Expedient synthesis of substituted (diphenylphosphinoylmethyl)benzenes.

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General protocols

All reactions requiring anhydrous conditions were conducted in flame-dried glass apparatus under an atmosphere of argon. Water refers to distilled water. All commercially available reagents were used as supplied.

The ^1H -spectra were recorded in ppm and referenced to the residual CHCl_3 signal located at δ 7.26. ^{13}C -NMR spectra were recorded in ppm and referenced to the residual CHCl_3 signal found at δ 77.00. Multiplicities in the NMR spectra are described as: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad; coupling constants are reported in Hz.

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Synthesis of **2**

Following the typical procedure outlined for **1**, chlorodiphenylphosphine (1 mL, 5.46 mmol) was added to benzoic acid (222 mg, 1.82 mmol) and water (65 μ L, 3.64 mmol). After microwave irradiation, work up and purification as outlined above, **2** was afforded as a white solid in a 79% yield (420 mg). Uncorrected melting point mp 189 - 193°C (Lit¹ 192 – 193°C), ¹H NMR (400 MHz, CDCl₃): δ 3.66 (d, 2H, J = 13), 7.10 – 7.20 (m, 5H), 7.40 – 7.54 (m, 6H), 7.66 – 7.73 (m, 4H); ¹³C NMR (75 MHz, CDCl₃): δ 133.1, 131.9 (d J_{PC} = 2.9), 131.2 (d J_{PC} = 9.1), 130.2 (d J_{PC} = 5.7), 128.6, 128.5, 128.4, 126.8 (d J_{PC} = 2.9), 38.0 (d J_{PC} = 66.0); IR (KBr) ν 3058, 1600, 1492, 1484, 1453, 1440, 1191, 1179, 1137, 1118, 1105 cm⁻¹; m/z (EI+) 292.2 (14), 291.1 (33), 202.1 (13), 201.1 (100), 183.0 (4), 152.1 (4), 91.1 (41), 84.0 (18), 77.1 (26), 65.1 (14), 51.2 (19), 49.2 (27); HRMS calcd for C₁₉H₁₇OP 293.1090, found 293.1091.

Synthesis of **3**

Following the typical procedure outlined for **1**, chlorodiphenylphosphine (1 mL, 5.46 mmol) was added to 2-toluic acid (248 mg, 1.82 mmol) and water (65 μ L, 3.64 mmol). After microwave irradiation, work up and purification as outlined above, **3** was afforded as a white solid in a 71% yield (394 mg). Uncorrected melting point mp 123 - 125°C, (Lit¹ 122 – 123°C), ¹H NMR (400 MHz, CDCl₃): δ 2.14 (s, 3H), 3.68 (d, 2H, J = 13), 7.07 (m, 1H), 7.25 – 7.30 (m, 2H), 7.39 – 7.85 (m, 20H); ¹³C NMR (75 MHz, CDCl₃): δ 134.2, 133.8, 133.6, 132.7, 132.0, 131.3, 131.2, 131.1, 130.3, 128.6 (d J_{PC} = 2.9), 128.4, 37.1 (d J_{PC} = 66.0); IR (KBr) ν 1590, 1493, 1430, 1405, 1318, 1238, 1185, 1100, 1170 cm⁻¹; m/z (EI+) 342.2 (23), 217.1 (8), 202.1 (20), 201.1 (100), 183.0 (3), 141.0 (32),

115.1 (28), 77.1 (18), 51.2 (6), 51.2 (19), 49.2 (27); HRMS calcd for $C_{32}H_{27}^{79}BrO_2P_2$ 585.0742, found 585.0740.

Synthesis of **4**.

Following the typical procedure outlined for **1**, chlorodiphenylphosphine (1 mL, 5.46 mmol) was added to 2-naphthoic acid (313 mg, 1.82 mmol) and water (65 μ L, 3.64 mmol). After microwave irradiation, work up and purification as outlined above, **4** was afforded as a white solid in a 68% yield (423 mg). Uncorrected melting point mp > 250°C, 1H NMR (400 MHz, $CDCl_3$): δ 3.84 (d, 2H, $J = 13$), 7.20 – 7.28 (m, 2H), 7.40 – 7.77 (m, 15H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 133.5 (d $J_{PC} = 3.1$), 133.0, 132.4 (d $J_{PC} = 2.3$), 132.1 (d $J_{PC} = 3.1$), 132.0, 131.4 (d $J_{PC} = 9.1$), 129.2 (d $J_{PC} = 6.8$), 129.0 (d $J_{PC} = 7.6$), 128.8 (d $J_{PC} = 12.2$), 128.4 (d $J_{PC} = 4.6$), 128.2 (d $J_{PC} = 2.3$), 127.8 (d $J_{PC} = 8.3$), 125.9, 126.0, 38.5 (d $J_{PC} = 66.0$); IR (KBr) ν 2935, 1510, 1438, 1185 cm^{-1} ; m/z (EI+) 342.2 (23), 217.1 (8), 202.1 (20), 201.1 (100), 183.0 (3), 141.0 (32), 115.1 (28), 77.1 (18), 51.2 (6), 51.2 (19), 49.2 (27); HRMS calcd for $C_{23}H_{19}OP$ 343.1246, found 343.1244.

Synthesis of **5**.

Following the typical procedure outlined for **1**, chlorodiphenylphosphine (1.67 mL, 9.10 mmol) was added to phthalic acid (302 mg, 1.82 mmol) and water (100 μ L, 5.46 mmol). After microwave irradiation, work up and purification as outlined above, **5** was afforded as a white solid in a 62% yield (574 mg). Uncorrected melting point mp > 250°C, (Lit² 283°C) 1H NMR (400 MHz, $CDCl_3$): δ 3.98 (d, 4H, $J =$), 6.70 – 6.75 (m, 2H), 6.84 – 6.88 (m, 2H), 7.36 – 7.53 (m, 12H), 7.64 – 7.73 (m, 8H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 133.3, 132.0, 131.8, 131.7, 131.6, 131.3 (d, $J_{PC} = 9.1$), 128.6 (d

$J_{\text{PC}} = 11.9$), 126.7, 35.3 (d $J_{\text{PC}} = 66.0$); IR (KBr) ν 2949, 1599, 1485, 1435, 1411, 1180, 1151, 1122, 1103 cm^{-1} ; m/z (EI+) 506.2 (17), 382.2 (7), 321.2 (11), 305.1 (10), 201.1 (82), 181.1 (22), 166.1 (11), 104.1 (13), 84.0 (26), 77.1 (42), 49.2 (37), 44.2 (100); HRMS calcd for $\text{C}_{32}\text{H}_{28}\text{O}_2\text{P}_2$ 507.1637, found 507.1636

Synthesis of **7**.

Following the typical procedure outlined for **1**, chlorodiphenylphosphine (7.48 mL, 40.81 mmol) was added to 4-bromoisophthalic acid (2g, 8.61 mmol) and water (440 μL , 24.49 mmol). After microwave irradiation, work up and purification as outlined above, **7** was afforded as a white solid in a 54% yield (2.58g). Uncorrected melting point $\text{mp} > 250^\circ\text{C}$, ^1H NMR (400 MHz, CDCl_3): δ 3.62 (d, 2H, $J = 13$), 3.77 (d, 2H, $J = 13$), 7.07 (m, 1H), 7.25 – 7.30 (m, 2H), 7.39 – 7.85 (m, 20H); ^{13}C NMR (75 MHz, CDCl_3): δ 134.2, 133.9, 133.6, 132.7, 132.0, 131.3, 131.2, 131.1, 130.3, 128.6 (d $J_{\text{PC}} = 2.9$), 128.4, 37.1 (d $J_{\text{PC}} = 66.0$); IR (KBr) ν 2946, 1522, 1493, 1436, 1160 cm^{-1} ; m/z (EI+) 342.2 (23), 217.1 (8), 202.1 (20), 201.1 (100), 183.0 (3), 141.0 (32), 15.1 (28), 77.1 (18), 51.2 (6), 51.2 (19), 49.2 (27); HRMS calcd for $\text{C}_{32}\text{H}_{27}^{79}\text{BrO}_2\text{P}_2$ 585.0742, found 585.0740

Microwave procedure for the synthesis of **8**

A mixture of **7** (300 mg, 0.5mmol), phenylacetylene (0.07mL, 0.61 mmol), $\text{PdCl}_2(\text{PPh}_3)_2$ (17.9 mg, 0.025 mmol, 5 mol%), CuI (4.7 mg, 0.025 mmol, 5 mol%), $(i\text{Pr})_2\text{NH}$ (0.2 mL), $t\text{Bu}_3\text{P}$ (10.3 mg, 1M solution in toluene, 10 mol%) in DMF (2mL) was stirred under argon in a sealed (aluminum crimp cap) Smith reaction tube for 10 min, after which the reaction was heated in a Personal Chemistry

microwave synthesizer at 120°C for 30 min. The mixture was cooled to room temperature and diluted with water (10 mL). The reaction products were extracted with ethyl acetate (3 x 5 mL). The combined ethyl acetate extracts were washed successively with water (2 x 5 mL), dilute HCl (5 mL), water (5 mL) and brine (5 mL). The organic phase was dried over anhydrous MgSO₄, filtered and solvent removed under reduced pressure. Flash column chromatography using dichloromethane-methanol (3% MeOH) solvent system afforded the product as pale brown oil (278 mg, 90%). $\nu_{\max}$ FT-IR (film / cm⁻¹) 3429, 3055, 2907, 1595, 1499, 1437; δ_{H} (400 MHz, CDCl₃) 3.64 (2H, d, J_{PH} 14), 3.84 (2H, d, J_{PH} 14), 7.13 (1H, d, J 8.1), 7.20 (1H, d, J 8.1), 7.31 – 7.70 (26H, m, ArH); δ_{C} (100 MHz, CDCl₃) 36.2 (d, J_{PC} 65), 38.2 (d, J_{PC} 65), 88.0, 93.7, 123.2, 128.5, 128.7, 128.8, 128.9, 131.2, 131.3, 131.4, 131.5 (d, J_{PC} 16), 131.9 (d, J_{PC} 16), 132.0, 132.1, 132.2, 132.3, 133.4, 132.6, 132.7, 132.8; m/z (CI) 607.0 [M+H]⁺ (100), 707.3 [M+iPr₂NH]⁺ (22), 810.5 [M+ (iPr₂NH)₂]⁺ (20); HRMS calcd for C₄₀H₃₂O₂P₂ 607.1950, found 607.1950

Conventional heating procedure for the Sonagashira synthesis of **9**

A mixture of **7** (200 mg, 0.34 mmol), *O*-THP protected propargyl alcohol (56 mg, 0.41 mmol), PdCl₂(PPh₃)₂ (12 mg, 0.017 mmol, 5 mol%), CuI (3.3 mg, 0.017 mmol, 5 mol%), ^tBu₃P (6.8 mg, 1M solution in toluene, 10 mol%) was added to freshly distilled and degassed Et₃N (2mL) and stirred under argon. The reaction was heated at 60°C for 12 hours, cooled to room temperature and the solvent evaporated under reduced pressure and water added (10 mL). The aqueous phase was extracted with ethyl acetate (3 x 5 mL). The combined ethyl acetate extracts were washed successively with water (2 x 5 mL) and brine (5 mL). The organic phase was dried over anhydrous MgSO₄, filtered and solvent removed under reduced pressure. Flash column chromatography using dichloromethane-methanol (3% MeOH) as the eluent solvent system afforded **9** as a pale brown oil (195 mg, 88 %). $\nu_{\max}$ FT-IR (film / cm⁻¹) 3412, 3044, 2948, 2894, 1476, 1437, 1193; δ_{H} (300 MHz, CDCl₃) 1.27 – 1.73 (6H, m), 3.50 (2H, d, J_{PH} 13.5), 3.57 – 3.72 (4H, m), 4.23 (1H, d, J 15.8), 4.34 (1H, d, J 15.8), 4.72 (1H, s), 6.91 (1H, d, J 2.8), 7.01 (1H, dd, J 8.2 and 2.8), 7.13 (1H, d, J 8.2), 6.28 – 7.62 (20H, m, ArH); δ_{C} (75 MHz, CDCl₃) 18.8, 25.1, 28.8, 30.1, 36.6 (d, J_{PC} 65.3), 37.5 (d, J_{PC} 65.3), 54.6, 61.9, 84.1, 89.3, 96.8, 121.5, 123.7, 128.2, 128.3, 128.5, 128.7, 130.2, 130.9 (d, J_{PC}

9.7), 131.2 (d, J_{PC} 9), 131.4, 131.8, 132.2, 132.6, 133.7; m/z (ES⁺) 644.7 (M⁺); HRMS calcd for C₄₀H₃₈O₄P₂ 645.2318, found 645.2317

Synthesis of **10**

To a solution of **7** (2g, 3.42 mmol), vinyl tri-*n*-butyltin (1.1 mL, 3.76 mmol) in dry toluene (35 mL) under an inert atmosphere was added *tris*(dibenzylideneacetone)palladium(0) (156 mg, 0.17 mmol) and triphenylphosphine (179 mg, 0.68 mmol). The reaction was heated at 120°C for 24 hours, followed by removal of the solvent *in vacuo*. The oily residue was redissolved in dichloromethane (50 mL), filtered through a Celite pad and then washed with dichloromethane (50 mL). The organic solution was washed with 10% aqueous ammonia (3 x 50 mL), water (3 x 50 mL), dried over magnesium sulphate, filtered and concentrated *in vacuo*. The product was purified by flash column chromatography on silica gel (dichloromethane / methanol 97 : 3) affording **10** as a white solid (920 mg, 51%). Mp decomposed > 80°C, $\nu_{\max}$ FT-IR (film / cm⁻¹) 3437; δ_{H} (400 MHz, CDCl₃) 3.48 (2H, d, J_{PH} 14), 3.55 (2H, d, J_{PH} 14), 4.99 (1H, d, J 11), 5.26 (1H, d, J 17), 6.63 (1H, dd, J 11 and 17), 6.87 – 7.67 (23H, m, ArH); δ_{C} (100 MHz, CDCl₃) 35 (d, J_{PC} 65), 37.7 (d, J_{PC} 65), 116.5, 126.6, 128.6, 128.7, 128.8, 128.9, 129.1, 131.3 (d, J_{PC} 9), 131.3, 131.4, 131.5, 131.7, 132.0, 132.7, 132.9, 133.3, 134.2; m/z (CI/NH₃) 532.2 (M⁺, 6%), 408, (4), 331 (18), 202 (15), 201 (100), 183 (18), 173 (11), 152 (8), 128 (20), 115 (14), 78 (10), 77 (39), 51 (24); HRMS calcd for C₄₀H₃₄O₂P₂ 609.2107, found 609.2115.

Synthesis of **11**³

To a solution of **10** (550 mg, 1.03 mmol), potassium hexacyanoferrate(III) (1.02 g, 3.10 mmol), potassium carbonate (430 mg, 3.10 mmol), and quinuclidine (34 mg, 0.31 mmol) in a mixture of water / *tert*-butanol (1 : 1, 7 mL each) was added osmium(III) chloride (5 mg, 0.017 mmol). The

resulting mixture was stirred vigorously at room temperature until TLC indicated consumption of the starting material. Sodium sulfite (2.08 g, 16.54 mmol) was added and the stirring maintained for a further 1h before the addition of dichloromethane (10 mL). The aqueous phase was further extracted with dichloromethane (3 x 10 mL). The combined organic phases were dried over MgSO₄ and evaporated *in vacuo*. The residue was purified using flash column chromatography on silica gel (dichloromethane / methanol, 95 : 5) affording the product as a white solid (480 mg, 52%). mp 123°C. FT-IR (thin layer): 3409, 2227, 1640, 1435, 1176, 1120 cm⁻¹; δ_{H} (300 MHz, CDCl₃) 2.95 (OH), 3.20 (1H, t, J_{PH} 14 and J_{HH} 14), 3.34 (1H, dd, J_{PH} 15 and J_{HH} 13), 3.55-3.69 (2H, m), 3.79-3.93 (2H, m), 4.93 (1H, dd, J 10 and 8), 5.25 (OH), 6.46 (1H, d, J 3), 6.73 (1H, dd, J 11 and 3), 7.19 (1H, d, J 11), 7.35 – 7.63 (18H, m, ArH), 7.73 (t, 2H, J 11); δ_{C} (75 MHz, CDCl₃) 34.4 (d, J_{PC} 65), 36.3 (d, J_{PC} 65), 65.2, 69.3, 126.46 (d, J_{PC} 2), 126.50 (d, J_{PC} 3), 127.66, 127.73, 127.8, 127.9, 128.0, 128.1 (d, J_{PC} 3), 128.1, 128.2 (d, J_{PC} 3), 128.4 (d, J_{PC} 3), 128.5 (d, J_{PC} 3), 129.2, 129.5 (d, J_{PC} 3), 129.6 (d, J_{PC} 3), 130.18 (d, J_{PC} 3), 130.26 (d, J_{PC} 3), 130.34 (d, J_{PC} 3), 130.5, 130.6, 131.0, 131.1 (d, J_{PC} 3), 131.3 (d, J_{PC} 3), 131.5 (d, J_{PC} 3), 131.9, 132.3, 132.7 (d, J_{PC} 5), 132.8 (d, J_{PC} 5), 139.36 (d, J_{PC} 4), 139.43 (d, J_{PC} 4); m/z (LC/ MS) 566.8 (M⁺), 548.8, 304, 279.9, 142.95. HRMS calcd for C₃₄H₃₂O₄P₂ 567.1849, found 567.1848.

Synthesis of **12**

A solution of sodium periodate (340 mg, 1.59 mmol) in water (1.5 mL) was added to silica gel (3 g) and the solid stirred for 1h. The resulting silica was added to a solution of **11** (450 mg, 0.79 mmol) in DCM (15 mL) and the suspension stirred vigorously at room temperature for 2h, after which time TLC indicated complete consumption of **11**. Filtering the reaction and evaporation of the solvent *in vacuo* afforded **12** as a clear oil (325 mg, 76%). ν_{max} FT-IR (film / cm⁻¹) 1694; δ_{H} (400 MHz, CDCl₃) 3.72 (2H, d, J_{PH} 14), 4.23 (2H, d, J_{PH} 15), 7.27 – 7.74 (23H, m, ArH), 9.65 (1H, s, CHO); δ_{C} (75 MHz, CDCl₃) 33.5 (d, J_{PC} 63), 37.1 (d, J_{PC} 66), 128.3, 128.4, 128.6 (d, J_{PC} 3), 128.7, 128.8, 128.9, 130.2, 131.0, 131.1 (d, J_{PC} 9), 131.2, 131.3 (d, J_{PC} 6), 131.4, 132.0, 132.1, 132.5, 132.7, 134.8, 192.8; m/z (CI/NH₃) 535.1 (M⁺, 30%), 522, (20), 507 (10), 220 (70), 203 (100), 187 (20), 96 (50); HRMS: found (ES⁺) [M+H]⁺ 535.1587. C₃₃H₂₈O₃P₂ requires [M+H]⁺ 535.1586.

Synthesis of **14**

To a degassed solution of **7** (200 mg, 0.27 mmol) and *para*-vinylphenylboronic acid (49 mg, 0.33 mmol) in a mixture of toluene : ethanol : water (10 : 2 : 1 mL respectively) was added potassium carbonate (491 mg, 3.55 mmol) and tetrakis(triphenylphosphine)palladium(0) (32 mg, 0.27 mmol). The resulting mixture was heated at 85°C under an atmosphere of argon for 16 hours. The reaction was cooled to ambient temperature, partitioned between dichloromethane / water and extracted with aliquots (3 x 10 mL) of dichloromethane. The combined organic layers were dried (MgSO₄), filtered and the solvent removed *in vacuo*. Flash column chromatography on silica gel, using 5% methanol in dichloromethane afforded the product as clear oil (94 mg, 56% yield). $\nu_{\max}$ FT-IR (film / cm⁻¹) 3426, 3057, 2221, 1629, 1489, 1437, 1119; δ_{H} (400 MHz, CDCl₃) 3.52 (2H, d, J_{PH} 14), 3.60 (2H, d, J_{PH} 14), 5.20 (1H, d, J 11), 5.70 (1H, d, J 17), 6.63 (1H, dd, J 11 and 17), 6.73 – 7.67 (27H, m, ArH); δ_{C} (100 MHz, CDCl₃) 32 (d, J_{PC} 66), 35.6 (d, J_{PC} 66), 112.2, 124.2, 128.3, 128.4, 128.5, 128.7, 129.6, 130.4, 131.0, 131.1, 131.2, 131.3, 131.6, 131.7, 132.9, 133.1; m/z (LC/MS) 609.1, 582.75, 557, 540, 506, 454.7, 422.7, 310.7

MW5009
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SW 600.6
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fb 3400 dp n n
bs 16 hs n n
dl 2.256 n n
nt 16 fn n n
ct TRANSMITTER H1 SP
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sfrq 399.918 rf1 1000.4
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pw 5.000 1p 23.7
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dn 0 SC 940
dm nnn VS 38
dmm C th
dpwr 42 a1 cdc ph
dmf 29412

MW5009

MU5003B

MU5024/A1
STANDARD 1H OBSERVE

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fb 3400 dp y
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dl 2.218 fn
ct 18 not used
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tn 18
sfrq 389.918 rfl
tof 0 rfp
tpw 5.000 lp
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dn 0 SC 0
dof 0 VS 0
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dpr 42 a1 cdc ph
dat 29412

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Solvent: cdcl3
Ambient temperature
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File: 0301
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Acq. time 5.444 sec
Vch 600.6 Hz
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OBSERVE H1, 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 36 sec

B.
13C OBSERVE

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File: 0402

Pulse Sequence: s2pul

Solvent: cdcl3

Sample name: temperature

Sample #4

File: 0401

UNITYplus-400 "chesun4"

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Pulse 45.0 degrees

Acq. time 1.199 sec

Width 25000.0 Hz

900 repetitions

OBSERVE C13, 100.625000 MHz

DECOUPLE H1, 399.9181668 MHz

Power 36 dB

continuously on

WALTZ-16 modulated

DATA PROCESSING

Line broadening 1.0 Hz

FT size 65536

Total time 1 hr, 10 sec

MV5007A
STANDARD 1H OBSERVE

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Sample directory: auto_31Jan2005
pulse Sequence: s2pu1
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MW5007

MW5048 main gear
STANDARD 1H OBSERVE

exp2 szpu1

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at	3.744	in	n
np	4492	dp	n
bs	3416	ns	mn
dl	2.256	fn	not used
nt	16	fn	not used
ct	16	fn	not used
PROCESSING			
DISPLAY			
TRANSMITTER			
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dmm	c	th	12
dpr	42	al	cdc
dpr	29412	ph

7

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4151_MICROTHP
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Ambient temperature:
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Puls. prg. 1708sec
Puls. width 1.708 sec
Width 18761.7 Hz
128 repetitions
DECOUPLE C13, 75.48045
Power 30 dB
Continuously on
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 minutes
Date: 03/10/1906

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sv_41368_M10CPh_reCC
STANDARD_1H OBSERVE

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File: 3202

Pulse Sequence: s2pu1
Solvent: cdcl3
Ambient temperature
Sample #32
File: 3201
UNITYplus-400 "chesun4"

Relax. delay 2.256 sec
Pulse 50.0 degrees
Acq. time 3.744 sec
Width 6000.6 Hz
SFO 399.999999 MHz
OBSERVED F1 399.9161069 MHz
DATA PROCESSING
FT size 65536
Total time 1 min. 36 sec

MW5852 dried
STANDARD 1H OBSERVE

FULL spectrum

Automation directory: /export/home/walkup/auto/auto_01.04.05

File : 1702

Pulse Sequence: s2pul

11362

exp2 std1h

SAMPLE		DEC. & VT	
date	Oct 6 06	dfrq	300.053
solvent	CDCl3	dn	H1
file	exp	dm	nmn
ACQUISITION			
sfrq	300.053	dm	C
tn	H1	dmf	200
PROCESSING			
at	3.747	wtfile	
np	33728	proc	ft
sw	4500.5	fn	not used
bs	2216	werr	
pw	20.6	wexp	react
d1	2.253	wbs	procplot
tof	0	wnt	
nt	16		
ct	16		
alock a			
gain	not used		
FLAGS			
il	n		
in	y		
dp	y		
DISPLAY			
sp	-768.5		
wp	4500.5		
sc	7140		
wc	250		
hzmm	18.00		
ls	6766.44		
rfl	768.5		
rflp	40		
th	41		
is	1.000		
ai	cdc ph

```

11379
exp3 std13c
SAMPLE
date Oct 6 06 DEC. & VT
solvent CDC13 dn 300.053
file exp H1
sfrq 456 1.0
acq 456 0
t1 73.813 dmf yyy
t2 1.706 dmf 11552
np 64000
sw 18761.7 lb PROCESSING 1.00
fb 9500 wfile ft
bs 116 proc not used
dt 2.290 fn
tof 0 werr react(1)
ct 10000 wexp procp1ot
gain not used
alock a
flags not used
il n
in y
dp DISPLAY
sp -1821.7
wp 18761.7
vs 15622
sc 0
hz 250
h2am 75.05
ls 500.00
rfl 1879.2
th 0
ins 19
al no ph
  
```

11386
STANDARD 1H OBSERVE

Archive directory: /export/home/walakup/vnmrsys/data
Sample directory: auto_01Jun2006

Pulse Sequence: s2pul

Solvent: cdcl3

Ambient temperature

File: 1201

UNITYplus-400 "chesun4"

Relax. delay 2.256 sec

Phs. 50.0 degrees

Acq. time 3.794 sec

Width 6000.6 Hz

16 repetitions

OBSERVE H1, 399.9161280 MHz

DATA PROCESSING

FT size 65536

Total time 1 min, 36 sec

```

7044
exp2 stdih
SAMPLE
date Oct 5 06 dfrq DEC. & VT
solvent CDCl3 dn 300.053 H1
file exp dm 0
ACQUISITION
sfrq 300.053 dnm nnn
at 3.747 dmf PROCESSING 200
np 33728 wfile ft
sw 4500.5 proc not used
fb 2250 fn
bs 20.6 werr
pw 2.250 wexp
di 0 wps
nt 16 wnt
ct 16
alock a
gain not used
FLAGS
il n
in y
dp y
DISPLAY
sp -770.2
wp 4500.5
vs 3650
sc 0
wc 250
hsc 18.36
ls 4558.96
rf1 770.2
rfp 0
th 51
ins 1.000
a1 cdc ph

```

Figure 1. X-ray crystal structure of (diphenylphosphinoylmethyl)benzene **1**

Figure 2. X-ray crystal structure of 1,2-*bis*(diphenylphosphinonylmethyl)benzene **2**

References

1. Ningning, Z.; Neckers, D. C. *J. Org. Chem.*, **2000**, 65, 2145 - 2150
2. Aguiar, A. M.; Nair, M. G. R. *J. Org. Chem.*, **1968**, 33, 579 – 582
3. *J. Chem. Soc., Perkin Trans 1.*, **1999**, 1095 – 1103