Supporting Information

Dithiofunctionalization: A Versatile Approach for Constructing Complex Disulfides from Alkenes and Alkynes

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Contents

General Remarks

All reactions were performed with dry glassware under atmosphere of nitrogen unless otherwise noted. The ^{1}H NMR spectra were obtained with a Varian Mercury 400 NMR spectrometer at 400 MHz, a JEOL JNM-ECZL-400S at 400 MHz or a JEOL JNM-ECA-600 at 600 MHz. The ^{13}C NMR spectra were obtained with a JEOL JNM-ECA-600 at 150 MHz. The ^{19}F NMR spectra were obtained with a Varian Mercury 400 NMR spectrometer at 376 MHz. All NMR measurements were carried out at 25 ºC unless otherwise noted. Chemical shifts are reported in parts per million (ppm) downfield from $(CH_3)_4Si$ (δ 0.00 for ^{1}H NMR in $CDCl_3$) or the solvent peak (δ 77.16 for ^{13}C NMR in $CDCl_3$). ^{19}F NMR spectra are referenced to external standard (CF_3CO_2H, –76.6 ppm in CDCl3). The abbreviations s, d, t, q, m, and br signify singlet, doublet, triplet, quartet, multiplet, and broad, respectively. Melting points were determined with an MPA100 OptiMelt apparatus. High-resolution mass spectra (HRMS) were recorded on a JEOL JMS-DX-303, a JEOL JMS-700, or a JEOL JMS-T100GC spectrometer with magnetic sector time-of-flight mass analyzer. Column chromatography was performed with silica gel (Kanto Chemical Co., Inc., 60 (spherical, neutral), 37563-84) or florisil (Kanto Chemical Co., Inc., 16231-08).

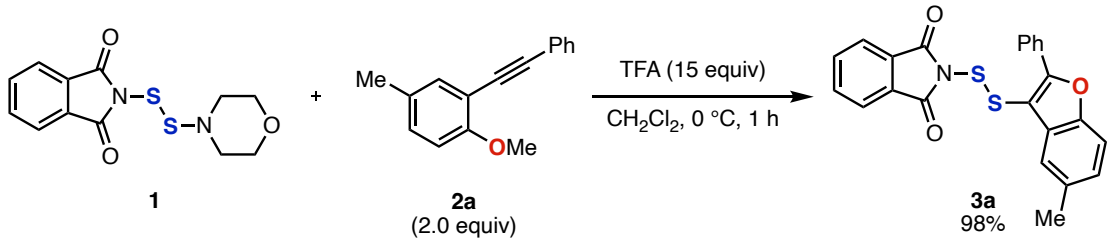
Unless otherwise noted, commercial reagents were purchased from Tokyo Chemical Industry Co., Ltd., Kanto Chemical Co., Inc., Signa-Aldrich Japan, FUJIFILM Wako Pure

Chemical Corporation, and other commercial suppliers and were used as received. Anhydrous CH_2Cl_2, MeCN, $CHCl_3$, DMF and 1,4-dioxane were purchased from FUJIFILM Wako Pure Chemical Corporation and were used as received.

N-(Morpholine-4-dithio)phthalimide **(1)**, [1] 1-methoxy-4-methyl-2-(phenylethynyl)benzene **(2a)**, [2] 4-chloro-1-methoxy-2-(phenylethynyl)benzene **(2b)**,[2] 1-methoxy-2-(phenylethynyl)benzene **(2c)**, [3] 1-methoxy-2-((4-methoxyphenyl)ethynyl)benzene **(2d)**, [4] methyl 4-((2-methoxyphenyl)ethynyl)benzoate **(2e)**,[4,5] 1-methoxy-2-(*o*-tolylethynyl)benzene **(2f)**,[5,6] 2-((2-methoxyphenyl)ethynyl)naphthalene **(2g)**,[6] 2-((2-methoxyphenyl)ethynyl)thiophene **(2h)**,[5] ((2-methoxyphenyl)ethynyl)(p-tolyl)sulfane **(2i)**, [7] methyl(2-(phenylethynyl)phenyl)sulfane **(2j)**,[2] 4-methyl-*N*-(2-(phenylethynyl)phenyl)benzenesulfonamide **(2k)**, [8] 3',5'-dimethoxy-2-(phenylethynyl)-1,1'-biphenyl **(2l)**, [9] 4-phenylpent-4-enoic acid **(4a)**, [10] 4-methylpent-4-enoic acid **(4b)**,[10] 5-phenylhex-5-enoic acid **(4c)**,[10] 4-phenylpent-4-en-1-ol **(4d)**, [11] 2-(3-phenylbut-3-en-1-yl)phenol **(4e)**,[12] (*E*)-2-(3-phenylprop-1-en-1-yl)phenol **(4f)**,[13] 4-methyl-*N*-(4-phenylpent-4-en-1-yl)benzenesulfonamide **(4g)**,[14] 5-(4-methylpent-3-en-1-yl)benzo[*d*][1,3]dioxole **(4h)**,[15,16] 4,4'-(ethene-1,1-diyl)bis(fluorobenzene) **(8b)**,[16] 4,4'-(ethene-1,1-diyl)bis(methoxybenzene) **(8c)**, [17] (*E*)-2-(3,7-dimethylocta-2,6-dien-1-yl)phenol **(10)**, [18] *N*-(1*H*-indol-5-yl)benzamide **(12)**,[19] 1,4-dimethoxy-2-((4-methoxyphenyl)ethynyl)benzene **(2m)**,[20] 4-(*p*-tolyl)pent-4-enoic acid **(4i)**, [21] 4-(2-(methylthio)ethyl)-2-phenyloxazol-5(4*H*)-one **(16)**, [22] 1-methoxy-2-((3-methoxyphenyl)ethynyl)benzene **(2n)**[23] were prepared according to the reported methods. All other chemical reagents used were commercial grade and used as received.

Experimental Procedures

*A general procedure for the dithiocyclization of alkynes (**2**) or olefin (**4**) with N-(morpholine-4-dithio)phthalimide (**1**)*

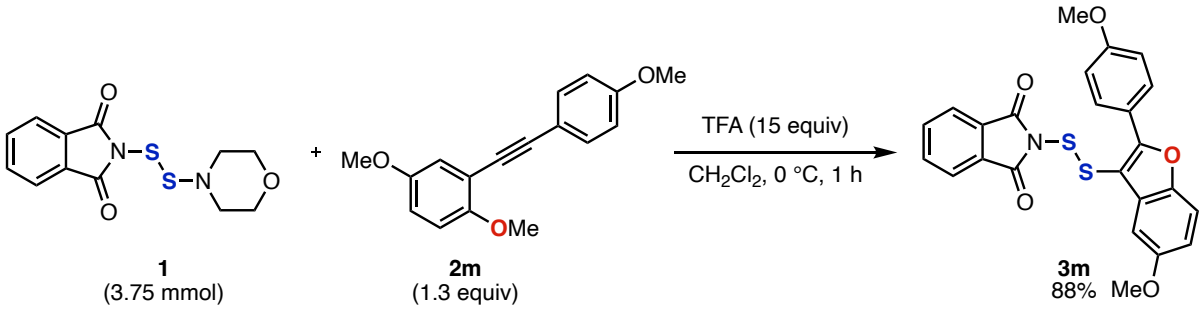


To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv) and 1-methoxy-4-methyl-2-(phenylethynyl)benzene (**2a**) (44.5 mg, 0.20 mmol, 2.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 μL, 1.5 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (Florisil, 3 g, hexane/EtOAc = 8/1 to 2/1) to afford 2-((5-methyl-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3a**) (40.9 mg, 98%) as a yellow solid.

According to the procedure for preparing 2-((5-methyl-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3a**), 2-((5-chloro-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3b**) (36.3 mg, 83%, performed at room temperature for 2 hours), 2-((2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3c**) (33.6 mg, 83%), 2-((2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3d**) (41.1 mg, 95%), methyl 4-(3-((1,3-dioxoisoindolin-2-yl)disulfanyl)benzofuran-2-yl)benzoate (**3e**) (22.2 mg, 48%), 2-((2-(*o*-tolyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3f**) (39.4 mg, 94%), 2-((2-(naphthalen-2-yl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3g**) (46.3 mg, quant), 2-((2-(thiophen-2-yl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3h**) (39.0 mg, 95%), 2-((2-(*p*-tolylthio)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3i**) (32.3 mg, 72%), 2-((2-phenylbenzo[*b*]thiophen-3-yl)disulfanyl)isoindoline-1,3-dione (**3j**) (41.0 mg, 98%), 2-((2-phenyl-1-tosyl-1*H*-indol-3-yl)disulfanyl)isoindoline-1,3-dione (**3k**) (50.2 mg, 90%), 2-((1,3-dimethoxy-10-phenylphenanthren-9-yl)disulfanyl)isoindoline-1,3-dione (**3l**) (52.2 mg, quant, performed for 2 hours), 2-(((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5a**) (36.3 mg, 94%), 2-(((2-methyl-5-oxotetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5b**) (21.7 mg, 67%), 2-(((6-oxo-2-phenyltetrahydro-2*H*-pyran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5c**) (36.0 mg, 90%), 2-(((2-phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5d**) (33.4 mg, 94%), 2-(((2-phenylchroman-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5e**) (18.8 mg, 43%), 2-(((2*S**,3*R**)-2-phenylchroman-3-yl)disulfanyl)isoindoline-1,3-dione (**5f**) (38.2 mg, 91%), 2-(((2-phenyl-1-tosylpyrrolidin-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5g**) (48.1 mg, 92%), 2-((5,5-dimethyl-5,6,7,8-tetrahydronaphtho[2,3-*d*][1,3]dioxol-6-yl)disulfanyl)isoindoline-1,3-dione (**5h**) (41.7 mg, quant, performed at room temperature for 3 hours), 2-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3m**) (41.2 mg, 89%), 2-(((5-oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5i**) (43.5 mg, quant), and 2-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3n**) (622 mg, 96%) were

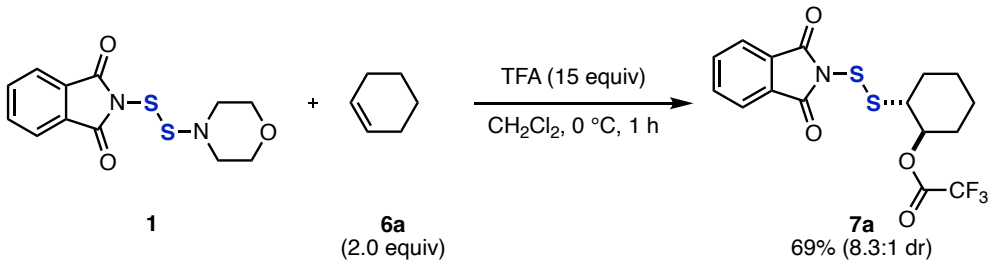
prepared from the *N*-(morpholine-4-dithio)phthalimide (**1**) and corresponding alkyne (**2**) or olefin (**4**).

A procedure for the 3.75 mmol scale reaction of dithiocyclization



To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (1.11 g, 3.75 mmol, 1.0 equiv) and 1,4-dimethoxy-2-((4-methoxyphenyl)ethynyl)benzene (**2m**) (1.31 g, 4.88 mmol, 1.3 equiv) suspended in CH_2Cl_2 (38mL) was slowly added TFA (4.31 mL, 57.3 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (50 mL) and extracted with $CHCl_3$ (50 mL × 3). The combined organic layer was washed with brine (50 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (Florisil, 30 g, hexane/EtOAc = 8/1 to 2/1) to afford 2-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3m**) (1.53 g, 3.3 mmol, 88%) as a yellow solid.

*A general procedure for the three-component reaction using olefin (**6**) and N-(morpholine-4-dithio)phthalimide (**1**)*

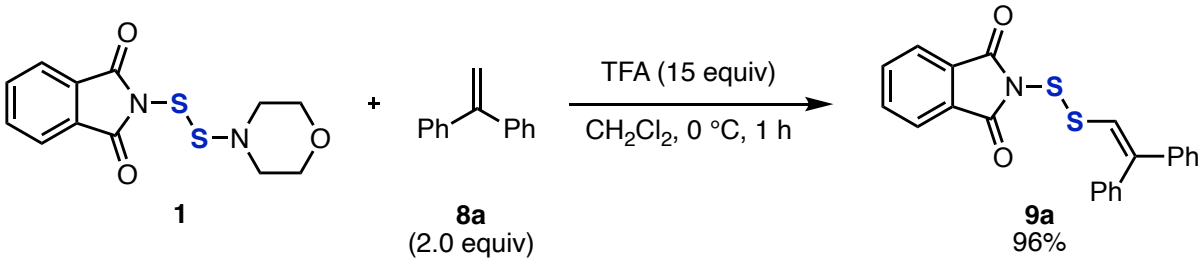


To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv) and cyclohexene (**6a**) (16.4 mg, 0.20 mmol, 2.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 μL, 1.5 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, 3 g; hexane/EtOAc = 15:1 to 5:1), followed by GPC, to afford 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)cyclohexyl 2,2,2-trifluoroacetate (**7a**) (28.0 mg, 69%, 8.3:1 dr) as a colorless oil.

According to the procedure for preparing 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)cyclohexyl 2,2,2-trifluoroacetate (**7a**), 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)-

1,2,3,4-tetrahydronaphthalen-1-yl 2,2,2-trifluoroacetate (**7b**) (26.2 mg, 58%, 2:1 dr), 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)cyclopentyl 2,2,2-trifluoroacetate (**7c**) (25.9 mg, 66%, 1.6:1 dr), 5-((1,3-dioxoisoindolin-2-yl)disulfanyl)octan-4-yl 2,2,2-trifluoroacetate (**7d**) (34.6 mg, 80%, 2.6:1 dr), 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)-1-phenylpropyl 2,2,2-trifluoroacetate (**7e**) (37.1 mg, 83%, 1.1:1 dr), 3-((1,3-dioxoisoindolin-2-yl)disulfanyl)-2,3-dimethylbutan-2-yl 2,2,2-trifluoroacetate (**7f**) (16.0 mg, 39%), 1-((1,3-dioxoisoindolin-2-yl)disulfanyl)-2-(4-methyl-5-oxocyclohex-3-en-1-yl)propan-2-yl 2,2,2-trifluoroacetate (**7g**) (37.8 mg, 80%, 1.2:1 dr).

*A general procedure for the Addition-elimination reaction of olefin (**8**) with N-(morpholine-4-dithio)phthalimide (**1**)*

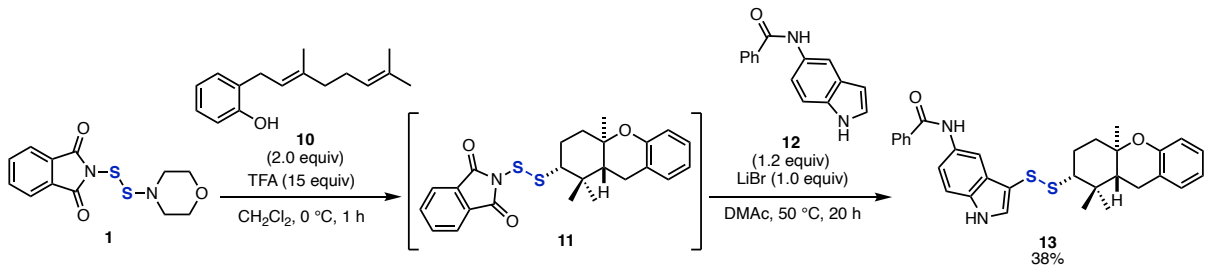


1 **8a** (2.0 equiv) **9a** 96%

To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv) and ethene-1,1-diyldibenzene (**8a**) (36.1 mg, 0.20 mmol, 2.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 μL, 1.5 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, 3 g; hexane/EtOAc = 15:1 to 5:1) to afford 2-((2,2-diphenylvinyl)disulfanyl)isoindoline-1,3-dione (**9a**) (37.2 mg, 96%) as a colorless solid.

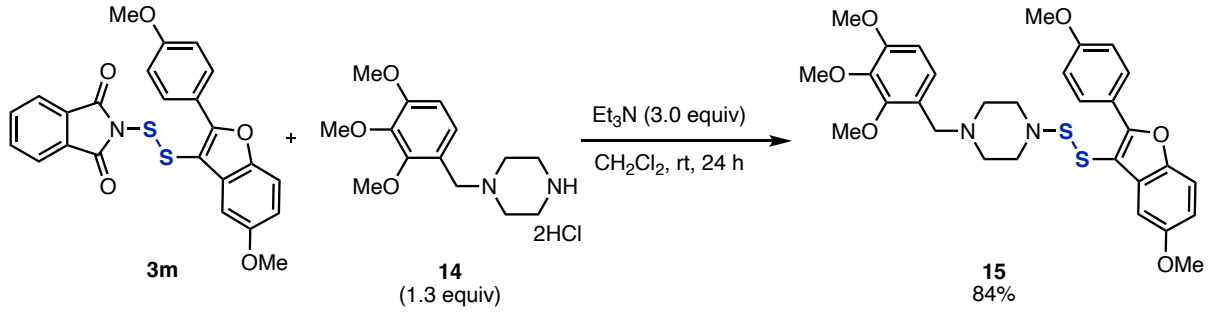
According to the procedure for preparing 2-((2,2-diphenylvinyl)disulfanyl)isoindoline-1,3-dione (**9a**), 2-((2,2-bis(4-fluorophenyl)vinyl)disulfanyl)isoindoline-1,3-dione (**9b**) (38.9 mg, 89%), 2-((2,2-bis(4-methoxyphenyl)vinyl)disulfanyl)isoindoline-1,3-dione (**9c**) (43.8 mg, 97%) were prepared from the *N*-(morpholine-4-dithio)phthalimide (**1**) and corresponding olefins (**8**).

Synthesis of N-(3-(((2R,4aR*,9aR*)-1,1,4a-trimethyl-2,3,4,4a,9,9a-hexahydro-1H-xanthen-2-yl)disulfanyl)-1H-indol-5-yl)benzamide (**13**) from (E)-2-(3,7-dimethylocta-2,6-dien-1-yl)phenol (**10**) with N-(morpholine-4-dithio)phthalimide (**1**) and N-(1H-indol-5-yl)benzamide (**12**)*[24]



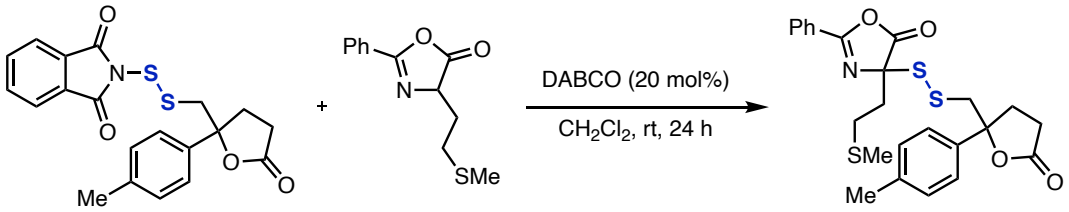
To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv) and (*E*)-2-(3,7-dimethylocta-2,6-dien-1-yl)phenol (**12**) (46.1 mg, 0.20 mmol, 2.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 μL, 1.5 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by short column chromatography and used in the next step without further purification. Under an argon atmosphere, crude **11** and *N*-(1*H*-indol-5-yl)benzamide (**12**) suspended in Dry DMAc (1mL) was added LiBr (8.9 mg, 0.10 mmol, 1.0 equiv) at 50 °C. After stirring at the same temperature for 20 h, the reaction mixture was quenched with an aqueous saturated solution of H_2O (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The two step yield was determined by ^1H NMR based on 1,1,2,2,-tetrachloroethane (38%).

*Synthesis of 1-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)-4-(2,3,4-trimethoxybenzyl)piperazine (**15**) from 2-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3m**) with trimetazidine Dihydrochloride (**14**)[25]*



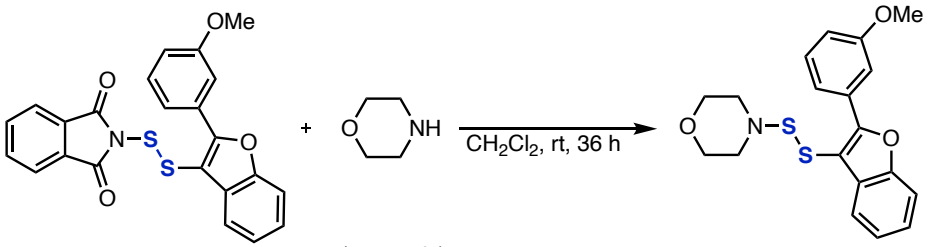
To a mixture of 2-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione **(3m)** (46.4 mg, 0.10 mmol, 1.0 equiv) and trimetazidine dihydrochloride **(20)** (44.3 mg, 0.13 mmol, 1.3 equiv) suspended in CH_2Cl_2 (1mL) was added Et_3N (42.0 μL, 0.30 mmol, 3.0 equiv) at room temperature. After stirring at the same temperature for 24 h, the reaction mixture was added EtOAc (5 mL), washed with 10% aqueous solution of K_2CO_3 (5 mL × 6), brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, 3 g; hexane/EtOAc = 8:1 to 2:1) to afford 1-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)-4-(2,3,4-trimethoxybenzyl)piperazine (**15**) (49.0 mg, 84%) as a yellow oil.

*Synthesis of 4-(2-(methylthio)ethyl)-4-(((5-oxo-2-(p-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)-2-phenyloxazol-5(4H)-one (**17**) from 2-(((5-oxo-2-(p-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**3i**) with 4-(2-(methylthio)ethyl)-2-phenyloxazol-5(4H)-one (**16**)*[22]



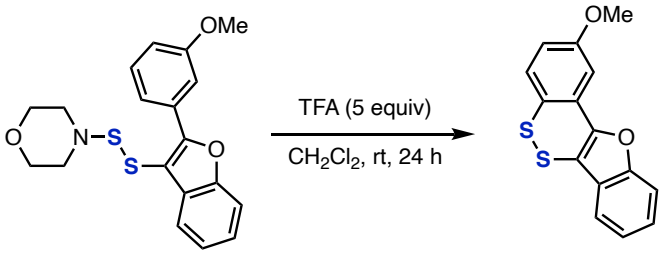
To a mixture of 2-(((5-oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione **(3i)** (39.9 mg, 0.10 mmol, 1.0 equiv) and 4-(2-(methylthio)ethyl)-2-phenyloxazol-5(4*H*)-one (**24**) (47.1 mg, 0.20 mmol, 2.0 equiv) suspended in CH_2Cl_2 (1mL) was added DABCO (2.2 mg, 0.020 mmol, 20 mol%) at room temperature. After stirring at the same temperature for 24 h, the reaction mixture was added EtOAc (5 mL), washed with 10% aqueous solution of K_2CO_3 (5 mL × 6), brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, 3 g; hexane/EtOAc = 5:1 to 1:1) to afford 4-(2-(methylthio)ethyl)-4-(((5-oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)-2-phenyloxazol-5(4*H*)-one (**17**) (41.7 mg, 86%) as a colorless oil.

Synthesis of 4-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfaneyl)morpholine (27) from 2-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfaneyl)isoindoline-1,3-dione (26) with Morpholine[25]



To a mixture of morpholine (131 μL, 1.5 mmol, 3.0 equiv) dissolved in CH_2Cl_2 (5 mL) was added 2-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3j**) (217 mg, 0.50 mmol, 1.0 equiv) at room temperature. After stirring for 24 h at the same temperature, the reaction mixture was washed with 10% aqueous K_2CO_3 (10 mL × 6), followed by brine (20 mL), dried over $MgSO_4$, and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, 15 g; hexane/EtOAc = 20:1 to 8:1) to afford 4-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)morpholine (**18**) (142.5 mg, 76%) as a colorless oil.

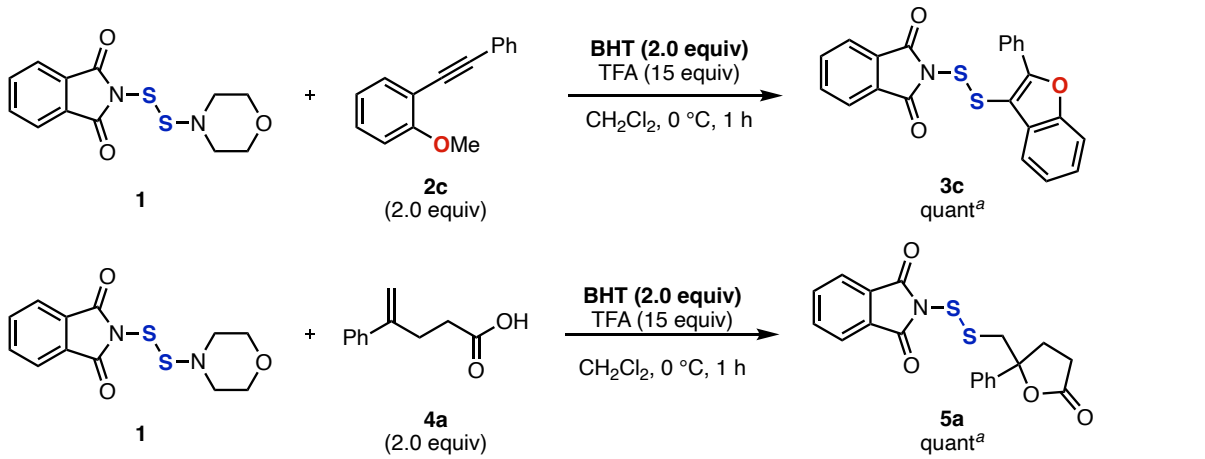
*Synthesis of 2-methoxybenzo[5,6][1,2]dithiino[4,3-b]benzofuran (**19**) via intramolecular cyclization of 4-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)morpholine (**18**)*



To a mixture of 4-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)morpholine (**18**) (38.8 mg, 0.10 mmol, 1.0 equiv) suspended in CH_2Cl_2 (2mL) was slowly added TFA (38.3 µL, 0.51 mmol, 4.9 equiv) at room temperature. After stirring at the same temperature for 24 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (silica gel, 3 g; hexane/EtOAc = 10:1 to 3:1), followed by GPC, to afford 2-methoxybenzo[5,6][1,2]dithiino[4,3-*b*]benzofuran (**19**) (9.2 mg, 31%) as a colorless oil.

Mechanistic study

*Procedures for the dithiocyclization of alkynes (**2a**) or olefin (**4a**) with N-(morpholine-4-dithio)phthalimide (**1**) in the presence of BHT*



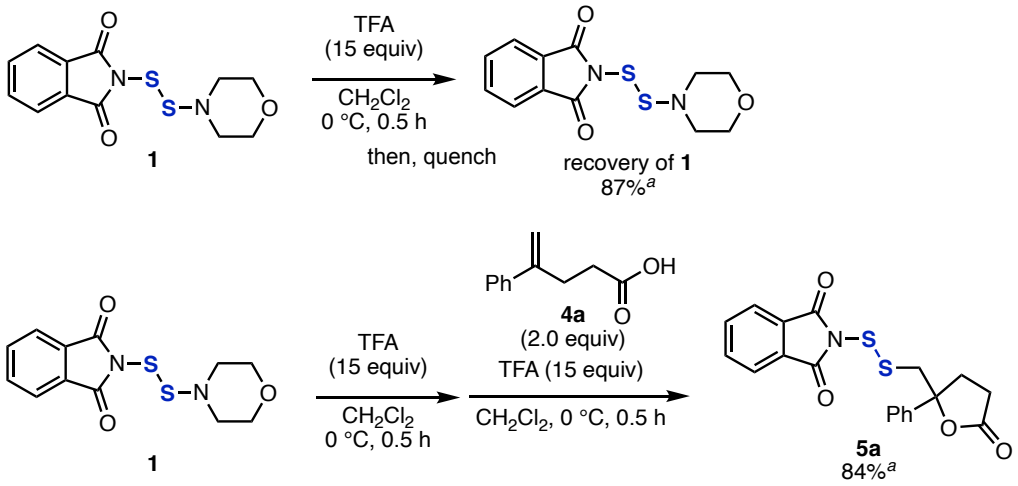
[a]The yield determined by ^{1}H NMR based on 1,1,2,2,-tetrachloroethane

Scheme S1. Dithiocyclization of alkynes (**2a**) or olefin (**4a**) with *N*-(morpholine-4-dithio)phthalimide (**1**) in the presence of BHT.

To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv), BHT (44.0 mg, 0.20 mmol, 2.0 equiv) and 1-methoxy-4-methyl-2-(phenylethynyl)benzene (**2a**) (44.5 mg, 0.20 mmol, 2.0 equiv) or 4-phenylpent-4-enoic acid (**4a**) (35.2 mg, 0.20 mmol, 2.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 µL, 1.5 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 1 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The residue was purified by column chromatography (Florisil, 3 g,

hexane/EtOAc = 8/1 to 2/1) to afford 2-((5-methyl-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3a**) (quant) or 2-(((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)disulfaneyl)isoindoline-1,3-dione (**5a**) (quant). The yields were determined by ^{1}H NMR based on 1,1,2,2,-tetrachloroethane.

*Procedures for the dithiocyclization via the stepwise addition of TFA and 4-phenylpent-4-enoic acid (**4a**)*



Scheme S2. Dithiocyclization via the stepwise addition of TFA and 4-phenylpent-4-enoic acid (**4a**)

To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 μL, 1.5 mmol, 15 equiv) at 0 °C. After stirring at the same temperature for 0.5 h, the reaction mixture was quenched with an aqueous saturated solution of $NaHCO_3$ (5 mL) and extracted with $CHCl_3$ (5 mL × 3). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The yield was determined by ^{1}H NMR based on 1,1,2,2,-tetrachloroethane (87%).

To a mixture of *N*-(morpholine-4-dithio)phthalimide (**1**) (29.6 mg, 0.10 mmol, 1.0 equiv) suspended in CH_2Cl_2 (1mL) was slowly added TFA (115 μL, 1.5 mmol, 15 equiv) at 0 °C. After the mixture was stirred at the same temperature for 0.5 h, 4-phenylpent-4-enoic acid (**4a**) (35.2 mg, 0.20 mmol, 2.0 equiv) was added. The reaction mixture was further stirred at the same temperature for 0.5 h, quenched with saturated aqueous $NaHCO_3$ solution (5 mL), and extracted with $CHCl_3$ (3 × 5 mL). The combined organic layer was washed with brine (5 mL), dried ($MgSO_4$), and concentrated under reduced pressure. The yield was determined by ^{1}H NMR based on 1,1,2,2,-tetrachloroethane (84%).

X-Ray Crystallographic Analysis

X-ray crystal structure analysis of 3j

Single crystals suitable for X-ray crystallography were obtained by recrystallization from CH_2Cl_2/ pentane. A suitable crystal was mounted on a Rigaku XtaLAB Synergy-R single crystal X-ray diffractometer equipped with a Cu Kα rotating anode X-ray source. The crystal was kept at 99.95(10) K during data collection. Using Olex2,[26] the structure was solved with the olex2. solve[27] structure solution program using Charge Flipping and refined with the SHELXL[28] refinement package using Least Squares minimization.

Crystallographic data of **3j** has been deposited on Cambridge Crystallographic Data Center, deposition no. CCDC 2481179.

Table S1. Crystal data and structure refinements for 2-((2-phenylbenzo[*b*]thiophen-3-yl)disulfanyl)isoindoline-1,3-dione (**3j**):

CCDC number	2481179
Empirical formula	$C_{22}H_{13}NO_2S_3$
Formula weight	419.51
Space system	monoclinic
Space group	$P2_1$
a/Å	10.96180(10)
b/Å	7.62930(10)
c/Å	11.57010(10)
α/°	90
β/°	108.5130(10)
γ/°	90
Volume/Å^3	917.544(17)
Z	2
Temperature/K	99.95(10)
2Θ range for data collection/°	8.058 to 152.008
ρ_{calcd} g/cm^3	1.518
μ/mm^{-1}	3.853
F_000	432.0
Crystal_size/mm^3	0.2 x 0.1 x 0.1
Radiation	Cu K\a (λ = 1.54184)
Reflections collected	22715
Independent	3612
Index ranges	–13≦h≦11, –9≦k≦9, –14≦l≦14
Data/restraints/parameters	3612/1/254
Final R indexes R [I>2σ(I)]gt	R_1 = 0.0250, wR_2 = 0.0667
Final R indexes R [all data]	R_1 = 0.0252, wR_2 = 0.0669
Goodness-of-fit on F^2	1.064
Largest peak/deepest hole eÅ^{-3}	0.23/–0.22

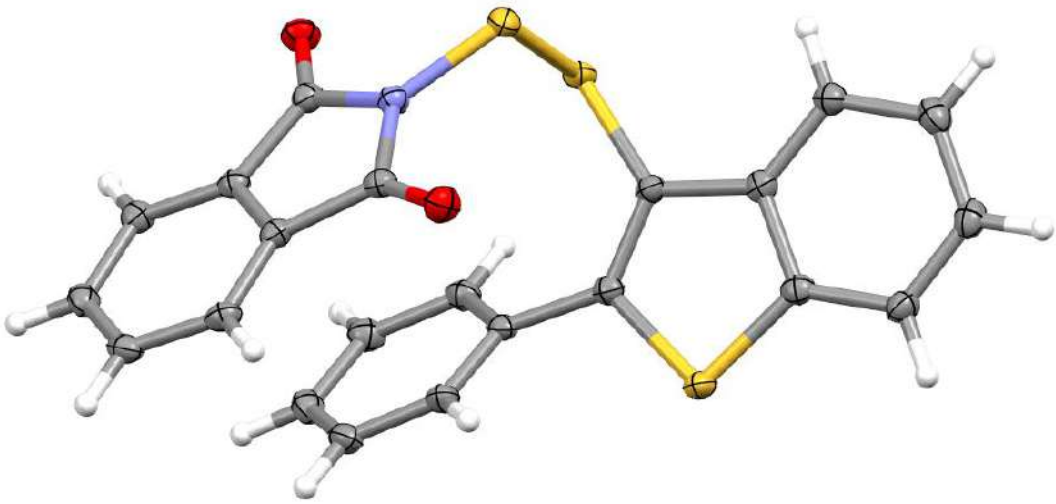


Figure S1. Thermal ellipsoid (50 % probability) plot of **3j**, CCDC No. 2481179. Color code of atoms: hydrogen, white; carbon, gray; nitrogen, purple; oxygen, red; sulfur, yellow.

X-ray crystal structure analysis of 5a

Single crystals suitable for X-ray crystallography were obtained by recrystallization from $CHCl_3$/ pentane. A suitable crystal was selected and mounted on a Rigaku XtaLAB Synergy-R diffractometer equipped with a Mo Kα rotating anode X-ray source. The crystal was kept at 99.9(2) K during data collection. Using Olex2,[26] the structure was solved with the SHELXT[27] structure solution program using Intrinsic Phasing and refined with the SHELXL[28] refinement package using Least Squares minimization.

Crystallographic data of **5a** has been deposited on Cambridge Crystallographic Data Center, deposition no. CCDC 2481173.

Table S2. Crystal data and structure refinements for 2-(((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5a**):

CCDC number	2481173
Empirical formula	$C_{76}H_{60}N_4O_{16}S8$
Formula weight	1541.76
Space system	triclinic
Space group	P-1
a/Å	9.38418(6)
b/Å	13.64407(9)
c/Å	28.2584(3)
α/°	77.1795(7)
β/°	88.5778(7)
γ/°	87.1848(6)
Volume/Å^3	3523.34(5)
Z	2
Temperature/K	99.9(2)

2Θ range for data collection/°	4.57 to 62.76
ρ_{calcd} g/cm^3	1.453
μ/mm^{-1}	0.327
F_(000)	1600.0
Crystal size/mm^3	0.2 x 0.2 x 0.1
Radiation	Mo Kα (λ = 0.71073)
Reflections collected	242652
Independent	20740
Index ranges	$-13 \leq h \leq 13, -19 \leq k \leq 18, -39 \leq l \leq 39$
Data/restraints/parameters	20740/0/937
Final R indexes R [I>2σ(I)]gt	$R_1 = 0.0820$, $wR_2 = 0.2074$
Final R indexes R [all data]	$R_1 = 0.0915$, $wR_2 = 0.2118$
Goodness-of-fit on F^2	1.150
Largest peak/deepest hole eÅ^{-3}	3.02/-0.55

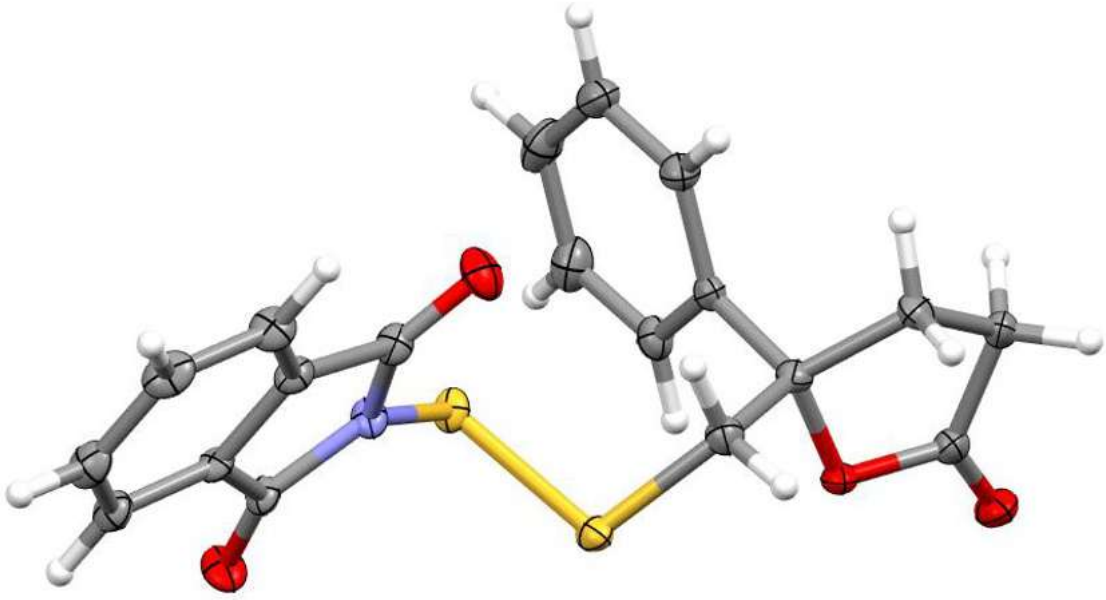


Figure S1. Thermal ellipsoid (50 % probability) plot of **5a**, CCDC No. 2481173. Color code of atoms: hydrogen, white; carbon, gray; nitrogen, purple; oxygen, red; sulfur, yellow.

X-ray crystal structure analysis of 5f

Single crystals suitable for X-ray crystallography were obtained by recrystallization from $CHCl_3$/ pentane. A suitable crystal was selected and mounted on a Rigaku XtaLAB Synergy-R diffractometer equipped with a Mo Kα rotating anode X-ray source. The crystal was kept at 99.95(10) K during data collection. Using Olex2,[26] the structure was solved with the olex2. solve[27] structure solution program using Charge Flipping and refined with the SHELXL[28] refinement package using Least Squares minimization.

Crystallographic data of **5f** has been deposited on Cambridge Crystallographic Data Center, deposition no. CCDC 2481174.

Table S3. Crystal data and structure refinements for 2-(((2*S**,3*R**)-2-phenylchroman-3-yl)disulfanyl)isoindoline-1,3-dione (**5f**):

CCDC number	2481174
Empirical formula	$C_{23}H_{17}NO_3S_2$
Formula weight	419.49
Space system	triclinic
Space group	P-1
a/Å	8.95830(10)
b/Å	9.87370(10)
c/Å	12.4450(2)
α/°	94.2530(10)
β/°	106.4280(10)
γ/°	112.5770(10)
Volume/Å^3	953.95(2)
Z	2
Temperature/K	99.95(10)
2Θ range for data collection/°	4.566 to 62.908
ρ_{calcd} g/cm^3	1.460
μ/mm^{-1}	0.305
F (000)	436.0
Crystal_size/mm^3	0.2 x 0.2 x 0.1
Radiation	Mo Kα (λ = 0.71073)
Reflections collected	55360
Independent	5628
Index ranges	$-12 \le h \le 12$, $-14 \le k \le 14$, $-18 \le l \le 17$
Data/restraints/parameters	5628/0/262
Final R indexes R [I>2σ(I)]gt	$R_1 = 0.0381$, $wR_2 = 0.1006$
Final R indexes R [all data]	$R_1 = 0.0484$, $wR_2 = 0.1060$
Goodness-of-fit on F^2	1.062
Largest peak/deepest hole eÅ^{-3}	0.47/–0.24

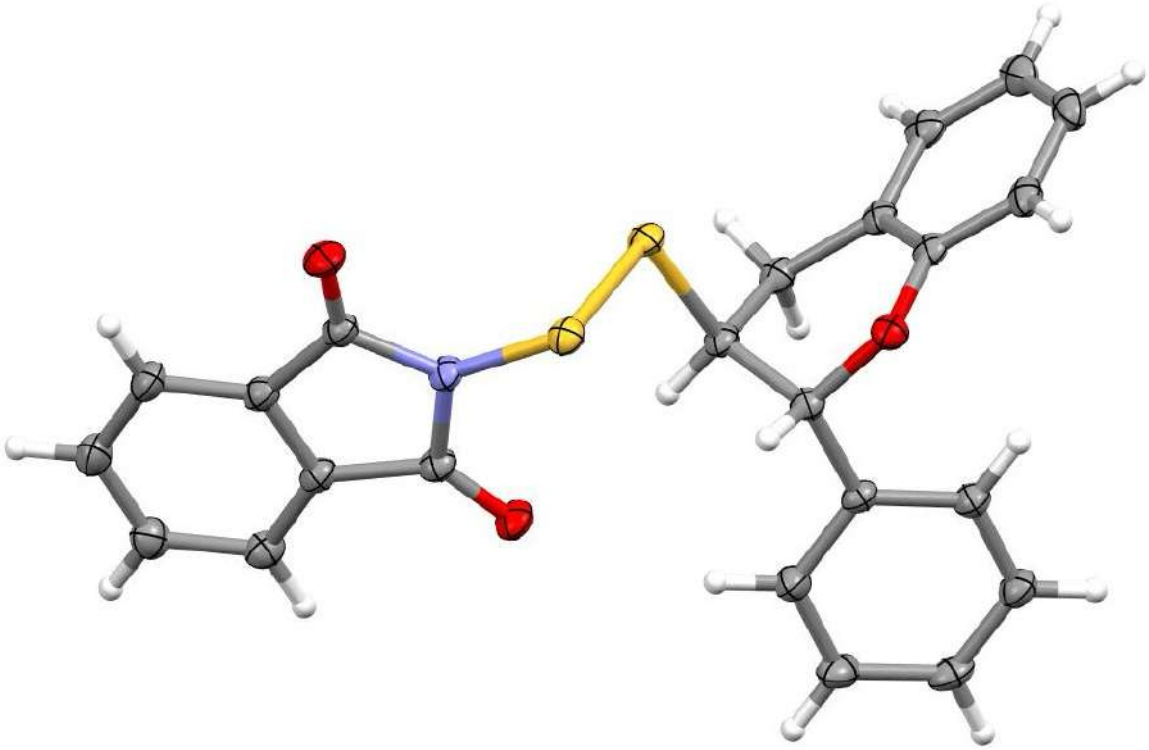
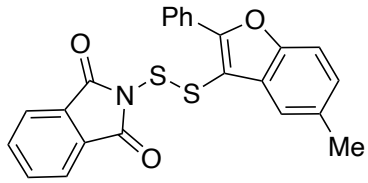
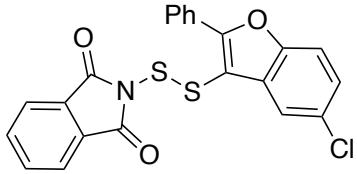


Figure S3. Thermal ellipsoid (50 % probability) plot of **5f**, CCDC No. 2481174. Color code of atoms: hydrogen, white; carbon, gray; nitrogen, purple; oxygen, red; sulfur, yellow.

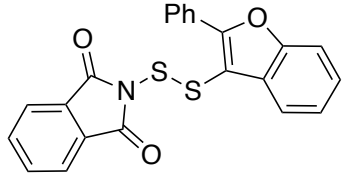
Characterization Data of New Compounds



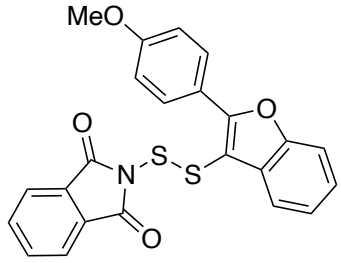
2-((5-Methyl-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3a): Yellow solid; R_f 0.35 (hexane/EtOAc = 3/1); m.p. 168-172 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, J = 7.3 Hz, 2H), 7.71-7.67 (m, 4H), 7.43-7.40 (m, 2H), 7.17 (d, J = 8.8 Hz, 1H), 7.06 (app. t, J = 7.9 Hz, 2H), 6.92 (app. t, J = 7.3 Hz, 1H), 2.40 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 166.8, 158.1, 152.4, 134.5, 133.6, 132.0, 130.0, 129.4, 128.9, 128.4, 127.5, 127.1, 124.0, 119.8, 111.2, 108.2, 21.5; **HRMS** (EI^+) Calcd for $C_{23}H_{15}NO_3S_2^+$ $[M]^+$ 417.0488, found 417.0476.



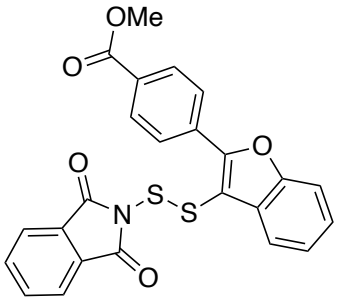
2-((5-Chloro-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3b): Yellow Soild; R_f 0.30 (hexane/EtOAc = 3/1); m.p. 154-156 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98 (d, J = 8.4 Hz, 2H), 7.74-7.67 (m, 4H), 7.59 (s, 1H), 7.47 (d, J = 8.7 Hz, 1H) 7.33 (d, J = 8.7 Hz, 1H), 7.09 (app. t, J = 7.5 Hz, 2H), 6.96 (app. t, J = 7.4 Hz, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 166.7, 159.4, 152.3, 134.6, 131.9, 131.4, 129.8, 129.4, 128.8, 128.5, 127.6, 126.0, 124.1, 119.8, 112.7, 108.3; **HRMS** (EI^+) Calcd for $C_{22}H_{12}{}^{35}ClNO_3S_2^+$ $[M]^+$ 436.9942, found 436.9950.



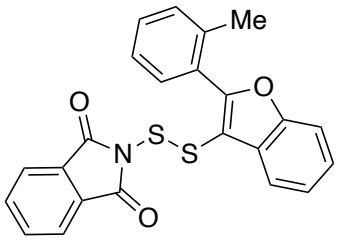
2-((2-Phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3c): Yellow solid; R_f 0.29 (hexane/EtOAc = 3/1); m.p. 168-172 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.99 (d, J = 7.4, 2H), 7.74-7.67 (m, 5H), 7.55 (d, J = 8.2 Hz, 1H), 7.38 (app. t, J = 7.4 Hz, 1H), 7.32 (app. t, J = 7.6 Hz, 1H), 7.05 (app. t, J = 7.6 Hz, 2H), 6.90 (app. t, J = 7.4 Hz, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 166.8, 158.1, 154.0, 134.5, 132.0, 129.9, 129.3, 129.0, 128.4, 127.5, 125.8, 124.04, 123.96, 120.1, 111.7, 108.6; **HRMS** (EI^+) Calcd for $C_{22}H_{13}NO_3S_2^+$ $[M]^+$ 403.0331, found 403.0325.



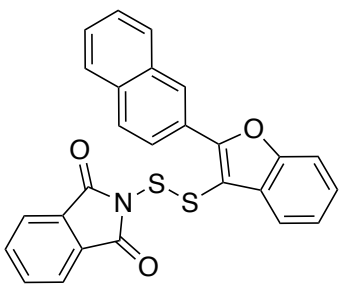
2-((2-(4-Methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3d): Yellow solid; R_f 0.25 (hexane/EtOAc = 3/1); m.p. 181-185 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.8 Hz, 2H), 7.71-7.68 (m, 4H), 7.65 (d, *J* = 6.9 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.35 (app. dt, *J* = 7.4, 1.4 Hz, 1H), 7.33-7.28 (m, 1H), 6.55 (d, *J* = 8.9 Hz, 2H), 3.61 (s, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 166.8, 160.2, 158.4, 153.8, 134.4, 132.1, 130.1, 129.1, 125.3, 123.94, 123.87, 122.0, 119.8, 113.8, 111.5, 106.9, 55.1; **HRMS** (EI^+) Calcd for $C_{23}H_{15}NO_4S_2^+$ $[M]^+$ 433.0437, found 433.0456.



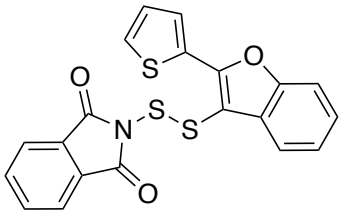
Methyl 4-(3-((1,3-dioxoisoindolin-2-yl)disulfanyl)benzofuran-2-yl)benzoate (3e): Colorless solid; R_f 0.23 (hexane/EtOAc = 3/1); m.p. 199-201 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.07 (d, *J* = 8.5 Hz, 2H), 7.73 (d, *J* = 7.6 Hz, 1H), 7.68 (d, *J* = 8.5 Hz, 2H), 7.66-7.61 (m, 4H), 7.57 (d, *J* = 8.1 Hz, 1H), 7.43 (app. t, *J* = 7.3 Hz, 1H), 7.36 (app. t, *J* = 7.7 Hz, 1H), 3.88 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 166.7, 166.0, 156.6, 154.2, 134.6, 133.5, 131.7, 129.9, 129.7, 127.2, 126.5, 124.3, 124.1, 120.5, 111.8, 110.8, 52.2; **HRMS** (EI^+) Calcd for $C_{24}H_{15}NO_5S_2^+$ $[M]^+$ 461.0386, found 461.0391.



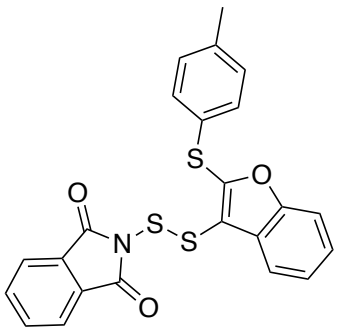
2-((2-(*o*-Tolyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3f): Colorless solid; R_f 0.43 (hexane/EtOAc = 3/1); m.p. 161-163 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.76-7.71 (m, 4H), 7.66 (d, *J* = 7.5 Hz, 1H), 7.53 (d, *J* = 8.1 Hz, 1H), 7.48-7.45 (m, 1H), 7.38 (app. t, *J* = 7.3 Hz, 1H), 7.31 (app. t, *J* = 7.6 Hz, 1H), 6.94-6.86 (m, 3H), 2.37 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 166.7, 160.4, 154.2, 138.1, 134.6, 132.1, 131.2, 130.9, 129.3, 129.0, 128.1, 125.5, 125.2, 123.9, 123.8, 120.1, 111.7, 110.4, 20.8; **HRMS** (EI^+) Calcd for $C_{23}H_{15}NO_3S_2^+$ $[M]^+$ 417.0488, found 417.0507.



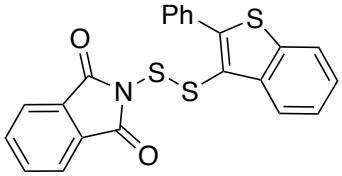
2-((2-(Naphthalen-2-yl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3g): Yellow solid; R_f 0.38 (hexane/EtOAc = 3/1); m.p. 159-162 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.49 (s, 1H), 8.13 (d, J = 8.8 Hz, 1H), 7.82-7.70 (m, 2H), 7.59 (d, J = 7.6 Hz, 1H), 7.50-7.32 (m, 8H), 7.30-7.19 (m, 2H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 166.8, 158.1, 154.1, 134.0, 133.0, 132.9, 131.1, 129.9, 128.7, 128.3, 127.7, 127.5, 126.9, 126.8, 126.6, 125.9, 124.2, 124.1, 123.2, 120.2, 111.6, 109.0; **HRMS** (EI^+) Calcd for $C_{26}H_{15}NO_3S_2^+$ $[M]^+$ 453.0488, found 453.0511.



2-((2-(Thiophen-2-yl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3h): Yellow solid; R_f 0.27 (hexane/EtOAc = 3/1); m.p. 197-199 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.79-7.75 (m, 2H), 7.75-7.69 (m, 3H), 7.59 (d, J = 7.7 Hz, 1H), 7.52 (d, J = 8.2 Hz, 1H), 7.36 (app. dt, J = 7.2, 1.2 Hz, 1H), 7.29 (app. t, J = 7.8 Hz, 1H), 6.99 (dd, J = 5.0, 1.0 Hz, 1H), 6.75 (app. t, J = 5.0 Hz ,1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.0, 154.2, 153.9, 134.7, 132.2, 130.9, 129.6, 128.1, 127.9, 127.4, 125.8, 124.11, 124.10, 119.8, 111.6, 107.2; **HRMS** (EI^+) Calcd for $C_{20}H_{11}NO_3S_3^+$ $[M]^+$ 408.9896, found 408.9905.

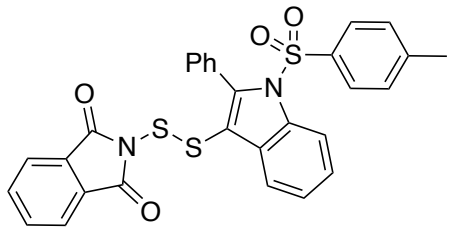


2-((2-(*p*-Tolylthio)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3i): Yellow solid; R_f 0.38 (hexane/EtOAc = 3/1); m.p. decomp.; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90-7.88 (m, 2H), 7.79-7.77 (m, 2H), 7.57 (d, J = 7.2 Hz, 1H), 7.43 (d, J = 8.2 Hz, 1H), 7.33 (app. t, J = 7.3 Hz, 1H), 7.28-7.24 (m, 1H), 7.21 (d, J = 8.2 Hz, 2H), 7.03 (d, J = 8.4 Hz, 2H), 2.28 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 162.5, 151.4, 150.1, 133.8, 130.4, 127.8, 126.9, 125.7, 124.1, 123.5, 121.4, 119.8, 119.4, 115.4, 112.8, 107.3, 16.7; **HRMS** (EI^+) Calcd for $C_{23}H_{15}NO_3S_3^+$ $[M]^+$ 449.0209, found 449.0208.

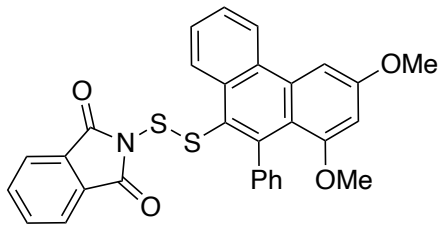


2-((2-Phenylbenzo[*b*]thiophen-3-yl)disulfanyl)isoindoline-1,3-dione (3j): Colorless solid; R_f 0.40 (hexane/EtOAc = 3/1); m.p. 200-203 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.98-7.95 (m, 1H), 7.88-7.86 (m, 1H), 7.75-7.71 (m, 4H), 7.51 (d, J = 7.2 Hz, 2H), 7.46-7.40 (m, 2H), 7.01 (app. t, J = 7.6 Hz, 2H), 6.86 (app. t, J = 7.4 Hz, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ

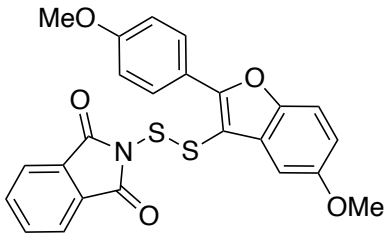
166.6, 150.9, 140.6, 138.5, 134.5, 132.8, 132.2, 130.2, 128.3, 128.2, 125.6, 125.4, 124.0, 123.5, 122.5, 122.1; **HRMS** (EI$^+$) Calcd for $C_{22}H_{13}NO_2S_3^+$ [M]$^+$ 419.0103, found 419.0119.



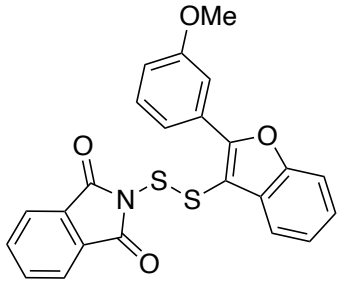
2-((2-Phenyl-1-tosyl-1*H*-indol-3-yl)disulfanyl)isoindoline-1,3-dione (3k): Pale yellow solid; R_f 0.33 (hexane/EtOAc = 3/1); m.p. 204-207 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.36 (d, J = 8.4 Hz, 1H), 7.83-7.75 (m, 4H), 7.65 (d, J = 7.8 Hz, 1H), 7.46 (app. dt, J = 7.3, 1.2 Hz, 1H), 7.35 (app. t, J = 7.8 Hz, 1H), 7.24 (d, J = 8.4 Hz, 2H), 7.14 (d, J = 7.0, 2H), 7.07 (d, J = 8.5 Hz, 2H), 7.05-7.00 (m, 1H), 6.93 (app. t, J = 7.8 Hz, 2H), 2.31 (s, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 166.6, 145.2, 144.9, 137.3, 135.2, 134.7, 132.4, 131.8, 130.2, 129.6, 129.3, 128.8, 126.89, 126.88, 126.2, 124.9, 124.0, 119.9, 116.5, 116.4, 21.7; **HRMS** (EI$^+$) Calcd for $C_{29}H_{20}N_2O_4S_3^+$ [M]$^+$ 556.0580, found 556.0593.



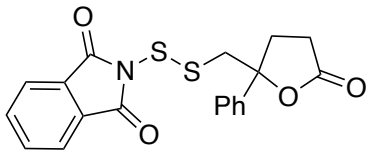
2-((1,3-Dimethoxy-10-phenylphenanthren-9-yl)disulfanyl)isoindoline-1,3-dione (3l): Colorless solid; R_f 0.10 (hexane/EtOAc = 3/1); m.p. 184-186 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.90-7.85 (m, 2H), 7.80-7.76 (m, 2H), 7.44 (d, J = 7.0 Hz, 1H), 7.24-7.23 (m, 5H), 7.19-7.13 (m, 2H), 6.93 (app. t, J = 7.4 Hz, 1H), 6.52 (d, J = 2.5 Hz, 1H), 6.48 (d, J = 2.5 Hz, 1H), 3.81 (s, 3H), 3.66 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 167.1, 162.1, 161.2, 147.7, 143.3, 134.5, 132.8, 131.6, 131.5, 129.6, 128.3, 128.2, 127.5, 127.4, 123.8, 123.32, 123.29, 115.5, 107.2, 98.9, 93.4, 88.8, 56.1, 55.6; **HRMS** (EI$^+$) Calcd for $C_{30}H_{22}NO_4S_2^+$ [M+H]$^+$ 524.0985, found 524.0986.



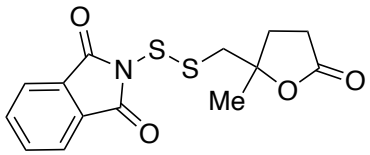
2-((5-Methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3m): Yellow solid; R_f 0.23 (hexane/EtOAc = 3/1); m.p. 154-156 °C; **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.94 (d, J = 8.9 Hz, 2H), 7.72-7.68 (m, 4H), 7.40 (d, J = 8.8 Hz, 1H), 7.05 (d, J = 2.6 Hz, 1H), 6.93 (dd, J = 8.8, 2.6 Hz, 1H), 6.55 (d, J = 8.9 Hz, 2H), 3.81 (s, 3H), 3.61 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 166.8, 160.1, 159.2, 157.0, 148.6, 134.4, 132.1, 130.7, 129.0, 123.9, 122.1, 114.4, 113.8, 112.1, 106.8, 101.8, 56.0, 55.1; **HRMS** (FAB$^+$) Calcd for $C_{24}H_{17}NO_5S_2^+$ [M]$^+$ 463.0543, found 463.0564.



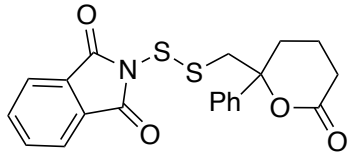
2-((2-(3-Methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (3n): Colorless solid; R_f 0.43 (hexane/EtOAc = 2/1); m.p. 161-163 °C;**^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.69-7.67 (m, 5H), 7.57-7.53 (m, 3H), 7.38 (app. t, J = 7.4 Hz, 1H), 7.33 (app. t, J = 7.5 Hz, 1H), 6.92 (app. t, J = 8.0 Hz, 1H), 6.37 (dd, J = 8.3, 2.4 Hz, 1H), 3.72 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 166.8, 159.2, 157.9, 153.9, 134.5, 131.9, 130.4, 129.8, 129.5, 125.8, 124.0, 123.9, 120.1, 120.0, 115.5, 111.9, 111.7, 108.7, 55.2; **HRMS** (FAB^+) Calcd for $C_{23}H_{15}NO_4S_2^+$ $[M]^+$ 433.0437, found 433.0437.



2-(((5-Oxo-2-phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5a): Colorless solid; R_f 0.10 (hexane/EtOAc = 3/1); m.p. decomp; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.97-7.91 (m, 2H), 7.85-7.78 (m, 2H), 7.43-7.33 (m, 4H), 7.30-7.26 (m, 1H), 3.84 (s, 2H), 2.75-2.67 (m, 2H), 2.59-2.46 (m, 2H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 175.5, 167.6, 141.3, 135.0, 132.4, 128.8, 128.4, 125.1, 124.3, 87.6, 54.2, 33.5, 28.7; **HRMS** (EI^+) Calcd for $C_{19}H_{15}NO_4S_2^+$ $[M]^+$ 385.0437, found 385.0440.

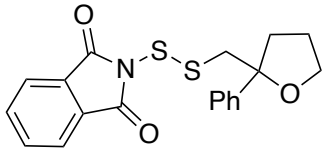


2-(((2-Methyl-5-oxotetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5b): Colorless oil; R_f 0.32 (hexane/EtOAc = 1/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96-7.94 (m, 2H), 7.82-7.80 (m, 2H), 3.59-3.50 (m, 2H), 2.78-2.59 (m, 2H), 2.41-2.33 (m, 1H), 2.12-2.04 (m, 1H), 1.51 (s, 3H),; **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 175.9, 167.6, 135.1, 132.3, 124.3, 85.2, 52.3, 32.3, 29.1, 26.0; **HRMS** (EI^+) Calcd for $C_{14}H_{14}NO_4S_2^+$ $[M+H]^+$ 324.0359, found 324.0357.

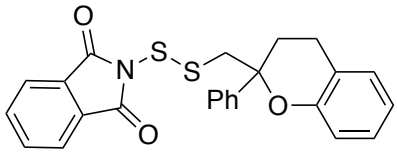


2-(((6-Oxo-2-phenyltetrahydro-2*H*-pyran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5c): Colorless oil; R_f 0.20 (hexane/EtOAc = 2/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.97-7.88 (m, 2H), 7.83-7.77 (m, 2H), 7.42-7.32 (m, 4H), 7.30-7.23 (m, 1H), 3.82 (d, J = 14.4 Hz, 1H),

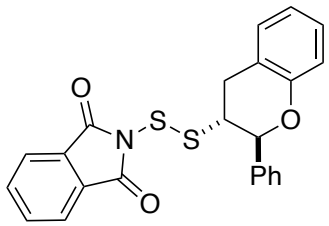
3.76 (d, J = 14.4 Hz, 1H), 2.52-2.35 (m, 3H), 2.25 (dt, J = 12.4, 4.2 Hz, 1H), 1.90-1.78 (m, 1H), 1.66-1.52 (m, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 170.6, 167.7, 141.2, 134.9, 132.4, 129.0, 128.2, 125.5, 124.2, 86.4, 55.6, 31.2, 29.2, 16.3; **HRMS** (FAB^+) Calcd for $C_{20}H_{18}NO_4S_2^+$ $[M+H]^+$ 400.0672, found 400.0684.



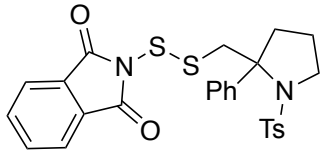
2-(((2-Phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5d): Colorless oil; R_f 0.25 (hexane/EtOAc = 5/1); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.95-7.88 (m, 2H), 7.83-7.76 (m, 2H), 7.39-7.35 (m, 2H), 7.30-7.26 (m, 2H), 7.15 (app. t, J = 7.5 Hz, 1H), 3.92 (dd, J = 14.2, 7.5 Hz, 1H), 3.81 (dd, J = 13.9, 7.8 Hz, 1H), 3.72 (s, 2H), 2.31-2.19 (m, 2H), 2.03-1.94 (m, 1H), 1.83-1.75 (m, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.8, 144.8, 134.8, 132.5, 128.3, 127.1, 125.5, 124.1, 86.0, 68.3, 55.0, 37.4, 25.9; **HRMS** (FAB^+) Calcd for $C_{19}H_{18}NO_3S_2^+$ $[M+H]^+$ 372.0723, found 372.0723.



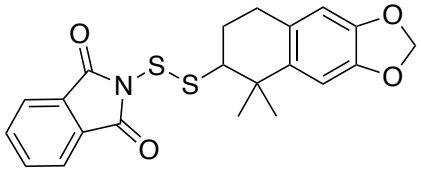
2-(((2-Phenylchroman-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5e): Colorless oil; R_f 0.23 (hexane/EtOAc = 5/1); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.90-7.88 (m, 2H), 7.78-7.76 (m, 2H), 7.35 (d, J = 7.3 Hz, 2H), 7.22 (app. t, J = 7.8 Hz, 2H), 7.05 (app. t, J = 7.7 Hz, 1H), 7.06 (app. t, J = 7.7 Hz, 1H), 6.88 (d, J = 3.6 Hz, 1H), 6.86 (d, J = 4.1 Hz, 1H), 6.78 (dt, J = 7.3, 1.1 Hz, 1H), 3.83 (d, J = 13.6 Hz, 1H), 3.80 (d, J = 13.6 Hz, 1H), 2.64-2.60 (m, 1H), 2.49-2.38 (m, 2H), 2.31-2.26 (m, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.7, 153.5, 141.7, 134.8, 132.4, 129.4, 128.6, 127.5, 127.5, 125.9, 124.1, 121.6, 120.5, 117.0, 80.1, 55.7, 29.9, 22.2; **HRMS** (EI^+) Calcd for $C_{24}H_{19}NO_3S_2^+$ $[M]^+$ 433.0801, found 433.0807.



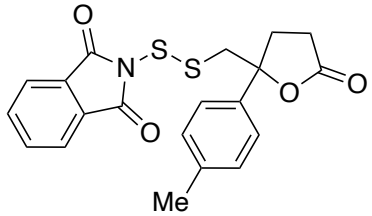
2-(((2*S,3*R**)-2-phenylchroman-3-yl)disulfanyl)isoindoline-1,3-dione (5f):** Colorless solid; R_f 0.33 (hexane/EtOAc = 3/1); m.p. 201-202 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94-7.90 (m, 2H), 7.82-7.78 (m, 2H), 7.41 (d, J = 7.4 Hz, 2H), 7.31 (app. t, J = 7.6 Hz, 2H), 7.27-7.23 (m, 1H), 7.17 (app. t, J = 7.2 Hz, 1H), 7.05 (d, J = 7.1 Hz, 1H), 6.93 (d, J = 8.2 Hz, 1H), 6.90 (app. t, J = 7.5 Hz, 1H), 5.43 (d, J = 5.8 Hz, 1H), 3.98 (q, J = 6.8 Hz, 1H), 3.23 (dd, J = 16.7, 4.6 Hz, 1H), 3.00 (dd, J = 16.7, 7.0 Hz, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.7, 153.9, 139.3, 135.0, 132.3, 129.7, 128.7, 128.3, 128.2, 126.8, 124.2, 121.0, 119.5, 116.7, 79.0, 50.7, 29.2; **HRMS** (EI^+) Calcd for $C_{23}H_{17}NO_3S_2^+$ $[M]^+$ 419.0644, found 419.0647.



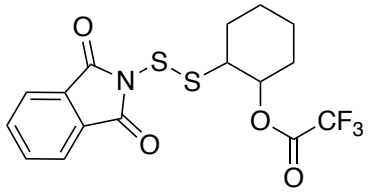
2-(((2-Phenyl-1-tosylpyrrolidin-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5g): Colorless oil; R_f 0.20 (hexane/EtOAc = 3/1); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.98-7.94 (m, 2H), 7.83-7.79 (m, 2H), 7.38 (d, *J* = 8.2 Hz, 2H), 7.35 (d, *J* = 7.1 Hz, 2H), 7.28-7.21 (m, 3H), 7.14 (d, *J* = 8.1 Hz, 2H), 4.48 (d, *J* = 13.8 Hz, 1H), 4.38 (d, *J* = 13.2 Hz, 1H), 3.71-3.66 (m, 1H), 3.56 (dd, *J* = 16.4, 7.4 Hz, 1H), 2.62-2.57(m, 1H), 2.39 (s, 3H), 2.19-2.14 (m, 1H), 2.01-1.96 (m, 1H), 1.92-1.86 (m, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.6, 143.2, 142.8, 137.3, 134.9, 132.1, 129.1, 128.2, 127.2, 127.0, 126.4, 124.1, 73.0, 52.0, 50.1, 40.7, 22.4, 21.4; **HRMS** (EI$^+$) Calcd for $C_{26}H_{24}KN_2O_4S_3^+$ [M+K]$^+$ 563.0530, found 563.0552.



2-((5,5-Dimethyl-5,6,7,8-tetrahydronaphtho[2,3-*d*][1,3]dioxol-6-yl)disulfanyl)isoindoline-1,3-dione (5h): Colorless solid; R_f 0.40 (hexane/EtOAc = 3/1); m.p. 187-189 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96-7.92 (m, 2H), 7.82-7.78 (m, 2H), 6.72 (s, 1H), 6.49 (s, 1H), 5.88 (s, 2H), 3.59 (dd, *J* = 10.3, 3.0 Hz, 1H), 2.95-2.81 (m, 2H), 2.53-2.46 (m, 1H), 2.27-2.16 (m, 1H), 1.37 (s, 3H), 1.23 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 167.9, 146.3, 145.8, 137.4, 134.9, 132.3, 128.0, 124.2, 108.4, 106.3, 100.9, 64.0, 38.8, 30.7, 29.2, 27.1, 26.2; **HRMS** (FAB$^+$) Calcd for $C_{21}H_{19}NO_4S_2^+$ [M]$^+$ 413.0750, found 413.0763.

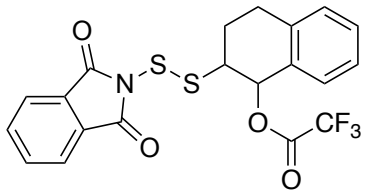


2-(((5-Oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (5i): Colorless solid; R_f 0.50 (hexane/EtOAc = 1/1); m.p. decomp; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94-7.92 (m, 2H), 7.82-7.79 (m, 2H), 7.26 (d, *J* = 8.1 Hz, 2H), 7.16 (d, *J* = 8.4 Hz, 2H), 3.82 (s, 2H), 2.71-2.62 (m, 2H), 2.58-2.43 (m, 2H), 2.30 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 175.7, 167.7, 138.30, 138.25, 135.0, 132.4, 129.5, 125.1, 124.3, 87.7, 54.3, 33.5, 28.7, 21.2; **HRMS** (FAB$^+$) Calcd for $C_{20}H_{18}NO_4S_2^+$ [M+H]$^+$ 400.0672, found 400.0683.

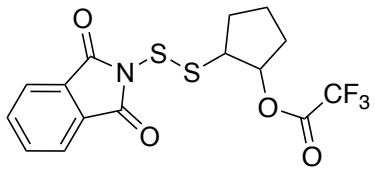


2-((1,3-Dioxoisoindolin-2-yl)disulfanyl)cyclohexyl 2,2,2-trifluoroacetate (7a): Colorless oil; R_f 0.43 (hexane/EtOAc = 3/1); 8.3:1 dr (determined by ^{19}F NMR); **^{1}H NMR** (600 MHz, $CDCl_3$, major) δ 7.95-7.92 (m, 2H), 7.82-7.79 (m, 2H), 5.00 (dt, *J* = 9.9, 4.3 Hz, 1H), 3.17 (dt,

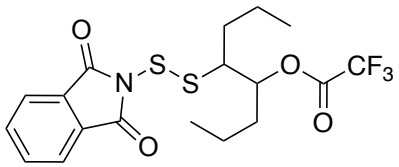
J = 10.7, 4.1 Hz, 1H), 2.28-2.13 (m, 2H), 2.07-1.97 (m, 1H), 1.89-1.72 (m, 2H), 1.61-1.38 (m, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, major) δ 167.7, 156.6 (q, J_{C-F} = 42.0 Hz), 134.9, 132.3, 124.1, 114.4 (q, J_{C-F} = 284.4 Hz), 78.4, 54.3, 31.4, 31.1, 25.2, 23.6; **^{9}F NMR** (376 MHz, $CDCl_3$, major) δ –75.3; **^{1}H NMR** (600 MHz, $CDCl_3$, minor) δ 7.95-7.92 (m, 2H), 7.82-7.79 (m, 2H), 4.88 (dt, *J* = 9.5, 4.3 Hz, 1H), 3.14-3.10 (m, 1H), 2.28-2.11 (m, 2H), 1.89-1.70 (m, 3H), 1.61-1.38 (m, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, minor) δ 168.7, 156.7 (q, J_{C-F} = 42.0 Hz), 134.9, 132.0, 124.1, 114.6 (q, J_{C-F} = 284.4 Hz), 79.4, 52.5, 30.6, 29.4, 24.8, 23.2; **^{19}F NMR** (376 MHz, $CDCl_3$, minor) δ –75.1; **HRMS** (EI^+) Calcd for $C_{16}H_{14}F_3NO_4S_2^+$ $[M]^+$ 405.0311, found 405.0309.



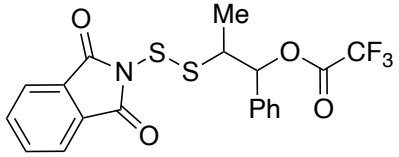
2-((1,3-Dioxoisoindolin-2-yl)disulfanyl)-1,2,3,4-tetrahydronaphthalen-1-yl 2,2,2-trifluoroacetate (7b): Colorless oi; R_f 0.29 (hexane/EtOAc = 5/1); 2:1 dr (determined by ^{1}H NMR) **^{1}H NMR** (400 MHz, $CDCl_3$, major) δ 7.96-7.94 (m, 2H), 7.82-7.79 (m, 2H), 7.33-7.15 (m, 4H), 6.41 (d, *J* = 5.8 Hz, 1H), 3.84-3.79 (m, 1H), 3.00-2.95 (m, 2H), 2.52-2.34 (m, 2H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, major) δ 167.6, 157.1 (q, J_{C-F} = 43.0 Hz), 136.8, 135.0, 132.2, 130.5, 129.8, 129.5, 129.2, 126.9, 124.2, 114.6 (q, J_{C-F} = 285.1 Hz), 76.2, 51.5, 26.3, 25.0; **^{1}H NMR** (400 MHz, $CDCl_3$, minor) δ 7.96-7.94 (m, 2H), 7.82-7.79 (m, 2H), 7.33-7.15 (m, 4H), 6.20 (d, *J* = 4.2 Hz, 1H), 3.72-3.67 (m, 1H), 3.00-2.93 (m, 2H), 2.17-2.09 (m, 2H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, minor) δ 168.3, 157.1 (q, J_{C-F} = 43.0 Hz), 137.0, 134.9, 132.0, 130.6, 129.7, 129.5, 129.1, 127.1, 124.2, 114.5 (q, J_{C-F} = 286.5 Hz), 76.5, 50.4, 25.9, 23.6; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –78.0; **HRMS** (EI^+) Calcd for $C_{20}H_{14}F_3NO_4S_2^+$ $[M]^+$ 453.0311, found 453.0323.



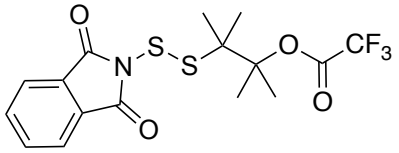
2-((1,3-Dioxoisoindolin-2-yl)disulfaneyl)cyclopentyl 2,2,2-trifluoroacetate (7c): Colorless oil; R_f 0.53 (hexane/EtOAc = 3/1); 1.6:1 dr (determined by ^{19}F NMR) **^{1}H NMR** (400 MHz, $CDCl_3$, major) δ 7.96-7.93 (m, 2H), 7.82-7.79 (m, 2H), 5.54-5.52 (m, 1H), 3.81-3.77 (m, 1H), 2.37-2.30 (m, 1H), 2.28-2.17 (m, 1H), 1.92-1.83 (m, 3H), 1.81-1.70 (m, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, major) δ 167.5, 156.8 (q, J_{C-F} = 42.4 Hz), 135.0, 132.2, 124.2, 114.6 (q, J_{C-F} = 284.4 Hz), 84.7, 56.1, 31.3, 30.1, 22.9; **^{9}F NMR** (376 MHz, $CDCl_3$, major) δ –75.1; **^{1}H NMR** (400 MHz, $CDCl_3$, minor) δ 7.96-7.93 (m, 2H), 7.82-7.79 (m, 2H), 5.47-5.45 (m, 1H), 3.73-3.70 (m, 1H), 2.37-2.30 (m, 1H), 2.28-2.17 (m, 1H), 1.92-1.83 (m, 3H), 1.81-1.70 (m, 1H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, minor) δ 167.2, 156.6 (q, J_{C-F} = 42.4 Hz), 134.9, 132.3, 124.2, 114.2 (q, J_{C-F} = 284.4 Hz), 85.0, 54.4, 30.9, 30.3, 22.8; **^{19}F NMR** (376 MHz, $CDCl_3$, minor) δ –75.4; **HRMS** (EI^+) Calcd for $C_{15}H_{12}F_3NO_4S_2^+$ $[M]^+$ 391.0154, found 391.0178.



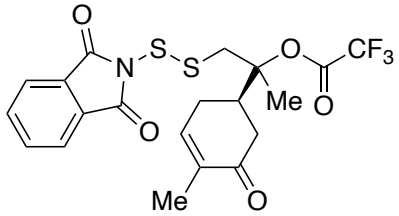
5-((1,3-Dioxoisoindolin-2-yl)disulfanyl)octan-4-yl 2,2,2-trifluoroacetate (7d): colorless oil; R_f 0.55 (hexane/EtOAc = 3/1); 2.6:1 dr (determined by ^{19}F NMR) **^{1}H NMR** (400 MHz, $CDCl_3$, major) δ 7.95-7.93 (m, 2H), 7.82-7.80 (m, 2H), 5.61-5.59 (m, 1H), 3.58-3.53 (m, 1H), 1.93-1.26 (m, 8H), 0.98 (t, J = 7.4 Hz, 3H), 0.90 (t, J = 7.2 Hz, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, major) δ 167.7, 157.3 (q, $J_{C\text{-}F}$ = 41.7 Hz), 135.0, 132.3, 124.2, 114.7 (q, $J_{C\text{-}F}$ = 284.4 Hz), 79.7, 55.3, 33.3, 30.9, 20.5, 18.9, 13.8; **^{9}F NMR** (376 MHz, $CDCl_3$, major) δ –75.0; **^{1}H NMR** (400 MHz, $CDCl_3$, minor) δ 7.98-7.96 (m, 2H), 7.84-7.82 (m, 2H), 5.38-5.34 (m, 1H), 3.30-3.26 (m, 1H), 1.93-1.26 (m, 8H), 1.00 (t, J = 7.2 Hz, 3H), 0.90 (t, J = 7.3 Hz, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, minor) δ 167.0, 157.3 (q, $J_{C\text{-}F}$ = 42.0 Hz), 135.1, 132.3, 124.4, 114.7 (q, $J_{C\text{-}F}$ = 284.0 Hz), 79.7, 58.0, 33.0, 31.2, 20.4, 18.7, 13.9, 13.7; **^{19}F NMR** (376 MHz, $CDCl_3$, minor) δ –75.0; **HRMS** (EI$^+$) Calcd for $C_{18}H_{20}F_3NO_4S_2^+$ $[M]^+$ 435.0780, found 435.0800.



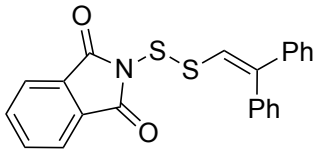
2-((1,3-Dioxoisoindolin-2-yl)disulfanyl)-1-phenylpropyl 2,2,2-trifluoroacetate (7e): Colorless oil; R_f 0.48 (hexane/EtOAc = 3/1); 1.1:1 dr (determined by ^{19}F NMR) **^{1}H NMR** (400 MHz, $CDCl_3$, major) δ 7.96-7.94 (m, 2H), 7.82-7.79 (m, 2H), 7.44-7.33 (m, 5H), 5.91 (d, J = 9.0 Hz, 1H), 3.73-3.65 (m, 1H), 1.37 (d, J = 7.1 Hz, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, major) δ 168.7, 156.3 (q, $J_{C\text{-}F}$ = 42.4 Hz), 135.6, 135.0, 132.3, 129.5, 129.0, 127.4, 124.2, 114.4 (q, $J_{C\text{-}F}$ = 284.4 Hz), 83.0, 49.9, 14.9; **^{19}F NMR** (376 MHz, $CDCl_3$, major) δ –75.1; **^{1}H NMR** (400 MHz, $CDCl_3$, minor) δ 7.96-7.94 (m, 2H), 7.82-7.79 (m, 2H), 7.44-7.33 (m, 5H), 5.82 (d, J = 8.5 Hz, 1H), 3.65-3.57 (m, 1H), 1.09 (d, J = 7.1 Hz, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, minor) δ 167.8, 156.3 (q, $J_{C\text{-}F}$ = 42.4 Hz), 135.5, 135.0, 132.0, 129.5, 129.0, 127.4, 124.2, 114.5 (q, $J_{C\text{-}F}$ = 284.0 Hz), 83.0, 49.9, 14.9 (q, $J_{C\text{-}F}$ = 285.1 Hz); **^{19}F NMR** (376 MHz, $CDCl_3$, minor) δ –74.9; **HRMS** (EI$^+$) Calcd for $C_{19}H_{14}F_3NO_4S_2^+$ $[M]^+$ 441.0311, found 441.0320.



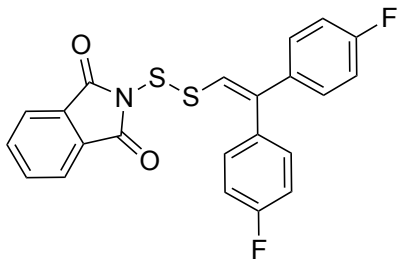
3-((1,3-Dioxoisoindolin-2-yl)disulfanyl)-2,3-dimethylbutan-2-yl 2,2,2-trifluoroacetate (7f): Colorless solid; R_f 0.48 (hexane/EtOAc = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94-7.93 (m, 2H), 7.83-7.88 (m, 2H), 1.70 (s, 6H), 1.56 (s, 6H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.5, 155.9 (q, $J_{C\text{-}F}$ = 41.7 Hz), 135.0, 132.1, 124.3, 114.4 (q, $J_{C\text{-}F}$ = 285.8 Hz), 92.9, 59.5, 24.0, 21.5; **^{19}F NMR** (376 MHz, $CDCl_3$) δ –75.6; **HRMS** (EI$^+$) Calcd for $C_{16}H_{16}F_3NO_4S_2^+$ $[M]^+$ 407.0473, found 407.0460.



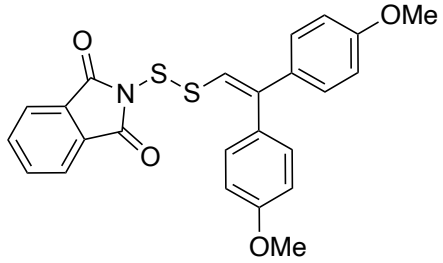
1-((1,3-Dioxoisoindolin-2-yl)disulfanyl)-2-(4-methyl-5-oxocyclohex-3-en-1-yl)propan-2-yl 2,2,2-trifluoroacetate (7g): Colorless oil; R_f 0.18 (hexane/EtOAc = 3/1); 1.2:1 dr (determined by ^{1}H NMR) **^{1}H NMR** (600 MHz, $CDCl_3$, major) δ 7.97-7.95 (m, 2H), 7.84-7.82 (m, 2H), 6.78-6.74 (m, 1H), 3.96 (d, J = 14.1 Hz, 1H), 3.88 (d, J = 14.1 Hz, 1H), 2.90-2.78 (m, 1H), 2.68-2.66 (m, 1H), 2.55-2.47 (m, 1H), 2.40-2.29 (m, 2H), 1.79 (s, 3H), 1.69 (s, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, major) δ 197.8, 167.6, 156.0 (q, $J_{C\text{-}F}$ = 42.0 Hz), 143.1, 136.1, 135.2, 132.2, 124.4, 114.3 (q, $J_{C\text{-}F}$ = 285.1 Hz), 90.1, 47.2, 41.4, 38.6, 26.8, 20.5, 15.7; **^{19}F NMR** (376 MHz, $CDCl_3$, major) δ –75.3; **^{1}H NMR** (400 MHz, $CDCl_3$, minor) δ 7.97-7.95 (m, 2H), 7.84-7.82 (m, 2H), 6.78-6.74 (m, 1H), 3.97 (d, J = 14.1 Hz, 1H), 3.88 (d, J = 14.1 Hz, 1H), 2.90-2.78 (m, 1H), 2.65-2.60 (m, 1H), 2.55-2.47 (m, 1H), 2.40-2.29 (m, 2H), 1.79 (s, 3H), 1.69 (s, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$, minor) δ 197.6, 167.5, 155.9 (q, $J_{C\text{-}F}$ = 42.0 Hz), 143.7, 135.9, 135.2, 132.2, 124.4, 114.3 (q, $J_{C\text{-}F}$ = 285.5 Hz), 90.2, 47.1, 41.6, 38.7, 26.6, 20.5, 15.7; **^{19}F NMR** (376 MHz, $CDCl_3$, minor) δ –75.3; **HRMS** (FAB^+) Calcd for $C_{20}H_{19}F_3NO_5S_2^+$ $[M+H]^+$ 474.0651, found 474.0654.



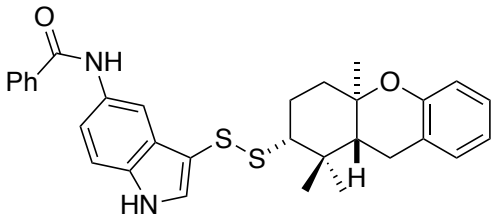
2-((2,2-Diphenylvinyl)disulfanyl)isoindoline-1,3-dione (9a): Colorless solid; R_f 0.43 (hexane/EtOAc = 3/1); m.p. 165-167 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.95-7.93 (m, 2H), 7.82-7.78 (m, 2H), 7.38 (s, 1H), 7.35-7.27 (m, 8H), 7.14-7.10 (m, 2H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.5, 142.9, 140.5, 138.1, 135.0, 132.3, 129.8, 128.6, 128.5, 128.4, 128.3, 128.0, 127.7, 124.3; **HRMS** (EI^+) Calcd for $C_{22}H_{15}NO_2S_2^+$ $[M]^+$ 389.0539, found 389.0549.



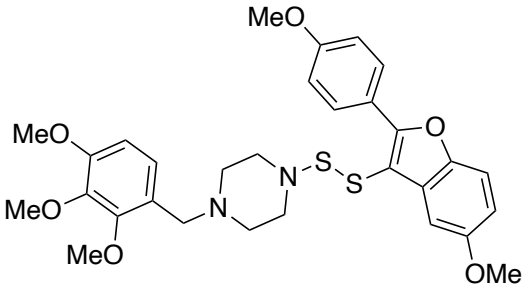
2-((2,2-Bis(4-fluorophenyl)vinyl)disulfanyl)isoindoline-1,3-dione (9b): Colorless solid; R_f 0.15 (hexane/EtOAc = 8/1); m.p. 121-123 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.96-7.92 (m, 2H), 7.82-7.78 (m, 2H), 7.31 (s, 1H), 7.29-7.25 (m, 2H), 7.13-7.06 (m, 2H), 7.05-6.98 (m, 4H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.5, 162.8 (d, $J_{C\text{-}F}$ = 246.7 Hz), 162.7 (d, $J_{C\text{-}F}$ = 247.0 Hz), 141.0, 136.7 (d, $J_{C\text{-}F}$ = 3.3 Hz), 135.0, 133.8 (d, $J_{C\text{-}F}$ = 3.3 Hz), 132.3, 131.6 (d, $J_{C\text{-}F}$ = 7.9 Hz), 129.4 (d, $J_{C\text{-}F}$ = 8.3 Hz), 128.3, 124.3, 115.7 (d, $J_{C\text{-}F}$ = 21.6 Hz), 115.6 (d, $J_{C\text{-}F}$ = 21.6 Hz); **^{19}F NMR** (376 MHz, CDCl3) δ –112.6-–112.7 (m), –113.8-–113.9 (m); **HRMS** (EI^+) Calcd for $C_{22}H_{13}F_2NO_2S_2^+$ $[M]^+$ 425.0350, found 425.0372.



2-((2,2-Bis(4-methoxyphenyl)vinyl)disulfanyl)isoindoline-1,3-dione (9c): Yellow solid; R_f 0.13 (hexane/EtOAc = 8/1); m.p. 168-171 °C; **^{1}H NMR** (400 MHz, $CDCl_3$) δ 7.94-7.90 (m, 2H), 7.80-7.76 (m, 2H), 7.26 (d, *J* = 8.7 Hz, 2H), 7.17 (s, 1H), 7.05 (d, *J* = 8.8 Hz, 2H), 6.87 (d, *J* = 8.8 Hz, 2H), 6.82 (d, *J* = 8.8 Hz, 2H), 3.82 (s, 3H), 3.80 (s, 3H); **^{13}C{^{1}H} NMR** (150 MHz, $CDCl_3$) δ 167.6, 159.62, 159.59, 142.9, 134.9, 133.7, 132.4, 131.2, 130.6, 129.1, 125.1, 124.2, 113.9, 113.8, 55.5, 55.4; **HRMS** (EI^+) Calcd for $C_{24}H_{19}NO_4S_2^+$ $[M]^+$ 449.0750, found 449.0756.

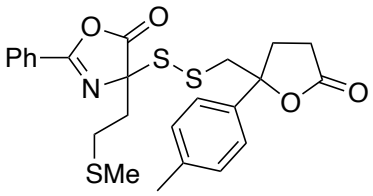


***N*-(3-(((2*R**,4a*R**,9a*R**)-1,1,4a-Trimethyl-2,3,4,4a,9,9a-hexahydro-1*H*-xanthen-2-yl)disulfanyl)-1*H*-indol-5-yl)benzamide (13):** Colorless oli; R_f 0.28 (hexane/EtOAc = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 9.54 (s, 1H), 8.47 (s, 1H), 8.06 (s, 1H), 7.90 (d, *J* = 7.0 Hz, 2H), 7.74 (d, *J* = 8.4 Hz, 1H), 7.57 (app. t, *J* = 7.3 Hz, 1H), 7.50 (app. t, *J* = 7.6 Hz, 2H), 7.35 (d, *J* = 2.4 Hz, 1H), 7.07 (app. t, *J* = 7.2 Hz, 1H), 7.03 (d, *J* = 7.6 Hz, 1H), 6.96 (dd, *J* = 8.4, 1.7 Hz, 1H), 6.82 (app. dt, *J* = 7.4, 1.1 Hz, 1H), 6.76 (d, *J* = 8.2 Hz, 1H), 2.78-2.74 (m, 1H), 2.63-2.60 (m, 2H), 2.35-2.31 (m, 1H), 2.07-2.04 (m,1H), 1.75-1.64 (m, 3H), 1.18 (s, 3H), 0.92 (s, 3H), 0.79 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 166.3, 153.0, 136.9, 135.3, 133.5, 132.1, 130.7, 129.8, 129.1, 127.4, 127.1, 126.0, 122.1, 120.0, 119.8, 117.1, 114.3, 108.5, 104.3, 76.5, 65.2, 49.4, 40.4, 38.3, 28.8, 27.8, 23.6, 19.9, 16.6; **HRMS** (FAB^+) Calcd for $C_{31}H_{33}N_2O_2S_2^+$ $[M+H]^+$ 529.1978, found 529.1981.

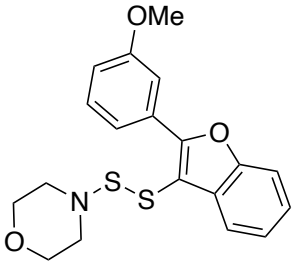


1-((5-Methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)-4-(2,3,4-trimethoxybenzyl)piperazine (15): Yellow oil; R_f 0.25 (hexane/EtOAc = 3/1); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.19 (d, *J* = 9.0 Hz, 2H), 7.36 (d, *J* = 8.9 Hz, 1H), 7.16 (d, *J* = 2.6 Hz, 1H), 7.02 (d, *J* = 9.0 Hz, 2H), 6.90 (dd, *J* = 8.9, 2.6 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 1H), 6.60 (d, *J* = 8.5 Hz, 1H), 3.89 (s, 3H), 3.88 (s, 3H), 3.85 (s, 6H), 3.74 (s, 3H), 3.32 (s, 2H), 2.84-2.79 (m, 4H), 2.38-2.29 (m, 4H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 160.3, 156.5, 155.7, 153.0, 152.7, 148.5, 142.6, 131.4, 129.1, 124.8, 124.4, 123.1, 114.1, 113.7, 111.7, 110.1, 107.0, 102.7, 61.4,

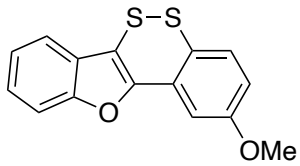
60.9, 56.3, 56.1, 55.9, 55.5, 53.2; **HRMS** (FAB$^+$) Calcd for $C_{30}H_{35}N_2O_6S_2^+$ $[M+H]^+$ 583.1931, found 583.1942.



4-(2-(Methylthio)ethyl)-4-(((5-oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)-2-phenyloxazol-5(4H)-one (17): Colorless oil; R_f 0.13 (hexane/EtOAc = 3/1); 1:1 dr (determined by ^{13}C NMR analysis of the isolated product); **^{1}H NMR** (400 MHz, $CDCl_3$) δ 8.05-8.01 (m, 2H+2H), 7.66-7.62 (m, 1H+1H), 7.54-7.50 (m, 2H+2H), 7.13-7.11 (m, 4H+4H), 3.38-3.33 (m, 2H), 3.28-3.21 (m, 2H), 2.61-2.34 (m, 8H+8H), 2.33(s, 3H), 2.32(s, 3H), 2.043 (s, 3H), 2.038 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 176.6, 176.5, 175.6, 162.8, 162.7, 138.3, 138.2, 138.1, 133.6, 129.4, 129.14, 129.12, 128.50, 128.48, 125.3, 125.2, 124.9, 124.8, 87.7, 87.6, 79.69, 79.67, 52.7, 52.5, 33.42, 33.38, 32.9, 32.8, 29.69, 29.66, 28.6, 21.2, 21.1, 15.08, 15.07; **HRMS** (FAB$^+$) Calcd for $C_{24}H_{26}NO_4S_3^+$ $[M+H]^+$ 488.1018, found 488.1003.



4-((2-(3-Methoxyphenyl)benzofuran-3-yl)disulfanyl)morpholine (18): Colorless oil; R_f 0.25 (hexane/EtOAc = 10/1); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.83 (d, J = 7.7 Hz, 1H), 7.80 (s, 1H), 7.75 (d, J = 6.5 Hz, 1H), 7.50 (d, J = 6.7 Hz, 1H), 7.42 (app. t, J = 7.9 Hz, 1H), 7.35-7.31 (m, 2H), 7.00 (dd, J = 8.2, 2.5 Hz, 1H), 3.89 (s, 3H), 3.51-3.50 (m, 4H), 2.79-2.77 (m, 4H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 159.8, 154.6, 153.7, 131.2, 130.4, 129.8, 125.4, 123.4, 120.6, 120.0, 115.6, 112.5, 112.0, 111.4, 66.9, 55.54, 55.51; **HRMS** (FAB$^+$) Calcd for $C_{19}H_{19}NO_3S_2^+$ $[M]^+$ 373.0801, found 373.0795.



2-Methoxybenzo[5,6][1,2]dithiino[4,3-*b*]benzofuran (19): Colorless oil; R_f 0.53 (hexane/EtOAc = 3/1); **^{1}H NMR** (600 MHz, $CDCl_3$) δ 7.53 (d, J = 8.3 Hz, 1H), 7.52 (d, J = 7.8 Hz, 1H), 7.36 (app. t, J = 7.7 Hz, 1H), 7.34 (d, J = 8.4 Hz, 1H), 7.30 (app. t, J = 7.5 Hz, 1H), 7.28 (d, J = 2.8 Hz, 1H), 6.84 (dd, J = 8.6, 2.8 Hz, 1H), 3.88 (s, 3H); **^{13}C{^{1}H} NMR** (150MHz, $CDCl_3$) δ 160.4, 154.3, 153.5, 130.6, 129.8, 126.6, 125.9, 123.7, 120.3, 120.1, 115.6, 112.5, 111.9, 109.8, 55.8; **HRMS** (FAB$^+$) Calcd for $C_{15}H_{10}O_2S_2^+$ $[M]^+$ 286.0117, found 286.0111.

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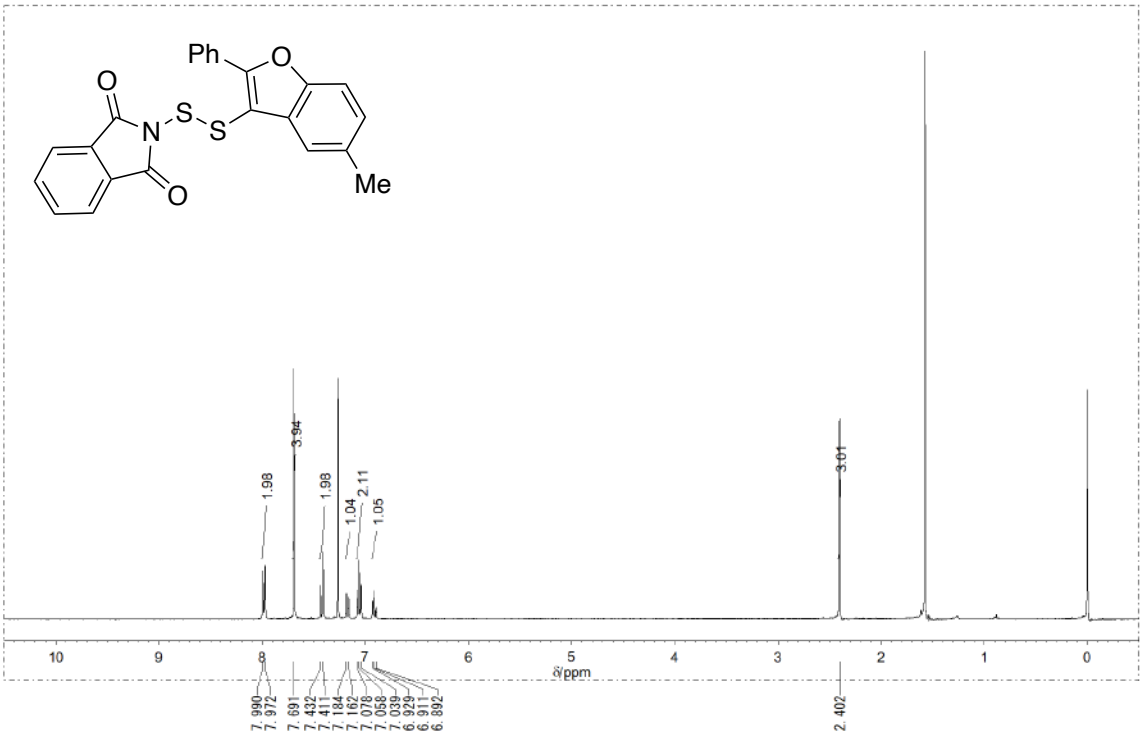
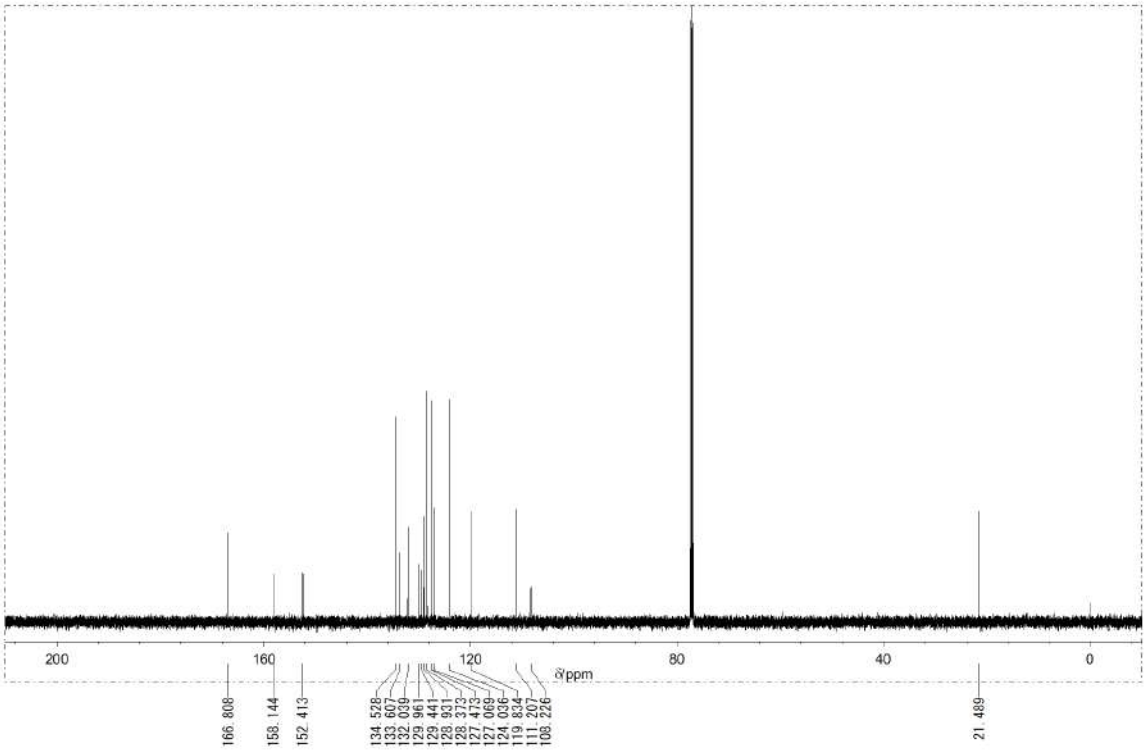
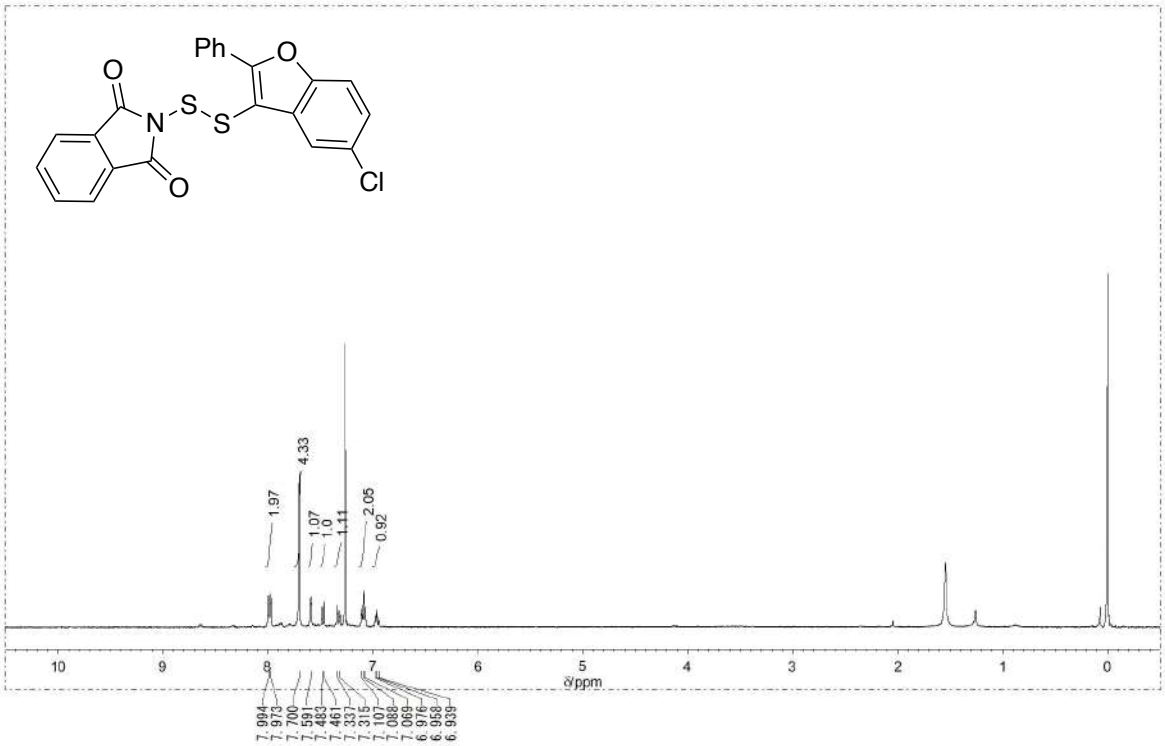
^{1}H and ^{13}C NMR Spectra of Compounds

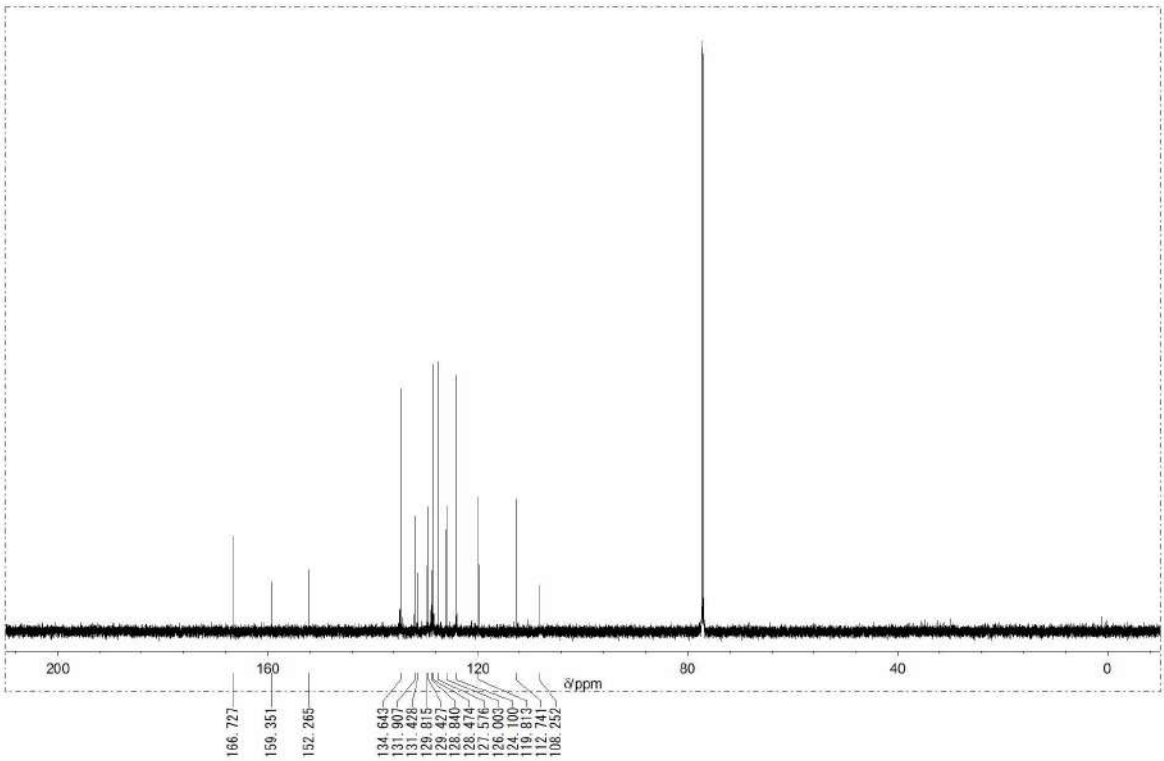
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((5-methyl-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3a**) ($CDCl_3$)



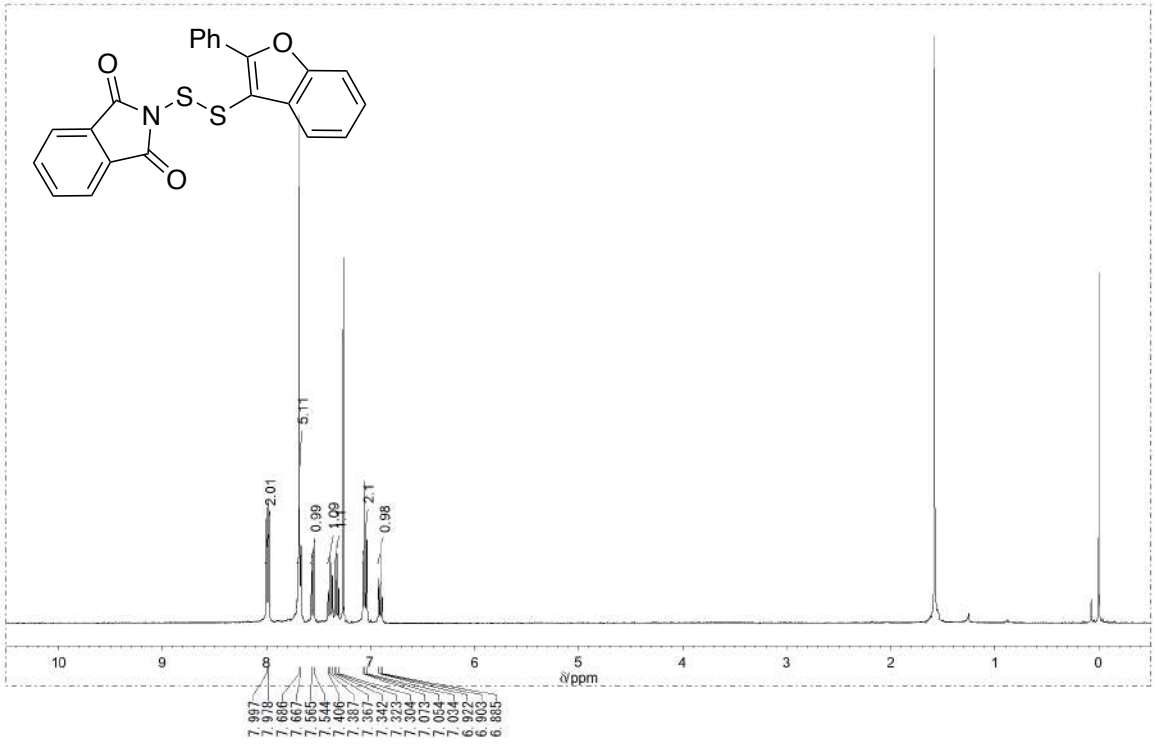


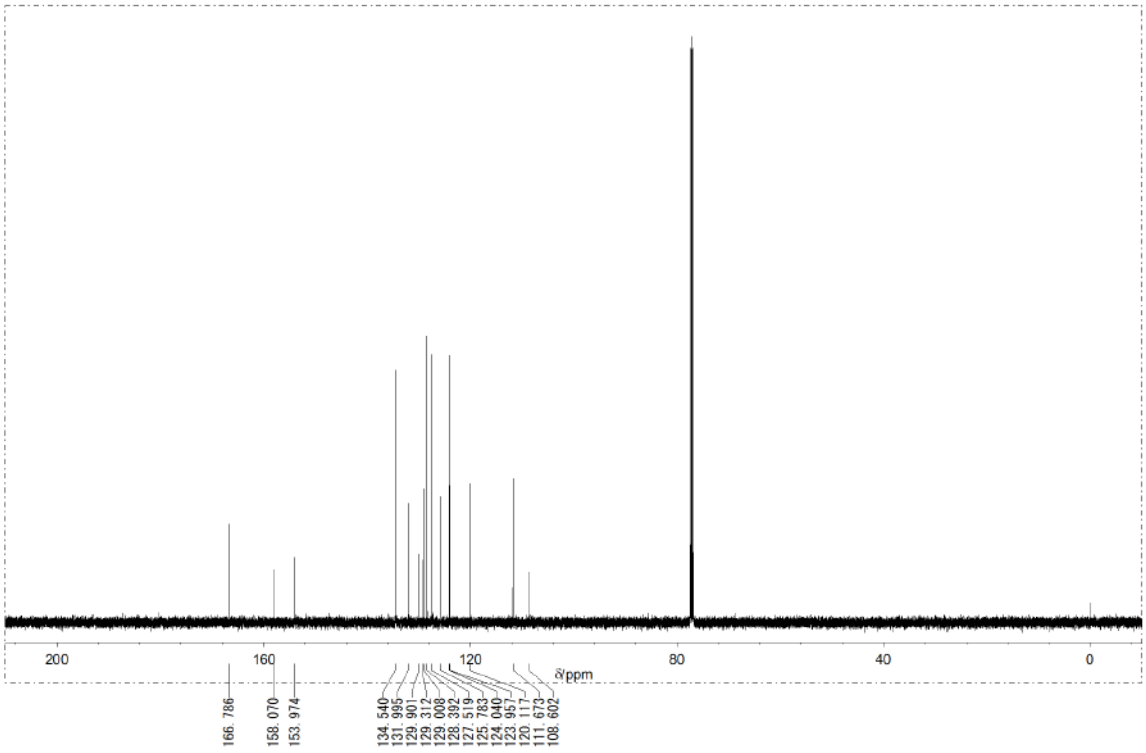
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((5-chloro-2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3b**) ($CDCl_3$)



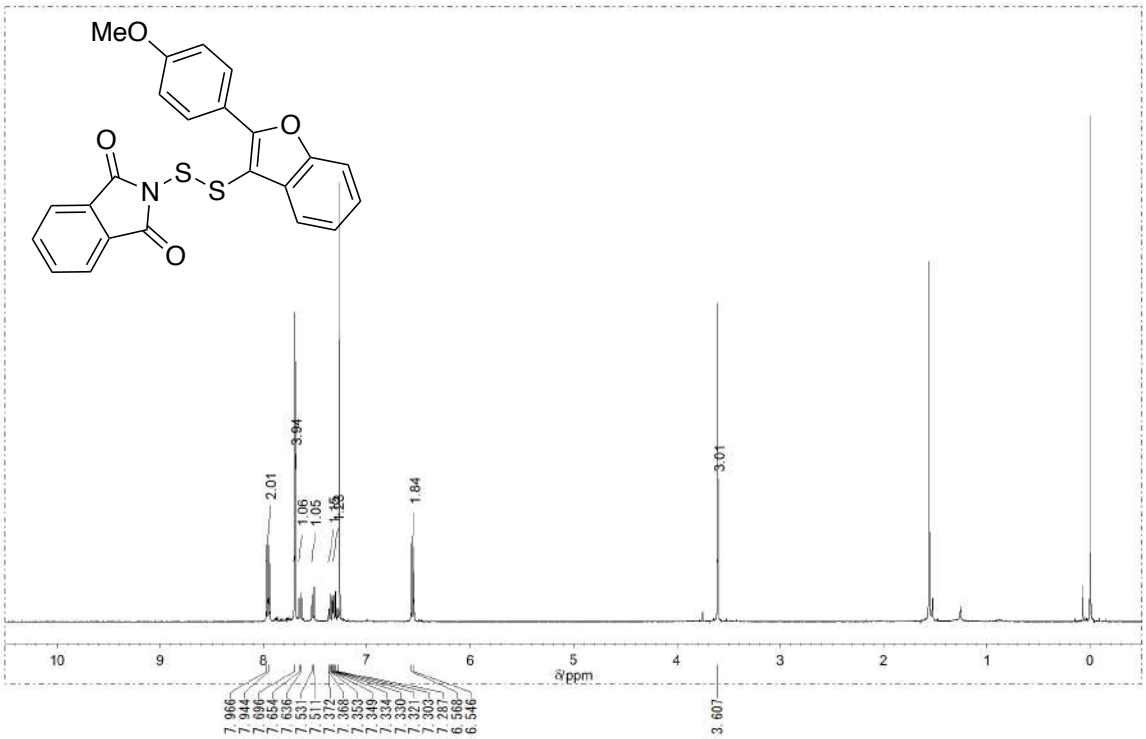


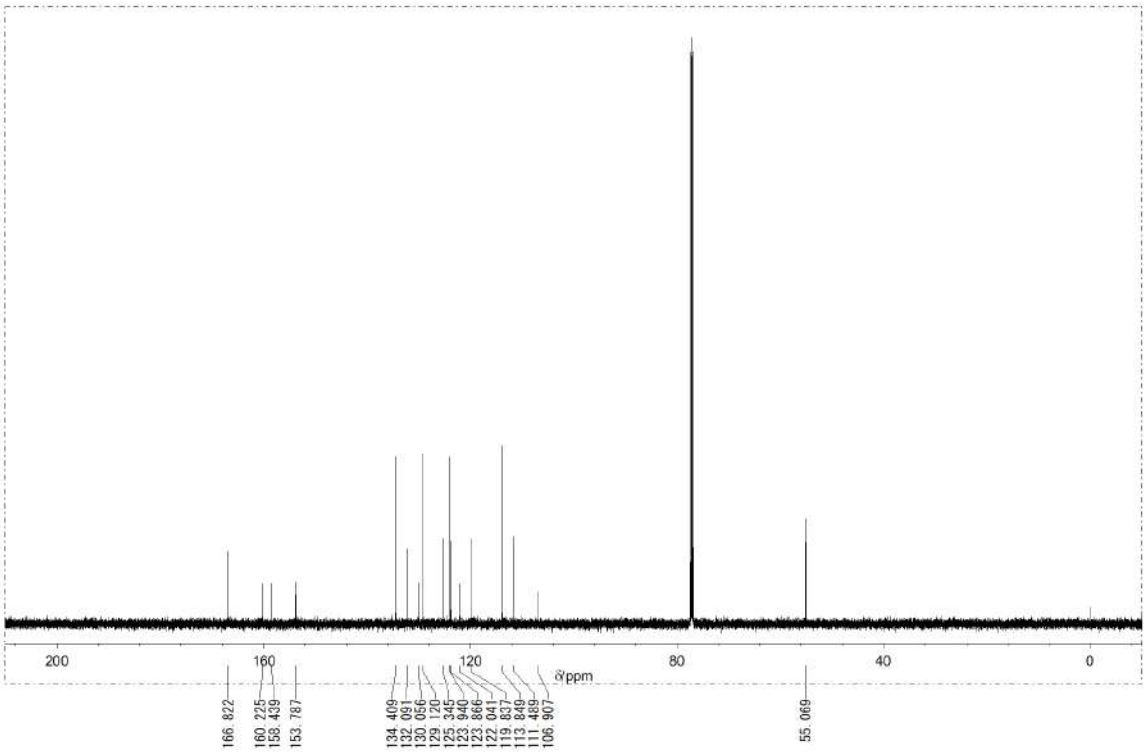
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2-phenylbenzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3c**) ($CDCl_3$)



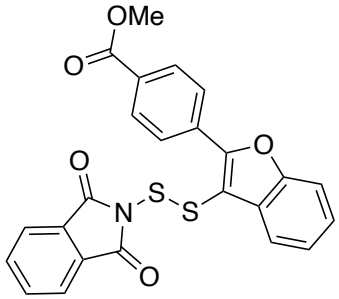


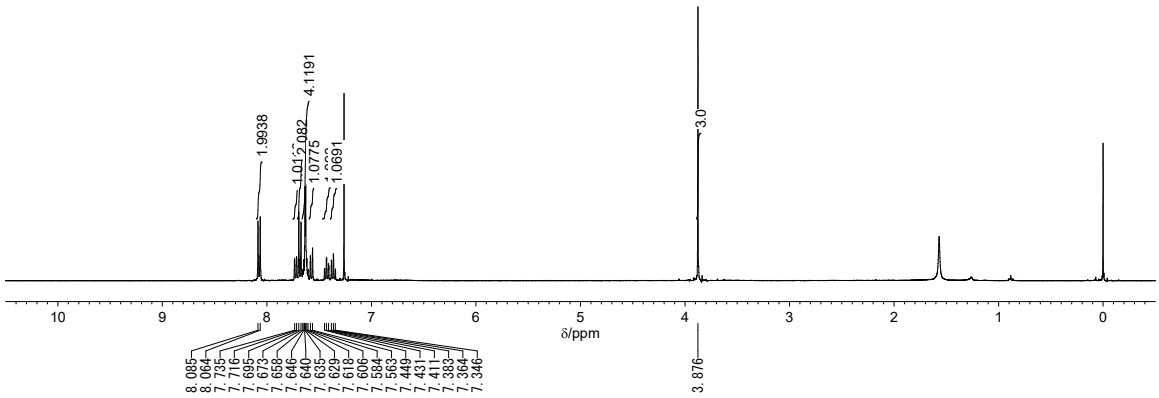
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3d**) ($CDCl_3$)

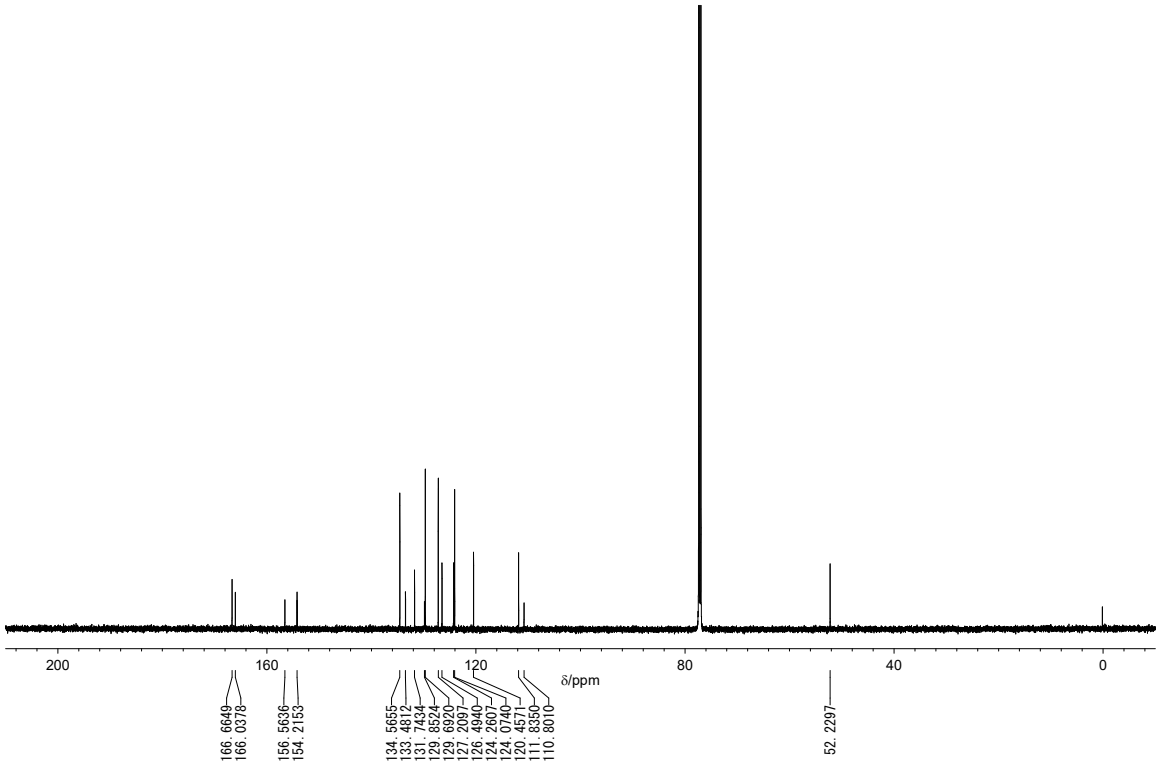




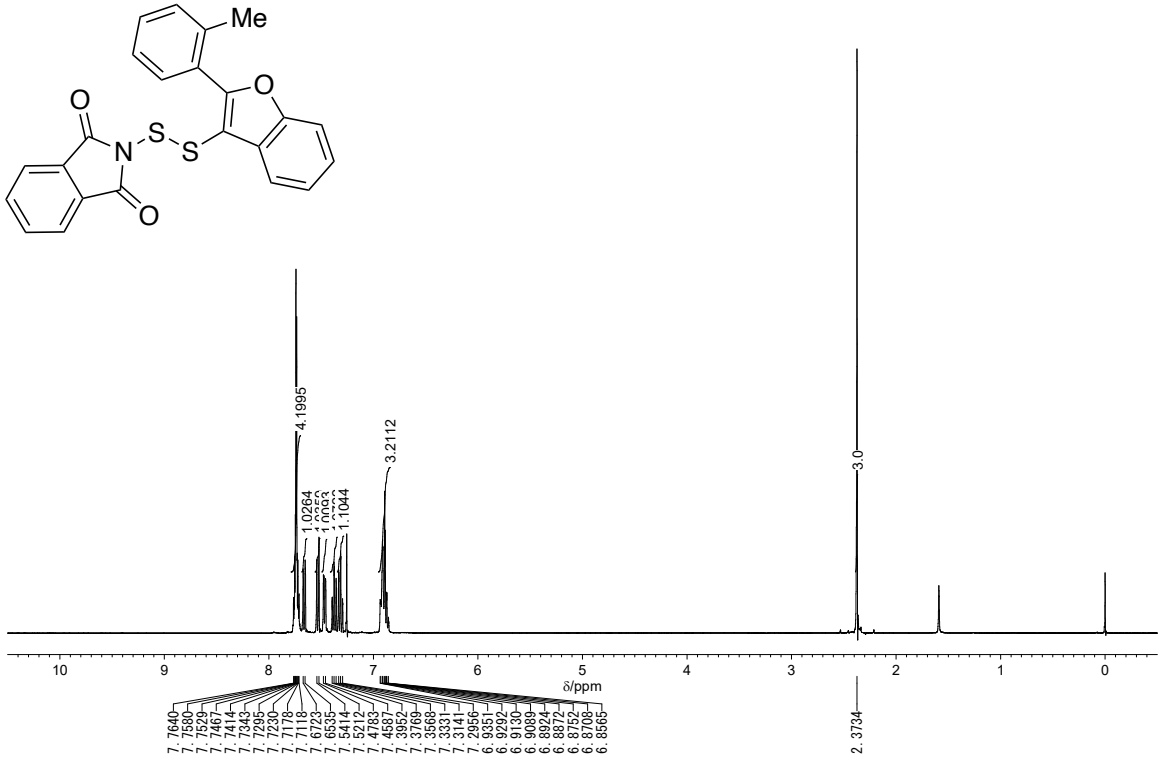
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of methyl 4-(3-((1,3-dioxoisoindolin-2-yl)disulfanyl)benzofuran-2-yl)benzoate (**3e**) ($CDCl_3$)

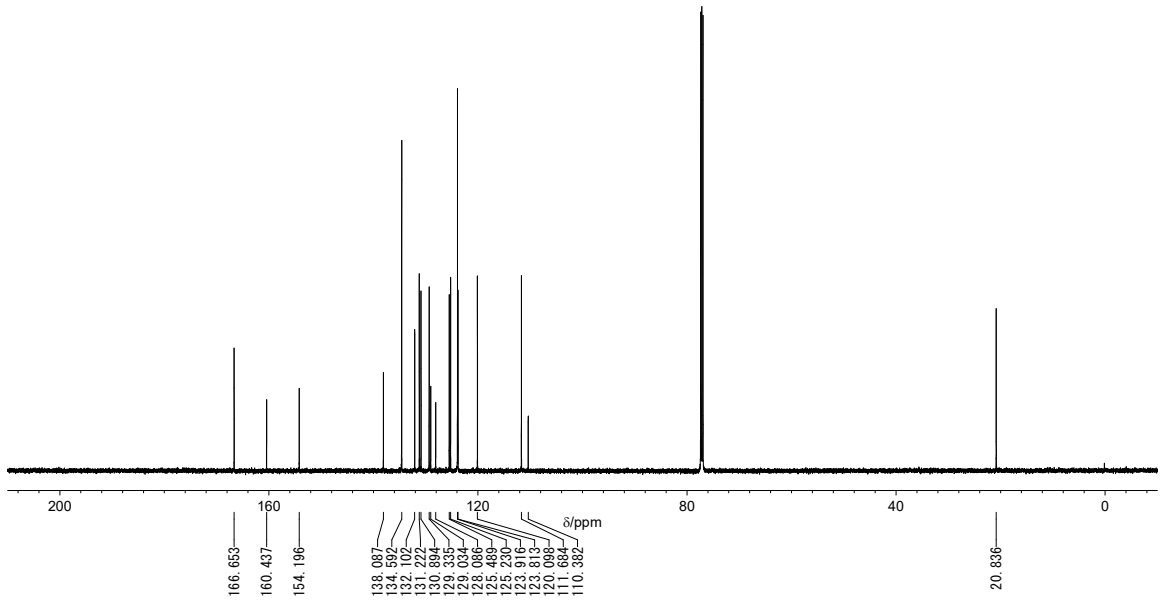




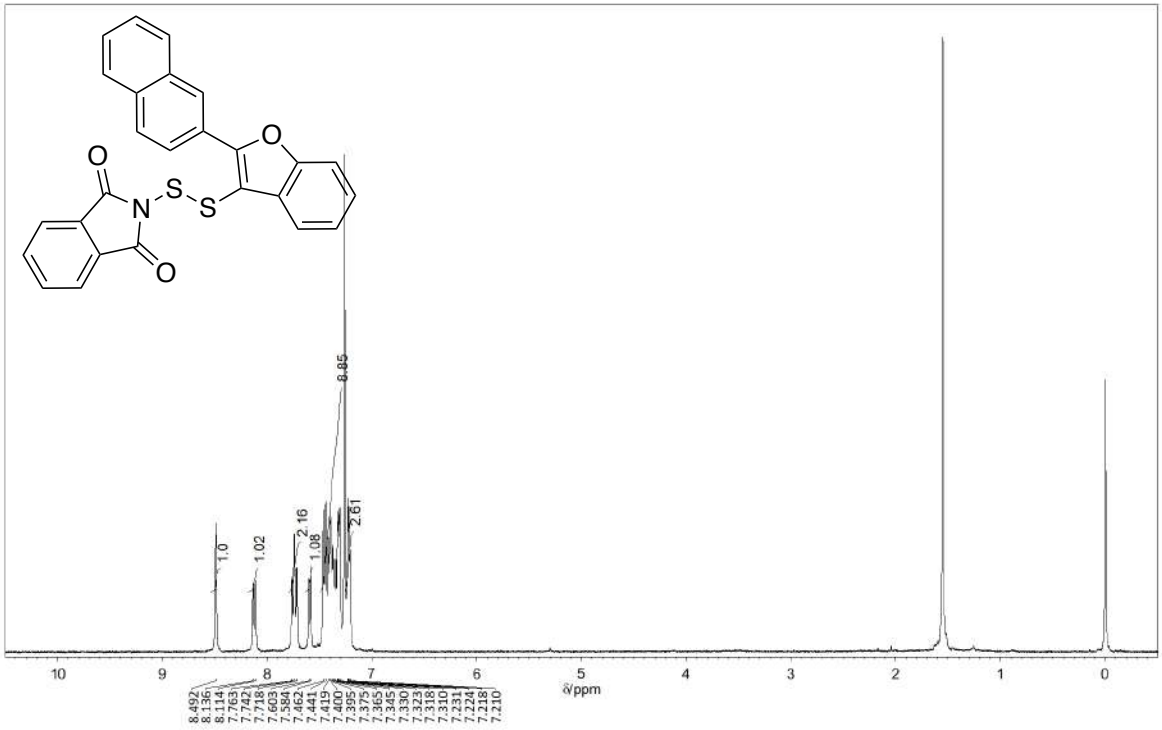


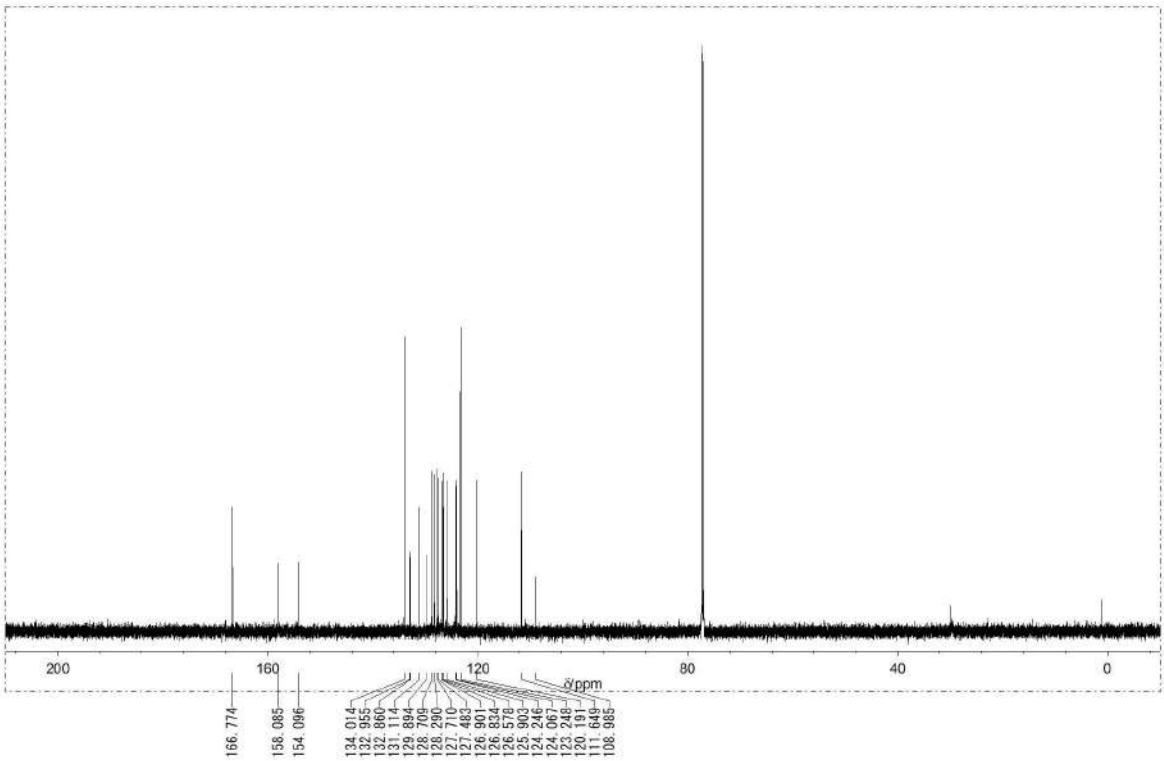
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((2-(*o*-tolyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3f**) ($CDCl_3$)



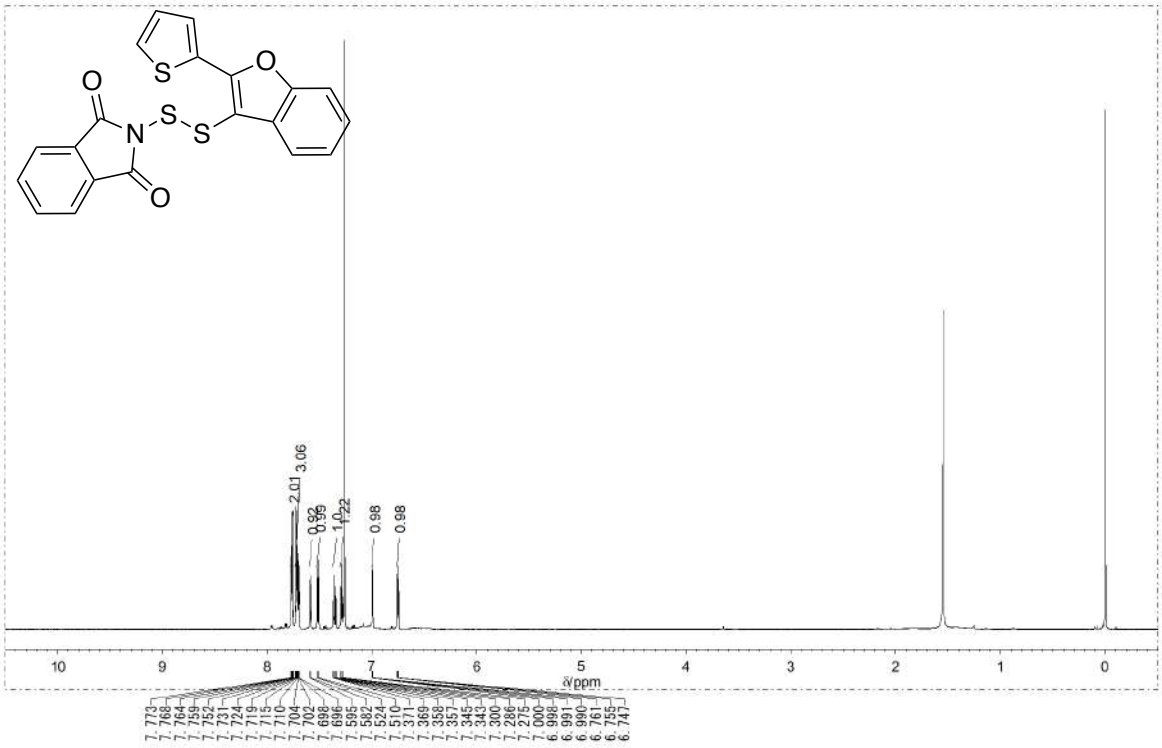


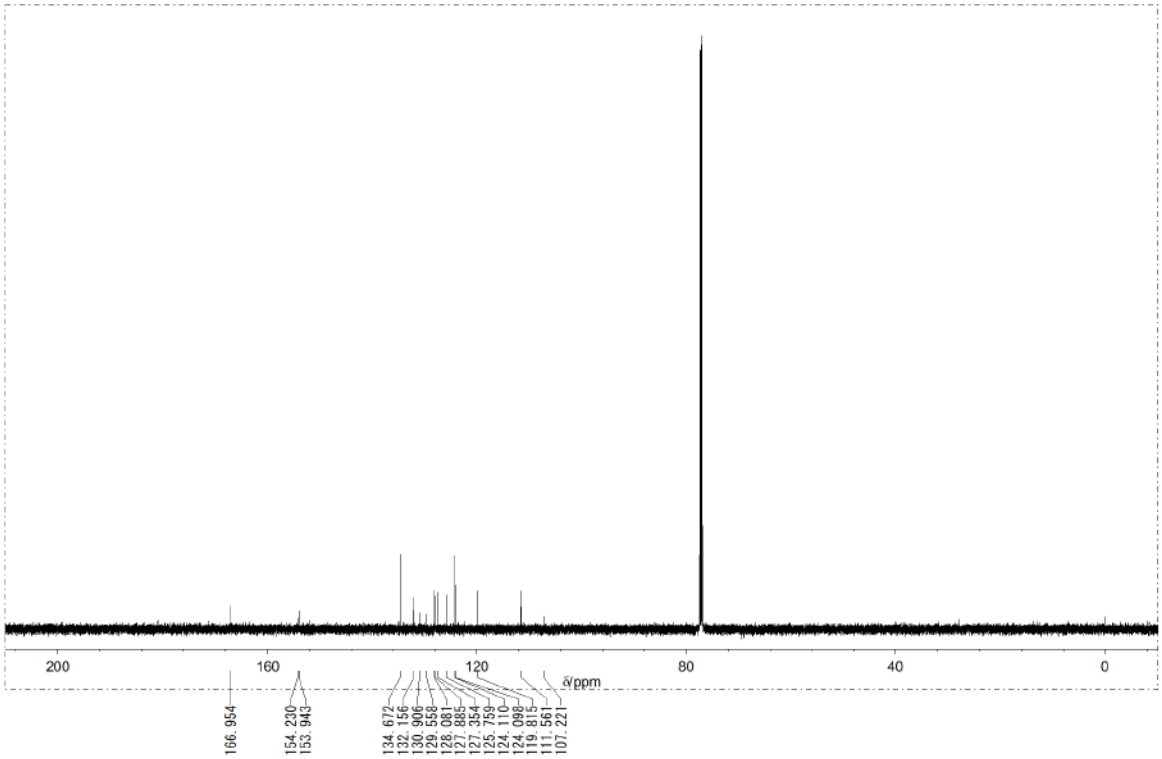
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2-(naphthalen-2-yl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3g**) ($CDCl_3$)



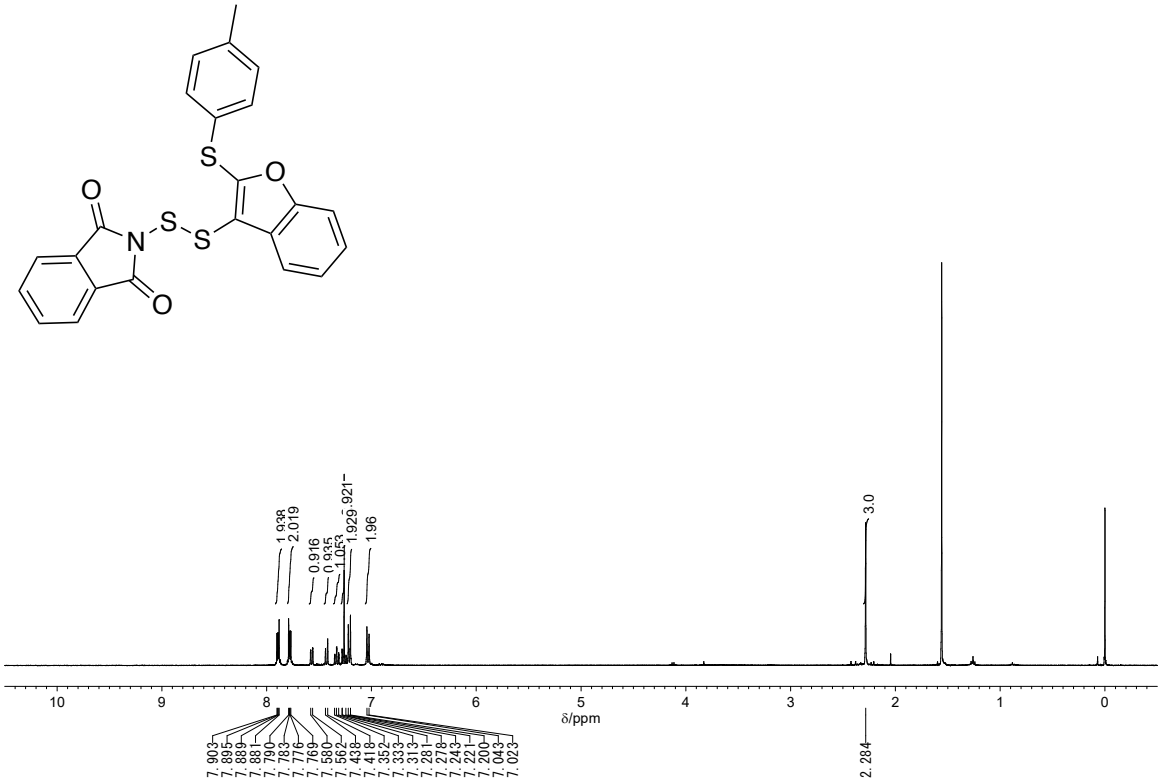


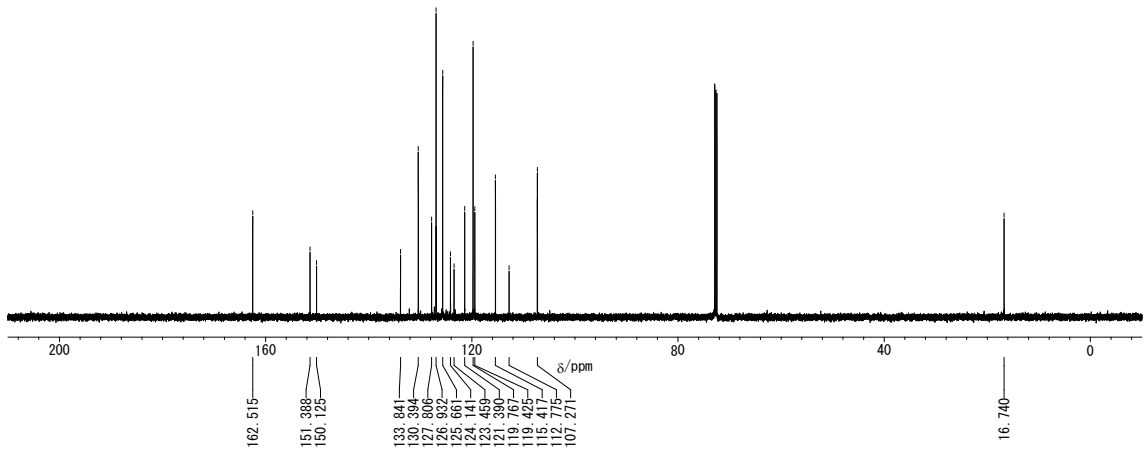
^{1}H NMR (600 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2-(thiophen-2-yl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3h**) ($CDCl_3$)



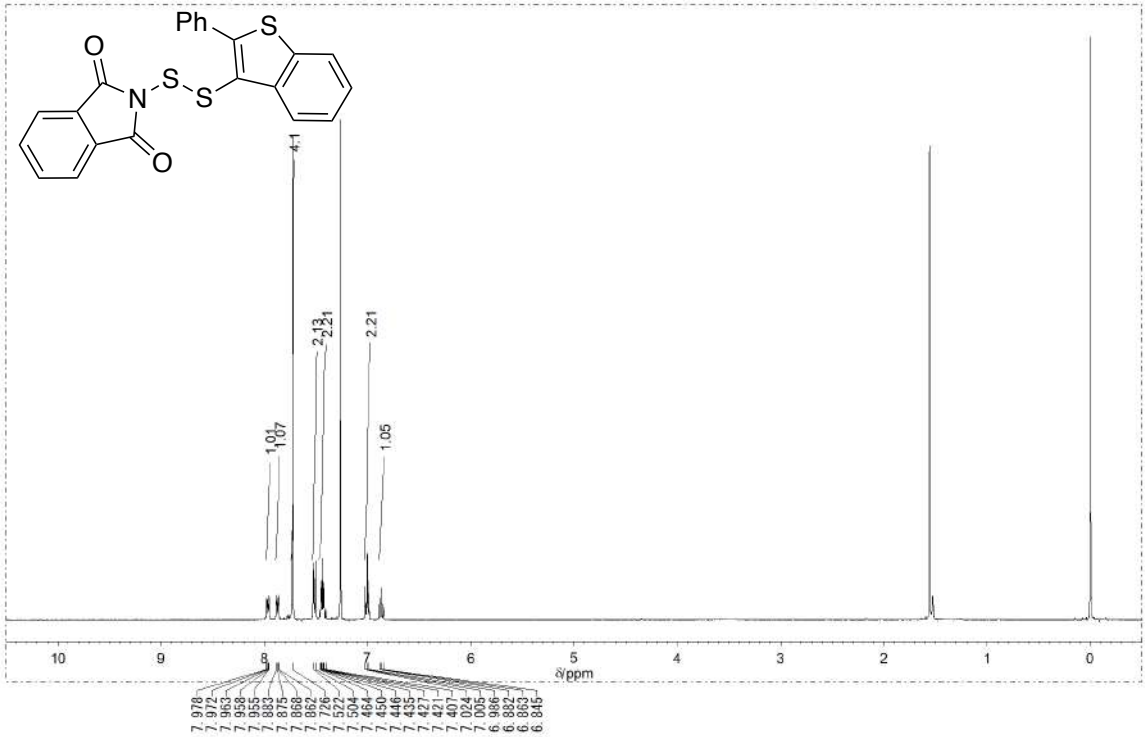


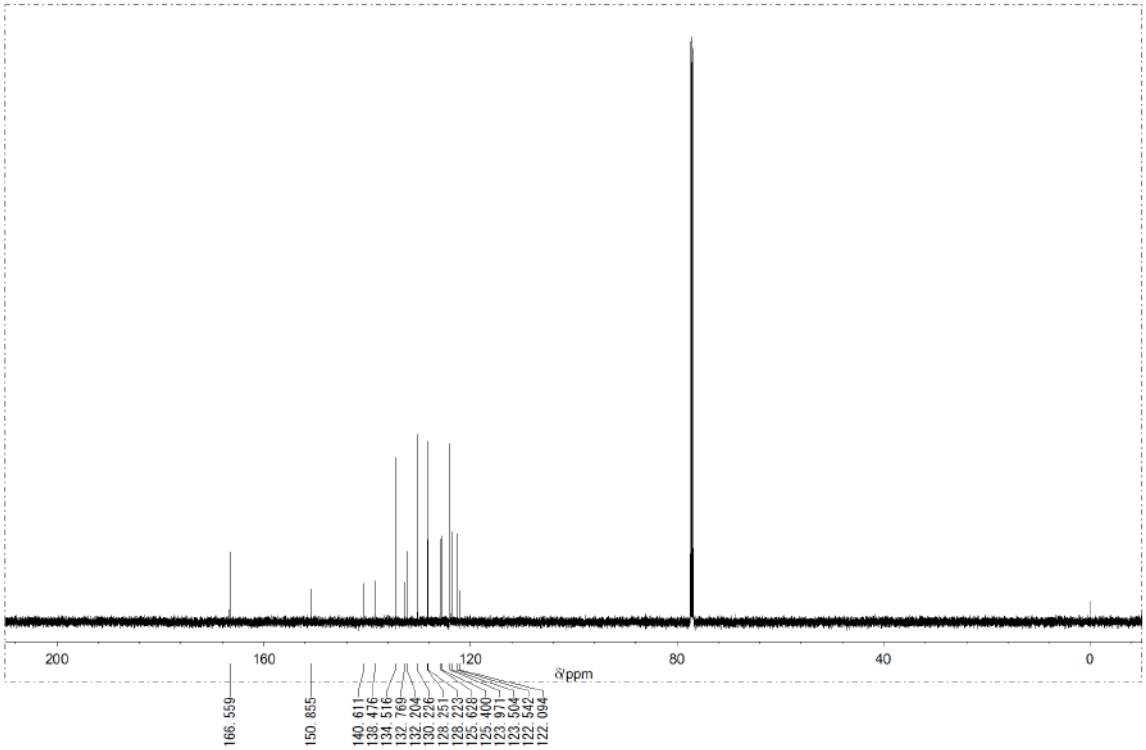
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((2-(*p*-tolylthio)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3i**) ($CDCl_3$)



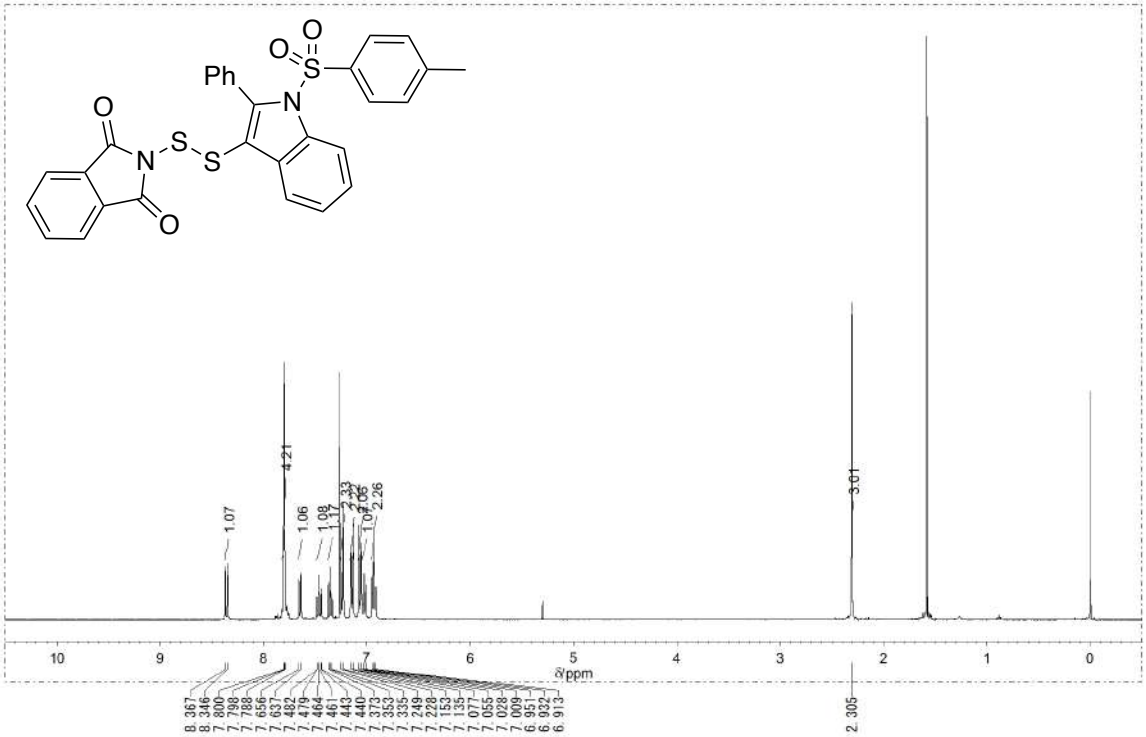


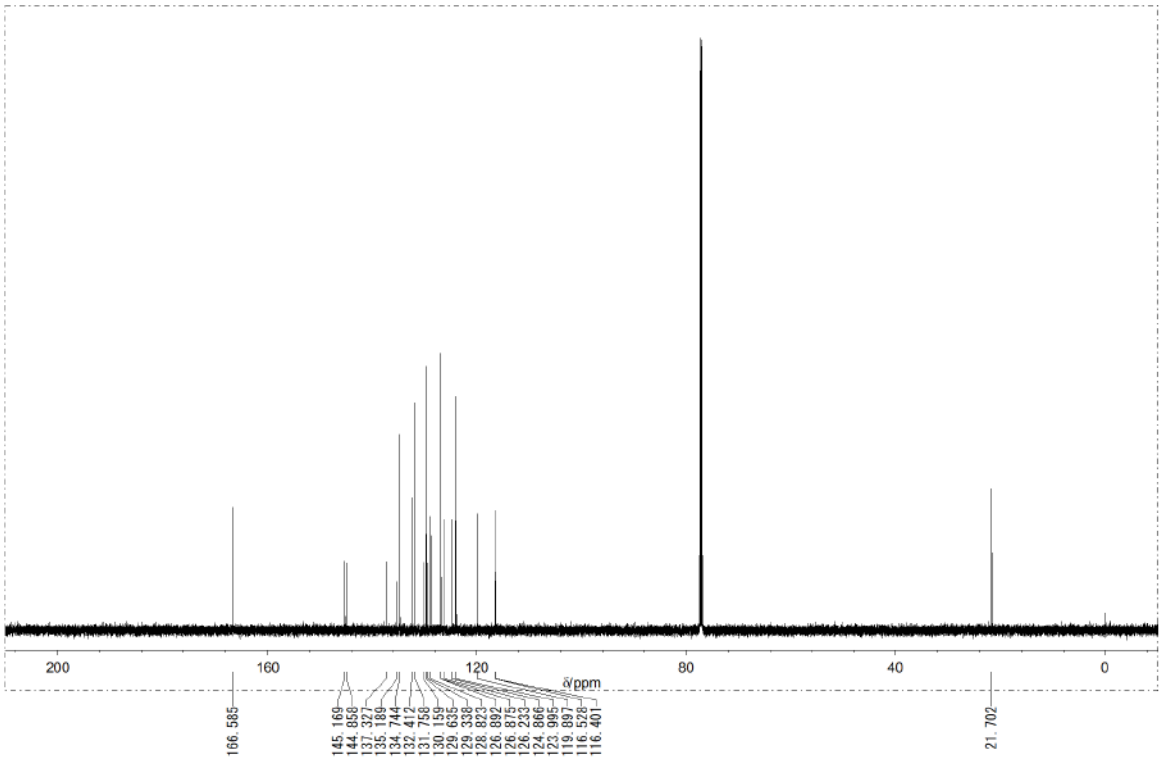
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((2-phenylbenzo[*b*]thiophen-3-yl)disulfanyl)isoindoline-1,3-dione (**3j**) ($CDCl_3$)



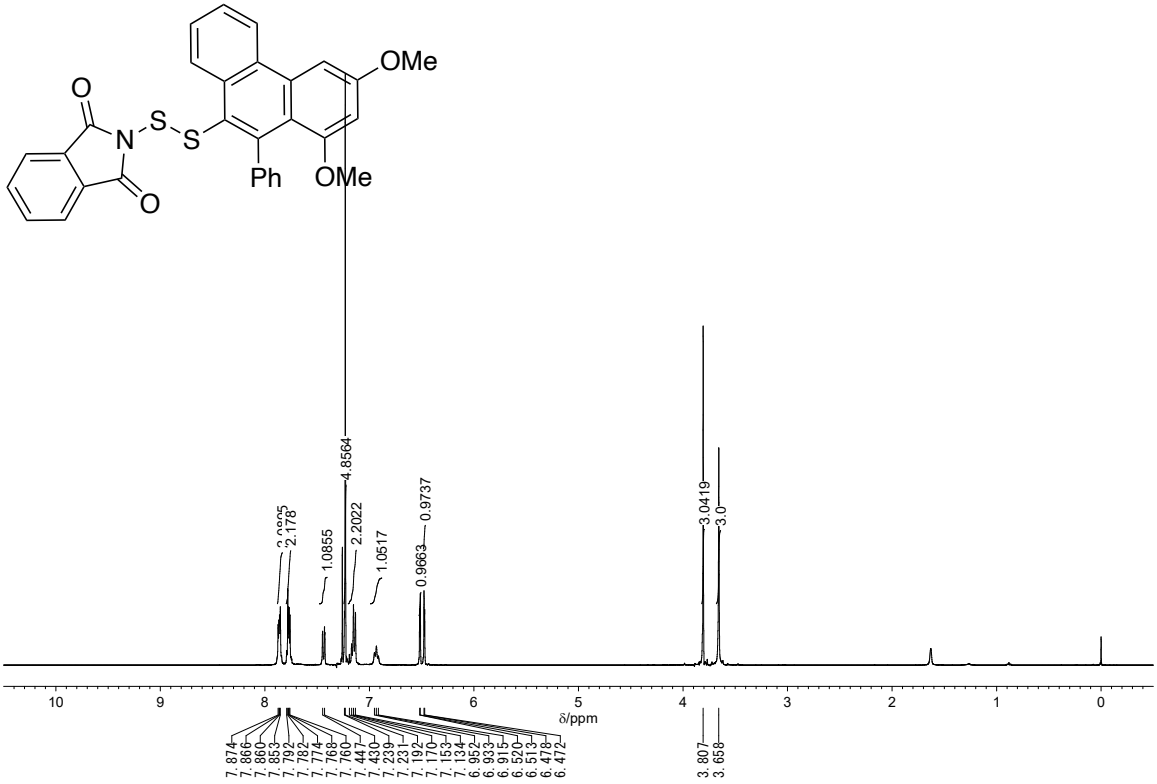


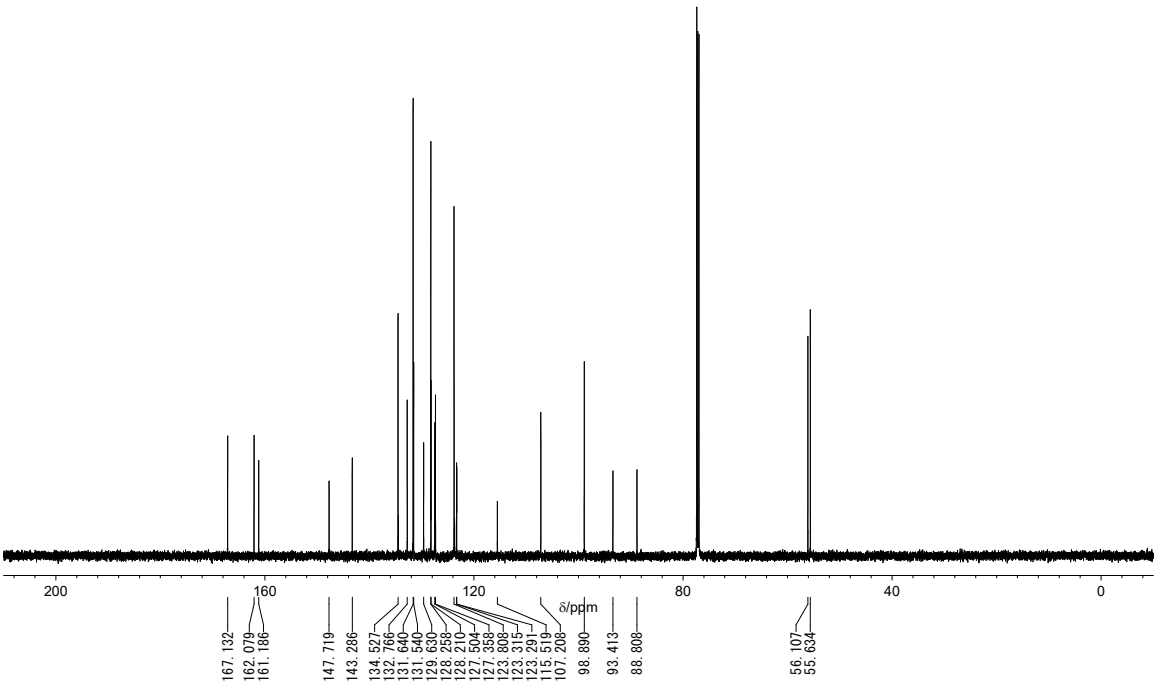
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((2-phenyl-1-tosyl-1*H*-indol-3-yl)disulfanyl)isoindoline-1,3-dione (**3k**) ($CDCl_3$)



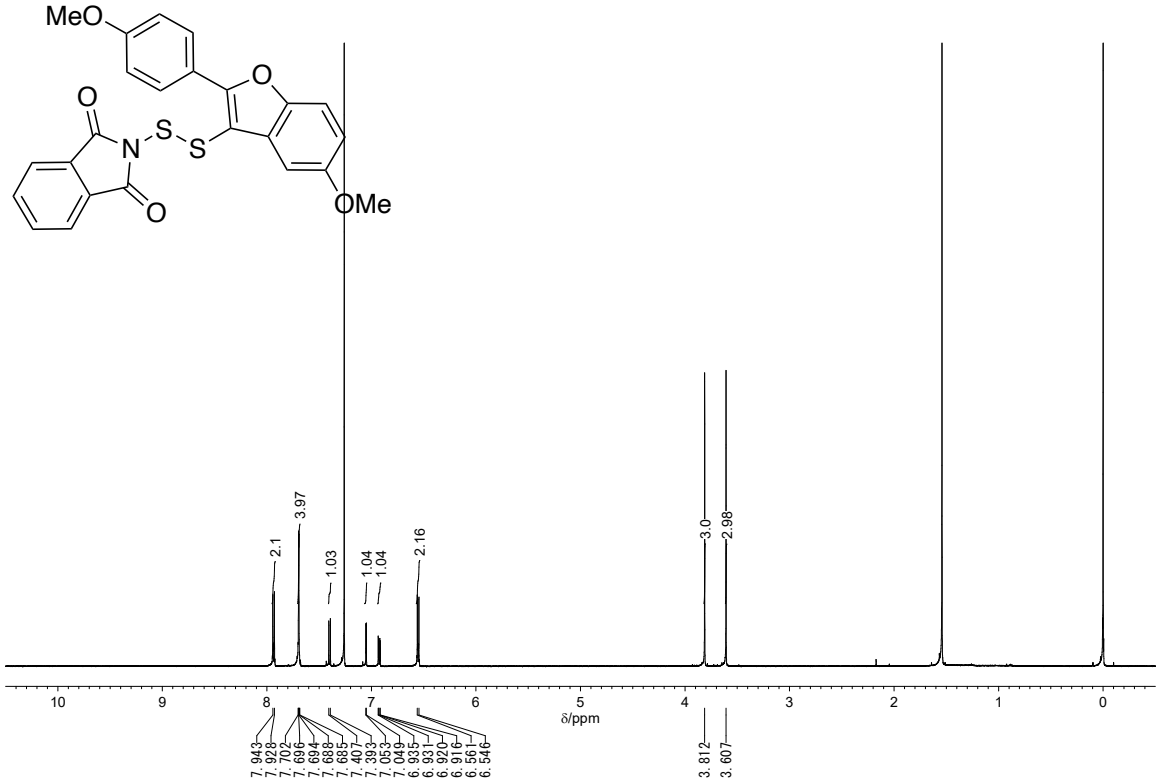


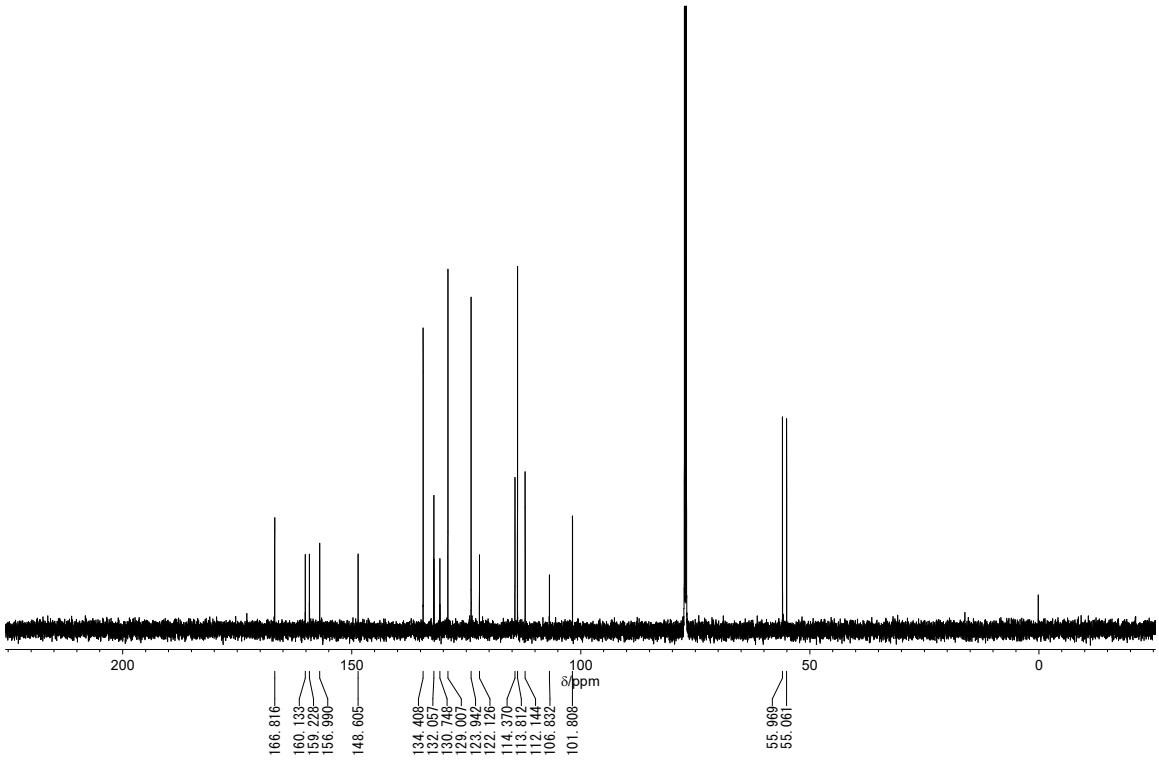
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((1,3-dimethoxy-10-phenylphenanthren-9-yl)disulfanyl)isoindoline-1,3-dione (**3l**) ($CDCl_3$)



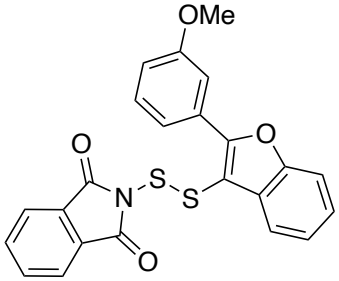


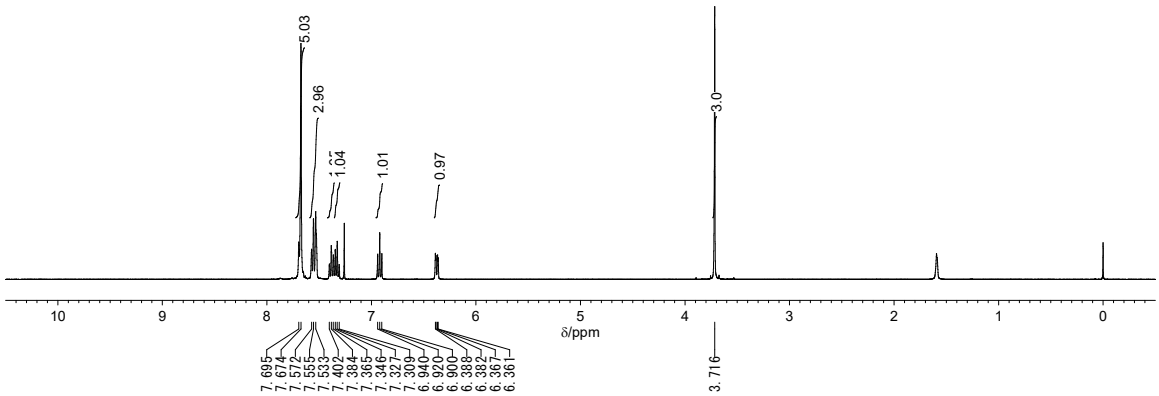
^{1}H NMR (600 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3m**) ($CDCl_3$)

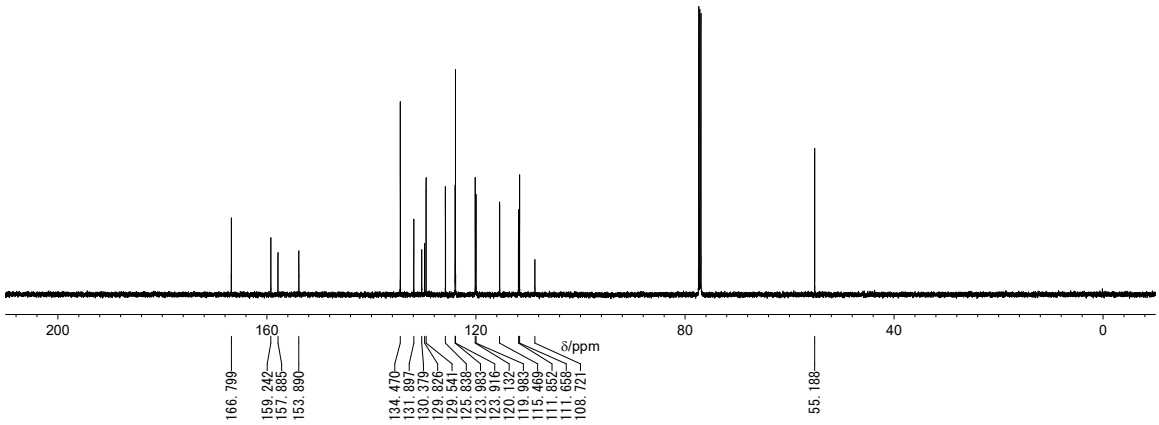




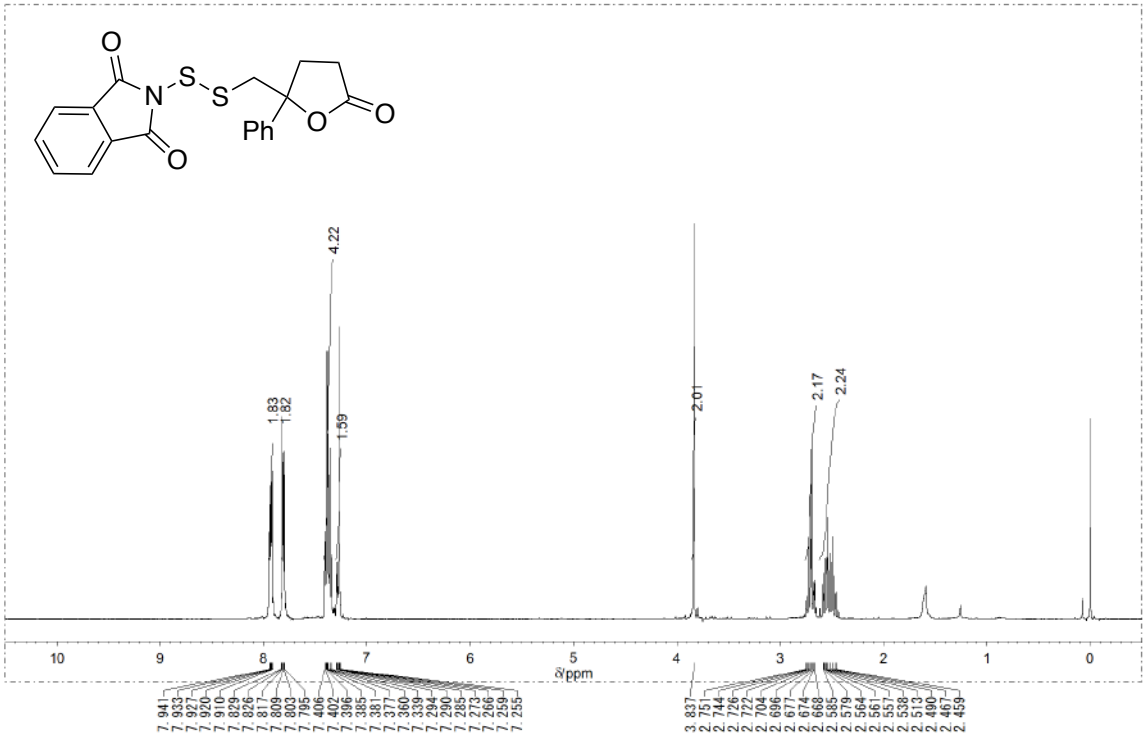
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)isoindoline-1,3-dione (**3n**) ($CDCl_3$)

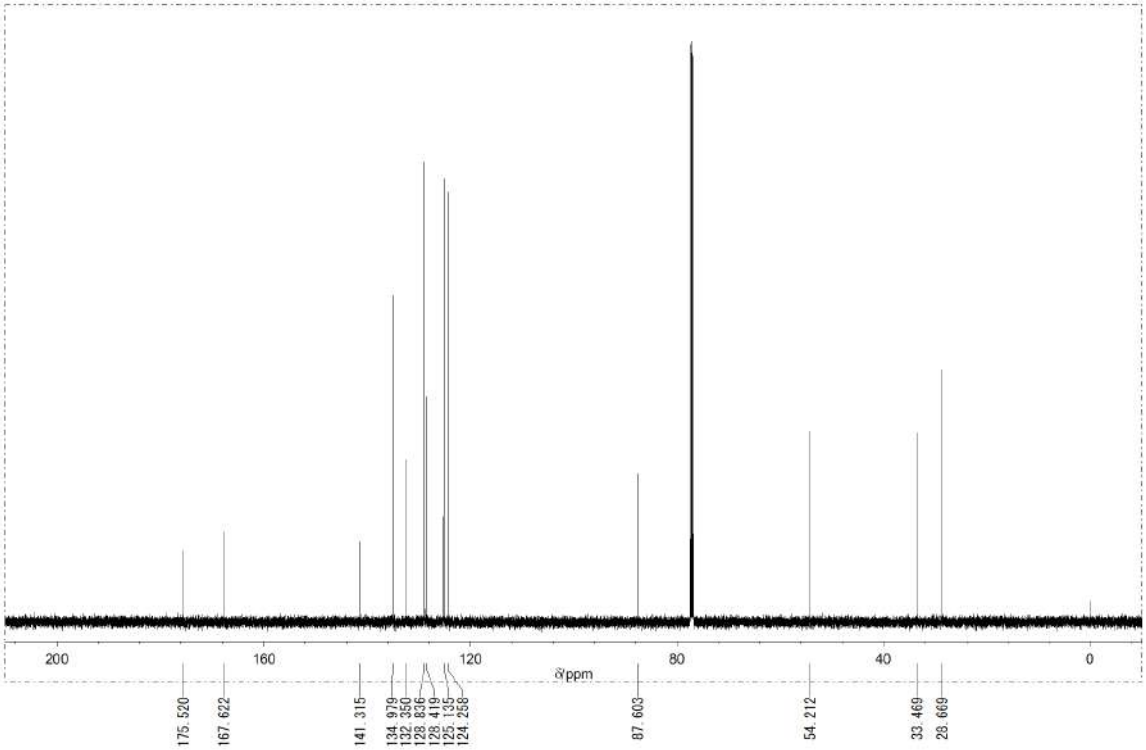




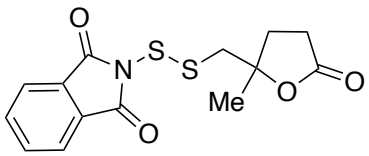


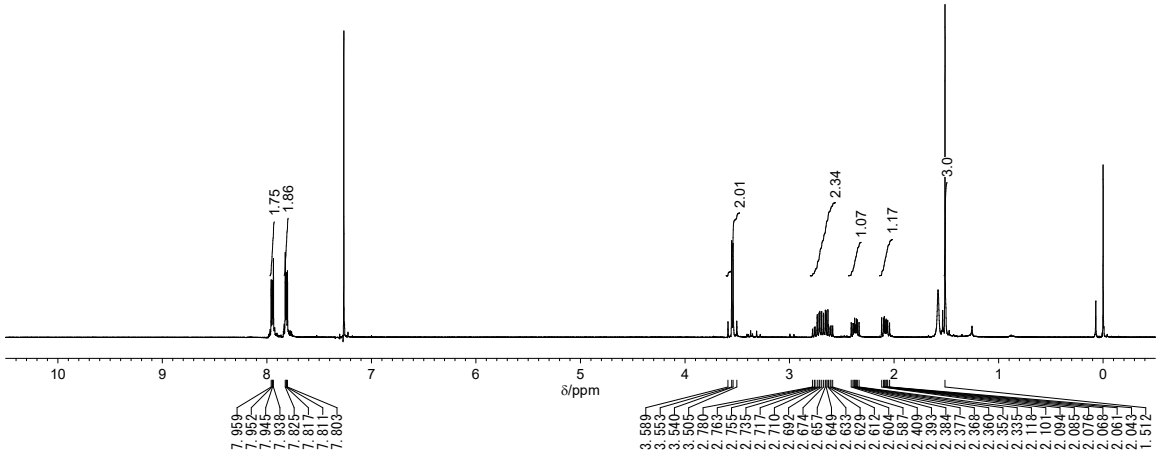
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-(((5-oxo-2-phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5a**) ($CDCl_3$)

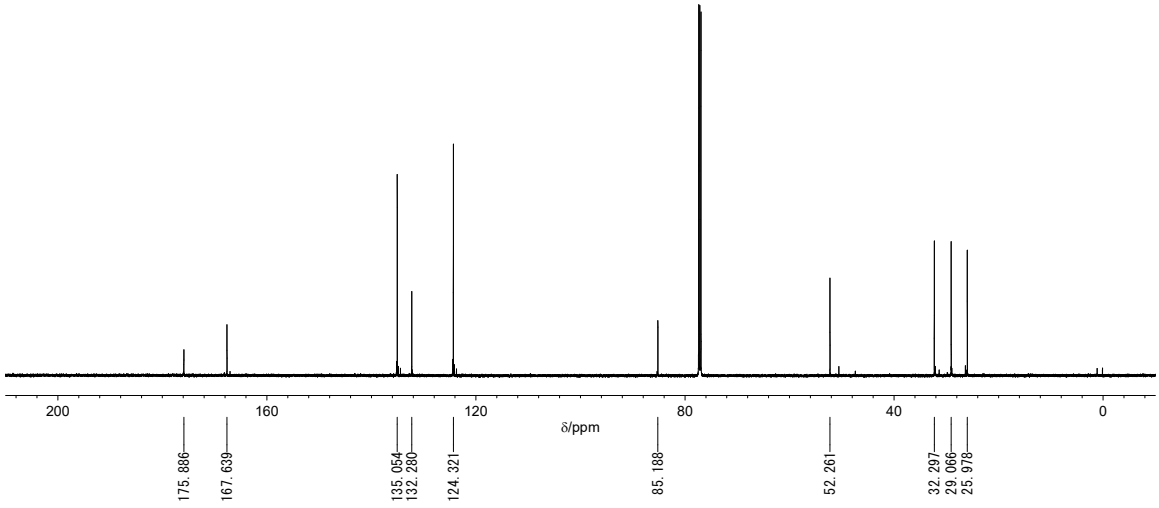




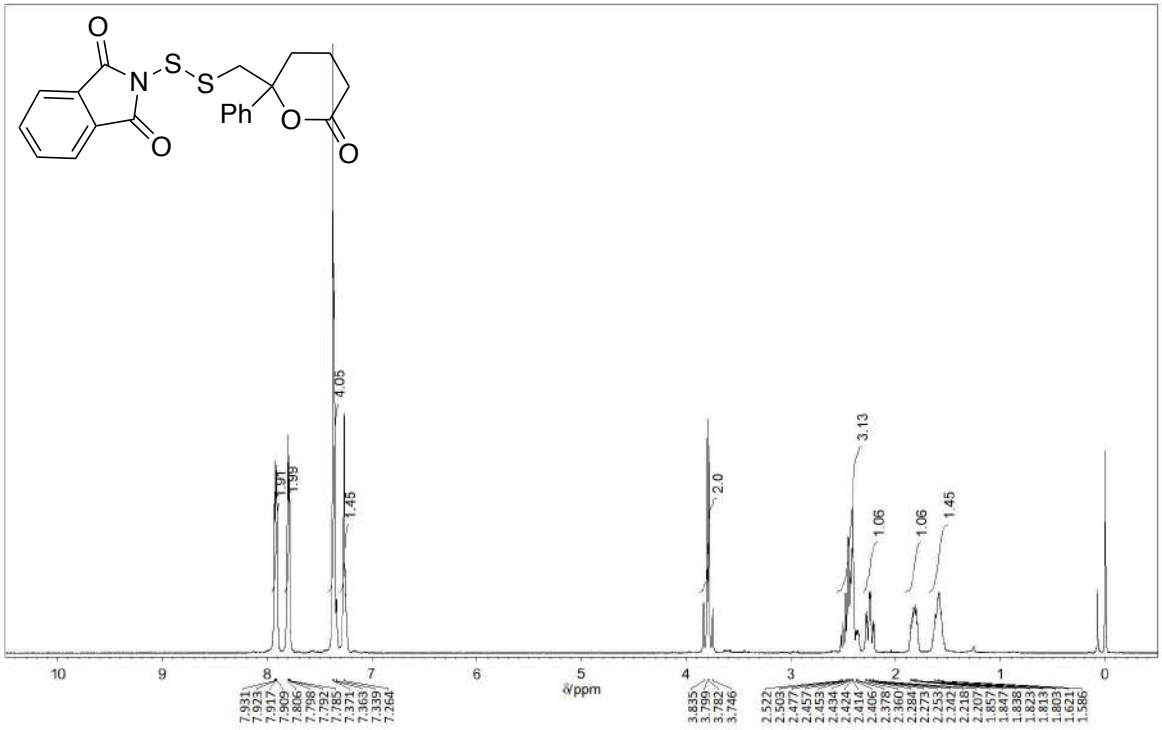
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-(((2-methyl-5-oxotetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5b**) ($CDCl_3$)

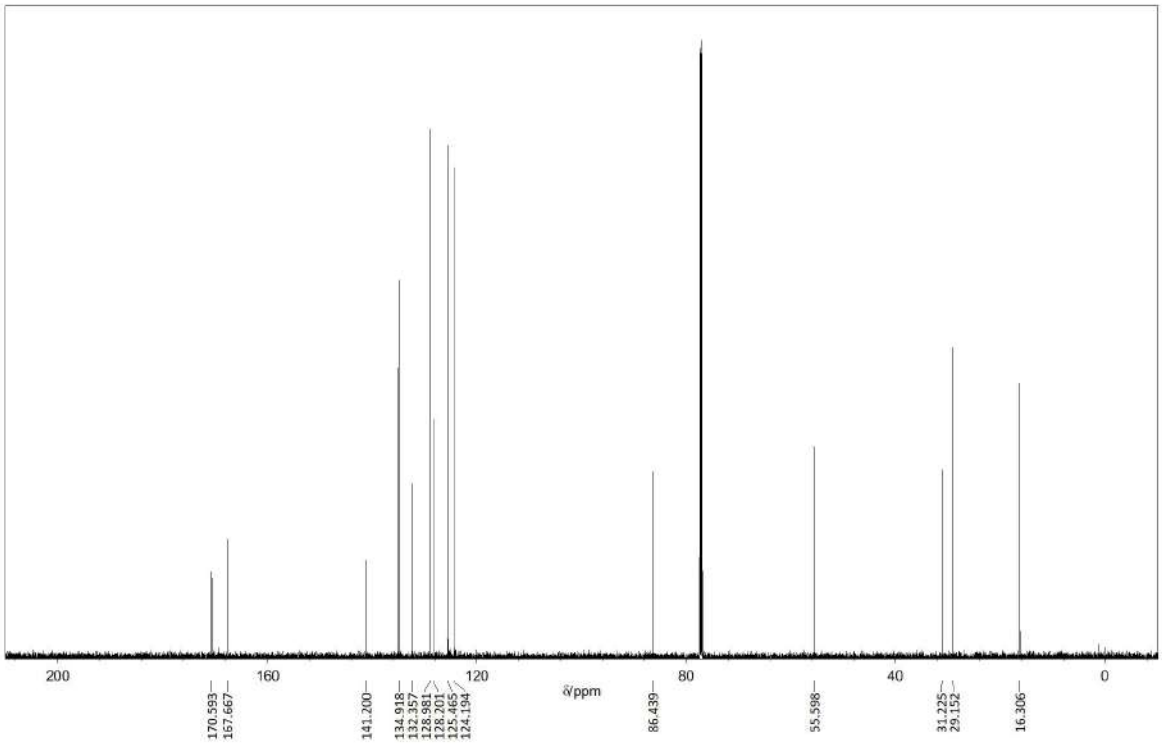




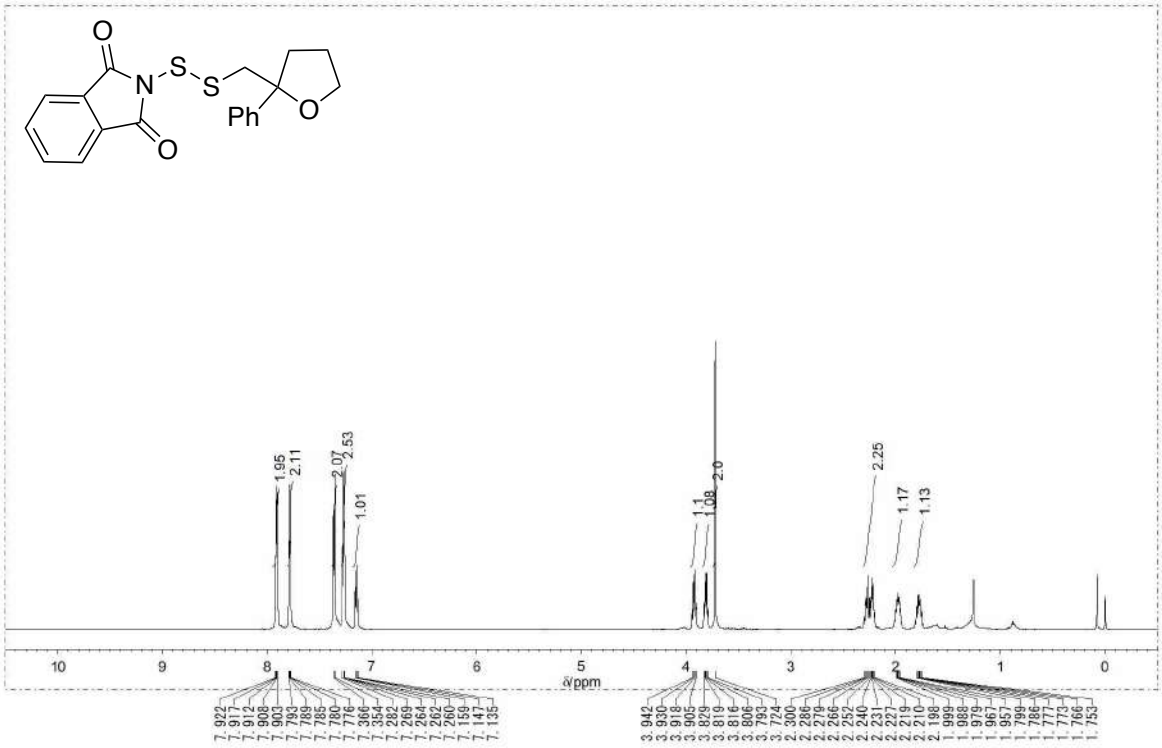


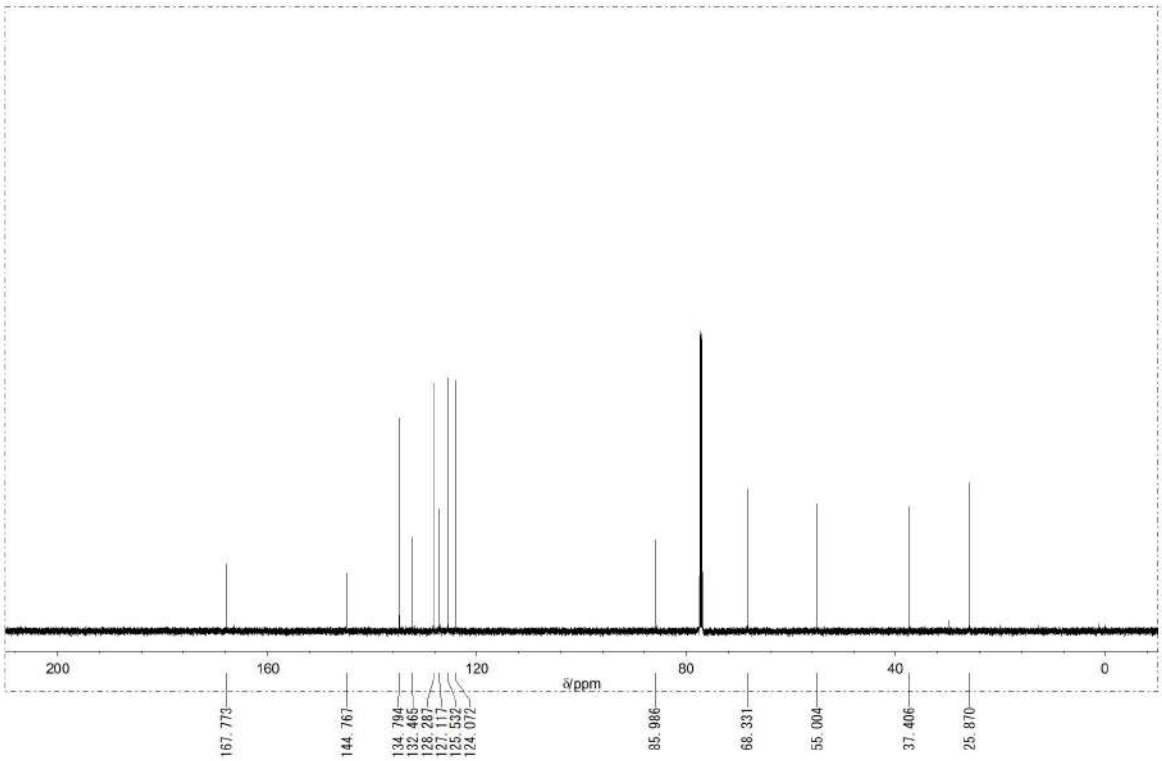
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-(((6-oxo-2-phenyltetrahydro-2*H*-pyran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5c**) ($CDCl_3$)



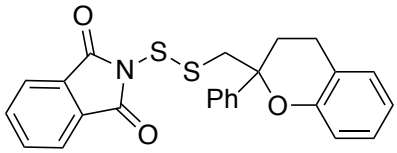


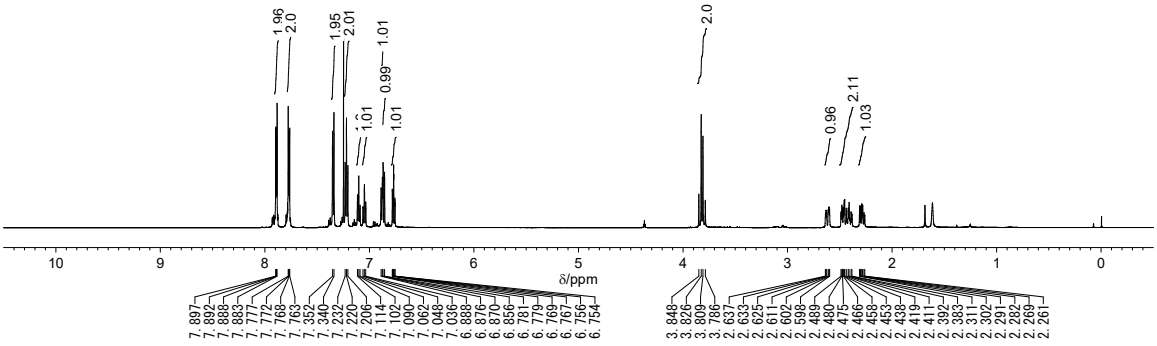
^{1}H NMR (600 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-(((2-phenyltetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5d**) ($CDCl_3$)

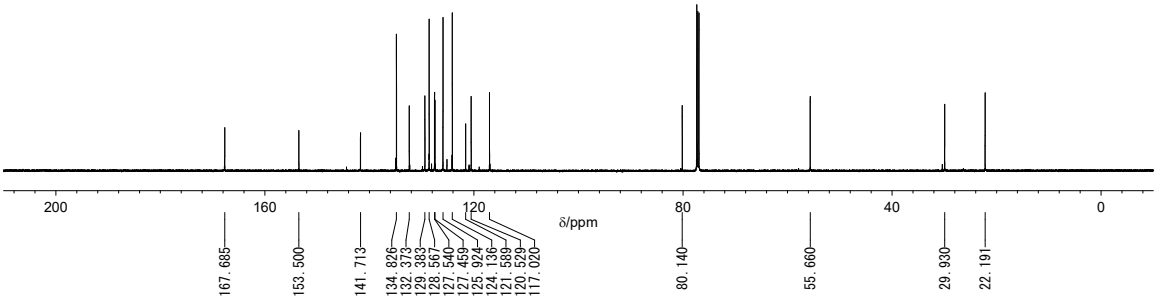




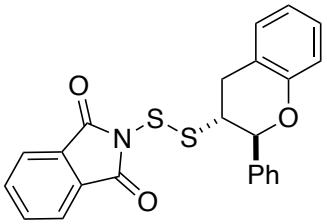
^{1}H NMR (600 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-(((2-phenylchroman-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5e**) ($CDCl_3$)

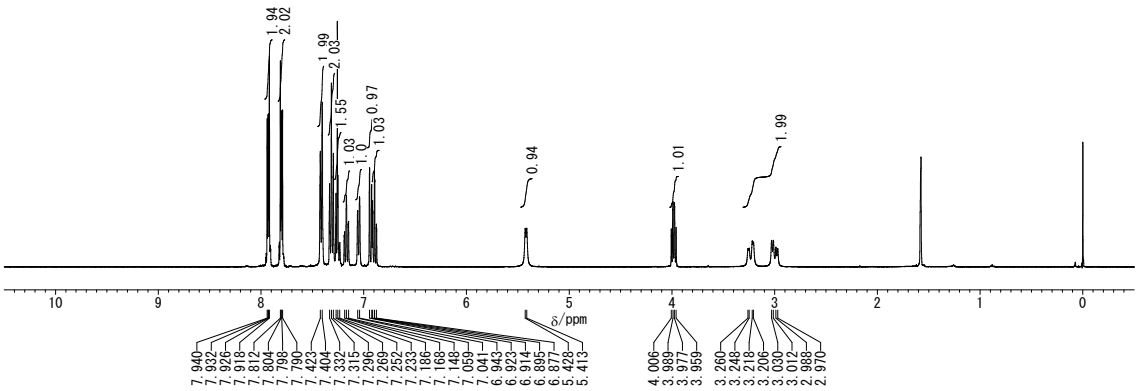


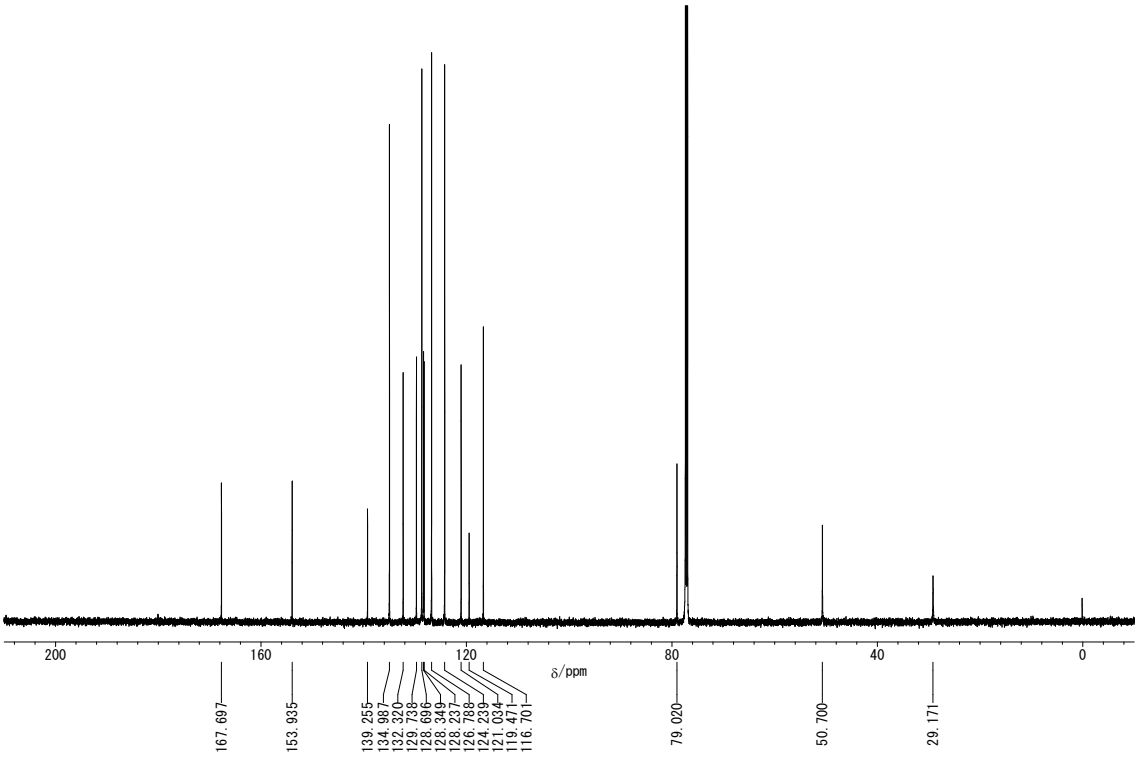




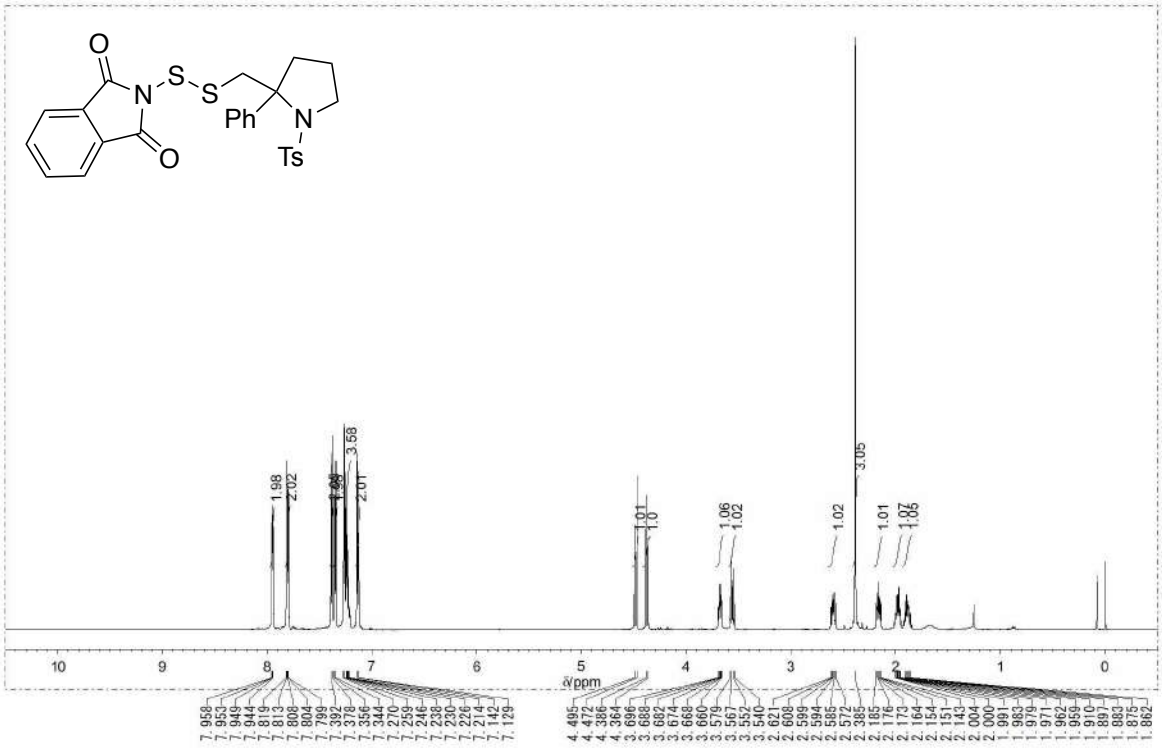
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-(((2*S**,3*R**)-2-phenylchroman-3-yl)disulfanyl)isoindoline-1,3-dione (**5f**) ($CDCl_3$)

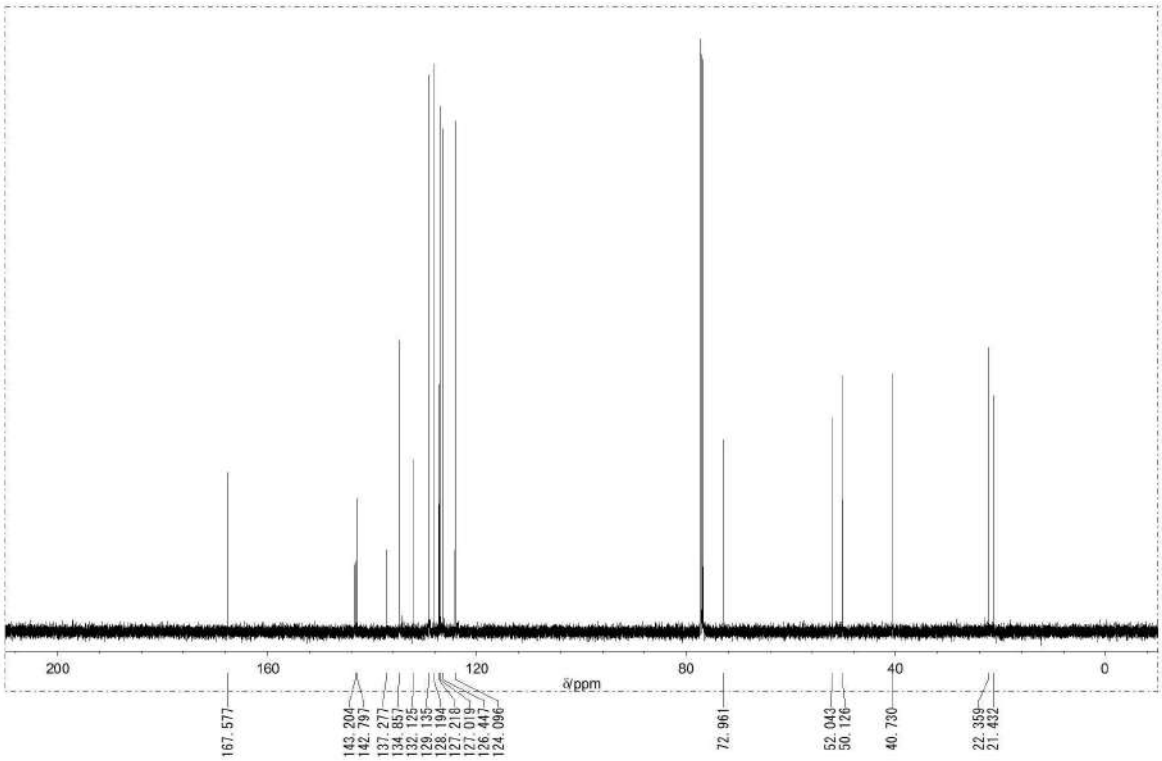




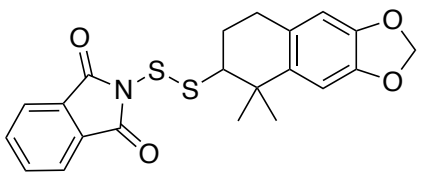


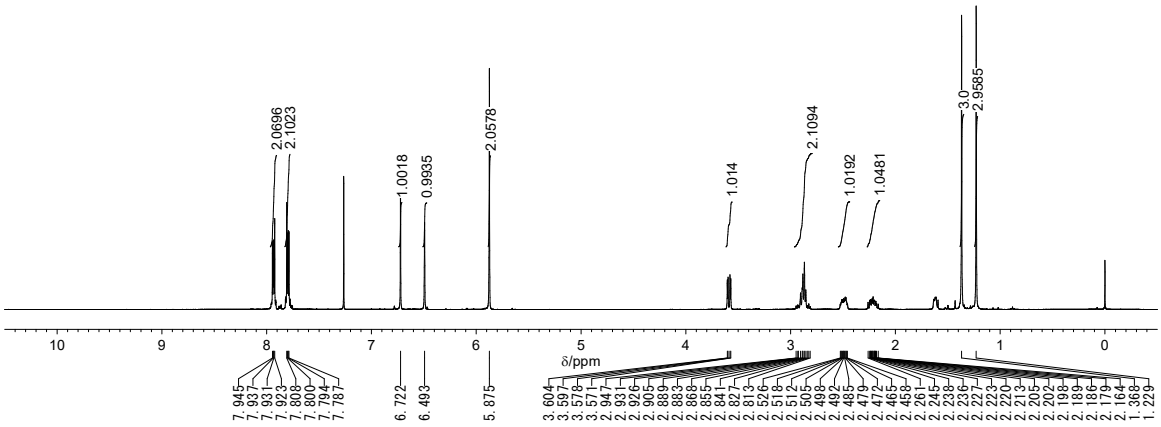
^{1}H NMR (600 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-(((2-phenyl-1-tosylpyrrolidin-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5g**) ($CDCl_3$)

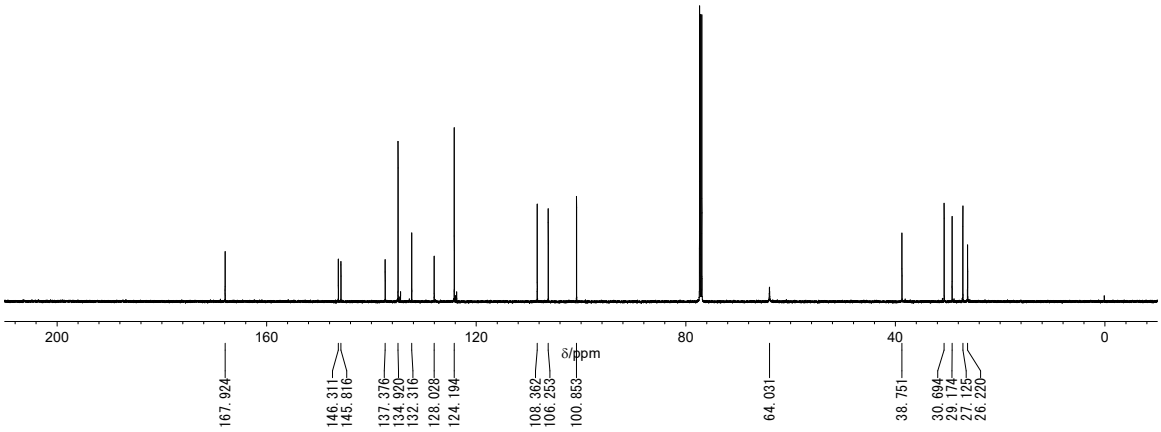




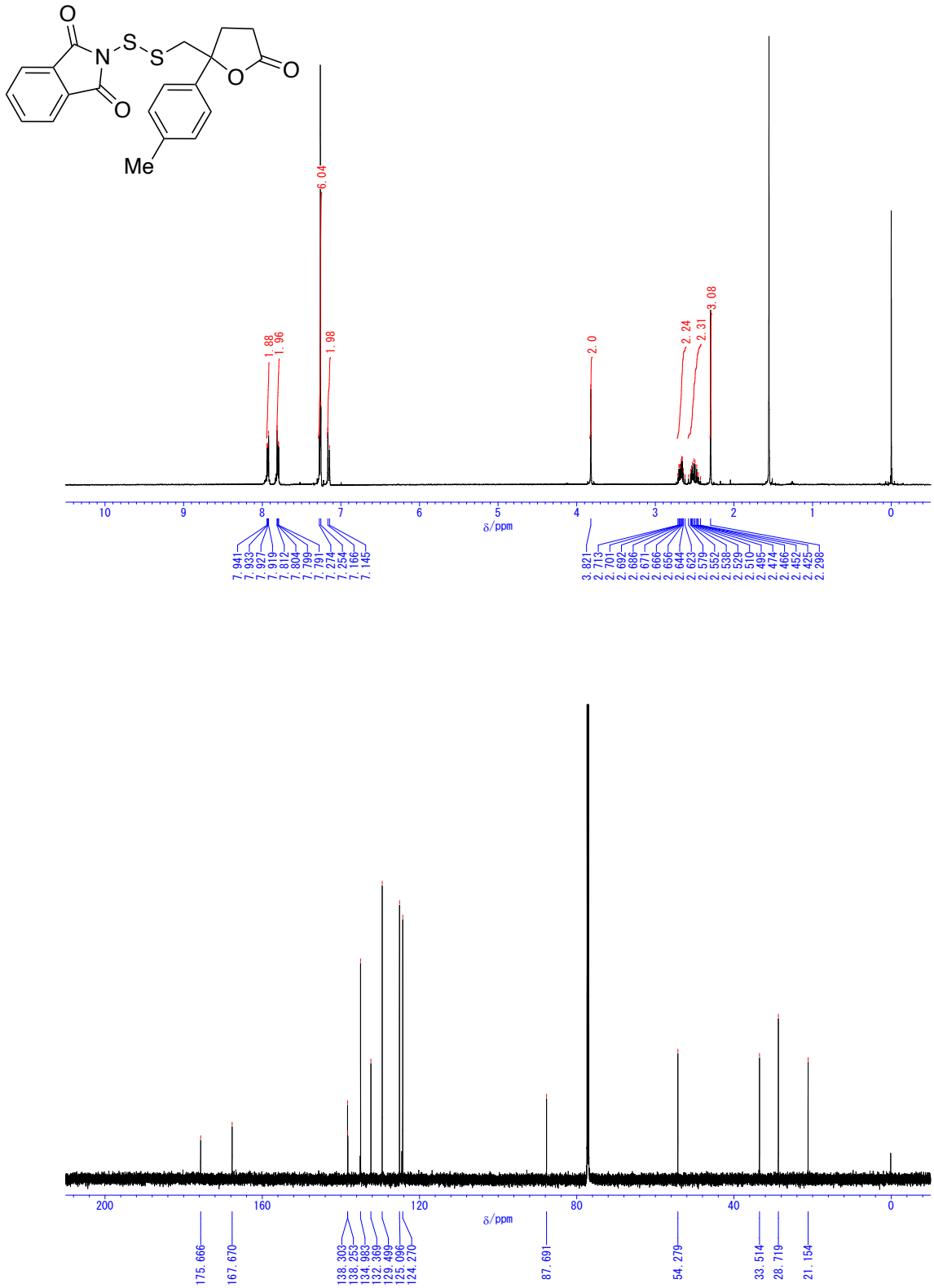
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((5,5-dimethyl-5,6,7,8-tetrahydronaphtho[2,3-*d*][1,3]dioxol-6-yl)disulfanyl)isoindoline-1,3-dione (**5h**) ($CDCl_3$)



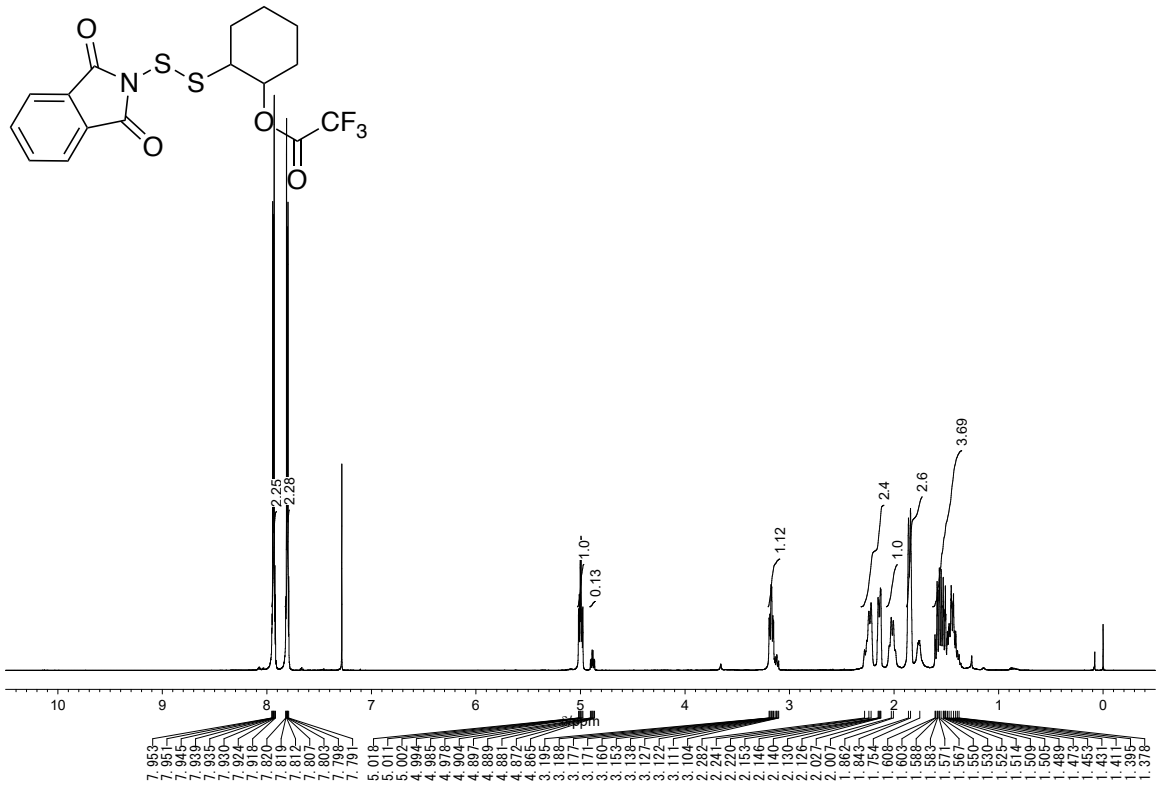


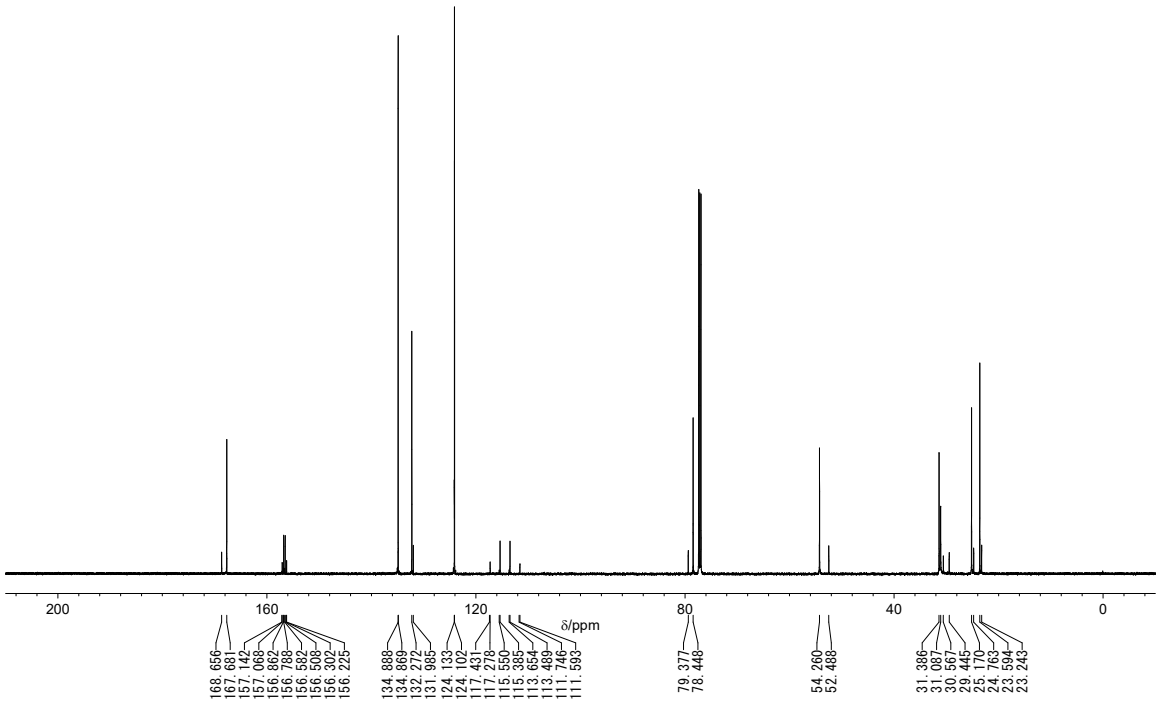


^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-(((5-oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)isoindoline-1,3-dione (**5i**) ($CDCl_3$)

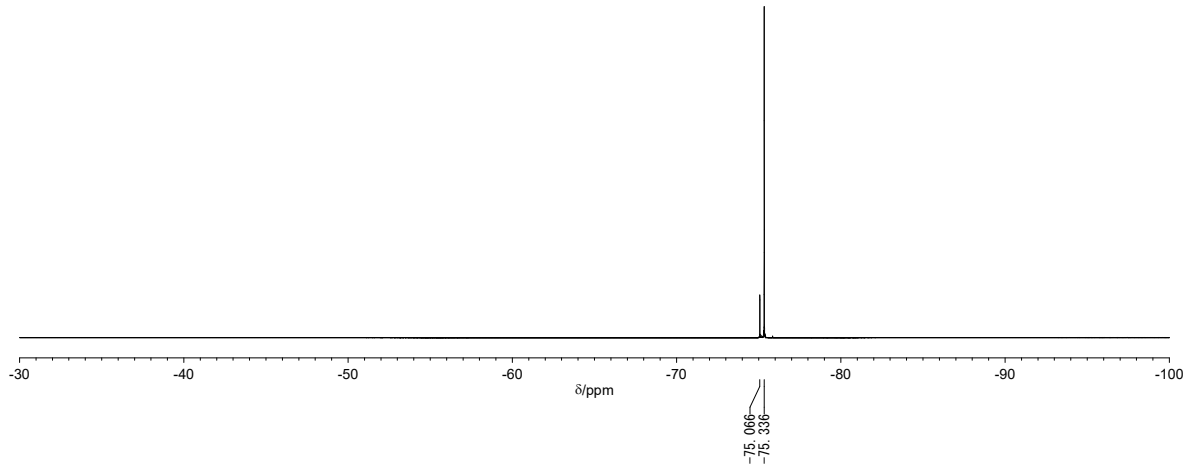


^{1}H NMR (600 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)cyclohexyl 2,2,2-trifluoroacetate (**7a**) ($CDCl_3$)

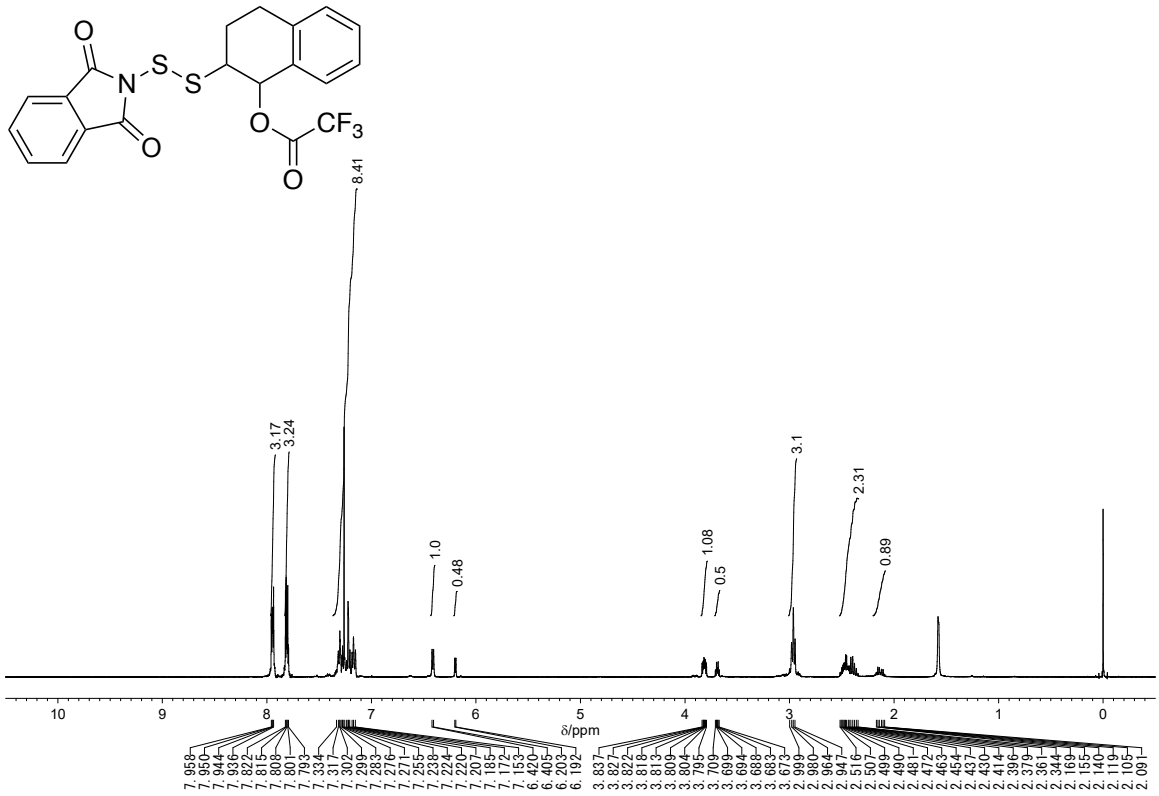


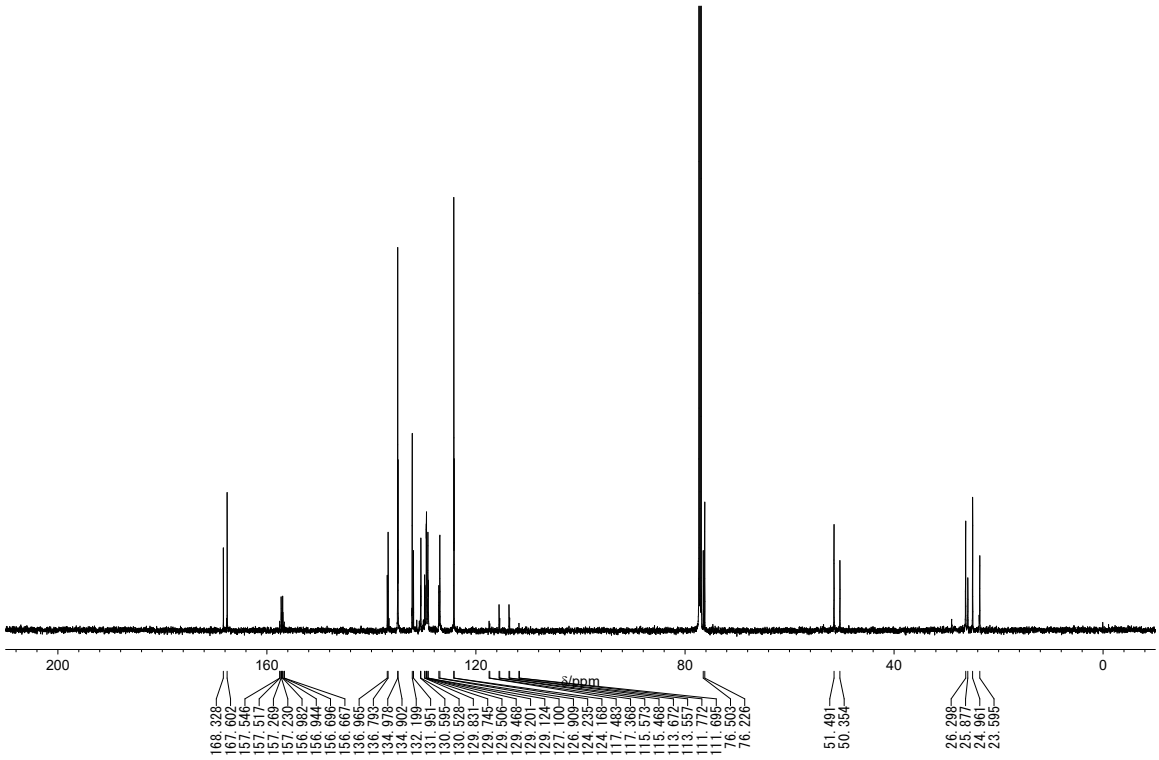


^{19}F NMR (376 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)cyclohexyl 2,2,2-trifluoroacetate (**7a**) ($CDCl_3$)

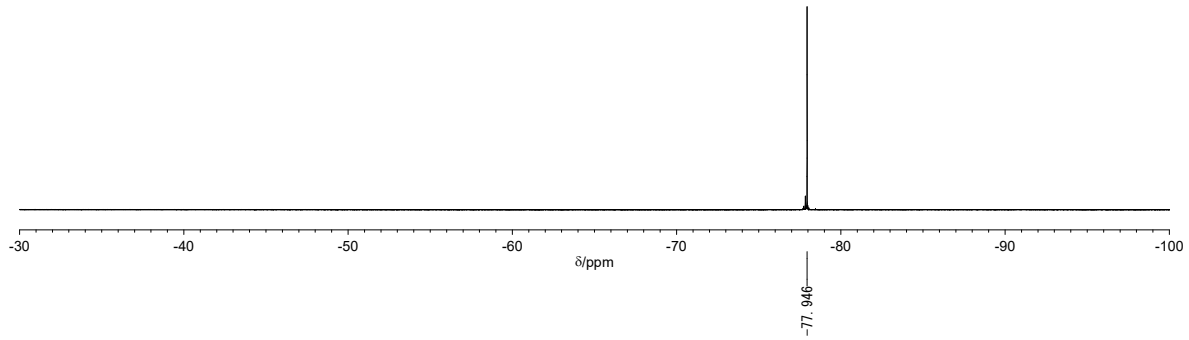


^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)-1,2,3,4-tetrahydronaphthalen-1-yl 2,2,2-trifluoroacetate (**7b**) ($CDCl_3$)

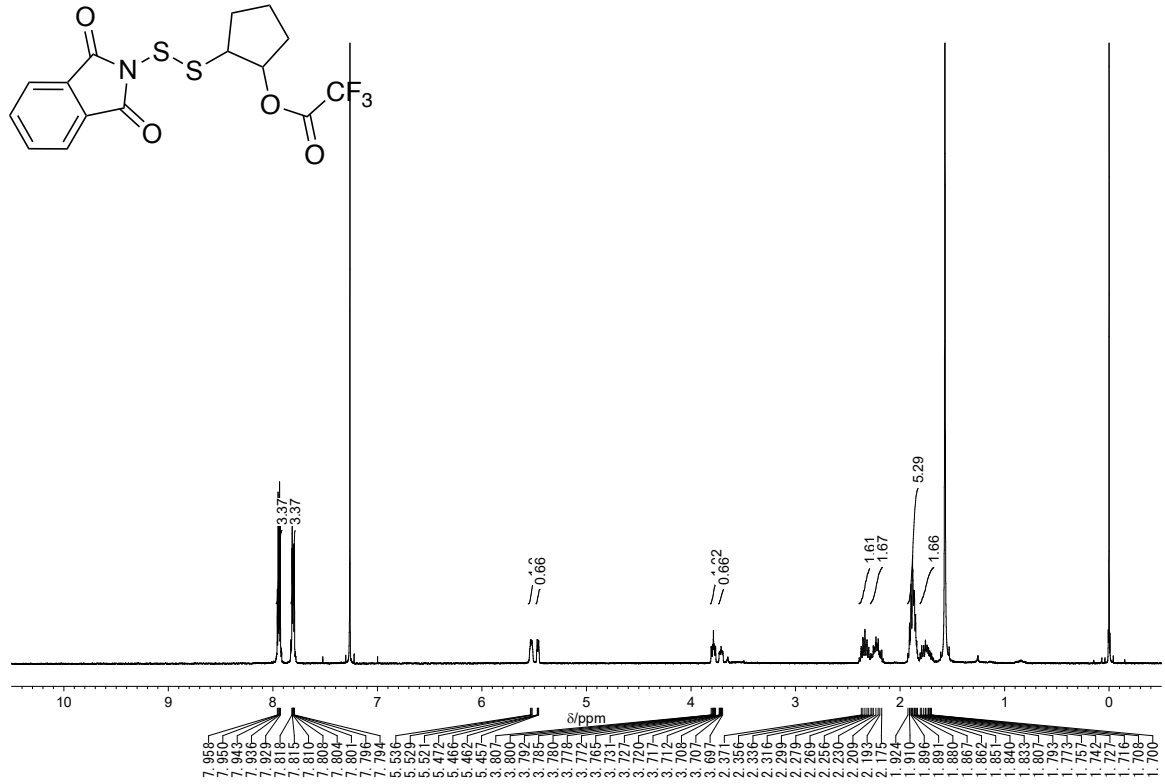


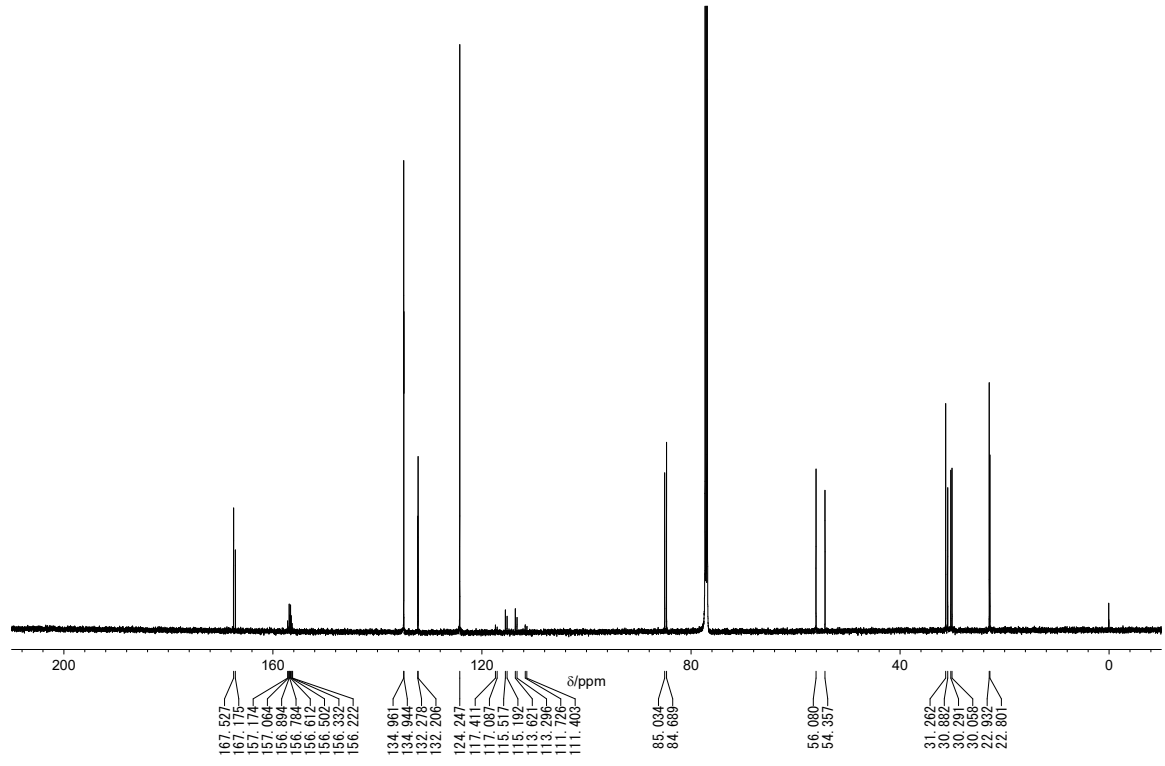


^{19}F NMR (376 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)-1,2,3,4-tetrahydronaphthalen-1-yl 2,2,2-trifluoroacetate (**7b**) ($CDCl_3$)

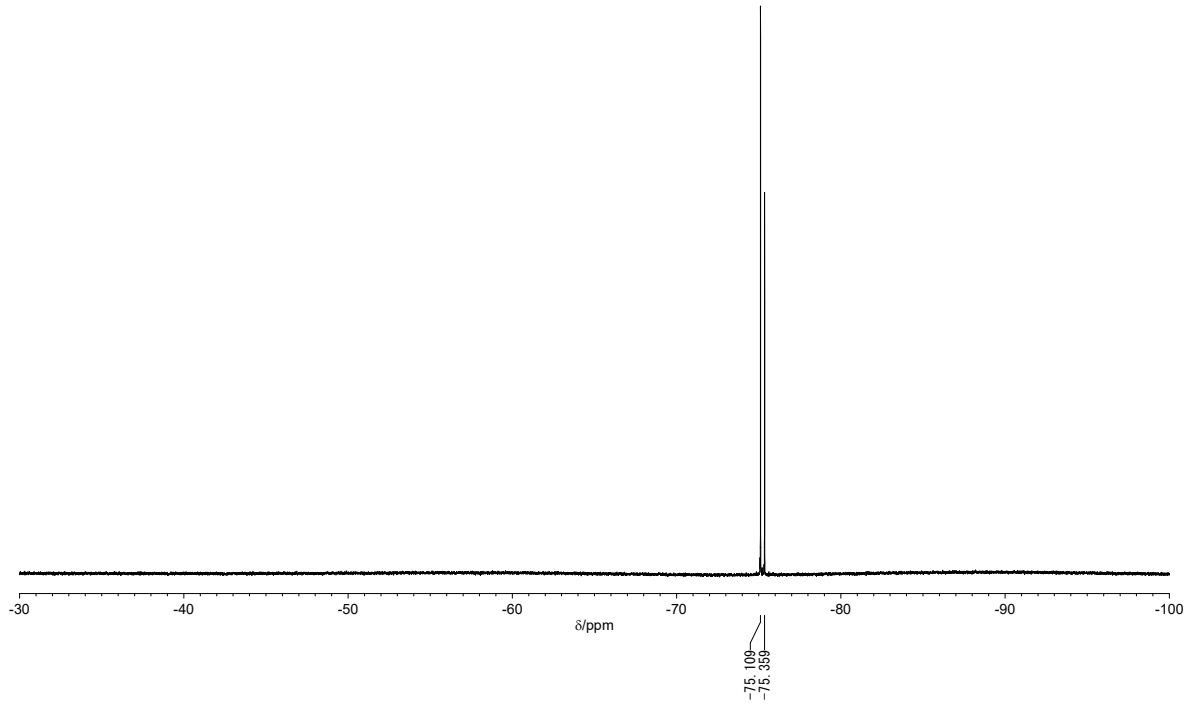


^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfaneyl)cyclopentyl 2,2,2-trifluoroacetate (**7c**) ($CDCl_3$)

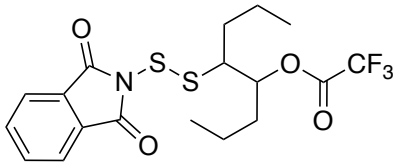


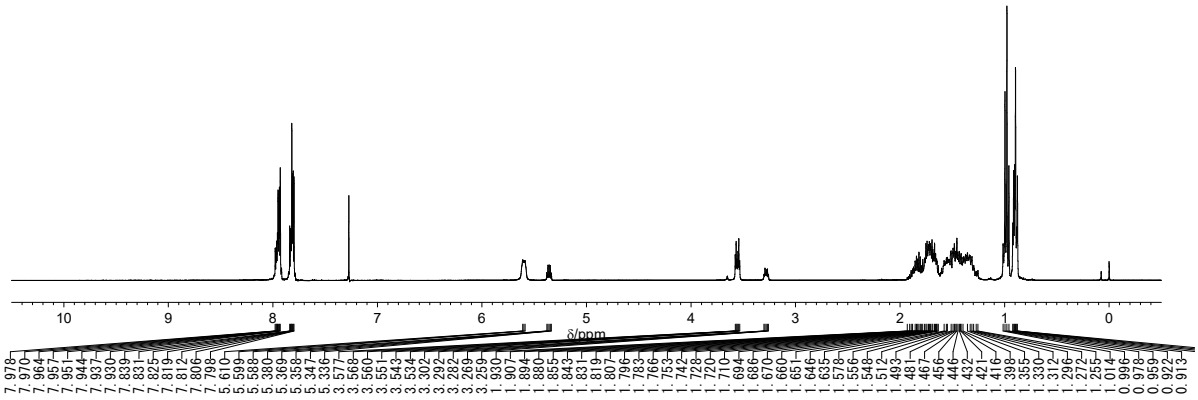


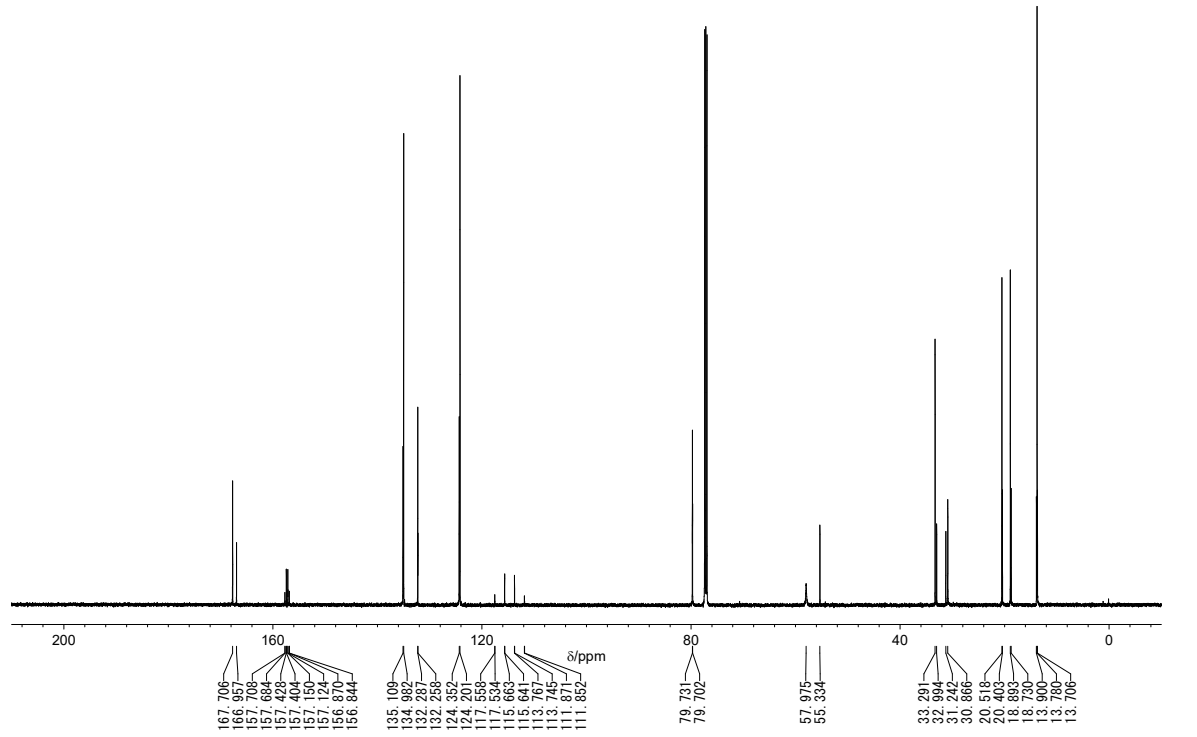
^{19}F NMR (376 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfaneyl)cyclopentyl 2,2,2-trifluoroacetate (**7c**) ($CDCl_3$)



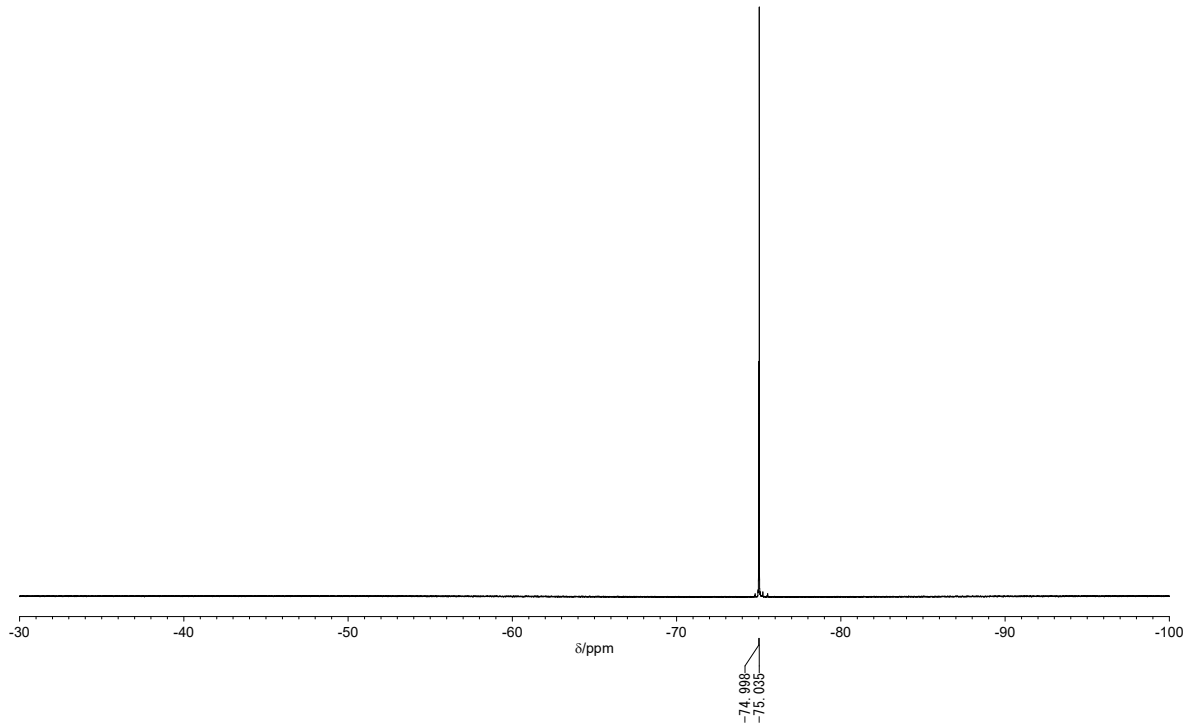
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 5-((1,3-dioxoisoindolin-2-yl)disulfanyl)octan-4-yl 2,2,2-trifluoroacetate (**7d**) ($CDCl_3$)



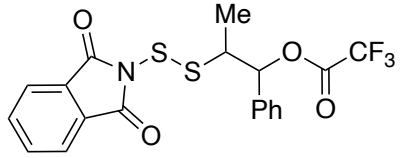


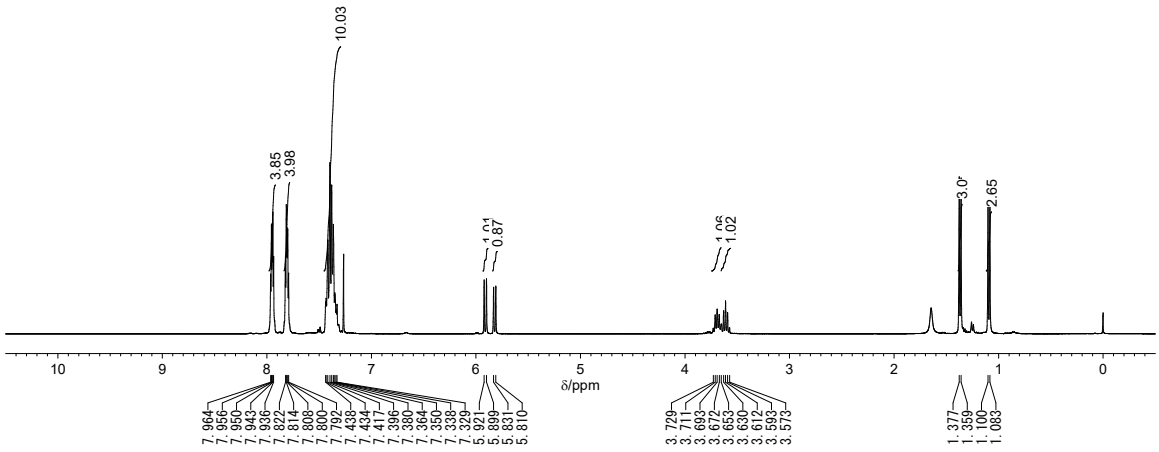


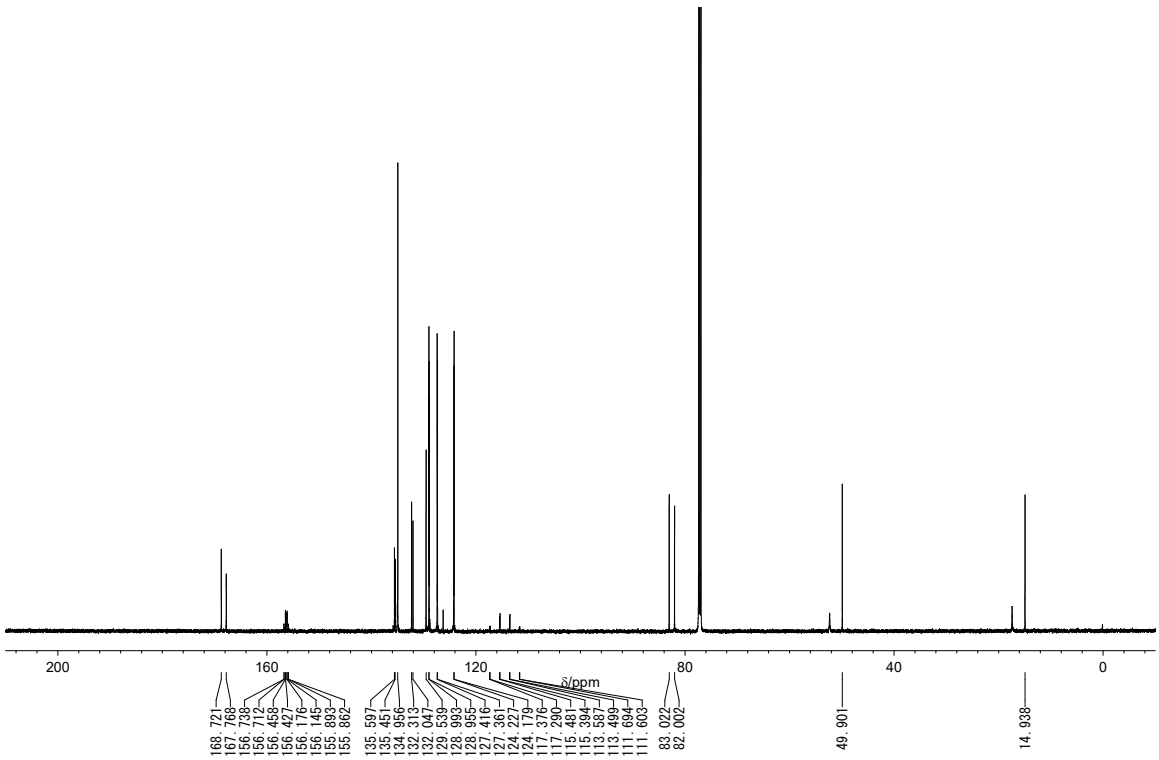
^{19}F NMR (376 MHz) spectra of 5-((1,3-dioxoisoindolin-2-yl)disulfanyl)octan-4-yl 2,2,2-trifluoroacetate (**7d**) ($CDCl_3$)



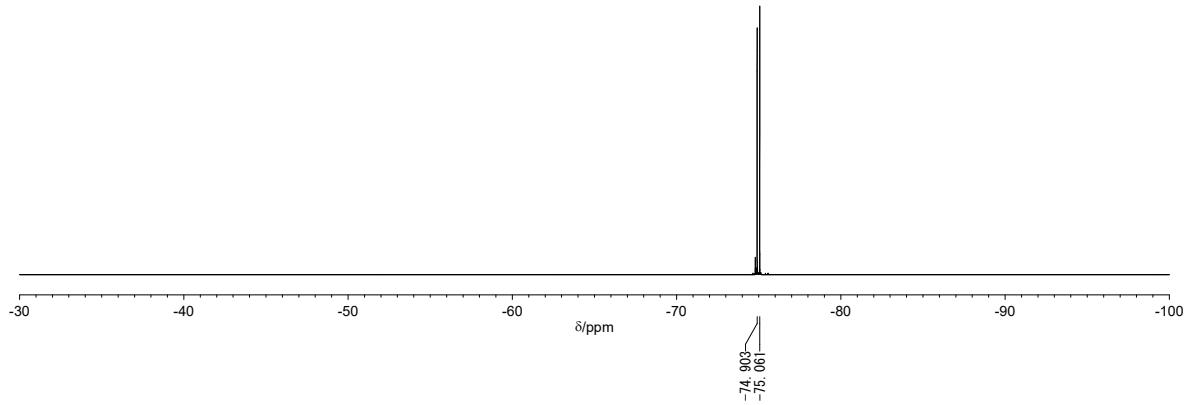
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)-1-phenylpropyl 2,2,2-trifluoroacetate (**7e**) ($CDCl_3$)



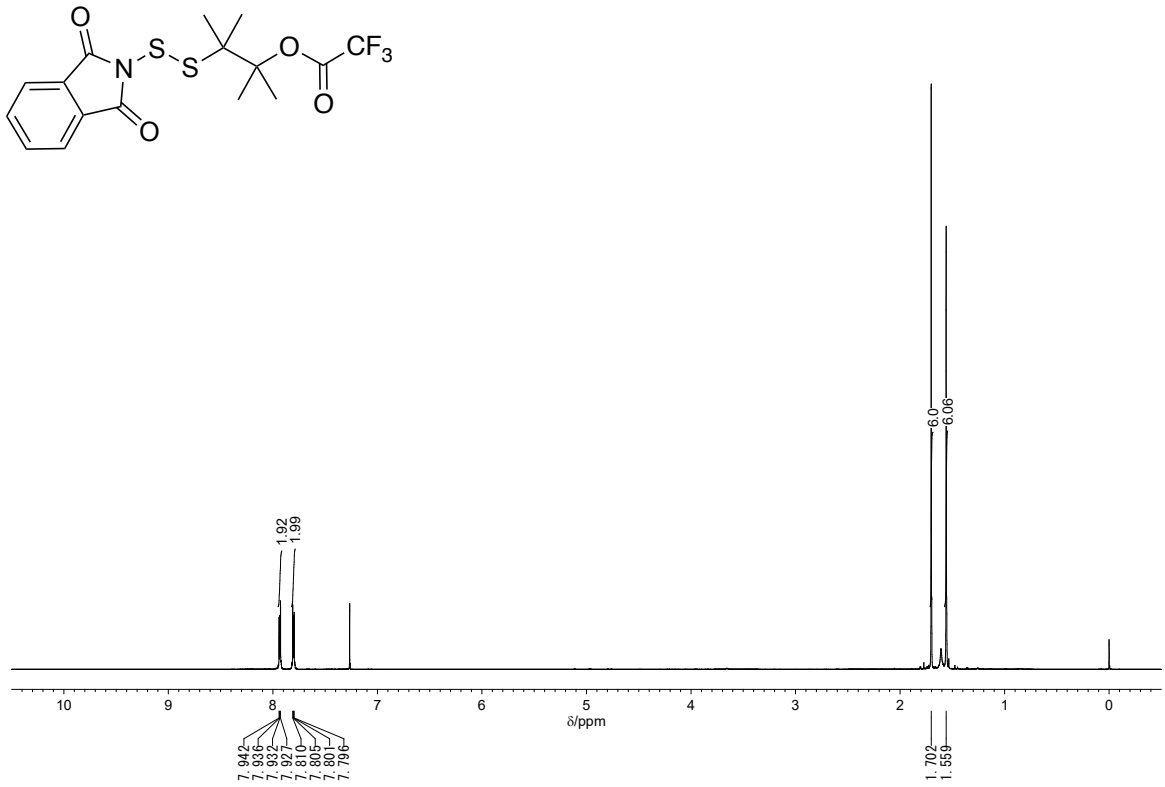


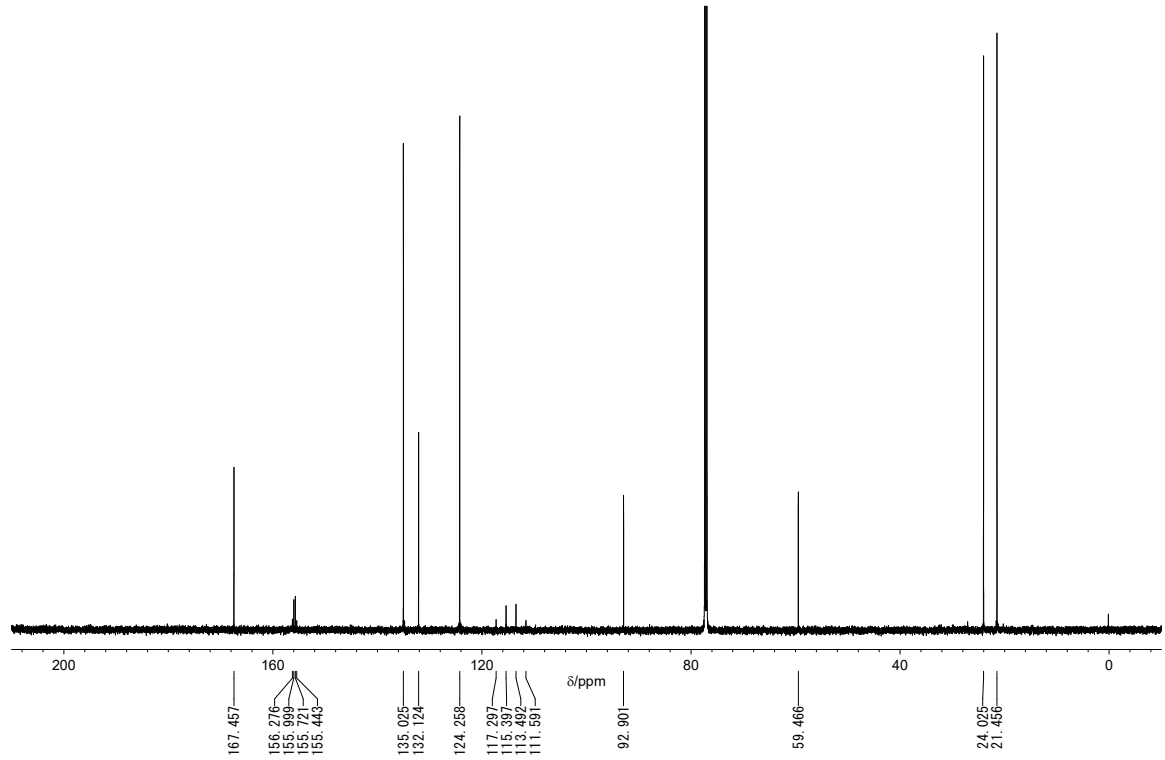


^{19}F NMR (376 MHz) spectra of 2-((1,3-dioxoisoindolin-2-yl)disulfanyl)-1-phenylpropyl 2,2,2-trifluoroacetate (**7e**) ($CDCl_3$)

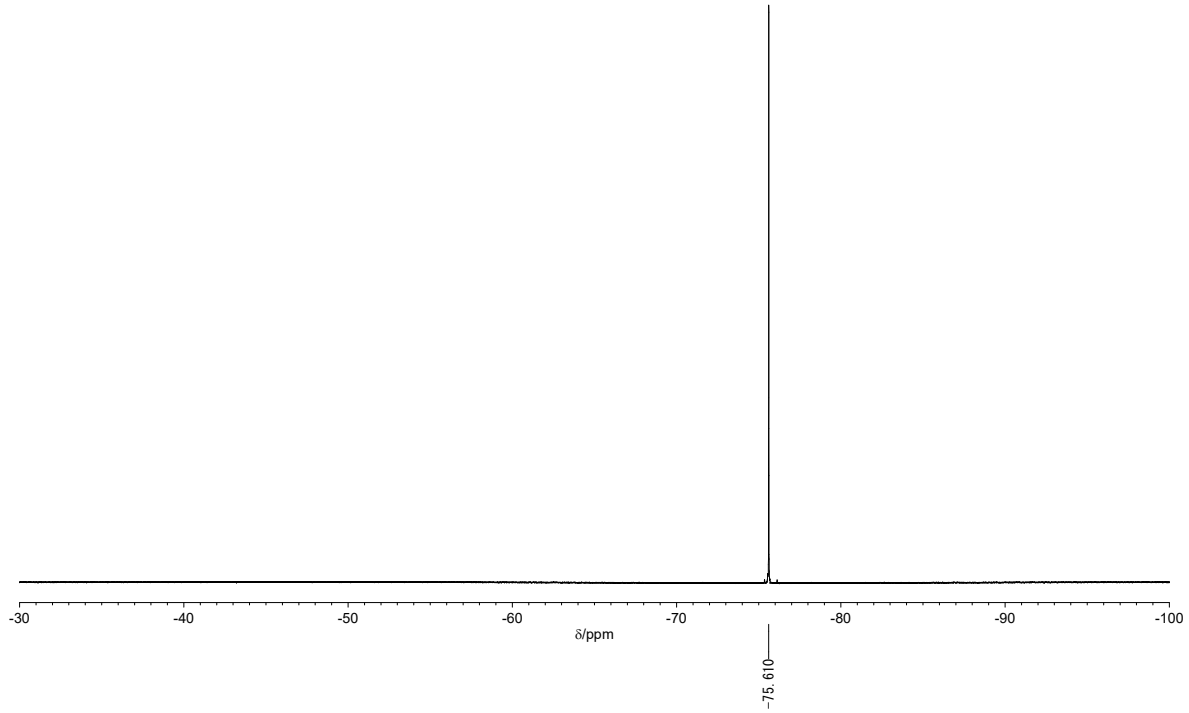


^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 3-((1,3-dioxoisoindolin-2-yl)disulfanyl)-2,3-dimethylbutan-2-yl 2,2,2-trifluoroacetate (**7f**) ($CDCl_3$)

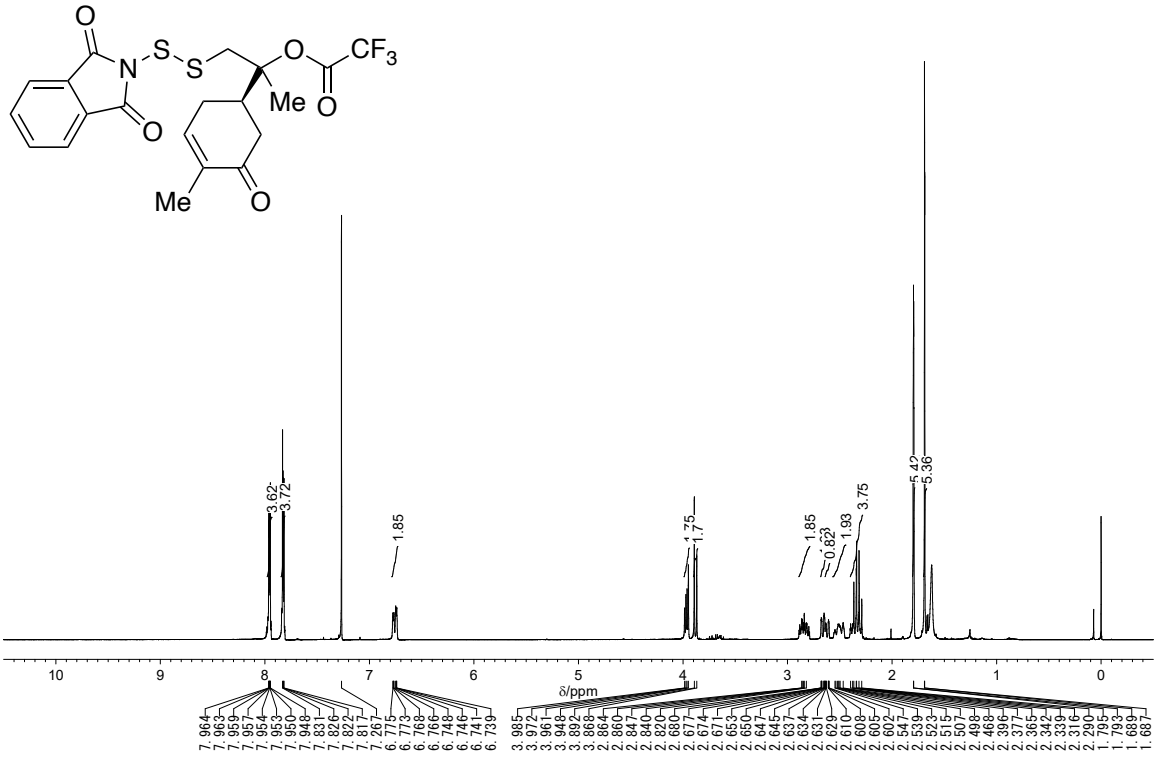


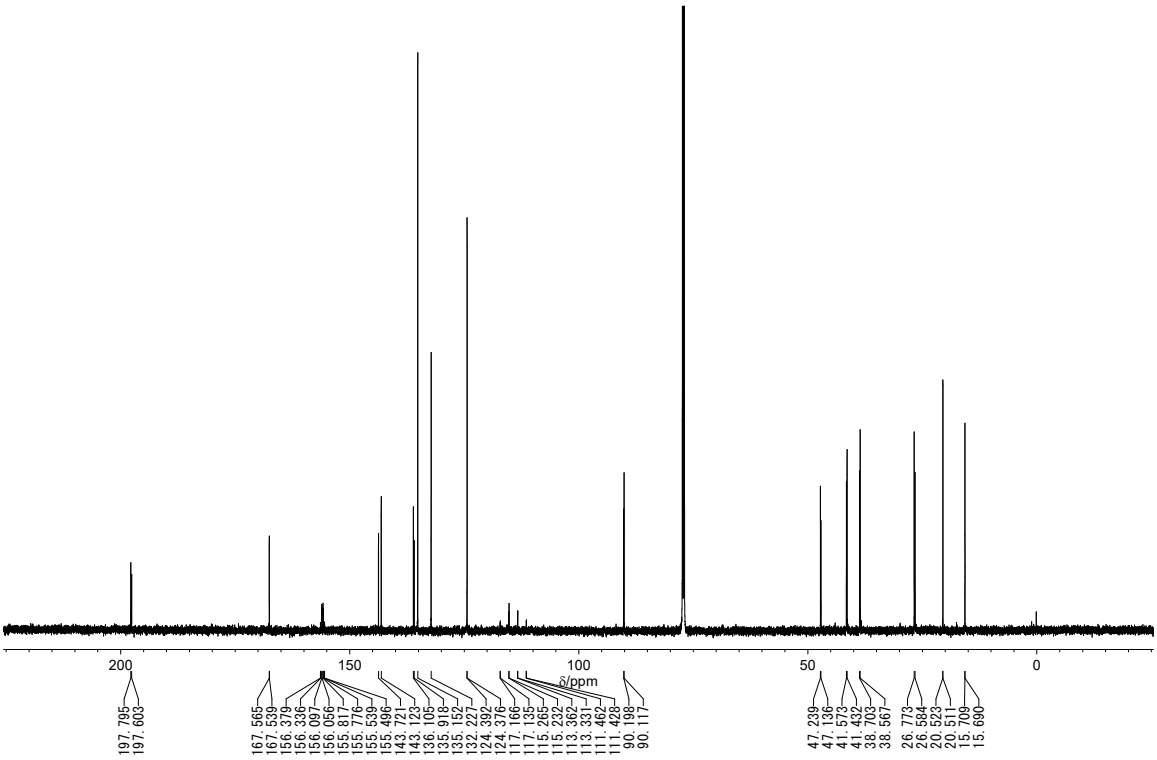


^{19}F NMR (376 MHz) spectra of 3-((1,3-dioxoisoindolin-2-yl)disulfanyl)-2,3-dimethylbutan-2-yl 2,2,2-trifluoroacetate (**7f**) ($CDCl_3$)

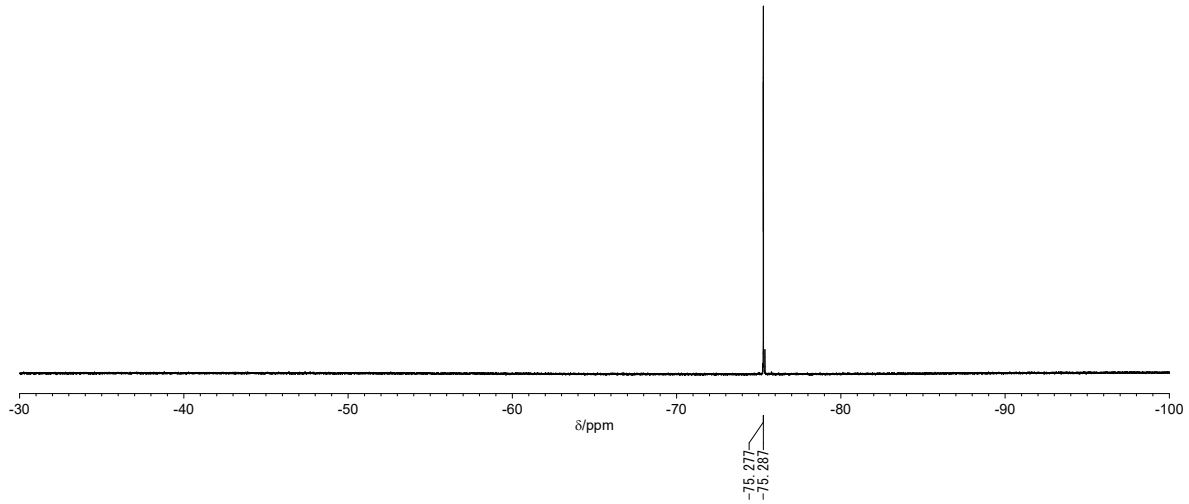


^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 1-((1,3-dioxoisoindolin-2-yl)disulfanyl)-2-(4-methyl-5-oxocyclohex-3-en-1-yl)propan-2-yl 2,2,2-trifluoroacetate (**7g**) ($CDCl_3$)

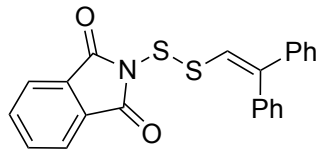


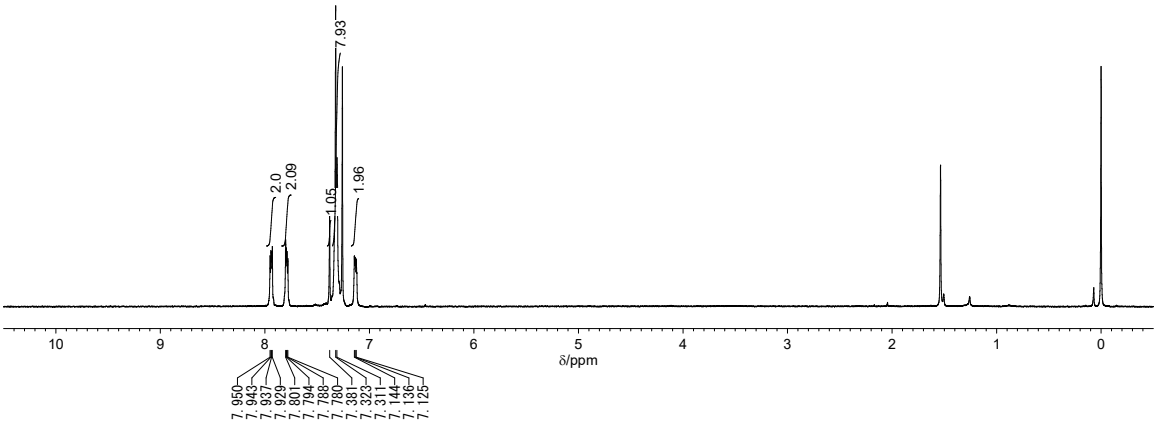


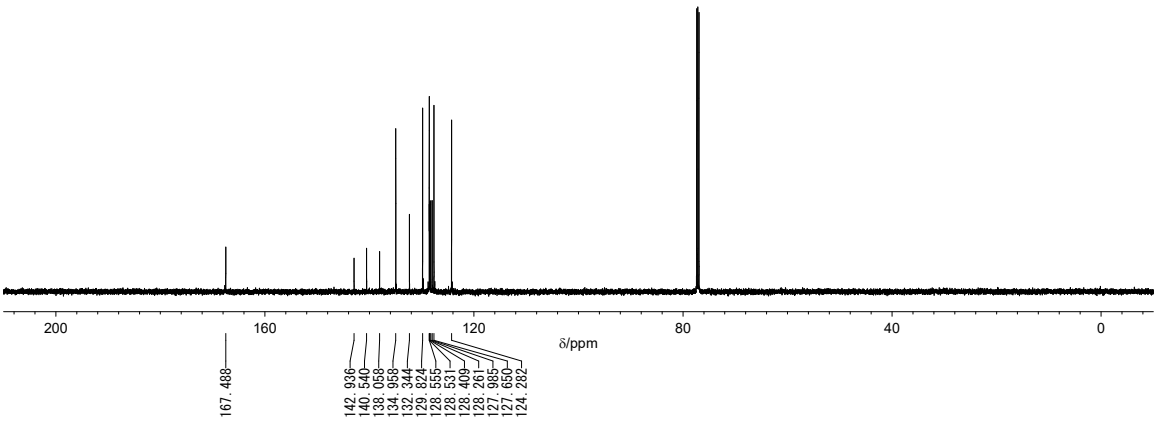
^{19}F NMR (376 MHz) spectra of 1-((1,3-dioxoisoindolin-2-yl)disulfanyl)-2-(4-methyl-5-oxocyclohex-3-en-1-yl)propan-2-yl 2,2,2-trifluoroacetate (**7g**) ($CDCl_3$)



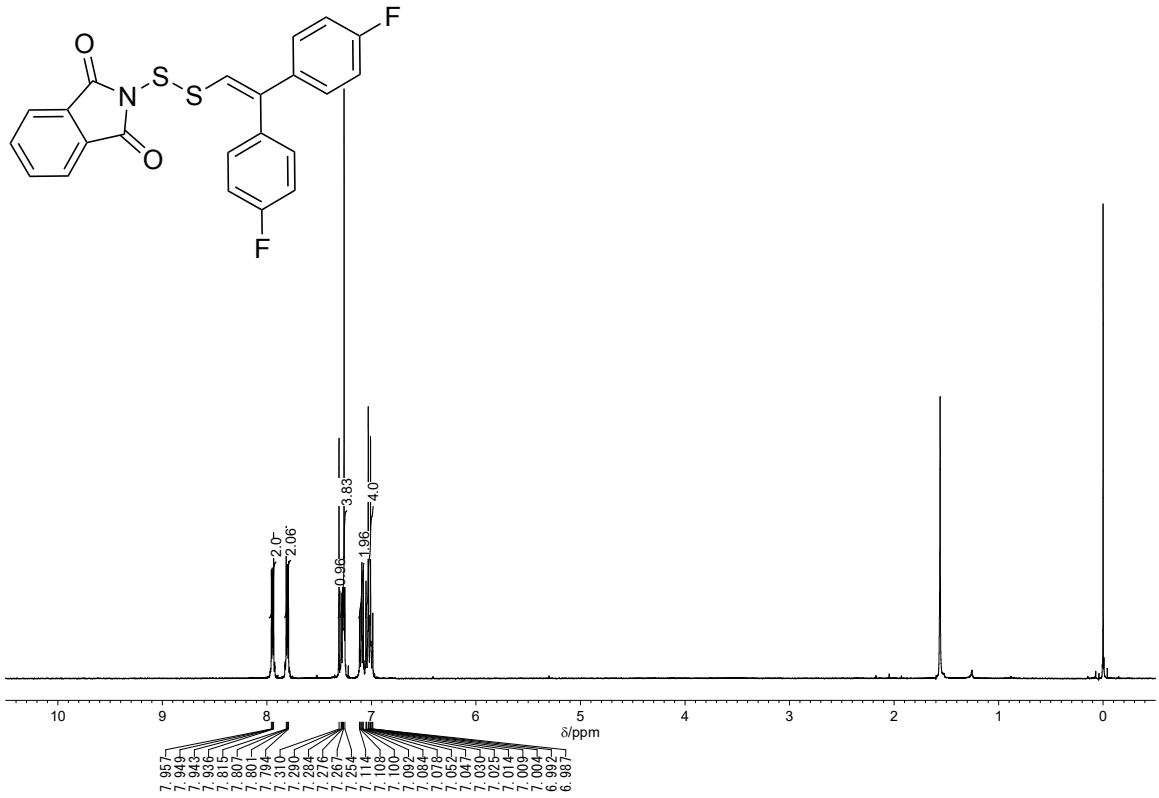
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2,2-diphenylvinyl)disulfanyl)isoindoline-1,3-dione (**9a**) ($CDCl_3$)

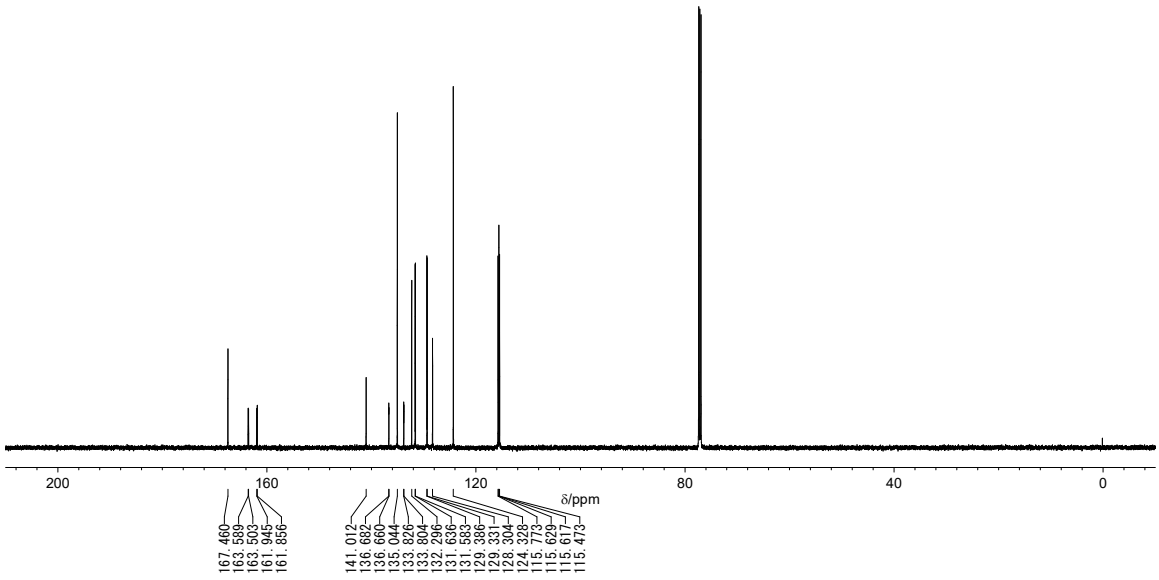




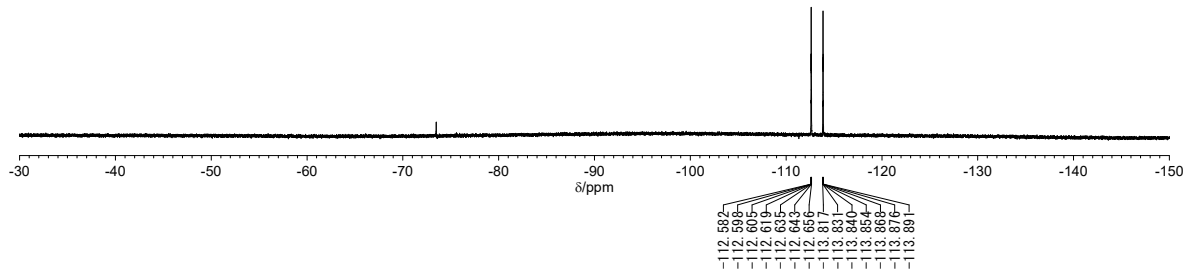


^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2,2-bis(4-fluorophenyl)vinyl)disulfanyl)isoindoline-1,3-dione (**9b**) ($CDCl_3$)

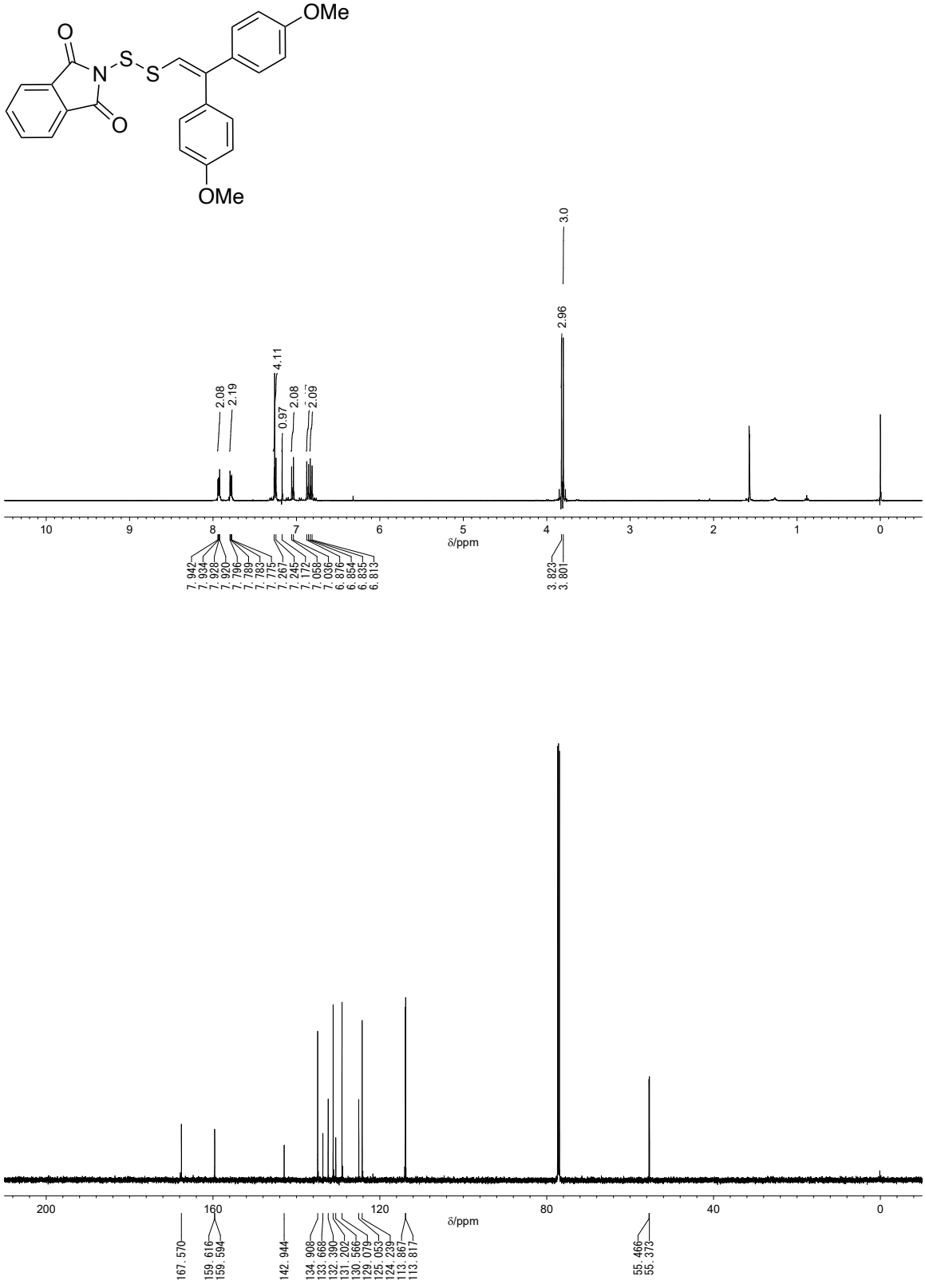




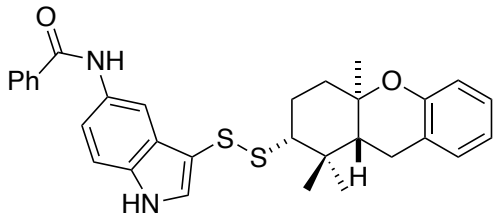
^{19}F NMR (376 MHz) spectra of 2-((2,2-bis(4-fluorophenyl)vinyl)disulfanyl)isoindoline-1,3-dione (**9b**) ($CDCl_3$)

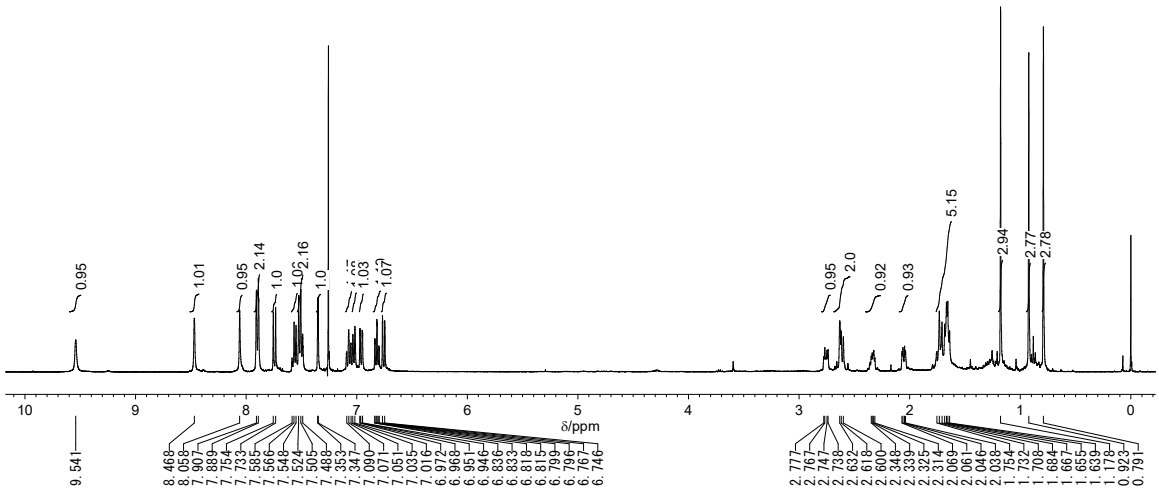


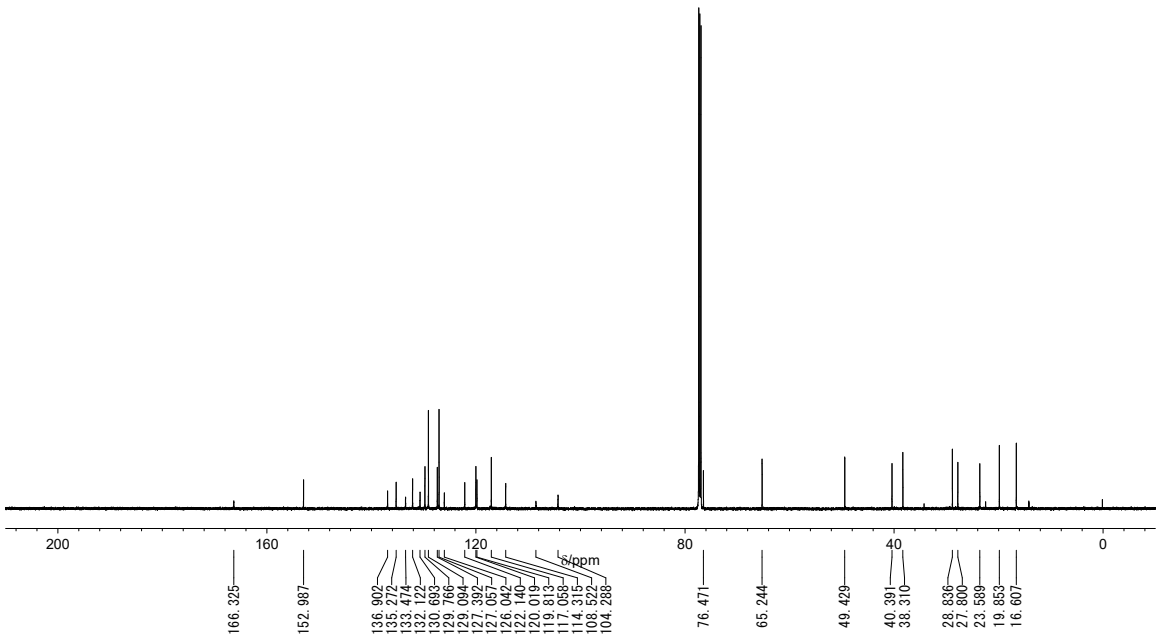
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 2-((2,2-bis(4-methoxyphenyl)vinyl)disulfanyl)isoindoline-1,3-dione (**9c**) ($CDCl_3$)



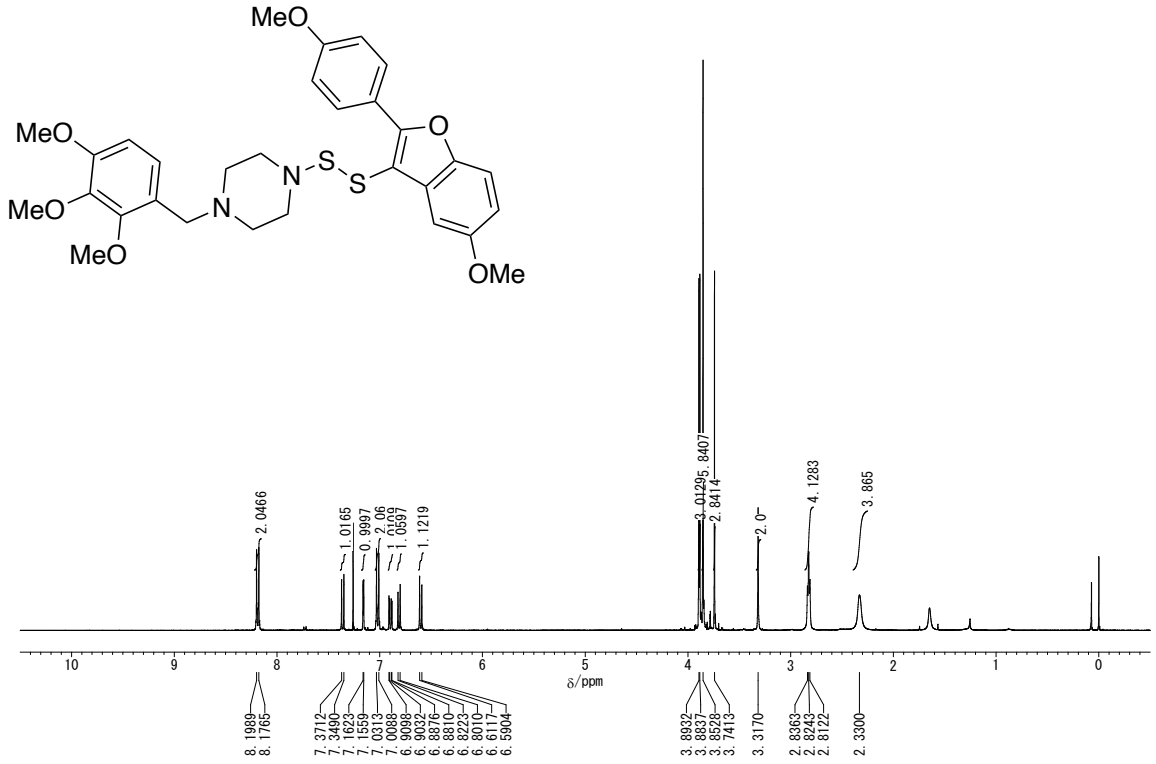
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of *N*-(3-(((2*R**,4a*R**,9a*R**)-1,1,4a-trimethyl-2,3,4,4a,9,9a-hexahydro-1*H*-xanthen-2-yl)disulfanyl)-1*H*-indol-5-yl)benzamide (**13**) ($CDCl_3$)

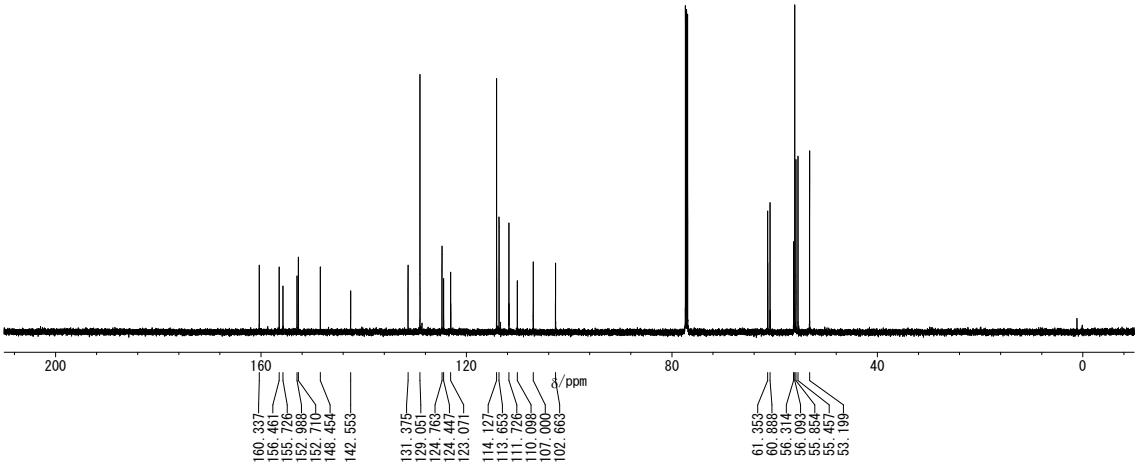




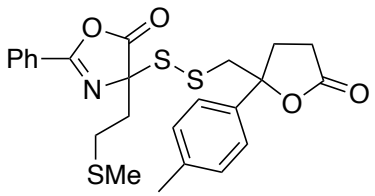


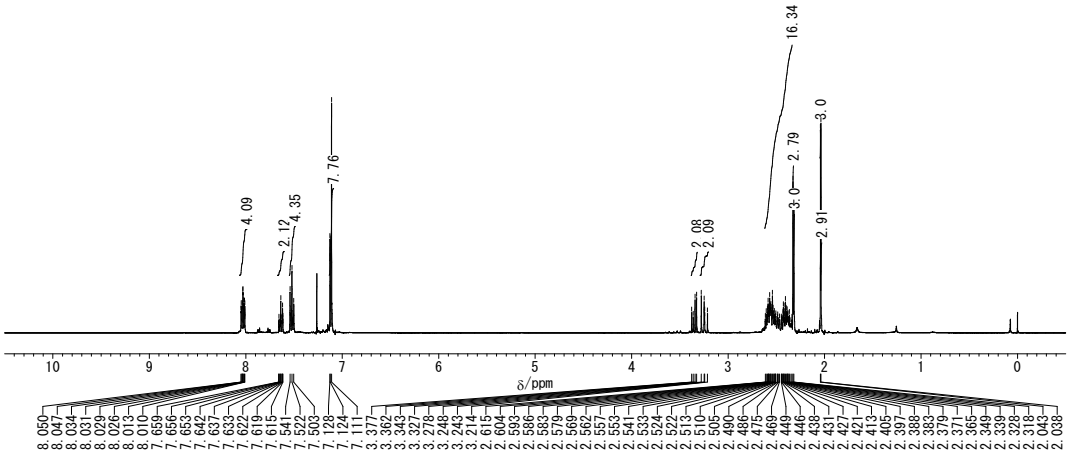
^{1}H NMR (400 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 1-((5-methoxy-2-(4-methoxyphenyl)benzofuran-3-yl)disulfanyl)-4-(2,3,4-trimethoxybenzyl)piperazine (**15**) ($CDCl_3$)

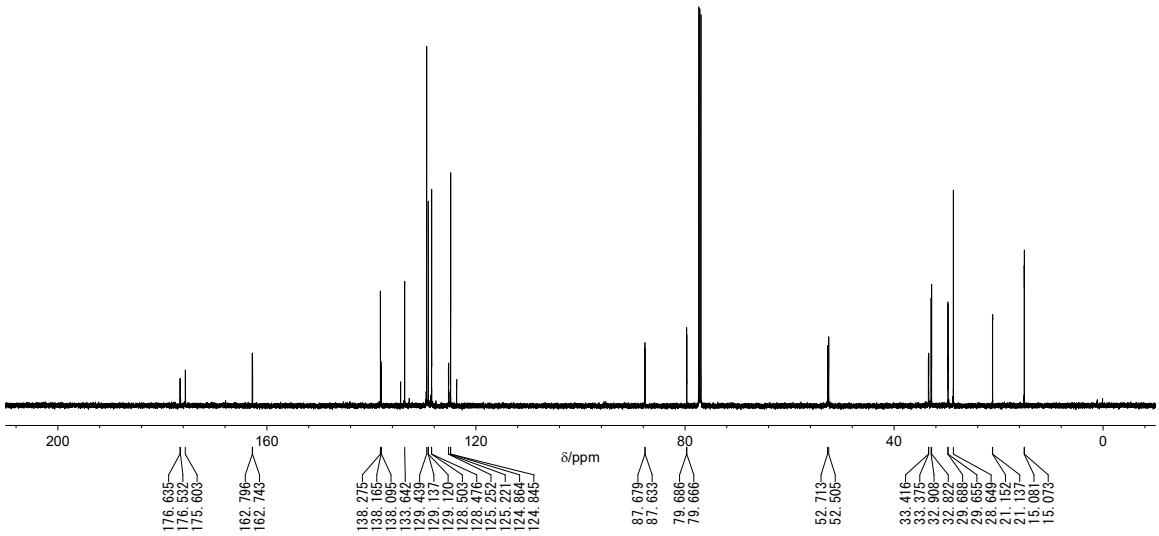




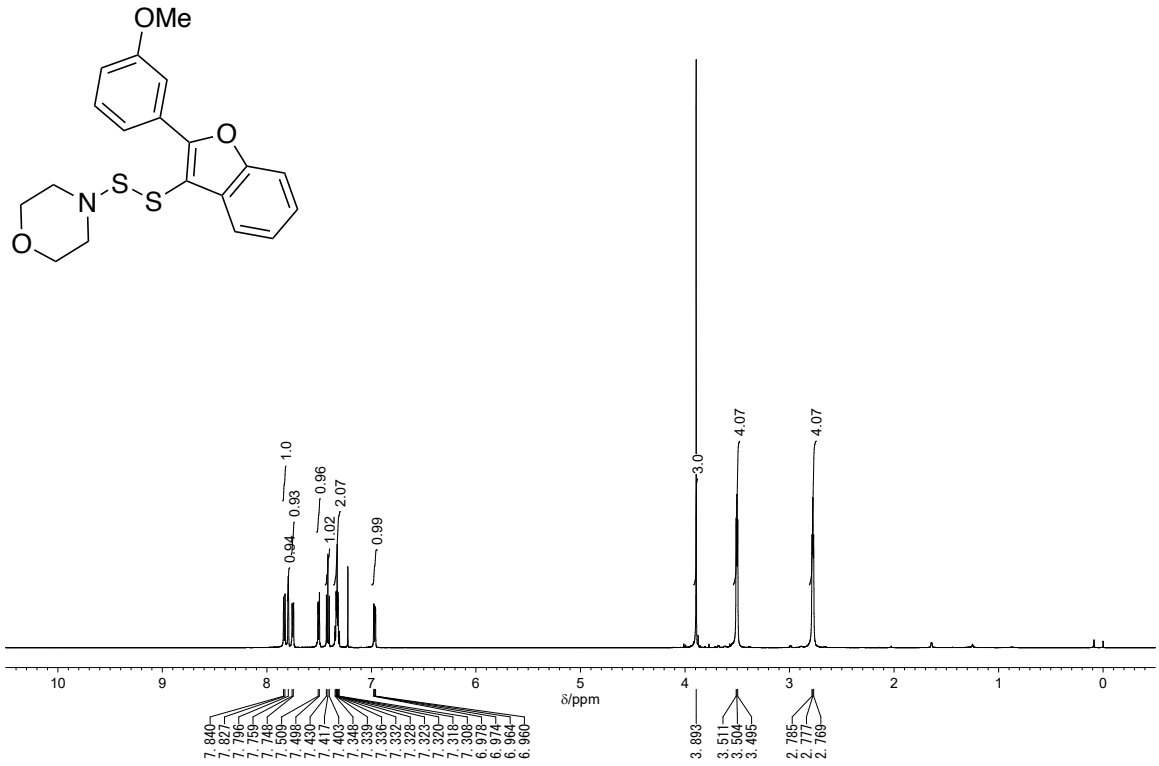
^{1}H NMR (400 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 4-(2-(methylthio)ethyl)-4-(((5-oxo-2-(*p*-tolyl)tetrahydrofuran-2-yl)methyl)disulfanyl)-2-phenyloxazol-5(4H)-one (**17**) ($CDCl_3$)

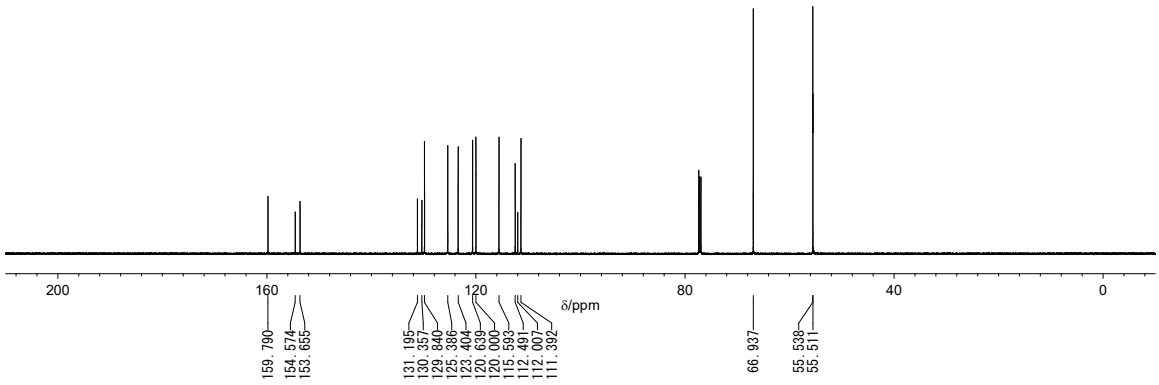






^{1}H NMR (600 MHz) and $^{13}C\{^{1}H\}$ NMR (150 MHz) spectra of 4-((2-(3-methoxyphenyl)benzofuran-3-yl)disulfanyl)morpholine (**18**) ($CDCl_3$)





^{1}H NMR (600 MHz) and ^{13}C{^{1}H} NMR (150 MHz) spectra of 2-methoxybenzo[5,6][1,2]dithiino[4,3-*b*]benzofuran (**19**) ($CDCl_3$)