Synthesis of *N*-(2-(methylamino)ethyl)- derivatives of 2*H*-phthalazin-1-ones

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Supplementary Information

Section A General Information

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Section C ¹H, ¹³C NMR , IR Spectra and element analysis of compounds **9**, **10**, **11**

Section A General Information

The melting points were determined on a Boetius hot stage apparatus and are uncorrected. ^1H NMR spectra were recorded at 200 MHz and ^{13}C NMR spectra at 50 MHz on a Varian Gemini 200 BB spectrometer with TMS as an internal reference. IR spectra were recorded on a Nexus FT-IR spectrometer. Mass spectra analyses were performed on a Mass Spectrometer MAT95 - Finnigan. The analytical thin layer chromatography tests (TLC) were carried out on Merck silica gel plates (Kieselgel 60 F₂₅₄, layer thickness 0.2 mm) and the spots were visualised using UV lamp.

Commercially available (Aldrich, Fluka) hydrazine monohydrate, *N,N,N',N'*-tetramethylethylenediamine, triphenylphosphite (TPP), diethyl azodicarboxylate (DEAD), 2-acetylbenzoic acid, 2-benzoylbenzoic acid were used without further purification. Tetrahydrofuran (THF) was distilled from sodium benzophenone ketyl immediately before use. ω -Aminoethylating agents **6**, **7**, **8** were prepared by standard methods¹⁸⁻²⁰ from the commercially available 2-(methylamino)ethanol.

2*H*-Phthalazin-1-ones **1**, were prepared according to an already reported procedure from appropriate 3-hydroxy-1*H*-isoindolin-1-ones or ketoacids.¹¹⁻¹⁴

Section B Experimental procedures and spectral data

General Procedure for Preparation of Benzenesulfonamides 9

To a solution of sodium methoxide (0.0165 mol) in dry MeOH (70 ml) was added 2*H*-phthalazin-1-one **1** (0.0153 mol). The mixture was heated to boiling for 30 minutes. Afterwards, toluene-4-sulfonic acid 2-[methyl-(toluene-4-sulfonyl)-amino]-ethyl ester (**6**) (0.0230 mol) was added and heating was continued for next 7 hrs. After this time, the mixture was cooled to ambient temperature. The separated product was filtrated off, washed with dry methanol and purified by crystallization or column chromatography.

***N*,4-Dimethyl-*N*-[2-(1-oxo-1*H*-phthalazin-2-yl)ethyl]benzenesulfonamide (9a):**

Yield 50%; mp 136-139°C (MeOH) (lit. ^[16]); FT-IR (KBr, cm⁻¹) ν : 1651 (C=O), 1338, 1165 (SO₂); ¹H NMR (200 MHz, CDCl₃, ppm) δ : 8.41-8.36 (m, 1H, 5-ArH), 8.15 (s, 1H, 4-ArH), 7.86-7.66 (m, 3H, 6,7,8-ArH), 7.61 (d, 2H, TsH, *J*=8.0 Hz), 7.18 (d, 2H, TsH, *J*=8.0 Hz), 4.41 (t, 2H, CH₂, *J*=6.3 Hz), 3.51 (t, 2H, CH₂, *J*=6.3 Hz), 2.91 (s, 3H, NMe), 2.34 (s, 3H, TsMe); ¹³C NMR (50 MHz, CDCl₃, ppm) δ : 159.3, 143.0, 137.8, 134.9, 133.7, 133.0, 131.5, 129.5, 129.4, 127.0, 126.5, 125.9, 48.4, 48.1, 35.0, 21.4. Anal. calcd. for (C₁₈H₁₉N₃O₃S): C 60.49, H 5.36, N 11.76, S 8.97%. Found: C 60.39, H 5.42, N 11.63, S 9.08%.

***N*,4-Dimethyl-*N*-[2-(4-methyl-1-oxo-1*H*-phthalazin-2-yl)ethyl]benzenesulfonamide (9b):**

Yield 45%; mp 150-152°C; R_f=0.46 (AcOEt-CH₂Cl₂=1:2); FT-IR (KBr, cm⁻¹) ν : 1643 (C=O), 1340, 1159 (SO₂); ¹H NMR (200 MHz, CDCl₃, ppm) δ : 8.43-8.38 (m, 1H, 8-ArH), 7.86-7.70 (m, 3H, 5,6,7-ArH), 7.60 (d, 2H, TsH, *J*=8.2 Hz), 7.15 (d, 2H, TsH, *J*=8.2 Hz), 4.36 (t, 2H, CH₂, *J*=6.3 Hz), 3.52 (t, 2H, CH₂, *J*=6.3 Hz), 2.92 (s, 3H, NMe), 2.56 (s, 3H, Me), 2.32 (s, 3H, TsMe); ¹³C NMR (50 MHz, CDCl₃, ppm) δ : 159.2, 143.6, 142.9, 135.0, 132.8, 131.1,

129.7, 129.3, 127.4, 127.0, 126.9, 124.6, 48.2, 48.0, 34.9, 21.4, 18.7. Anal. calcd. for (C₁₉H₂₁N₃O₃S): C 61.44, H 5.70, N 11.31, S 8.63%. Found: C 61.32; H 5.82, N 11.32, S 8.42%.

4,*N*-Dimethyl-*N*-[2-(1-oxo-4-phenyl-1*H*-phthalazin-2-yl)-ethyl]benzenesulfonamide (9c):

Yield 64%; mp 128-131°C (MeOH); FT-IR (KBr, cm⁻¹) v: 1654 (C=O), 1336, 1163 (SO₂); ¹H NMR (200 MHz, CDCl₃, ppm) δ: 8.46-8.51 (m, 1H, 8-ArH), 7.82-7.72 (m, 3H, Ph, ArH), 7.67-7.52 (m, 7H; Ph, Ar, TsH), 7.15 (m, 2H, TsH), 4.48 (t, 2H, CH₂, *J*=6.0 Hz), 3.59 (t, 2H, CH₂, *J*=6.0 Hz), 2.93 (s, 3H, NMe), 2.31 (s, 3H, TsMe); ¹³C NMR (50 MHz, CDCl₃, ppm) δ: 159.1, 142.9, 135.0, 134.9, 132.7, 131.2, 129.4, 129.0, 128.5, 127.9, 127.0, 126.6, 48.1, 34.8, 21.4. Anal. calcd. for (C₂₄H₂₃N₃O₃S): C 66.49, H 5.35, N 9.69, S 7.40%. Found: C 66.45; H 5.42, N 9.73, S 7.47%.

***N*-{2-[4-(4-Methoxy-phenyl)-1-oxo-1*H*-phthalazin-2-yl]-ethyl}-4,*N*-dimethylbenzenesulfonamide (9d):**

Yield 47%; mp 149-151°C; *R*_f=0.42 (CHCl₃-AcOEt=4:1); FT-IR (KBr, cm⁻¹) v: 1652 (C=O), 1341, 1159 (SO₂); ¹H NMR (200 MHz, CDCl₃, ppm) δ: 8.50-8.45 (m, 1H, ArH), 7.80-7.73 (m, 3H, ArH), 7.62-7.53 (m, 4H, ArH), 7.16-7.04 (m, 4H, ArH), 4.47 (t, 2H, CH₂, *J*=6.0 Hz), 3.90 (s, 3H, OMe), 3.58 (t, 2H, CH₂, *J*=6.0 Hz), 2.92 (s, 3H, NMe), 2.31 (s, 3H, TsMe); ¹³C NMR (50 MHz, CDCl₃, ppm) δ: 160.4, 159.3, 147.0, 143.1, 135.2, 132.8, 131.3, 130.8, 129.5, 129.5, 128.2, 127.2, 127.1, 126.8, 114.1, 55.4, 48.2, 48.1, 34.8, 21.4. Anal. calcd. for (C₂₄H₂₅N₃O₃S): C 64.78, H 5.44, N 9.06, S 6.92%. Found: C 64.58, H 5.63, N 8.85, S 6.98%.

***N*-{2-[4-(4-Chlorophenyl)-1-oxo-1*H*-phthalazin-2-yl]-ethyl}-4,*N*-dimethylbenzenesulfonamide (9e):**

Yield: 40%; mp 151-152°C; R_f =0.78 (CHCl₃-AcOEt=4:1); FT-IR (KBr, cm⁻¹) ν : 1659 (C=O), 1159, 1341 (SO₂); ¹H NMR (200 MHz, CDCl₃, ppm) δ 8.52-8.47 (m, 1H, ArH), 7.81-7.15 (m, 9H, ArH), 7.19-7.15 (m, 2H, ArH), 4.47 (t, 2H, CH₂, J =6.0 Hz), 3.57 (t, 2H, CH₂, J =6.0 Hz), 2.91 (s, 3H, NMe), 2.33 (s, 3H, TsMe); ¹³C NMR (50 MHz, CDCl₃, ppm) δ 159.0, 145.8, 143.0, 135.1, 134.9, 133.4, 132.8, 131.3, 130.8, 129.4, 128.7, 127.9, 127.1, 127.0, 126.2, 48.1, 47.9, 34.8, 21.4. Anal. calcd. for (C₂₄H₂₂N₃ClO₃S): C 61.60, H 4.74, N 8.98, Cl 7.58, S 6.85%. Found: C 61.25, H 4.97, N 9.01, Cl 7.50, S 6.75%.

***N*-[2-(6-Methoxy-1-oxo-4-pyridin-2-yl)-1*H*-phthalazin-2-yl]-ethyl]-4,*N*-dimethylbenzenesulfonamide (9f):**

Yield: 45%; mp 117-120°C; R_f =0.76 (AcOEt); FT-IR (KBr, cm⁻¹) ν : 1656 (C=O), 1162, 1335 (SO₂); ¹H NMR (200 MHz, CDCl₃, ppm) δ : 8.78-8.60 (m, 1H, 6Py-H), 8.38 (d, 1H, ArH, J =8.8 Hz), 7.96-7.93 (m, 3H, ArH), 7.62-7.59 (m, 2H, ArH), 7.43-7.28 (m, 2H, ArH), 7.18-7.14 (m, 2H, ArH), 4.50 (t, 2H, CH₂, J =6.0 Hz), 3.90 (s, 3H, OMe), 3.59 (t, 2H, CH₂, J =6.0 Hz), 2.92 (s, 3H, NMe), 2.32 (s, 3H, TsMe); ¹³C NMR (50 MHz, CDCl₃, ppm) δ : 163.1, 159.3, 154.9, 148.4, 143.7, 143.1, 137.3, 135.0, 130.6, 129.5, 128.9, 127.1, 124.7, 123.6, 121.8, 120.3, 108.8, 55.6, 48.2, 48.1, 34.9, 21.4. Anal. calcd. for (C₂₄H₂₄N₄O₄S): C 62.05, H 5.21, N 12.06, S 6.90%. Found: C 62.17, H 5.32, N 11.96, S 7.07%.

General Procedure for Preparation of Acetyl- and *tert*-Butoxycarbonyl- Derivatives of 2-[(Methylamino)ethyl]-2*H*-phthalazin-1-ones 11 (Mitsunobu Procedure)

The Mitsunobu reaction was carried out under argon. To a stirred solution of TPP (0,0102 mol) in dry THF (10 ml) was slowly added DEAD (0,0102 mol, solution in toluene c $\approx$ 40%) at -10°C. Then a solution of phthalazinone **1** (0.0068 mol) in THF (44 ml) was added

dropwise. The whole lot was mixed for 15 min at -10°C and next was added the appropriate derivative of *N*-methylethanolamine **7** or **8** (0.00748 mol) in THF (5 ml) at -10°C. The mixture was stirred during 2 hrs at -10°C, after which time, the reaction mixture was warmed to ambient temperature and stirred in this conditions for 20 hrs. All volatile materials were removed under reduced pressure, ethyl ether (20ml) was added to the residue and the whole lot was stirred for 0.5 hrs at ambient temperature. The separate white solid was collected by filtration, washed with ether and filtrate was evaporated to dryness. The residue was subjected to column chromatography to give the pure product **11**.

***N*-Methyl-*N*-[2-(1-oxo-1*H*-phthalazin-2-yl)-ethyl]-acetamide (**11a**):**

Yield: 30%; mp 101-105°C; R_f =0.62 (AcOEt-MeOH=3:1); FT-IR (film, cm^{-1}) ν : 1736, 1648 (C=O); ^1H NMR (200 MHz, CDCl_3 , ppm, mixture of rotamers) δ : 8.33 (d, 1H, ArH, J =7.4 Hz), 8.10 (s, 1H, 4H), 7.76-7.59 (m, 3H, ArH), 4.34 (t, 2H, CH_2 , J =6.2 Hz), 3.78-3.65 (m, 2H, CH_2), 2.92, 2.90 (2 $\times$ s, 3H, NMe, two rotamers), 1.92 (s, 3H, Me); ^{13}C NMR (50 MHz, CDCl_3 , ppm, mixture of rotamers) δ : 170.7, 170.5, 159.5, 159.3, 138.1, 137.7, 133.3, 132.9, 131.8, 131.4, 129.4, 129.4, 127.4, 127.3, 126.4, 126.3, 126.1, 125.9, 48.7, 48.5, 48.3, 46.0, 36.7, 33.2, 21.6, 20.8. Anal. calcd. for ($\text{C}_{13}\text{H}_{15}\text{N}_3\text{O}_2$) C 63.66, H 6.16, N 17.13%. Found: C 63.37, H 6.18N 17.01%.

Methyl-[2-(1-oxo-1*H*-phthalazin-2-yl)-ethyl]-carbamic acid *tert*-butyl ester (11b**):**

Yield: 45%; mp 73-75°C; R_f =0.5 (CH_2Cl_2 -acetone=9:1); FT-IR (KBr, cm^{-1}) ν : 1690, 1659 (C=O); ^1H NMR (200 MHz, CDCl_3 , ppm, mixture of rotamers) δ : 8.46-8.43 (m, 1H, ArH), 8.16 (s, 1H, ArH), 7.80-7.72 (m, 3H, ArH), 4.42-4.38 (m, 2H, CH_2), 3.72-3.66 (m, 2H, CH_2), 2.91 (s, 3H, NMe), 1.30, 1.21 (2 $\times$ s, 9H, Boc-Me, two rotamers); ^{13}C NMR (50 MHz, CDCl_3 , ppm, mixture of rotamers) δ : 159.3, 155.4, 137.4, 133.0, 131.4, 129.6, 127.7, 126.5, 125.8,

79.2, 49.1, 48.7, 47.1, 34.8, 34.3, 28.1. Anal. calcd. for (C₁₆H₂₁N₃O₃): C 63.35, H 6.98, N 13.85%. Found: C 63.31, H 6.62, N 13.85%.

***N*-Methyl-*N*-[2-(1-oxo-4-phenyl-1*H*-phthalazin-2-yl)-ethyl]-acetamide (11c):**

Yield: 55%; mp 138-140°C; *R*_f=0.12 (CH₂Cl₂-acetone=9:1); FT-IR (KBr, cm⁻¹) *v*: 1652, 1649 (C=O); ¹H NMR (200 MHz, CDCl₃, ppm, mixture of rotamers) *δ*: 8.53-8.50 (m, 1H, ArH), 7.81-7.72 (m, 3H, ArH), 7.61-7.50 (m, 5H, ArH), 4.50 (m, 2H, CH₂), 3.93-3.77 (m, 2H, CH₂), 3.04, 3.02 (2×s, 3H, NMe, two rotamers), 2.02, 1.97 (2×s, 3H, Ac-Me, two rotamers); ¹³C NMR (50 MHz, CDCl₃, ppm, mixture of rotamers) *δ*: 171.1, 170.9, 159.4, 159.3, 147.8, 147.1, 135.1, 134.8, 133.1, 132.8, 131.7, 131.3, 129.4, 129.3, 129.0, 128.7, 128.6, 127.1, 126.7, 49.0, 48.6, 46.2, 36.7, 33.5, 21.7, 20.9. Anal. calcd. for (C₁₉H₁₉N₃O₂): C 71.01, H 5.96, N 13.07%. Found: C 70.88; H 5.99, N 12.81%.

Methyl-[2-(1-oxo-4-phenyl-1*H*-phthalazin-2-yl)-ethyl]-carbamic acid *tert*-butyl ester (11d):

Yield: 50%; mp 169-170°C (MeOH-H₂O) (lit.^[16]); *R*_f=0.42 (CH₂Cl₂-acetone=9:1); FT-IR (KBr, cm⁻¹) *v*: 1690, 1645 (C=O); ¹H NMR (200 MHz, CDCl₃, ppm, mixture of rotamers) *δ*: 8.57-8.54 (m, 1H, ArH), 7.70-7.90 (m, 3H, ArH), 7.61-7.53 (m, 5H, ArH), 4.49-4.44 (m, 2H, CH₂), 3.77-3.72 (m, 2H, CH₂), 2.95, 2.89 (2×s, 3H, NMe, two rotamers), 1.27, 1.13 (2×s, 9H, Boc-Me, two rotamers); ¹³C NMR (50 MHz, CDCl₃, ppm, mixture of rotamers) *δ*: 158.9, 155.4, 146.6, 134.9, 132.7, 131.1, 129.4, 128.9, 128.5, 127.0, 126.5, 79.2, 48.9, 47.1, 46.8, 34.9, 34.2, 28.0. Anal. calcd. for (C₂₂H₂₅N₃O₃): C 69.64, H 6.64, N 11.07%. Found: C 69.57, H 6.77, N 11.09%.

[2-(6-Methoxy-1-oxo-4-pyridin-2-yl-1*H*-phthalazin-2-yl)-ethyl]-methyl-carbamic acid *tert*-butyl ester (11e):

Yield: 55%; mp 119-121°C; R_f =0.74 (AcOEt-acetone=3:1); FT-IR (KBr, cm^{-1}) ν : 1686, 1654 (C=O); ^1H NMR (200 MHz, CDCl_3 , ppm, mixture of rotamers) δ : 8.78-8.73 (m, 1H, ArH), 8.43 (d, 1H, ArH, J =8.8 Hz), 7.93-7.87 (m, 3H, ArH), 7.43-7.30 (m, 2H, ArH), 4.47-4.42 (m, 2H, CH_2), 3.90 (s, 3H, OMe), 3.76-3.70 (m, 2H, CH_2), 2.94, 2.86 (2 $\times$ s, 3H, NMe, two rotamers), 1.30, 1.17 (2 $\times$ s, 9H, Boc-Me, two rotamers); ^{13}C NMR (50 MHz, CDCl_3 , ppm, mixture of rotamers) δ : 163.1, 155.6, 154.9, 148.3, 137.4, 137.2, 130.7, 128.9, 124.6, 123.5, 121.9, 120.4, 108.5, 79.3, 55.6, 48.9, 48.7, 47.3, 47.0, 35.1, 34.3, 28.1. Anal. calcd. for ($\text{C}_{22}\text{H}_{23}\text{N}_4\text{O}_4$): C 64.38, H 6.38, N 13.65%. Found: C 64.65, H 6.41, N 14.19%.

General Procedure for Preparation of 2-[(Methylamino)ethyl]-2*H*-phthalazin-1-ones 10

Method A. Hydrolysis of Benzenesulfonamides 9

The benzenesulfonamide **9** (0.0106 mol) and H_2SO_4 (98%, 0.0318 mol) were heated up to 100-110°C over a period of 6 hrs. After this time the reaction mixture was cooled to ambient temperature, alkalized with 20% aqueous solution of NaOH and next extracted with CH_2Cl_2 (3 $\times$ 30ml). The combined extracts were dried over MgSO_4 and concentrated till dryness. The amine was separated by column chromatography.

Method B. Hydrolysis of *tert*-Butyl Methyl[2-(1-oxo-1*H*-phthalazin-2-yl)ethyl]-carbamates 11

To a solution of HCl_{aq} (20%, 36 ml) was added carbamate **11** (0.00275 mol). The mixture was stirred for 30 hrs at room temperature. Next reaction mixture was alkalised with 20% aqueous solution of NaOH and extracted with CH_2Cl_2 (3 $\times$ 50ml). The combined extracts were dried

over MgSO₄ and concentrated under vacuum. The amine was isolated by column chromatography.

2-[2-(Methylamino)-ethyl]-2*H*-phthalazin-1-one (10a):

Yield: 78% (Method A, oil); R_f =0.0 (AcOEt-MeOH=1:1, next MeOH); FT-IR (film, cm⁻¹) ν : 1652 (C=O), 3316 (NH); ¹H NMR (200 MHz, CDCl₃, ppm) δ : 8.46-8.42 (m, 1H, ArH), 8.19 (s, 1H, ArH), 7.87-7.69 (m, 3H, ArH), 4.40 (t, 2H, CH₂, J =6.0 Hz), 3.09 (t, 2H, CH₂, J =6.0 Hz), 2.48 (s, 3H, Me), 2.37 (brs, 1H, NH); ¹³C NMR (50 MHz, CDCl₃, ppm) δ : 159.3, 137.7, 132.8, 131.4, 129.3, 127.6, 126.4, 125.7, 50.1, 50.0, 36.0. HRMS Calcd. for (C₁₁H₁₃N₃O): 203.10586. Found: 203.10597.

4-Methyl-2-[2-(methylamino)-ethyl]-2*H*-phthalazin-1-one (10b):

Yield: 65% (Method A, oil); R_f =0.46 (AcOEt-CH₂Cl₂=1:2); FT-IR (KBr, cm⁻¹) ν : 1652 (C=O), 3319 (NH); ¹H NMR (200 MHz, CDCl₃, ppm) δ : 8.45-8.50 (m, 1H, ArH), 7.72-7.87 (m, 3H, ArH), 4.37 (t, 2H, CH₂, J =6.0 Hz), 3.08 (t, 2H, CH₂, J =6.0 Hz), 2.60 (s, 3H, NMe), 2.49 (s, 3H, Me), 1.58 (s, 1H, NH); ¹³C NMR (50 MHz, CDCl₃, ppm) δ : 158.4, 142.5, 131.8, 130.2, 128.6, 126.6, 125.9, 123.8, 49.4, 49.1, 35.3, 17.9. Anal. calcd. for (C₁₂H₁₅N₃O): C 66.34, H 6.96, N 19.34%. Found: C 66.42, H 6.97, N 19.27%.

2-[2-(Methylamino)-ethyl]-4-phenyl-2*H*-phthalazin-1-one (10c):

Yield: 56% (Method A); mp 96-98°C; R_f =0.01 (AcOEt-MeOH=1:1 next MeOH); FT-IR (KBr, cm⁻¹) ν : 1652 (C=O), 3332 (NH). ¹H NMR (200 MHz, CDCl₃, ppm) δ : 8.57-8.53 (m, 1H, ArH), 7.82-7.76 (m, 3H, ArH), 7.62-7.53 (m, 6H, ArH), 4.49 (t, 2H, CH₂, J =6.1 Hz), 3.16 (t, 2H, CH₂, J =6.1 Hz), 2.51 (s, 3H, NMe), 2.29 (s, 1H, NH); ¹³C NMR (50 MHz, CDCl₃, ppm) δ : 159.2, 146.9, 135.0, 132.6, 131.2, 129.3, 128.9, 128.4, 127.0, 126.5, 50.3,

50.2, 36.1. Anal. calcd. for (C₁₇H₁₇N₃O): C 73.10, H 6.13, N 15.04%. Found: C 73.11, H 5.98, N 15.11%.

6-Methoxy-2-[2-(methylamino)-ethyl]-4-(pyridin-2-yl)-2*H*-phthalazin-1-one (10d):

Yield: 55% (Method B); mp 94-96°C; R_f=0.0 (AcOEt next MeOH); FT-IR (KBr, cm⁻¹) ν: 1652 (C=O), 3316 (NH); ¹H NMR (200 MHz, CDCl₃, ppm) δ: 8.75-7.24 (m, 1H, ArH), 8.42 (d, 1H, ArH, *J*=8.8 Hz), 7.95-7.86 (m, 3H, ArH), 7.42-7.28 (m, 2H, ArH), 4.44 (t, 2H, CH₂, *J*=6.1 Hz), 3.88 (s, 3H, OMe), 3.10 (t, 2H, CH₂, *J*=6.1 Hz), 2.47 (s, 3H, NMe), 1.52 (brs, 1H, NH); ¹³C NMR (50 MHz, CDCl₃, ppm) δ: 162.9, 159.3, 154.9, 148.4, 143.3, 137.1, 130.4, 128.9, 124.2, 123.4, 121.9, 120.2, 108.6, 55.5, 50.3, 50.2, 36.0. Anal. calcd. for (C₁₇H₁₈N₃O₂): C 65.79, H 5.85, N 18.05%. Found: C 65.52, H 5.78, N 17.93%.

Section C ^1H , ^{13}C NMR , IR Spectra of compounds **9**, **10**, **11**

9a

EXP1 PULSE SEQUENCE: S2PUL
DATE 01-01-05
SOLVENT CDCl3
FILE H

Data: Śr Paź 11 14:31:40 2006

Skanów: 16

KBr

Rozdzielczość: 4.000

9b

Data: Pon Lis 20 13:53:27 2008

Skanów: 16

KBr

Rozdzielczość: 4,000

EXP1 PULSE SEQUENCE: S2PUL
DATE 01-09-05
SOLVENT CDCL3
FILE H

9c

Data: Śr Paź 11 14:05:54 2006

Skanów: 16

KBr

Rozdzielczość: 4.000

EXP1 PULSE SEQUENCE: S2PUL
DATE 02-08-09
SOLVENT CDCl3
FILE H

9d

Data: Czw Kwi 12 11:08:49

Skanów: 16

KBr

Rozdzielczość: 4,000

EXP1 PULSE SEQUENCE: S2PUL
DATE 03-15-07
SOLVENT CDCL3
FILE H

EXP4 PULSE SEQUENCE: S2PUL
DATE 03-16-07
SOLVENT CDCl3
FILE C

9e

Data: Czw Kwi 12 10:40:47

Skanów: 16

KBr

Rozdzielczość: 4,000

EXP4 PULSE SEQUENCE: S2PUL
DATE 01-24-97
SOLVENT CDCL3
FILE C

EXP5 PULSE SEQUENCE: S2PUL
DATE 03-23-07
SOLVENT CDCL3
FILE C

10a

Data: Pt Sty 12 10:35:34 ;

Skanów: 16

film

Rozdzielczość: 4,000

10t

Data: Wt Sty 23 12:31:2t

Skanów: 16

KBr

Rozdzielczość: 4,000

EXP2 PULSE SEQUENCE: S2PUL
DATE 01-01-07
SOLVENT CDCL3
FILE C

10c

10d

Data: Śr Maj 16 10:05:14 2007

Skanów: 16

film

Rozdzielczość: 4,000

EXP1 PULSE SEQUENCE: zgpg30
DATE 01-01-07
SOLVENT CDCl₃
FILE 1

EXP2 PULSE SEQUENCE: zgpg30
DATE 01-05-07
SOLVENT CDCl3
FILE C

11a

Data: Czw Luty 15 13:42:59 2007

Skanów: 16

film

Rozdzielczość: 4,000

EXP2 PULSE SEQUENCE: S2PUL

DATE 01-14-07
SOLVENT CDCL3
F1 C

11b

Data: Wt Lut 27 14:44.2

Skanów: 16

KBr

Rozdzielczość: 4,000

11c

Data: Wt Luty 27 14:04:40

Skandów: 16

KBr

Rozdzielczość: 4,000

EXP1 PULSE SEQUENCE: S2PUL
DATE 03-23-07
SOLVENT CDCL3
FILE H

EXP2 PULSE SEQUENCE: S2PUL
DATE 03-26-07
SOLVENT COCL3
FILE C

Data: Wt Sty 23 11:56:06

Skanów: 16

KBr

Rozdzielczość: 4,000

11e

Data: Czw Maj 31 13:31:06

Skanów: 16

Rozdzielczość: 4,000

KBr

EXP1 PULSE SEQUENCE: S2PUL
DATE 04-02-07
SOLVENT CDCL3
FILE H

EXP2 PULSE SEQUENCE: S2PUL
DATE 04-04-07
SOLVENT CDCL3
FILE C