Supporting Information

for

Diastereoselectivity in the Staudinger reaction of

pentafluorosulfanylaldimines and ketimines

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Detailed experimental procedures and spectroscopic data for 1a-e, 10, 5a-d, 7a-e and 11

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General methods:

All reactions were carried out under an inert atmosphere. Reagents were purchased from commercial sources and used without further purification. All solvents were purified by standard methods and freshly distilled under argon. NMR spectra were recorded in a 400 MHz spectrometer: ¹H (400 MHz), ¹³C (100 MHz), and ¹⁹F (376 MHz). The chemical shifts of ¹H and ¹³C NMR are reported relative to the residual signal of CDCl₃ (for ¹H: $\delta = 7.24$; ¹³C: $\delta = 77.00$) or C₆D₆ (for ¹H: $\delta = 7.15$; ¹³C: $\delta = 128.00$). All ¹³C NMR spectra were acquired in the proton-decoupled mode. ¹⁹F NMR spectra are reported relative to the resonance assigned to CFCl₃ ($\delta = 0$). Thin layer chromatography was performed with silica gel F₂₅₄ as the adsorbent on 0.2 mm thick, plastic-backed plates. The chromatograms were visualized under UV (254 nm) or by staining with a KMnO₄ aqueous solution followed by heating. Column chromatography was performed using

silica gel 60 (70–230 mesh). HRMS analyses were performed by the Proteomics Core Facility of the Center for Functional Genomics of the University at Albany.

2-Pentafluorosulfanylaldehydes

General procedure for preparation of 3a–c. To a solution of pentafluorosulfanyl chloride (2 mmol, 0.9 M in pentane) at -40 °C was added the enol ether **2** (1.54 mmol, 1 equiv) dissolved in 1 mL of pentane. After 5 min, this mixture was aerated by the bubbling of 2–3 mL of air by syringe, immediately after aeration 0.4 mL of triethylboron (1 M solution in hexanes, 0.1 equiv) in hexane was added via syringe pump over 30 min. The mixture was allowed to warm to rt with stirring over 1.5 h and then quenched with saturated NaHCO₃ solution. The mixture was then extracted with dichloromethane, the organic fractions washed with brine, dried (MgSO₄) and filtered and concentrated. The crude product was subjected to hydrolysis without further purification.

Preparation of 1a–c from 3: A mixture of the 1-chloro-2-pentafluorosulfanylalkyl ether (1–10 mmol), aqueous HCl (3.5 M, 9 mL) and glacial acetic acid (6 mL) was heated to 50 °C overnight. After cooling to room temperature, dichloromethane was added and the mixture poured into a beaker containing 40 mL of 20% Na₂CO₃. The aqueous layer was extracted with dichloromethane (2×) and the combined organic fractions washed with water (2×), brine, dried with Na₂SO₄, and filtered. The product was concentrated by cautious distillation of the dichloromethane by spinning band distillation, 10:1 reflux ratio, at ambient pressure.



(1a) 2-Pentafluorosulfanylacetaldehyde (yield 82%)

¹H-NMR (CDCl₃) δ /ppm: 9.79-9.77 (m, 1H, CHO), 4.37 (pd, 2H, ³*J*(H-F) = 8.0 Hz, ³*J*(H-H) = 2.5 Hz, CH₂).

¹⁹F-NMR (CDCl₃) δ/ppm: 80.4 (9 signals, 1F), 72.4 (dm, 4F, ${}^{2}J$ (F-F) = 147.9 Hz).



(**1b**) 2-Pentafluorosulfanylpropanal (yield 96%)

¹H-NMR (CDCl₃) δ ppm: 9.80-9.77 (m, 1H, CHO), 4.52-4.43 (m, 1H, CH), 1.64 (dp, ³*J*(H-H) = 7.2 Hz, ⁴*J*(H-F) = 2.8 Hz, CH₃).

¹⁹F-NMR (CDCl₃) δ/ppm: 82.1 (9 signals, 1F), 62.7 (dd, 4F, ${}^{2}J$ (F-F) = 144.6 Hz, ${}^{3}J$ (F-H) = 5.4 Hz).



(1c) 2- Pentafluorosulfanylbutanal (yield 73%)

¹H-NMR (CDCl₃) δ/ppm: 9.67-9.63 (m, 1H, CHO), 4.26-4.17 (m, 1H, CH), (m, CH₂), 1.01 (t, 3H, ${}^{3}J$ =7.5 Hz, CH₃).

¹⁹F-NMR (CDCl₃) δ/ppm: 82.6 (9 signals, 1F), 64.3 (dd, 4F, ${}^{2}J$ (F-F) = 144.6 Hz, ${}^{3}J$ (F-H) = 6.0 Hz).



(**1d**) 2- Pentafluorosulfanylnonanal¹

¹H-NMR (CDCl₃) δ/ppm: 9.66-9.61 (m, 1H, CHO), 4.33-4.21 (m, 1H, H_α), 2.21-2.12 (m, 2H, H_β), 1.39-1.21 (m, 10H), 0.92-0.87 (m, 3H).

¹⁹F-NMR (CDCl₃) δ/ppm: 82.7 (9 signals, 1F), 64.2 (dd, 4F, ${}^{2}J$ (F-F) = 144.6 Hz, ${}^{3}J$ (F-H) = 5.8 Hz).



(11) Ethyl 3-pentafluorosulfanyl-2-oxopropanoate (yield 79%)



Into a mixture of pentafluorosulfanyl bromide (0.4 M in pentane, 5 mL) and triethylboron (1 M in hexane, 0.2 mL) was added the enol acetate of ethyl pyruvate (237 mg, 1.5 mmol in 1 mL pentane) at 0 °C. The reaction mixture was stirred for 15 min then an additional portion of triethylboron (0.1 mL) was added. The mixture was stirred for another 15 min and then was quenched with sat. NaHCO₃. Following extraction with diethyl ether and driving with anhydrous MgSO₄, concentration in vacuo gave the crude product (525 mg, 96%) which was used in subsequent transformation without purification.

¹H NMR (CDCl₃) δ 5.13-5.00 (m, 1H), 4.79-4.67 (m, 1H), 4.46 – 4.31 (m, 2H), 2.24 (s, 3H), 1.37 (t, J = 7.2 Hz, 3H).

¹⁹F NMR (CDCl₃) δ 80.30 (p, J = 149.0, 1F), 68.66 – 68.12 (dt, J = 149.0, 7.3 Hz, 4F).



To a solution of the bromoacetate (960 mg, 2.6 mmol) in CH_2Cl_2 (10 mL) was added 1 drop of HCl (3.5 M in water) and 1 drop of water. The reaction mixture was heated under reflux overnight. After cooling to rt and drying with K_2CO_3 for 20 min, concentration in vacuo gave the crude product (518 mg, 82%).

¹H NMR (400 MHz, CDCl₃) δ 4.82 (p, *J* = 7.6 Hz, 1H), 4.42 (q, *J* = 7.2 Hz, 2H), 1.41(t, *J* = 7.2 Hz, 3H).

¹⁹F NMR (376 MHz, CDCl₃) δ 78.72 (p, *J* = 151.8, 1F), 68.66 – 68.12 (dm, *J* = 151.8 Hz, 4F).

General procedure for imine synthesis

To a flame dried round-bottom flask was added 5 equivalents of anhydrous magnesium sulfate followed by dry, distilled dichloromethane, to this suspension protected from atmospheric moisture was added a dichoromethane solution of the aldehyde (0.43 M). A dichloromethane solution (0.43 M) of the desired amine **6** was then added dropwise with stirring. Removal of the solids by filtration in the absence of atomospheric moisture, yielded a dichloromethane solution containing imine **7** that was utilized directly in the Staudinger reaction.

(5a) N-(2-Pentafluorosulfanylethylidene)-4-methoxybenzenamine

¹H-NMR (CDCl₃) δ/ppm: 8.00-7.95 (m, 1H), 7.15 (d, 2H, ${}^{3}J = 8.9$ Hz), 6.91 (d, 2H, ${}^{3}J = 8.9$ Hz), 4.67-4.57 (m, 2H, CH₂), 3.82 (s, 3H, CH₃).

¹⁹F-NMR (CDCl₃) δ/ppm: 81.3 (9 signals, 1F), 68.8 (dt, 4F, ${}^{2}J$ (F-F) = 146.1 Hz, ${}^{3}J$ (F-H) = 7.3 Hz).



(5b) N-(2-Pentafluorosulfanylpropylidene)-4-methoxybenzenamine

¹H-NMR (CDCl₃) δ/ppm: 7.97-7.93 (m, 1H), 7.12 (d, 2H, 3J = 8.8 Hz), 6.91 (d, 2H, 3J = 8.8 Hz), 4.83-4.74 (m, 1H, CH), 3.82 (s, 3H, OCH₃), 1.77 (dp, 3H, ³*J*(H-H) = 7.1 Hz, CH₃).

¹⁹F-NMR (CDCl₃) δ/ppm: 83.0 (9 signals, 1F), 64.5 (enamine) (d, 4F, ${}^{2}J$ (F-F) = 148.6 Hz), 58.7 (dd, 4F, ${}^{2}J$ (F-F) = 143.0 Hz, ${}^{3}J$ (F-H) = 4.4 Hz).



(5c) N-(2-Pentafluorosulfanylbutylidene)-4-methoxybenzenamine

¹H-NMR (CDCl₃) δ/ppm: 7.84-7.78 (m, 1H), 7.14 (d, 2H, ${}^{3}J = 8.4$ Hz), 6.92 (d, 2H, ${}^{3}J = 8.4$ Hz), 4.61-4.50 (m, 1H, CH), 3.82 (s, 3H, OCH₃), 2.42-2.32 (m, 1H, CH₂), 2.26-2.16 (m, 1H, CH₂), 1.02 (t, 3H, ${}^{3}J = 7.3$ Hz, CH₃).

¹⁹F-NMR (CDCl₃) δ/ppm: 83.5 (9 signals, 1F), 65.8 (enamine) (d, 4F, ${}^{2}J$ (F-F) = 148.7 Hz), 59.7 (dd, 4F, ${}^{2}J$ (F-F) = 143.3 Hz, ${}^{4}J$ (F-H) = 4.7 Hz).



(5d) N-(2-Pentafluorosulfanylnonylidene)-4-methoxybenzenamine

¹H-NMR (CDCl₃) δ /ppm: 7.79 (m, 1H, ³*J* = 6.4 Hz), 7.13 (d, 2H, ³*J* = 8.8 Hz), 6.92 (d, 2H, ³*J* = 8.6 Hz), 4.67-4.56 (m, 1H, H_a), 3.83 (s, 3H), 1.41-1.21 (m, 10H), 0.93-0.84 (m, 3H).

¹⁹F-NMR (CDCl₃) δ /ppm: 83.5 (9 signals, 1F), 65.7 (enamine) (d, 4F, ²*J*(F-F) = 148.8 Hz), 59.6 (dd, 4F, ²*J*(F-F) = 143.1 Hz, ³*J*(F-H) = 5.1 Hz).

General procedure for β -lactam synthesis.

In a typical procedure, to a dichloromethane solution of triethylamine (0.537 g, 5.37 mmol, 3 mL) was added benzyloxyacetyl chloride (0.978 g, 5.3 mmol) dissolved in another 3 mL of dichloromethane. The imine **5** (1.28 mmol) in 4.5 mL of dichloromethane was added dropwise to the previously prepared solution of triethylamine and benzyloxyacetyl chloride at 0 °C. The reaction mixture was allowed to warm to room temperature and to stir overnight. The reaction was quenched with ca 6 mL of saturated NaHCO₃, following separation of the phases, the aqueous phase was extracted three times with dichloromethane. The combined organic extracts were dried over MgSO₄, filtered and concentrated in vacuo. The desired β -lactam **7** was isolated by column chromatography using 4:1, ethyl acetate/hexanes.

(7a) (3SR,4RS)-3-(Benzyloxy)-4-(pentafluorosulfanylmethyl)-1-(4-

methoxyphenyl)azetidin-2-one (yield 17%)

¹H-NMR (CDCl₃) δ /ppm: 7.44-7.27 (m, 7H, H_{Bn}, H_{PMP}), 6.95-6.90 (m, 2H, H_{PMP}), 4.90 (AB, 2H, ²*J* = 11.7 Hz, H_{Bn}), 4.90 (d, 1H, ³*J* = 4.8 Hz, H_γ), 4.82 (ddd, 1H, ³*J* = 8.7 Hz, ³*J* = 4.8 Hz, ³*J* = 1.2 Hz, H_β), 4.43-4.29 (m, 1H, H_α), 4.05-3.91 (m, 1H, H_α), 3.80 (s, 3H, OCH₃).

¹³C{¹H}-NMR (CDCl₃) δ/ppm: 163.4 (<u>C</u>=O), 157.0, 136.5, 128.8, 128.6, 128.2, 127.9, 118.6, 114.9, 80.8 (C_γ), 73.6, 65.3 (²*J*(C-F) = 13.0 Hz, C_α), 55.5, 53.9 (³*J*(C-F) = 5.9 Hz, C_β).

¹⁹F-NMR (CDCl₃) δ/ppm: 83.3 (9 signals, 1F 146.7 Hz), 66.9 (dt, 4F, ${}^{2}J$ (F-F) = 146.7 Hz, ${}^{3}J$ (F-H) = 8.0 Hz).

HRMS (ESI, positive): Exact mass calcd. $C_{18}H_{18}F_5NO_3S$ [M]⁺ requires *m*/*z*: 423.0922, found *m*/*z*: 423.0912.

(7b) (3SR,4RS)-3-(Benzyloxy)-4-((1'SR)-1-pentafluorosulfanylethyl)-1-(4-

methoxyphenyl)azetidin-2-one (yield 9%)

¹H-NMR (CDCl₃) δ /ppm: 7.43-7.27 (m, 7H, H_{Bn}, H_{PMP}), 6.94-6.89 (m, 2H, H_{PMP}), 4.94 (d, 1H, ³*J* = 5.0 Hz, H_{γ}), 4.90 (dd, 1H, ³*J* = 5.2 Hz, ³*J* = 3.3 Hz, H_{β -ring}), (4.87 (AB, 2H, ²*J*)

= 11.7 Hz, H_{Bn}), 4.61-4.49 (m, 1H, H_a), 3.80 (s, 3H, OCH₃), 1.68 (d, 3H, ${}^{3}J$ = 6.9 Hz, H_β).

¹³C{¹H}-NMR (CDCl₃) δ/ppm: 164.6 (C=O), 157.0, 136.7, 128.8, 128.5, 128.1, 127.8, 118.8, 114.9, 82.6, 79.4 (m), 74.3, 56.9 (m), 55.5, 12.8.

¹⁹F-NMR (CDCl₃) δ /ppm: 84.6 (9 signals, 1F, 143.7 Hz), 57.9 (dd, 4F, ²*J*(F-F) = 143.7 Hz, ³*J*(F-H) = 6.3 Hz).

HRMS (ESI, positive): Exact mass calcd. $C_{19}H_{20}F_5NO_3S$ [M]⁺ requires *m*/*z*: 437.1079, found *m*/*z*: 437.1071.

(7c) (3SR,4RS)-3-(Benzyloxy)-4-((1'SR)-1-pentafluorosulfanylpropyl)-1-(4-

methoxyphenyl)azetidin-2-one (yield 2%)

¹H-NMR (CDCl₃) δ/ppm: 7.43-7.27 (m, 7H, H_{Bn}, H_{PMP}), 6.95-6.89 (m, 2H, H_{PMP}), 4.95 (dd, 1H, ${}^{3}J = 5.0$ Hz, ${}^{3}J = 2.9$ Hz, H_{β-ring}), 4.92 (d, 1H, ${}^{3}J = 5.0$ Hz, H_{γ-ring}), 4.86 (AB, 2H, ${}^{2}J = 11.8$ Hz, H_{Bn}), 4.41-4.30 (m, 1H, H_α), 3.80 (s, 3H, OCH₃), 2.54-2.40 (m, 1H, H_β), 2.17-2.04 (m, 1H, H_β), 0.96 (t, 3H, ${}^{3}J = 7.4$ Hz, H_γ).

¹³C{¹H}-NMR (CDCl₃) δ /ppm: 164.7 (C=O), 156.9, 136.8, 129.1, 128.5, 128.1, 127.8, 118.6, 114.9, 86.9 (m, C_{\alpha}), 82.6 (C_{\gamma-ring}), 74.2, 57.6 (m, C_{\beta-ring}), 55.5, 21.2 (m, C_{\beta}), 13.7 (m, C_{\gamma}).

¹⁹F-NMR (CDCl₃) δ/ppm: 85.9 (9 signals, 1F, ${}^{2}J$ (F-F) = 143.7 Hz) 60.9 (d, 4F, ${}^{2}J$ (F-F) = 143.6 Hz).

HRMS (ESI, positive): Exact mass calcd. $C_{20}H_{22}F_5NO_3S$ [M]⁺ requires *m*/*z*: 451.1235, found *m*/*z*: 451.1219.

(7d) (3SR,4RS)-3-(Benzyloxy)-4-((1'SR)-1-pentafluorosulfanyloctyl)-1-(4-

methoxyphenyl)azetidin-2-one (yield 4%)

¹H-NMR (CDCl₃) δ /ppm: 7.43-7.28 (m, 7H, H_{Bn}, H_{PMP}), 6.94-6.90 (m, 2H, H_{PMP}), 4.95 (dd, 1H, ³*J* = 5.1 Hz, ³*J* = 3.0 Hz, H_{β-ring}), 4.91 (d, 1H, ³*J* = 5.1 Hz, H_{γ-ring}), 4.87 (AB, 2H, ²*J* = 11.8 Hz, H_{Bn}), 4.49-4.39 (m, 1H, H_α), 3.80 (s, 3H, OCH₃), 2.48-2.36 (m, 1H, H_β), 2.09-1.96 (m, 1H, H_β), 1.12-1.02 (m, 6H), 0.94-0.84 (m, 6H), 0.81 (t, ³*J* = 7.1 Hz).

¹³C{¹H}-NMR (CDCl₃) δ/ppm: 164.7 (<u>C</u>=O), 156.9, 137.2, 128.6, 128.5, 128.1, 127.6, 118.6, 114.9, 82.5 (C_γ), 73.4, 67.2, 31.6, 29.7, 28.94, 28.64, 28.19, 27.5, 22.6, 14.03

¹⁹F-NMR (CDCl₃) δ/ppm: 86.0 (9 signals, 1F, 2J(F-F) = 143.7 Hz), 60.7 (d, 4F, ${}^{2}J$ (F-F) = 143.8 Hz).

HRMS (ESI, positive): Exact mass calcd. $C_{25}H_{32}F_5NO_3S$ [M]⁺ requires *m*/*z*: 521.2018, found *m*/*z*: 521.2013.

(7e) Ethyl 3-(benzyloxy)-2-(pentafluorosulfanylmethyl)-1-

(4-methoxyphenyl)-4-oxoazetidine-2-carboxylate (yield 8%) Diastereomers designated A & B

¹H-NMR (CDCl₃) δ /ppm: 7.45-7.32 (m, 7H, H_{Bn}, H_{PMP}, **A** + **B**), 6.89-6.82 (m, 2H, H_{PMP}, **A** + **B**), 4.93 (s, 1H, H_{γ}, **A** + **B**), 4.83 (AB, 2H, ²*J* = 11.8 Hz, H_{Bn}, **A** + **B**), 4.52-4.05 (m, 4H, H_{α}, CH₂-ester, **A** + **B**), 3.80 (s, 3H, OCH₃, **A**), 3.79 (s, 3H, OCH₃, **B**), 1.30 (t, 3H, ³*J* = 7.2 Hz, CH₃-ester, **A**), 1.25 (m, 3H, CH₃-ester, **B**).

¹³C{¹H}-NMR (CDCl₃) δ/ppm: 168.6(C=O_{Ester} B), 168.5 (C=O_{Ester}, A), 164.5 (C=O_{Ring}, A), 163.9 (C=O, C_{Ring}, B), 158.5 (A), 157.3 (B), 136.2 (B), 135.6 (A), 129.2, 128.8, 128.73, 127.68, 128.5, 128.3, 128.1, 127.1, 125.9, 121.4, 114.2 (B), 114.0 (A), 88.0 (A), 86.0 (B), 74.7 (A), 74.5 (B), 71.3 (m), 69.4 (m), 69.2, 63.0 (A), 62.6 (B), 55.42 (B), 55.38 (B), 42.3, 14.0 (B), 13.9 (A).

¹⁹F-NMR (CDCl₃) δ/ppm: 81.9 (9 signals, 1F, ²*J*(F-F) = 149.9-145.2 Hz A + B), 71.2 (dt, 4F, ²*J*(F-F) = 147.0, ³*J*(F-H) = 8.9 Hz).

HRMS (ESI, positive): Exact mass calcd. $C_{21}H_{22}F_5NO_5S$ [M]⁺ requires *m*/*z*: 495.1133, found *m*/*z*: 495.1163.



(12) (E)-ethyl 2-(4-methoxyphenylimino)-3-pentafluorosulfanylpropanoate

Procedure:

Into a solution of **11** (726 mg, 3 mmol) in DCM (15 mL) was added *p*-anisidine (1.10 g, 9 mmol) and MgSO₄ (1.8 g, 15 mmol). The reaction mixture was allowed to stir overnight. Filteration and concentration gave the crude product as a mixture of E/Z-isomers and unreacted of PMPNH₂.

Isomer 1: ¹H NMR (400 MHz, CDCl₃) δ 6.91 – 6.87 (m, 2H), 6.84 – 6.81 (m, 2H), 4.83 (p, *J* = 7.3 Hz, 2H), 4.13(q, *J* = 7.1 Hz, 1H), 3.81 (s, 3H), 1.06 (t, *J* = 7.1 Hz, 3H).

¹⁹F NMR (376 MHz, CDCl₃) δ 80. 03 (p, J = 152.0 Hz, 1F), 70.49 (dm , J = 152.0 Hz, 4F).

Isomer 2: ¹H NMR (400 MHz, CDCl₃) δ 6.96 – 6.92 (m, 2H), 6.79 – 6.76 (m, 2H), 4.76 (p, *J* = 7.4 Hz, 2H), 4.47(q, *J* = 7.0 Hz, 1H), 3.83 (s, 3H), 1.43 (t, *J* = 6.9 Hz, 3H). ¹⁹F NMR (376 MHz, CDCl₃) δ 80. 00 (p, *J* = 149.4 Hz, 1F), 69.50 (dm , *J* = 149.4 Hz,

4F).

Crystallography

The X-ray intensity data were collected for **7a** and **7c** on a Bruker APEX CCD X-ray diffractometer equipped with a graphite monochromated Mo K α radiation source ($\lambda = 0.71073$ Å). Data reduction and integration were performed with the software package *SAINT*,² and absorption corrections were applied using the program *SADABS*.³ All structures were solved and refined using the *SHELXTL* program package.⁴ All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included at idealized positions as a riding model. Crystallographic data and X-ray experimental conditions for **7a** and **7c** are listed in Table S1.



	7a	7c
Empirical formula	$C_{18}H_{18}F_5NO_3S$	$C_{20}H_{22}F_5NO_3S$
Formula weight	423.39	451.45
$T(\mathbf{K})$	100(2)	100(2)
Crystal system	monoclinic	triclinic
Space group	$P2_{1}/c$	<i>P</i> ⁻ 1
<i>a</i> (Å)	5.9580(11)	6.809 (7) Å
<i>b</i> (Å)	14.455(3)	10.145 (10) Å
<i>c</i> (Å)	20.381(4)	14.616 (15) Å
α (°)	90.00	93.168 (13)
β (°)	92.069(2)	95.116 (14)°
γ (°)	90.00	97.126 (13)
$V(\text{\AA}^3)$	1754.1(6)	995.6 (18)
Ζ	4	2
$R1^{\rm a}, wR2^{\rm b}$	0.0322, 0.0867	0.1262, 0.2922
$[I > 2\sigma(I)]$		
$R1^{\circ}, wR2^{\circ}$ (all	0.0352, 0.0898	0.2002, 0.3241
data)	, , , , , , , , , , , , , , , , , , , ,	·
Quality-of-fit	1.059	1.051
Largest diff. peak/hole, e/Å ³	0.382, -0.408	1.159, -0.956

Table S1. Crystallographic data and X-ray experimental conditions for 7a and 7c.

 $\frac{|Peak/Hole, C/A|}{|^{a}R1 = \Sigma| |F_{o}| - |F_{c}| |/\Sigma|F_{o}|}$ ${}^{b}wR2 = [\Sigma[w(F_{o}^{2} - F_{c}^{2})^{2}]/\Sigma[w(F_{o}^{2})^{2}]]^{\frac{1}{2}}$ ${}^{c}Quality-of-fit = [\Sigma[w(F_{o}^{2} - F_{c}^{2})^{2}]/(N_{obs} - N_{params})]^{\frac{1}{2}}, \text{ based on all data.}$



S13



S14





































References

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³ SADABS; Bruker AXS, Inc.: Madison, WI, 2001.

⁴ Sheldrick, G. M. *SHELXTL*, version 6.14; Bruker AXS, Inc.: Madison, WI, 2001.