# 1 Biological procedures

## 1.1 General Considerations

# 2 Synthetic Chemistry

## 2.1 General Considerations

Reactions were conducted with continuous magnetic stirring under an inert nitrogen atmosphere with dry solvents unless otherwise stated. Nitrogen was passed through a drying tube filled with calcium chloride before use. Glassware was oven-dried (115 °C), assembled and then allowed to cool to room temperature under a positive pressure of inert gas pressure or vacuum.

Cooling of reactions was carried out using low temperature baths: -10–5 °C was achieved by an ice-brine slush bath, -15 °C using acetone/ice slush. Quoted temperatures are oil bath temperatures or internal reaction temperatures to the nearest 5 °C if stated. Microwave heated reactions were carried out in a CEM Discover® microwave.

Reagents were purchased from Sigma-Aldrich Chemical Co. Ltd., Acros Organics Ltd, AJAX, Fisher xxx. and used as supplied. Toluene, acetonitrile and dimethylformamide were collected fresh from an in-house solvent purification system having been passed through anhydrous alumina columns or dried with freshly activated 4Å. Anhydrous THF was freshly distilled from sodium/benzonphenone and dichloromethane from calcium hydride. Hexanes were distilled (63–66 °C) prior to use. Petrol refers to petroleum ether 40–60 6°C.

Reactions were monitored by TLC until deemed complete using aluminium backed silica plates (Merck Kieselgel 60 F254). Plates were visualised under ultraviolet light (254 nm) followed by staining with vanillin or $KMnO_4$. Flash column chromatography was carried out using Grace Davisil® LC60A 40–63 micron silica, the compound to be purified either applied as an oil or pre-absorbed onto silica. Pressure was applied at the column head using ∼1 bar of nitrogen pressure.

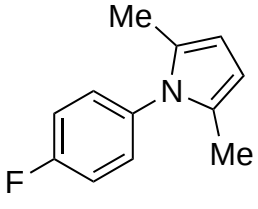
$^{1}H$ and $^{13}C$ nuclear magnetic resonance spectroscopy experiments were carried out using Bruker xxx NMR spectrometers. Acquisitions were carried out at room temperature unless otherwise stated. Chemical shifts are reported in parts per million from the resid-

ual solvent peak. Chemical shifts ($\delta$) are given in parts per million, ppm) and coupling constants ($J$) in Hertz (Hz). Proton multiplicity is assigned using the following abbreviations: singlet (s), doublet (d), triplet (t), quartet (q), septet (sep), multiplet (m), broad (br), apparent (app), overlapping (o) and aryl (Ar). Where required, proton assignment was achieved using 2D NMR spectroscopy techniques, predominantly COSY and HMQC spectroscopy. All samples were dried with high vacuum to remove traces of solvents and water present before weighing to give the reported yields. Minor impurities (such as silicone grease) may be present in some compound spectra.

Melting points were determined manually using a Stanford Research Systems Optimelt apparatus. Infrared measurements were carried out as a thin film or neat using a attenuated total reflectance module on a Bruker xxx with internal calibration in the range 4000–xxx00 $cm^{-1}$. Electrospray (ESI) and Atmospheric Pressure Chemical Ionisation (APCI) accurate mass measurements were carried out on a xxx mass spectrometer by the internal service at the School of Chemistry, University of Sydney. APCI xxx

## 2.2 Known Compounds

**General Procedure A as exemplified by 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole, OSM-S-1, PMY 1-1**



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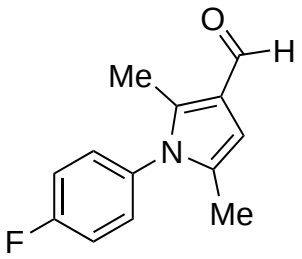
4-fluoroaniline (10.4 mL, 110 mmol, 1.1 equiv.) and 2,5-hexanedione (11.7 mL, 100 mmol, 1 equiv.) were heated to 110 °C (oil bath temp.). After 15 hours, reaction was cooled to room temperature. The mixture was dissolved in hot EtOH (15 mL) and a mixture of EtOH (30 mL) and aqueous solution of 10% citric acid (15 mL). The reaction was slowly cooled to approx. 10 °C with periodic shaking. The resulting crystals were filted and washed with water (approx. 200 mL) to obtain the product as pale tan crystals (15.0 g, 79%) after drying under vacuum at room temperature. A further crop of brown crystals (1.89 g, 10%) could be obtained from mother liquor. Mpt. 48–79 ($EtOH/H_2O$); $^1H$ NMR (300 MHz, $CDCl_3$) $\delta$ 7.21–7.11 (m, 4H), 5.89 (s, 2H), 2.01 (s, 6H); $^{13}C$ NMR (75 MHz, $CDCl_3$) $\delta$ 161.9 (d, $J$ 247.4), 135.0 (d, $J$ 2.7), 129.9 (d, $J$

8.5), 128.9, 116.0 (d, $J$ 22.6), 105.8, 12.9; $^{19}F\{^{1}H\}$ NMR (282 MHz, $CDCl_3$) $\delta$ -114.1; $m/z$ (APCI+) 190 $[M+H]^+$. InChi=1S/C12H12FN/c1-9-3-4-10(2)14(9)12-7-5-11(13)6-8-12/h3-8H,1-2H3. http://malaria.ourexperiment.org/uri/17. Data consistent with literature.http://dx.doi.org/10.1002/cmdc.200600026. NMR spectra on page 32.

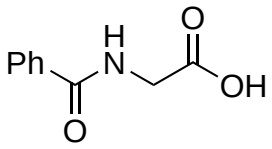
**General Prodedure B as exemplified by 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carbaldehyde, OSM-S-2, PMY 2-4**




xxx

DMF (5.0 mL) was stirred under a nitrogen atmosphere in an ice-bath. Phosphoryl chloride (1.18 mL, 12.7 mmol, 1.2 equiv.) was added and the reaction stirred for 25 minutes. Reaction still colourless. A solution of pyrrole PMY 1-6 (2.00 g, 10.5 mmol, 1 equiv.) in DMF (5 mL) was added dropwise over 5 minutes. The reaction was removed from the ice-bath and allowed to warm to room temperature. After 45 minutes, reaction was complete by TLC (20% EtOAc/hexane). The reaction was poured over ice (approx. 100 ml) and the pH adjusted to 6 (approx. 20% /ceNaOH(aq)) and left stirring overnight. 20% NaOH(aq) added until pH 11 and left for a further 30 minutes. The solid was filtered and washed with water and recrystallised (MeCN/water) to obtain a first crop of tan free-flowing powder (2.05 g, 89%). Mpt. 117–118 °C (MeCN/$H_2O$); $^{1}H$ NMR (300 MHz, $CDCl_3$) $\delta$ 9.87 (s, 1H), 7.22–7.20 (m, 4H), 6.38 (s, 1H), 2.27 (s, 3H), 1.98 (s, 3H); $^{13}C$ NMR (75 MHz, $CDCl_3$) $\delta$ 185.3, 162.5 (d, $J$ 249.7), 138.8, 132.8 (d, $J$ 2.2), 131.0, 129.7 (d, $J$ 8.7), 122.0, 116.6 (d, $J$ 22.8), 106.0, 12.6, 11.2; $^{19}F\{^{1}H\}$ NMR (282 MHz, $CDCl_3$) $\delta$ xxx; $m/z$ (APCI+) 218 $[M+H]^+$. InChi=1S/C13H12FNO/c1-9-7-11(8-16)10(2)15(9)13-5-3-12(14)4-6-13/h3-8H,1-2H3. http://malaria.ourexperiment.org/uri/1f and http://www.thesynapticleap.org/node/344#comment-712. Data consistent with literature.http://dx.doi.org/10.1021/jm00098a013http://dx.doi.org/10.1002/cmdc.200600026 NMR spectra on page 33.

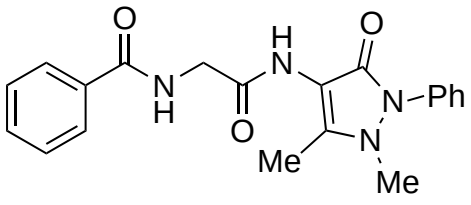
**Synthesis of hippuric acid, OSM-S-15, PMY 26-1**




xxx

Glycine (12.5 g, 0.17 mol, 1 equiv.) was dissolved in NaOH(aq) (13.3 g in approx 130 mL water, 0.33 mol, 2 equiv.). The flask was placed in a room temperature water bath and benzoyl chloride (21 mL, 0.18 mol, 1.06 equiv.) was added portionwise over 60 minutes keeping the temperature below 30 °C. The reaction was stirred for an hour and then cooled in ice. Conc. HCl(aq) (approx 20 mL) was added and the mixture stirred for 30 mins. The copious white precipitate was filtered and washed with water. The crude product was triturated with hot DCM (100 mL) for 10 minutes then filtered and washed with further DCM (2 × 20 mL). After air drying (10 mins), the product was dissolved in boiling water (approx 500 mL), hot filtered to remove some residual solid and allowed to crystallise slowly overnight. The crystals were filtered out (room temp.) and washed with water to obtain the product hippuric acid as white needles (22.4 g, 75%) after drying under a stream of nitrogen. Mpt. 189–190 °C ($H_2O$); $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; InChi=1S /C9H9NO3/c11-8(12)6-10-9(13)7-4-2-1-3-5-7/h1-5H,6H2,(H,10,13)(H,11,12). `http://malaria.ourexperiment.org/uri/5ef`. Data consistent with literature.`http://jocpr.com/vol2-iss4-2010/JCPR-2-4-410-414.pdf`http://www.orgsyn.org/orgsyn/prep.asp?prep=cv2p0328 NMR spectra on page **??**.

***N*-(2-((1,5-dimethyl-3-oxo-2-phenyl-2,3-dihydro-1*H*-pyrazol-4-yl)amino)-2-oxoethyl)benza**
**OSM-S-16, PMY 27-2**



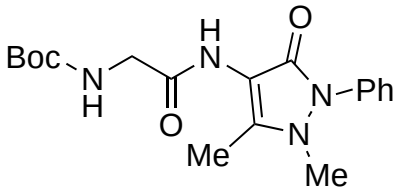

xxx

Prepared according to literature method.`http://www.ncbi.nlm.nih.gov/pubmed/8267666`

Mtp. xxx °C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; $\nu_{max}$ (neat)

/$cm^{-1}$ ; $m/z$ (ESI+) $[M+H]^+$. InChi=1S/C20H20N4O3/c1-14-18(20(27)24(23(14)2)16-11-7-4-8-12-16)22-17(25)13-21-19(26)15-9-5-3-6-10-15/h3-12H,13H2,1-2H3,(H,21,26)(H,22,25). http://malaria.ourexperiment.org/uri/75. See page **??** for NMR spectra. Data consistent with literature.http://www.ncbi.nlm.nih.gov/pubmed/8267666

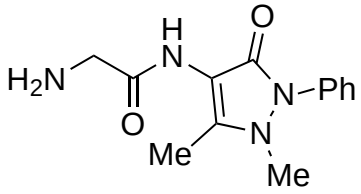
**OSM-S-17, PMY 29-1, PMY 29-2**




xxx

Mtp. xxx °C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; $\nu_{max}$ (neat) /$cm^{-1}$ ; $m/z$ (ESI+) $[M+H]^+$. InChi=1S/C18H24N4O4/c1-12-15(20-14(23)11-19-17(25)26-18(2,3)4)16(24)22(21(12)5)13-9-7-6-8-10-13/h6-10H,11H2,1-5H3,(H,19,25)(H,20,23). http://malaria.ourexperiment.org/uri/6c. See page **??** for NMR spectra. Data consistent with literature.http://dx.doi.org/10.1007/s00726-008-0074-1

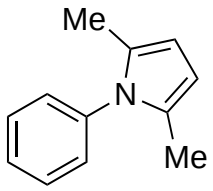
**$N$-(1,5-Dimethyl-3-oxo-2-phenyl-2,3-dihydro-1$H$-pyrazol-4-yl)glycinamide, OSM-S-18, PMY 30-3**




xxx

Mtp. xxx °C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; $\nu_{max}$ (neat) /$cm^{-1}$ ; $m/z$ (ESI+) $[M+H]^+$. InChi=1S/C13H16N4O2/c1-9-12(15-11(18)8-14)13(19)17(16(9)2)10-6-4-3-5-7-10/h3-7H,8,14H2,1-2H3,(H,15,18). http://malaria.ourexperiment.org/uri/8d. See page **??** for NMR spectra. Data consistent with literature.http://www.ncbi.nlm.nih.gov/pubmed/8267666

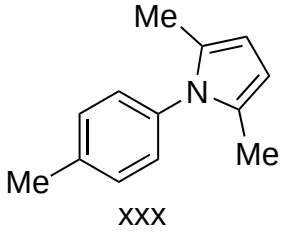
**OSM-S-25, LMW 1**




xxx

Mtp. 49–50 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.44–7.36 (s, 3H), 7.21–7.17 (t, 2H), 5.90 (s, 2H), 2.02 (s, 6H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 139.1, 129.1. 128.8, 128.3, 127.7, 105.8, 13.1; $\nu_{max}$ (neat) $/cm^{-1}$ 1597, 1520, 1494, 1401, 1318, 1037, 1006, 773, 747, 717, 695, 646; *m/z* (APCI) 173 $[M+H]^+$. InChi=1S/C12H13N/c1-10-8-9-11(2)13(10)12-6-4-3-5-7-12/h3-9H,1-2H3. `http://malaria.ourexperiment.org/uri/4c`. See page 34 for NMR spectra. Data consistent with literature.`http://dx.doi.org/10.1002/cmdc.200600026`

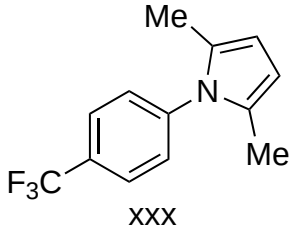
**OSM-S-26, LMW 2**




xxx

Mtp. 45–46 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.23–7.20 (m, 2H), 7.08–7.05 (m, 2H), 5.88 (s, 2H), 2.38 (s, 3H), 2.01 (s, 6H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 137.5, 136.6, 129.8, 128.9, 128.1, 105.7, 21.3, 13.2; $\nu_{max}$ (neat) $/cm^{-1}$ 1513.22, 1403, 1320, 826, 750; *m/z* (ESI+) 187 $[M+H]^+$. InChi=1S/C13H15N/c1-10-4-8-13(9-5-10)14-11(2)6-7-12(14)3/h4-9H,1-3H 3. `http://malaria.ourexperiment.org/uri/4b`. See page 35 for NMR spectra. Data consistent with literature.`http://dx.doi.org/10.1002/cmdc.200600026`

**OSM-S-27, LMW 3**

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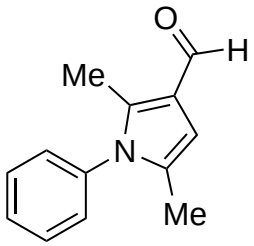
Mtp. 70–73 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.73 (d, $J$ 8.2, 2H), 7.34 (d, $J$ 8.3), 5.93 (s, 2H), 2.05 (s, 6H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 142.3, 129.8 (q, $J$ 32.7), 128.1, 128.6, 126.3 (m), 123.9 (q, $J$ 272.1), 106.6, 13.0; $\nu_{max}$ (neat) /$cm^{-1}$ 1615, 1402, 1325, 1128, 1064, 850, 759; $m/z$ (ESI+) 241 $[M+H]^+$. InChi=1S/C13H12F3N/c1-9-3-4-10(2)17(9)12-7-5-11(6-8-12)13(14,15)16/h3-8H,1-2H3. `http://malaria.ourexperiment.org/uri/4a`. See page 35 for NMR spectra. Data consistent with literature.`http://dx.doi.org/10.1002/cmdc.200600026`

**2,5-dimethyl-1*H*-phenyl pyrrole-3-carboxaldehyde, OSM-S-28, LMW 4**



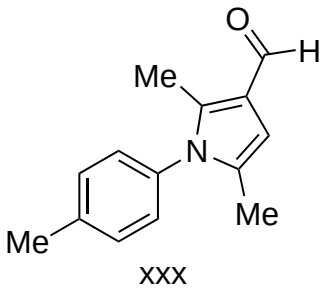

xxx

=== standardise ===

Prepared according to General Procedure B using DMF (1.5 mL, 19.4 mmol, 6.5 equiv.) was stirred under a nitrogen atmosphere in an ice-bath. Phosphoryl chloride (0.308 mL, 3.3 mmol, 1.1 equiv.) was added and the reaction stirred for 30 minutes. Reaction mixture was a very pale yellow. A solution of 2,5-dimethyl-1H-phenyl-pyrrole (514 mg, 3 mmols, 1 equiv.) in DMF (2 mL) was added dropwise over 1 minute. After 5 minutes, the flask was removed from ice and reaction stirred for a further 55 mins. TLC at 1 hr showed reaction to be complete. Reaction mixture was poured onto ice (50 mL) and 1M NaOH added (pH 11), until adjusted to pH 6. Total volume approximately 50 mL after ice has melted and was pH 3 the next day. 20% NaOH was added to achieve pH 11 and flask bathed in a brine ice-bath for 20 minutes. Product was filtered and washed with water to produce a wet brown paste. Product was dissolved in MeCN and concentrated under a nitrogen atmosphere. Brown-green solution with slight precipitation was observed overnight. Product was then extracted using a mixture of EtOAc (2 20 mL), Et2O (20 mL), water (20 mL) and brine (20 mL). Product was concentrated under reduced pressure and cooled in refrigerator overnight to crystallise. Product was dissolved in EtOH (3 mL) in a water bath at 40C. Solution was cooled in a brine ice bath and filtered. Filter cake was washed with water (3 10 mL) and product dried under vacuum to give a grey free-flowing powder (407 mg, 64%). Mtp. 88–90 °C; $^{1}$H NMR (300 MHz,

$CDCl_3$) $\delta$ 9.88 (s, 1H), 7.55–7.50 (m, 3H), 7.22–7.19 (m, 2H), 6.39 (s, 1H), 2.28 (s, 3H), 1.99 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 185.3, 138.9, 137.0, 131.0, 129.6, 128.9, 128.0, 121.9, 105.8, 12.7, 11.2; $\nu_{max}$ (neat) /$cm^{-1}$ 1650, 1422, 801, 669; $m/z$ (ESI+) 200 $[M+H]^+$. InChi=1S/C13H13NO/c1-10-8-12(9-15)11(2)14(10)13-6-4-3-5-7-13/h3-9H,1-2 H3. `http://malaria.ourexperiment.org/uri/4d`. See page 37 for NMR spectra. Data consistent with literature.`http://dx.doi.org/10.1002/cmdc.200600026`

**2,5-dimethyl-1*H*-(*p*-tolyl)-pyrrole-3-carboxaldehyde, OSM-S-29, LMW 5**



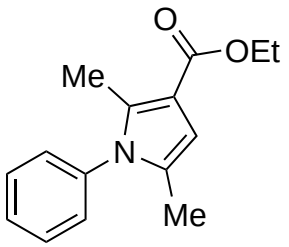

xxx

=== standardise ===

Prepared according to General Procedure B DMF (1.5 mL, 19.4 mmol, 6.5 equiv.) was stirred under a nitrogen atmosphere in an ice-bath. Phosphoryl chloride (0.308 mL, 3.3 mmol, 1.1 equiv.) was added and the reaction stirred for 30 minutes. Reaction mixture was a very pale yellow. A solution of 2,5-dimethyl-1H-(p-tolyl)-pyrrole (556 mg, 3 mmols, 1 equiv.) in DMF (2 mL) was added dropwise over 1 minute. After 5 minutes, flask removed from ice and reaction stirred for a further 55 mins. TLC at 1 hr showed reaction at completion. Reaction mixture was then poured onto ice (50 mL) and 1M NaOH added (pH 11), until adjusted to pH 6. Total volume approximately 50 mL after ice has melted and solution was pH 3 the next day. 20% NaOH was added to achieve pH 11 and flask bathed in a brine ice-bath for 20 minutes. Product filtered and washed with water to produce a wet brown paste. Product was dissolved in MeCN and concentrated under a nitrogen atmosphere. Brown solution with precipitation of fine crystals was observed overnight. Solution was cooled in an ice bath and filtered. Filter cake washed with water (3 × 10 mL). Filtered product was then dried under vacuum to produce a lumpy grey-brown powder (423 mg, 71%). Mtp. 109–111 °C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 9.86 (s, 1H), 7.31 (d, $J$ 7.9, 2H), 7.07 (d, $J$ 7.9, 2H), 6.37 (s, 1H), 2.44 (s, 3H), 2.27 (s, 3H), 1.98 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$185.2, 139.0, 138.9, 134.3, 131.1, 130.2, 127.7, 121.8, 105.6, 21.2, 12.6, 11.2; $\nu_{max}$ (neat) /$cm^{-1}$ 3935, 1651, 1515, 1422, 811; $m/z$ (ESI+) 214 $[M+H]^+$. InChi=1S/C14H15NO/c1-10-4-6-14(7-5-10)15-11(2)8-13(9-16)12(15)3/h4-9H

,1-3H3. http://malaria.ourexperiment.org/uri/4e. See page 38 for NMR spectra. Data consistent with literature.http://dx.doi.org/10.1002/cmdc.2006000265

**Synthesis of ethyl 2,5-dimethyl-1-phenyl-1*H*-pyrrole-3-carboxylate, OSM-S-31, LMW 8-1**




xxx

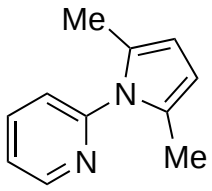
=== standardise ===

Prepared according to general method C using ethyl acetoacetate (6 mL,47 mmol, 1 equiv.) and $K_2CO_3$ (8.45 g, 61.1 mmol, 1.3 equiv.) were mixed in MeCN (55 mL). NaI (7.05 g, 47 mmol, 1 equiv.) and chloroacetone (4.8 mL, 51.7 mmol, 1.1 equiv.) were added and mixture heated to 80 °C in an oil bath. TLC at 2 hours showed reaction at completion. Reaction was allowed to cool to room temperature. Mixture washed with EtOAc (2 × 20 mL), water (2 × 20 mL), 1:1 water:brine (2 × 20 mL) and brine (2 × 20 mL) and dried with MgSO4 and concentrated under reduced pressure to form a yellow oil. Ethyl 2-acetyl-4-oxopentanoate intermediate (2 mL, 10.7 mmol, 1 equiv.) was added to aniline (1.17 mL, 12.89 mmol, 1.2 equiv.) and heated at 80 °C in an oil bath for 75 mins. TLC at 1 hour showed reaction at completion and reaction was allowed to cool to room temperature. Product was washed with EtOAc (2 × 20 mL), 10% citric acid (3 × 20 mL), water (20 mL) and brine (2 × 20 mL) and then concentrated under reduced pressure to form a dark brown oil. Product was purified by chromatography on silica (2-15% EtOAc/petrol). Product containing fractions concentrated under reduce pressure to produce a yellow oil (952 mg, approximate yield 37%) which was used without further purification.

$^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.50–7.41 (m, 2H), 7.18–7.15 (m, 2H), 6.38 (s, 1H), 4.28 (q, $J$ 7.1, 2H), 2.29 (s, 3H), 1.97 (s, 3H), 1.34 (t, $J$ 7.1, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 165.7, 137.8, 136.2, 129.4, 128.7, 128.5, 128.2, 111.5, 107.6, 59.2, 14.6, 12.6, 12.4; $\nu_{max}$ (neat) /$cm^{-1}$ 2978, 1693, 1411, 121; $m/z$ (APCI+) 244 $[M+H]^+$. InChi=1 S/C15H17NO2/c1-4-18-15(17)14-10-11(2)16(12(14)3)13-8-6-5-7-9-13/h5-10H,4H2,1-3H

3. http://malaria.ourexperiment.org/uri/50. Data consistent with literature.http://dx.doi.org/10.1016/0040-4020(80)80102-5

**OSM-S-41, ZYH 9-1**

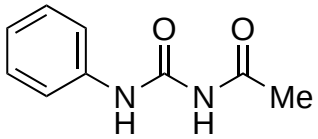



xxx

Prepared according to General Procedure A except

$^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) $[M+H]^+$. InChi=1S/C11H12N2/c1-9-6-7-10(2)13(9)11-5-3-4-8-12-11/h3-8H,1-2 H3. http://malaria.ourexperiment.org/uri/60. See page **??** for NMR spectra. Data consistent with literature.http://dx.doi.org/10.1590/S0103-50532008000500011

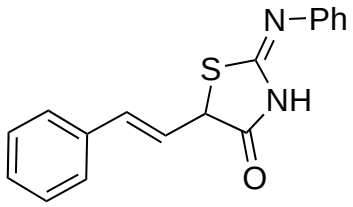
**OSM-S-53, ZYH 20-1**




xxx

Mtp. 166–167 °C; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) $[M+H]^+$. InChi=1S/C9H10N2OS/c1-7(12)10-9(13)11-8-5 -3-2-4-6-8/h2-6H,1H3,(H2,10,11,12,13). http://malaria.ourexperiment.org/uri/7b. See page **??** for NMR spectra. Data consistent with literature.http://dx.doi.org/10.1080/03086647708079937

**OSM-S-55, ZYH 23-1**



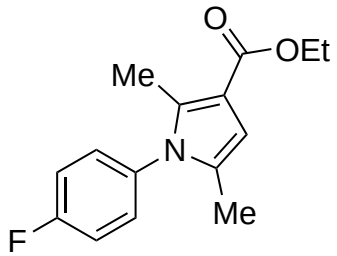

xxx

Prepared according to literature.http://dx.doi.org/10.1016/j.bmc.2008.10.032

Mtp. 260–261 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 303 $[M+Na]^+$. InChi=1S/C16H12N2OS/c19-15-14(11-12-7-3-1-4-8-12)20-16(18-15)17-13-9-5-2-6-10-13/h1-11H,(H,17,18,19)/b14-11-. http://malaria.ourexperiment.org/uri/85. See page **??** for NMR spectra. Data consistent with literature.http://dx.doi.org/10.1016/j.bmc.2008.10.032

# 3 Novel compounds or those without reported experimental data

**General procedure C for the synthesis of pyrrole-3-esters as exemplified by ethyl 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carboxylate, OSM-S-3, PMY 6-1**



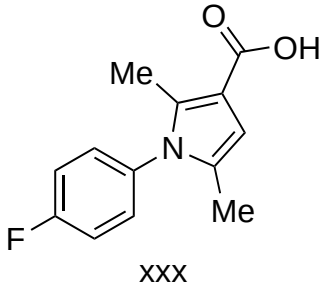

xxx

Ethyl acetoacetate (2.00 mL, 15.7 mmol, 1 equiv.) and $K_2CO_3$ (2.82 g, 20.4 mmol, 1.3 equiv.) in acetonitrile (30 mL) were cooled ice/brine. Chloroacetone (1.39, 17.2 mmol, 1.1 equiv.) was added dropwise. After stirring for 15 minutes, reaction was allowed to warm to room temperature. After 15 minutes, sodium iodide (2.58 g, 17.2 mmol, 1.1 equiv.) was added. After stirring for a further for 1.5 hours, reaction is now a pale yellow suspension

Heated to reflux. After 20 hours at reflux, reaction was allowed to cool and then filtered. The filtrate was concentrated under reduced pressure and then dissolved in EtOAc (40 mL). Washed with water (2 × 20 mL), 1:1 brine/water (20 mL) to break emulsion, brine and dried ($MgSO_4$) then concentrated under reduced pressure to a brown oil (2.95 g). $^1$H NMR consistent with product`http://dx.doi.org/10.1021/jo00391a003` which was used without further purification.

4-fluoroaniline (1.78 mL, 18.8 mmol, 1.2 equiv.) was added and the reaction heated to 90 °C. After 2 hours, reaction complete by TLC. Reaction cooled. Black oil with colouless liquid (water). Dissolved in EtOAc (30 mL) and washed with 10% citric acid (3 × 15 mL), water (3 × 15 mL) and brine then dried ($MgSO_4$) and concentrated under reduced pressure (3.70 g). Recrystallised EtOH/water; Dissolved in warm EtOH (4 mL) and cooled in ice until crystallisation initiated, then 40% EtOH (16 mL) added slowly with stirring. Cooled in ice for 30 minutes to complete crystallisation and filtered. Red/brown crystals washed with 20% EtOH and dried under vacuum (2.77 g, 61% over 2 steps). Mpt. 63–66 °C (EtOH/$H_2O$); $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.19–7.16 (m, 4H), 6.37 (s, 1H), 4.28 (q, $J$ 7.1, 2H), 2.28 (s, 3H), 1.96 (s, 3H), 1.34 (d, $J$ 7.1, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 165.6, 162.3 (d, $J$ 248.8), 136.2, 133.7 (d, $J$2.9), 129.9 (d, $J$ 8.7), 128.8, 116.4 (d, $J$ 22.8), 111.6, 107.6, 59.3, 14.5, 12.6, 12.3; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C15H16FNO2/c1-4-19-15(18)14-9-10(2)17(11(14)3)13-7-5-12(16)6-8-13/h5-9H,4H2,1-3H3. See page 39 for NMR spectra. `http://malaria.ourexperiment.org/uri/20`.

**General Procedure D for hydrolysis of pyrrole-3-esters to their corresponding acids as exemplified by 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carboxylic acid, OSM-S-4, PMY 8**

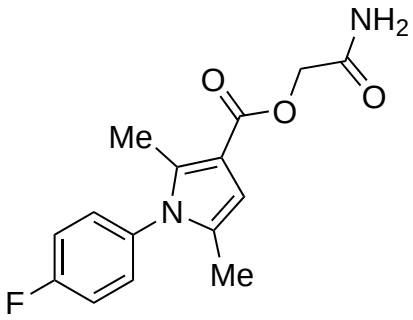


xxx

PMY 6 (2.16 g, 8.27 mmol, 1 equiv.) was dissolved in EtOH (approx 30 mL) and 20% NaOH(aq) (40 mL, approx 17 equiv.). The reaction was heated to reflux for approximately 16 hours. The reaction was then cooled in ice and 15% HCl was added slowly

until a pale precipitate formed (pH 1). The mixture was stirred for a further 15 minutes and then filtered. The pale brown cake was washed with water (2 × 25 mL). After drying under vacuum the product was obtained as a pale brown powder (1.73 g, 89%). Mpt.241–242°C (acetone/$H_2O$) decomposition; $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.19 (app d, $J$ 6.4, 4H), 6.42 (br s, 1H), 2.30 (s, 3H), 1.97 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 171.09, 162.4 (d, $J$ 249.0), 137.8, 133.6 (d, $J$ 2.7), 129.9 (d, $J$ 8.6), 129.2, 116.5 (d, $J$ 22.8), 110.8, 108.2, 12.6, 12.5; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C13H12FNO2/c1-8-7-12(13(16)17)9(2)15(8)11-5-3-10(14)4-6-11/h3-7H,1-2H3,(H,16,17). See page 40 for NMR spectra. `http://malaria.ourexperiment.org/uri/81`.

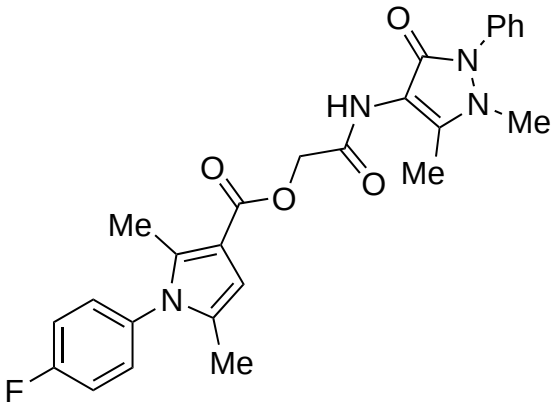
**General Procedure E for the Synthesis of pyrrole 3-esters or amides as exemplified by 2-amino-2-oxoethyl 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carboxylate, TCMDC-123812, OSM-S-5, PMY 10**




xxx

Mpt.xxx–xxx °C (xxx); $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.21–7.17 (m, 4H), 6.37 (s, 1H), 6.03 (app br d, $J$ 70.2, 2H), 4.74 (s, 2H), 2.30 (s, 3H), 1.98 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 171.1, 163.7, 162.5 (d, $J$ 249.3), 137.8, 133.3 (d, $J$ 3.4), 129.8 (d, $J$ 8.7), 129.4, 116.6 (d, $J$ 23.0), 109.9, 107.4, 61.8, 12.6, 12.4; $^{19}$F{$^1$H} NMR (282 MHz, $CDCl_3$) $\delta$ -111.92; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C15H15FN2O3/c1-9-7-13(15(20)21-8-14(17)19)10(2)18(9)12-5-3-11(16)4-6-12/h3-7H,8H2,1-2H3,(H2,17,19). See page 41 for NMR spectra. `http://malaria.ourexperiment.org/xxx`.

**TCMDC-123794, OSM-S-6, PMY 11-2**

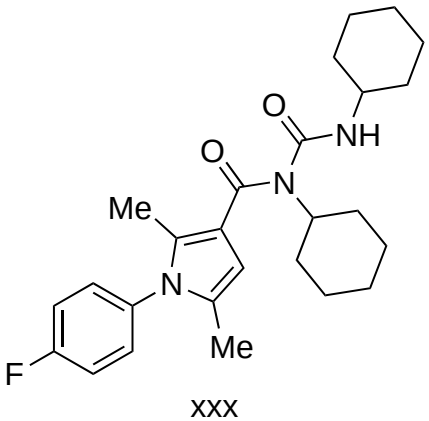



xxx

General procedure E

Mpt.xxx–xxx °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.64 (br s, 1H), 7.48–7.37 (m, 4H), 7.32–7.30 (m, 1H), 7.21–7.16 (m, 4H), 6.43 (s, 1H), 4.87 (s, 2H), 3.08 (s, 3H), 2.31 (s, 6H), 1.95 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ok but need a stronger one; $^{19}$F{$^{1}$H} NMR (282 MHz, $CDCl_3$) $\delta$ -112.14; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C26H25FN4O4/c1-16-14-22(17(2)30(16)20-12-10-19(27)11-13-20)26(34)35-15-23(32)28-24-18(3)29(4)31(25(24)33)21-8-6-5-7-9-21/h5-14H,15H2,1-4H3,(H,28,32). `http://malaria.ourexperiment.org/xxx`.

**OSM-S-7, PMY 12-1**

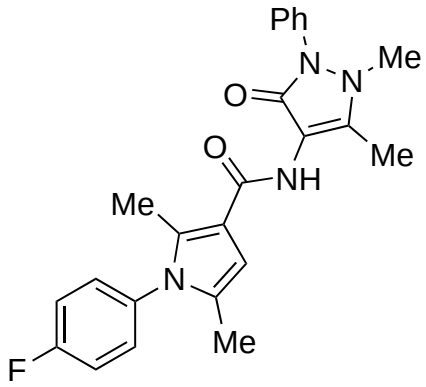



xxx

DCC coupling

Mpt.xxx–xxx °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.22–7.11 (m, 4H), 6.54 (br app d, $J$ 7.3, rotamers, 1H), 6.10 (s, 1H), 4.26 (br t, $J$ 11.8, 1H), 3.69–3.61 (m, 1H), 2.22–2.11 (m, 5H), 1.95 (br s, 3H), 1.83–1.79 (m, 6H), 1.63-1.55 (m, 4H), 1.37–1.08 (m, 8H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 169.5, 162.3 (d, $J$ 248.9), 15534, 134.0, 133.7 (d, $J$ 2.2), 129.8 (d, $J$ 8.7), 128.7, 116.4 (d, $J$ 23.2), 116.3, 106.1, 57.7, 49.3, 32.6, 31.1, 26.4, 25.6, 25.4, 24.5, 12.5, 12.2; $^{19}$F{$^{1}$H} NMR (282 MHz, $CDCl_3$) $\delta$ -112.41; $\nu_{max}$ (neat) /$cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C26H34FN3O2/c1-18-17-24(19(2)29(18)23-15-13-20(27)14-16-23)25(31)30(22-11-7-4-8-12-22)26(32)28-21-9-5-3-6-10-21/h13-17,21-22H,3-12H2,1-2H3,(H,28,32). See page 42 for NMR spectra.`http://malaria.ourexperiment.org/uri/30`.

**OSM-S-8, PMY 12-5**

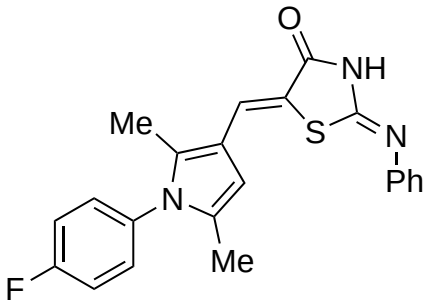



xxx

T3P coupling

Mpt.xxx–xxx °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 4.50–7.39 (m, 4H), 7.33–7.29 (m, 1H), 7.19–7.15 (m, 4H), 6.28 (s, 1H), 2.86 (s, 3H), 2.33 (s, 3H), 2.30 (s, 3H), 1.97 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $^{19}$F{$^{1}$H} NMR (282 MHz, $CDCl_3$) $\delta$ xxx; $\nu_{max}$ (neat) /$cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C24H23FN4O2/c1-15-14-21(16(2)28(15)19-12-10-18(25)11-13-19)23(30)26-22-17(3)27(4)29(24(22)31)20-8-6-5-7-9-20/h5-14H,1-4H3,(H,26,30). `http://malaria.ourexperiment.org/uri/65`.

**OSM-S-9, PMY 14-4**

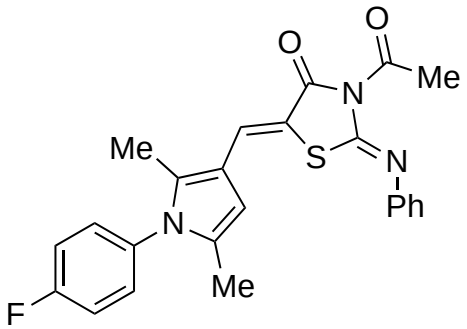



xxx

Prepared according to literature procedure.http://dx.doi.org/10.1016/j.bmcl.2011.09.049

Mpt.xxx–xxx °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $^{19}$F{$^{1}$H} NMR (282 MHz, $CDCl_3$) $\delta$ xxx; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 434 $[M+Na]^+$. InChI=1S/C22H18FN3OS/c1-14-12-16(15(2)26(14)19-10-8-17(23)9-11-19)13-20-21(27)25-22(28-20)24-18-6-4-3-5-7-18/h3-13H,1-2H3,(H,24,25,27)/b20-13-. http://malaria.ourexperiment.org/uri/33.

**General Procedure F as exemplified by (2*Z*,5*Z*)-5-((1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrol-3-yl)methylene)-2-(phenylimino)thiazolidin-4-one, OSM-S-10, PMY 35**



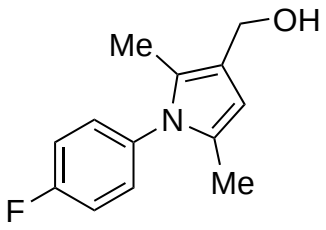

xxx

Prepared according to method reported by Roberts.http://dx.doi.org/10.1016/j.bmc.2008.10.032

Mpt.xxx–xxx °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.95 (s, 1H), 7.53–7.50 (m, 3H), 7.32–7.18 (m, 6H), 6.40 (s, 1H), 2.16 (s, 3H), 2.12 (s, 3H), 2.04 (s, 3H); $^{1}$H NMR (300

MHz, DMSO−$d_6$) $\delta$ 7.73 (s, 1H), 7.57–7.52 (m, 5H), 7.47–7.42 (m, 4H), 6.29 (s, 1H), 2.14 (s, 3H), 2.04 (s, 3H), 2.03 (s, 3H); $^{13}$C NMR (75 MHz, DMSO−$d_6$) $\delta$ 178.6, 174.5, 172.8, 131.8 (d, $J$ 245.9), 139.8, 137.1, 133.1 (d, $J$ 2.6), 132.0, 130.1 (d, $J$ 8.8), 129.9, 129.6, 129.2, 128.6, 119.4, 116.5 (d, $J$ 22.6), 115.7, 105.4, 25.1, 12.4, 10.7; $^{19}$F{$^1$H} NMR (282 MHz, DMSO−$d_6$) $\delta$ -112.7; $\nu_{max}$ (neat) /$cm^{-1}$ ; $m/z$ (ESI+) 434 [M+Na]$^+$. InChI=1S/C24H20FN3O2S/c1-15-13-18(16(2)27(15)21-11-9-19(25)10-12-21)14-22-23(30)28(17(3)29)24(31-22)26-20-7-5-4-6-8-20/h4-14H,1-3H3/b22-14-,26-24-. `http://malaria.ourexperiment.org/uri/95`.

**OSM-S-11, PMY 18-3, PMY 15-2**



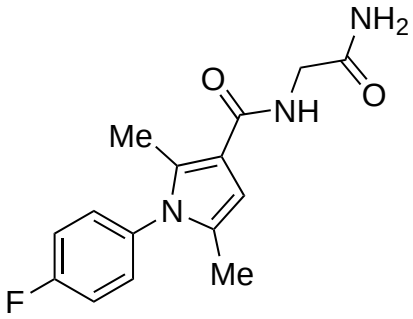

xxx

$LiAlH_4$ (72 mg, 1.91 mmol, 1 equiv, 4 hydride equiv.) was stirred in Et2O (5 mL) at room temperature (water bath). PMY 6-1 (500 mg, 1.91 mmol, 1 equiv.) in Et2O (10 mL) was added dropwise over 5 minutes. After 6 hours, a saturated solution of Rochelle's salt (approx 10 mL) was added, shaken and left to separate. Diluted with EtOAc (30 mL). Separated and aqueous extracted with EtOAc (3  20 mL), dried (MgSO4) and concentrated under reduced pressure. The residue was then purified by chromatography (2-30% EtOAc/hexane) to obtain an off-white solid (264 mg, 63%).

Alternatively the product can be prepared from xxx carbaldehyde using $NaBH_4$.

Mpt.xxx–xxx °C (xxx); $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.19–7.14 (m, 4H), 6.02 (s, 1H), 4.54 (s, 2H), 2.05 (s, 3H), 2.04 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 162.0 (d, $J$ 247.4), 134.9 (d, $J$ 2.9), 130.0 (d, $J$ 8.6), 128.3, 126.7, 119.1, 116.1 (d, $J$ 22.7), 106.8, 57.7, 12.7, 10.5; $^{19}$F{$^1$H} NMR (282 MHz, $CDCl_3$) $\delta$ -113.62; $\nu_{max}$ (neat) /$cm^{-1}$ ; $m/z$ (ESI+) xxx [M+H]$^+$. InChi=1S/C13H14FNO/c1-9-7-11(8-16)10(2)15(9)13-5-3-12(14)4-6-13/h3-7,16H,8H2,1-2H3. See page 44 for NMR spectra. `http://malaria.ourexperiment.org/uri/38`. $NaBH_4$ `http://malaria.ourexperiment.org/uri/3e`

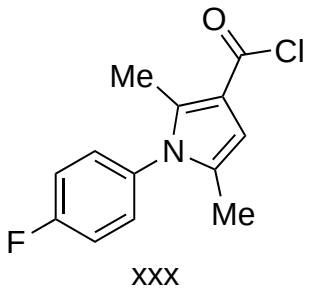
**OSM-S-19, PMY 31-5**




xxx

Mpt.193–195 °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 311 $[M+Na]^+$. InChi=1S/C15H16FN3O2/c1-9-7-13(15(21)18-8-14(17)20)10(2)19(9)12-5-3-11(16)4-6-12/h3-7H,8H2,1-2H3,(H2,17,20)(H,18,21). `http://malaria.ourexperiment.org/uri/8e`.

**OSM-S-20, PMY 32-3**

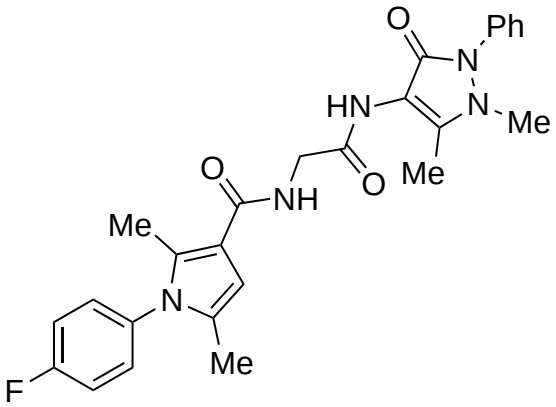



xxx

Prepared according to literature procedure.`http://www.wipo.int/patentscope/search/en/WO2006076202` Used without further purification.

$^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) xxx $[M+H]^+$. InChi=1S/C13H11ClFNO/c1-8-7-12(13(14)17)9(2)16(8)11-5-3-10(15)4-6-11/h3-7H,1-2H3. `http://malaria.ourexperiment.org/uri/bd`.

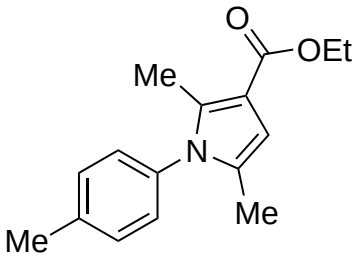
**OSM-S-21, PMY 34-1**




xxx

Mpt.xxx–xxx °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 498 $[M+Na]^+$. InChi=1S/C26H26FN5O3/c1-16-14-22(17(2)31(16)20-12-10-19(27)11-13-20)25(34)28-15-23(33)29-24-18(3)30(4)32(26(24)35)21-8-6-5-7-9-21/h5-14H,15H2,1-4H3,(H,28,34)(H,29,33). `http://malaria.ourexperiment.org/uri/8f`.

**Synthesis of ethyl 2,5-dimethyl-1-(*p*-tolyl)-1*H*-pyrrole-3-carboxylate, OSM-S-30, LMW 6-1**



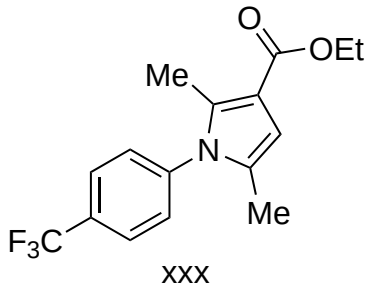

xxx

=== standardise ===

Prepared according to general procedure C using ethyl acetoacetate (2 mL, 15.7 mmol, 1 equiv.) and $K_2CO_3$ (2.82 g, 20.4 mmol, 1.3 equiv.) in MeCN (30 mL) were mixed. Chloroacetone (1.6 mL, 17.2 mmol, 1.1 equiv.) and NaI (2.7 g, 18 mmol, 1.15 equiv.) were added and heated in an oil bath at 80 °C. TLC showed reaction at completion after 2.5 hrs. After 3 hrs reflux, solution was allowed to cool to room temperature and then filtered and washed with EtOAc (30 mL). Mixture was concentrated under reduced

pressure, dissolved in EtOAc (40 mL) and washed with water (20 mL), 1:1 water:brine (20 mL) and brine (20 mL). Crude product was then concentrated under reduced pressure. p-toluidine (2.02 g, 18.8 mmol, 1.2 equiv.) was added to crude intermediate and heated in an oil bath at 90 °C. At 1.5 hrs, reaction was complete by TLC and reaction was allowed to cool to room temperature. Dark brown product was washed with EtOAc (2 × 20 mL), 10% citric acid (3 × 20 mL), water (2 × 20 mL) and brine (20 mL) and then concentrated under reduced pressure to form a black oil. Product was dissolved in EtOH and activated charcoal added to remove coloured impurities. Product was stirred for 1 hr then filtered and washed with EtOH. Filtrate was concentrated under reduced pressure to form a black oil. Oil was purified by chromatography on silica (2-10% EtOAc in petrol). Pure and impure fractions were taken separately and concentrated under reduced pressure to produce yellow and dark yellow oils respectively. Pure fraction crystallised overnight to a bright yellow crystalline solid (1.8 g, 45.6%). Mpt. 60–63 °C (EtOAc/petrol); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.27 (d, $J$ 7.6, 2H), 7.04 (d, $J$ 7.6, 2H), 6.36 (s, 1H), 4.27 (q, $J$ 7.0, 2H), 2.42 (s, 3H), 2.28 (s, 3H), 1.96 (s, 3H), 1.34 (t, $J$ 7.0, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 165.8, 138.5, 136.3, 135.1, 130.0, 128.8, 127.9, 111.3, 107.4, 59.2, 21.2, 14.6, 12.6, 12.4; $\nu_{max}$ (neat) /$cm^{-1}$ 2982, 1693, 1515, 1411, 1218, 1081, 767; $m/z$ (APCI+) 258 $[M+H]^+$. InChi=1S/C16H19NO2/c1-5-19-16(18)15-10-12(3)17(13(15)4)14-8-6-11(2)7-9-14/h6-10H,5H2,1-4H3. `http://malaria.ourexperiment.org/uri/4f`.

**ethyl 2,5-dimethyl-1-[*p*-(trifluoromethyl)phenyl]-1*H*-pyrrole-3-carboxylate, OSM-S-32, LMW 9**




xxx

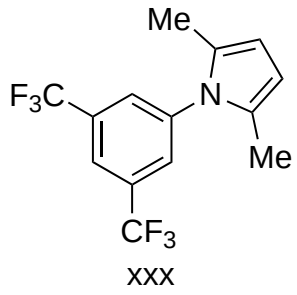
=== standardise ===

Prepared according to general procedure xxx using ethyl acetoacetate (6 mL, 47 mmol, 1 equiv.) and $K_2CO_3$ (8.45 g, 61.1 mmol, 1.3 equiv.) were mixed in MeCN (55 mL). NaI (7.05 g, 47 mmol, 1 equiv.) and Chloroacetone (4.8 mL, 51.7 mmol, 1.1 equiv.) were added and mixture heated to 80oC in an oil bath. TLC at 2 hours showed reaction at completion. Reaction was allowed to cool to room temperature. Mixture washed with

EtOAc (2 x 20 mL), water (2 x 20 mL), 1:1 water:brine (2 x 20 mL) and brine (2 x 20 mL) and dried with MgSO4 and concentrated under reduced pressure to form a yellow oil. Ethyl 2-acetyl-4-oxopentanoate intermediate (LMW 7-1) (2 mL, 10.7 mmol, 1 equiv.) was added to p-(trifluoromethyl)aniline (1.62 mL, 12.9 mmol, 1.2 equiv.) and heated at 80oC in an oil bath for 1.25 hrs. TLC at 1 hour showed reaction at completion and reaction was allowed to cool to room temperature. Product was washed with EtOAc (2 x 20 mL), 10% citric acid (3 x 20 mL), water (20 mL) and brine (2 x 20 mL) and then concentrated under reduced pressure to form a dark brown oil. Brown oil was dissolved in 20 mL EtOH and heated before filtering under heat to remove residual salts and washing with hot EtOH. Filtrate was concentrated under reduced pressure and purified by chromatography on silica (2-15% EtOAc in petrol). Pure fractions were taken and concentrated under reduced pressure to produce a light yellow oil. Product was cooled in a refrigerator overnight forming a pale yellow crystalline solid (1.85 g, 55%).

Mpt. 66–68 °C (EtOAc/petrol); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.78 (d, $J$ 8.0, 2H), 7.33 (d, $J$ 8.0, 2H), 6.41 (s, 1H), 4.28 (q, $J$ 7.0, 2H), 2.30 (s, 3H), 1.99 (s, 3H), 1.35 (t, $J$ 7.0, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 165.5, 141.0, 135.9, 131.0 (q, $J$ 32.9), 128.8, 128.5, 126.6 (q, $J$ 3.3), 123.7 (q, $J$ 272.6), 112.2, 108.2, 59.4, 14.5, 12.7, 12.4; $^{19}$F NMR (282 MHz, $CDCl_3$): -62.7; $\nu_{max}$ (neat) /$cm^{-1}$ 2928, 1681, 1614, 1414, 1322, 1215, 120, 1065, 541, 770; $m/z$ (APCI+) 312 $[M+H]^+$. InChi=1S/C16H16F3NO2/c1-4 -22-15(21)14-9-10(2)20(11(14)3)13-7-5-12(6-8-13)16(17,18)19/h5-9H,4H2,1-3H3. `http://malaria.ourexperiment.org/uri/51`.

**OSM-S-33, ZYH 1-1**



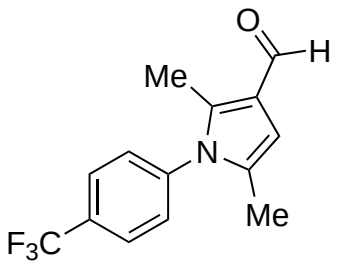
xxx

Prepared according to General Procedure A

$^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 7.72 (d $J$ 0.3Hz, 2H, rotamers), 5.96 (br d, $J$ 1.7, 2H rotamers), 2.07 (br d, rotamers 6H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 140.9, 133.0 (q, $J$ 33.9), 128.8, 128.6 (q, $J$ 3.6), 123.1 (q $J$ 272.6), 121.5 (dq $J$ 3.5 and 3.8), 107.6, 13.1; $\nu_{max}$ (neat)

/$cm^{-1}$ 1713, 1621, 1410, 1274, 1124, 900, 682; $m/z$ HRES (APCI) high res mass, reported is for M-H2 + H. ?!? InChi=1S/C14H11F6N/c1-8-3-4-9(2)21(8)12-6-10(13(15,16)17)5-11(7-12)14(18,19)20/h3-7H,1-2H3. http://malaria.ourexperiment.org/uri/52.

**OSM-S-34, ZYH 2-2**

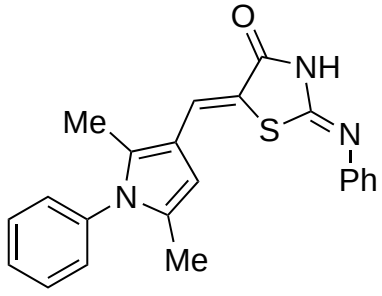



xxx

Prepared according to General Procedure B

Mpt. 85–87 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 9.90 (s, 1H), 7.81 (d, $J$ 8.4, 2H), 7.36 (d, $J$ 8.4, 2H), 6.42 (s, 1H), 2.30 (s, 3H), 2.00 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 185.4, 140.3, 138.4, 131.3 (q, $J$ 32.9), 130.8, 128.7 (2C), 126.9 (q, $J$ 3.6), 123.7 (q, $J$ 272.4), 122.5 (2C), 106.6, 12.8, 11.3; $\nu_{max}$ (neat) /$cm^{-1}$ 16558, 1614, 1520, 1424, 1401, 1321; $m/z$ (ESI+ )268 $[M+H]^+$; HRES (ESI+) 290.07619 $[M+Na]^+$, $C_{14}H_{12}F_3NNaO$ requires 290.07687. InChi=1S/C14H12F3NO/c1-9-7-11(8-19)10(2)18(9)13-5-3-12(4-6-13)14(15,16)17/h3-8H,1-2H3. http://malaria.ourexperiment.org/uri/58.

**(2$Z$,5$Z$)-5-((2,5-dimethyl-1-phenyl-1$H$-pyrrol-3-yl)methylene)-2-(phenylimino)thiazolidin-4-one, OSM-S-35, ZYH 3-1**

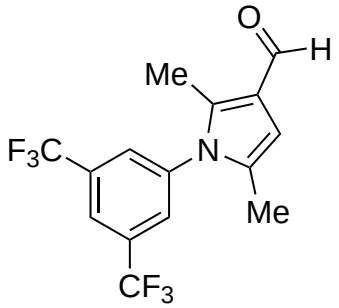



xxx

Prepared according to general procedure F

Mpt. 273 °C (decomposes); $^{1}$H NMR (300 MHz, DMSO−$d_6$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) /$cm^{-1}$ 1703, 1637, 1591, 1495, 1300, 1173; *m/z* (ESI+) 374 $[M+H]^+$; HRES (ESI+) 374.13105 $[M+H]^+$, $C_{22}H_{20}N_3OS$ requires 374.13271. InChi=1S/C22H19N3OS/c1-15-13-17(16(2)25(15)19-11-7-4-8-12-19)14-20-21(26)24-22(27-20)23-18-9-5-3-6-10-18/h3-14H,1-2H3,(H,23,24,26)/b20-14-. `http://malaria.ourexperiment.org/uri/54`.

**1-(3,5-bis(trifluoromethyl)phenyl)-2,5-dimethyl-1*H*-pyrrole-3-carbaldehyde, OSM-S-36, ZYH 4-2/4-3**

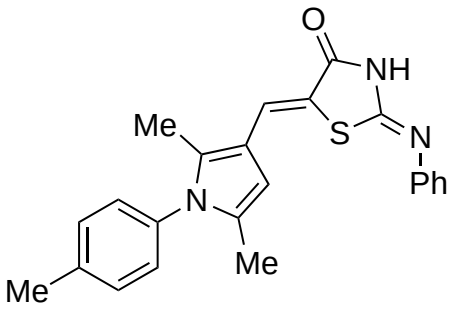



xxx

Prepared according to General Procedure B

Mpt. 95–96 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ 9.90 (s, 1H), 8.04 (s, 1H), 7.74 (s, 2H), 6.44 (app d, *J* 0.7, rotamers, 1H), 2.33 (s, 3H), 2.03 (s, 3H); $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ 185.4, 138.8, 138.0, 133.6 (q, *J* 34.3, 2C), 130.6, 128.7 (q, *J* 3.4, 2C), 123.0 (dq *J* 3.8 and 3.9, 2C), 122.9, 122.7 (q, *J* 273.1, 2C), 107.4, 12.8, 11.4; $\nu_{max}$ (neat) /$cm^{-1}$ ; *m/z* HRES (ESI+) found 336.08190 $[M+H]^+$, $C_{15}H_{11}F_6NO$ requires 336.08176. InChi=1S/C15H11F6NO/c1-8-3-10(7-23)9(2)22(8)13-5-11(14(16,17)18)4-12(6-13)15(19,20)21/h3-7H,1-2H3. `http://malaria.ourexperiment.org/uri/55`.

**(2*Z*,5*Z*4)-5-((2,5-dimethyl-1-(p-tolyl)-1*H*-pyrrol-3-yl)methylene)-2-(phenylimino)thiazolid 4-one, OSM-S-37, ZYH 5-1**

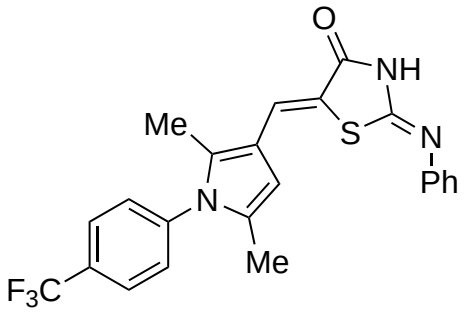



xxx

Prepared according to general procedure F

Mpt. 279 °C (decomposes); $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; *m/z* (ESI+) 388 $[M+H]^+$; HRES (ESI+) found 388.14765 $[M+H]^+$, $C_{23}H_{22}N_3OS$ requires 388.14836. InChi=1S/C23H21N3OS/c1-15-9-11-20(12-10-15)26-16 (2)13-18(17(26)3)14-21-22(27)25-23(28-21)24-19-7-5-4-6-8-19/h4-14H,1-3H3,(H,24,25,27) /b21-14-. `http://malaria.ourexperiment.org/uri/56`.

**(2*Z*,5*Z*)-5-((2,5-dimethyl-1-(4-(trifluoromethyl)phenyl)-1*H*-pyrrol-3-yl)meth ylene)-2-(phenylimino)thiazolidin-4-one,OSM-S-38,ZYH6**

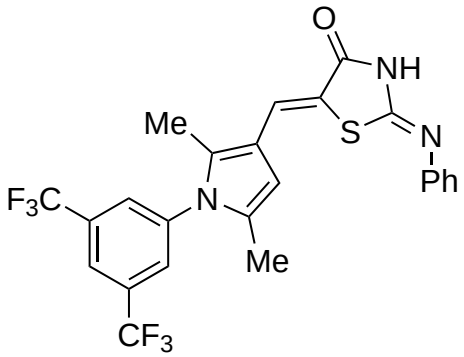



xxx

Prepared according to general procedure F

Mpt. 308 °C (decomp); $^1$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; *m/z* (ESI+) xxx $[M+H]^+$. InChi=1S/C23H18F3N3OS/c1-14-12-16 (15(2)29(14)19-10-8-17(9-11-19)23(24,25)26)13-20-21(30)28-22(31-20)27-18-6-4-3-5-7-18 /h3-13H,1-2H3,(H,27,28,30)/b20-13-. `http://malaria.ourexperiment.org/uri/59`.

**OSM-S-39, ZYH 7-2**

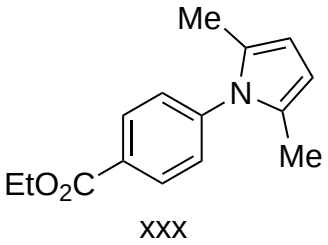



xxx

Prepared according to general procedure F

Mpt. 255 (decomposes) °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ HRES (ESI+) 510.10796 $[M+H]^+$, $C_{24}H_{18}F_6N_3OS$ requires 510.10748. InChi=1S/C24H17F6N3OS/c1-13-8-15(9-20-21(34)32-22(35-20)31-18-6-4-3-5-7-18)14(2)33(13)19-11-16(23(25,26)27)10-17(12-19)24(28,29)30/h3-12H,1-2H3,(H,31,32,34)/b20-9-. `http://malaria.ourexperiment.org/uri/64`.

**OSM-S-40, ZYH 8-1**

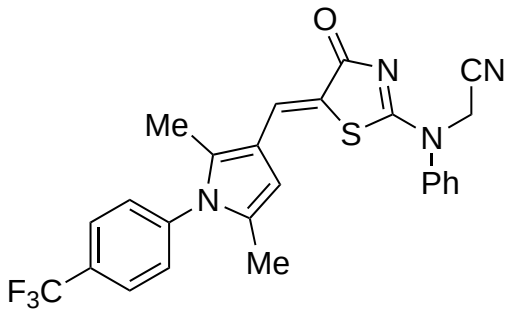



xxx

Prepared according to General Procedure A

Mpt. 60–61 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (APCI) 244 $[M+H]^+$. InChi=1S/C15H17NO2/c1-4-18-15(17)13-7-9-14(10-8-13)16-11(2)5-6-12(16)3/h5-10H,4H2,1-3H3. `http://malaria.ourexperiment.org/uri/5f`.

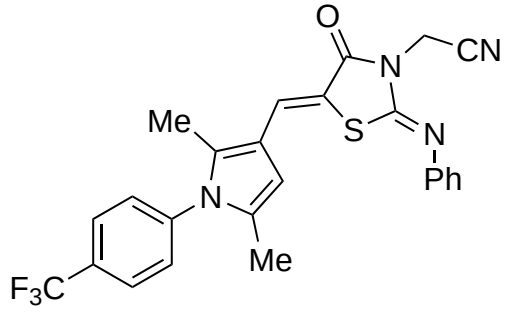
**OSM-S-42, ZYH 10-1 A**




xxx

Mpt. 182–183 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 982 $[2M+Na]^+$; HRES (ESI+) 503.11264 $[M+Na]^+$, $C_{25}H_{19}F_3N_4NaOS$ requires 503.11294. InChi=1S/C25H19F3N4OS/c1-16-14-18(17(2)32(16)21-10-8-19(9-11-21)25(26,27)28)15-22-23(33)30-24(34-22)31(13-12-29)20-6-4-3-5-7-20/h3-11,14-15H,13H2,1-2H3/b22-15-. `http://malaria.ourexperiment.org/uri/61`.

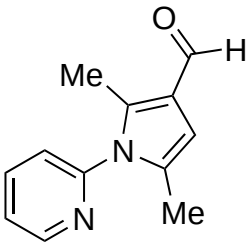
**OSM-S-43, ZYH 10-1 B**




xxx

Mpt. 72–74 °C (xxx); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 503 $[M+Na]^+$; HRES (ESI+) 503.11295 $[M+Na]^+$, $C_{25}H_{19}F_3N_4NaOS$ requires 503.11294. InChi=1S/C25H19F3N4OS/c1-16-14-18(17(2)32(16)21-10-8-19(9-11-21)25(26,27)28)15-22-23(33)31(13-12-29)24(34-22)30-20-6-4-3-5-7-20/h3-11,14-15H,13H2,1-2H3/b22-15-,30-24-. `http://malaria.ourexperiment.org/uri/61`.

**OSM-S-44, ZYH 11-1**

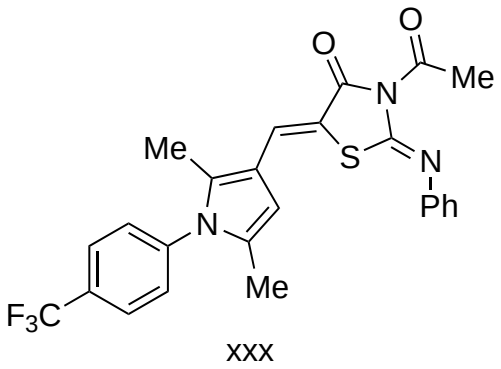



xxx

Prepared accoding to General Procedure B

Mpt. 61–64 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ HRES (APCI) 201.10165 $[M+H]^+$, $C_{12}H_{13}N_2O$ requires 201.10279. InChi=1S/C12H12N2O/c1-9-7-11(8-15)10(2)14(9)12-5-3-4-6-13-12/h3-8H,1-2H3. `http://malaria.ourexperiment.org/uri/67`.

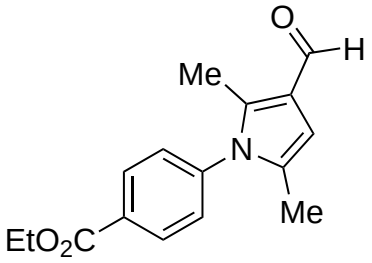
**ZYH 12-1/12-2**




xxx

Mpt. 309–311 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ HRES (ESI+) 506.11167 $[M+Na]^+$, $C_{25}H_{20}F_3N_3NaO_2S$ requires 506.11260. InChi=1S/C25H20F3N3O2S/c1-15-13-18(16(2)30(15)21-11-9-19(10-12-21)25(26,27)28)14-22-23(33)31(17(3)32)24(34-22)29-20-7-5-4-6-8-20/h4-14H,1-3H3/b22-14+,29-24-. `http://malaria.ourexperiment.org/uri/74`.

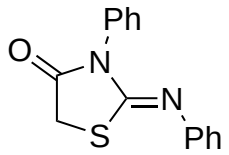
**OSM-S-46, ZYH 13-1**




xxx

Mpt. 112–114 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (APCI) 272 $[M+H]^+$. InChi=1S/C16H17NO3/c1-4-20-16(19)13-5-7-15(8-6-13)17-11(2)9-14(10-18)12(17)3/h5-10H,4H2,1-3H3. `http://malaria.ourexperiment.org/uri/69`.

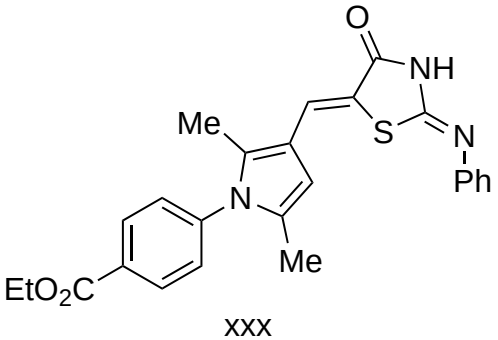
**OSM-S-47, ZYH 14-1**




xxx

Mpt. 175–176 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (APCI) 269 $[M+H]^+$. InChi=1S/C15H12N2OS/c18-14-11-19-15(16-12-7-3-1-4-8-12)17(14)13-9-5-2-6-10-13/h1-10H,11H2/b16-15-. `http://malaria.ourexperiment.org/uri/70`.

**OSM-S-48, ZYH 15-1**

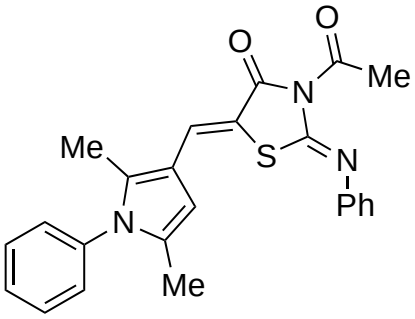



xxx

Prepared according to general procedure F

Mpt. 248 °C (decomposes); $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 913 $[2M+Na]^+$; HRES (ESI+) 468.13527 $[M+Na]^+$, $C_{25}H_{23}N_3NaO_3S$ requires 468.13578. InChi=1S/C25H23N3O3S/c1-4-31-24(30)18-10-12-21(13-11-18)28-16(2)14-19(17(28)3)15-22-23(29)27-25(32-22)26-20-8-6-5-7-9-20/h5-15H,4H2,1-3H3,(H,26,27,29)/b22-15-. `http://malaria.ourexperiment.org/uri/71`.

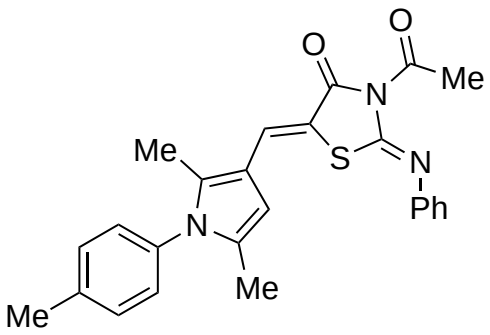
**OSM-S-49, ZYH 16-1**




xxx

Mpt. 237–238 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ HRES (ESI+) 416.14285 $[M+H]^+$, $C_{24}H_{22}N_3O_2S$ requires 416.14327. InChi=1S/C24H21N3O2S/c1-16-14-19(17(2)26(16)21-12-8-5-9-13-21)15-22-23(29)27(18(3)28)24(30-22)25-20-10-6-4-7-11-20/h4-15H,1-3H3/b22-15-,25-24-. `http://malaria.ourexperiment.org/uri/77`.

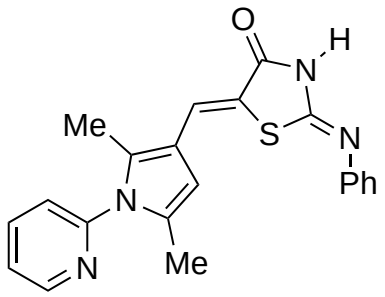
**OSM-S-50, ZYH 17-1**




xxx

Mpt. 256–258 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ HRES (ESI+) 388.14793 [M-$C_2H_3O$+H]$^+$, $C_{23}H_{22}N_3OS$ requires 388.14836. InChi=1S/C25H23N3O2S/c1-16-10-12-22(13-11-16)27-17(2)14-20(18(27)3)15-23-24(30)28(19(4)29)25(31-23)26-21-8-6-5-7-9-21/h5-15H,1-4H3/b23-15-,26-25-. `http://malaria.ourexperiment.org/uri/78`.

**OSM-S-51, ZYH 18-1**

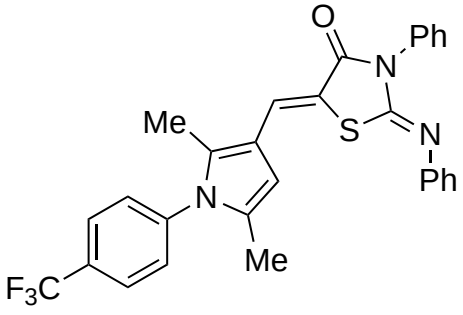



xxx

Prepared according to general procedure F

Mpt. 276–278 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 397 [M+Na]$^+$; HRES (ESI+) 375.12680 [M+H]$^+$, $C_{21}H_{19}N_4OS$ requires 375.12796. InChi=1S/C21H18N4OS/c1-14-12-16(15(2)25(14)19-10-6-7-11-22-19)13-18-20(26)24-21(27-18)23-17-8-4-3-5-9-17/h3-13H,1-2H3,(H,23,24,26)/b18-13-. `http://malaria.ourexperiment.org/uri/79`.

**OSM-S-52, ZYH 19-1**

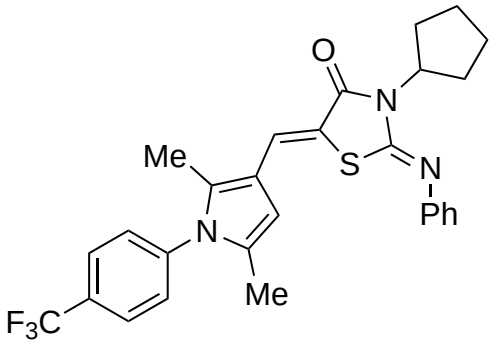



xxx

Prepared according to general procedure F

Mpt. 226–227 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ (ESI+) 540 $[M+Na]^+$; HRES (ESI+) 540.13240 $[M+Na]^+$, $C_{29}H_{22}F_3N_3NaOS$ requires 540.13334. InChi=1S/C29H22F3N3OS/c1-19-17-21(20(2)34(19)25-15-13-22(14-16-25)29(30,31)32)18-26-27(36)35(24-11-7-4-8-12-24)28(37-26)33-23-9-5-3-6-10-23/h3-18H,1-2H3/b26-18-,33-28-. http://malaria.ourexperiment.org/uri/7a.

**OSM-S-54, ZYH 22-3**

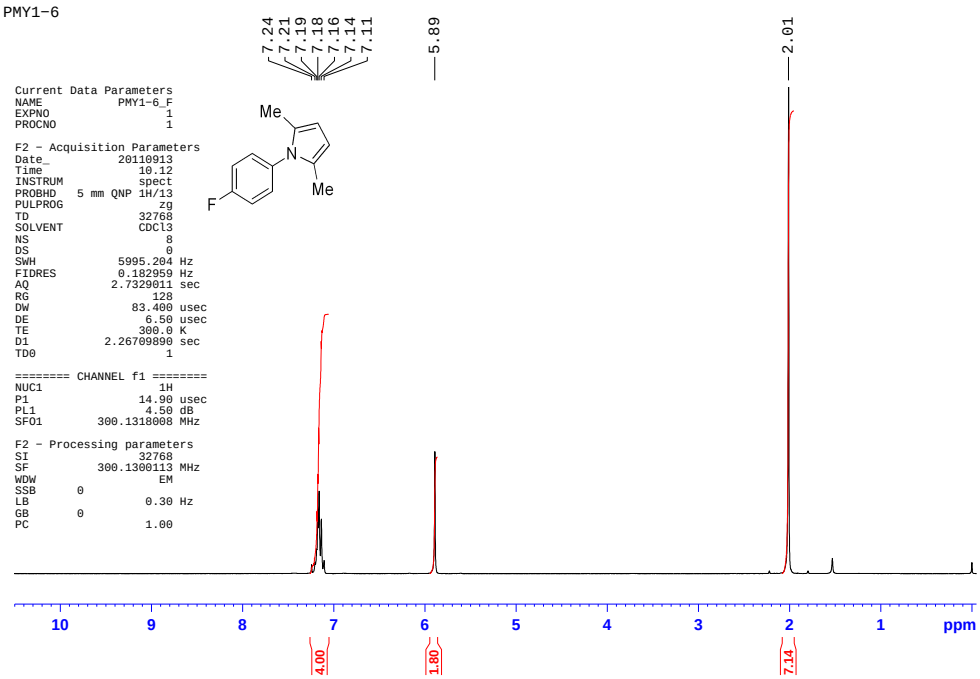


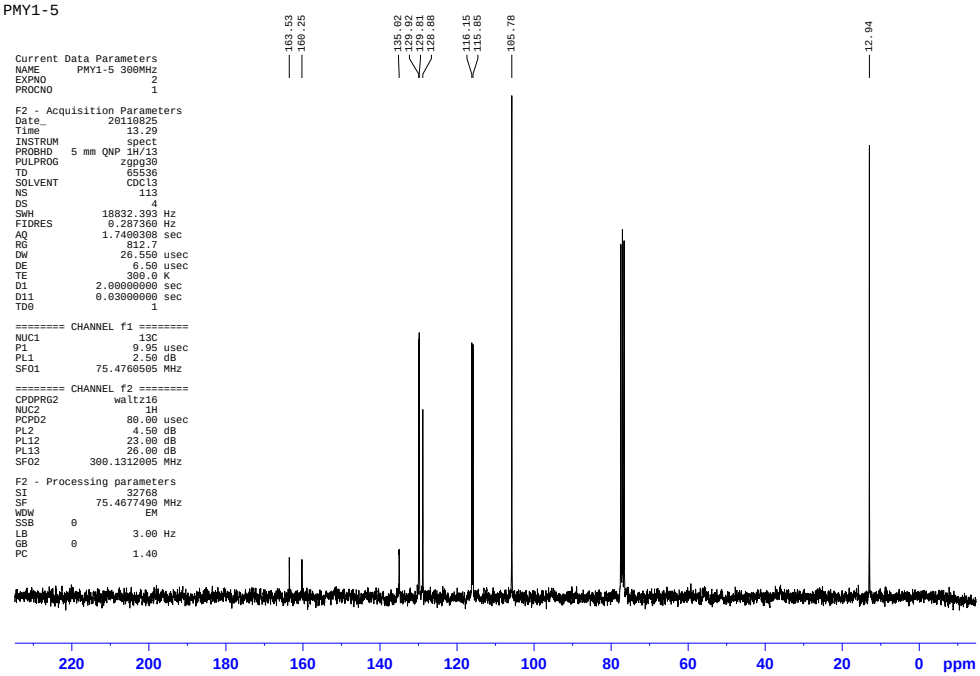

xxx

Mpt. 71–72 °C; $^{1}$H NMR (300 MHz, $CDCl_3$) $\delta$ ; $^{13}$C NMR (75 MHz, $CDCl_3$) $\delta$ ; $\nu_{max}$ (neat) $/cm^{-1}$ ; $m/z$ HRES (ESI+) 510.18203 $[M+H]^+$, $C_{28}H_{27}F_3N_3OS$ requires 510.18269. InChi=1S/C28H26F3N3OS/c1-18-16-20(19(2)33(18)24-14-12-21(13-15-24)28(29,30)31)17-25-26(35)34(23-10-6-7-11-23)27(36-25)32-22-8-4-3-5-9-22/h3-5,8-9,12-17,23H,6-7,10-11H2,1-2H3/b25-17-,32-27-. http://malaria.ourexperiment.org/uri/83.

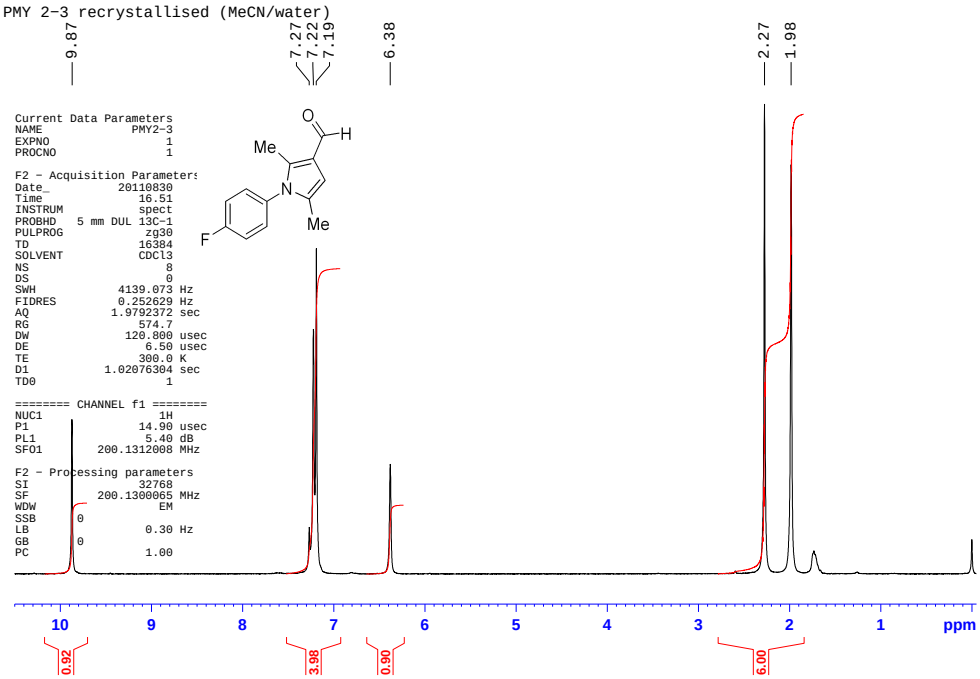
# 4 NMR Spectra

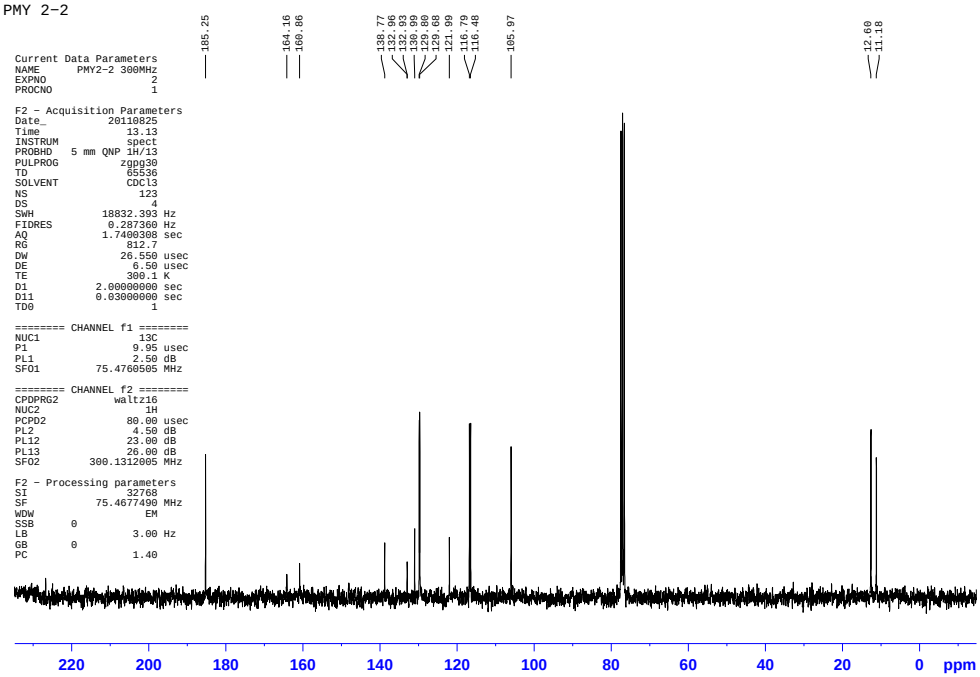
# PMY 1, 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole,l page 2



# PMY 2, 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carbaldehyde, page 3



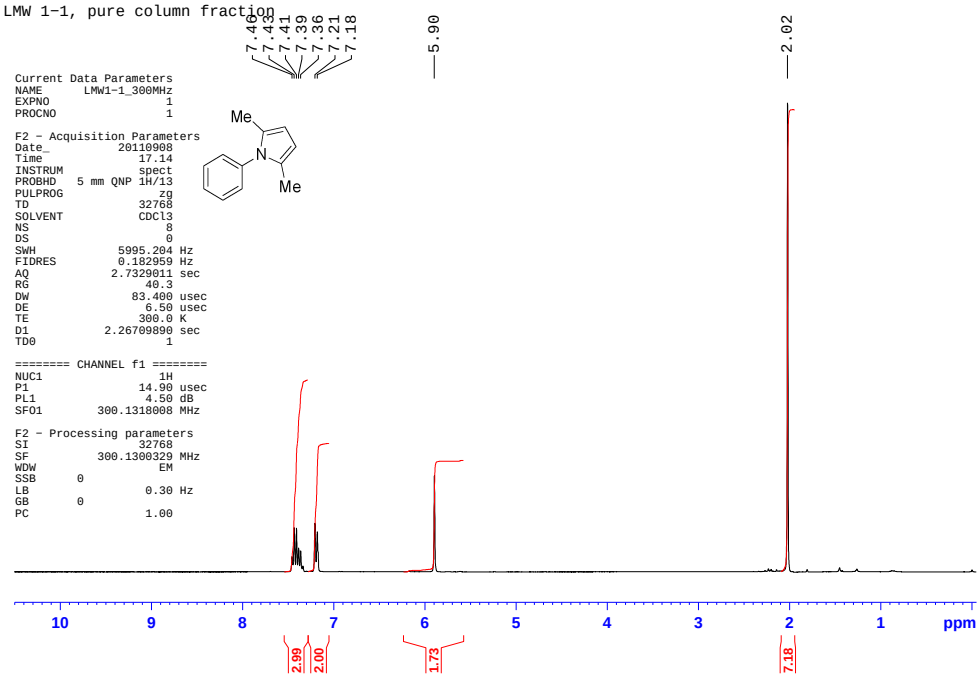
## PMY 26-1

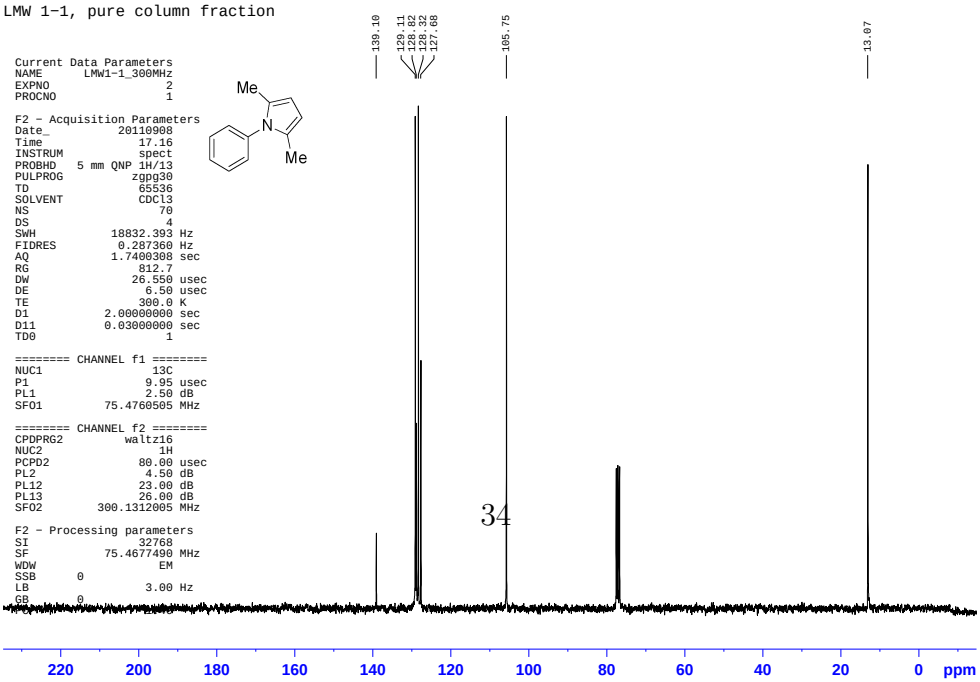
## PMY 27-2

## PMY 29-1, PMY 29-2

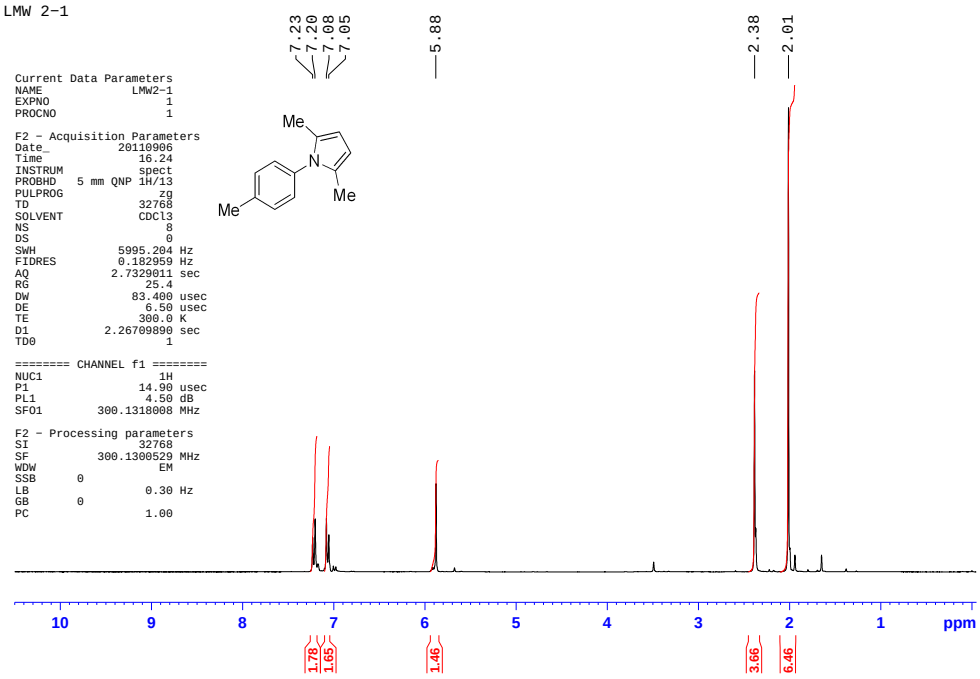
## PMY 30-3

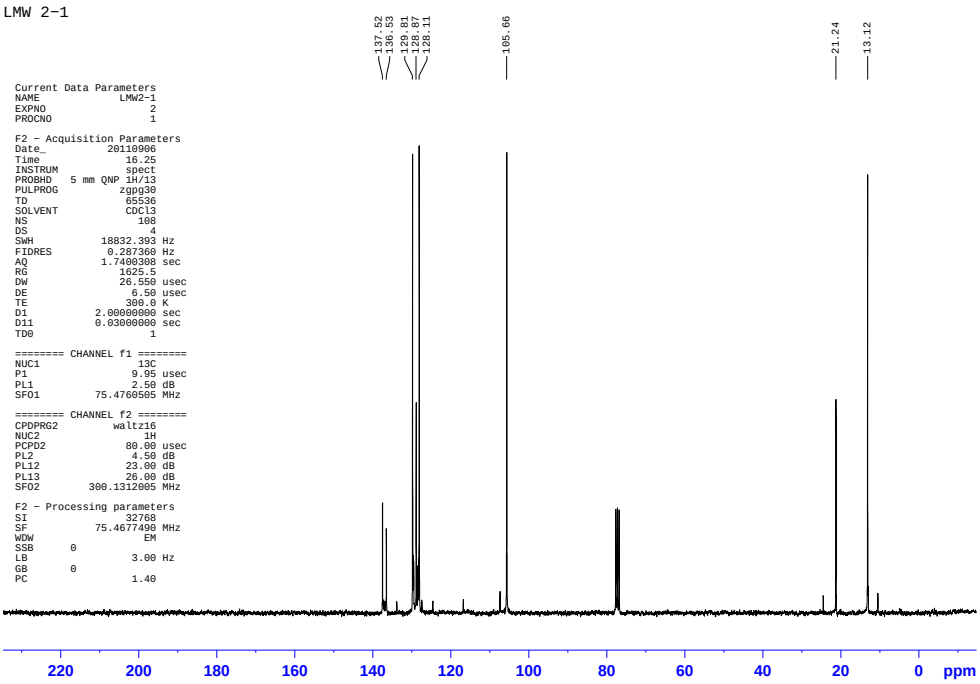
## LMW 1, 6



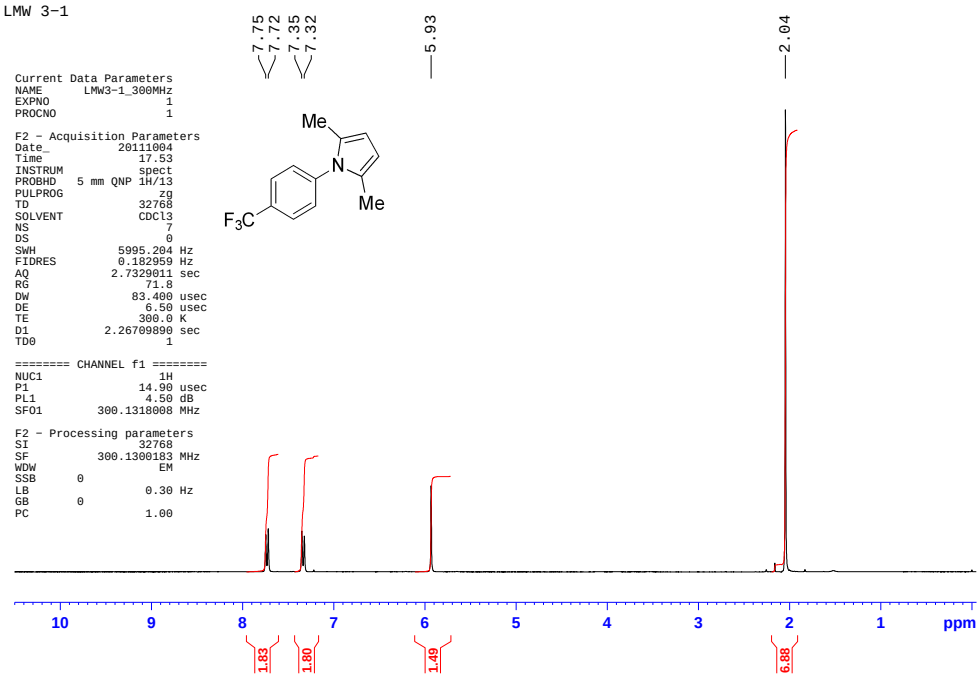



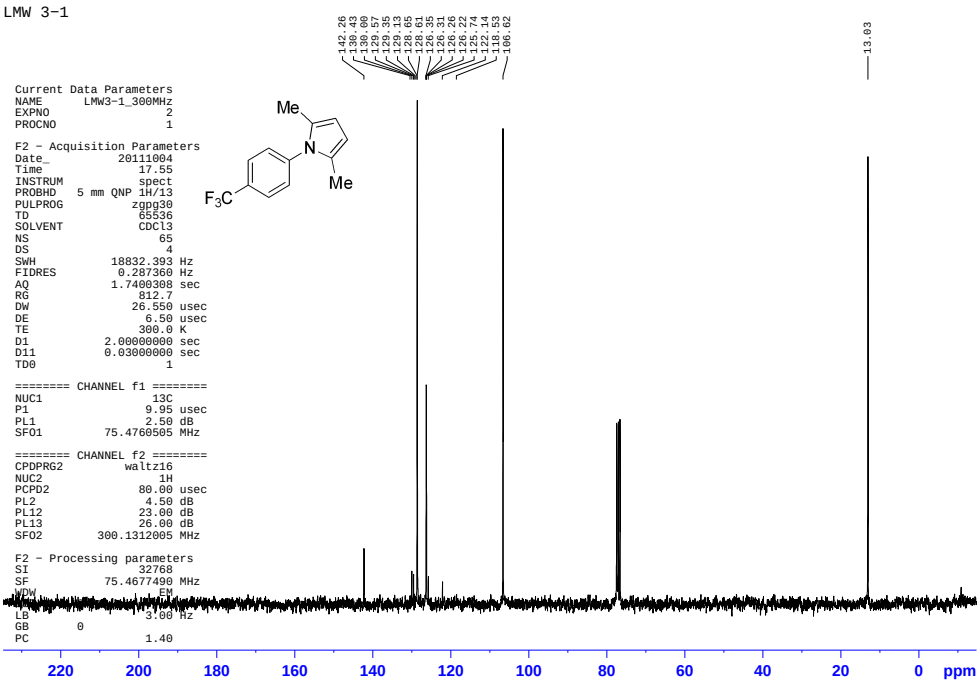
# LMW 2, 6


LMW 2-1
7.23
7.20
7.08
7.05
5.88
2.38
2.01
Me
N
Me
Me
Current Data Parameters
NAME LMW2-1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110906
Time 16.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 25.4
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 2.26709890 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 4.50 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 32768
SF 300.1300529 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.78
1.65
1.46
3.66
6.46

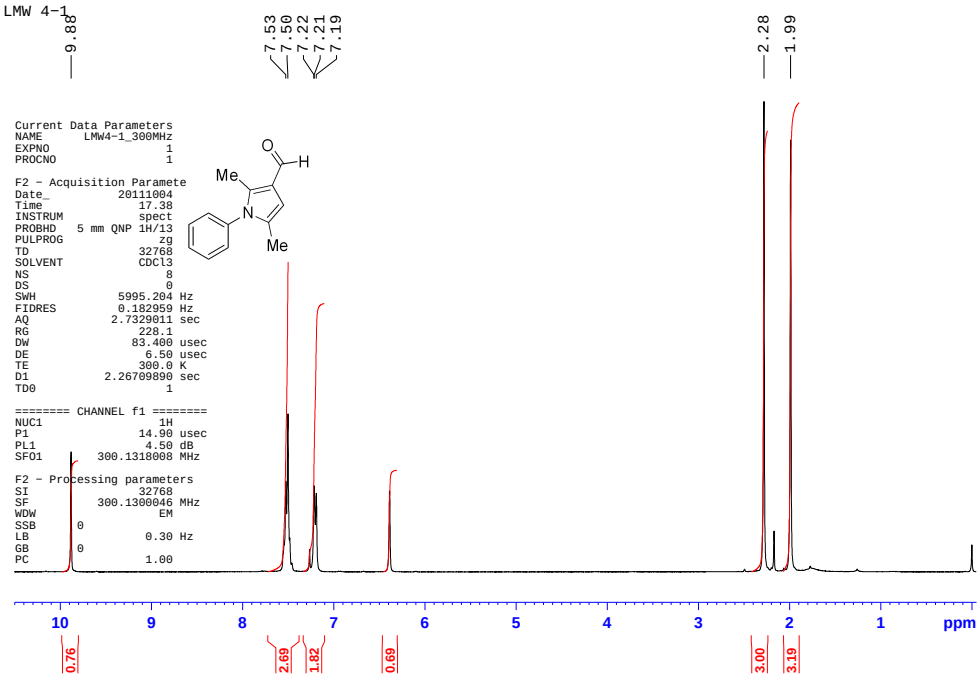

LMW 2-1
137.52
136.53
129.81
128.87
128.11
105.66
21.24
13.12
Current Data Parameters
NAME LMW2-1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20110906
Time 16.25
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 108
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 1625.5
DW 26.550 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.95 usec
PL1 2.50 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.50 dB
PL12 23.00 dB
PL13 26.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
0
ppm

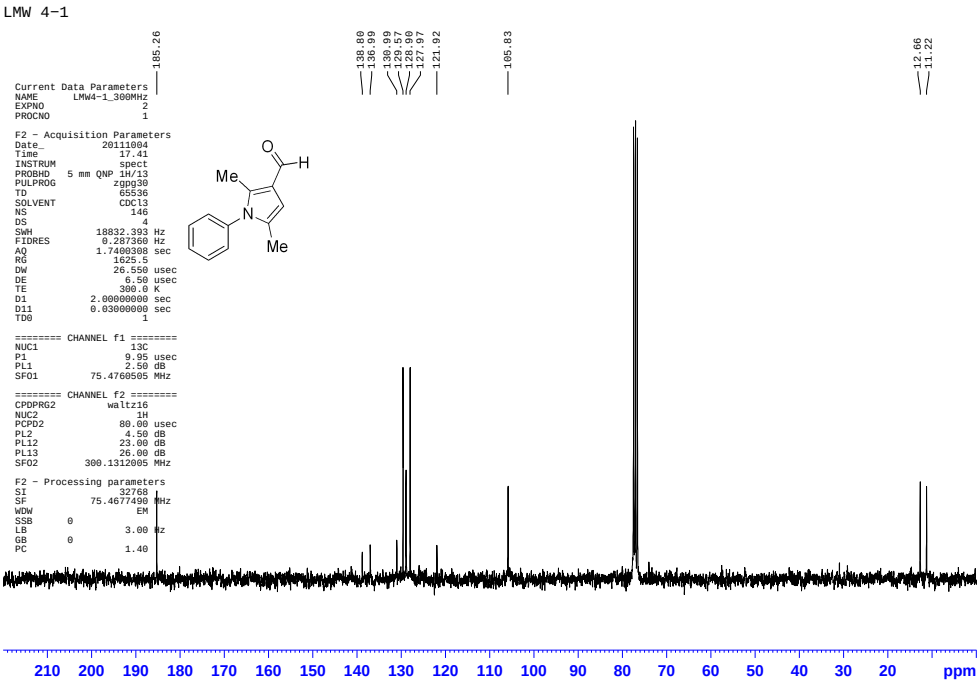
# LMW 3, 6


LMW 3-1
7.75
7.72
7.35
7.32
5.93
2.04
Me
N
Me
F3C
Current Data Parameters
NAME LMW3-1_300MHz
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 17.53
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 7
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 71.8
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 2.26709890 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 4.50 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 32768
SF 300.1300183 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
10
9
8
7
6
5
4
3
2
1
ppm
1.83
1.80
1.49
6.88

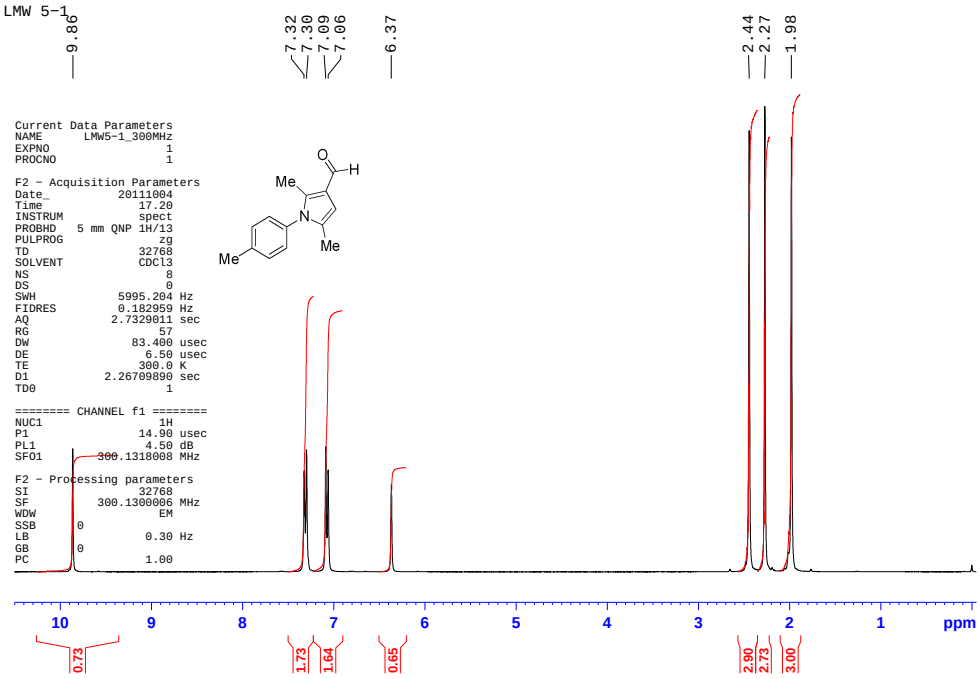

LMW 3-1
142.26
130.43
130.00
129.57
129.35
129.13
128.65
128.61
126.35
126.31
126.26
126.22
125.74
122.14
118.53
106.62
13.03
Me
N
Me
F3C
Current Data Parameters
NAME LMW3-1_300MHz
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 17.55
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 65
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 812.7
DW 26.550 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.95 usec
PL1 2.50 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.50 dB
PL12 23.00 dB
PL13 26.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
LB 3.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
0
ppm

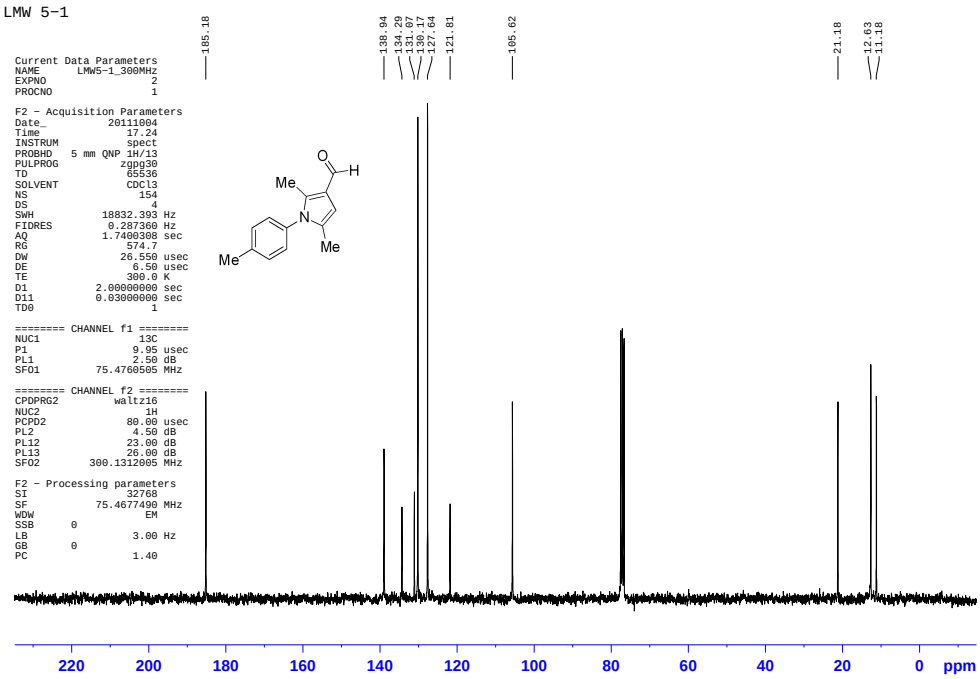
# LMW 4-1, 7


LMW 4-1
9.88
7.53
7.50
7.22
7.21
7.19
2.28
1.99
Current Data Parameters
NAME LMW4-1_300MHz
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 17.38
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 228.1
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 2.26709890 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 4.50 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 32768
SF 300.1300046 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
H
Me
N
Me
10
9
8
7
6
5
4
3
2
1
ppm
0.76
2.69
1.82
0.69
3.00
3.19


LMW 4-1
185.26
138.80
136.99
130.99
129.57
128.90
127.97
121.92
105.83
12.66
11.22
Current Data Parameters
NAME LMW4-1_300MHz
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 17.41
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 146
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7400308 sec
RG 1625.5
DW 26.550 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.95 usec
PL1 2.50 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.50 dB
PL12 23.00 dB
PL13 26.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
O
H
Me
N
Me
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

# LMW 5-1, 8


LMW 5-1
9.86
7.32
7.30
7.09
7.06
6.37
2.44
2.27
1.98
Current Data Parameters
NAME LMW5-1_300MHz
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 17.20
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 57
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 2.26709890 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 4.50 dB
SFO1 300.1318008 MHz
F2 - Processing parameters
SI 32768
SF 300.1300006 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
Me
Me
Me
O
H
N
10
9
8
7
6
5
4
3
2
1
ppm
0.73
1.73
1.64
0.65
2.90
2.73
3.00


LMW 5-1
185.18
138.94
134.29
131.67
130.17
127.64
121.81
105.62
21.18
12.63
11.18
Current Data Parameters
NAME LMW5-1_300MHz
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111004
Time 17.24
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 154
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7408308 sec
RG 574.7
DW 26.550 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.95 usec
PL1 2.50 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.50 dB
PL12 23.00 dB
PL13 26.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
Me
Me
Me
O
H
N
220
200
180
160
140
120
100
80
60
40
20
0
ppm

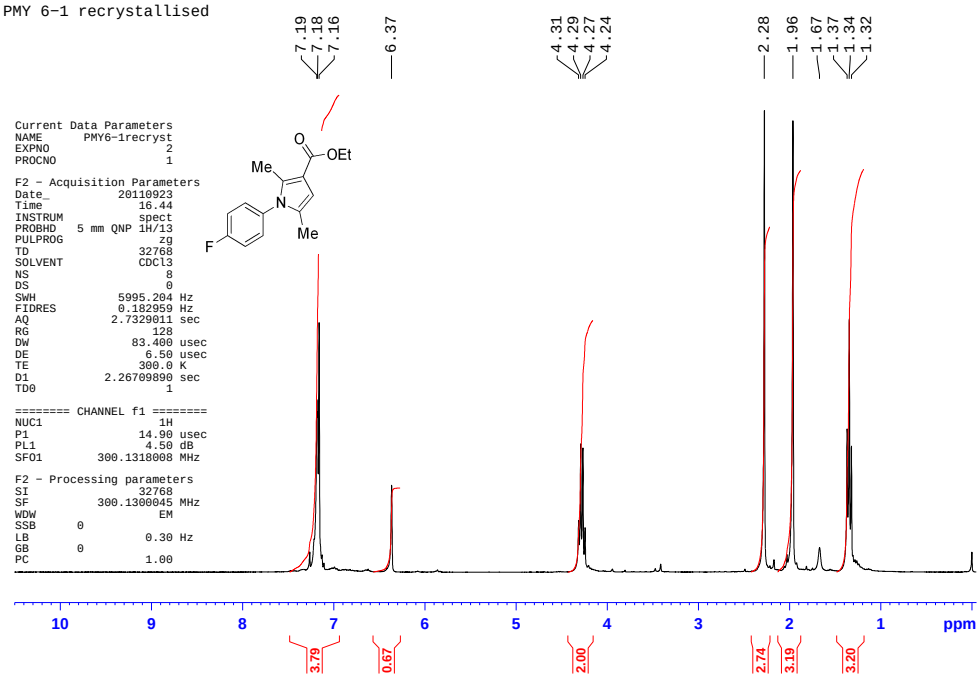
**LMW 8-1**

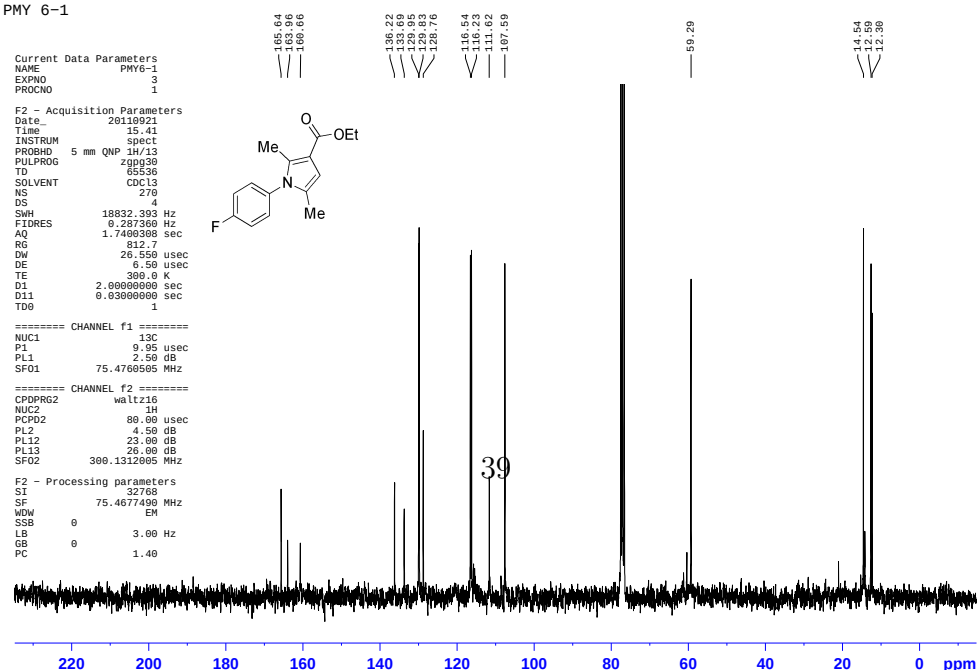
**ZYH 9-1**

**ZYH 20-1**

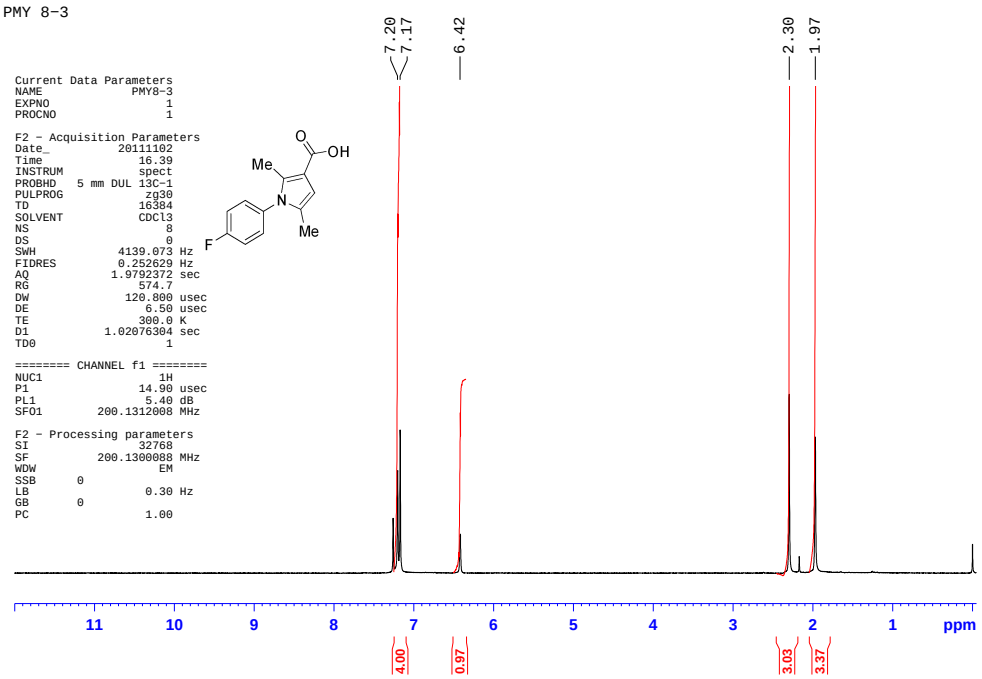
**ZYH 23-1**

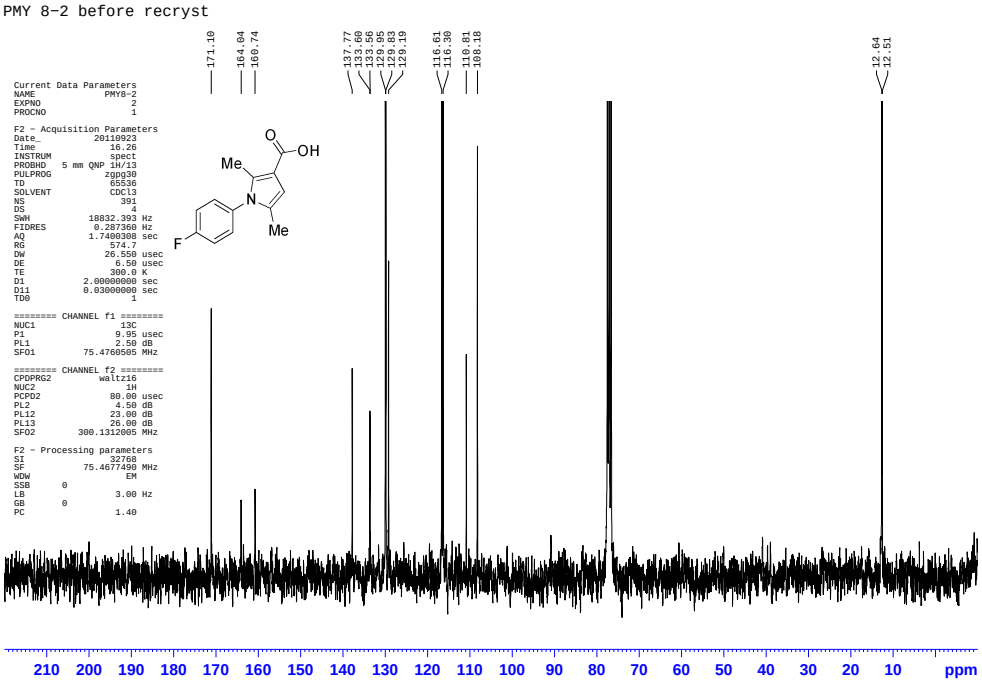
**OSM-S-3, PMY 6-1, ethyl 1-(4-fluorophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carboxylate, page 11**

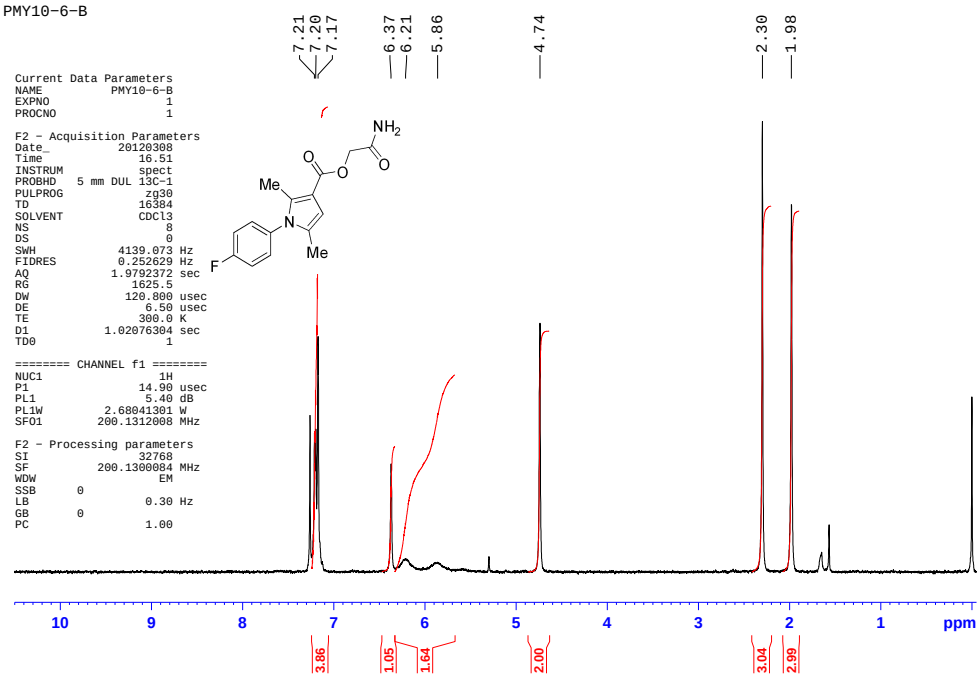


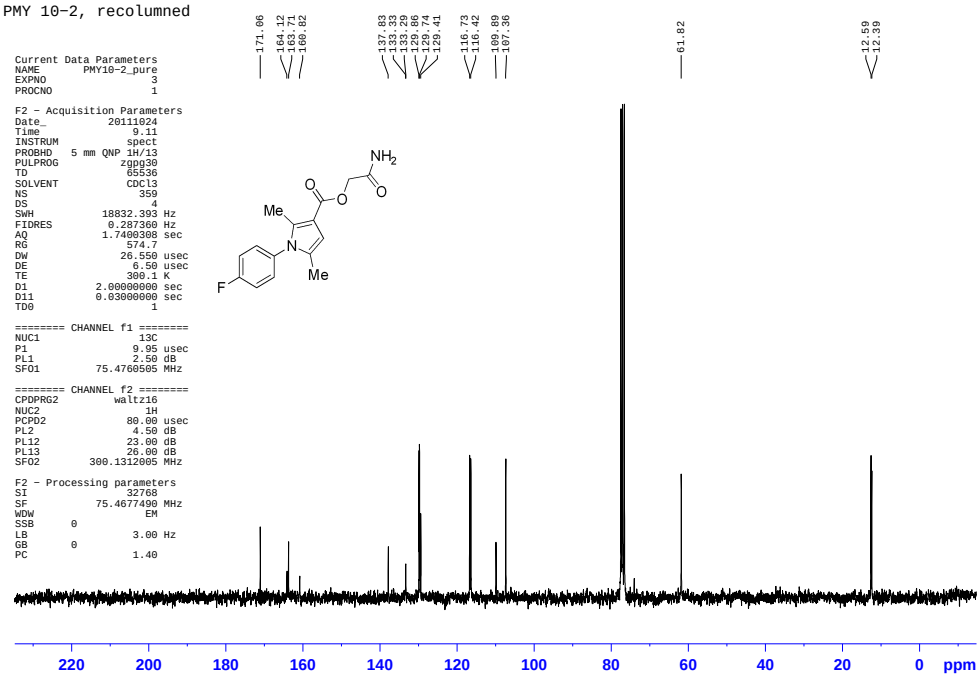



## OSM-S-8, PMY 8-2, 1-(4-fluorophenyl)-2,5-dimethyl-1$H$-pyrrole-3-carboxylic acid, page 12



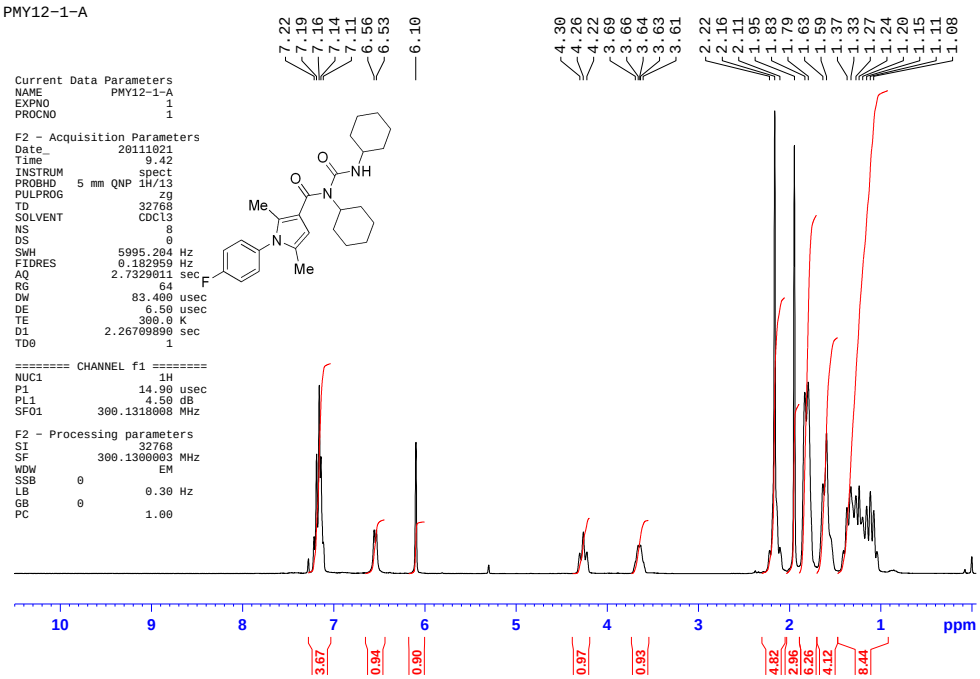



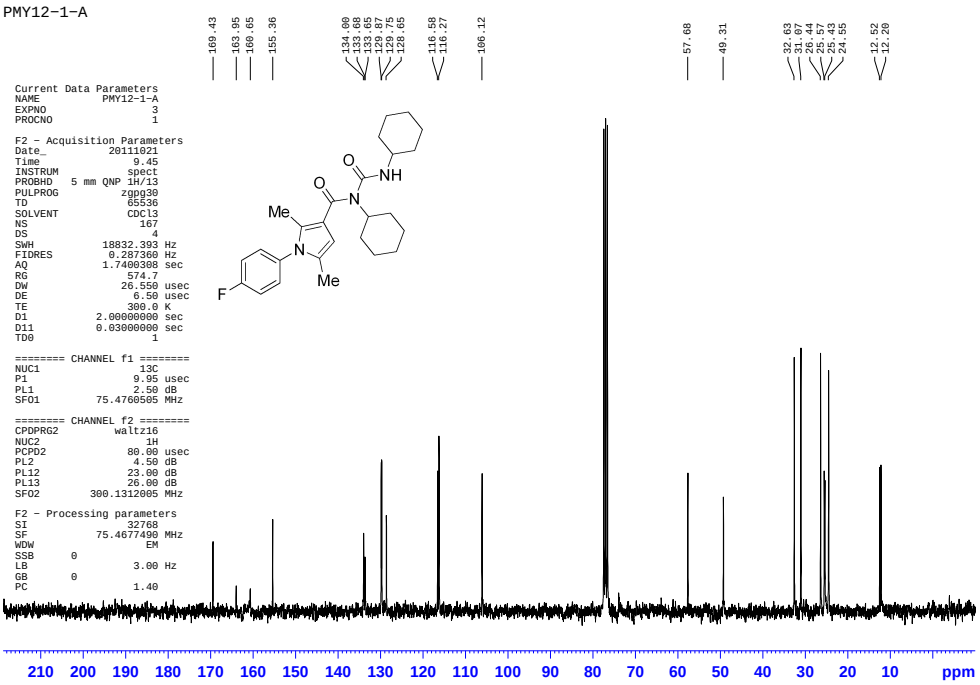

PMY10-6-B
7.21
7.20
7.17
6.37
6.21
5.86
4.74
2.30
1.98
Current Data Parameters
NAME PMY10-6-B
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20120308
Time 16.51
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 4139.073 Hz
FIDRES 0.252629 Hz
AQ 1.9792372 sec
RG 1625.5
DW 120.800 usec
DE 6.50 usec
TE 300.0 K
D1 1.02076304 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 14.90 usec
PL1 5.40 dB
PL1W 2.68041301 W
SFO1 200.1312008 MHz
F2 - Processing parameters
SI 32768
SF 200.1300084 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
NH2
O
O
O
Me
N
Me
F
10
9
8
7
6
5
4
3
2
1
ppm
3.86
1.05
1.64
2.00
3.04
2.99


PMY 10-2, recolumned
171.06
164.12
163.71
160.62
137.83
133.33
133.29
129.86
129.74
129.41
116.73
116.42
109.89
107.36
61.82
12.59
12.39
Current Data Parameters
NAME PMY10-2_pure
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20111024
Time 9.11
INSTRUM spect
PROBHD 5 mm QNP 1H/13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 359
DS 4
SWH 18832.393 Hz
FIDRES 0.287360 Hz
AQ 1.7408308 sec
RG 574.7
DW 26.550 usec
DE 6.50 usec
TE 300.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.95 usec
PL1 2.50 dB
SFO1 75.4760505 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 4.50 dB
PL12 23.00 dB
PL13 26.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 75.4677490 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.40
NH2
O
O
O
Me
N
Me
F
220
200
180
160
140
120
100
80
60
40
20
0
ppm

## PMY 11-2

## PMY 12-1, page 14

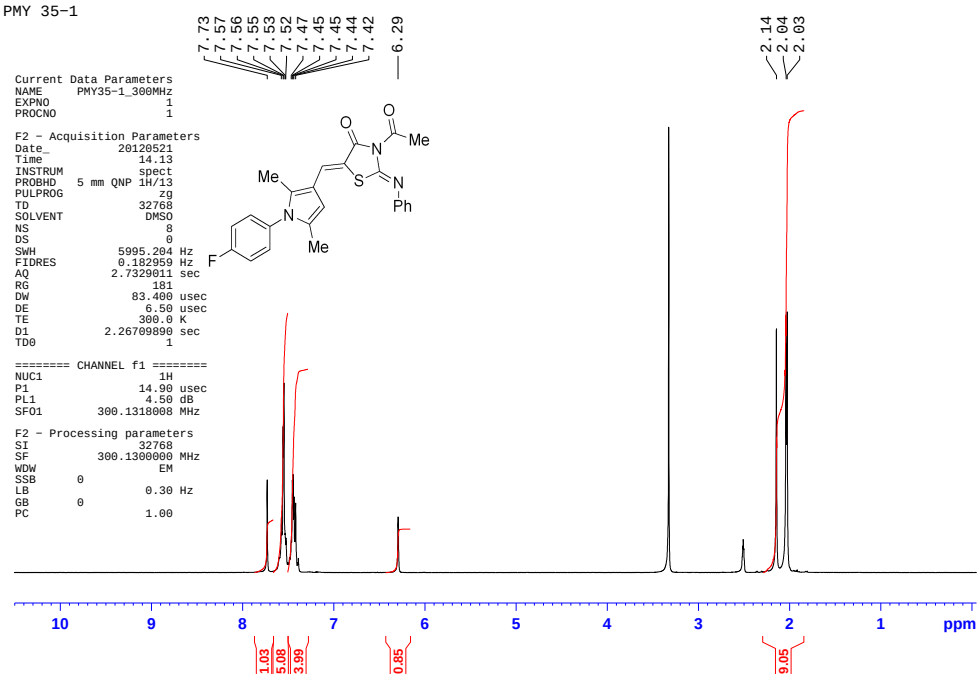


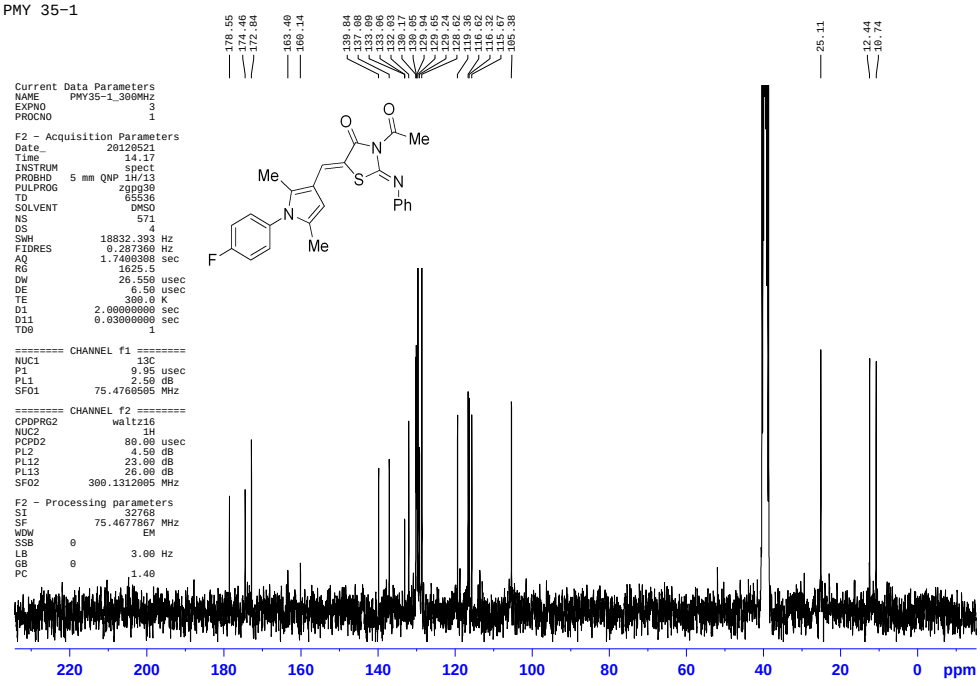



## PMY 12-5

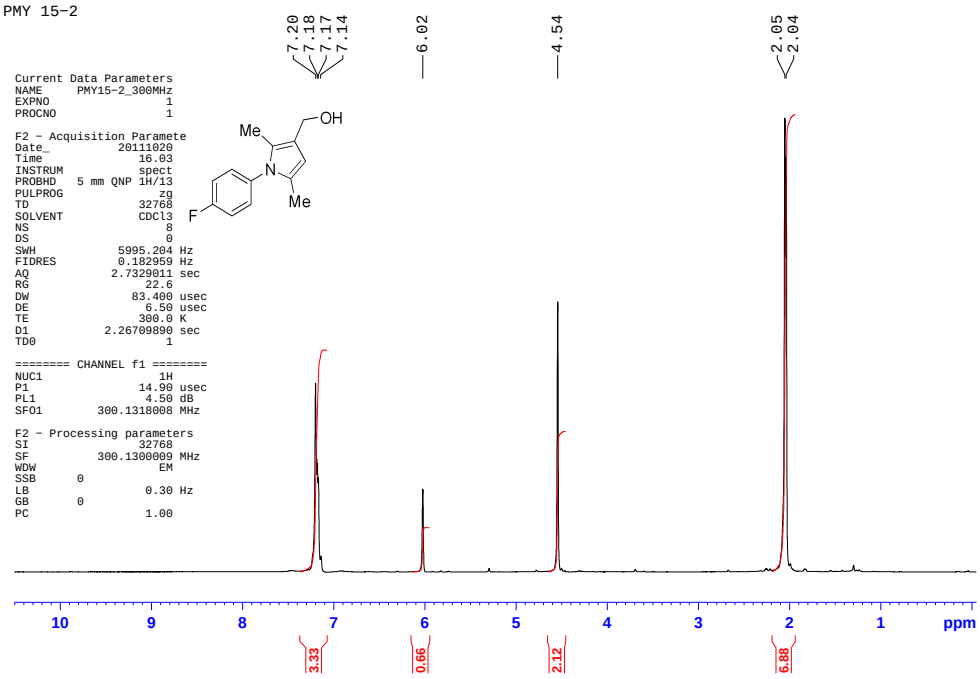
## OSM-S-10, PMY 14-4

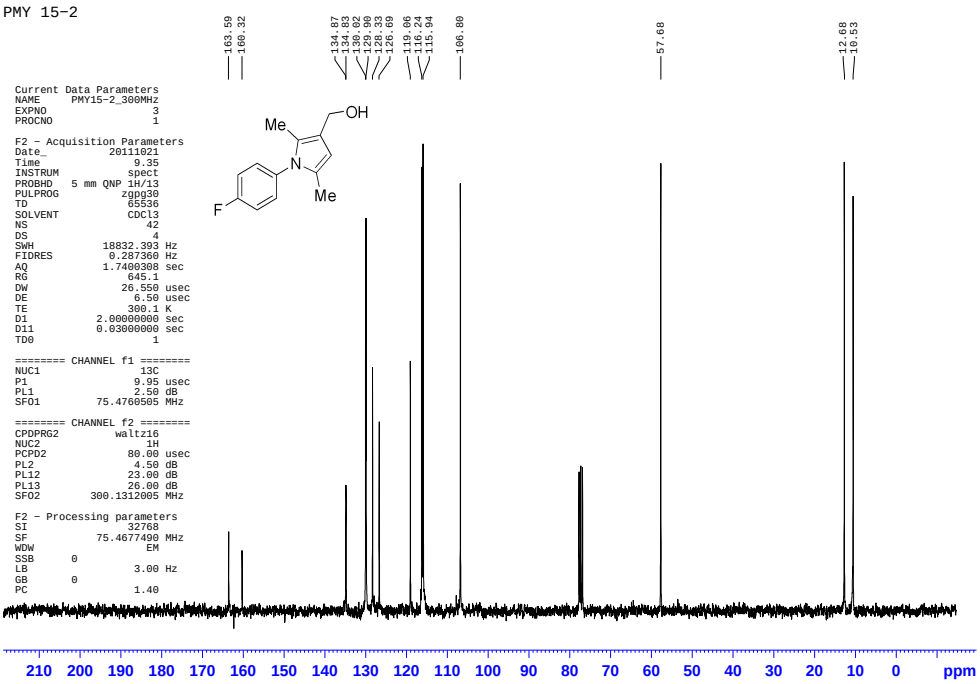
## OSM-S-9, PMY 35



# PMY 18-3, PMY 15-2, page 17



**PMY 31-5**

**PMY 32-3**

**PMY 34-1**

**LMW 6-1**

**LMW 9-1**

**ZYH 1-1**

**ZYH 2-2**

**ZYH 3-1, PMY 47-1**

**ZYH 4-2/4-3**

**ZYH 5-1**

**ZYH 6-1/6-2**

**ZYH 7-2**

**ZYH 8-1**

**ZYH 10-1 A**

**ZYH 10-1 B**

**ZYH 11-1**

**ZYH 12-1/12-2**

**ZYH 13-1**



**ZYH 14-1**

**ZYH 15-1**