Supporting Information for

*CuCl*-Mediated tandem CO insertion and annulation of 1,4-dilithio-1,3-dienes: formation of multiply substituted cyclopentadienones and/or their head-to-head dimers

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Contents

1. Experimental details and characterization data for all compounds.
2. Copies of $^1$H NMR and $^{13}$C NMR spectra for all new compounds.
3. X-ray crystallographic studies of $3a$ and $3d$. 
1. **Experimental details and characterization data for all new compounds.**

**General Methods.** All reactions were conducted under a slightly positive pressure of dry, prepurified nitrogen using standard Schlenk line techniques when appropriate. Unless otherwise noted, all starting materials were commercially available and were used without further purification. Diethyl ether was refluxed and distilled from sodium/benzophenone ketyl under a nitrogen atmosphere. 1,4-Dihalo-1,3-butadiene compounds are prepared by the reported ways. $^1$t-BuLi was obtained from Acros Organics. $^1$H NMR, and $^{13}$C NMR spectra were recorded on a JEOL-AL300 spectrometer (FT, 300 MHz for $^1$H; 75.4 MHz for $^{13}$C) in CDCl$_3$ at room temperature using Me$_4$Si as an internal standard, unless otherwise noted. IR spectra were obtained on a Thermo Nicolet AVATAR 330 FT-IR spectrophotometer. HRMS were recorded on a ZAB-HS instrument.

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A typical procedure for syntheses of cyclopentadienones 2a–c and/or their head-to-head dimers 3a–c: To a 10 mL solution of 1,4-diodo-1,3-diene compound (1.0 mmol) in Et$_2$O at −78 °C was added t-BuLi (4.0 mmol, 1.5 mol/L in pentane). After this reaction mixture was stirred at −78 °C for 1 h, CuCl (2.0 mmol) was added and kept at −78 °C for 0.5 h. Then CO was bubbled into the vessel for 5 min, followed by addition of tBuOOtBu (2.0 mmol) to this reaction mixture. After 1 h of stirring at 0 °C, the reaction mixture was quenched with water and extracted with Et$_2$O. The extraction was washed with brine and dried over MgSO$_4$. The solvent was then evaporated in vacuo and the residue was purified by column chromatograph using silica gel (hexane: Et$_2$O = 20:1) to afford the final products 2a–c and 3a–c.

**2a:** red oil (25 mg, 10%); $^1$H NMR (300 MHz, CDCl$_3$): $\delta = 0.88$ (t, $J = 7.2$ Hz, 6H, CH$_3$), 0.99 (t, $J = 7.2$ Hz, 6H, CH$_3$), 1.33–1.54 (m, 8H, CH$_2$), 2.04 (t, $J = 7.5$ Hz, 4H, CH$_2$), 2.23 (t, $J = 7.5$ Hz, 4H, CH$_2$); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta = 14.23$ (2 CH$_3$),
14.43 (2 CH₃), 22.52 (2 CH₂), 22.85 (2 CH₂), 24.91 (2 CH₂), 28.30 (2 CH₂), 125.78 (2 quat. C), 154.95 (2 quat. C), 204.94 (1 ketone C=O). IR (neat): \( \nu (C=O) = 1716 \text{ cm}^{-1} \).

HRMS (IE): calcd. for C₁₇H₂₈O: 248.2140, found 248.2145. The NMR data are identical with the literature. ²

**2b:** red oil (33 mg, 11%); \(^1\)H NMR (300 MHz, CDCl₃): \( \delta = 0.87–1.00 \) (m, 12H, CH₃), 1.23–1.44 (m, 16H, CH₂), 2.05 (t, \( J = 7.2 \) Hz, 4H, CH₂), 2.24 (t, \( J = 7.2 \) Hz, 4H, CH₂); \(^{13}\)C NMR (75 MHz, CDCl₃): \( \delta = 14.03 \) (2 CH₃), 14.12 (2 CH₃), 23.15 (2 CH₂), 23.21 (2 CH₂), 23.27 (2 CH₂), 26.61 (2 CH₂), 31.64 (2 CH₂), 32.27 (2 CH₂), 126.27 (2 quat. C), 154.57 (2 quat. C), 203.89 (1 ketone C=O). IR (neat): \( \nu (C=O) = 1714 \text{ cm}^{-1} \).

HRMS (IE): calcd. for C₂₁H₃₆O: 304.2766, found 304.2757. The NMR data are identical with the literature. ²

**2c:** orange oil (23 mg, 12%); \(^1\)H NMR (300 MHz, CDCl₃): \( \delta = 0.99 \) (t, \( J = 7.5 \) Hz, 6H, CH₃), 1.11 (t, \( J = 7.5 \) Hz, 6H, CH₃), 2.10 (q, \( J = 7.5 \) Hz, 4H, CH₂), 2.29 (q, \( J = 7.5 \) Hz, 4H, CH₂); \(^{13}\)C NMR (75 MHz, CDCl₃): \( \delta = 13.79 \) (2 CH₃), 14.45 (2 CH₃), 15.88 (2 CH₂), 19.07 (2 CH₂), 126.68 (2 quat. C), 155.85 (2 quat. C), 204.62 (1 ketone C=O). IR (neat): \( \nu (C=O) = 1682 \text{ cm}^{-1} \).

HRMS (IE): calcd. for C₁₃H₂₀O: 192.1514, found 192.1516.

**3a:** Colorless solid (151 mg, 61%); m. p. 82.1–83.4 °C; \(^1\)H NMR (300 MHz, CDCl₃): \( \delta = 0.74 \) (t, \( J = 7.2 \) Hz, 6H, CH₃), 0.82 (t, \( J = 7.2 \) Hz, 6H, CH₃), 0.97 (t, \( J = 7.2 \) Hz, 6H, CH₃), 1.09 (t, \( J = 7.2 \) Hz, 6H, CH₃), 1.36–1.73 (m, 24H, CH₂), 2.18–2.41 (m, 8H, CH₂); \(^{13}\)C NMR (75 MHz, CDCl₃): \( \delta = 14.70 \) (2 CH₃), 15.05 (2 CH₃), 15.40 (2 CH₃), 15.66 (2 CH₃), 17.56 (2 CH₂), 19.22 (2 CH₂), 21.49 (2 CH₂), 21.72 (2 CH₂), 26.56 (2 CH₂), 32.49 (2 CH₂), 32.92 (2 CH₂), 33.30 (2 CH₂), 53.83 (2 quat. C), 60.85 (2 quat. C), 144.84 (2 quat. C), 173.41 (2quat. C), 209.36 (2 ketone C=O). IR (neat): \( \nu (C=O) = 1692 \text{ cm}^{-1} \).

HRMS (EI): calcd. for C₃₄H₅₆O₂: 496.4280, found 496.4277.

**3b:** Colorless solid (128 mg, 42%); m. p. 65.6–67.0 °C; \(^1\)H NMR (300 MHz, CDCl₃):
\( \delta = 0.82 \ (t, J = 7.2 \ Hz, \ 6H, \ CH_3), \ 0.83 \ (t, J = 7.2 \ Hz, \ 6H, \ CH_3), \ 0.94 \ (t, J = 6.9 \ Hz, \ 6H, \ CH_3), \ 1.00 \ (t, J = 7.2 \ Hz, \ 6H, \ CH_3), \ 1.11-1.64 \ (m, \ 40H, \ CH_2), \ 2.20-2.38 \ (m, \ 8H, \ CH_2) \); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta = 13.85 \ (2 \ CH_3), \ 13.87 \ (2 \ CH_3), \ 13.91 \ (4 \ CH_3), \ 23.29 \ (2 \ CH_2), \ 23.83 \ (2 \ CH_2), \ 24.16 \ (6 \ CH_2), \ 26.30 \ (2 \ CH_2), \ 28.01 \ (2 \ CH_2), \ 29.95 \ (2 \ CH_2), \ 30.21 \ (2 \ CH_2), \ 30.31 \ (2 \ CH_2), \ 30.46 \ (2 \ CH_2), \ 30.73 \ (2 \ CH_2), \ 53.73 \ (2 \ quat. \ C), \ 60.77 \ (2 \ quat. \ C), \ 144.90 \ (2 \ quat. \ C), \ 173.44 \ (2 \ quat. \ C), \ 209.27 \ (2 \ ketone \ C=O). \) IR (neat): \( \nu \ (C=O) = 1694 \ \text{cm}^{-1}. \) HRMS (EI): calcd. for C\(_{42}H_72O_2\): 608.5532, found 608.5525.

**3c:** Colorless solid (92 mg, 48%); m. p. 92.3–93.5 °C; \(^{1}H\) NMR (300 MHz, CDCl\(_3\)): \( \delta = 0.62 \ (t, J = 7.2 \ Hz, \ 6H, \ CH_3), \ 1.08 \ (t, J = 7.5 \ Hz, \ 6H, \ CH_3), \ 1.11 \ (t, J = 7.5 \ Hz, \ 6H, \ CH_3), \ 1.28 \ (t, J = 7.5 \ Hz, \ 6H, \ CH_3), \ 1.43-1.85 \ (m, \ 8H, \ CH_2), \ 2.26-2.54 \ (m, \ 8H, \ CH_2); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta = 9.36 \ (2 \ CH_3), \ 10.92 \ (2 \ CH_3), \ 12.74 \ (4 \ CH_3), \ 17.31 \ (2 \ CH_2), \ 21.91 \ (2 \ CH_2), \ 23.13 \ (2 \ CH_2), \ 23.32 \ (2 \ CH_2), \ 53.80 \ (2 \ quat. \ C), \ 61.19 \ (2 \ quat. \ C), \ 146.33 \ (2 \ quat. \ C), \ 173.90 \ (2 \ quat. \ C), \ 209.45 \ (2 \ ketone \ C=O). \) IR (neat): \( \nu \ (C=O) = 1681 \ \text{cm}^{-1}. \) HRMS (EI): calcd. for C\(_{26}H_40O_2\): 384.3028, found 384.3019.

**3d:** Obtained as colorless solid (135 mg, 55%) in a manner analogous to that described for the synthesis of 3a. It was noted that only trace amount of cyclopentadienone was observed in this process. M. p. 117.1–118.5 °C; \(^{1}H\) NMR (300 MHz, CDCl\(_3\)): \( \delta = 0.77 \ (t, J = 7.2 \ Hz, \ 6H, \ CH_3), \ 0.91 \ (t, J = 7.2 \ Hz, \ 6H, \ CH_3), \ 1.12-1.37 \ (m, \ 20H, \ CH_2), \ 1.71-1.92 \ (m, \ 8H, \ CH_2), \ 2.08-2.91 \ (m, \ 12H, \ CH_2); \(^{13}\)C NMR (75 MHz, CDCl\(_3\)): \( \delta = 13.87 \ (2 \ CH_3), \ 13.90 \ (2 \ CH_3), \ 22.70 \ (2 \ CH_2), \ 22.77 \ (2 \ CH_2), \ 23.53 \ (2 \ CH_2), \ 24.09 \ (2 \ CH_2), \ 26.65 \ (2 \ CH_2), \ 27.62 \ (2 \ CH_2), \ 27.99 \ (2 \ CH_2), \ 28.35 \ (2 \ CH_2), \ 29.13 \ (2 \ CH_2), \ 31.11 \ (2 \ CH_2), \ 54.49 \ (2 \ quat. \ C), \ 54.57 \ (2 \ quat. \ C), \ 141.90 \ (2 \ quat. \ C), \ 174.79 \ (2 \ quat. \ C), \ 208.19 \ (2 \ ketone \ C=O). \) IR (neat): \( \nu \ (C=O) = 1692 \ \text{cm}^{-1}. \) HRMS (IE): calcd. for C\(_{34}H_52O_2\): 492.3967, found 492.3968.

**A typical procedure for syntheses of cyclopentadienones 2d–h:** To a 10 mL solution of 1,4-diiodo-1,3-diene compound (1.0 mmol) in Et\(_2\)O at −78 °C was added
\( \text{t-BuLi (4.0 mmol, 1.5 mol/L in pentane). After this reaction mixture was stirred at } \)
\(-78^\circ\text{C for 1 h, CuCl (2.0 mmol) was added and kept at } -78^\circ\text{C for 0.5 h. Then CO was}

bubbled into the vessel for 5 min, followed by addition of \( \text{t-BuOOtBu (2.0 mmol) to this reaction mixture. After 1 h of stirring at 0 } \)

\({}^\circ\text{C, the reaction mixture was}

quenched with water and extracted with \( \text{Et}_2\text{O. The extraction was washed with brine}

and dried over MgSO}_4. The solvent was then evaporated in vacuo and the residue was

purified by column chromatograph using silica gel (hexane: \( \text{Et}_2\text{O = 20:1} \) to afford

exclusively the final products 2d–h.}

\textbf{2d}: red solid (269 mg, 70%); \( ^1\text{H NMR (300 MHz, CDCl}_3\)): \( \delta = 6.90\text{–}6.94 \) (m, 4H, CH), 7.15–7.19 (m, 4H, CH), 7.23–7.26 (m, 12H, CH); \( ^{13}\text{C NMR (75 MHz, CDCl}_3\)): \( \delta = 125.30 \) (2 quat. C), 127.45 (2 CH), 127.98 (4 CH), 128.03 (4 CH), 128.50 (2 CH), 129.33 (4 CH), 130.14 (4 CH), 130.73 (2 quat. C), 133.05 (2 quat. C), 154.47 (2 quat. C), 200.24 (1 ketone C=O). IR (neat): \( v \) (C=O) = 1702 cm\(^{-1}\). HRMS (IE): calcd. for C\(_{29}\)H\(_{20}\)O: 384.1514, found 384.1522. The NMR data are identical with the literature. \(^3\)

\textbf{2e}: orange oil (236 mg, 70%); \( ^1\text{H NMR (300 MHz, CDCl}_3\)): \( \delta = 0.40 \) (s, 18H, TMS), 0.92 (t, \( J = 6.6 \) Hz, 6H, CH\(_3\)), 1.33–1.48 (m, 8H, CH\(_2\)), 2.40 (t, \( J = 7.2 \) Hz, 4H, CH\(_2\)); \( ^{13}\text{C NMR (75 MHz, CDCl}_3\)): \( \delta = 0.06 \) (6 CH\(_3\)) 13.86 (2 CH\(_3\)), 23.18 (2 CH\(_2\)), 28.05 (2 CH\(_2\)), 32.87 (2 CH\(_2\)), 128.21 (2 quat. C), 172.85 (2 quat. C), 211.12 (1 ketone C=O). IR (neat): \( v \) (C=O) = 1700 cm\(^{-1}\). HRMS (IE): calcd. for C\(_{19}\)H\(_{36}\)OSi\(_2\): 336.2305, found 336.2317. The NMR data are identical with the literature. \(^2\)

\textbf{2f}: red oil (145 mg, 50%); \( ^1\text{H NMR (300 MHz, CDCl}_3\)): \( \delta = 0.83 \) (t, \( J = 7.5 \) Hz, 3H, CH\(_3\)), 1.10 (t, \( J = 7.5 \) Hz, 3H, CH\(_3\)), 2.24–2.39 (m, 4H, CH\(_2\)), 7.15–7.39 (m, 10H, CH); \( ^{13}\text{C NMR (75 MHz, CDCl}_3\)): \( \delta = 13.09 \) (1 CH\(_3\)), 14.45 (1 CH\(_3\)), 16.22 (1 CH\(_2\)), 19.59 (1 CH\(_2\)), 124.79 (1 quat. C), 126.95 (1 CH), 127.35 (1 quat. C), 127.86 (2 CH), 128.17 (2 CH), 128.22 (2 CH), 128.63 (2 CH), 129.65 (1 CH), 131.00 (1 quat. C), 134.52 (1 quat. C), 154.05 (1 quat. C), 157.48 (1 quat. C), 202.56 (1 ketone C=O). IR (neat): \( v \) (C=O) = 1711 cm\(^{-1}\). HRMS (IE): calcd. for C\(_{21}\)H\(_{20}\)O: 288.1514, found

85
288.1512.

2g: red oil (234 mg, 68%); $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ = 0.57 (t, $J$ = 6.9 Hz, 3H, CH$_3$), 0.75 (t, $J$ = 6.9 Hz, 3H, CH$_3$), 0.80–1.53 (m, 8H, CH$_2$), 2.12 (t, $J$ = 6.9 Hz, 2H, CH$_2$), 2.42 (t, $J$ = 6.9 Hz, 2H, CH$_2$), 7.22–7.43 (m, 10H, CH); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ = 13.39 (1 CH$_3$), 13.75 (1 CH$_3$), 22.41 (1 CH$_2$), 22.66 (1 CH$_2$), 22.84 (1 CH$_2$), 26.69 (1 CH$_2$), 29.90 (1 CH$_2$), 31.62 (1 CH$_2$), 125.00 (1 quat. C), 127.05 (1 CH), 127.85 (2 CH), 128.07 (1 CH), 128.17 (2 CH), 128.35 (2 CH), 129.30 (2 CH), 131.72 (1 quat. C), 134.17 (1 quat. C), 153.47 (1 quat. C), 158.28 (2 quat. C), 202.53 (1 ketone C=O). IR (neat): $\nu$ (C=O) = 1690 cm$^{-1}$. HRMS (IE): calcd. for C$_{25}$H$_{28}$O: 344.2140, found 344.2145. The NMR data are identical with the literature.

2h: green oil (109 mg, 45%); $^1$H NMR (300 MHz, CDCl$_3$): $\delta$ = 0.85 (t, $J$ = 7.2 Hz, 3H, CH$_3$), 0.90 (t, $J$ = 7.2 Hz, 3H, CH$_3$), 1.23–1.55 (m, 8H, CH$_2$), 2.17 (t, $J$ = 7.5 Hz, 2H, CH$_2$) 2.45 (t, $J$ = 7.5 Hz, 2H, CH$_2$) 6.93–7.29 (m, 4H, CH); $^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ = 13.88 (2 CH$_3$), 22.58 (1 CH$_2$), 22.75 (1 CH$_2$), 22.99 (1 CH$_2$), 25.97 (1 CH$_2$), 29.94 (1 CH$_2$), 31.42 (1 CH$_2$), 118.90 (1 CH), 121.59 (1 CH), 127.79 (1 CH), 131.11 (1 quat. C), 133.08 (1 CH), 134.75 (1 quat. C), 145.64 (1 quat. C), 157.71 (1quat. C), 198.50 (1 ketone C=O). IR (neat): $\nu$ (C=O) = 1718 cm$^{-1}$. HRMS (IE): calcd. for C$_{17}$H$_{22}$O: 242.1671, found 242.1679. The NMR data are identical with the literature.

References:

2. Copies of $^1$H NMR and $^{13}$C NMR spectra for all new compounds.
3. **X-ray crystallographic studies of 3a and 3d:** Crystals for X-ray analyses of 3a and 3d were obtained as described in the preparations. The crystals were sealed in thin-walled glass capillaries. Data collections were performed at 20 °C on a Rigaku RAXIS RAPID IP, using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). The determination of crystal class and unit cell parameters was carried out by the SMART program package. The raw frame data were processed using SAINT and SADABS to yield the reflection data file. These structures were solved by use of SHELXTL program. Refinement was performed on F² anisotropically for all the non-hydrogen atoms by the full-matrix least-squares method. The hydrogen atoms were placed at the calculated positions and were included in the structure calculation without further refinement of the parameters. Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication nos. CCDC-668730 (3a) and CCDC-668731. Copies of these data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

![EFig. 1 ORTEP drawing of 3a with 30% thermal ellipsoids.](image)

**Table 1.** Crystal data and structure refinement for 3a.

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<th>Identification code</th>
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<td>CCDC number</td>
<td>668730</td>
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<tr>
<td>Empirical formula</td>
<td>C₃₄H₅₆O₂</td>
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Formula weight 496.79
Temperature 293(2) K
Wavelength 0.71073 Å
Crystal system, space group Orthorhombic, Aba2
Unit cell dimensions
\[ a = 19.655(4) \text{ Å} \quad \alpha = 90^{\circ} \]
\[ b = 20.326(4) \text{ Å} \quad \beta = 90^{\circ} \]
\[ c = 16.036(3) \text{ Å} \quad \gamma = 90^{\circ} \]
Volume 6407(2) Å³

Refls. No. for cell measurement 11916
Theta range for cell measurement 1.92 to 25.02 °
Z, Calculated density 8, 1.030 Mg/m³
Absorption coefficient 0.061 mm⁻¹
F(000) 2208

Crystal shape / Crystal colour block / colourless
Crystal size 0.45 x 0.20 x 0.18 mm
Theta range for data collection 1.92 to 25.02 °
Limiting indices \(-23 \leq h \leq 23, -24 \leq k \leq 24, -19 \leq l \leq 19\)
Reflections collected / unique 11916 / 2929 \([R_{int} = 0.0506]\)
Reflections with I>2σ(I) 1209
Completeness to theta = 25.02 ° 99.6 %
Decay correction (%) 0
Absorption correction Empirical
Max. and min. transmission 0.989 and 0.973
Method for primary solution direct
Method for secondary solution difmap
Hydrogen addition / treatment geom / constr
Refinement method Full-matrix least-squares on F²
Data / restraints / parameters 2929 / 2 / 334
Goodness-of-fit on F² 0.985
Final R indices [I>2σ(I)] \( R = 0.0527, \quad wR = 0.1108 \)
R indices (all data) \( R = 0.1280, \quad wR = 0.1210 \)
Absolute structure parameter -1(3)
Extinction coefficient 0.0044(2)
Largest diff. peak and hole 0.398 and -0.167 e.Å⁻³
Max. and mean shift/sigma 0.000 and 0.000
Measurement device Rigaku RAXIS RAPID IP
Measurement method \( \backslash W \)
Program for data collection Rapid-AUTO (Rigaku, 2000)
Program for cell refinement Rapid-AUTO
Program for data reduction CrystalStructure (Rigaku/MSC, 2000)
Program for structure solution SHELXS-97, (Sheldrick, 1997)
Program for structure refinement SHELXL-97, (Sheldrick, 1997)
Program for molecular graphics Siemens SHELXTL V4.2, (Sheldrick, 1990)
Program for publication material SHELXL-97, (Sheldrick, 1997)
Table 5. Crystal data and structure refinement for 3d.

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<tr>
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<td>Crystal shape / Crystal colour</td>
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<tr>
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<td>Reflections collected / unique</td>
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Decay correction (%)              0
Absorption correction             Empirical
Max. and min. transmission        0.992 and 0.978
Method for primary solution       direct
Method for secondary solution     difmap
Hydrogen addition / treatment     geom / constr
Refinement method                 Full-matrix least-squares on $F^2$
Data / restraints / parameters    2700 / 7 / 174
Goodness-of-fit on $F^2$          1.037
Final R indices [I>2sigma(I)]    $R = 0.0685$, $wR = 0.1558$
R indices (all data)              $R = 0.1758$, $wR = 0.1757$
Extinction coefficient            0.0024(4)
Largest diff. peak and hole       0.296 and -0.168 e.Å$^{-3}$
Max. and mean shift/sigma         0.000 and 0.000
Measurement device                Rigaku RAXIS RAPID IP
Measurement method                Rapid-AUTO (Rigaku, 2000)
Program for data collection       Rapid-AUTO (Rigaku, 2000)
Program for cell refinement       Rapid-AUTO
Program for data reduction        CrystalStructure (Rigaku/MSC, 2000)
Program for structure solution    SHELXS-97, (Sheldrick, 1997)
Program for structure refinement  SHELXL-97, (Sheldrick, 1997)
Program for molecular graphics    Siemens SHELXTL V4.2, (Sheldrick, 1990)
Program for publication material  SHELXL-97, (Sheldrick, 1997)