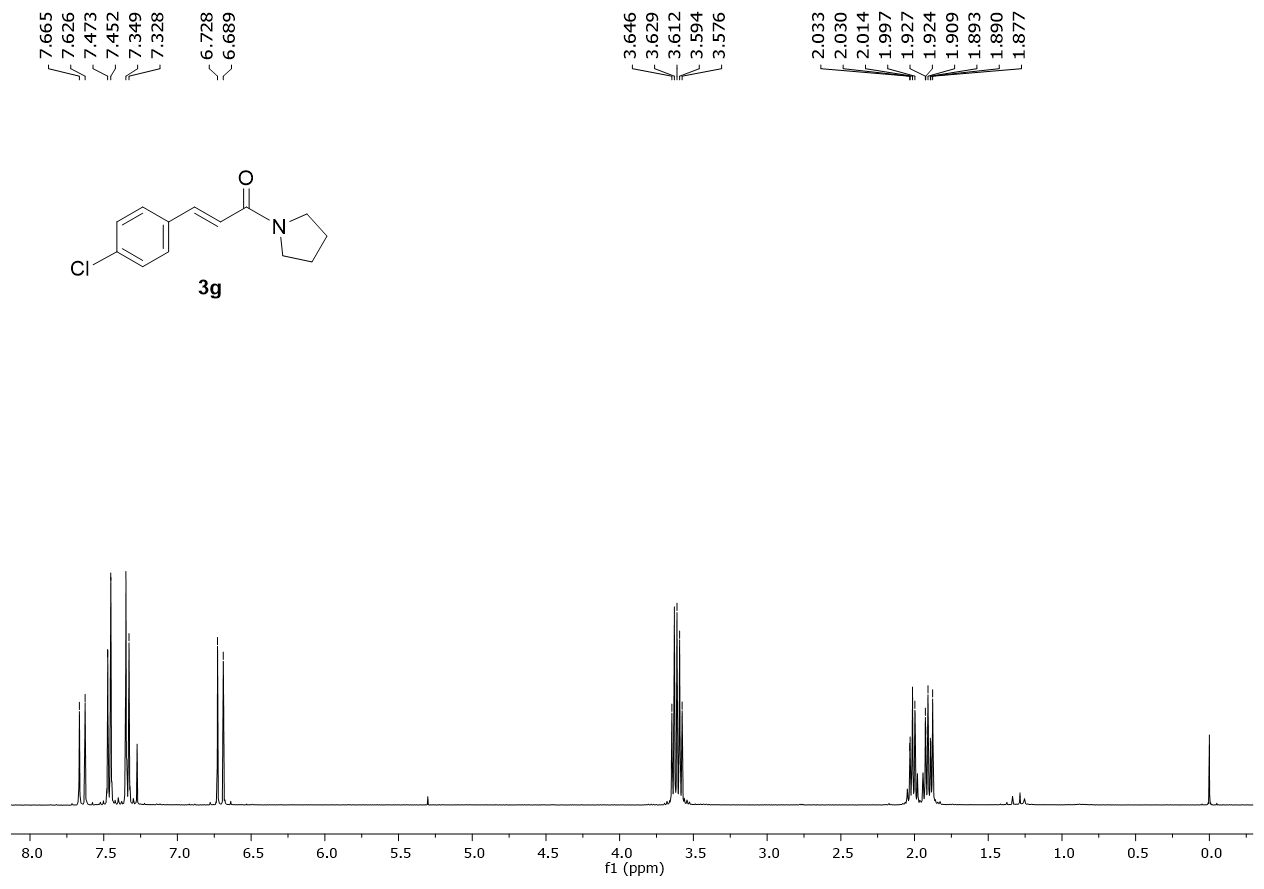


Fig. 26 1H NMR spectra of *(E)-3-(4-chlorophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3g.**

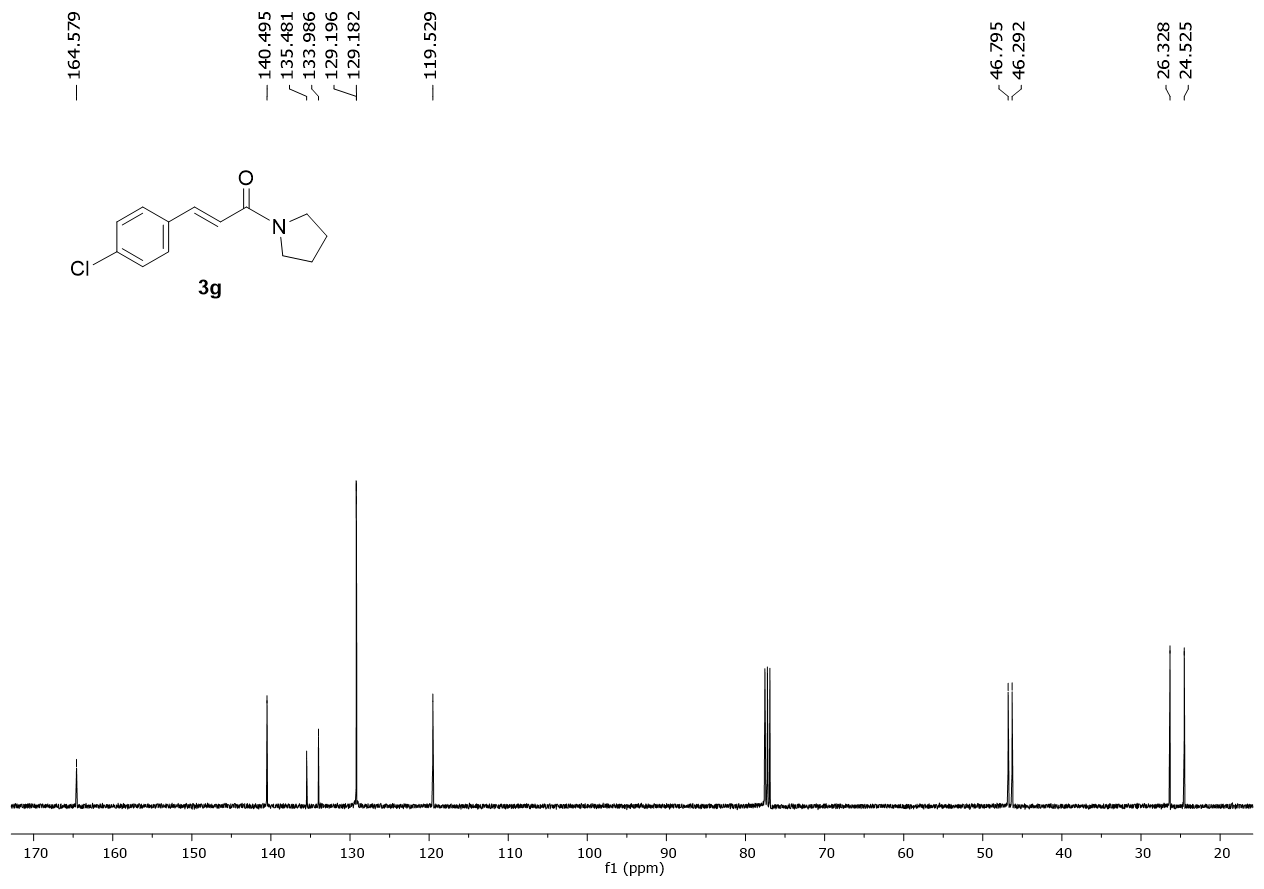


Fig. 27 13C NMR spectra of *(E)-3-(4-chlorophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3g.**

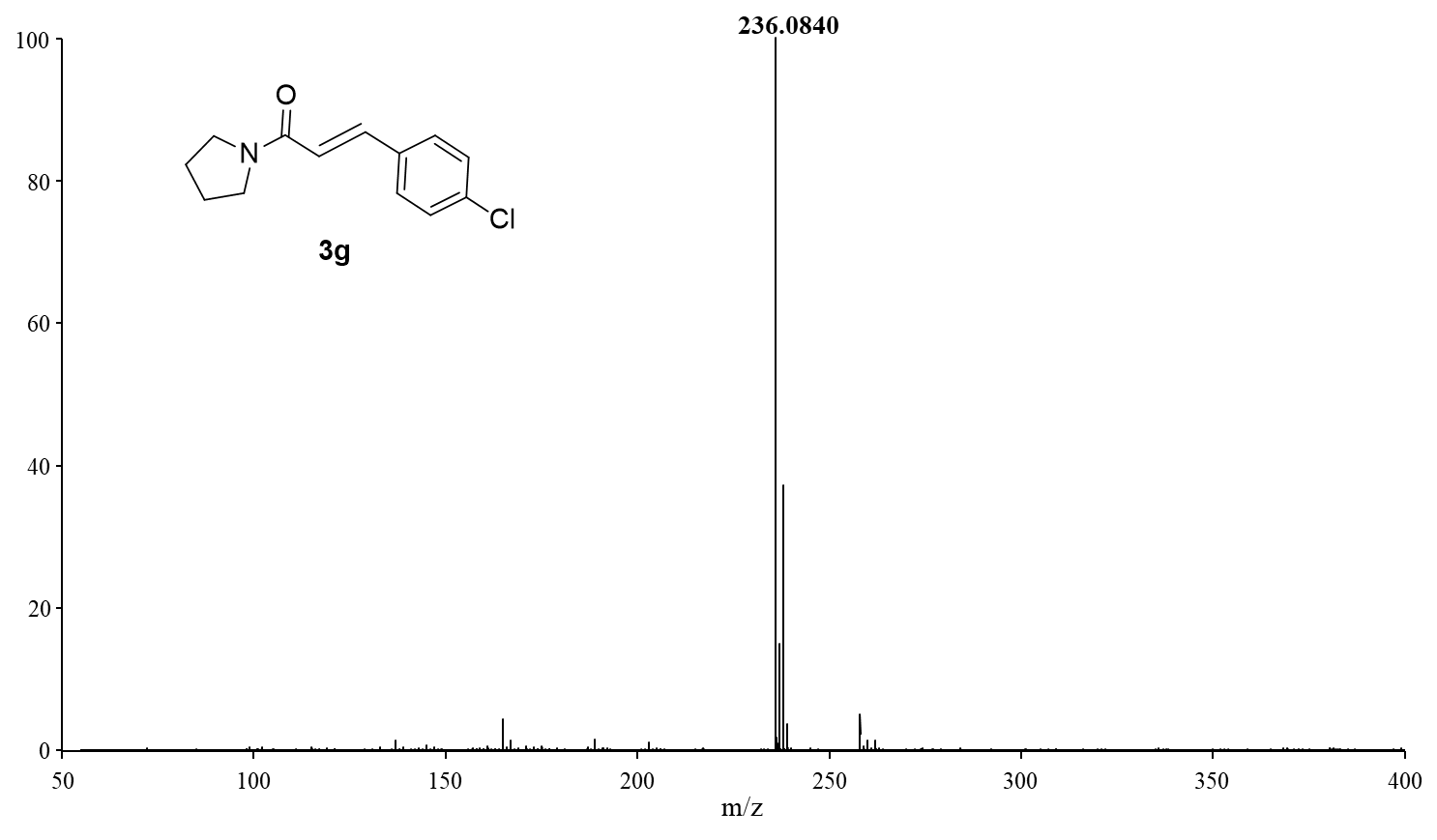
****

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

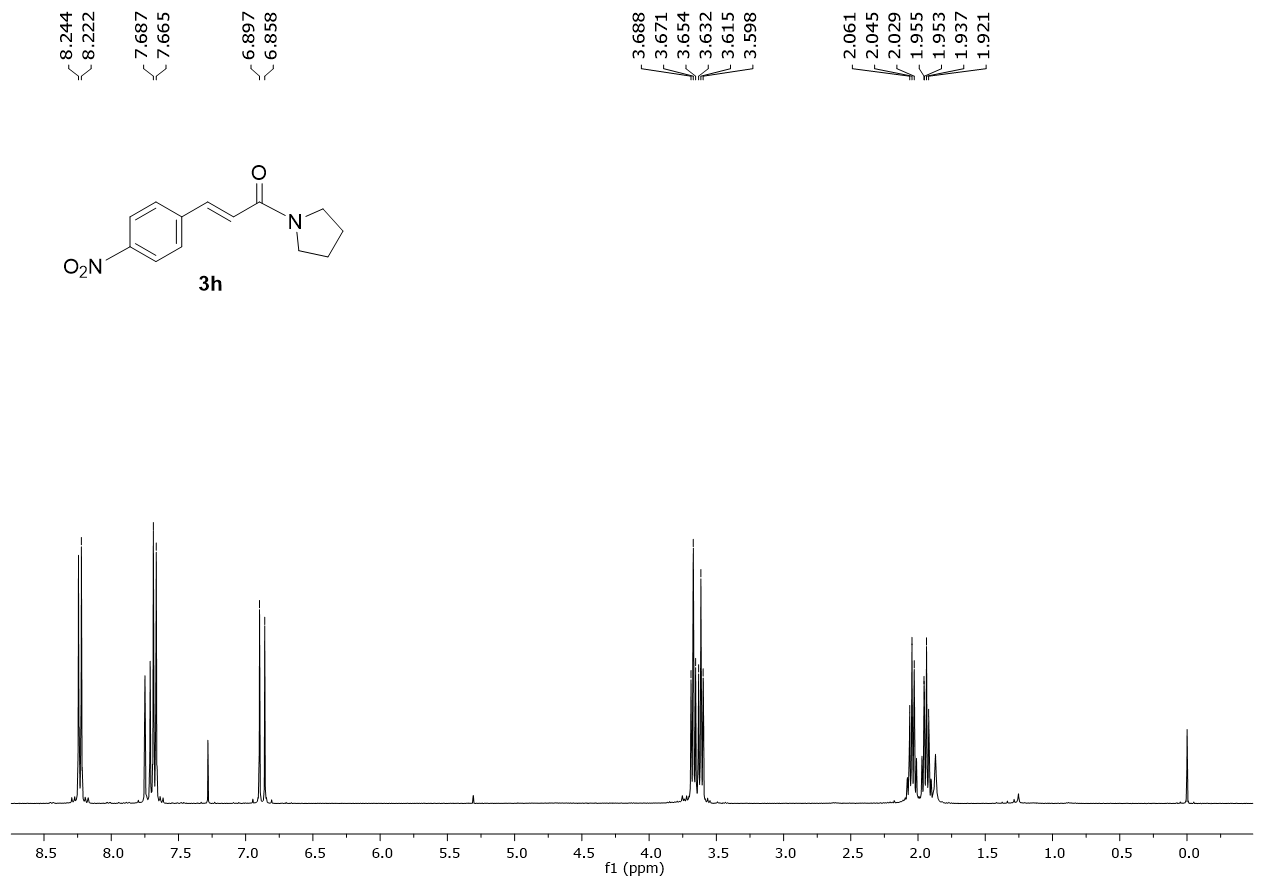
****

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

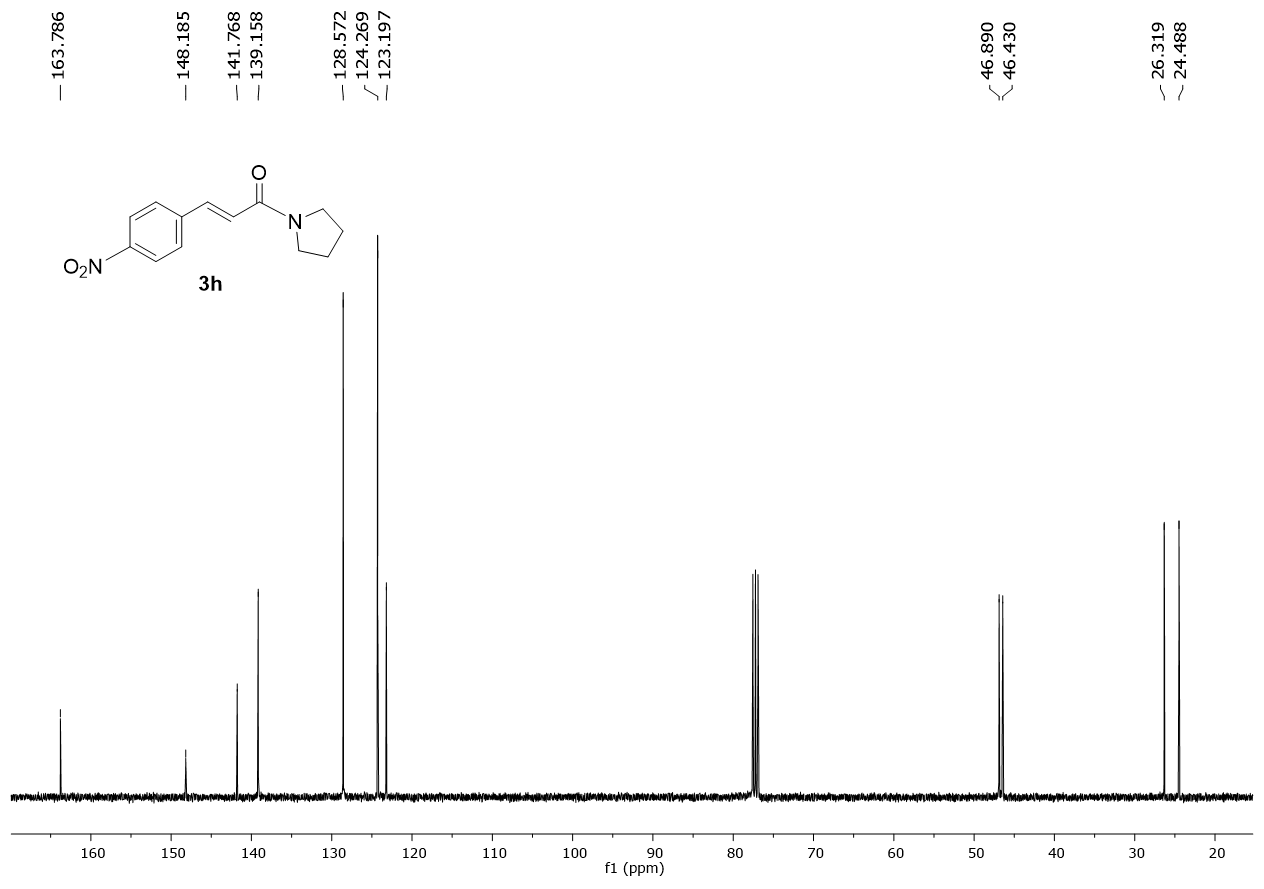


Fig. 30 13C NMR spectra of *(E)-3-(4-nitrophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3h.**

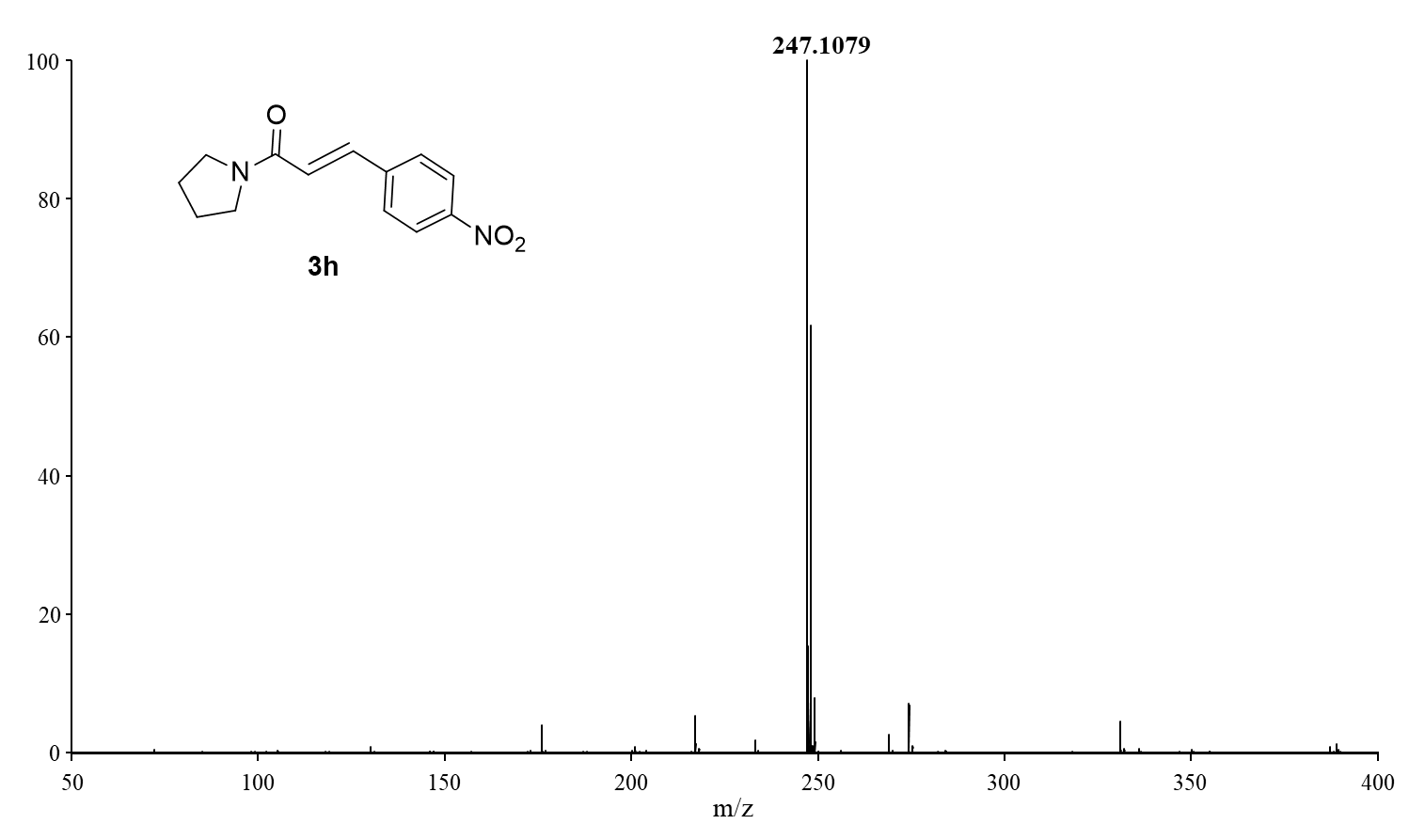


Fig. 31 HRMS spectra of *(E)-3-(4-nitrophenyl)-1-(pyrrolidin-1-yl)prop-2-en-1-one* **3h.**

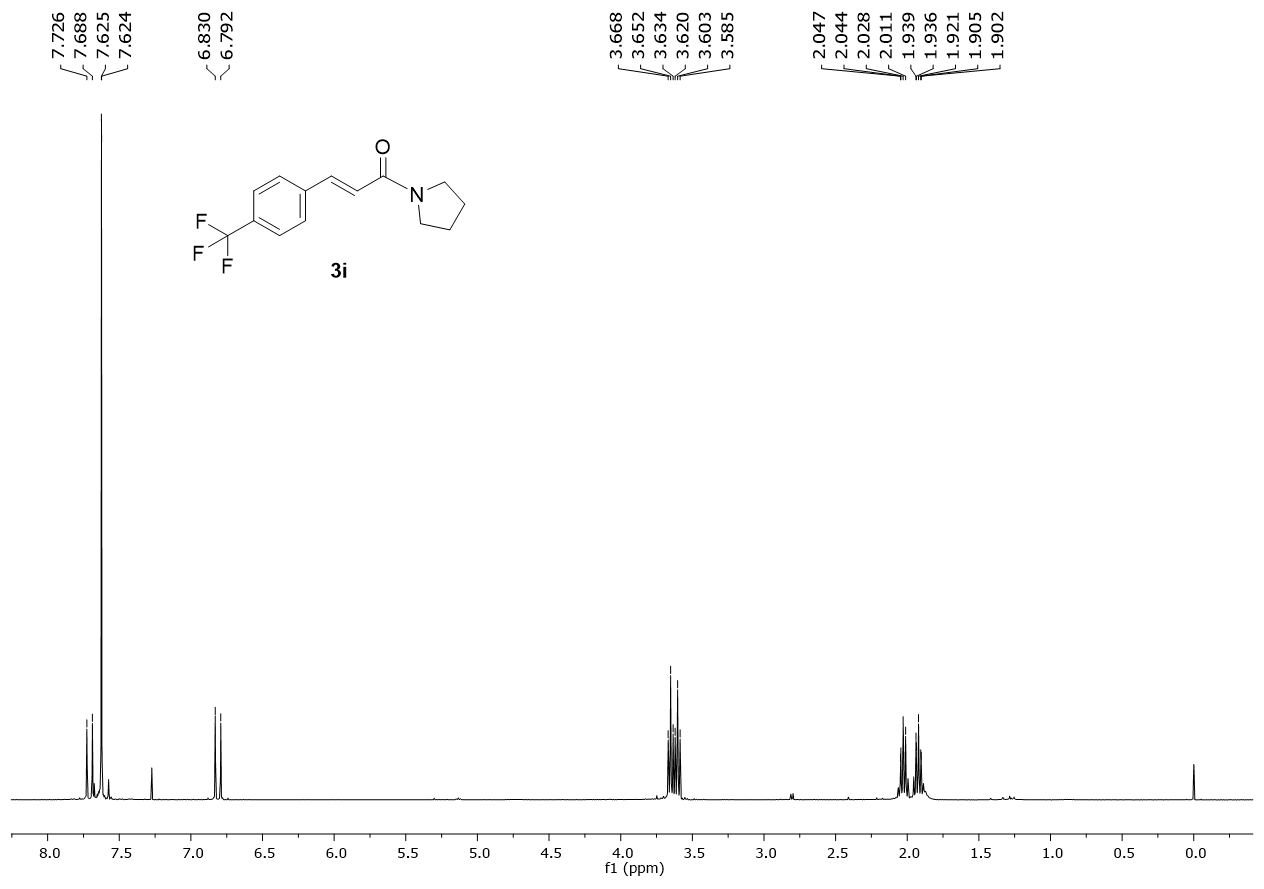


Fig. 32 1H NMR spectra of *(E)-1-(pyrrolidin-1-yl)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one* **3i.**



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

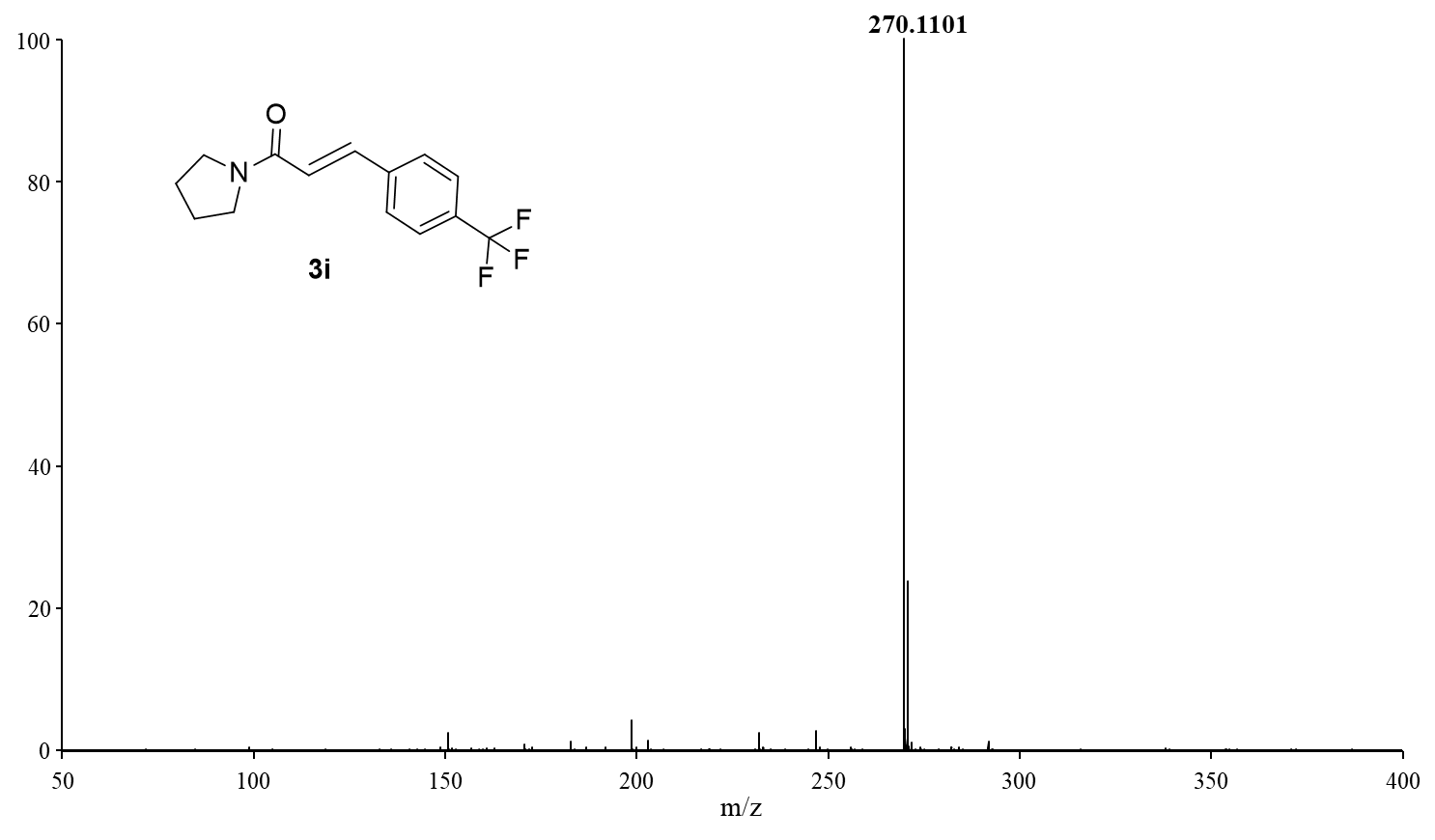


Fig. 34 HRMS spectra of *(E)-1-(pyrrolidin-1-yl)-3-(4-(trifluoromethyl)phenyl)prop-2-en-1-one* **3i.**