Supplementary Information

Activity of \(N,N'\)-dialkyl-2-trifluoromethylthioimidazolium salts as phase-transfer catalysts for the alkylation of active methylene compounds

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1. General Information

All NMR spectra were recorded on Varian 500PS spectrometers. \(^1\)H \(^13\)C and \(^19\)F NMR spectra are reported as chemical shifts (\(\delta\)) in parts per million (ppm) relative to the solvent peak using tetramethylsilane, and trichlorofluoromethane (\(^19\)F) as an internal standard. Chemical shifts (\(\delta\)) are quoted in parts per million (ppm) and coupling constants (\(J\)) are measured in hertz (Hz). The following abbreviations are used to describe multiplicities s=singlet, d=doublet, t=triplet, q=quartet,
quint.=quintet, sext.=sextet, sept.=septet br=broad, m=multiplet. NMR spectra were processed in ACD/SpecManager. High resolution mass spectra (HRMS, m/z) were obtained on JEOL JMS-700N for FAB using m-nitrobenzylalcohol as a matrix or on JEOL JMS-T100TD for electrospray ionization (ESI+).

All reactions were performed in apparatuses with magnetic stirring under an inert atmosphere. Flash column chromatography was performed over Fuji Silysia Chemical Ltd. silica gel C60 (50-200 μm) using an eluent system as described for each experiment. Thin-layer chromatography was performed using TLC Silica gel 60 F254 aluminum sheets (Merck) and silica gel F254 glass plates (Merck).

Materials.
Imidazole-2(3H)-thiones 3, Methyl 1-indanone-2-carboxylate 5 and Methyl 1-methyl-2-oxoindoline-3-carboxylate 6 were known compounds and were prepared according literature procedures.[1-3] Unless noted otherwise, all starting materials and reagents were obtained from commercial suppliers and were used without further purification. All chemicals were purchased from Aldrich, Nacalai tesque, Tokyo Chemical Industry and Wako Pure Chemical Industries and used as received. All solvents were purchased from Wako Pure Chemical Industries.

2. Experimental Procedures and Characterization Data

2-1. General procedure for synthesis of \(N,N'\)-dialkyl-2-trifluoromethylthioimidazolium salts

\[
\begin{align*}
\text{S} & \quad \text{X}^+ \\
& \quad \text{S} \quad \text{N} \quad \text{N} \\
\text{Me} & \quad \text{Me} \\
\text{R} \quad \text{R} & \quad \text{MeCN} & \quad 80^\circ C, 16 h
\end{align*}
\]

A 30 mL test tube was charged with a solution of imidazole-2(3H)-thiones 3 (1.0 mmol) and S-(trifluoromethyl)dibenzothiophenium salts 1 (1.0 mmol) in MeCN (5.0 mL). After a cap is mounted, the mixture was heated to 80 °C and stirred for 16 h. The reaction mixture was cooled to room temperature followed by evaporation to remove the solvent. The residue was purified by SiO\(_2\) gel column chromatography to afford the corresponding \(N,N'\)-dialkyl-2-trifluoromethylthioimidazolium salts.
**1,3,4,5-Tetramethyl-2-trifluoromethylthioimidazolium tetrafluoroborate (4a)**

Synthesized following procedure. A mixture of imidazole-2(3H)-thiones 3a (150 mg, 1.0 mmol), and S-(trifluoromethyl)dibenzothiophenium salts 1a (340 mg, 1.0 mmol) in MeCN (5.0 mL) was stirred at 80 °C for 16hr. Purified by SiO₂ gel column chromatography (DCM : MeOH=90 : 10) to give 200 mg (64% yield) of the title compounds as a brown solid. ¹H NMR (500 MHz, CDCl₃) δ 2.39 (s, 6H), 3.91 (s, 6H); ¹³C NMR (125 MHz, CDCl₃) δ 9.43, 34.2, 126.7 (m), 126.9 (q, J = 313Hz), 132.8; ¹⁹F NMR (470 MHz, CDCl₃) δ -40.3 (s, 3F, S-CF₃), -155.2 (s, 10BF₄), -155.2 (s, 11BF₄); HRMS (FAB) m/z Calcd for C₈H₁₂F₃N₂S [M]⁺ 225.0668, found 225.0672.

**1,3-Dibutyl-4,5-dimethyl-2-trifluoromethylthioimidazolium tetrafluoroborate (4b)**

Synthesized following procedure. A mixture of imidazole-2(3H)-thiones 3b (381 mg, 1.5 mmol), and S-(trifluoromethyl)dibenzothiophenium salts 1a (510 mg, 1.5 mmol) in MeCN (8.0 mL) was stirred at 80 °C for 16hr. Purified by SiO₂ gel column chromatography (DCM : MeOH=95 : 5) to give 257 mg (43% yield) of the title compounds as a brown oil. ¹H NMR (500 MHz, CDCl₃) δ 1.01 (t, J = 7.6 Hz, 6H), 1.47 (q, J = 7.4 Hz, 4H), 1.75–1.85 (m, 4H), 2.41 (s, 6H), 4.29 (t, J = 8.3 Hz, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 9.61, 13.3, 19.8, 31.0, 47.9, 124.8 (m), 126.5 (q, J = 313 Hz), 132.9; ¹⁹F NMR (470 MHz, CDCl₃) δ -39.4 (s, 3F, S-CF₃), -154.6 (s, 10BF₄), -154.6 (s, 11BF₄); HRMS (FAB) m/z Calcd for C₁₄H₂₄F₃N₂S [M]⁺ 309.1607, found 309.1612.

**1,3-Diisopropyl-4,5-dimethyl-2-trifluoromethylthioimidazolium tetrafluoroborate (4c)**

Synthesized following procedure. A mixture of imidazole-2(3H)-thiones 3c (1.0 g, 4.7 mmol), and S-(trifluoromethyl)dibenzothiophenium salts 1a (1.6 g, 1.5 mmol) in MeCN (25.0 mL) was stirred at 80 °C for 16hr. Purified by SiO₂ gel column chromatography (DCM : MeOH=95 : 5) to give 796 mg (46% yield) of the title compound as a white solid. Single crystals suitable for X-ray crystallography were grown from a mixture solution of MeOH/DCM. ¹H NMR (500 MHz, CDCl₃) δ 1.63 (d, J = 4.9 Hz, 6H), 1.75 (d, J = 5.2 Hz, 6H), 2.51 (s, 6H), 5.37 (sept., 2H); ¹³C NMR (125 MHz, CDCl₃) δ 10.7, 20.5, 20.7, 54.6, 126.6 (q, J = 313Hz), 133.0; ¹⁹F NMR (470 MHz, CDCl₃) δ -40.6 (s, 3F, S-CF₃), -153.6 (s, 10BF₄), -153.6 (s, 11BF₄); HRMS (FAB) m/z Calcd for C₁₂H₂₀F₃N₂S [M]⁺ 281.1294, found 281.1299.

**1,3-Diisopropyl-4,5-dimethyl-2-trifluoromethylthioimidazolium trifluoromethanesulfonate**
Synthesized following procedure. A mixture of imidazole-2(3H)-thiones 3c (212 mg, 1.0 mmol), and S-(trifluoromethyl)dibenzo thiophenium salts 1b (402 mg, 1.0 mmol) in MeCN (5.0 mL) was stirred at 80 °C for 16 hr. Purified by SiO₂ gel column chromatography (DCM : MeOH = 95 : 5) to give 134 mg (31 % yield) of the title compound as a white solid. 

\[ ^{1}H \text{ NMR (500 MHz, CDCl}_3 \delta 1.60–1.76 \text{ (m, 12H), 2.50} \text{ (s, 6H), 5.35} \text{ (sept., 2H); } ^{13}C \text{ NMR (125 MHz, CDCl}_3 \delta 10.8, 20.5, 20.9, 54.6, 126.6} \text{ (q, J } = 313 \text{Hz), 132.7; } ^{19}F \text{ NMR (470 MHz, CDCl}_3 \delta -40.6} \text{ (s, 3F, S-CF}_3 \text{), -78.4} \text{ (s, OTf); HRMS (FAB) m/z Calcd for C}_{12}H_{20}F_3N_2S [M]^{+} 281.1294, \text{ found 281.1297.} \]

**1,3-Dicyclohexyl-4,5-dimethyl-2-trifluoromethylthioimidazolium tetrafluoroborate (4e)**

Synthesized following procedure. A mixture of imidazole-2(3H)-thiones 3d (439 mg, 1.5 mmol), and S-(trifluoromethyl)dibenzo thiophenium salts 1a (510 mg, 1.5 mmol) in MeCN (8.0 mL) was stirred at 80 °C for 16 hr. Purified by SiO₂ gel column chromatography (DCM : MeOH = 95 : 5) to give 302 mg (67% yield) of the title compounds as a brown oil.

\[ ^{1}H \text{ NMR (500 MHz, CDCl}_3 \delta 1.22–1.28 \text{ (m, 2H), 1.40–1.46} \text{ (m, 4H), 1.77–2.29} \text{ (m, 14H), 2.50} \text{ (s, 6H), 4.82–4.97} \text{ (m, 2H); } ^{13}C \text{ NMR (125 MHz, CDCl}_3 \delta 11.2, 24.3, 24.8, 25.4, 25.8, 25.9, 26.1, 30.1, 30.7, 32.6, 62.9, 126.5} \text{ (q, J } = 313 \text{Hz), 133.1; } ^{19}F \text{ NMR (470 MHz, CDCl}_3 \delta -41.0} \text{ (s, 3F, S-CF}_3 \text{), -153.6} \text{ (s, } ^{10}\text{BF}_4 \text{), -153.7} \text{ (s, } ^{11}\text{BF}_4 \text{); HRMS (FAB) m/z Calcd for C}_{18}H_{28}F_3N_2S [M]^{+} 361.1920, \text{ found 361.1924.} \]

**2-2. Experiment of benzylation of 5 in the presence of 4c**

To verify the catalytic activity of 4c, the benzylation of indanone carboxylate 5 with KOH as base in the presence of a catalytic amount of 4c was performed. The rate of reaction to convert 5 into benzylation product 6 was confirmed. The reaction was completed within 2 hrs. In contrast, the reaction in the absence of catalyst afford the product 6 in 47% after 24 h duration. These results demonstrated the efficacy of 4c as phase- transfer catalyst.
Indanone 5 (47.5 mg, 0.25 mmol), benzyllbromide (45 μl, 0.38 mmol), 4c (9.2 mg, 0.025 mmol), and KOH (42 mg, 0.75 mmol) in CHCl₃ (1.2 mL) was stirred for 2 hrs.

2-3. Experiment of benzylation of 7 and reaction of diethylmalonate and 1,4-dibromobutane without imidazolium salt catalyst 4c

These experiments that includes a comparison of reaction under conditions without catalyst 4c performed, and these results demonstrated the efficacy of 4c as phase- transfer catalyst in comparison with that showed in Table 1.

Indanone 7(41 mg, 0.20 mmol), benzyllbromide (26 μl, 0.22 mmol) and KOH (34 mg, 0.60 mmol) in CHCl₃ (1.0 mL) was stirred for 24 hrs. When the reaction was stirred for 1 hr, the isolated yield was 12%. The duration 24 hrs gave the product in 58% yield.
A mixture of dimethylmalonate (39.0 mg, 0.30 mmol), 1,4-dibromobutane (39 μl, 0.33 mmol) and K₂CO₃ (104 mg, 0.75 mmol) in DMF (1.5 mL) was stirred for 24 hrs. When the reaction was stirred for 8 hr, the isolated yield was 30%. The duration 24 hrs gave the product in 74% yield. In contrast, the reaction using catalyst 4c stirring for 8 hour afford the product 10 in 83% duration (Table 1).

2-4. General procedure for the alkylation of α-keto esters

General procedure A: GP-A
To a stirred suspension of active methylene compounds, 4c (10 mol%), and KOH (3.0 equiv) in CHCl₃ (0.2 M) was added alkyl halide (1.5 equiv) at room temperature. After the reaction completed, the mixture was diluted with sat. NH₄Cl solution and extracted EtOAc. The organic layers were dried over MgSO₄, filtered, concentrated. The residue was purified by column chromatography on SiO₂ gel to afford the alkylation product.

General procedure A: GP-B
To a stirred suspension of active methylene compounds, 4c (10 mol%), and K₂CO₃ (3.0 equiv) in DMF (0.2 M) was added alkyl halide (1.5 equiv) at room temperature. After the reaction completed, the mixture was diluted with sat. NH₄Cl solution and extracted EtOAc. The organic layers were dried over MgSO₄, filtered, concentrated. The residue was purified by column chromatography on SiO₂ gel to afford the alkylation product.

Table 1. Alkylation of a variety of active methylene compounds using 4c as a catalyst
Methyl 2-benzyl-1-oxo-2,3-dihydro-1H-indene-2-carboxylate (6a)

Synthesized following GP-A (0.25 mmol scale). A mixture of indanone 5 (47.5 mg, 0.25 mmol), benzylbromide (45 μl, 0.38 mmol), 4c (9.2 mg, 0.025 mmol) and KOH (42 mg, 0.75 mmol) in CHCl₃ (1.2 mL) was stirred for 1hr. Purified by SiO₂ gel column chromatography (acetone : n-hexane=1 : 9) to give 65.8 mg (94 % yield) of the title compound as a colorless oil.

1H NMR (500 MHz, CDCl₃) δ 3.18 (d, J = 17.1 Hz, 1H), 3.30 (d, J = 14.1 Hz, 1H), 3.50 (d, J = 14.0 Hz, 1H), 3.63 (d, J = 17.1 Hz, 1H), 3.73 (s, 3H), 7.13–7.20 (m, 5H), 7.34–7.37 (m, 2H), 7.54 (dt, J = 1.2 , 7.6 Hz, 1H), 7.75 (d, J = 7.6 Hz, 1H); 13C NMR (125 MHz, CDCl₃) δ 35.4, 39.7, 52.9, 124.7, 126.2, 126.8, 127.6, 128.3, 129.9, 135.3, 136.3, 153.1, 171.2, 202.1. NMR data were corresponded with those previously described.⁴

Methyl 1-oxo-2-(prop-2-yn-1-yl)-2,3-dihydro-1H-indene-2-carboxylate (6b)
Synthesized following GP-A (0.25 mmol scale). A mixture of indanone 5 (47.5 mg, 0.25 mmol), propargyl bromide (28 μl, 0.38 mmol), 4c (9.2 mg, 0.025 mmol) and KOH (42 mg, 0.75 mmol) in CHCl₃ (1.2 mL) was stirred for 6 hrs. Purified by SiO₂ gel column chromatography (EtOAc : n-hexane=1 : 4) to give 54.0 mg (95 % yield) of the title compound as a yellow oil.

**1H NMR** (500 MHz, CDCl₃) δ 1.82 (t, J = 2.7 Hz, 1H), 2.84 (dd, J = 2.7, 16.9 Hz, 1H), 3.01 (dd, J = 2.7, 16.9 Hz, 1H), 3.39 (d, J = 17.3 Hz, 1H), 3.68 (s, 3H), 3.70 (d, J = 17.3 Hz, 1H), 7.39–7.42 (m, 1H), 7.52 (dt, J = 1.0, 7.8 Hz, 1H), 7.65 (dt, J = 1.2, 7.6 Hz, 1H), 7.78–7.79 (m, 1H);

**13C NMR** (125 MHz, CDCl₃) δ 23.9, 36.6, 52.9 , 58.9 , 70.5, 79.2, 124.8, 126.3, 127.8, 135.6, 153.3, 170.4, 200.8.

**Methyl 3-benzyl-1-methyl-2-oxoindoline-3-carboxylate (8a)**

Synthesized following GP-A (0.20 mmol scale). A mixture of 7 (41.0 mg, 0.20 mmol), benzyl bromide (36 μl, 0.30 mmol), 4c (7.4 mg, 0.02 mmol) and KOH (34 mg, 0.60 mmol) in CHCl₃ (1.0 mL) was stirred for 30 min. Purified by SiO₂ gel column chromatography (EtOAc : n-hexane=1 : 2) to give 56.0 mg (95 % yield) of the title compound as a white solid.

**1H NMR** (500 MHz, CDCl₃) δ 2.95 (s, 3H), 3.56 (s, 2H), 3.71 (s, 3H), 6.58 (d, J=7.8 Hz, 1H), 6.84–6.89 (m, 2H), 6.99–7.05 (m, 3H), 7.08 (dt, J = 1.0, 7.6 Hz, 1H), 7.23 (dt, J = 1.2, 7.6 Hz, 1H), 7.33–7.35 (m, 1H);

**13C NMR** (125 MHz, CDCl₃) δ 26.1, 40.0, 53.2, 60.7, 108.1, 122.5, 123.9, 126.7, 127.2, 127.5, 129.0, 129.9, 134.2, 143.9, 169.7, 173.3. NMR data were corresponded with those previously described.²

**Methyl 1-methyl-2-oxo-3-(prop-2-yn-1-yl)indoline-3-carboxylate (8b)**

Synthesized following GP-A (0.20 mmol scale). A mixture of 7 (41.0 mg, 0.20 mmol), propargyl bromide (23 μl, 0.30 mmol), 4c (7.4 mg, 0.02 mmol) and KOH (34 mg, 0.60 mmol) in CHCl₃ (1.0 mL) was stirred for 30 min. Purified by SiO₂ gel column chromatography (EtOAc : n-hexane=1 : 4) to give 46.6 mg (96 % yield) of the title compound as a yellow solid.

**1H NMR** (500 MHz, CDCl₃) δ 1.78 (t, J = 2.7 Hz, 1H), 3.04 (dd, J = 2.7, 16.6 Hz, 1H), 3.20 (dd, J = 2.7, 16.6 Hz, 1H), 3.27 (s, 3H), 3.67 (s, 3H), 6.90 (d, J = 7.8 Hz, 1H), 7.11 (dt, J = 0.8, 7.6 Hz, 1H), 7.33 (d, J = 7.6 Hz, 1H), 7.38 (dt, J = 1.3, 7.6 Hz, 1H);

**13C NMR** (125 MHz, CDCl₃) δ 24.2, 26.5, 53.2, 57.8, 70.6, 77.9, 108.4, 123.0, 123.5, 127.0, 129.5, 168.6, 172.8.

**Dimethyl 2,2-dibenzylmalonate (9)**

Synthesized following GP-B (0.25 mmol scale). A mixture of dimethylmalonate (33.0 mg, 0.25 mmol), benzyl bromide (89 μl, 0.75 mmol), 4c (9.2 mg, 0.025 mmol) and K₂CO₃ (104 mg, 0.75 mol)
mmol) in DMF (1.2 mL) was stirred for 10 hrs. Purified by SiO$_2$ gel column chromatography (EtOAc : n-hexane=1 : 4) to give 77.3 mg (99 % yield) of the title compound as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 3.23 (s, 4H), 3.64 (s, 6H), 7.16 (d, $J = 7.1$Hz, 4H), 7.24–7.29 (m, 6H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 39.2, 52.2, 60.4, 127.0, 128.2, 130.0, 136.1, 171.2. NMR data were corresponded with those previously described.$^6$

**Dimethyl cyclopentane-1,1-dicarboxylate (10)**

Synthesized following GP-B (0.20 mmol scale). A mixture of dimethylmalonate (39.0 mg, 0.30 mmol), 1,4-dibromobutane (39 $\mu$l, 0.33 mmol), 4c (11.0 mg, 0.03 mmol) and K$_2$CO$_3$ (104 mg, 0.75 mmol) in DMF (1.5 mL) was stirred for 8 hrs. Purified by SiO$_2$ gel column chromatography (EtOAc : n-hexane=1 : 4) to give 46.3 mg (83 % yield) of the title compound as a colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 1.67–1.70, 2.18–2.20, 3.72 (s, 6H). NMR data were corresponded with those previously described.$^7$

**5,5-Dibenzyl-2,2-dimethyl-1,3-dioxane-4,6-dione (11)**

Synthesized following GP-B (0.25 mmol scale). A mixture of Merdrum's acid (36.0 mg, 0.25 mmol), benzyl bromide (89 $\mu$l, 0.75 mmol), 4c (9.2 mg, 0.025 mmol) and K$_2$CO$_3$ (104 mg, 0.75 mmol) in DMF (1.2 mL) was stirred for 8 hrs. Purified by SiO$_2$ gel column chromatography (EtOAc : n-hexane=1 : 2) to give 74.3 mg (92 % yield) of the title compound as a yellow solid.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 0.63 (s, 6H), 3.46 (s, 4H), 7.20–7.28 (m, 10H); $^{13}$C NMR (125 MHz, CDCl$_3$) $\delta$ 28.5, 44.9, 60.0, 105.8, 127.7, 128.8, 130.1, 134.8, 168.1. NMR data were corresponded with those previously described.$^8$

**2-5. Experiment of equilibrium between 4c with KOH**

Imidazolium salt 4c (11.0 mg, 0.03 mmol), KOH (16.8 mg 0.30 mmol) and CHCl$_3$ (1.0 mL) were placed in a vial which was equipped with a magnetic stir bar. The mixture was stirred for 1 and 10 hrs. The reaction mixture was quenched with 1M HCl aq., and the aqueous phase was extracted with AcOEt. The combined organic phases were washed with water and brine, then dried over MgSO$_4$, filtered and concentrated in vacuo. The relative intensity of peaks was determined by $^{19}$F NMR analysis of the crude mixture. The result indicated that most of 4c remained and ca.10% of 4c converted into likely ion exchange compound when stirring for 1hour. Besides, trace amount of thiourea ([M+1]$^+$ = 213) and unidentified products (243, 393) were detectable by Mass spectrometry, but not urea derivative. When stirring for stirring 10 hours, the reaction resulted in the
decomposition to give multi-compounds. In addition, the characterization of 4c for sufficient alkaline stability was not ascertained.

(top) $^{19}$F NMR spectrum of 4c. (middle) $^{19}$F NMR spectrum of crude mixture for while stirring for 1h. (bottom) $^{19}$F NMR spectrum of crude mixture for while stirring for 10 hrs.

3. Crystal data for 4c
Figure1. ORTEP drawing of the X-ray structure of 4c. Hydrogen atoms are omitted for clarity and ellipsoids displayed at 50% probability. Selected bond distances (Å), angles [deg] and torsional angles[deg] in 4c, C1–S1, 1.732 (2); S1–C12, 1.808 (3); F1–C12, 1.323(3); F2–C12, 1.342(3); F3–C12, 1.333(3); C1–S1–C12, 97.48 (11); C12–S1–C1-N1, −93.99 (19); C12–S1–C1-N2, 89.66 (19)

A. Crystal Data

Empirical Formula C_{12}H_{20}BF_{7}N_{2}S
Formula Weight 368.16
Crystal Color, Habit colorless, block
Crystal Dimensions 0.150 X 0.070 X 0.040 mm
Crystal System monoclinic
Lattice Type Primitive
Lattice Parameters
\begin{align*}
a &= 7.926(2) \text{ Å} \\
b &= 15.738(5) \text{ Å} \\
c &= 13.422(4) \text{ Å} \\
\beta &= 100.404(3) ^\circ \\
V &= 1646.7(8) \text{ Å}^3 \\
\end{align*}

Space Group P2_1/n (\#14)
Z value 4
D_{calc} 1.485 g/cm^3
F_{000} 760.00
\mu (\text{MoK}\alpha) 2.643 cm^{-1}

B. Intensity Measurements

Diffractometer Saturn724
Radiation MoK\alpha (\lambda = 0.71075 \text{ Å})
multi-layer mirror monochromated
Voltage, Current 50kV, 24mA
Temperature -180.0^\circ C
Detector Aperture 72.8 x 72.8 mm
Data Images 720 exposures
\omega oscillation Range (\chi=45.0, \phi=0.0) -110.0 - 70.0^\circ
Exposure Rate 16.0 sec./^\circ
Detector Swing Angle: -20.09°

ω oscillation Range (χ=45.0, φ=90.0): -110.0 - 70.0°

Exposure Rate: 16.0 sec./°

Detector Swing Angle: -20.09°

Detector Position: 44.99 mm

Pixel Size: 0.141 mm

$2\theta_{\text{max}}$: 54.9°

No. of Reflections Measured:
- Total: 13471
- Unique: 3722 (R(int) = 0.0402)

Corrections:
- Lorentz-polarization
- Absorption
  (trans. factors: 0.873 - 0.989)

**C. Structure Solution and Refinement**

Structure Solution: Direct Methods (SHELXS2013)

Refinement: Full-matrix least-squares on F²

Function Minimized: $\sum w (F_o^2 - F_c^2)^2$

Least Squares Weights:
$$ w = \frac{1}{\left[ \sigma^2(F_o^2) + (0.0576 \cdot P)^2 \right] + 1.0654 \cdot P} $$

where $P = (\text{Max}(F_o^2,0) + 2F_c^2)/3$

$2\theta_{\text{max}}$ cutoff: 54.9°

Anomalous Dispersion: All non-hydrogen atoms

No. Observations (All reflections): 3722

No. Variables: 214

Reflection/Parameter Ratio: 17.39

Residuals: R1 (I>2.0σ(I)): 0.0516

Residuals: R (All reflections): 0.0685

Residuals: wR2 (All reflections): 0.1267

Goodness of Fit Indicator: 1.074

Max Shift/Error in Final Cycle: 0.001

Maximum peak in Final Diff. Map: 0.60 e/Å³

Minimum peak in Final Diff. Map: -0.34 e/Å³
4. References


5. NMR spectra