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Non-symmetric diphosphines based on the imidazole scaffold: Unusual group interchange involving Pd-CH_3 and (imidazole)P-Ph cleavage

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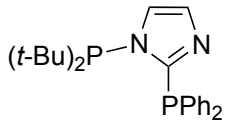
Contents

1. Synthesis and characterisation

1.1 General methods

All manipulations involving organometallics were performed under nitrogen or argon in a Braun glove-box or using standard Schlenk techniques. Solvents were dried using standard methods and distilled under nitrogen prior use or passed through columns of activated alumina and subsequently purged with nitrogen or argon. 1-(di-*t*-butylphosphino)imidazole was made as we previously reported.[1] The starting materials di-*t*-butyl-(imidazole-2-yl)phosphine[2] and [PdCl(Me)(1,5-cod)][3] were prepared according to the literature.

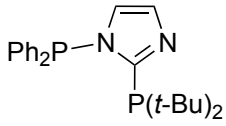
1.2 1-(di-*t*-butylphosphino)-2-(diphenylphosphino)-*1H*-imidazole (L1)



L1

A direct, higher yield synthesis of this compound compared to a previous one is described below.[1] To a solution of 1-(di-*t*-butylphosphino)imidazole (1.060 g, 5.00 mmol) in THF (20 ml) at -50 °C was added dropwise *n*-BuLi (1.6 M solution in hexane 3.2 ml, 5.12 mmol) over 1 min. The reaction mixture was stirred for 30 min until the temperature increased to -20 °C, then the yellow solution was cooled again to -50 °C and chlorodiphenylphosphine (1.103 g, 5.00 mmol) was added dropwise over 1 min. The reaction mixture was allowed to warm to room temperature and stirred overnight. Evaporation of the volatiles under reduced pressure, extraction of the residue with pentane (3×20 ml), evaporation of the extracts to dryness under reduced pressure and washing of the remaining solid with pentane (3 ml) at -50 °C gave **L1** as a colourless powder (1.754 g, 88%). X-ray quality crystals were obtained by slow cooling (-20 °C) of dilute pentane solutions for two days. Analysis: Found (Calc. for $C_{23}H_{30}N_2P_2$) (%): C, 69.42 (69.68), H, 7.52 (7.63), N, 6.90 (7.07). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.45 (dd, 1H, $^3J_{HP}$ = 2.1 Hz, $^3J_{HH}$ = 1.3 Hz, im-*H*5), 7.43-7.38 (4H, Ar-*H*), 7.32-7.29 (6H, Ar-*H*), 7.26 (t, 1H, $^3J_{HH}$ = $^4J_{HP}$ = 1.3 Hz, im-*H*4), 1.11 (d, 18H, $^3J_{HP}$ = 12.6 Hz, C(C*H*$_3$)$_3$). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 155.7 (d, $^1J_{CP}$ = 22.8 Hz, N*C*N), 137.0 (dd, $^2J_{CP}$ = 8.2 Hz, $^3J_{CP}$ = 5.3 Hz, im-*C*5), 134.5 (d, $^1J_{CP}$ = 21.0 Hz, *ipso*-C PPh_2), 132.2, 129.0, 128.5 (d, $^2J_{CP}$ = 7.3 Hz, *o*-PPh_2), 125.4 (d, $^3J_{CP}$ = 7.4 Hz, im-*C*4), 35.6 (dd, $^1J_{CP}$ = 28.7 Hz, $^4J_{CP}$ = 2.1 Hz, *C*(CH$_3$)$_3$), 29.1 (d, $^2J_{CP}$ = 16.2 Hz, C(*C*H$_3$)$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 80.0 (d, $^3J_{PP}$ = 115.4 Hz, P(*t*-Bu)$_2$), -28.2 (d, $^3J_{PP}$ = 115.4 Hz, PPh_2).

1.3 1-(diphenylphosphino)-2-(di-*t*-butylphosphino)-*1H*-imidazole (L2)

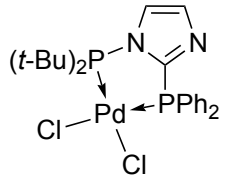


L2

To a solution of di-*t*-butyl-(imidazol-2-yl)phosphine (0.546 g, 2.57 mmol) and triethylamine (0.286 g, 2.83 mmol) in Et_2O (20 ml) at -10 °C was added a solution of diphenylchlorophosphine (0.567 g, 2.57 mmol) in Et_2O (10 ml). The reaction mixture was

allowed to warm to room temperature and stirred overnight. Evaporation of the volatiles under reduced pressure, extraction of the residue into pentane (3×15 ml), evaporation of the extracts to dryness and washing of the remaining solid with pentane (5 ml) at -40 °C gave **L2** as a colourless powder (0.480 g, 47%). X-ray quality crystals were obtained by slow cooling (-40 °C) of dilute pentane solutions for two days in the glovebox. This compound is very air-sensitive. Analysis: Found (Calc. for $C_{23}H_{30}N_2P_2$) (%): C, 69.47 (69.68); H, 7.74 (7.63); N, 6.89 (7.07). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.42- 7.36 (6H, Ar-*H*), 7.27-7.23 (5H, im-*H*4 and Ar-*H*), 6.74 (dd, 1H, $^3J_{HP}$ = 2.9 Hz, $^3J_{HH}$ = 1.2 Hz, im-*H*5), 1.19 (d, 18H, $^3J_{HP}$ = 12.3 Hz, C(C*H*$_3$)$_3$). ^{13}C{^{1}H} NMR (75 MHz, C_6D_6): δ 154.5 (t, $^1J_{CP}$ = $^2J_{CP}$ = 18.7 Hz, N*C*N), 137.7 (dd, $^2J_{CP}$ = 18.8 Hz, $^3J_{CP}$ = 3.6 Hz, im-*C*5), 133.0 (d, $^1J_{CP}$ = 22.3 Hz, *ipso*-C PPh_2), 132.8, 130.3, 129.3 (d, $^2J_{CP}$ = 6.3 Hz, *o*-PPh_2), 124.0 (d, $^3J_{CP}$ = 7.0 Hz, im-*C*4), 34.5 (dd, $^1J_{CP}$ = 18.8 Hz, $^4J_{CP}$ = 3.2 Hz, *C*(CH$_3$)$_3$), 30.9 (d, $^2J_{CP}$ = 14.6 Hz, C(*C*H$_3$)$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 36.4 (d, $^3J_{PP}$ = 112 Hz, PPh_2), 4.4 (d, $^3J_{PP}$ = 112 Hz, P(*t*-Bu)$_2$).

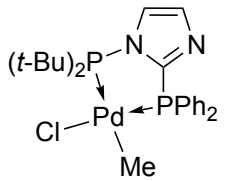
1.4 [PdCl$_2$L1] (1a)



1a

To a suspension of [$PdCl_2$(cod)] (0.051 g, 0.18 mmol) in THF (8 ml) was added a solution of **L1** (0.076 g, 0.19 mmol) in THF (8 ml) at -78 °C. The reaction mixture was allowed to warm slowly to room temperature and stirred for 2 h. After evaporation of the solvent under reduced pressure, the residue was washed with Et_2O (4 ml) and dried under vacuum to give **1a** as a pale yellow powder (0.090 g, 85%). Analysis: Found (Calc. for $C_{23}H_{30}Cl_2N_2P_2Pd$) (%): C, 47.78 (48.15); H, 5.27 (5.27); N, 4.98 (4.88). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.93-7.88 (4H, Ar-*H*), 7.71 (s, 1H, im-*H*5), 7.58-7.55 (3H, im-*H*4 and Ar-*H*), 7.48-7.43 (4H, Ar-*H*), 1.55 (d, 18H, $^3J_{HP}$ = 16.9 Hz, C(C*H*$_3$)$_3$). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 152.7 (dd, $^1J_{CP}$ = 87.8 Hz, $^2J_{CP}$ = 26.2 Hz, N*C*N), 141.5 (dd, $^2J_{CP}$ = 9.5 Hz, $^3J_{CP}$ = 3.2 Hz, im-*C*5), 134.6 (d, J_{CP} = 11.5 Hz), 132.5 (d, J_{CP} = 2.4 Hz), 128.8 (d, J_{CP} = 12.7 Hz), 127.5 (d, $^1J_{CP}$ = 68.1 Hz, *ipso*-C Ph_2P), 126.1 (dd, $^3J_{CP}$ = 6.2 Hz, $^3J_{CP'}$ = 2.4 Hz, im-*C*4), 42.3 (d, $^1J_{CP}$ = 12 Hz, *C*(CH$_3$)$_3$), 29.5 (d, $^2J_{CP}$ = 4.6 Hz, C(*C*H$_3$)$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 136.5 (d, $^{3+2}J_{PP}$ = 13.8 Hz, P(*t*-Bu)$_2$), 24.8 (d, $^{3+2}J_{PP}$ = 13.8 Hz, PPh_2).

1.5 [PdCl(Me)L1] (1b)

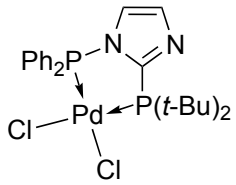


1b

To a solution of [PdCl(Me)(cod)] (0.132 g, 0.50 mmol) in THF (8 ml) was added a solution of **L1** (0.200 g, 0.50 mmol) in THF (8 ml) at -78 °C. The reaction mixture was allowed to warm slowly to room temperature and was stirred for 2 h. After evaporation of the solvent

under reduced pressure, the residue was washed with Et_2O (3 ml) and dried under vacuum to give **1b** as a white powder (0.241 g, 87%). X-ray quality crystals were obtained by slow diffusion of ether into a dichloromethane solution of **1b**. Analysis: Found (Calc. for $C_{24}H_{33}ClN_2P_2Pd$) (%): C, 52.44 (52.09); H, 6.08 (6.01); N, 5.06 (5.06). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.73-7.68 (4H, Ar-*H*), 7.57 (d, 1H, $^3J_{HP}$ = 1.2 Hz, im-*H*5), 7.53-7.49 (3H, im-*H*4 and Ar-*H*), 7.46-7.41 (4H, Ar-*H*), 1.44 (d, 18H, $^3J_{HP}$ = 15.1 Hz, C(C*H*$_3$)$_3$), 0.84 (dd, 3H, $^3J_{HPtrans}$ = 7.5 Hz, $^3J_{HPcis}$ = 3.3 Hz, PdC*H*$_3$). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 155.7 (dd, $^1J_{CP}$ = 82.1 Hz, $^2J_{CP}$ = 35.1 Hz, N*C*N), 139.3 (dd, $^2J_{CP}$ = 9.1 Hz, $^{3+4}J_{CP}$ = 2 Hz, im-*C*5), 134.3 (d, J_{CP} = 12.4 Hz), 131.8 (d, J_{CP} = 2.2 Hz), 129.5 (d, $^1J_{CP}$ = 61.7 Hz, *ipso*-C Ph_2P), 129.0 (d, J_{CP} = 12 Hz), 125.6 (t, $^3J_{CP}$ = $^3J_{CP'}$ = 4.8 Hz, im-*C*4), 39.4 (*C*(CH$_3$)$_3$), 29.3 (d, $^2J_{CP}$ = 7.4 Hz, C(*C*H$_3$)$_3$), 11.7 (d, $^2J_{CPtrans}$ = 97.8 Hz, Pd*C*H$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 114.9 (d, $^{2+3}J_{PP}$ = 37.2 Hz, P(*t*-Bu)$_2$), 27.4 (d, $^{2+3}J_{PP}$ = 37.2 Hz, PPh_2).

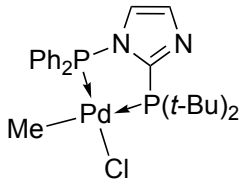
1.6 [PdCl$_2$L2] (2a)



2a

To a suspension of [$PdCl_2$(cod)] (0.051 g, 0.18 mmol) in THF (8 ml) was added a solution of **L2** (0.076 g, 0.19 mmol) in THF (8 ml) at -78 ^{o}C. The reaction mixture was allowed to warm slowly to room temperature and stirred for 2 h. Evaporation of the volatiles under reduced pressure, extraction of the residue with CH_2Cl_2 (10 ml), evaporation of the extracts to dryness and washing of the remaining solid with Et_2O (4 ml) gave **2a** as a pale yellow powder (0.085 g, 80%). X-ray quality crystals were obtained by slow diffusion of ether into dichloromethane solution of **2a**. Analysis: Found (Calc. for $C_{23}H_{30}Cl_2N_2P_2Pd$) (%): C, 47.75 (48.15); H, 5.28 (5.27); N, 4.64 (4.88). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.80-7.74 (4H, Ar-*H*), 7.68-7.64 (3H, im-*H*4 and Ar-*H*), 7.56-7.51 (4H, Ar-*H*), 7.10 (d, 1H, $^3J_{HP}$ = 1.4 Hz, im-*H*5), 1.59 (d, 18H, $^3J_{HP}$ = 16.6 Hz, C(C*H*$_3$)$_3$). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 149.8 (dd, $^1J_{CP}$ = 64.3 Hz, $^2J_{CP}$ = 32.6 Hz, N*C*N), 141.4 (dd, $^2J_{CP}$ = 7.3 Hz, $^{3+4}J_{CP}$ = 5.1 Hz, im-*C*5), 133.9 (d, J_{CP} = 12.5 Hz), 133.6 (d, J_{CP} = 2.3 Hz), 129.2 (d, J_{CP} = 12.6 Hz), 128.3 (d, $^1J_{CP}$ = 65.1 Hz, *ipso*-C Ph_2P), 122.7 (d, $^3J_{CP}$ = 4.3 Hz, im-*C*4), 40.6 (d, $^1J_{CP}$ = 18.6 Hz, *C*(CH$_3$)$_3$), 29.9 (d, $^2J_{CP}$ = 3.3 Hz, C(*C*H$_3$)$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 76.2 (d, $^{2+3}J_{PP}$ = 19.4 Hz, PPh_2), 73.4 (d, $^{2+3}J_{PP}$ = 19.4 Hz, P(*t*-Bu)$_2$).

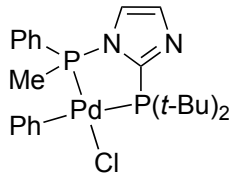
1.7 [PdCl(Me)L2] (2b) and [PdCl(Ph) Ph(Me)PNCH=CHNCP(*t*-Bu)$_2$] (2c)



2b

To a solution of [PdCl(Me)(cod)] (0.098 g, 0.37 mmol) in THF (8 ml) was added a solution of **L2** (0.150 g, 0.38 mmol) in THF (8 ml) at -78 ^{o}C. The reaction mixture was allowed to warm slowly to room temperature and was stirred for 2 h. The reaction mixture was concentrated to 2 ml under reduced pressure, and Et_2O was added (10 ml). After

supernatant was removed, the solid was dried under vacuum to give **2b** as a white powder (0.164 g, 80%). X-ray quality crystals were obtained by slow diffusion of Et_2O into THF solution of **2b** at -38 °C. Analysis: Found (Calc. for $C_{24}H_{33}ClN_2P_2Pd$) (%): C, 51.54 (52.09), H, 5.83 (6.01), N, 5.39 (5.06). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.62-7.57 (6H, Ar-*H*), 7.53-7.48 (5H, im-*H*4 and Ar-*H*), 7.08 (d, 1H, $^3J_{HP}$ = 1.3 Hz, im-*H*5), 1.45 (d, 18H, $^3J_{HP}$ = 14.8 Hz, C(CH_3)$_3$), 0.7 (dd, 3H, $^3J_{HPtrans}$ = 7.0 Hz, $^3J_{HPcis}$ = 4.6 Hz, PdCH_3). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 152.2 (dd, $^1J_{CP}$ = 46.1 Hz, $^2J_{CP}$ = 26.9 Hz, N*C*N), 139.4 (t, $^2J_{CP}$ = $^{3+4}J_{CP}$ = 5.5 Hz, im-*C*5), 133.5 (d, J_{CP} = 13.7 Hz), 132.9 (d, J_{CP} = 2.0 Hz), 130.3 (d, $^1J_{CP}$ = 58 Hz, *ipso*-C, PPh_2), 129.5 (d, J_{CP} = 11.9 Hz), 122.5 (d, J_{CP} = 5.1 Hz, im-*C*4), 37.6 (d, $^1J_{CP}$ = 9.7 Hz, *C*(CH$_3$)$_3$), 30.0 (d, $^2J_{CP}$ = 5.8 Hz, C(*C*H$_3$)$_3$), 12.3 (d, $^2J_{CPtrans}$ = 90.6 Hz, Pd*C*H$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 85.9 (d, $^{2+3}J_{PP}$ = 35.4 Hz, PPh_2), 41.4 (d, $^{2+3}J_{PP}$ = 35.4 Hz, P(*t*-Bu)$_2$).



2c

The complex **2b** is not stable in THF or CH_2Cl_2 solution for a long period of time and is completely and cleanly transformed to **2c** after 5 days. X-ray quality crystals were obtained by slow diffusion of pentane into a THF solution of **2b** at room temperature. Solid **2c** is air stable. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.77 (dd, 1H, *J* = 13.4 Hz, *J* = 8.5 Hz), 7.54 (d, 1H, *J* = 1.1 Hz), 7.43-7.34 (3H), 7.18-7.13 (2H), 7.08-7.03 (2H), 6.95 (quart., 1H, *J* = 1.5 Hz), 6.93-6.90 (2H), 2.04 (d, 3H, $^2J_{HP}$ = 9.7 Hz, P-CH_3), 1.56 (dd, 18H, $^3J_{HP}$ = 20.4 Hz, $^5J_{HP}$ = 14.9 Hz, C(CH_3)$_3$). ^{13}C{^{1}H} NMR (125 MHz, CD_2Cl_2): δ 157.1 (dd, $^1J_{CP}$ = 113.7 Hz, $^2J_{CP}$ = 3.6 Hz, N*C*N), 139.8 (t, $^2J_{CP}$ = $^{3+4}J_{CP}$ = 5.5 Hz, im-*C*5), 136.8 (d, J_{CP} = 3.5 Hz), 134.2 (d, J_{CP} = 12.6 Hz), 133.4 (d, J_{CP} = 13.5 Hz), 132.4 (d, J_{CP} = 1.9 Hz), 129.2 (d, J_{CP} = 11.6 Hz), 128.0 (d, J_{CP} = 7.7 Hz), 124.2, 121.5 (d, J_{CP} = 3.9 Hz, im-*C*4), 37.5 (dd, $^1J_{CP}$ = 27.6 Hz, $^{3+4}J_{CP}$ = 10.5 Hz, *C*(CH$_3$)$_3$), 29.9 (dd, $^2J_{CP}$ = 19.7 Hz, $^{4+5}J_{CP}$ = 5.9 Hz, C(*C*H$_3$)$_3$), 15.9 (d, $^1J_{CP}$ = 37.4 Hz, P-*C*H$_3$). ^{31}P{^{1}H} NMR (161 MHz, CD_2Cl_2): δ 72.1 (d, $^{2+3}J_{PP}$ = 35.3 Hz, PPh_2), 42.0 (d, $^{2+3}J_{PP}$ = 35.3 Hz, P(*t*-Bu)$_2$).

2. X-ray crystallography

Summary of the crystal data, data collection and refinement for structures of **L1**, **L2**, **1b, 2a**, **2b** and **2c** are given in Table S1. For **L1**, **L2**, **2a** and **2b**, X-ray diffraction data collection was carried out on a Bruker APEX II DUO Kappa-CCD diffractometer equipped with an Oxford Cryosystem liquid N_2 device, using Mo-Kα radiation (λ = 0.71073 Å). The crystal-detector distance was 38 mm. The cell parameters were determined (APEX2 software)[4] from reflections taken from three sets of 12 frames, each at 10s exposure. The structure was solved by Direct methods using the program SHELXS-97.[5] The refinement and all further calculations were carried out using SHELXL-97.[6] The H-atoms were included in calculated positions and

treated as riding atoms using SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-squares on F^2. A semi-empirical absorption correction was applied using SADABS in APEX2.[4]

For **1b** and **2c**, X-ray diffraction data collection was carried out on a Nonius Kappa-CCD diffractometer equipped with an Oxford Cryosystem liquid N_2 device, using Mo-Kα radiation (λ = 0.71073 Å). The crystal-detector distance was 36 mm. The cell parameters were determined (Denzo software)[7] from reflections taken from one set of 10 frames (1.0° steps in phi angle), each at 20s exposure. The structures were solved by Direct methods using the program SHELXS-97.[5] The refinement and all further calculations were carried out using SHELXL-97.[6] The H-atoms were included in calculated positions and treated as riding atoms using SHELXL default parameters. The non-H atoms were refined anisotropically, using weighted full-matrix least-squares on F^2. A semi-empirical absorption correction was applied using MULscanABS in PLATON.[8] In **2c**, a squeeze was made. The residual electron density was assigned to half a molecule of pentane.

Table S1. Crystal data for compounds **L1**, **L2, 1b, 2b** and **2c**.

	L1	**L2**	**1b**	**2a**	**2b**	**2c**
Chemical formula	C_{23} H_{30} N_2 P_2	C_{23} H_{30} N_2 P_2	C_{24} $H_{33}Cl$ N_2 P_2 Pd	C_{23} H_{30} Cl_2 N_2 P_2 Pd	C_{24} $H_{33}Cl$ N_2 P_2 Pd	C_{24} $H_{33}Cl$ N_2 P_2 Pd
CCDC Number	968367	968368	968369	968370	968371	968372
Formula Mass	396.43	396.43	553.31	573.73	553.31	553.31
Crystal system	Monoclinic	Orthorhombic	Monoclinic	Orthorhombic	Monoclinic	Trigonal
a/Å	17.4036(7)	12.1693(18)	8.0415(2)	9.7407(3)	8.7612(5)	25.9649(6)
b/Å	9.2806(3)	21.844(3)	14.3830(4)	14.4492(4)	36.145(2)	25.9649(6)
c/Å	16.0476(6)	8.3719(11)	21.4302(5)	17.6018(5)	9.6899(4)	20.5993(7)
α/°	90	90	90	90	90	90
β/°	121.1790(10)	90	96.664(2)	90	123.139(4)	90
γ/°	90	90	90	90	90	120
Unit cell volume/Å^3	2217.55(14)	2225.5(5)	2461.89(11)	2477.37(12)	2569.4(2)	12027.0(6)
Temperature/K	173(2)	173(2)	173(2)	173(2)	173(2)	173(2)
Space group	*C c*	*P n a 21*	*P 21/c*	*P 21 21 21*	*P 21/c*	*R -3*
Formula units / cell, *Z*	4	4	4	4	4	18
Absorption coefficient, μ/mm^{-1}	0.206	0.205	1.006	1.107	0.964	0.927
No. of reflections measured	14989	16073	15023	24097	29287	24630
No. of independent reflections	7200	4118	5627	7875	7465	6126
R_{int}	0.0213	0.0784	0.0486	0.0221	0.0697	0.0796
Final R_1 values ($I > 2\sigma(I)$)	0.0338	0.0444	0.0314	0.0190	0.0671	0.0477
Final $wR(F^2)$ values ($I > 2\sigma(I)$)	0.0762	0.0863	0.0783	0.0412	0.1005	0.1248
Final R_1 values (all data)	0.0425	0.0747	0.0401	0.0222	0.1024	0.0872
Final $wR(F^2)$ values (all data)	0.0805	0.0953	0.0849	0.0423	0.1078	0.1401
Goodness of fit on F^2	1.058	1.017	1.086	1.053	1.185	1.053

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