Supporting Information for

Trimerization and Cyclization of Reactive P-Functionalities Confined Within OCO Pincers

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1. Experiments and NMR Spectra for New Compounds (page S1-S43)
2. Simulated ¹⁹F NMR Spectra using MestreNova (S44-S49)
3. Experimental Details for Crystal Structure Refinement and Acquisition (S50 – S114)
1. Experimentals and NMR Spectra for New Compounds:

General Experimental Details

Unless otherwise specified, all reactions were performed under an atmosphere of nitrogen in an MBraun or Vacuum Atmospheres glovebox or using standard Schlenk techniques. All glassware was dried overnight in an oven at 140 °C prior to use. Solvents used in the glove box were purchased directly from chemical suppliers (Aldrich or Acros), pumped directly into the glove box, and stored over oven-activated 4 or 5 Å molecular sieves (Aldrich). Solvents used outside the glove box were purged with N₂ for 30 min and stored over molecular sieves. TMSCF₃ was dried by cryogenic transfer. ¹H, ¹³C{¹H}, ¹⁹F, and ³¹P{¹H} NMR spectra were recorded on a Varian Mercury-300 (300/75/282/121 MHz), Varian Unity Inova-500 (500/126/470/202 MHz), or Agilent 600 DD2 (600/151/565/243 MHz) spectrometer at ambient temperature. Chemical shifts are reported in ppm downfield of tetramethylsilane using the solvent as internal standard (¹H CDCl₃ = 7.27 ppm, ¹H C₆D₆ = 7.16 ppm, ¹³C CDCl₃ = 77.16 ppm, ¹³C C₆D₆ = 128.06 ppm). Multiplicities are abbreviated as br (broad), s (singlet), d (doublet), t (triplet), q (quartet), or m (multiplet). Coupling constants (J) are reported in Hertz (Hz). Flash column chromatography was performed on silica gel (40-63 μm, SiliCycle). High-resolution mass spectrometry (HRMS) was recorded on an Agilent 6545 LC-MS Q-ToF spectrometer (NSF-1532310). Dimethyl 2-bromoisophthalate and [Me₄N][F] were synthesized according to the published procedures listed below.¹² All other chemicals were used as received, unless otherwise noted.

¹ Courchay, F.C.; Sworen, J.C.; Ghiviriga, I.; Abboud, K.A.; Wagener, K.B. Understanding Structural Isomerization during Ruthenium-Catalyzed Olefin Metathesis: A Deuterium Labeling Study. Organometallics 2006, 25, 6074-6086.
² Kolomeitsev, A.A.; Seifert, F.U.; Roeschenthaler, G.-V. Simple Preparation of Difluorophosphoranes Using Anhydrous Zinc and Tetramethylammonium Fluorides. J. Fluorine Chem. 1995, 71 (1), 47-49.
Synthesis of 1.

In the glovebox, 2,4,6-trimethylphenol (3.00 g, 22.03 mmol, 2.5 equiv.) was dissolved in THF (100 mL), and a 1.6 M solution of n-BuLi in hexanes (13.0 mL, 20.8 mmol, 2.3 equiv.) was added dropwise at room temperature. The reaction mixture was stirred for 15 min. Subsequently, tribromide D (3.086 g, 9.0 mmol) was added to the stirred solution, and the reaction mixture was transferred to a Schlenk bomb equipped with a Teflon screw cap and heated at 100 °C for 1 d. The volatiles were then removed under reduced pressure, and the crude residue was extracted with a toluene:hexane mixture (1:1, 3 x 15 mL). The combined extracts were filtered through a Celite plug, and the filtrate was concentrated under vacuum then purified by column chromatography (silica gel, toluene:hexane 1:1, Rf = 0.63). The title product (2.64 g, 5.822 mmol) was obtained as white plates in 65% yield. X-Ray quality crystals were obtained by slow evaporation from a concentrated solution of 1 in n-hexanes.

Anal. Calcd for C_{26}H_{29}BrO_{2}: C, 68.87; H, 6.45. Found: C, 68.77; H, 6.27. \(^1\)H NMR (500 MHz, CDCl\(_3\)): \(\delta\) 7.78 (d, \(J = 7.6\) Hz, 2H, Ar), 7.51 (t, \(J = 7.6\) Hz, 1H, Ar), 6.89 (s, 4H, Ar), 4.93 (s, 4H, CH\(_2\)), 2.30 (s, 12H, Me), 2.29 (s, 6H, Me). \(^{13}\)C\(^1\)H NMR (126 MHz, CDCl\(_3\)): \(\delta\) 153.5 (Ar), 137.8 (Ar), 133.6 (Ar), 130.9 (Ar), 129.6 (Ar), 127.9 (Ar), 127.7 (Ar), 121.6 (Ar), 73.3 (CH\(_2\)), 20.9 (CH\(_3\)), 16.4 (CH\(_3\)).
$^{1}$H NMR Spectrum of 1

![1H NMR Spectrum](image)

Mes \[\text{O} \begin{array}{c} \text{Br} \\ \text{O} \end{array} \text{Mes} \]

\[ \text{1} \]
$^{13}$C\{$^1$H\} NMR Spectrum of 1

\[
\begin{array}{c}
\text{Mes} - O - \text{Br} - O - \text{Mes} \\
\end{array}
\]
DEPT of 1

Mes-O-Br-O-Mes
Chemical Formula: C_{26}H_{29}BrO_{2}
Exact Mass: 452.1351
Molecular Weight: 453.4200
Elemental Analysis: C, 68.87; H, 6.45; Br, 17.62; O, 7.06

Acknowledgment
Analytical data were obtained from the CENTC Elemental Analysis Facility at the University of Rochester, funded by NSF CHE-0650456.

Instrumentation
Microanalysis samples were weighed with a PerkinElmer Model AD-6 Autobalance and their compositions were determined with a PerkinElmer 2400 Series II Analyzer.
Synthesis of 2, Its "Half" Trifluoromethylated Derivative, and Diol E:

Synthesis of "Half" Trifluoromethylated.

Initial attempts to prepare diol E (via its recently reported literature prep\textsuperscript{3}) using six equiv of Ruppert’s reagent (TMSCF\textsubscript{3}) and four equiv of [Me\textsubscript{4}N][F] yielded a mixture of trifluoromethylated products; the “half” trifluoromethylated derivative could be isolated cleanly in low yield.

In the glovebox, dimethyl 2-bromoisophthalate (4.40 g, 16.1 mmol) and [Me\textsubscript{4}N][F] (6.00 g, 64.4 mmol, 4 equiv.) were suspended in DME (65 mL) in a Schlenk bomb with a stir bar. The bomb was taken out of the glove box, placed on the Schlenk line, and cooled to -40 °C in a dry ice/acetonitrile bath. TMSCF\textsubscript{3} (9.52 mL, 64.4 mmol, 4 equiv.) was injected \textit{via} syringe through a rubber septum, and the mixture was stirred at -40 °C for 4 h. A second aliquot of TMSCF\textsubscript{3} (4.76 mL, 32.2 mmol, 2 equiv.) was injected in a similar fashion, and the mixture was stirred at -40 °C for another 6 h. After warming to room temperature, NH\textsubscript{4}Cl (aq) was added at 0 °C, and the mixture was extracted with toluene (3 x 50 mL). The combined organic layers were washed with water then brine, dried over MgSO\textsubscript{4}, filtered, and the filtrate was concentrated under vacuum. The crude product mixture was purified by column chromatography with DCM/hexanes (1:2) as the eluent. The pure fractions were recrystallized from dichloromethane (8 mL) layered with hexanes (50 mL) at 4 °C. The colorless precipitate was filtered and rinsed with cold pentane (40 mL), affording crystals suitable for X-ray diffraction (0.24 g, 0.49 mmol, 3%).

Anal. Calcd for C\textsubscript{11}H\textsubscript{7}BrF\textsubscript{6}O\textsubscript{3}: C, 34.67; H, 1.85. Found: C, 34.76; H, 1.57. \textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}): δ 7.81 (br d, J = 15 Hz, 1H, Ar), 7.60 (dd, J = 5, 15 Hz, 1H, Ar), 7.50 (t, J = 15 Hz, 1H, Ar), 5.34 (br s, 1H, OH), 3.99 (s, 3H, Me). \textsuperscript{13}C NMR (126 MHz, CDCl\textsubscript{3}): δ 138.9 (Ar), 132.1 (Ar), 130.9 (Ar), 128.7 (Ar), 127.6 (Ar), 122.4 (q, J = 290 Hz, CF\textsubscript{3}), 118.0 (Ar), 53.1 (Me), the quaternary carbon and C=O were not observed. \textsuperscript{19}F NMR (470 MHz, CDCl\textsubscript{3}): δ -73.2 (s).

\textsuperscript{3} Y. Imada, T. Kukita, H. Nakano and Y. Yamamoto, \textit{Bull. Chem. Soc. Jpn.}, 2016, 89, 546-548.
\(^1\)H NMR (300 MHz, CDCl\(_3\)) spectrum
$^{13}$C NMR (126 MHz, CDCl$_3$) spectrum
(inset displays CF$_3$ quartet)
$^{19}$F NMR (282 MHz, CDCl$_3$) spectrum
**Acknowledgment**

Analytical data were obtained from the CENTC Elemental Analysis Facility at the University of Rochester, funded by NSF CHE-0650456.

**Instrumentation**

Microanalysis samples were weighed with a PerkinElmer Model AD6000 Autobalance and their compositions were determined with a PerkinElmer 2400 Series II Analyzer.
Synthesis of Diol E.

In order to access diol E, the published prep\textsuperscript{4} had to be modified. Specifically, a large excess of TMSCF\textsubscript{3} was required, and the solvent was changed from DME to DMF.

In the glovebox, dimethyl 2-bromoisophthalate (3.20 g, 11.7 mmol) and [Me\textsubscript{4}N][F] (21.83 g, 234.4 mmol, 20 equiv.) were dissolved in DMF (300 mL) in a Schlenk flask with a stir bar. The flask was taken out of the glove box, placed on the Schlenk line, and fitted with an air-free addition funnel under positive N\textsubscript{2} pressure. The flask was cooled to -40 °C in a dry ice/acetonitrile bath with vigorous stirring, TMSCF\textsubscript{3} (34.6 mL, 234 mmol, 20 equiv.) was transferred to the addition funnel via cannula and added dropwise to the Schlenk flask over several min. The mixture was stirred at -40 °C for 4 h then another dose of TMSCF\textsubscript{3} (17.3 mL, 117 mmol, 10 equiv.) was added in the same manner. The mixture was stirred overnight at -40 °C, warmed to RT, and stirred for an additional 1 h. NH\textsubscript{4}Cl (aq) was added at 0 °C, and the reaction mixture was extracted with toluene (3 x 50 mL). The combined organic layers were washed with water, brine, dried over MgSO\textsubscript{4}, filtered, and the filtrate was concentrated under vacuum. The resulting yellow oil was purified by column chromatography using Et\textsubscript{2}O/n-hexane (1:2) as the eluent to give pale yellow solid 7 (4.67 g, 9.55 mmol, 82%). The \textsuperscript{1}H,\textsuperscript{13}C{[\textsuperscript{1}H]}, and \textsuperscript{19}F NMR spectra were consistent with the published report,\textsuperscript{4} but a slight impurity at 3.99 ppm in the \textsuperscript{1}H NMR spectrum persisted.

\textsuperscript{4} Y. Imada, T. Kukita, H. Nakano and Y. Yamamoto, Bull. Chem. Soc. Jpn., 2016, 89, 546-548.
Synthesis of 2.

Diol E (3.61 g, 7.38 mmol) and DMF (180 mL) were combined in a Schlenk flask with a stir bar. The solution was purged with N₂ on the Schlenk line for 30 min, then K₂CO₃ (5.10 g, 36.9 mmol, 5 equiv.) was added with stirring under positive N₂ pressure. Next, MeI (2.30 mL, 36.9 mmol, 5 equiv.) was injected via syringe, and the reaction mixture was stirred overnight, then quenched with NH₄Cl (aq) and extracted with toluene (3 x 50 mL). The combined organic layers were washed with water, brine, dried over MgSO₄, filtered, and the filtrate concentrated under vacuum, resulting in pale yellow solid 2 (2.97 g, 5.74 mmol, 78%). Recrystallization from acetonitrile (8 mL) at 4 °C afforded crystals suitable for X-ray diffraction.

Anal. Calcd for C₁₄H₉BrO₂F₁₂: C, 32.52; H, 1.75. Found: C, 32.71; H, 1.77. ¹H NMR (500 MHz, CDCl₃): δ 7.76 (d, J = 10 Hz, 2H, Ar), 7.51 (t, J = 10 Hz, 1H, Ar), 3.52 (s, 6H, Me). ¹³C{¹H} NMR (151 MHz, CDCl₃): δ 134.1 (Ar), 129.6 (Ar), 127.0 (Ar), 123.5 (Ar), 122.4 (q, J = 290 Hz, CF₃), 86.5 (septet, J = 29 Hz, C(CF₃)₂), 54.8 (OMe). ¹⁹F NMR (471 MHz, CDCl₃): δ -66.9 (s).
$^1$H NMR (500 MHz, CDCl$_3$) spectrum
$^{13}\text{C}^1\text{H}$ NMR (151 MHz, CDCl$_3$) spectrum
(insets display CF$_3$ quartet and quaternary carbon atoms)

Trace ether and grease are present.
$^{19}$F NMR (471 MHz, CDCl$_3$) spectrum

![Chemical Structure](image)
Acknowledgment

Analytical data were obtained from the CENTC Elemental Analysis Facility at the University of Rochester, funded by NSF CHE-0650456.

Instrumentation

Microanalysis samples were weighed with a PerkinElmer Model AD6000 Autobalance and their compositions were determined with a PerkinElmer 2400 Series II Analyzer.

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**Chemical Formula:** C$_{14}$H$_9$BrF$_{12}$O$_2$

**Exact Mass:** 515.9594

**Molecular Weight:** 517.1088

m/z: 515.9594 (100.0%), 517.9574 (97.3%), 516.9628 (15.1%), 518.9607 (14.7%), 517.9661 (1.1%), 519.9641 (1.0%)

**Elemental Analysis:** C, 32.52; H, 1.75; Br, 15.45; F, 44.09; O, 6.19
Synthesis of 3.

OCO-supported aryl bromide 1 (1.134 g, 2.50 mmol) was loaded into a Schlenk flask, dissolved in Et₂O (60 mL), taken outside of the glovebox, and cooled to -78 °C. A 1.6 M solution of n-BuLi in hexanes (1.7 mL, 2.72 mmol, 1.1 equiv) was injected (under N₂), and the reaction mixture was stirred at -78 °C for 5 min. A second Schlenk flask containing a solution of PCl₃ (550 mg, 4.01 mmol, 1.6 equiv.) in Et₂O (5 mL) was transferred via cannula to the cooled reaction mixture. The reaction mixture was stirred at -78 °C for 10 min and then at room temperature (RT) for 1 h, leading to the precipitation of a white solid. The volatiles were removed under reduced pressure, the Schlenk flask was brought back into the glovebox, the residue was triturated with Et₂O (10 mL), and the volatiles were again removed. The residue was dissolved in THF (20 mL), and PMe₃ was added (485 mg, 6.375 mmol, 2.6 equiv.). The reaction mixture was stirred at RT for 1 d. The organic volatiles were removed under reduced pressure, and the residue was extracted with toluene (3 x 20 mL). The combined extracts were filtered through a Celite plug, the filtrate was concentrated to dryness, triturated with n-pentane (10 mL), and again concentrated to dryness under reduced pressure. The resulting residue was triturated with acetonitrile (10 mL) and stirred at RT for 1 h, generating a white precipitate that was collected by filtration and dried (565 mg, 0.466 mmol, 56%). X-Ray quality crystals of 3 were obtained by recrystallization from a solution of hot acetonitrile.

Anal. Calcd for C₇₆H₆₇O₆P₃: C, 77.20; H, 7.23. Found: C, 76.89; H, 7.14. ³¹P{¹H} NMR (202 MHz, CDCl₃): δ 116.2 (d, Jpp = 186 Hz), -144.0 (t, Jpp = 186 Hz). ¹H NMR (500 MHz, CDCl₃): δ 7.71 (d, J = 7.7 Hz, 2H, Ar¹), 7.40 (t, J = 7.7 Hz, 1H, Ar¹), 7.33-7.28 (m, 4H, Ar²), 7.28-7.22 (m, 2H, Ar²), 6.71 (s, 8H, Ar²), 6.62 (s, 4H, Ar²), 5.39 (s, 4H, CH₂-Ar²), 4.71 (br, 8H, CH₂-Ar²), 2.25 (s, 12H, Ar³), 2.20 (s, 6H, Ar³), 2.02 (s, 12H, Ar³), 1.95 (br, 24H, Ar³). ¹³C NMR (126 MHz, CDCl₃): δ 153.46 (Ar), 153.44 (Ar), 143.5 (Ar), 143.4 (d, JPC = 6.9 Hz, Ar), 133.6 (Ar), 133.0 (Ar), 132.9 (Ar), 130.7 (Ar), 130.5 (Ar), 130.4 (Ar), 129.6 (Ar), 129.4 (Ar), 129.0 (Ar), 128.4 (d, JPC = 10.0 Hz, Ar), 127.9 (Ar), 126.4 (Ar), 73.2 (CH₂), 72.8 (d, JPC = 7.6 Hz, CH₂), 20.9 (CH₃), 16.7 (CH₃), 16.4 (CH₃), 16.3 (CH₃).

Ar¹ refers to the aryl groups associated with the Blue P
Ar² refers to the aryl groups associated with the Red Ps.
$^{1}H$ NMR Spectrum of 3
$^{13}$C$\{^1\text{H}\}$ NMR Spectrum of 3
$^{31}$P$^1$H NMR Spectrum of 3
Special Handling

The sample was transferred under argon and was combusted in a tin capsule that was crimp-sealed with a die apparatus.

Acknowledgment

Analytical data were obtained from the CENTC Elemental Analysis Facility at the University of Rochester and funded by NSF CHE-0650456.

Instrumentation

Microanalysis samples were weighed with a PerkinElmer Model AD-6 Autobalance and their compositions were determined with a PerkinElmer 2400 Series II Analyzer. Air-sensitive samples were handled in a VAC Atmospheres glovebox.
Generation and Observation of 5 by $^{31}$P{$^1$H} and $^{19}$F NMR Spectroscopy.

Fluorinated aryl bromide 2 (500 mg, 0.967 mmol) was dissolved in 6 mL of THF in a vial with a stir bar inside the glovebox and (i-Pr)MgCl·LiCl was added dropwise via syringe (0.82 mL, 1.064 mmol, 1.1 equiv, 1.3 M in THF), resulting in a homogeneous, yellow reaction mixture. The reaction mixture was stirred for 1 h at RT then directly filtered into 2 mL of a pre-cooled solution (-35 °C, 1 h) of PCl₃ (146 mg, 1.064 mmol, 1.1 equiv) in THF. The solution was warmed to RT for 1 h then analyzed by $^{31}$P{$^1$H} NMR spectroscopy, revealing the presence of unreacted PCl₃ (218 ppm), i-PrPCl₂ (202 ppm) and the chlorophosphine (171 ppm). The “intermediate” product mixture was then concentrated under vacuum to remove the volatile and unwanted phosphorus byproducts (PCl₃ and i-PrPCl₂), leaving a pale yellow solid (405 mg, 0.751 mmol, 78%) that was reanalyzed by $^{31}$P{$^1$H} and $^{19}$F NMR spectroscopy in C₆D₆.

$^{19}$F NMR (282 MHz, C₆D₆):* δ -68.9 (m, $J_{FF}$ ~ 8 Hz, $J_{PF}$ ~ 2 Hz, $J_{HF}$ ~ 0.5-1.5 Hz), -70.4 (m, $J_{FF}$ ~ 8 Hz, $J_{PF}$ ~ 4 Hz, $J_{HF}$ ~ 0.2-2.6 Hz), 70.9 (s, impurity**), -73.4 (m, $J_{FF}$ ~ 9 Hz, $J_{PF}$ ~ 2 Hz, $J_{HF}$ ~ 1 Hz), -76.3 (m, $J_{FF}$ ~ 9 Hz, $J_{PF}$ ~ 2 Hz, $J_{HF}$ ~ 0.5-2 Hz). $^{31}$P{$^1$H} NMR (121 MHz, C₆D₆): δ 166.4 (s).

*Reported coupling constants in the $^{19}$F NMR spectrum were determined by simulation with Mestrenova.

**The impurity at 70.9 ppm in the $^{19}$F NMR spectrum is the product of protodemetalation.
$^{19}\text{F NMR (282 MHz, C$_6$D$_6$)}$ spectra

* = impurity (see Experimental for structure)
$^{31}\text{P}^{(1)}\text{H} \text{NMR (121 MHz, C}_6\text{D}_6\text{)} \text{ spectrum}$
Synthesis of 6.

Fluorinated aryl bromide 2 (500 mg, 0.967 mmol) was dissolved in 6 mL of THF in a vial with a stir bar inside the glovebox and (i-Pr)MgCl-LiCl was added dropwise via syringe (0.82 mL, 1.064 mmol, 1.1 equiv, 1.3 M in THF), resulting in a homogeneous, yellow reaction mixture. The reaction mixture was stirred for 1 h at room temperature (RT) then directly filtered into 2 mL of a pre-cooled solution (-35 °C, 1 h) of PCl$_3$ (146 mg, 1.064 mmol, 1.1 equiv) in THF. The solution was warmed to RT for 1 h then analyzed by $^{31}$P($^1$H) NMR spectroscopy, revealing the presence of unreacted PCl$_3$ (218 ppm), i-PrPCl$_2$ (202 ppm) and the chlorophosphine (170 ppm). The “intermediate” product mixture was then concentrated under vacuum to remove the volatile and unwanted phosphorus byproducts (PCl$_3$ and i-PrPCl$_2$), leaving a pale yellow residue that was dissolved in 6 mL of THF and transferred to a Schlenk bomb fitted with a screw-top Teflon cap. The bomb was taken outside of the glovebox, cooled to 0 °C, and (p-CH$_3$)C$_6$H$_4$MgBr (1.06 mL, 1.064 mmol, 1.1 equiv, 1.0 M in THF) was injected via syringe under positive N$_2$ pressure, affording a light orange reaction mixture, which was subsequently warmed to RT. The Schlenk bomb was resealed (under positive N$_2$ pressure), brought back inside the glove box, and an aliquot was analyzed by $^{31}$P($^1$H) NMR spectroscopy displaying a prominent signal at 129 ppm with slight impurities at -2 and -8 ppm. The entire reaction mixture was concentrated under vacuum, extracted with pentane (3 x 50 mL), and filtered through a Kimwipe plug. The filtrate was concentrated under vacuum, dissolved in ether (2 mL) and cooled to -35 °C overnight, resulting in large white/colorless blocks suitable for X-ray diffraction (202 mg, 0.371 mmol, 38% yield).

Anal. Calcd for C$_{39}$H$_{13}$F$_{12}$O$_2$P: C, 44.14; H, 2.41. Found: C, 44.09; H, 2.41. $^1$H NMR (600 MHz, C$_6$D$_6$): $^\delta$ 7.57 (br d, $^J$ = 8 Hz, 1H, Ar), 7.52 (d, $^J$ = 8 Hz, 1H, Ar), 7.10 (apparent t, $^J$ = 8 Hz, 2H, Ar), 6.88 (t, $^J$ = 8 Hz, 1H, Ar), 6.81 (d, $^J$ = 8 Hz, 2H, Ar), 3.11 (s, 3H, OMe), 1.92 (s, 3H, p-Me). $^{13}$C($^1$H) NMR (151 MHz, C$_6$D$_6$): $^\delta$ 143.4 (d, $^J$ = 33 Hz, Ar), 141.2 (Ar), 137.8 (d, $^J$ = 41 Hz, Ar), 135.7 (d, $^J$ = 6 Hz, Ar), 133.0 (d, $^J$ = 5 Hz, Ar), 132.2 (d, $^J$ = 25 Hz, Ar), 131.1 (Ar), 130.0 (Ar), 129.0 (d, $^J$ = 8 Hz, Ar), 126.9 (Ar), four overlapping CF$_3$ signals*: 123.1 (qd, $^J$ = 290 and 9 Hz), 123.0 (q, $^J$ = 290 Hz), 122.5 (q, $^J$ = 290 Hz), and 122.4 (qd, $^J$ = 290 and 3 Hz), 89.5 (septet of doublets, $^J$ = 31.5 and 16.5 Hz, C(CF$_3$)$_2$ in P-ring), 80.8 (septet, $^J$ = 28 Hz, C(CF$_3$)$_2$), 55.3 (OMe), 21.2 (Me). $^{19}$F NMR (282 MHz, C$_6$D$_6$): $^\delta$ -69.5 (m, $^J_{FF}$ ~ 8 Hz, $^J_{HF}$ ~ 0.5-2 Hz), -69.6 (m, $^J_{FF}$ ~ 8 Hz, $^J_{HF}$ ~ 7 Hz, $^J_{HF}$ ~ 0.5-2.5 Hz), -74.0 (m, $^J_{FF}$ ~ 9 Hz, $^J_{HF}$ ~ 0.5-1 Hz), and -76.5 (m, $^J_{FF}$ ~ 9 Hz, $^J_{HF}$ ~ 1-5 Hz). $^{31}$P($^1$H) NMR (121 MHz, C$_6$D$_6$): $^\delta$ 129.4 (septet, $^J$ = 6 Hz). *In CDCl$_3$, the overlapping CF$_3$ signals in the $^{13}$C($^1$H) NMR spectrum are better resolved: $^\delta$ 122.5 (q, $^J$ = 290 Hz), 122.3 (qd, $^J$ = 290 and 9 Hz), 122.0 (q, $^J$ = 290 Hz), 121.6 (qd, $^J$ = 290 and 2 Hz). **Reported coupling constants in the $^{19}$F NMR spectrum were determined by simulation with Mestrenova.
$^1\text{H NMR (600 MHz, C}_6\text{D}_6\text{)}$ spectrum
$^{13}$C{$^1$H} NMR (151 MHz, CD$_6$) spectra
This is the CF$_3$ region in CDCl$_3$. The signals are better resolved.
DEPT-135
$^{19}$F NMR (282 MHz, CD$_6$) spectrum
$^{31}\text{P}^{('}\text{H})$ NMR (121 MHz, C$_6$D$_6$) spectrum

![NMR spectrum image]
Special Handling

The sample was transferred under argon and was combusted in a tin capsule that was crimp-sealed with a die apparatus.

Acknowledgment

Analytical data were obtained from the CENTC Elemental Analysis Facility at the University of Rochester, funded by NSF CHE-0650456.

Instrumentation

Microanalysis samples were weighed with a PerkinElmer Model AD6000 Autobalance and their compositions were determined with a PerkinElmer 2400 Series II Analyzer. Air-sensitive samples were handled in a VAC Atmospheres glovebox.
2. Simulated $^{19}$F NMR Spectra using MestreNova

The $^{19}$F spectra for 5 and 6 were simulated with MestreNova. All $^{19}$F and $^{31}$P spins were included in the simulation along with the aromatic protons. Number of points was matched to the experimental spectrum (16 K to 512 K depending on the experiment). Line widths were set at 0.9 Hz for spectra collected at 282 MHz and 1.1 Hz for spectra collected at 564 MHz. $^{31}$P and $^1$H chemical shifts were set to zero in the $^{19}$F simulations. Initial chemical shifts and coupling constants were estimated from the experimental spectrum then iteratively refined until the simulated pattern matched the experimental pattern. After simulating the $^{19}$F spectra at 282 MHz the resultant coupling constants were tested against the experimental $^{19}$F spectra collected at 564 MHz. The spectra were refined further through slight adjustments to the coupling constants until a suitable match was obtained for the low and high field spectrum.

Table S1. Simulated $^{19}$F chemical shifts and coupling constants for 5 at 282 MHz.

| $^{19}$F (ppm) | Label | CF$_3$ A | CF$_3$ A’ | CF$_3$ B | CF$_3$ B’ | P$_c$ | H$_d$ | H$_e$ | H$_f$ |
|---------------|-------|----------|-----------|----------|-----------|------|------|------|------|
| -68.9237      | CF$_3$ A |          |           |          |           |      |      |      |      |
| -70.4093      | CF$_3$ A’ | 7.93     |           |          |           |      |      |      |      |
| -73.3518      | CF$_3$ B |          |           |          |           |      |      |      |      |
| -76.2722      | CF$_3$ B’ | 9.34     |           |          |           |      |      |      |      |
| P$_c$         | 1.65   | 3.86     | 1.9       | 1.77     |           |      |      |      |      |
| H$_d$         | 1.25   | 2.58     |           |          |           |      |      |      |      |
| H$_e$         | 0.60   | 0.17     | 1.22      | 1.60     |           |      |      |      |      |
| H$_f$         | 1.35   | 2.25     | 0.25      | 0.35     | 0.10      | 0.70 |      |      |      |
**Figure S1.** Experimental (red) and simulated (blue) $^{19}$F signals at -68.9237 (A), -70.4093 (B), -73.3518 (C) and -76.2722 (D) for 5 at 282 MHz.
Figure S2. Experimental (red) and simulated (blue) $^{19}\text{F}$ signals at -68.9460 (A), -70.4300 (B), -73.3746 (C) and -76.2950 (D) for 5 at 564 MHz.
Table S2. Simulated $^{19}$F chemical shifts and coupling constants for 6 at 282 MHz.

![Chemical Structure of 6]

| $^{19}$F (ppm) | Label | CF$_3$ A | CF$_3$ A' | CF$_3$ B | CF$_3$ B' | P$_c$ | H$_d$ | H$_e$ | H$_f$ |
|---------------|-------|----------|-----------|----------|-----------|-------|-------|-------|-------|
| -68.4819      | CF$_3$ A   |          |           |          |           |       |       |       |       |
| -69.6176      | CF$_3$ A'  | 7.90     |           |          |           |       |       |       |       |
| -73.9876      | CF$_3$ B   |          |           |          |           |       |       |       |       |
| -76.5368      | CF$_3$ B'  |          |           |          | 9.30      |       |       |       |       |
| P$_c$         | 2.00    | 7.28     |           |          |           |       |       |       |       |
| H$_d$         | 1.65    | 2.36     |           |          |           |       |       |       |       |
| H$_e$         | 0.45    | 0.54     | 0.88      | 5.00     |           |       |       |       |       |
| H$_f$         | 0.9     | 1.35     | 0.4       | 1.30     | 0.1       | 0.5   | 0.5   |       |
Figure S3. Experimental (red) and simulated (blue) $^{19}$F signals at -69.4819 and -69.6176 (A), -73.9876 (B), and -76.5368 (C) for 6 at 282 MHz.
**Figure S4.** Experimental (red) and simulated (blue) $^{19}$F signals at -71.988 and -72.1280 (A), -76.4970 (B), and -79.0490 (C) for 6 at 564 MHz.
3. Experimental Details for Crystal Structure Refinement and Acquisition

All data were collected on Bruker Micro-Star rotating anode systems using micro-focus optics and APEX detectors. All, except CAIN100 (6), were collected using Mo-Kα radiation. Cu-Kα radiation was used for CAIN100 (6). CAIN58 (3) diffracted only weakly and no high-angle data were available (resolution = 0.86Å). All data sets were processed using current Bruker software and refinement was completed using OLEX2 programs. (1) Pseudo-mirror plane disorder was found in CAIN100 (6).

(1) Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H., OLEX2: A complete structure solution, refinement and analysis program (2009). J. Appl. Cryst., 42, 339-341.
Table 1. Crystal data and structure refinement for cain57_0m_a (1).

| Property                        | Value                        |
|---------------------------------|------------------------------|
| Identification code             | JH154                        |
| Empirical formula               | C26 H29 Br O2                |
| Formula weight                  | 453.40                       |
| Temperature                     | 100.0 K                      |
| Wavelength                      | 0.71073 Å                    |
| Crystal system                  | Triclinic                    |
| Space group                     | P-1                          |
| Unit cell dimensions            | a = 7.4093(13) Å, α = 101.293(4)°. |
|                                 | b = 8.2707(14) Å, β = 100.471(5)°. |
|                                 | c = 18.706(3) Å, γ = 94.032(6)°. |
| Volume                          | 1098.7(3) Å³                 |
| Z                               | 2                            |
| Density (calculated)            | 1.371 Mg/m³                  |
| Absorption coefficient          | 1.890 mm⁻¹                   |
| F(000)                          | 472                          |
| Crystal size                    | 0.32 x 0.3 x 0.28 mm³        |
| Theta range for data collection | 2.813 to 25.372°.            |
| Index ranges                    | -8 <= h <= 8, -8 <= k <= 9, -22 <= l <= 22 |
| Reflections collected           | 11445                        |
| Independent reflections         | 3990 [R(int) = 0.0341]       |
| Completeness to theta = 25.242° | 99.5 %                       |
| Absorption correction           | Semi-empirical from equivalents |
| Max. and min. transmission      | 0.2590 and 0.2112            |
| Refinement method               | Full-matrix least-squares on F² |
| Data / restraints / parameters  | 3990 / 0 / 268               |
| Goodness-of-fit on F²           | 1.092                        |
| Final R indices [I>2sigma(I)]   | R1 = 0.0465, wR2 = 0.1194    |
| R indices (all data)            | R1 = 0.0553, wR2 = 0.1231    |
| Extinction coefficient          | n/a                          |
| Largest diff. peak and hole     | 2.092 and -0.456 e.Å⁻³       |
Table 2. Atomic coordinates ($10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for cain57_0m_a (I). U(eq) is defined as one third of the trace of the orthogonalized $U^0$ tensor.

|     | x     | y     | z     | U(eq) |
|-----|-------|-------|-------|-------|
| Br(1)| 2058(1)| 1429(1)| 5060(1)| 22(1) |
| O(1) | 3198(3)| 4135(3)| 6829(1)| 17(1) |
| O(2) | 1719(3)| 2549(3)| 3248(1)| 18(1) |
| C(9) | 4142(5)| 3949(5)| 7521(2)| 17(1) |
| C(18)| 838(5) | 1614(4)| 2553(2)| 17(1) |
| C(13)| 5742(5)| 5178(5)| 8762(2)| 24(1) |
| C(24)| 386(5) | 4310(5)| 2127(2)| 23(1) |
| C(14)| 4820(5)| 5373(5)| 8068(2)| 19(1) |
| C(10)| 4363(5)| 2357(5)| 7652(2)| 20(1) |
| C(17)| 4616(5)| 7078(5)| 7915(2)| 24(1) |
| C(19)| 193(5) | 2451(5)| 1989(2)| 19(1) |
| C(2) | 3472(5)| 4811(4)| 5661(2)| 14(1) |
| C(12)| 6002(5)| 3620(5)| 8905(2)| 26(1) |
| C(21)| -882(5)| -224(5)| 1154(2)| 24(1) |
| C(6) | 1519(5)| 4236(4)| 4410(2)| 15(1) |
| C(5) | 1739(5)| 5923(4)| 4417(2)| 18(1) |
| C(20)| -663(5)| 1495(5)| 1296(2)| 23(1) |
| C(23)| 678(5) | -109(5)| 2425(2)| 20(1) |
| C(8) | 438(5) | 3069(4)| 3723(2)| 18(1) |
| C(11)| 5307(5)| 2227(5)| 8346(2)| 25(1) |
| C(7) | 2378(5)| 3714(4)| 5038(2)| 14(1) |
| C(3) | 3678(5)| 6474(4)| 5627(2)| 17(1) |
| C(15)| 3635(6)| 824(5) | 7058(2)| 27(1) |
| C(1) | 4457(5)| 4257(4)| 6333(2)| 16(1) |
| C(4) | 2820(5)| 7032(4)| 5021(2)| 20(1) |
| C(22)| -183(6)| -1003(5)| 1723(2)| 26(1) |
| C(26)| 1386(6)| -1006(5)| 3031(2)| 28(1) |
| C(25)| -1889(6)| -1229(6)| 403(2) | 37(1) |
| C(16)| 7060(6)| 3434(6)| 9649(2)| 37(1) |
Table 3. Bond lengths [Å] and angles [°] for cain57_0m_a (1).

| Bond | Length (Å) |
|------|------------|
| Br(1)-C(7) | 1.897(3) |
| O(1)-C(9) | 1.397(4) |
| O(1)-C(1) | 1.443(4) |
| O(2)-C(18) | 1.393(4) |
| O(2)-C(8) | 1.445(4) |
| C(9)-C(14) | 1.395(5) |
| C(9)-C(10) | 1.401(5) |
| C(18)-C(19) | 1.403(5) |
| C(18)-C(23) | 1.391(5) |
| C(13)-H(13) | 0.9500 |
| C(13)-C(14) | 1.396(5) |
| C(13)-C(12) | 1.386(6) |
| C(24)-H(24A) | 0.9800 |
| C(24)-H(24B) | 0.9800 |
| C(24)-H(24C) | 0.9800 |
| C(24)-C(19) | 1.501(5) |
| C(14)-C(17) | 1.506(5) |
| C(10)-C(11) | 1.387(5) |
| C(10)-C(15) | 1.506(5) |
| C(17)-H(17A) | 0.9800 |
| C(17)-H(17B) | 0.9800 |
| C(17)-H(17C) | 0.9800 |
| C(19)-C(20) | 1.391(5) |
| C(2)-C(7) | 1.406(5) |
| C(2)-C(3) | 1.389(5) |
| C(2)-C(1) | 1.503(5) |
| C(12)-C(11) | 1.391(6) |
| C(12)-C(16) | 1.510(5) |
| C(21)-C(20) | 1.387(5) |
| C(21)-C(22) | 1.389(6) |
| C(21)-C(25) | 1.516(5) |
| C(6)-C(5) | 1.390(5) |
| C(6)-C(8) | 1.502(5) |
| C(6)-C(7) | 1.392(5) |
C(5)-H(5) 0.9500
C(5)-C(4) 1.386(5)
C(20)-H(20) 0.9500
C(23)-C(22) 1.388(5)
C(23)-C(26) 1.510(5)
C(8)-H(8A) 0.9900
C(8)-H(8B) 0.9900
C(11)-H(11) 0.9500
C(3)-H(3) 0.9500
C(3)-C(4) 1.374(5)
C(15)-H(15A) 0.9800
C(15)-H(15B) 0.9800
C(15)-H(15C) 0.9800
C(1)-H(1A) 0.9900
C(1)-H(1B) 0.9900
C(4)-H(4) 0.9500
C(22)-H(22) 0.9500
C(26)-H(26A) 0.9800
C(26)-H(26B) 0.9800
C(26)-H(26C) 0.9800
C(25)-H(25A) 0.9800
C(25)-H(25B) 0.9800
C(25)-H(25C) 0.9800
C(16)-H(16A) 0.9800
C(16)-H(16B) 0.9800
C(16)-H(16C) 0.9800

C(9)-O(1)-C(1) 110.7(2)
C(18)-O(2)-C(8) 112.6(3)
O(1)-C(9)-C(10) 119.7(3)
C(14)-C(9)-O(1) 118.4(3)
C(14)-C(9)-C(10) 121.8(3)
O(2)-C(18)-C(19) 118.5(3)
C(23)-C(18)-O(2) 119.8(3)
C(23)-C(18)-C(19) 121.7(3)
C(14)-C(13)-H(13) 119.3
| Bond Description      | Distance (Å) |
|-----------------------|--------------|
| C(12)-C(13)-H(13)     | 119.3        |
| C(12)-C(13)-C(14)     | 121.4(4)     |
| H(24A)-C(24)-H(24B)   | 109.5        |
| H(24A)-C(24)-H(24C)   | 109.5        |
| H(24B)-C(24)-H(24C)   | 109.5        |
| C(19)-C(24)-H(24A)    | 109.5        |
| C(19)-C(24)-H(24B)    | 109.5        |
| C(19)-C(24)-H(24C)    | 109.5        |
| C(9)-C(14)-C(13)      | 118.1(3)     |
| C(9)-C(14)-C(17)      | 121.3(3)     |
| C(13)-C(14)-C(17)     | 120.6(3)     |
| C(9)-C(10)-C(15)      | 121.6(3)     |
| C(11)-C(10)-C(9)      | 117.9(3)     |
| C(11)-C(10)-C(15)     | 120.4(3)     |
| C(14)-C(17)-H(17A)    | 109.5        |
| C(14)-C(17)-H(17B)    | 109.5        |
| C(14)-C(17)-H(17C)    | 109.5        |
| H(17A)-C(17)-H(17B)   | 109.5        |
| H(17A)-C(17)-H(17C)   | 109.5        |
| H(17B)-C(17)-H(17C)   | 109.5        |
| C(18)-C(19)-C(24)     | 121.5(3)     |
| C(20)-C(19)-C(18)     | 117.7(3)     |
| C(20)-C(19)-C(24)     | 120.8(3)     |
| C(7)-C(2)-C(1)        | 123.3(3)     |
| C(3)-C(2)-C(7)        | 116.9(3)     |
| C(3)-C(2)-C(1)        | 119.7(3)     |
| C(13)-C(12)-C(11)     | 118.9(4)     |
| C(13)-C(12)-C(16)     | 120.7(4)     |
| C(11)-C(12)-C(16)     | 120.4(4)     |
| C(20)-C(21)-C(22)     | 118.4(3)     |
| C(20)-C(21)-C(25)     | 120.8(4)     |
| C(22)-C(21)-C(25)     | 120.8(4)     |
| C(5)-C(6)-C(8)        | 119.0(3)     |
| C(5)-C(6)-C(7)        | 117.6(3)     |
| C(7)-C(6)-C(8)        | 123.4(3)     |
| C(6)-C(5)-H(5)        | 119.5        |
| Bond                  | Angle (°)  |
|----------------------|------------|
| C(4)-C(5)-C(6)       | 121.0(3)  |
| C(4)-C(5)-H(5)       | 119.5     |
| C(19)-C(20)-H(20)    | 118.9     |
| C(21)-C(20)-C(19)    | 122.1(4)  |
| C(21)-C(20)-H(20)    | 118.9     |
| C(18)-C(23)-C(26)    | 121.6(3)  |
| C(22)-C(23)-C(18)    | 118.3(3)  |
| C(22)-C(23)-C(26)    | 120.1(3)  |
| O(2)-C(8)-C(6)       | 107.5(3)  |
| O(2)-C(8)-H(8A)      | 110.2     |
| O(2)-C(8)-H(8B)      | 110.2     |
| C(6)-C(8)-H(8A)      | 110.2     |
| C(6)-C(8)-H(8B)      | 110.2     |
| H(8A)-C(8)-H(8B)     | 108.5     |
| C(10)-C(11)-C(12)    | 121.8(4)  |
| C(10)-C(11)-H(11)    | 119.1     |
| C(12)-C(11)-H(11)    | 119.1     |
| C(2)-C(7)-Br(1)      | 118.3(3)  |
| C(6)-C(7)-Br(1)      | 118.9(3)  |
| C(6)-C(7)-C(2)       | 122.8(3)  |
| C(2)-C(3)-H(3)       | 119.2     |
| C(4)-C(3)-C(2)       | 121.7(3)  |
| C(4)-C(3)-H(3)       | 119.2     |
| C(10)-C(15)-H(15A)   | 109.5     |
| C(10)-C(15)-H(15B)   | 109.5     |
| C(10)-C(15)-H(15C)   | 109.5     |
| H(15A)-C(15)-H(15B)  | 109.5     |
| H(15A)-C(15)-H(15C)  | 109.5     |
| H(15B)-C(15)-H(15C)  | 109.5     |
| O(1)-C(1)-C(2)       | 109.0(3)  |
| O(1)-C(1)-H(1A)      | 109.9     |
| O(1)-C(1)-H(1B)      | 109.9     |
| C(2)-C(1)-H(1A)      | 109.9     |
| C(2)-C(1)-H(1B)      | 109.9     |
| H(1A)-C(1)-H(1B)     | 108.3     |
| C(5)-C(4)-H(4)       | 120.0     |
C(3)-C(4)-C(5) 120.1(3)
C(3)-C(4)-H(4) 120.0
C(21)-C(22)-H(22) 119.1
C(23)-C(22)-C(21) 121.8(4)
C(23)-C(22)-H(22) 119.1
C(23)-C(26)-H(26A) 109.5
C(23)-C(26)-H(26B) 109.5
C(23)-C(26)-H(26C) 109.5
H(26A)-C(26)-H(26B) 109.5
H(26A)-C(26)-H(26C) 109.5
H(26B)-C(26)-H(26C) 109.5
C(21)-C(25)-H(25A) 109.5
C(21)-C(25)-H(25B) 109.5
C(21)-C(25)-H(25C) 109.5
H(25A)-C(25)-H(25B) 109.5
H(25A)-C(25)-H(25C) 109.5
H(25B)-C(25)-H(25C) 109.5
C(12)-C(16)-H(16A) 109.5
C(12)-C(16)-H(16B) 109.5
C(12)-C(16)-H(16C) 109.5
H(16A)-C(16)-H(16B) 109.5
H(16A)-C(16)-H(16C) 109.5
H(16B)-C(16)-H(16C) 109.5

Symmetry transformations used to generate equivalent atoms:
Table 4. Anisotropic displacement parameters (Å$^2 \times 10^3$) for cain57_0m_a (1). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^2 U^{11} + \ldots + 2hk a^* b^* U^{12}]$

|          | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{23}$ | $U^{13}$ | $U^{12}$ |
|----------|----------|----------|----------|----------|----------|----------|
| Br(1)    | 33(1)    | 12(1)    | 20(1)    | 4(1)     | 1(1)     | -1(1)    |
| O(1)     | 15(1)    | 24(1)    | 13(1)    | 7(1)     | 3(1)     | 2(1)     |
| O(2)     | 14(1)    | 24(1)    | 14(1)    | 0(1)     | 3(1)     | 4(1)     |
| C(9)     | 13(2)    | 26(2)    | 13(2)    | 4(1)     | 3(1)     | 2(1)     |
| C(18)    | 12(2)    | 25(2)    | 11(2)    | -2(1)    | 1(1)     | -1(1)    |
| C(13)    | 20(2)    | 35(2)    | 16(2)    | 2(2)     | 4(2)     | 2(2)     |
| C(24)    | 24(2)    | 24(2)    | 22(2)    | 6(2)     | 4(2)     | 2(2)     |
| C(14)    | 16(2)    | 24(2)    | 17(2)    | 3(2)     | 6(2)     | 4(2)     |
| C(10)    | 20(2)    | 23(2)    | 20(2)    | 5(2)     | 7(2)     | 3(2)     |
| C(17)    | 25(2)    | 21(2)    | 24(2)    | 0(2)     | 6(2)     | 2(2)     |
| C(19)    | 16(2)    | 24(2)    | 17(2)    | 5(2)     | 6(2)     | 3(2)     |
| C(2)     | 12(2)    | 16(2)    | 14(2)    | 1(1)     | 5(1)     | 2(1)     |
| C(12)    | 22(2)    | 41(2)    | 17(2)    | 9(2)     | 4(2)     | 5(2)     |
| C(21)    | 23(2)    | 29(2)    | 18(2)    | -4(2)    | 6(2)     | -3(2)    |
| C(6)     | 10(2)    | 21(2)    | 16(2)    | 3(1)     | 6(1)     | 3(1)     |
| C(5)     | 20(2)    | 24(2)    | 15(2)    | 8(2)     | 6(2)     | 8(2)     |
| C(20)    | 24(2)    | 31(2)    | 14(2)    | 5(2)     | 3(2)     | 5(2)     |
| C(23)    | 21(2)    | 23(2)    | 20(2)    | 6(2)     | 8(2)     | 4(2)     |
| C(8)     | 14(2)    | 25(2)    | 14(2)    | 4(2)     | 2(1)     | 3(1)     |
| C(11)    | 28(2)    | 27(2)    | 24(2)    | 12(2)    | 7(2)     | 7(2)     |
| C(7)     | 15(2)    | 10(2)    | 17(2)    | 2(1)     | 7(1)     | 2(1)     |
| C(3)     | 20(2)    | 15(2)    | 18(2)    | 0(1)     | 7(2)     | 1(1)     |
| C(15)    | 31(2)    | 21(2)    | 28(2)    | 5(2)     | 6(2)     | -1(2)    |
| C(1)     | 14(2)    | 18(2)    | 16(2)    | 3(1)     | 4(1)     | 1(1)     |
| C(4)     | 24(2)    | 14(2)    | 25(2)    | 6(2)     | 10(2)    | 4(2)     |
| C(22)    | 33(2)    | 19(2)    | 24(2)    | -2(2)    | 10(2)    | 0(2)     |
| C(26)    | 36(2)    | 28(2)    | 23(2)    | 9(2)     | 6(2)     | 12(2)    |
| C(25)    | 39(3)    | 42(3)    | 21(2)    | -7(2)    | 1(2)     | -5(2)    |
| C(16)    | 37(3)    | 51(3)    | 23(2)    | 16(2)    | -1(2)    | 6(2)     |
Table 5. Hydrogen coordinates (x $10^4$) and isotropic displacement parameters (Å$^2x 10^{-3}$) for cain57_0m_a (1).

|     | x     | y     | z     | U(eq) |
|-----|-------|-------|-------|-------|
| H(13)| 6202  | 6133  | 9143  | 29    |
| H(24A)| -582 | 4731  | 2383  | 35    |
| H(24B)| 264  | 4661  | 1651  | 35    |
| H(24C)| 1600 | 4753  | 2436  | 35    |
| H(17A)| 5573 | 7379  | 7651  | 36    |
| H(17B)| 4746 | 7883  | 8386  | 36    |
| H(17C)| 3395 | 7082  | 7608  | 36    |
| H(5) | 1139  | 6320  | 4002  | 22    |
| H(20)| -1112 | 2038  | 907   | 27    |
| H(8A)| -164 | 2095  | 3857  | 21    |
| H(8B)| -530 | 3634  | 3463  | 21    |
| H(11)| 5483 | 1158  | 8443  | 30    |
| H(3) | 4431 | 7246  | 6033  | 21    |
| H(15A)| 2684 | 1098  | 6676  | 40    |
| H(15B)| 3100 | -26   | 7280  | 40    |
| H(15C)| 4648 | 403   | 6830  | 40    |
| H(1A)| 4900 | 3166  | 6178  | 19    |
| H(1B)| 5540 | 5066  | 6590  | 19    |
| H(4) | 2967 | 8180  | 5015  | 24    |
| H(22)| -298 | -2181 | 1629  | 31    |
| H(26A)| 2231 | -233  | 3438  | 42    |
| H(26B)| 2042 | -1924 | 2830  | 42    |
| H(26C)| 343  | -1444 | 3219  | 42    |
| H(25A)| -2900 | -640  | 202   | 55    |
| H(25B)| -2391 | -2314 | 459   | 55    |
| H(25C)| -1026 | -1381 | 61    | 55    |
| H(16A)| 8368 | 3818  | 9700  | 56    |
| H(16B)| 6932 | 2264  | 9682  | 56    |
| H(16C)| 6566 | 4098  | 10049 | 56    |
**X-RAY OF “HALF TRIFLUOROMETHYLATED”**

Table 1. Crystal data and structure refinement for cain80_0m_a (“HALF”).

| Identification code        | RC.BiZ      |
|----------------------------|-------------|
| Empirical formula          | C11 H7 Br F6 O3 |
| Formula weight             | 381.08      |
| Temperature                | 100.0 K     |
| Wavelength                 | 0.71073 Å   |
| Crystal system             | Monoclinic  |
| Space group                | C c         |
| Unit cell dimensions       |             |
| a = 13.9233(7) Å           | α = 90°     |
| b = 14.4415(9) Å           | β = 100.826(2)° |
| c = 12.9349(7) Å           | γ = 90°     |
| Volume                     | 2554.6(2) Å³|
| Z                          | 8           |
| Density (calculated)       | 1.982 Mg/m³ |
| Absorption coefficient     | 3.300 mm⁻¹ |
| F(000)                     | 1488        |
| Crystal size               | 0.32 x 0.31 x 0.28 mm³ |
| Theta range for data collection | 2.051 to 28.287° |
| Index ranges               | -16<=h<=18, -19<=k<=16, -16<=l<=17 |
| Reflections collected      | 8847        |
| Independent reflections    | 4616 [R(int) = 0.0348] |
| Completeness to theta = 25.000° | 99.9 %   |
| Absorption correction      | Semi-empirical from equivalents |
| Max. and min. transmission | 0.7457 and 0.5761 |
| Refinement method          | Full-matrix least-squares on F² |
| Data / restraints / parameters | 4616 / 2 / 386 |
| Goodness-of-fit on F²      | 0.833       |
| Final R indices [I>2sigma(I)] | R1 = 0.0230, wR2 = 0.0533 |
| R indices (all data)       | R1 = 0.0251, wR2 = 0.0541 |
| Absolute structure parameter | 0.007(5)    |
| Extinction coefficient     | n/a         |
| Largest diff. peak and hole | 0.414 and -0.312 e.Å⁻³ |
Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for cain80_0m_a ("HALF"). U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

|   | x       | y       | z       | U(eq) |
|---|---------|---------|---------|-------|
| Br(1') | 5669(1) | 5628(1) | 6811(1) | 14(1) |
| Br(1)  | 5256(1) | 1266(1) | 6661(1) | 14(1) |
| F(1)   | 5475(2) | -755(2) | 5767(2) | 19(1) |
| F(4')  | 2685(2) | 6101(2) | 4523(2) | 23(1) |
| F(2')  | 2736(2) | 4670(2) | 3218(2) | 23(1) |
| F(4)   | 3386(2) | -766(2) | 3017(2) | 23(1) |
| F(6')  | 3417(2) | 5943(2) | 6139(2) | 23(1) |
| O(1')  | 4166(2) | 4280(2) | 5760(2) | 14(1) |
| F(6)   | 2601(2) | 365(2)  | 3590(2) | 21(1) |
| F(3)   | 4336(2) | -1719(2) | 5146(2) | 23(1) |
| O(1)   | 3687(2) | -25(2)  | 5679(2) | 14(1) |
| F(1')  | 4032(2) | 3812(2) | 3564(2) | 22(1) |
| O(3)   | 5924(2) | 3525(2) | 5804(2) | 20(1) |
| F(2)   | 5198(2) | -1092(2) | 4116(2) | 23(1) |
| O(3')  | 7750(2) | 6609(2) | 5833(2) | 18(1) |
| F(3')  | 2821(2) | 3569(2) | 4340(2) | 28(1) |
| F(5)   | 2584(2) | -996(2) | 4257(2) | 24(1) |
| O(2)   | 7081(2) | 2437(2) | 6001(2) | 16(1) |
| O(2')  | 6683(2) | 7612(2) | 6297(2) | 15(1) |
| F(5')  | 2288(2) | 4985(2) | 5464(2) | 32(1) |
| C(10)  | 6211(2) | 2765(2) | 5604(2) | 12(1) |
| C(11') | 7487(2) | 8046(3) | 7012(3) | 19(1) |
| C(11)  | 7693(2) | 3018(3) | 6772(3) | 19(1) |
| C(10') | 6923(2) | 6889(2) | 5762(2) | 12(1) |
| C(5')  | 5403(2) | 5886(2) | 5345(2) | 11(1) |
| C(9)   | 4632(2) | 886(3)  | 3376(3) | 14(1) |
| C(8)   | 5130(2) | 1629(3) | 3045(3) | 15(1) |
| C(3)   | 4774(3) | -937(3) | 4945(3) | 16(1) |
| C(4')  | 4622(2) | 5516(2) | 4612(3) | 10(1) |
| C(5)   | 5130(2) | 1355(2) | 5178(2) | 10(1) |
| C(4)   | 4602(2) | 738(2)  | 4441(2) | 10(1) |
|   |      |      |      |        |
|---|------|------|------|--------|
| C(6) | 5633(2) | 2104(2) | 4833(3) | 12(1) |
| C(9') | 4529(2) | 5805(2) | 3567(3) | 14(1) |
| C(2') | 3835(2) | 4872(2) | 4916(3) | 12(1) |
| C(7') | 5938(2) | 6781(3) | 3974(3) | 15(1) |
| C(6') | 6061(2) | 6507(2) | 5014(3) | 11(1) |
| C(8') | 5169(3) | 6434(3) | 3242(3) | 16(1) |
| C(3') | 3339(3) | 4235(3) | 3989(3) | 18(1) |
| C(1') | 3041(3) | 5484(3) | 5266(3) | 18(1) |
| C(2) | 4052(2) | -115(2) | 4749(2) | 12(1) |
| C(1) | 3148(3) | -381(3) | 3887(3) | 17(1) |
| C(7) | 5624(2) | 2247(2) | 3776(3) | 14(1) |
Table 3. Bond lengths [Å] and angles [°] for cain80_0m_a ("HALF").

| Bond                  | Length  |
|-----------------------|---------|
| Br(1')-C(5)           | 1.900(3) |
| Br(1)-C(5)            | 1.898(3) |
| F(1)-C(3)             | 1.327(4) |
| F(4')-C(1')           | 1.336(4) |
| F(2')-C(3')           | 1.334(4) |
| F(4)-C(1)             | 1.352(4) |
| F(6')-C(1')           | 1.330(4) |
| O(1')-H(1')           | 0.8400   |
| O(1')-C(2')           | 1.395(4) |
| F(6)-C(1)             | 1.333(4) |
| F(3)-C(3)             | 1.332(4) |
| O(1)-C(2)             | 1.396(4) |
| O(1)-H(1)             | 0.78(4)  |
| F(1')-C(3')           | 1.344(4) |
| O(3)-C(10)            | 1.211(4) |
| F(2)-C(3)             | 1.336(4) |
| O(3')-C(10')          | 1.208(4) |
| F(3')-C(3')           | 1.333(4) |
| F(5)-C(1)             | 1.332(4) |
| O(2)-C(10)            | 1.312(4) |
| O(2)-C(11)            | 1.451(4) |
| O(2')-C(11')          | 1.453(4) |
| O(2')-C(10')          | 1.329(4) |
| F(5')-C(1')           | 1.336(4) |
| C(10)-C(6)            | 1.502(5) |
| C(11')-H(11A)         | 0.9800   |
| C(11')-H(11B)         | 0.9800   |
| C(11')-H(11C)         | 0.9800   |
| C(11)-H(11D)          | 0.9800   |
| C(11)-H(11E)          | 0.9800   |
| C(11)-H(11F)          | 0.9800   |
| C(10')-C(6')          | 1.498(5) |
| C(5')-C(4')           | 1.407(5) |
| C(5')-C(6')           | 1.405(4) |
C(9)-H(9) 0.9500
C(9)-C(8) 1.388(5)
C(9)-C(4) 1.403(4)
C(8)-H(8) 0.9500
C(8)-C(7) 1.385(5)
C(3)-C(2) 1.546(5)
C(4')-C(9') 1.396(4)
C(4')-C(2') 1.544(4)
C(5)-C(4) 1.408(5)
C(5)-C(6) 1.406(5)
C(4)-C(2) 1.542(4)
C(6)-C(7) 1.380(4)
C(9')-H(9') 0.9500
C(9')-C(8') 1.392(5)
C(2')-C(3') 1.565(5)
C(2')-C(1') 1.547(5)
C(7')-H(7') 0.9500
C(7')-C(6') 1.383(5)
C(7')-C(8') 1.383(5)
C(8')-H(8') 0.9500
C(2)-C(1) 1.565(5)
C(7)-H(7) 0.9500

C(2')-O(1')-H(1') 109.5
C(2)-O(1)-H(1) 108(3)
C(10)-O(2)-C(11) 116.1(3)
C(10')-O(2')-C(11') 115.5(3)
O(3)-C(10)-O(2) 124.1(3)
O(3)-C(10)-C(6) 124.1(3)
O(2)-C(10)-C(6) 111.7(3)
O(2')-C(11')-H(11A) 109.5
O(2')-C(11')-H(11B) 109.5
O(2')-C(11')-H(11C) 109.5
H(11A)-C(11')-H(11B) 109.5
H(11A)-C(11')-H(11C) 109.5
H(11B)-C(11')-H(11C) 109.5
O(2)-C(11)-H(11D) 109.5
O(2)-C(11)-H(11E) 109.5
O(2)-C(11)-H(11F) 109.5
H(11D)-C(11)-H(11E) 109.5
H(11D)-C(11)-H(11F) 109.5
H(11E)-C(11)-H(11F) 109.5
O(3')-C(10')-O(2') 123.6(3)
O(3')-C(10')-C(6') 124.2(3)
O(2')-C(10')-C(6') 112.1(3)
C(4')-C(5')-Br(1') 125.4(2)
C(6')-C(5')-Br(1') 114.4(2)
C(6')-C(5')-C(4') 120.2(3)
C(8)-C(9)-H(9) 119.0
C(8)-C(9)-C(4) 121.9(3)
C(4)-C(9)-H(9) 119.0
C(9)-C(8)-H(8) 120.0
C(7)-C(8)-C(9) 120.1(3)
C(7)-C(8)-H(8) 120.0
F(1)-C(3)-F(3) 107.9(3)
F(1)-C(3)-F(2) 107.9(3)
F(1)-C(3)-C(2) 109.7(3)
F(3)-C(3)-F(2) 107.6(3)
F(3)-C(3)-C(2) 112.0(3)
F(2)-C(3)-C(2) 111.6(3)
C(5')-C(4')-C(2') 123.6(3)
C(9')-C(4')-C(5') 117.3(3)
C(9')-C(4')-C(2') 119.0(3)
C(4)-C(5)-Br(1) 125.4(2)
C(6)-C(5)-Br(1) 114.6(2)
C(6)-C(5)-C(4) 120.0(3)
C(9)-C(4)-C(5) 117.4(3)
C(9)-C(4)-C(2) 118.9(3)
C(5)-C(4)-C(2) 123.5(3)
C(5)-C(6)-C(10) 121.0(3)
C(7)-C(6)-C(10) 117.8(3)
C(7)-C(6)-C(5) 121.2(3)
| Bond                  | Angle (°) |
|-----------------------|-----------|
| C(4')-C(9')-H(9')    | 118.7     |
| C(8')-C(9')-C(4')    | 122.6(3)  |
| C(8')-C(9')-H(9')    | 118.7     |
| O(1')-C(2')-C(4')    | 115.0(3)  |
| O(1')-C(2')-C(3')    | 105.8(3)  |
| O(1')-C(2')-C(1')    | 105.6(3)  |
| C(4')-C(2')-C(3')    | 113.0(3)  |
| C(4')-C(2')-C(1')    | 108.1(3)  |
| C(1')-C(2')-C(3')    | 108.9(3)  |
| C(6')-C(7')-H(7')    | 120.0     |
| C(6')-C(7')-C(8')    | 120.0(3)  |
| C(8')-C(7')-H(7')    | 120.0     |
| C(5')-C(6')-C(10')   | 121.9(3)  |
| C(7')-C(6')-C(10')   | 117.4(3)  |
| C(7')-C(6')-C(5')    | 120.7(3)  |
| C(9')-C(8')-H(8')    | 120.4     |
| C(7')-C(8')-C(9')    | 119.2(3)  |
| C(7')-C(8')-H(8')    | 120.4     |
| F(2')-C(3')-F(1')    | 107.9(3)  |
| F(2')-C(3')-C(2')    | 114.9(3)  |
| F(1')-C(3')-C(2')    | 109.5(3)  |
| F(3')-C(3')-F(2')    | 106.8(3)  |
| F(3')-C(3')-F(1')    | 106.7(3)  |
| F(3')-C(3')-C(2')    | 110.6(3)  |
| F(4')-C(1')-F(5')    | 107.4(3)  |
| F(4')-C(1')-C(2')    | 111.1(3)  |
| F(6')-C(1')-F(4')    | 108.1(3)  |
| F(6')-C(1')-F(5')    | 107.7(3)  |
| F(6')-C(1')-C(2')    | 110.2(3)  |
| F(5')-C(1')-C(2')    | 112.2(3)  |
| O(1)-C(2)-C(3)       | 105.1(3)  |
| O(1)-C(2)-C(4)       | 115.3(3)  |
| O(1)-C(2)-C(1)       | 105.2(2)  |
| C(3)-C(2)-C(1)       | 109.9(3)  |
| C(4)-C(2)-C(3)       | 108.4(2)  |
| C(4)-C(2)-C(1)       | 112.6(3)  |
F(4)-C(1)-C(2)  113.8(3)
F(6)-C(1)-F(4)  107.9(3)
F(6)-C(1)-C(2)  110.3(3)
F(5)-C(1)-F(4)  106.1(3)
F(5)-C(1)-F(6)  107.4(3)
F(5)-C(1)-C(2)  110.9(3)
C(8)-C(7)-H(7)  120.3
C(6)-C(7)-C(8)  119.4(3)
C(6)-C(7)-H(7)  120.3

Symmetry transformations used to generate equivalent atoms:
Table 4. Anisotropic displacement parameters (Å² x 10³) for cain80_0m_a ("HALF"). The anisotropic displacement factor exponent takes the form: $-2\pi^2[ h^2 a^* U_{11} + ... + 2h k a^* b^* U_{12} ]$

|        | U_{11} | U_{22} | U_{33} | U_{23} | U_{13} | U_{12} |
|--------|--------|--------|--------|--------|--------|--------|
| Br(1') | 18(1)  | 13(1)  | 10(1)  | 1(1)   | 1(1)   | -2(1)  |
| Br(1)  | 17(1)  | 17(1)  | 9(1)   | -1(1)  | 2(1)   | -5(1)  |
| F(1)   | 17(1)  | 17(1)  | 23(1)  | 3(1)   | 2(1)   | 5(1)   |
| F(4')  | 22(1)  | 28(1)  | 20(1)  | 4(1)   | 3(1)   | 15(1)  |
| F(2')  | 17(1)  | 30(1)  | 18(1)  | -1(1)  | -4(1)  | -1(1)  |
| F(4)   | 27(1)  | 23(1)  | 18(1)  | -9(1)  | 4(1)   | -7(1)  |
| F(6')  | 22(1)  | 31(1)  | 17(1)  | -5(1)  | 3(1)   | 14(1)  |
| O(1')  | 12(1)  | 15(1)  | 16(1)  | 5(1)   | 4(1)   | 1(1)   |
| F(6)   | 14(1)  | 22(1)  | 25(1)  | -1(1)  | -4(1)  | 2(1)   |
| O(3)   | 28(1)  | 10(1)  | 34(1)  | 2(1)   | 11(1)  | 0(1)   |
| O(1)   | 16(1)  | 13(1)  | 14(1)  | -1(1)  | 6(1)   | -1(1)  |
| F(1')  | 23(1)  | 17(1)  | 26(1)  | -9(1)  | 3(1)   | 1(1)   |
| O(3')  | 18(1)  | 13(1)  | 28(1)  | -6(1)  | 3(1)   | 2(1)   |
| F(2)   | 30(1)  | 19(1)  | 23(1)  | 1(1)   | 14(1)  | 11(1)  |
| O(3')  | 12(1)  | 18(1)  | 23(1)  | -3(1)  | 2(1)   | 4(1)   |
| F(3)   | 27(1)  | 28(1)  | 27(1)  | 3(1)   | -1(1)  | -17(1) |
| F(5)   | 22(1)  | 25(1)  | 25(1)  | -2(1)  | 4(1)   | -12(1) |
| O(2)   | 10(1)  | 14(1)  | 23(1)  | -6(1)  | -1(1)  | 2(1)   |
| O(2')  | 13(1)  | 11(1)  | 21(1)  | -6(1)  | 2(1)   | 1(1)   |
| F(5')  | 15(1)  | 40(2)  | 45(1)  | 6(1)   | 16(1)  | 2(1)   |
| C(10)  | 12(1)  | 12(2)  | 14(2)  | -2(1)  | 5(1)   | -2(1)  |
| C(11') | 16(2)  | 16(2)  | 23(2)  | -7(1)  | 1(1)   | -3(1)  |
| C(11)  | 14(2)  | 21(2)  | 22(2)  | -5(2)  | -1(1)  | -4(1)  |
| C(10') | 15(2)  | 11(2)  | 11(1)  | 1(1)   | 3(1)   | 1(1)   |
| C(5')  | 14(2)  | 11(2)  | 7(1)   | -1(1)  | 2(1)   | 3(1)   |
| C(9)   | 13(2)  | 13(2)  | 13(2)  | -1(1)  | 1(1)   | 3(1)   |
| C(8)   | 15(2)  | 20(2)  | 9(2)   | 4(1)   | 2(1)   | 2(1)   |
| C(3)   | 22(2)  | 12(2)  | 15(2)  | 1(1)   | 8(1)   | 2(1)   |
| C(4')  | 10(1)  | 7(2)   | 14(2)  | 0(1)   | 4(1)   | 3(1)   |
| C(5)   | 9(1)   | 11(2)  | 9(1)   | 1(1)   | 1(1)   | 5(1)   |
| C(4)   | 9(1)   | 10(2)  | 13(2)  | -1(1)  | 1(1)   | 2(1)   |
|     |     |     |     |     |     |     |
|-----|-----|-----|-----|-----|-----|-----|
| C(6)| 7(1)| 12(2)| 16(2)| -2(1)| 2(1)| 3(1)|
| C(9')| 16(2)| 12(2)| 14(2)| 0(1)| 3(1)| 3(1)|
| C(2')| 9(1)| 12(2)| 16(2)| 1(1)| 3(1)| -1(1)|
| C(7')| 17(2)| 12(2)| 16(2)| 0(1)| 7(1)| -1(1)|
| C(6')| 10(1)| 9(2)| 16(2)| -3(1)| 3(1)| 2(1)|
| C(8')| 22(2)| 14(2)| 13(2)| -1(1)| 6(1)| -1(1)|
| C(3')| 18(2)| 16(2)| 17(2)| 2(1)| 0(1)| -4(1)|
| C(1')| 14(2)| 20(2)| 18(2)| 1(1)| 3(1)| 5(1)|
| C(2)| 12(1)| 10(2)| 13(2)| -1(1)| 3(1)| 0(1)|
| C(1)| 17(2)| 16(2)| 16(2)| -2(1)| 4(1)| -1(1)|
| C(7)| 12(2)| 14(2)| 16(2)| 2(1)| 4(1)| 1(1)|
Table 5. Hydrogen coordinates \( \times 10^4 \) and isotropic displacement parameters \( \AA^2 \times 10^{-3} \) for cain80_0m_a ("HALF").

|     | x     | y     | z     | U(eq) |
|-----|-------|-------|-------|-------|
| H(1') | 4705  | 4050  | 5694  | 21    |
| H(11A) | 7985  | 8249  | 6617  | 28    |
| H(11B) | 7775  | 7598  | 7552  | 28    |
| H(11C) | 7243  | 8582  | 7348  | 28    |
| H(11D) | 7844  | 3594  | 6436  | 29    |
| H(11E) | 8302  | 2690  | 7055  | 29    |
| H(11F) | 7347  | 3161  | 7346  | 29    |
| H(9)   | 4301  | 465   | 2865  | 16    |
| H(8)   | 5132  | 1713  | 2317  | 18    |
| H(9')  | 4008  | 5563  | 3059  | 17    |
| H(7')  | 6381  | 7209  | 3761  | 18    |
| H(8')  | 5078  | 6622  | 2527  | 19    |
| H(7)   | 5953  | 2764  | 3552  | 17    |
| H(1)   | 3450(30) | 460(30) | 5680(30) | 12(11) |
X-RAY OF 2

Table 1. Crystal data and structure refinement for cain84_0m_a (2).

| Identification code | B31Cs |
|---------------------|-------|
| Empirical formula   | C14 H9 Br F12 O2 |
| Formula weight      | 517.12 |
| Temperature         | 100.0 K |
| Wavelength          | 0.71073 Å |
| Crystal system      | Monoclinic |
| Space group         | P 21/n |
| Unit cell dimensions|          |
| a                   | 11.9324(10) Å  α = 90°. |
| b                   | 12.4694(12) Å  β = 116.905(4)°. |
| c                   | 12.4948(10) Å  γ = 90°. |
| Volume              | 1657.9(3) Å³ |
| Z                   | 4 |
| Density (calculated)| 2.072 Mg/m³ |
| Absorption coefficient| 2.618 mm⁻¹ |
| F(000)              | 1008 |
| Crystal size        | 0.33 x 0.32 x 0.25 mm³ |
| Theta range for data collection | 1.959 to 26.386°. |
| Index ranges        | -14<=h<=14, -13<=k<=15, -15<=l<=11 |
| Reflections collected| 8685 |
| Independent reflections | 3370 [R(int) = 0.0365] |
| Completeness to theta = 25.242° | 99.7 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.7454 and 0.6400 |
| Refinement method   | Full-matrix least-squares on F² |
| Data / restraints / parameters | 3370 / 0 / 264 |
| Goodness-of-fit on F² | 1.075 |
| Final R indices [I>2σ(I)] | R1 = 0.0464, wR2 = 0.1265 |
| R indices (all data) | R1 = 0.0573, wR2 = 0.1341 |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 1.690 and -0.525 e.Å⁻³ |
Table 2. Atomic coordinates $(x \times 10^4)$ and equivalent isotropic displacement parameters $(\text{Å}^2 \times 10^3)$ for cain84_0m_a (2). $U(eq)$ is defined as one third of the trace of the orthogonalized $U^\alpha$ tensor.

|    | x     | y     | z     | $U(eq)$ |
|----|-------|-------|-------|---------|
| Br(1) | 5171(1) | 2633(1) | 2871(1) | 17(1) |
| F(12) | 8013(2) | 3404(2) | 4264(2) | 24(1) |
| F(11) | 9253(2) | 3791(2) | 3493(2) | 25(1) |
| F(8) | 8153(2) | 5668(2) | 1954(3) | 27(1) |
| F(7) | 6437(2) | 5126(2) | 449(2) | 22(1) |
| F(10) | 8458(2) | 5052(2) | 4111(2) | 25(1) |
| F(9) | 8120(2) | 4180(2) | 1070(2) | 26(1) |
| O(2) | 6912(3) | 3082(2) | 1916(2) | 14(1) |
| F(5) | 1598(3) | 5632(2) | 2774(3) | 36(1) |
| F(1) | 4091(3) | 3270(2) | 4573(2) | 32(1) |
| O(1) | 2554(3) | 3103(3) | 2224(3) | 21(1) |
| F(3) | 3582(3) | 4911(2) | 4645(3) | 33(1) |
| F(6) | 636(3) | 4208(3) | 1895(3) | 39(1) |
| F(4) | 1446(3) | 5233(2) | 1045(3) | 36(1) |
| F(2) | 2202(3) | 3658(3) | 4177(3) | 37(1) |
| C(1) | 5002(3) | 4135(3) | 2566(3) | 10(1) |
| C(6) | 5938(3) | 4689(3) | 2399(3) | 9(1) |
| C(4) | 4934(4) | 6364(3) | 2454(4) | 15(1) |
| C(10) | 2187(4) | 2988(4) | 949(4) | 20(1) |
| C(5) | 5899(4) | 5808(3) | 2377(4) | 13(1) |
| C(2) | 3971(4) | 4685(3) | 2578(3) | 11(1) |
| C(12) | 8208(4) | 4099(3) | 3547(4) | 17(1) |
| C(3) | 3967(4) | 5803(3) | 2528(4) | 13(1) |
| C(13) | 7449(4) | 4792(3) | 1437(4) | 18(1) |
| C(11) | 7061(3) | 4152(3) | 2295(3) | 10(1) |
| C(14) | 5956(4) | 2851(4) | 723(4) | 21(1) |
| C(7) | 2861(4) | 4132(3) | 2683(4) | 13(1) |
| C(8) | 1632(4) | 4819(4) | 2112(5) | 27(1) |
| C(9) | 3192(4) | 3993(4) | 4042(4) | 25(1) |
Table 3. Bond lengths [Å] and angles [°] for cain84_0m_a (2).

| Bond                  | Distance  |
|-----------------------|-----------|
| Br(1)-C(1)            | 1.905(4)  |
| F(12)-C(12)           | 1.340(5)  |
| F(11)-C(12)           | 1.336(5)  |
| F(8)-C(13)            | 1.351(5)  |
| F(7)-C(13)            | 1.344(5)  |
| F(10)-C(12)           | 1.345(5)  |
| F(9)-C(13)            | 1.328(5)  |
| O(2)-C(11)            | 1.400(5)  |
| O(2)-C(14)            | 1.438(5)  |
| F(5)-C(8)             | 1.320(6)  |
| F(1)-C(9)             | 1.326(6)  |
| O(1)-C(10)            | 1.455(5)  |
| O(1)-C(7)             | 1.386(5)  |
| F(3)-C(9)             | 1.333(5)  |
| F(6)-C(8)             | 1.332(5)  |
| F(4)-C(8)             | 1.351(6)  |
| F(2)-C(9)             | 1.334(5)  |
| C(1)-C(6)             | 1.408(5)  |
| C(1)-C(2)             | 1.414(5)  |
| C(6)-C(5)             | 1.396(5)  |
| C(6)-C(11)            | 1.554(5)  |
| C(4)-H(4)             | 0.9500    |
| C(4)-C(5)             | 1.384(6)  |
| C(4)-C(3)             | 1.388(6)  |
| C(10)-H(10A)          | 0.9800    |
| C(10)-H(10B)          | 0.9800    |
| C(10)-H(10C)          | 0.9800    |
| C(5)-H(5)             | 0.9500    |
| C(2)-C(3)             | 1.395(5)  |
| C(2)-C(7)             | 1.552(5)  |
| C(12)-C(11)           | 1.545(5)  |
| C(3)-H(3)             | 0.9500    |
| C(13)-C(11)           | 1.566(5)  |
| C(14)-H(14A)          | 0.9800    |
C(14)-H(14B) 0.9800
C(14)-H(14C) 0.9800
C(7)-C(8) 1.564(6)
C(7)-C(9) 1.569(6)

C(11)-O(2)-C(14) 117.5(3)
C(7)-O(1)-C(10) 115.9(3)
C(6)-C(1)-Br(1) 119.5(3)
C(6)-C(1)-C(2) 121.1(4)
C(2)-C(1)-Br(1) 119.3(3)
C(1)-C(6)-C(11) 124.9(3)
C(5)-C(6)-C(1) 118.1(3)
C(5)-C(6)-C(11) 116.8(3)
C(5)-C(4)-H(4) 120.1
C(5)-C(4)-C(3) 119.7(4)
C(3)-C(4)-H(4) 120.1
O(1)-C(10)-H(10A) 109.5
O(1)-C(10)-H(10B) 109.5
O(1)-C(10)-H(10C) 109.5
H(10A)-C(10)-H(10B) 109.5
H(10A)-C(10)-H(10C) 109.5
H(10B)-C(10)-H(10C) 109.5
C(6)-C(5)-H(5) 119.3
C(4)-C(5)-C(6) 121.4(4)
C(4)-C(5)-H(5) 119.3
C(1)-C(2)-C(7) 124.4(3)
C(3)-C(2)-C(1) 118.0(4)
C(3)-C(2)-C(7) 117.5(3)
F(12)-C(12)-F(10) 106.6(3)
F(12)-C(12)-C(11) 111.3(3)
F(11)-C(12)-F(12) 107.3(3)
F(11)-C(12)-F(10) 106.8(3)
F(11)-C(12)-C(11) 112.4(3)
F(10)-C(12)-C(11) 112.2(3)
C(4)-C(3)-C(2) 121.3(4)
C(4)-C(3)-H(3) 119.3
| Bond                        | Angle (°)  |
|-----------------------------|------------|
| C(2)-C(3)-H(3)             | 119.3      |
| F(8)-C(13)-C(11)           | 113.4(3)   |
| F(7)-C(13)-F(8)            | 107.1(3)   |
| F(7)-C(13)-C(11)           | 111.4(3)   |
| F(9)-C(13)-F(8)            | 106.7(3)   |
| F(9)-C(13)-F(7)            | 107.0(3)   |
| F(9)-C(13)-C(11)           | 110.8(3)   |
| O(2)-C(11)-C(6)            | 117.2(3)   |
| O(2)-C(11)-C(12)           | 102.7(3)   |
| O(2)-C(11)-C(13)           | 106.5(3)   |
| C(6)-C(11)-C(13)           | 111.8(3)   |
| C(12)-C(11)-C(6)           | 109.7(3)   |
| C(12)-C(11)-C(13)          | 108.3(3)   |
| O(2)-C(14)-H(14A)          | 109.5      |
| O(2)-C(14)-H(14B)          | 109.5      |
| O(2)-C(14)-H(14C)          | 109.5      |
| H(14A)-C(14)-H(14B)        | 109.5      |
| H(14A)-C(14)-H(14C)        | 109.5      |
| H(14B)-C(14)-H(14C)        | 109.5      |
| O(1)-C(7)-C(2)             | 116.9(3)   |
| O(1)-C(7)-C(8)             | 107.5(3)   |
| O(1)-C(7)-C(9)             | 103.7(3)   |
| C(2)-C(7)-C(8)             | 112.2(3)   |
| C(2)-C(7)-C(9)             | 109.5(3)   |
| C(8)-C(7)-C(9)             | 106.3(3)   |
| F(5)-C(8)-F(6)             | 106.8(4)   |
| F(5)-C(8)-F(4)             | 106.7(4)   |
| F(5)-C(8)-C(7)             | 115.0(4)   |
| F(6)-C(8)-F(4)             | 106.5(4)   |
| F(6)-C(8)-C(7)             | 110.2(4)   |
| F(4)-C(8)-C(7)             | 111.2(4)   |
| F(1)-C(9)-F(3)             | 107.8(4)   |
| F(1)-C(9)-F(2)             | 106.6(4)   |
| F(1)-C(9)-C(7)             | 111.5(4)   |
| F(3)-C(9)-F(2)             | 107.5(4)   |
| F(3)-C(9)-C(7)             | 111.6(4)   |
F(2)-C(9)-C(7) 111.5(4)

Symmetry transformations used to generate equivalent atoms:
Table 4. Anisotropic displacement parameters (Å\(^2\times10^3\)) for cain84_0m_a (2). The anisotropic displacement factor exponent takes the form:  
\[-2\pi^2[ h^2 a^*^2 U^{11} + ... + 2h k a^* b^* U^{12} ]\]

| Atom | U\(^{11}\)  | U\(^{22}\)  | U\(^{33}\)  | U\(^{23}\)  | U\(^{13}\)  | U\(^{12}\)  |
|------|-------------|-------------|-------------|-------------|-------------|-------------|
| Br(1) | 16(1)       | 9(1)        | 29(1)       | 5(1)        | 13(1)       | 2(1)        |
| F(12) | 20(1)       | 31(2)       | 15(1)       | 8(1)        | 3(1)        | 0(1)        |
| F(11) | 10(1)       | 39(2)       | 23(1)       | 0(1)        | 5(1)        | 4(1)        |
| F(8)  | 22(1)       | 24(1)       | 40(2)       | 1(1)        | 18(1)       | -10(1)      |
| F(7)  | 24(1)       | 28(1)       | 16(1)       | 8(1)        | 11(1)       | 5(1)        |
| F(10) | 18(1)       | 25(1)       | 22(1)       | -9(1)       | 0(1)        | -4(1)       |
| F(9)  | 22(1)       | 36(2)       | 30(2)       | 5(1)        | 20(1)       | 6(1)        |
| O(2)  | 14(1)       | 13(1)       | 12(1)       | -1(1)       | 3(1)        | 1(1)        |
| F(5)  | 31(2)       | 24(2)       | 66(2)       | -2(1)       | 32(2)       | 8(1)        |
| F(1)  | 44(2)       | 36(2)       | 16(1)       | 9(1)        | 12(1)       | 11(1)       |
| O(1)  | 22(2)       | 21(2)       | 20(2)       | -4(1)       | 9(1)        | -5(1)       |
| F(3)  | 47(2)       | 32(2)       | 28(2)       | -14(1)      | 25(2)       | -14(1)      |
| F(6)  | 18(1)       | 41(2)       | 56(2)       | 1(2)        | 16(1)       | -1(1)       |
| F(4)  | 23(2)       | 39(2)       | 36(2)       | 15(1)       | 5(1)        | 5(1)        |
| F(2)  | 43(2)       | 45(2)       | 38(2)       | -6(2)       | 31(2)       | -15(2)      |
| C(1)  | 9(2)        | 9(2)        | 7(2)        | -1(1)       | 1(2)        | 0(1)        |
| C(6)  | 9(2)        | 9(2)        | 6(2)        | 0(1)        | 1(2)        | 0(1)        |
| C(4)  | 24(2)       | 8(2)        | 15(2)       | 1(2)        | 10(2)       | 1(2)        |
| C(10) | 27(2)       | 20(2)       | 9(2)        | -4(2)       | 5(2)        | -6(2)       |
| C(5)  | 17(2)       | 13(2)       | 11(2)       | 1(2)        | 7(2)        | -3(2)       |
| C(2)  | 10(2)       | 14(2)       | 7(2)        | -1(2)       | 4(2)        | 0(2)        |
| C(12) | 12(2)       | 19(2)       | 18(2)       | -2(2)       | 5(2)        | -1(2)       |
| C(3)  | 18(2)       | 9(2)        | 13(2)       | 0(2)        | 8(2)        | 6(2)        |
| C(13) | 17(2)       | 19(2)       | 20(2)       | 1(2)        | 11(2)       | -1(2)       |
| C(11) | 8(2)        | 12(2)       | 8(2)        | 2(2)        | 3(2)        | 1(1)        |
| C(14) | 18(2)       | 21(2)       | 15(2)       | -7(2)       | 1(2)        | 1(2)        |
| C(7)  | 10(2)       | 12(2)       | 18(2)       | 0(2)        | 8(2)        | 1(2)        |
| C(8)  | 19(2)       | 27(3)       | 35(3)       | 3(2)        | 13(2)       | 1(2)        |
| C(9)  | 25(2)       | 30(2)       | 24(2)       | -3(2)       | 14(2)       | -3(2)       |
Table 5. Hydrogen coordinates ($x \times 10^4$) and isotropic displacement parameters ($Å^2 \times 10^{-3}$) for cain84_0m_a (2).

|    | x     | y     | z     | U(eq) |
|----|-------|-------|-------|-------|
| H(4)| 4934  | 7126  | 2456  | 18    |
| H(10A)| 1349  | 3296  | 482   | 30    |
| H(10B)| 2172  | 2225  | 752   | 30    |
| H(10C)| 2793  | 3364  | 755   | 30    |
| H(5)| 6548  | 6196  | 2307  | 16    |
| H(3)| 3288  | 6188  | 2544  | 16    |
| H(14A)| 6270  | 3022  | 142   | 31    |
| H(14B)| 5208  | 3284  | 552   | 31    |
| H(14C)| 5737  | 2088  | 664   | 31    |
### X-RAY OF 3

Table 1. Crystal data and structure refinement for Cain58 (3).

| Property                              | Value                  |
|---------------------------------------|------------------------|
| Identification code                   | JH167                  |
| Empirical formula                     | C78 H87 O6 P3          |
| Formula weight                        | 1213.38                |
| Temperature                           | 100.0 K                |
| Wavelength                            | 0.71073 Å              |
| Crystal system                        | Triclinic              |
| Space group                           | P-1                    |
| Unit cell dimensions                  |                        |
| a = 10.515(5) Å                      | α= 100.80(3)°          |
| b = 15.037(8) Å                      | β= 94.78(2)°           |
| c = 22.719(11) Å                     | γ = 103.351(19)°       |
| Volume                                | 3403(3) Å³             |
| Z                                      | 2                      |
| Density (calculated)                  | 1.184 Mg/m³            |
| Absorption coefficient                | 0.140 mm⁻¹             |
| F(000)                                | 1296                   |
| Crystal size                          | 0.28 x 0.08 x 0.04 mm³ |
| Theta range for data collection       | 1.425 to 23.409°       |
| Index ranges                          | -11<=h<=11, -16<=k<=16, -25<=l<=25 |
| Reflections collected                 | 45744                  |
| Independent reflections               | 9821 [R(int) = 0.0824] |
| Completeness to theta = 23.409°      | 99.0 %                 |
| Absorption correction                 | Semi-empirical from equivalents |
| Max. and min. transmission            | 0.2571 and 0.2038      |
| Refinement method                     | Full-matrix least-squares on F² |
| Data / restraints / parameters        | 9821 / 0 / 803         |
| Goodness-of-fit on F²                 | 1.096                  |
| Final R indices [I>2sigma(I)]         | R1 = 0.0788, wR2 = 0.1660 |
| R indices (all data)                  | R1 = 0.1154, wR2 = 0.1799 |
| Extinction coefficient                | n/a                    |
| Largest diff. peak and hole           | 0.408 and -0.375 e.Å⁻³ |
Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for Cain58 (3). U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

|          | x       | y       | z       | U(eq)  |
|----------|---------|---------|---------|--------|
| P(2)     | 2753(1) | 7331(1) | 7046(1) | 23(1)  |
| P(3)     | 1107(1) | 6059(1) | 6916(1) | 23(1)  |
| P(1)     | 2046(1) | 6854(1) | 7843(1) | 23(1)  |
| O(6)     | -511(3) | 3706(2) | 7037(2) | 24(1)  |
| O(2)     | 5561(3) | 7449(2) | 7824(2) | 25(1)  |
| O(1)     | 898(3)  | 5570(2) | 9336(2) | 27(1)  |
| O(4)     | 1390(3) | 7449(3) | 5688(2) | 33(1)  |
| O(3)     | 4426(3) | 10065(2)| 8127(2) | 34(1)  |
| O(5)     | 3542(3) | 5448(3) | 5367(2) | 32(1)  |
| C(6)     | 2497(5) | 5803(4) | 8649(2) | 24(1)  |
| C(75)    | -2555(5)| 3940(4) | 7362(2) | 24(1)  |
| C(58)    | 1750(5) | 4409(4) | 7056(2) | 23(1)  |
| C(57)    | 2456(5) | 3728(4) | 6975(2) | 25(1)  |
| C(17)    | 5027(5) | 6476(4) | 7585(2) | 24(1)  |
| C(3)     | 4848(5) | 5459(4) | 8333(2) | 26(1)  |
| C(28)    | 942(5) | 8331(4) | 6632(2) | 29(1)  |
| C(1)     | 3045(4) | 6163(3) | 8166(2) | 19(1)  |
| C(2)     | 4265(5) | 6012(3) | 8025(2) | 22(1)  |
| C(53)    | 2001(4) | 5136(3) | 6746(2) | 20(1)  |
| C(18)    | 6811(4) | 7648(3) | 8159(2) | 21(1)  |
| C(27)    | 1925(4) | 8299(4) | 7089(2) | 22(1)  |
| C(7)     | 1280(5) | 6038(4) | 8869(2) | 27(1)  |
| C(60)    | 3811(5) | 6111(4) | 5010(2) | 26(1)  |
| C(23)    | 7901(5) | 7646(3) | 7849(2) | 22(1)  |
| C(55)    | 3564(5) | 4428(4) | 6238(2) | 28(1)  |
| C(59)    | 3093(5) | 5827(4) | 5915(2) | 30(1)  |
| C(39)    | 6619(5) | 9907(4) | 8017(2) | 28(1)  |
| C(20)    | 8191(5) | 8114(3) | 9097(2) | 26(1)  |
| C(19)    | 6942(5) | 7892(3) | 8783(2) | 26(1)  |
| C(70)    | -1604(5)| 3427(4) | 7332(2) | 25(1)  |
| C(65)    | 5060(5) | 6697(4) | 5086(2) | 29(1)  |
|   |   |   |   |   |
|---|---|---|---|---|
| C(22) | 9142(5) | 7891(4) | 8187(3) | 30(1) |
| C(32) | 2440(5) | 9083(4) | 7557(2) | 28(1) |
| C(54) | 2880(5) | 5118(4) | 6306(2) | 25(1) |
| C(35) | 6034(5) | 10509(4) | 8999(2) | 32(1) |
| C(74) | -3683(5) | 3586(4) | 7614(2) | 29(1) |
| C(72) | -2897(5) | 2291(4) | 7796(2) | 32(1) |
| C(76) | -2388(5) | 4816(4) | 7134(2) | 30(1) |
| C(44) | 1583(5) | 8097(4) | 5317(2) | 31(1) |
| C(71) | -1767(5) | 2599(4) | 7538(2) | 29(1) |
| C(34) | 5710(5) | 10146(3) | 8389(2) | 27(1) |
| C(38) | 7912(5) | 10052(3) | 8288(2) | 31(1) |
| C(69) | 643(5) | 4260(4) | 7437(2) | 25(1) |
| C(33) | 3593(5) | 9139(4) | 8021(2) | 29(1) |
| C(8) | 154(5) | 6008(4) | 9724(2) | 29(1) |
| C(21) | 9309(5) | 8120(4) | 8811(2) | 30(1) |
| C(5) | 3102(5) | 5243(4) | 8938(2) | 31(1) |
| C(73) | -3870(5) | 2778(4) | 7835(2) | 28(1) |
| C(56) | 3381(5) | 3748(4) | 6576(2) | 31(1) |
| C(61) | 2816(5) | 6148(4) | 4585(2) | 34(1) |
| C(9) | -1204(5) | 5853(4) | 9544(3) | 34(1) |
| C(49) | 2738(5) | 8801(4) | 5427(2) | 32(1) |
| C(24) | 5755(5) | 7929(4) | 9116(2) | 34(1) |
| C(64) | 5312(5) | 7364(4) | 4730(2) | 33(1) |
| C(4) | 4280(5) | 5081(4) | 8778(3) | 33(1) |
| C(31) | 1936(5) | 9864(4) | 7601(3) | 38(2) |
| C(37) | 8271(5) | 10414(4) | 8905(3) | 34(1) |
| C(45) | 634(5) | 7981(5) | 4822(3) | 41(2) |
| C(26) | 7757(5) | 7437(4) | 7167(2) | 34(1) |
| C(63) | 4327(6) | 7431(4) | 4305(2) | 35(1) |
| C(13) | 7886(6) | 6547(4) | 10271(2) | 33(1) |
| C(62) | 3089(6) | 6820(4) | 4239(2) | 38(2) |
| C(36) | 7336(6) | 10647(4) | 9252(3) | 35(1) |
| C(43) | 410(5) | 7563(4) | 6086(2) | 34(1) |
| C(29) | 479(5) | 9137(4) | 6693(3) | 43(2) |
| C(48) | 2907(5) | 9418(5) | 5046(3) | 42(2) |
| C(50) | 3800(5) | 8880(4) | 5938(2) | 36(1) |
|   |     |     |     |   |
|---|-----|-----|-----|---|
| C(10) | -1902(6) | 6281(5) | 9961(3) | 48(2) |
| C(40) | 6242(6) | 9554(4) | 7349(2) | 39(2) |
| C(25) | 10672(5) | 8394(4) | 9172(3) | 38(2) |
| C(66) | 6150(5) | 6626(5) | 5536(3) | 42(2) |
| C(78) | -760(5) | 2026(4) | 7466(3) | 39(2) |
| C(16) | 2250(6) | 6728(4) | 10431(3) | 43(2) |
| C(77) | -5102(5) | 2428(4) | 8110(3) | 43(2) |
| C(11) | -1315(7) | 6814(4) | 10516(3) | 44(2) |
| C(68) | 1465(6) | 5471(5) | 4502(3) | 51(2) |
| C(41) | 9665(6) | 10547(4) | 9186(3) | 47(2) |
| C(67) | 4589(7) | 8155(4) | 3920(3) | 48(2) |
| C(30) | 944(6) | 9881(5) | 7166(3) | 47(2) |
| C(12) | 39(7) | 6950(4) | 10670(3) | 47(2) |
| C(47) | 1982(6) | 9362(5) | 4566(3) | 55(2) |
| C(14) | -1846(5) | 5290(5) | 8933(3) | 49(2) |
| C(42) | 5015(6) | 10786(5) | 9380(3) | 52(2) |
| C(46) | 867(6) | 8643(5) | 4462(3) | 54(2) |
| C(52) | -590(6) | 7177(5) | 4662(3) | 54(2) |
| C(15) | -2133(8) | 7242(5) | 10962(4) | 74(2) |
| C(51) | 2196(7) | 10080(7) | 4175(4) | 107(4) |
Table 3. Bond lengths [Å] and angles [°] for Cain58 (3).

| Bond                  | Length/Distance |
|-----------------------|-----------------|
| P(2)-P(3)             | 2.217(2)        |
| P(2)-P(1)             | 2.194(2)        |
| P(2)-C(27)            | 1.853(5)        |
| P(3)-P(1)             | 2.237(2)        |
| P(3)-C(53)            | 1.850(5)        |
| P(1)-C(1)             | 1.839(5)        |
| O(6)-C(70)            | 1.399(6)        |
| O(6)-C(69)            | 1.434(6)        |
| O(2)-C(17)            | 1.422(6)        |
| O(2)-C(18)            | 1.400(6)        |
| O(1)-C(7)             | 1.415(6)        |
| O(1)-C(8)             | 1.400(6)        |
| O(4)-C(44)            | 1.395(6)        |
| O(4)-C(43)            | 1.445(6)        |
| O(3)-C(34)            | 1.398(6)        |
| O(3)-C(33)            | 1.428(6)        |
| O(5)-C(60)            | 1.393(6)        |
| O(5)-C(59)            | 1.432(6)        |
| C(6)-C(1)             | 1.420(7)        |
| C(6)-C(7)             | 1.504(7)        |
| C(6)-C(5)             | 1.388(7)        |
| C(75)-C(70)           | 1.395(7)        |
| C(75)-C(74)           | 1.398(7)        |
| C(75)-C(76)           | 1.483(7)        |
| C(58)-C(57)           | 1.392(7)        |
| C(58)-C(53)           | 1.396(7)        |
| C(58)-C(69)           | 1.508(7)        |
| C(57)-H(57)           | 0.9500          |
| C(57)-C(56)           | 1.384(7)        |
| C(17)-H(17A)          | 0.9900          |
| C(17)-H(17B)          | 0.9900          |
| C(17)-C(2)            | 1.505(7)        |
| C(3)-H(3)             | 0.9500          |
| C(3)-C(2)             | 1.391(7)        |
C(3)-C(4) 1.365(7)
C(28)-C(27) 1.418(7)
C(28)-C(43) 1.494(8)
C(28)-C(29) 1.395(8)
C(1)-C(2) 1.406(7)
C(53)-C(54) 1.417(7)
C(18)-C(23) 1.395(7)
C(18)-C(19) 1.385(7)
C(27)-C(32) 1.398(7)
C(7)-H(7A) 0.9900
C(7)-H(7B) 0.9900
C(60)-C(65) 1.379(7)
C(60)-C(61) 1.382(7)
C(23)-C(22) 1.391(7)
C(23)-C(26) 1.509(7)
C(55)-H(55) 0.9500
C(55)-C(54) 1.386(7)
C(55)-C(56) 1.377(7)
C(59)-H(59A) 0.9900
C(59)-H(59B) 0.9900
C(59)-C(54) 1.502(7)
C(39)-C(34) 1.391(7)
C(39)-C(38) 1.397(7)
C(39)-C(40) 1.496(7)
C(20)-H(20) 0.9500
C(20)-C(19) 1.378(7)
C(20)-C(21) 1.389(7)
C(19)-C(24) 1.518(7)
C(70)-C(71) 1.390(7)
C(65)-C(64) 1.397(7)
C(65)-C(66) 1.507(7)
C(22)-H(22) 0.9500
C(22)-C(21) 1.380(7)
C(32)-C(33) 1.515(7)
C(32)-C(31) 1.387(7)
C(35)-C(34) 1.374(7)
C(35)-C(36)  1.394(7)
C(35)-C(42)  1.515(8)
C(74)-H(74)  0.9500
C(74)-C(73)  1.379(7)
C(72)-H(72)  0.9500
C(72)-C(71)  1.390(7)
C(72)-C(73)  1.389(7)
C(76)-H(76A)  0.9800
C(76)-H(76B)  0.9800
C(76)-H(76C)  0.9800
C(44)-C(49)  1.384(7)
C(44)-C(45)  1.397(7)
C(71)-C(78)  1.513(7)
C(38)-H(38)  0.9500
C(38)-C(37)  1.389(8)
C(69)-H(69A)  0.9900
C(69)-H(69B)  0.9900
C(33)-H(33A)  0.9900
C(33)-H(33B)  0.9900
C(8)-C(9)  1.404(7)
C(8)-C(13)  1.372(8)
C(21)-C(25)  1.517(7)
C(5)-H(5)  0.9500
C(5)-C(4)  1.380(7)
C(73)-C(77)  1.513(7)
C(56)-H(56)  0.9500
C(61)-C(62)  1.388(8)
C(61)-C(68)  1.519(8)
C(9)-C(10)  1.399(8)
C(9)-C(14)  1.496(8)
C(49)-C(48)  1.375(8)
C(49)-C(50)  1.509(7)
C(24)-H(24A)  0.9800
C(24)-H(24B)  0.9800
C(24)-H(24C)  0.9800
C(64)-H(64)  0.9500
C(64)-C(63)  1.390(8)  
C(4)-H(4)  0.9500  
C(31)-H(31)  0.9500  
C(31)-C(30)  1.384(8)  
C(37)-C(36)  1.376(8)  
C(37)-C(41)  1.503(8)  
C(45)-C(46)  1.395(8)  
C(45)-C(52)  1.512(8)  
C(26)-H(26A)  0.9800  
C(26)-H(26B)  0.9800  
C(26)-H(26C)  0.9800  
C(63)-C(62)  1.387(8)  
C(63)-C(67)  1.511(8)  
C(13)-C(16)  1.499(8)  
C(13)-C(12)  1.394(8)  
C(62)-H(62)  0.9500  
C(36)-H(36)  0.9500  
C(43)-H(43A)  0.9900  
C(43)-H(43B)  0.9900  
C(29)-H(29)  0.9500  
C(29)-C(30)  1.361(9)  
C(48)-H(48)  0.9500  
C(48)-C(47)  1.376(8)  
C(50)-H(50A)  0.9800  
C(50)-H(50B)  0.9800  
C(50)-H(50C)  0.9800  
C(10)-H(10)  0.9500  
C(10)-C(11)  1.371(9)  
C(40)-H(40A)  0.9800  
C(40)-H(40B)  0.9800  
C(40)-H(40C)  0.9800  
C(25)-H(25A)  0.9800  
C(25)-H(25B)  0.9800  
C(25)-H(25C)  0.9800  
C(66)-H(66A)  0.9800  
C(66)-H(66B)  0.9800
| Bond                  | Distance |
|-----------------------|----------|
| C(66)-H(66C)          | 0.9800   |
| C(78)-H(78A)          | 0.9800   |
| C(78)-H(78B)          | 0.9800   |
| C(78)-H(78C)          | 0.9800   |
| C(16)-H(16A)          | 0.9800   |
| C(16)-H(16B)          | 0.9800   |
| C(16)-H(16C)          | 0.9800   |
| C(77)-H(77A)          | 0.9800   |
| C(77)-H(77B)          | 0.9800   |
| C(77)-H(77C)          | 0.9800   |
| C(11)-C(12)           | 1.396(9) |
| C(11)-C(15)           | 1.527(8) |
| C(68)-H(68A)          | 0.9800   |
| C(68)-H(68B)          | 0.9800   |
| C(68)-H(68C)          | 0.9800   |
| C(41)-H(41A)          | 0.9800   |
| C(41)-H(41B)          | 0.9800   |
| C(41)-H(41C)          | 0.9800   |
| C(67)-H(67A)          | 0.9800   |
| C(67)-H(67B)          | 0.9800   |
| C(67)-H(67C)          | 0.9800   |
| C(30)-H(30)           | 0.9500   |
| C(12)-H(12)           | 0.9500   |
| C(47)-C(46)           | 1.368(9) |
| C(47)-C(51)           | 1.513(9) |
| C(14)-H(14A)          | 0.9800   |
| C(14)-H(14B)          | 0.9800   |
| C(14)-H(14C)          | 0.9800   |
| C(42)-H(42A)          | 0.9800   |
| C(42)-H(42B)          | 0.9800   |
| C(42)-H(42C)          | 0.9800   |
| C(46)-H(46)           | 0.9500   |
| C(52)-H(52A)          | 0.9800   |
| C(52)-H(52B)          | 0.9800   |
| C(52)-H(52C)          | 0.9800   |
| C(15)-H(15A)          | 0.9800   |
C(15)-H(15B)  0.9800
C(15)-H(15C)  0.9800
C(51)-H(51A)  0.9800
C(51)-H(51B)  0.9800
C(51)-H(51C)  0.9800
P(1)-P(2)-P(3)  60.95(7)
P(2)-P(3)-P(1)  59.02(7)
C(27)-P(2)-P(1)  96.46(16)
P(2)-P(3)-P(1)  104.08(16)
P(2)-P(1)-P(3)  60.03(7)
C(1)-P(1)-P(2)  113.78(16)
P(1)-P(2)-P(3)  108.26(17)
C(70)-O(6)-C(69)  113.9(4)
C(18)-O(2)-C(17)  112.8(4)
C(8)-O(1)-C(7)  113.9(4)
C(44)-O(4)-C(43)  113.6(4)
C(34)-O(3)-C(33)  113.8(4)
C(60)-O(5)-C(59)  110.9(4)
C(1)-C(6)-C(7)  121.0(4)
C(5)-C(6)-C(1)  120.1(5)
C(5)-C(6)-C(7)  118.9(5)
C(70)-C(75)-C(74)  116.9(5)
C(70)-C(75)-C(76)  121.9(4)
C(74)-C(75)-C(76)  121.2(5)
C(57)-C(58)-C(53)  120.0(5)
C(57)-C(58)-C(69)  116.7(5)
C(53)-C(58)-C(69)  123.1(5)
C(58)-C(57)-H(57)  119.7
C(56)-C(57)-C(58)  120.6(5)
C(56)-C(57)-H(57)  119.7
O(2)-C(17)-H(17A)  109.4
O(2)-C(17)-H(17B)  109.4
O(2)-C(17)-C(2)  111.3(4)
| Bond                                      | Angle  |
|-------------------------------------------|--------|
| H(17A)-C(17)-H(17B)                      | 108.0  |
| C(2)-C(17)-H(17A)                        | 109.4  |
| C(2)-C(17)-H(17B)                        | 109.4  |
| C(2)-C(3)-H(3)                            | 119.2  |
| C(4)-C(3)-H(3)                            | 119.2  |
| C(4)-C(3)-C(2)                            | 121.6(5)|
| C(27)-C(28)-C(43)                         | 123.6(5)|
| C(29)-C(28)-C(27)                         | 117.6(5)|
| C(29)-C(28)-C(43)                         | 118.8(5)|
| C(6)-C(1)-P(1)                            | 112.1(3)|
| C(2)-C(1)-P(1)                            | 129.1(4)|
| C(2)-C(1)-C(6)                            | 118.6(4)|
| C(3)-C(2)-C(17)                           | 117.8(4)|
| C(3)-C(2)-C(1)                            | 119.1(5)|
| C(1)-C(2)-C(17)                           | 123.0(4)|
| C(58)-C(53)-P(3)                          | 117.4(4)|
| C(58)-C(53)-C(54)                         | 119.1(5)|
| C(54)-C(53)-P(3)                          | 123.4(4)|
| C(23)-C(18)-O(2)                          | 118.7(4)|
| C(19)-C(18)-O(2)                          | 119.4(4)|
| C(19)-C(18)-C(23)                         | 121.8(4)|
| C(28)-C(27)-P(2)                          | 123.2(4)|
| C(32)-C(27)-P(2)                          | 116.8(4)|
| C(32)-C(27)-C(28)                         | 119.4(5)|
| O(1)-C(7)-C(6)                            | 110.4(4)|
| O(1)-C(7)-H(7A)                           | 109.6  |
| O(1)-C(7)-H(7B)                           | 109.6  |
| C(6)-C(7)-H(7A)                           | 109.6  |
| C(6)-C(7)-H(7B)                           | 109.6  |
| H(7A)-C(7)-H(7B)                          | 108.1  |
| C(65)-C(60)-O(5)                          | 119.2(5)|
| C(65)-C(60)-C(61)                         | 122.3(5)|
| C(61)-C(60)-O(5)                          | 118.5(5)|
| C(18)-C(23)-C(26)                         | 121.4(4)|
| C(22)-C(23)-C(18)                         | 117.9(5)|
| C(22)-C(23)-C(26)                         | 120.6(4)|
C(54)-C(55)-H(55) 119.2
C(56)-C(55)-H(55) 119.2
C(56)-C(55)-C(54) 121.6(5)
O(5)-C(59)-H(59A) 109.7
O(5)-C(59)-H(59B) 109.7
O(5)-C(59)-C(54) 109.8(4)
H(59A)-C(59)-H(59B) 108.2
C(54)-C(59)-H(59A) 109.7
C(54)-C(59)-H(59B) 109.7
C(34)-C(39)-C(38) 117.2(5)
C(34)-C(39)-C(40) 121.4(5)
C(38)-C(39)-C(40) 121.4(5)
C(19)-C(20)-H(20) 118.7
C(19)-C(20)-C(21) 122.6(5)
C(21)-C(20)-H(20) 118.7
C(18)-C(19)-C(24) 121.5(5)
C(20)-C(19)-C(18) 117.9(5)
C(20)-C(19)-C(24) 120.6(5)
C(75)-C(70)-O(6) 119.1(4)
C(71)-C(70)-O(6) 118.7(4)
C(71)-C(70)-C(75) 121.9(5)
C(60)-C(65)-C(64) 118.5(5)
C(60)-C(65)-C(66) 121.7(5)
C(64)-C(65)-C(66) 119.7(5)
C(23)-C(22)-H(22) 119.0
C(21)-C(22)-C(23) 122.0(5)
C(21)-C(22)-H(22) 119.0
C(27)-C(32)-C(33) 122.3(5)
C(31)-C(32)-C(27) 120.6(5)
C(31)-C(32)-C(33) 117.0(5)
C(53)-C(54)-C(59) 121.8(5)
C(55)-C(54)-C(53) 119.0(5)
C(55)-C(54)-C(59) 119.3(4)
C(34)-C(35)-C(36) 118.0(5)
C(34)-C(35)-C(42) 120.8(5)
C(36)-C(35)-C(42) 121.1(5)
C(75)-C(74)-H(74)  118.5
C(73)-C(74)-C(75)  123.0(5)
C(73)-C(74)-H(74)  118.5
C(71)-C(72)-H(72)  119.3
C(73)-C(72)-H(72)  119.3
C(73)-C(72)-C(71)  121.4(5)
C(75)-C(76)-H(76A)  109.5
C(75)-C(76)-H(76B)  109.5
C(75)-C(76)-H(76C)  109.5
H(76A)-C(76)-H(76B)  109.5
H(76A)-C(76)-H(76C)  109.5
O(4)-C(44)-C(45)  118.7(5)
C(49)-C(44)-O(4)  119.3(4)
C(49)-C(44)-C(45)  121.9(5)
C(70)-C(71)-C(78)  120.9(5)
C(72)-C(71)-C(70)  118.7(5)
C(72)-C(71)-C(78)  120.4(5)
C(39)-C(34)-O(3)  118.8(5)
C(35)-C(34)-O(3)  118.1(5)
C(35)-C(34)-C(39)  123.0(5)
C(39)-C(38)-H(38)  119.4
C(37)-C(38)-C(39)  121.3(5)
C(37)-C(38)-H(38)  119.4
O(6)-C(69)-C(58)  106.8(4)
O(6)-C(69)-H(69A)  110.4
O(6)-C(69)-H(69B)  110.4
C(58)-C(69)-H(69A)  110.4
C(58)-C(69)-H(69B)  110.4
H(69A)-C(69)-H(69B)  108.6
O(3)-C(33)-C(32)  107.3(4)
O(3)-C(33)-H(33A)  110.2
O(3)-C(33)-H(33B)  110.2
C(32)-C(33)-H(33A)  110.2
C(32)-C(33)-H(33B)  110.2
H(33A)-C(33)-H(33B)  108.5
O(1)-C(8)-C(9)  118.7(5)
C(13)-C(8)-O(1)  117.9(5)
C(13)-C(8)-C(9)  123.4(5)
C(20)-C(21)-C(25)  121.0(5)
C(22)-C(21)-C(20)  117.8(5)
C(22)-C(21)-C(25)  121.1(5)
C(6)-C(5)-H(5)  120.0
C(4)-C(5)-C(6)  120.0(5)
C(4)-C(5)-H(5)  120.0
C(74)-C(73)-C(72)  118.2(5)
C(74)-C(73)-C(77)  121.1(5)
C(72)-C(73)-C(77)  120.8(5)
C(57)-C(56)-H(56)  120.3
C(55)-C(56)-C(57)  119.5(5)
C(55)-C(56)-H(56)  120.3
C(60)-C(61)-C(62)  118.1(5)
C(60)-C(61)-C(68)  120.8(5)
C(62)-C(61)-C(68)  121.2(5)
C(8)-C(9)-C(14)  121.5(5)
C(10)-C(9)-C(8)  116.0(6)
C(10)-C(9)-C(14)  122.4(5)
C(44)-C(49)-C(50)  121.5(5)
C(48)-C(49)-C(44)  117.9(5)
C(48)-C(49)-C(50)  120.7(5)
C(19)-C(24)-H(24A)  109.5
C(19)-C(24)-H(24B)  109.5
C(19)-C(24)-H(24C)  109.5
H(24A)-C(24)-H(24B)  109.5
H(24A)-C(24)-H(24C)  109.5
H(24B)-C(24)-H(24C)  109.5
C(65)-C(64)-H(64)  119.7
C(63)-C(64)-C(65)  120.7(5)
C(63)-C(64)-H(64)  119.7
C(3)-C(4)-C(5)  120.4(5)
C(3)-C(4)-H(4)  119.8
C(5)-C(4)-H(4)  119.8
C(32)-C(31)-H(31) 120.1
C(30)-C(31)-C(32) 119.7(6)
C(30)-C(31)-H(31) 120.1
C(38)-C(37)-C(41) 120.1(5)
C(36)-C(37)-C(38) 119.2(5)
C(36)-C(37)-C(41) 120.6(5)
C(44)-C(45)-C(52) 123.2(6)
C(46)-C(45)-C(44) 116.8(6)
C(46)-C(45)-C(52) 120.0(5)
C(23)-C(26)-H(26A) 109.5
C(23)-C(26)-H(26B) 109.5
C(23)-C(26)-H(26C) 109.5
H(26A)-C(26)-H(26B) 109.5
H(26A)-C(26)-H(26C) 109.5
H(26B)-C(26)-H(26C) 109.5
C(64)-C(63)-C(67) 121.0(5)
C(62)-C(63)-C(64) 118.8(5)
C(62)-C(63)-C(67) 120.1(5)
C(8)-C(13)-C(16) 121.4(5)
C(8)-C(13)-C(12) 118.1(6)
C(12)-C(13)-C(16) 120.5(5)
C(61)-C(62)-H(62) 119.2
C(63)-C(62)-C(61) 121.6(5)
C(63)-C(62)-H(62) 119.2
C(35)-C(36)-H(36) 119.4
C(37)-C(36)-C(35) 121.3(5)
C(37)-C(36)-H(36) 119.4
O(4)-C(43)-C(28) 112.5(4)
O(4)-C(43)-H(43A) 109.1
O(4)-C(43)-H(43B) 109.1
C(28)-C(43)-H(43A) 109.1
C(28)-C(43)-H(43B) 109.1
H(43A)-C(43)-H(43B) 107.8
C(28)-C(29)-H(29) 118.7
C(30)-C(29)-C(28) 122.5(6)
C(30)-C(29)-H(29) 118.7
C(49)-C(48)-H(48) 118.6
C(49)-C(48)-C(47) 122.8(6)
C(47)-C(48)-H(48) 118.6
C(49)-C(50)-H(50A) 109.5
C(49)-C(50)-H(50B) 109.5
C(49)-C(50)-H(50C) 109.5
H(50A)-C(50)-H(50B) 109.5
H(50A)-C(50)-H(50C) 109.5
H(50B)-C(50)-H(50C) 109.5
C(9)-C(10)-H(10) 118.6
C(11)-C(10)-C(9) 122.7(6)
C(11)-C(10)-H(10) 118.6
C(39)-C(40)-H(40A) 109.5
C(39)-C(40)-H(40B) 109.5
C(39)-C(40)-H(40C) 109.5
H(40A)-C(40)-H(40B) 109.5
H(40A)-C(40)-H(40C) 109.5
H(40B)-C(40)-H(40C) 109.5
C(21)-C(25)-H(25A) 109.5
C(21)-C(25)-H(25B) 109.5
C(21)-C(25)-H(25C) 109.5
H(25A)-C(25)-H(25B) 109.5
H(25A)-C(25)-H(25C) 109.5
H(25B)-C(25)-H(25C) 109.5
C(65)-C(66)-H(66A) 109.5
C(65)-C(66)-H(66B) 109.5
C(65)-C(66)-H(66C) 109.5
H(66A)-C(66)-H(66B) 109.5
H(66A)-C(66)-H(66C) 109.5
H(66B)-C(66)-H(66C) 109.5
C(71)-C(78)-H(78A) 109.5
C(71)-C(78)-H(78B) 109.5
C(71)-C(78)-H(78C) 109.5
H(78A)-C(78)-H(78B) 109.5
H(78A)-C(78)-H(78C) 109.5
H(78B)-C(78)-H(78C) 109.5
| Bond                  | Angle  |
|-----------------------|--------|
| C(13)-C(16)-H(16A)   | 109.5  |
| C(13)-C(16)-H(16B)   | 109.5  |
| C(13)-C(16)-H(16C)   | 109.5  |
| H(16A)-C(16)-H(16B)  | 109.5  |
| H(16A)-C(16)-H(16C)  | 109.5  |
| H(16B)-C(16)-H(16C)  | 109.5  |
| C(73)-C(77)-H(77A)   | 109.5  |
| C(73)-C(77)-H(77B)   | 109.5  |
| C(73)-C(77)-H(77C)   | 109.5  |
| H(77A)-C(77)-H(77B)  | 109.5  |
| H(77A)-C(77)-H(77C)  | 109.5  |
| H(77B)-C(77)-H(77C)  | 109.5  |
| C(10)-C(11)-C(12)    | 118.9(6) |
| C(10)-C(11)-C(15)    | 120.5(7) |
| C(12)-C(11)-C(15)    | 120.6(7) |
| C(61)-C(68)-H(68A)   | 109.5  |
| C(61)-C(68)-H(68B)   | 109.5  |
| C(61)-C(68)-H(68C)   | 109.5  |
| H(68A)-C(68)-H(68B)  | 109.5  |
| H(68A)-C(68)-H(68C)  | 109.5  |
| H(68B)-C(68)-H(68C)  | 109.5  |
| C(37)-C(41)-H(41A)   | 109.5  |
| C(37)-C(41)-H(41B)   | 109.5  |
| C(37)-C(41)-H(41C)   | 109.5  |
| H(41A)-C(41)-H(41B)  | 109.5  |
| H(41A)-C(41)-H(41C)  | 109.5  |
| H(41B)-C(41)-H(41C)  | 109.5  |
| C(63)-C(67)-H(67A)   | 109.5  |
| C(63)-C(67)-H(67B)   | 109.5  |
| C(63)-C(67)-H(67C)   | 109.5  |
| H(67A)-C(67)-H(67B)  | 109.5  |
| H(67A)-C(67)-H(67C)  | 109.5  |
| H(67B)-C(67)-H(67C)  | 109.5  |
| C(31)-C(30)-H(30)    | 120.0  |
| C(29)-C(30)-C(31)    | 120.0(6) |
| C(29)-C(30)-H(30)    | 120.0  |
| Bond                  | Angle (°) |
|-----------------------|-----------|
| C(13)-C(12)-C(11)    | 121.0(6)  |
| C(13)-C(12)-H(12)    | 119.5     |
| C(11)-C(12)-H(12)    | 119.5     |
| C(48)-C(47)-C(51)    | 120.7(6)  |
| C(46)-C(47)-C(48)    | 117.7(6)  |
| C(46)-C(47)-C(51)    | 121.6(6)  |
| C(9)-C(14)-H(14A)    | 109.5     |
| C(9)-C(14)-H(14B)    | 109.5     |
| C(9)-C(14)-H(14C)    | 109.5     |
| H(14A)-C(14)-H(14B)  | 109.5     |
| H(14A)-C(14)-H(14C)  | 109.5     |
| H(14B)-C(14)-H(14C)  | 109.5     |
| C(35)-C(42)-H(42A)   | 109.5     |
| C(35)-C(42)-H(42B)   | 109.5     |
| C(35)-C(42)-H(42C)   | 109.5     |
| H(42A)-C(42)-H(42B)  | 109.5     |
| H(42A)-C(42)-H(42C)  | 109.5     |
| H(42B)-C(42)-H(42C)  | 109.5     |
| C(45)-C(46)-H(46)    | 118.6     |
| C(47)-C(46)-C(45)    | 122.8(5)  |
| C(47)-C(46)-H(46)    | 118.6     |
| C(45)-C(52)-H(52A)   | 109.5     |
| C(45)-C(52)-H(52B)   | 109.5     |
| C(45)-C(52)-H(52C)   | 109.5     |
| H(52A)-C(52)-H(52B)  | 109.5     |
| H(52A)-C(52)-H(52C)  | 109.5     |
| H(52B)-C(52)-H(52C)  | 109.5     |
| C(11)-C(15)-H(15A)   | 109.5     |
| C(11)-C(15)-H(15B)   | 109.5     |
| C(11)-C(15)-H(15C)   | 109.5     |
| H(15A)-C(15)-H(15B)  | 109.5     |
| H(15A)-C(15)-H(15C)  | 109.5     |
| H(15B)-C(15)-H(15C)  | 109.5     |
| C(47)-C(51)-H(51A)   | 109.5     |
| C(47)-C(51)-H(51B)   | 109.5     |
| C(47)-C(51)-H(51C)   | 109.5     |
H(51A)-C(51)-H(51B)  109.5
H(51A)-C(51)-H(51C)  109.5
H(51B)-C(51)-H(51C)  109.5

Symmetry transformations used to generate equivalent atoms:
Table 4. Anisotropic displacement parameters (Å² x 10³) for Cain58 (3). The anisotropic displacement factor exponent takes the form: 

\(-2\pi²[h²a*²U₁₁ + ... + 2hk a*b* U₁₂]

|       | U₁₁  | U₂₂  | U₃₃  | U₁₂  | U₁₃  | U₁₂  |
|-------|------|------|------|------|------|------|
| P(2)  | 12(1)| 32(1)| 24(1)| 7(1) | 4(1) | 6(1) |
| P(3)  | 12(1)| 32(1)| 25(1)| 7(1) | 3(1) | 4(1) |
| P(1)  | 14(1)| 32(1)| 24(1)| 7(1) | 5(1) | 8(1) |
| O(6)  | 15(2)| 33(2)| 24(2)| 6(2) | 7(2) | 4(2) |
| O(2)  | 10(2)| 33(2)| 28(2)| 4(2) | 1(2) | 4(2) |
| O(1)  | 24(2)| 35(2)| 29(2)| 16(2)| 11(2)| 12(2)|
| O(4)  | 21(2)| 40(2)| 39(2)| 14(2)| 1(2) | 6(2) |
| O(3)  | 22(2)| 23(2)| 51(2)| 8(2) | 4(2) | 0(2) |
| O(5)  | 32(2)| 39(2)| 27(2)| 6(2) | 12(2)| 10(2)|
| C(6)  | 16(3)| 28(3)| 27(3)| 6(2) | 1(2) | 5(2) |
| C(75) | 20(3)| 25(3)| 26(3)| 5(2) | 2(2) | 7(2) |
| C(58) | 14(3)| 31(3)| 21(3)| 4(2) | 4(2) | 2(2) |
| C(57) | 20(3)| 27(3)| 27(3)| 6(2) | 0(2) | 3(2) |
| C(17) | 17(3)| 33(3)| 25(3)| 4(2) | 9(2) | 9(2) |
| C(3)  | 11(3)| 28(3)| 41(3)| 7(3) | 4(2) | 6(2) |
| C(28) | 13(3)| 40(4)| 38(3)| 17(3)| 10(2)| 5(2) |
| C(1)  | 15(3)| 22(3)| 19(3)| 2(2) | 1(2) | 6(2) |
| C(2)  | 14(3)| 20(3)| 29(3)| 1(2) | 2(2) | 1(2) |
| C(53) | 14(3)| 25(3)| 17(3)| -1(2)| -4(2)| 2(2) |
| C(18) | 14(3)| 21(3)| 26(3)| 4(2) | 2(2) | 2(2) |
| C(27) | 12(3)| 27(3)| 30(3)| 11(3)| 9(2) | 4(2) |
| C(7)  | 25(3)| 38(3)| 25(3)| 14(3)| 10(2)| 12(3)|
| C(60) | 28(3)| 33(3)| 18(3)| 3(2) | 7(2) | 7(3) |
| C(23) | 18(3)| 24(3)| 27(3)| 5(2) | 8(2) | 10(2)|
| C(55) | 19(3)| 35(3)| 26(3)| 2(3) | 4(2) | 7(2) |
| C(59) | 30(3)| 42(4)| 23(3)| 8(3) | 9(2) | 14(3)|
| C(39) | 29(3)| 20(3)| 34(3)| 7(2) | 4(3) | 5(2) |
| C(20) | 26(3)| 19(3)| 27(3)| 2(2) | -3(2)| -2(2)|
| C(19) | 23(3)| 17(3)| 37(3)| 6(2) | 9(2) | 5(2) |
| C(70) | 20(3)| 32(3)| 20(3)| 1(2) | 5(2) | 6(2) |
| C(65) | 24(3)| 36(3)| 25(3)| 0(3) | 7(2) | 8(3)|
| C(22) | 14(3) | 29(3) | 45(4) | 1(3) | 6(2) | 4(2) |
|-------|-------|-------|-------|------|------|------|
| C(32) | 19(3) | 31(3) | 38(3) | 14(3) | 9(2) | 6(2) |
| C(54) | 15(3) | 35(3) | 23(3) | 6(2) | 1(2) | 4(2) |
| C(35) | 29(3) | 31(3) | 36(4) | 6(3) | 5(3) | 9(3) |
| C(74) | 20(3) | 30(3) | 37(3) | 3(3) | 6(2) | 10(2) |
| C(72) | 29(3) | 27(3) | 41(3) | 11(3) | 12(3) | 7(3) |
| C(76) | 23(3) | 32(3) | 40(3) | 10(3) | 8(3) | 12(3) |
| C(44) | 20(3) | 46(4) | 30(3) | 12(3) | 1(2) | 9(3) |
| C(71) | 24(3) | 30(3) | 36(3) | 9(3) | 11(2) | 12(2) |
| C(34) | 22(3) | 19(3) | 37(3) | 9(3) | -3(3) | -1(2) |
| C(38) | 24(3) | 18(3) | 54(4) | 14(3) | 11(3) | 3(2) |
| C(69) | 19(3) | 29(3) | 25(3) | 6(2) | 1(2) | 3(2) |
| C(33) | 27(3) | 27(3) | 34(3) | 10(3) | 5(3) | 5(3) |
| C(8)  | 23(3) | 38(3) | 37(3) | 23(3) | 15(3) | 16(3) |
| C(21) | 24(3) | 25(3) | 35(3) | 3(3) | -3(3) | 8(2) |
| C(5)  | 30(3) | 31(3) | 37(3) | 15(3) | 6(3) | 10(3) |
| C(73) | 24(3) | 33(3) | 31(3) | 7(3) | 11(2) | 10(3) |
| C(56) | 24(3) | 37(3) | 29(3) | -1(3) | -2(2) | 12(3) |
| C(61) | 29(3) | 46(4) | 22(3) | 3(3) | 6(3) | 5(3) |
| C(9)  | 26(3) | 44(4) | 45(4) | 28(3) | 17(3) | 12(3) |
| C(49) | 23(3) | 50(4) | 27(3) | 13(3) | 4(2) | 15(3) |
| C(24) | 36(3) | 34(3) | 31(3) | 2(3) | 15(3) | 10(3) |
| C(64) | 29(3) | 37(4) | 25(3) | 4(3) | 8(3) | -1(3) |
| C(4)  | 24(3) | 38(4) | 44(4) | 18(3) | 2(3) | 14(3) |
| C(31) | 30(3) | 32(3) | 54(4) | 8(3) | 6(3) | 17(3) |
| C(37) | 34(3) | 20(3) | 46(4) | 10(3) | -8(3) | 2(3) |
| C(45) | 23(3) | 71(5) | 30(3) | 15(3) | -1(3) | 12(3) |
| C(26) | 22(3) | 45(4) | 34(3) | 10(3) | 11(3) | 7(3) |
| C(63) | 48(4) | 29(3) | 28(3) | 2(3) | 13(3) | 11(3) |
| C(13) | 41(3) | 30(3) | 29(3) | 10(3) | 13(3) | 7(3) |
| C(62) | 35(3) | 54(4) | 24(3) | 4(3) | 3(3) | 13(3) |
| C(36) | 44(4) | 29(3) | 29(3) | 2(3) | -1(3) | 10(3) |
| C(43) | 18(3) | 51(4) | 37(3) | 20(3) | 6(3) | 10(3) |
| C(29) | 21(3) | 52(4) | 66(5) | 30(4) | 4(3) | 16(3) |
| C(48) | 24(3) | 63(4) | 46(4) | 27(3) | 8(3) | 11(3) |
| C(50) | 26(3) | 48(4) | 33(3) | 12(3) | 2(3) | 5(3) |
| C(10) | 39(4) | 57(4) | 74(5) | 46(4) | 36(4) | 30(3) |
|-------|-------|-------|-------|-------|-------|-------|
| C(40) | 43(4) | 46(4) | 29(3) | 7(3)  | 10(3) | 14(3) |
| C(25) | 25(3) | 28(3) | 53(4) | -4(3) | -8(3) | 8(3)  |
| C(66) | 28(3) | 62(4) | 35(3) | 12(3) | 4(3)  | 5(3)  |
| C(78) | 33(3) | 37(4) | 56(4) | 17(3) | 18(3) | 20(3) |
| C(16) | 41(4) | 49(4) | 35(4) | 3(3)  | 1(3)  | 7(3)  |
| C(77) | 28(3) | 43(4) | 64(4) | 20(3) | 22(3) | 12(3) |
| C(11) | 58(4) | 32(4) | 57(5) | 22(3) | 40(4) | 21(3) |
| C(68) | 37(4) | 69(5) | 37(4) | 9(3)  | -4(3) | -2(3) |
| C(41) | 36(4) | 31(4) | 68(5) | 5(3)  | -6(3) | 7(3)  |
| C(67) | 67(5) | 42(4) | 42(4) | 11(3) | 16(3) | 21(3) |
| C(30) | 27(3) | 48(4) | 70(5) | 16(4) | 9(3)  | 18(3) |
| C(12) | 62(5) | 37(4) | 47(4) | 12(3) | 29(3) | 14(3) |
| C(47) | 30(4) | 98(6) | 54(4) | 48(4) | 12(3) | 19(4) |
| C(14) | 22(3) | 80(5) | 50(4) | 32(4) | 4(3)  | 6(3)  |
| C(42) | 59(4) | 66(5) | 35(4) | 5(3)  | 15(3) | 24(4) |
| C(46) | 24(3) | 108(6) | 39(4) | 35(4) | 0(3)  | 20(4) |
| C(52) | 33(4) | 78(5) | 42(4) | 14(4) | -8(3) | 1(3)  |
| C(15) | 83(6) | 58(5) | 99(6) | 22(5) | 66(5) | 31(4) |
| C(51) | 41(5) | 180(10) | 126(8) | 122(8) | 1(5) | 13(5) |
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^-3) for Cain58 (3).

|       | x     | y     | z     | U(eq) |
|-------|-------|-------|-------|-------|
| H(57) | 2301  | 3245  | 7196  | 30    |
| H(17A)| 5753  | 6176  | 7490  | 29    |
| H(17B)| 4438  | 6387  | 7204  | 29    |
| H(3)  | 5661  | 5343  | 8231  | 32    |
| H(7A) | 1456  | 6722  | 9021  | 33    |
| H(7B) | 553   | 5850  | 8529  | 33    |
| H(55) | 4174  | 4424  | 5953  | 33    |
| H(59A)| 2257  | 5998  | 5818  | 36    |
| H(59B)| 3755  | 6400  | 6134  | 36    |
| H(20) | 8292  | 8268  | 9527  | 31    |
| H(22) | 9896  | 7902  | 7983  | 36    |
| H(74) | -4351 | 3918  | 7634  | 35    |
| H(72) | -3005 | 1736  | 7949  | 38    |
| H(76A)| -2081 | 5355  | 7474  | 46    |
| H(76B)| -3234 | 4838  | 6929  | 46    |
| H(76C)| -1738 | 4835  | 6849  | 46    |
| H(38) | 8558  | 9899  | 8047  | 37    |
| H(69A)| 858   | 3931  | 7756  | 30    |
| H(69B)| 498   | 4868  | 7632  | 30    |
| H(33A)| 3275  | 9006  | 8402  | 35    |
| H(33B)| 4085  | 8676  | 7868  | 35    |
| H(5)  | 2706  | 4972  | 9245  | 37    |
| H(56) | 3886  | 3296  | 6534  | 37    |
| H(24A)| 5172  | 8225  | 8906  | 50    |
| H(24B)| 6053  | 8294  | 9530  | 50    |
| H(24C)| 5276  | 7292  | 9128  | 50    |
| H(64) | 6165  | 7777  | 4778  | 40    |
| H(4)  | 4698  | 4704  | 8980  | 39    |
| H(31) | 2269  | 10386 | 7929  | 45    |
| H(26A)| 7839  | 6800  | 7018  | 50    |
| Location | H(26B) | H(26C) | H(62) | H(36) | H(43A) | H(43B) | H(29) | H(48) | H(50A) | H(50B) | H(50C) | H(10) | H(40A) | H(40B) | H(40C) | H(25A) | H(25B) | H(25C) | H(66A) | H(66B) | H(66C) | H(78A) | H(78B) | H(78C) | H(16A) | H(16B) | H(16C) | H(77A) | H(77B) | H(77C) | H(68A) | H(68B) | H(68C) | H(41A) | H(41B) | H(41C) |
|----------|--------|--------|-------|-------|--------|--------|-------|-------|--------|--------|--------|-------|--------|-------|-------|--------|--------|--------|--------|--------|-------|-------|--------|-------|--------|-------|--------|-------|--------|-------|--------|-------|--------|-------|
|          | 8448   | 7884   | 7033  | 50    |        |        | -187  | 52    |        |        |        | 46    |        | 42    | 40    |        |        | 54    |        | 54    | 54    | 63    | 63    | 63    | 64    | 64    | 77    | 77    | 77    | 77    | 70    | 70    | 70    |
|        |        |        |        |        |
|--------|--------|--------|--------|--------|
| H(67A) | 4138   | 8645   | 4054   | 72     |
| H(67B) | 4258   | 7857   | 3496   | 72     |
| H(67C) | 5539   | 8434   | 3960   | 72     |
| H(30)  | 588    | 10411  | 7197   | 56     |
| H(12)  | 457    | 7323   | 11053  | 57     |
| H(14A) | -1779  | 5697   | 8641   | 74     |
| H(14B) | -2776  | 5009   | 8951   | 74     |
| H(14C) | -1401  | 4794   | 8806   | 74     |
| H(42A) | 4591   | 11201  | 9194   | 78     |
| H(42B) | 5447   | 11112  | 9787   | 78     |
| H(42C) | 4347   | 10224  | 9404   | 78     |
| H(46)  | 223    | 8593   | 4130   | 65     |
| H(52A) | -475   | 6684   | 4874   | 81     |
| H(52B) | -734   | 6927   | 4225   | 81     |
| H(52C) | -1353  | 7400   | 4785   | 81     |
| H(15A) | -2986  | 7231   | 10746  | 111    |
| H(15B) | -1662  | 7889   | 11147  | 111    |
| H(15C) | -2277  | 6879   | 11277  | 111    |
| H(51A) | 1694   | 10542  | 4298   | 160    |
| H(51B) | 1896   | 9768   | 3751   | 160    |
| H(51C) | 3136   | 10395  | 4223   | 160    |
# X-RAY OF 6

Table 1. Crystal data and structure refinement for cain100_0m_b (6).

| Property                        | Value                                      |
|---------------------------------|--------------------------------------------|
| Identification code             | BII                                        |
| Empirical formula               | C20 H13 F12 O2 P                           |
| Formula weight                  | 544.27                                     |
| Temperature                     | 100.0 K                                    |
| Wavelength                      | 1.54178 Å                                  |
| Crystal system                  | Monoclinic                                 |
| Space group                     | P 21                                       |
| Unit cell dimensions            | a = 6.4519(4) Å, b = 14.3030(9) Å, c = 11.4008(6) Å |
|                                | α = 90°, β = 92.203(4)°, γ = 90°.          |
| Volume                          | 1051.31(11) Å³                            |
| Z                               | 2                                          |
| Density (calculated)            | 1.719 Mg/m³                                 |
| Absorption coefficient          | 2.334 mm⁻¹                                 |
| F(000)                          | 544                                        |
| Crystal size                    | 0.29 x 0.26 x 0.23 mm³                     |
| Theta range for data collection | 3.880 to 70.023°.                          |
| Index ranges                    | -7≤h≤7, -17≤k≤17, -13≤l≤13                 |
| Reflections collected           | 6831                                       |
| Independent reflections         | 3721 [R(int) = 0.0264]                     |
| Completeness to theta = 67.679° | 97.6 %                                     |
| Absorption correction           | None                                       |
| Max. and min. transmission      | 0.5220 and 0.4062                          |
| Refinement method               | Full-matrix least-squares on F²            |
| Data / restraints / parameters  | 3721 / 1 / 333                            |
| Goodness-of-fit on F²           | 1.086                                      |
| Final R indices [I>2sigma(I)]   | R1 = 0.0508, wR2 = 0.1294                  |
| R indices (all data)            | R1 = 0.0583, wR2 = 0.1345                  |
| Absolute structure parameter    | 0.14(3)                                    |
| Extinction coefficient          | n/a                                        |
| Largest diff. peak and hole     | 0.478 and -0.463 e.Å⁻³                    |
Table 2. Atomic coordinates (x $10^4$) and equivalent isotropic displacement parameters (Å$^2 	imes 10^3$) for cain100_0m_b (6). U(eq) is defined as one third of the trace of the orthogonalized $U^{ij}$ tensor.

|       | x     | y     | z     | U(eq)   |
|-------|-------|-------|-------|---------|
| F(1)  | 5175(18) | 6976(4) | 6142(6) | 112(4) |
| F(2)  | 3119(14) | 8130(4) | 6482(6) | 84(3)  |
| F(3)  | 5550(11) | 7759(4) | 7724(6) | 62(2)  |
| F(4)  | 685(14)  | 7966(4) | 8341(5) | 80(3)  |
| F(5)  | 3006(9)  | 7381(4) | 9475(4) | 51(1)  |
| F(6)  | 311(9)   | 6609(5) | 9080(6) | 61(2)  |
| F(7)  | 334(10)  | 3373(5) | 9067(7) | 70(2)  |
| F(8)  | 2999(9)  | 2549(5) | 9391(5) | 62(2)  |
| F(9)  | 504(13)  | 2040(5) | 8289(5) | 74(2)  |
| F(10) | 5556(12) | 2277(5) | 7593(6) | 71(2)  |
| F(11) | 3089(14) | 1853(4) | 6444(6) | 84(3)  |
| F(12) | 4781(15)| 3052(5) | 5983(5) | 84(2)  |
| O(1)  | 921(18)  | 6450(7) | 6835(9) | 31(3)  |
| O(1') | 1540(40) | 6529(17)| 6550(20)| 32(2)  |
| O(2)  | 770(20)  | 3587(10)| 6825(10)| 30(2)  |
| O(2') | 1360(50)| 3490(20)| 6550(30)| 30(2)  |
| C(2)  | 1325(6)  | 5050(9) | 5146(3) | 36(1)  |
| C(3)  | 3350(6)  | 5035(9) | 4785(3) | 39(1)  |
| C(4)  | 3750(6)  | 5024(9) | 3599(3) | 34(1)  |
| C(5)  | 2150(6)  | 4991(7) | 2747(3) | 28(1)  |
| C(6)  | 141(6)   | 5005(8) | 3115(3) | 32(1)  |
| C(7)  | -280(6)  | 5012(8) | 4298(3) | 32(1)  |
| C(8)  | 2618(7)  | 5001(9) | 1462(3) | 37(1)  |
| C(9)  | 2599(13)| 3261(6) | 7506(7) | 31(2)  |
| C(10) | 4030(20)| 2603(7) | 6873(8) | 53(2)  |
| C(11) | 1621(15)| 2792(6) | 8575(8) | 36(2)  |
| C(12) | 2871(5)  | 4995(7) | 7548(3) | 20(1)  |
| C(13) | 3709(12)| 4155(5) | 7903(6) | 22(2)  |
| C(14) | 5524(12)| 4142(5) | 8651(6) | 24(2)  |
| C(15) | 6415(6)  | 4979(8) | 8995(3) | 30(1)  |
| C(16) | 5554(13)| 5817(6) | 8658(7) | 30(2)  |
|      |      |      |      |      |
|------|------|------|------|------|
| C(17)| 3790(12) | 5833(5) | 7960(6) | 23(1) |
| C(18)| 2662(14) | 6713(6) | 7561(6) | 32(2) |
| C(19)| 1655(14) | 7187(6) | 8613(7) | 33(2) |
| C(20)| 4130(20) | 7403(7) | 6970(7) | 55(3) |
| P(1’) | 505(9) | 4798(5) | 6673(5) | 22(1) |
| P(1)  | 496(6) | 5280(3) | 6649(3) | 22(1) |
| C(1)  | -110(30) | 2882(12) | 5942(15) | 65(4) |
| C(1’) | -20(30) | 7134(15) | 6016(16) | 23(1) |
Table 3. Bond lengths [Å] and angles [°] for cain100_0m_b (6).

| Bond                  | Distance [Å] |
|-----------------------|--------------|
| F(1)-C(20)            | 1.328(12)    |
| F(2)-C(20)            | 1.337(12)    |
| F(3)-C(20)            | 1.334(13)    |
| F(4)-C(19)            | 1.309(10)    |
| F(5)-C(19)            | 1.318(9)     |
| F(6)-C(19)            | 1.325(12)    |
| F(7)-C(11)            | 1.316(12)    |
| F(8)-C(11)            | 1.308(9)     |
| F(9)-C(11)            | 1.329(10)    |
| F(10)-C(10)           | 1.341(13)    |
| F(11)-C(10)           | 1.317(11)    |
| F(12)-C(10)           | 1.310(12)    |
| O(1)-C(18)            | 1.421(13)    |
| O(1')-C(18)           | 1.37(2)      |
| O(1')-P(1)            | 1.91(2)      |
| O(1')-C(1')           | 1.44(3)      |
| O(2)-C(9)             | 1.463(16)    |
| O(2)-P(1')            | 1.749(16)    |
| O(2)-C(1)             | 1.520(19)    |
| O(2')-C(9)            | 1.36(3)      |
| C(2)-C(3)             | 1.385(6)     |
| C(2)-C(7)             | 1.390(5)     |
| C(2)-P(1')            | 1.874(7)     |
| C(2)-P(1)             | 1.844(6)     |
| C(3)-H(3)             | 0.9500       |
| C(3)-C(4)             | 1.387(5)     |
| C(4)-H(4)             | 0.9500       |
| C(4)-C(5)             | 1.391(6)     |
| C(5)-C(6)             | 1.378(6)     |
| C(5)-C(8)             | 1.508(5)     |
| C(6)-H(6)             | 0.9500       |
| C(6)-C(7)             | 1.385(5)     |
| C(7)-H(7)             | 0.9500       |
| C(8)-H(8A)            | 0.9800       |
| Bond                | Length  |
|---------------------|---------|
| C(8)-H(8B)         | 0.9800  |
| C(8)-H(8C)         | 0.9800  |
| C(9)-C(10)         | 1.520(14)|
| C(9)-C(11)         | 1.548(12)|
| C(9)-C(13)         | 1.526(10)|
| C(12)-C(13)        | 1.372(11)|
| C(12)-C(17)        | 1.410(11)|
| C(12)-P(1')        | 1.814(7) |
| C(12)-P(1)         | 1.856(6) |
| C(13)-C(14)        | 1.422(11)|
| C(14)-H(14)        | 0.9500  |
| C(14)-C(15)        | 1.379(13)|
| C(15)-H(15)        | 0.9500  |
| C(15)-C(16)        | 1.370(13)|
| C(16)-H(16)        | 0.9500  |
| C(16)-C(17)        | 1.364(11)|
| C(17)-C(18)        | 1.515(10)|
| C(18)-C(19)        | 1.542(11)|
| C(18)-C(20)        | 1.541(14)|
| C(1)-H(1A)         | 0.9800  |
| C(1)-H(1B)         | 0.9800  |
| C(1)-H(1C)         | 0.9800  |
| C(1')-H(1'A)       | 0.9800  |
| C(1')-H(1'B)       | 0.9800  |
| C(1')-H(1'C)       | 0.9800  |
| C(18)-O(1')-P(1)   | 107.8(15)|
| C(18)-O(1')-C(1')  | 125(2)   |
| C(1')-O(1')-P(1)   | 110.2(16)|
| C(9)-O(2)-P(1')    | 116.2(9) |
| C(9)-O(2)-C(1)     | 114.5(11)|
| C(1)-O(2)-P(1')    | 124.0(11)|
| C(3)-C(2)-C(7)     | 118.6(4) |
| C(3)-C(2)-P(1')    | 125.1(4) |
| C(3)-C(2)-P(1)     | 126.1(4) |
| C(7)-C(2)-P(1')    | 114.1(4) |
C(7)-C(2)-P(1) 114.7(4)
C(2)-C(3)-H(3)  119.9
C(2)-C(3)-C(4)  120.2(4)
C(4)-C(3)-H(3)  119.9
C(3)-C(4)-H(4)  119.3
C(3)-C(4)-C(5)  121.4(4)
C(5)-C(4)-H(4)  119.3
C(4)-C(5)-C(8)  120.5(4)
C(6)-C(5)-C(4)  117.9(3)
C(6)-C(5)-C(8)  121.5(4)
C(5)-C(6)-H(6)  119.4
C(5)-C(6)-C(7)  121.2(4)
C(7)-C(6)-H(6)  119.4
C(2)-C(7)-H(7)  119.7
C(6)-C(7)-C(2)  120.6(4)
C(6)-C(7)-H(7)  119.7
C(5)-C(8)-H(8A)  109.5
C(5)-C(8)-H(8B)  109.5
C(5)-C(8)-H(8C)  109.5
H(8A)-C(8)-H(8B)  109.5
H(8A)-C(8)-H(8C)  109.5
H(8B)-C(8)-H(8C)  109.5
O(2)-C(9)-C(10)  116.0(8)
O(2)-C(9)-C(11)  102.2(8)
O(2)-C(9)-C(13)  104.5(8)
O(2')-C(9)-C(10)  96.9(13)
O(2')-C(9)-C(11)  119.3(15)
O(2')-C(9)-C(13)  106.7(16)
C(10)-C(9)-C(11)  112.2(7)
C(10)-C(9)-C(13)  111.9(8)
C(13)-C(9)-C(11)  109.3(6)
F(10)-C(10)-C(9)  111.7(7)
F(11)-C(10)-F(10)  105.2(8)
F(11)-C(10)-C(9)  113.7(10)
F(12)-C(10)-F(10)  111.1(11)
F(12)-C(10)-F(11)  106.8(8)
F(12)-C(10)-C(9) 108.3(8)
F(7)-C(11)-F(9) 105.7(9)
F(7)-C(11)-C(9) 110.1(7)
F(8)-C(11)-F(7) 106.7(8)
F(8)-C(11)-F(9) 107.9(8)
F(8)-C(11)-C(9) 112.9(8)
F(9)-C(11)-C(9) 113.0(7)
C(13)-C(12)-C(17) 119.4(3)
C(13)-C(12)-P(1') 109.9(6)
C(13)-C(12)-P(1) 131.5(6)
C(17)-C(12)-P(1) 130.5(7)
C(17)-C(12)-P(1') 109.0(6)
C(12)-C(13)-C(9) 118.1(7)
C(12)-C(13)-C(14) 119.6(6)
C(14)-C(13)-C(9) 122.3(7)
C(13)-C(14)-H(14) 120.5
C(15)-C(14)-C(13) 119.0(7)
C(15)-C(14)-H(14) 120.5
C(14)-C(15)-H(15) 119.3
C(16)-C(15)-C(14) 121.3(4)
C(16)-C(15)-H(15) 119.3
C(15)-C(16)-H(16) 120.0
C(17)-C(16)-C(15) 119.9(8)
C(17)-C(16)-H(16) 120.0
C(12)-C(17)-C(18) 114.6(6)
C(16)-C(17)-C(12) 120.7(7)
C(16)-C(17)-C(18) 124.7(7)
O(1)-C(18)-C(17) 108.3(7)
O(1)-C(18)-C(19) 102.8(8)
O(1)-C(18)-C(20) 113.6(8)
O(1')-C(18)-C(17) 109.1(12)
O(1')-C(18)-C(19) 121.1(12)
O(1')-C(18)-C(20) 94.0(11)
C(17)-C(18)-C(19) 110.1(6)
C(17)-C(18)-C(20) 111.5(8)
C(20)-C(18)-C(19) 110.1(7)
F(4)-C(19)-F(5)  107.1(7)
F(4)-C(19)-F(6)  108.2(9)
F(4)-C(19)-C(18)  113.7(7)
F(5)-C(19)-F(6)  104.8(8)
F(5)-C(19)-C(18)  112.7(7)
F(6)-C(19)-C(18)  109.9(7)
F(1)-C(20)-F(2)  108.4(8)
F(1)-C(20)-F(3)  106.2(12)
F(1)-C(20)-C(18)  110.8(8)
F(2)-C(20)-C(18)  112.4(11)
F(3)-C(20)-F(2)  106.4(8)
F(3)-C(20)-C(18)  112.4(7)
O(2)-P(1')-C(2)  104.6(7)
O(2)-P(1')-C(12)  91.3(6)
C(12)-P(1')-C(2)  102.7(4)
C(2)-P(1)-O(1')  89.7(8)
C(2)-P(1)-C(12)  102.2(3)
C(12)-P(1)-O(1')  87.3(8)
O(2)-C(1)-H(1A)  109.5
O(2)-C(1)-H(1B)  109.5
O(2)-C(1)-H(1C)  109.5
H(1A)-C(1)-H(1B)  109.5
H(1A)-C(1)-H(1C)  109.5
H(1B)-C(1)-H(1C)  109.5
O(1')-C(1')-H(1'A)  109.5
O(1')-C(1')-H(1'B)  109.5
O(1')-C(1')-H(1'C)  109.5
H(1'A)-C(1')-H(1'B)  109.5
H(1'A)-C(1')-H(1'C)  109.5
H(1'B)-C(1')-H(1'C)  109.5

Symmetry transformations used to generate equivalent atoms:
Table 4. Anisotropic displacement parameters ($\AA^2 \times 10^3$) for cain100_0m_b (6). The anisotropic displacement factor exponent takes the form: $-2\pi^2 [ h^2 a^* U^{11} + ... + 2hk a^* b^* U^{12} ]$

|      | $U^{11}$  | $U^{22}$  | $U^{33}$  | $U^{23}$  | $U^{13}$  | $U^{12}$  |
|------|-----------|-----------|-----------|-----------|-----------|-----------|
| F(1) | 231(10)   | 35(4)     | 79(4)     | 18(3)     | 99(6)     | 16(4)     |
| F(2) | 171(9)    | 20(3)     | 58(4)     | 11(3)     | -26(4)    | 10(4)     |
| F(3) | 63(4)     | 40(3)     | 85(4)     | 17(3)     | 12(3)     | -12(3)    |
| F(4) | 139(7)    | 49(4)     | 51(3)     | -18(3)    | -19(4)    | 63(4)     |
| F(5) | 39(3)     | 80(4)     | 33(2)     | -23(2)    | -6(2)     | 1(3)      |
| F(6) | 37(3)     | 54(4)     | 94(5)     | -33(3)    | 25(3)     | -3(3)     |
| F(7) | 43(4)     | 64(4)     | 105(5)    | 38(4)     | 32(4)     | 6(3)      |
| F(8) | 38(3)     | 88(5)     | 60(3)     | 52(3)     | -15(2)    | -10(3)    |
| F(9) | 108(6)    | 62(4)     | 53(3)     | 20(3)     | -8(3)     | -54(4)    |
| F(10)| 70(5)     | 54(4)     | 88(4)     | -31(3)    | 0(3)      | 23(3)     |
| F(11)| 165(9)    | 31(3)     | 53(3)     | -8(3)     | -23(4)    | -20(4)    |
| F(12)| 153(7)    | 46(4)     | 57(3)     | -10(3)    | 49(4)     | -1(3)     |
| O(1) | 34(6)     | 26(5)     | 32(5)     | -13(4)    | -14(4)    | 13(4)     |
| O(1')| 40(5)     | 27(4)     | 29(3)     | -8(3)     | -10(3)    | 4(3)      |
| O(2) | 37(7)     | 26(4)     | 27(6)     | 5(4)      | -10(4)    | -8(5)     |
| O(2')| 37(7)     | 26(4)     | 27(6)     | 5(4)      | -10(4)    | -8(5)     |
| C(2) | 25(2)     | 63(4)     | 18(2)     | 4(4)      | -2(1)     | -3(4)     |
| C(3) | 25(2)     | 69(4)     | 21(2)     | -1(4)     | -4(2)     | 5(5)      |
| C(4) | 26(2)     | 47(3)     | 28(2)     | 0(4)      | 4(2)      | 4(4)      |
| C(5) | 40(2)     | 23(2)     | 21(2)     | 1(3)      | 1(2)      | -1(4)     |
| C(6) | 35(2)     | 36(2)     | 23(2)     | -10(4)    | -8(2)     | 3(4)      |
| C(7) | 23(2)     | 48(3)     | 24(2)     | -1(4)     | -3(1)     | -6(4)     |
| C(8) | 46(3)     | 42(3)     | 24(2)     | 3(4)      | 2(2)      | -1(5)     |
| C(9) | 38(5)     | 21(4)     | 33(3)     | 4(3)      | -10(3)    | -10(3)    |
| C(10)| 85(8)     | 24(5)     | 50(5)     | -8(4)     | 6(4)      | 1(5)      |
| C(11)| 31(5)     | 29(5)     | 47(4)     | 8(3)      | -9(3)     | -9(3)     |
| C(12)| 21(2)     | 23(2)     | 16(2)     | 0(3)      | 2(1)      | -3(4)     |
| C(13)| 26(4)     | 22(4)     | 19(3)     | 0(2)      | 4(2)      | 1(3)      |
| C(14)| 26(4)     | 22(4)     | 23(3)     | 3(3)      | -2(3)     | 5(3)      |
| C(15)| 26(2)     | 37(2)     | 25(2)     | -2(4)     | -9(1)     | 15(4)     |
| C(16)| 27(4)     | 28(4)     | 33(4)     | -3(3)     | -3(3)     | -3(3)     |
|        |      |      |      |      |      |      |
|--------|------|------|------|------|------|------|
| C(17)  | 26(4)| 18(4)| 24(3)| -2(2)| -2(2)| 4(3) |
| C(18)  | 40(5)| 27(4)| 29(3)| -8(3)| -10(3)| 4(3) |
| C(19)  | 29(4)| 32(5)| 37(4)| -17(3)| -14(3)| 7(3) |
| C(20)  | 107(9)| 27(5)| 32(4)| 4(4)| 6(4)| 12(5) |
| P(1')  | 19(1)| 26(1)| 19(1)| -5(2)| -2(1)| 2(2) |
| P(1)   | 19(1)| 26(1)| 19(1)| -5(2)| -2(1)| 2(2) |
| C(1)   | 61(8)| 57(7)| 76(8)| -12(6)| -20(6)| -31(6) |
| C(1')  | 26(4)| 18(4)| 24(3)| -2(2)| -2(2)| 4(3) |
Table 5. Hydrogen coordinates ( x $10^4$) and isotropic displacement parameters ($\AA^2$ x $10^{-3}$) for cain100_0m_b (6).

|     | x    | y    | z    | U(eq) |
|-----|------|------|------|-------|
| H(3) | 4468 | 5031 | 5352 | 46    |
| H(4) | 5146 | 5040 | 3363 | 40    |
| H(6) | -977 | 5009 | 2548 | 38    |
| H(7) | -1676 | 4990 | 4531 | 38    |
| H(8A)| 2789 | 5648 | 1201 | 56    |
| H(8B)| 1470 | 4709 | 1008 | 56    |
| H(8C)| 3899 | 4651 | 1341 | 56    |
| H(14)| 6113 | 3566 | 8910 | 29    |
| H(15)| 7650 | 4975 | 9476 | 36    |
| H(16)| 6185 | 6387 | 8910 | 35    |
| H(1A)| 665  | 2914 | 5221 | 98    |
| H(1B)| -1575 | 3026 | 5761 | 98    |
| H(1C)| 3    | 2251 | 6274 | 98    |
| H(1'A)| -593  | 7538 | 6618 | 34    |
| H(1'B)| -1137 | 6754 | 5653 | 34    |
| H(1'C)| 604   | 7521 | 5415 | 34    |