Supporting Information for

Alkali-Metal- and Halide- Free Dinuclear Mixed-Valent Samarium and Europium Complexes

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Experimental Section

Chemicals and reagents

Pyrrole (C₅H₅N, 98%) and 3-pentanone (C₅H₁₀O, 99%) were purchased from Alfa Aesar. Mercury (Hg, 99.99%) and 12-crown-4-ether(C₆H₁₆O₄, 98%) were purchased from sigma Aldrich. SmCl₃ (anhydrous, 100%) and EuCl₃ (anhydrous, 100%) were purchased from Strem Chemicals. Methanesulfonic acid (CH₃SO₃H, 70%) was purchased from Acros Organics. Lithium bis(trimethylsilyl)amide (C₆H₁₅Si₂NLi, 97%) was purchased from Aldrich. Toluene was refluxed over sodium and distilled under nitrogen before use. Any other solvents used for experiments under nitrogen were dried using a solvent purification system from Pure Process Technologies. All reactions except the ligand synthesis were performed under nitrogen.

Apparatus

All NMR spectra were obtained using a Bruker AVANCE 300 MHz or Bruker AVANCE III 400 MHz Spectrometer. Absorption and emission spectra were collected on a HORIBA Duetta Spectrophotometer using HORIBA EzSpec software. Cyclic voltammograms were collected on a CH Instruments Electrochemical Analyzer using a three-electrode system. Single-crystal X-ray diffraction studies were performed at Vanderbilt University, all measurements were made on a Rigaku Oxford Diffraction Supernova Eos CCD with filtered Cu Kα or Mo Kα radiation at a temperature of 100 K.

Synthesis of calix[4]-pyrrole: The synthesis of calix[4]pyrrole was adapted from Jacoby et al.¹ Freshly distilled pyrrole (11.6 mL, 167.2 mmol), 3-pentanone (17.7 mL, 167.2 mmol), and methanesulfonic acid (55.5 µL/ 10mmol of pyrrole) were mixed in ethanol and refluxed for 4.5 hrs. The dark brown reaction mixture was allowed to cool down to room temperature and placed in the refrigerator overnight. The white crystalline solid formed was filtered off, recrystallized from ethanol several times to remove unreacted pyrrole, washed with cold ethanol and dried under vacuum (17.4 g, 77%). ¹H NMR (CDCl₃, 400 MHz): δ (ppm) 0.63 (t, J = 9.7 Hz, CH₃, 24H), 1.8 (br s, CH₂, 16H), 5.93 (s, C₆H₃NH, 8H), 6.98 (s, C₆H₅NH, 4H).

Preparation of Sm(N(SiMe₃)₂)₃ (µ-Cl)Li(thf)₃: The synthesis of Sm(N(SiMe₃)₂)₃ (µ-Cl)Li(thf)₃ was adapted from Bradley et al.² Lithium bis(trimethylsilyl)amide (1.163 g, 6.950 mmol) was dissolved in dry THF (15 mL) under nitrogen and cooled down to 0 °C. Samarium(III) chloride (0.595 g, 2.317 mmol) was added portion wise over 30 minutes. Then the reaction mixture was stirred at room temperature for 24 hours. THF was removed under vacuum and the residue was extracted with n-pentane. Off white needle like crystals were obtained by slow evaporation of pentane (1.983 g, 96%). ¹H NMR (C₆D₆, 400 MHz): δ (ppm) -1.56 (s, SiCH₃, 54H), 1.35 (s, THF, 12H), 3.41 (s, THF, 12H).

Preparation of Eu(N(SiMe₃)₂)₃ (µ-Cl)Li(thf)₃: Prepared according to the same procedure described for Sm(N(SiMe₃)₂)₃ (µ-Cl)Li(thf)₃ (0.620 g, 90%). ¹H NMR (C₆D₆, 400 MHz): δ (ppm) 1.31 (s, THF, 12H), 3.61 (s, THF, 12H), 6.95 (s, SiCH₃, 54H).

Preparation of (η⁵:η¹:η⁵:η¹ Et₂-calix[4]pyrrolyl)Sm(N(SiMe₃)₂)₂: The synthesis was adapted from the procedure reported by Zhou et al.³ A toluene solution of calix[4]pyrrole (0.124 g, 0.23 mmol in 2.0 mL of toluene) was added dropwise to a toluene solution of Sm(N(SiMe₃)₂)₃ (µ-
Needle like crystals were obtained by slow evaporation of pentane (0.600 g, 23%). $^1$H NMR (C$_6$D$_6$, 400 MHz): δ (ppm) -0.91 (s, CH$_3$, 36H), -0.63 (t, J = 6.7 Hz, CH$_3$, 24H), 0.29 (s, CH$_2$, 16H), 10.14 (s, C$_4$H$_2$N, 8H). Elem. Anal. for C$_{48}$H$_{64}$N$_5$Si$_4$Sm$_2$ calcd. C, 49.77; H, 7.31; N, 7.26; found: C, 49.82; H, 7.38; N, 7.36.

Preparation of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{EuN(SiMe$_3$)$_2$}$_2$: Prepared according to the same procedure described for ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{SmN(SiMe$_3$)$_2$}$_2$ (0.231 g, 59%). $^1$H NMR (C$_6$D$_6$, 400 MHz): δ (ppm) -5.97 (s, C$_4$H$_2$N, 4H), 8.69 (s, CH$_2$, 16H), 10.69 (s, CH$_3$, 24H), 45.19 (s, C$_4$H$_2$N, 4H). Elem. Anal. for C$_{48}$H$_{64}$N$_5$Si$_4$Eu$_2$ calcd. C, 49.64; H, 7.29; N, 7.24; found: C, 49.64; H, 7.30; N, 7.24.

Preparation of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Sm$_2$(N(SiMe$_3$)$_2$)(thf): To a THF solution of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Sm$_2$(N(SiMe$_3$)$_2$)(thf) (0.036 g, 0.031 mmol) under nitrogen was added sodium amalgam (0.007 g, 0.304 mmol Na(s) in Hg) at room temperature and stirred overnight. After sodium amalgam was separated from the reaction solution solvent was removed under vacuum. Then the brown solid obtained was dissolved in pentane and treated with a drop of 12-crown-4 ether diluted in pentane. The solid that precipitated out was filtered and solvent was removed under vacuum (27 mg, 77%). 1H NMR (C$_6$D$_6$, 400 MHz): δ (ppm) -18.05 (s, THF, 4H), -7.71 (s, THF, 4H), -4.31 (s, CH$_3$, 12H), -2.60 (s, CH$_3$, 12H), 1.35 (s, CH$_2$, 8H), 2.55 (s, CH$_2$, 8H), 2.94 (s, CH$_3$, 18H), 12.96 (s, C$_4$H$_2$N, 4H). Elem. Anal. for C$_{48}$H$_{74}$N$_5$OSi$_2$Sm$_2$ calcd. C, 51.64; H, 6.97; N, 6.55; found: C, 52.02; H, 6.88; N, 6.43.

Preparation of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Eu$_2$(N(SiMe$_3$)$_2$)(thf): To a THF solution of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Eu$_2$(N(SiMe$_3$)$_2$) (0.035 g, 0.029 mmol) under nitrogen was added sodium amalgam (0.007 g, 0.304 mmol Na(s) in Hg) at room temperature and stirred overnight. After sodium amalgam was separated from the reaction solution solvent was removed under vacuum. Then the dark brown solid obtained was dissolved in pentane and treated with a drop of 12-crown-4 ether diluted in pentane. The solid that precipitated out was filtered and the filtrate was concentrated by slow evaporation to get needle like crystals (29 mg, 90%). NMR signals for this are extremely broad due to high paramagnetic nature of the complex. Elem. Anal. for C$_{48}$H$_{74}$N$_5$OSi$_2$Eu$_2$ calcd. C, 51.48; H, 6.95; N, 6.53; found: C, 51.28; H, 6.90; N, 6.42.

Preparation of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Sm$_4$(μ-O)$_2$}(thf) with silanol: To a THF solution of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Sm$_2$(N(SiMe$_3$)$_2$)(thf) (0.036 g, 0.034 mmol) under nitrogen was added a THF solution of silanol (0.003 g, 0.034 mmol) dropwise at room temperature. The reaction mixture was stirred for eight days at room temperature and the solvent was removed under vacuum. Then the greenish brown crude obtained was dissolved in pentane and concentrated by slow evaporation to get needle like crystals (18 mg, 58%). $^1$H NMR (C$_6$D$_6$, 400 MHz): δ (ppm) -20.70 (s, THF, 8H), -11.76 (s, THF, 8H), -9.52 (s, CH$_3$, 24H), -5.71 (s, CH$_2$, 16H), -5.09 (s, CH$_2$, 16H), 0.22 (s, CH$_3$, 24H), 4.28 (s, C$_4$H$_2$N, 8H), 10.79 (s, C$_4$H$_2$N, 8H). NMR signals for this are broad due to paramagnetic nature of the compound. Elem. Anal. for C$_{90}$H$_{112}$N$_6$O$_4$Sm$_4$ calcd. C, 51.90; H, 6.10; N, 6.05; found: C, 51.92; H, 6.15; N, 5.95.

Preparation of ($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$) Et$_8$ -calix[4]pyrrolyl|{Sm$_4$(μ-O)$_2$}(thf) with water: Nitrogen was bubbled through a few mL of DI water and a dilute solution of water in THF was prepared. One
equivalent of the above solution was added dropwise to a THF solution of \((\eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8\text{-calix}[4]\text{pyrrolyl})\text{Sm}_2\text{(N(SiMe}_3)_2\text{)(thf)})\) (0.036 g, 0.034 mmol) under nitrogen at 0 °C. The reaction mixture was stirred for an hour at 0 °C and the solvent was removed under vacuum. Then the greenish brown crude obtained was dissolved in pentane and concentrated by slow evaporation to get crystals (10 mg, 33%). ¹H NMR \((C_6D_6, 400 \text{ MHz})\): δ (ppm) -20.78 (s, THF, 8H), -11.52 (s, THF, 8H), -10.13 (s, CH₃, 24H), -5.76 (s, CH₂, 16H), -5.08 (s, CH₂, 16H), 0.22 (s, CH₃, 24H), 4.13 (s, C₄H₆N, 8H), 10.82 (s, C₄H₂N, 8H). NMR signals for this are broad due to paramagnetic nature of the compound. Elem. Anal. for C₈₀H₁₁₂N₈O₄Sm₄ calcld. C, 51.90; H, 6.10; N, 6.05; found: C, 51.97; H, 6.14; N, 6.00.

NMR

Calix[4]pyrrole:
¹H (400.144 MHz, CDCl₃)

Figure S1: Proton NMR spectrum of Calix[4]pyrrole in CDCl₃. †residual CHCl₃, ‡H₂O, *iPrOH
Sm(\text{N(SiMe}_3\text{)}_2\text{)}_3 (\mu-\text{Cl})\text{Li(thf)}_3:\n\text{\textsuperscript{3}H (400.144 MHz, C}_6\text{D}_6)\\

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{sm_complex.png}
\caption{Proton NMR spectrum of Sm(\text{N(SiMe}_3\text{)}_2\text{)}_3 (\mu-\text{Cl})\text{Li(thf)}_3 in C}_6\text{D}_6. \dagger\text{residual C}_6\text{H}_6}
\end{figure}
Eu(N(SiMe$_3$)$_2$)$_3$ ($\mu$-Cl)Li(thf)$_3$:
$^1$H (400.144 MHz, C$_6$D$_6$)

Figure S3: Proton NMR spectrum of Eu(N(SiMe$_3$)$_2$)$_3$ ($\mu$-Cl)Li(thf)$_3$ in C$_6$D$_6$, †residual C$_6$H$_6$, ‡grease, *pentane.
\((\eta^5: \eta^1: \eta^5: \eta^1)\text{Et}_8\text{-calix}[4]\text{pyrrolyl})\{\text{SmN(SiMe}_3)_2\}_2\):  
\(^1\text{H}\) (400.144 MHz, \text{C}_6\text{D}_6)

**Figure S4:** Proton NMR spectrum of \((\eta^5: \eta^1: \eta^5: \eta^1)\text{Et}_8\text{-calix}[4]\text{pyrrolyl})\{\text{SmN(SiMe}_3)_2\}_2\) in \text{C}_6\text{D}_6,  
†residual \text{C}_6\text{H}_6
\( \eta^5: \eta^1: \eta^5: \eta^1 \text{ Et}_8\text{-calix}[4]\text{pyrrolyl})\{\text{EuN(SiMe}_3\}_2\}_2: \)

\(^1H\ (400.144\text{ MHz, } C_6D_6)\)

**Figure S5:** Proton NMR spectrum of \( \eta^5: \eta^1: \eta^5: \eta^1 \text{ Et}_8\text{-calix}[4]\text{pyrrolyl})\{\text{EuN(SiMe}_3\}_2\}_2\) in \( C_6D_6\), †residual \( C_6H_6\), ‡grease, *thf.
\( \eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8\text{-calix}[4]pyrrolyl}\{\text{Sm}_2(\text{N(SiMe}_3)_2)(\text{thf})\}:

\(^1\text{H} (400.144 \text{ MHz, C}_{6}\text{D}_6)\)

Figure S6: Proton NMR spectrum of \( \eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8\text{-calix}[4]pyrrolyl}\{\text{Sm}_2(\text{N(SiMe}_3)_2)(\text{thf})\} in C_{6}\text{D}_6, \dagger \text{residual } C_{6}\text{H}_6, \ddagger \text{unidentified impurity, } ^* \text{pentane.}
(η⁵: η¹: η⁵: η¹ Et₈-calix[4]pyrrolyl)Sm₄(μ-O)₂(thf)₂
³H (400.144 MHz, C₆D₆)

**Figure S7:** Proton NMR spectrum of (η⁵: η¹: η⁵: η¹ Et₈-calix[4]pyrrolyl)Sm₄(μ-O)₂(thf)₂ in C₆D₆,
†residual C₆H₆, *pentane.
**Figure S8**: Proton NMR spectrum of the precipitate generated after addition of 12-crown-4, *pentane, ‡grease
Photophysical Data

Absorption and emission of Sm complexes

**Figure S9**: Absorption spectrum of $(\eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8 \text{-calix[4]pyrrolyl})\{\text{SmN(SiMe}_3\}_2\}_2$ in pentane

**Figure S10**: Emission spectrum of $(\eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8 \text{-calix[4]pyrrolyl})\{\text{SmN(SiMe}_3\}_2\}_2$ in pentane. Excitation at 320 nm
**Figure S11:** Absorption spectrum of \((\eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8 \text{-calix[4]pyrrolyl})\text{Sm}_2(\text{N(SiMe}_3)_2)(\text{thf})\) in pentane

**Figure S12:** Emission spectrum of \((\eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8 \text{-calix[4]pyrrolyl})\text{Sm}_2(\text{N(SiMe}_3)_2)(\text{thf})\) in pentane. Excitation at 327 nm
Electrochemical Data

Cyclic voltammogram of $\eta^5: \eta^1: \eta^5: \eta^1$ Et$_8$-calix[4]pyrrolyl){SmN(SiMe$_3$)$_2$)$_2$

Figure S13: One electron reduction of $\eta^5: \eta^1: \eta^5: \eta^1$ Et$_8$-calix[4]pyrrolyl){SmN(SiMe$_3$)$_2$)$_2$

Cyclic voltammogram of $\eta^5: \eta^1: \eta^5: \eta^1$ Et$_8$-calix[4]pyrrolyl){EuN(SiMe$_3$)$_2$)$_2$

Figure S14: One electron reduction of $\eta^5: \eta^1: \eta^5: \eta^1$ Et$_8$-calix[4]pyrrolyl){EuN(SiMe$_3$)$_2$)$_2$
**Figure S15**: Extended voltammogram of $(\eta^5: \eta^1: \eta^5: \eta^1 \text{Et}_8 \text{-calix[4]pyrrolyl})\{\text{SmN(SiMe}_3\}_2\}_2$

**Figure S16**: Extended voltammogram of $(\eta^5: \eta^4: \eta^5: \eta^1 \text{Et}_8 \text{-calix[4]pyrrolyl})\{\text{EuN(SiMe}_3\}_2\}_2$
Mass Spectrometry

Figure S17: Electronic ionization high resolution mass spectrum of the volatiles obtained from the reaction with silanol.
X-Ray Crystallography

($\eta^5$: $\eta^1$: $\eta^5$: $\eta^1$ Et$_8$-calix[4]pyrrolyl){SmN(SiMe$_3$)$_2$)$_2$ Complex.

Crystal data and structure refinement for NM132 (1).

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<td>Radiation</td>
<td>MoKα ($\lambda = 0.71073$)</td>
</tr>
<tr>
<td>2$\Theta$ range for data collection/°</td>
<td>4.576 to 60.552</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-16 ≤ h ≤ 16, -16 ≤ k ≤ 15, -29 ≤ l ≤ 30</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>65776</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>14653 [R$<em>{\text{int}}$ = 0.0552, R$</em>{\text{sigma}}$ = 0.0440]</td>
</tr>
<tr>
<td>Data/restraints/parameters</td>
<td>14653/0/561</td>
</tr>
<tr>
<td>Goodness-of-fit on F$^2$</td>
<td>1.116</td>
</tr>
<tr>
<td>Final R indexes [I≥2σ (I)]</td>
<td>R$_1$ = 0.0395, wR$_2$ = 0.0823</td>
</tr>
<tr>
<td>Final R indexes [all data]</td>
<td>R$_1$ = 0.0460, wR$_2$ = 0.0850</td>
</tr>
<tr>
<td>Largest diff. peak/hole / e Å$^{-3}$</td>
<td>2.71/-1.25</td>
</tr>
</tbody>
</table>
\( \eta^5 : \eta^1 : \eta^1 \): Et_8 -calix[4]pyrrolyl\{EuN(SiMe_3)_2\}_2 Complex.

**Crystal data and structure refinement for NM210b (1).**

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Identification code</td>
<td>NM210b (1)</td>
</tr>
<tr>
<td>Empirical formula</td>
<td>C_{48}H_{84}Eu_2N_6Si_4</td>
</tr>
<tr>
<td>Formula weight</td>
<td>1161.49</td>
</tr>
<tr>
<td>Temperature/K</td>
<td>100.01(10)</td>
</tr>
<tr>
<td>Crystal system</td>
<td>triclinic</td>
</tr>
<tr>
<td>Space group</td>
<td>P 1</td>
</tr>
<tr>
<td>a/Å</td>
<td>11.4426(5)</td>
</tr>
<tr>
<td>b/Å</td>
<td>11.6392(5)</td>
</tr>
<tr>
<td>c/Å</td>
<td>21.2371(9)</td>
</tr>
<tr>
<td>α/°</td>
<td>79.337(4)</td>
</tr>
<tr>
<td>β/°</td>
<td>79.682(4)</td>
</tr>
<tr>
<td>γ/°</td>
<td>77.099(4)</td>
</tr>
<tr>
<td>Volume/Å³</td>
<td>2681.2(2)</td>
</tr>
<tr>
<td>Z</td>
<td>2</td>
</tr>
<tr>
<td>( \rho_{\text{calc}} )/g/cm³</td>
<td>1.439</td>
</tr>
<tr>
<td>μ/mm⁻¹</td>
<td>2.444</td>
</tr>
<tr>
<td>F(000)</td>
<td>1192.0</td>
</tr>
<tr>
<td>Crystal size/mm³</td>
<td>0.154 × 0.046 × 0.038</td>
</tr>
<tr>
<td>Radiation</td>
<td>MoKα (λ = 0.71073)</td>
</tr>
<tr>
<td>2θ range for data collection/°</td>
<td>4.942 to 60.59</td>
</tr>
<tr>
<td>Index ranges</td>
<td>-14 ≤ h ≤ 16, -15 ≤ k ≤ 16, -29 ≤ l ≤ 29</td>
</tr>
<tr>
<td>Reflections collected</td>
<td>14131</td>
</tr>
<tr>
<td>Independent reflections</td>
<td>14131 [R_int = ?, R_\text{sigma} = 0.1208]</td>
</tr>
<tr>
<td>Data/restraints/parameters</td>
<td>14131/0/562</td>
</tr>
<tr>
<td>Goodness-of-fit on F²</td>
<td>1.029</td>
</tr>
<tr>
<td>Final R indexes [I&gt;=2σ (I)]</td>
<td>R₁ = 0.0585, wR₂ = 0.1056</td>
</tr>
<tr>
<td>Final R indexes [all data]</td>
<td>R₁ = 0.1028, wR₂ = 0.1237</td>
</tr>
<tr>
<td>Largest diff. peak/hole / e Å³</td>
<td>2.88/-2.00</td>
</tr>
</tbody>
</table>
Crystal data and structure refinement for NM100_2.

Identification code  NM100_2
Empirical formula  C_{46}H_{74}Eu_{2}N_{5}O_{2}Si_{2}
Formula weight  1073.20
Temperature/K  100.00(10)
Crystal system  monoclinic
Space group  C2/c
a/Å  13.7438(4)
b/Å  20.1221(6)
c/Å  17.6945(4)
α/°  90
β/°  90.855(2)
γ/°  90
Volume/Å³  4892.9(2)
Z  4
ρ_{calc}g/cm³  1.457
μ/mm⁻¹  2.626
F(000)  2188.0
Crystal size/mm³  0.306 × 0.249 × 0.045
Radiation  MoKα (λ = 0.71073)
2Θ range for data collection/°  4.658 to 60.48
Index ranges  -18 ≤ h ≤ 18, -27 ≤ k ≤ 28, -23 ≤ l ≤ 24
Reflections collected  30304
Independent reflections  6620 [R_{int} = 0.0589, R_{sigma} = 0.0486]
Data/restraints/parameters  6620/0/262
Goodness-of-fit on F²  1.097
Final R indexes [I>2σ(I)]  R₁ = 0.0407, wR₂ = 0.0939
Final R indexes [all data]  R₁ = 0.0515, wR₂ = 0.0998
Largest diff. peak/hole / e Å⁻³  3.20/-1.20
(η⁵: η¹: η⁵: η¹ Et₈-calix[4]pyrrolyl)Sm₄(μ-O)₂(thf)₂

Crystal data and structure refinement for NM229_twin1_hklf4.

Identification code: NM229_twin1_hklf4
Empirical formula: C₄₇H₆₄N₄O₂Sm₂
Formula weight: 1017.72
Temperature/K: 100.01(10)
Crystal system: monoclinic
Space group: C2/c

a/Å: 17.9470(8)
b/Å: 21.5868(6)
c/Å: 23.2649(9)
α/°: 90
β/°: 108.706(4)
γ/°: 90

Volume/Å³: 8537.1(6)
Z: 8
ρ(calc) g/cm³: 1.584
μ/mm⁻¹: 20.763
F(000): 4112.0

Crystal size/mm³: 0.159 × 0.122 × 0.08
Radiation: CuKα (λ = 1.54184)

2Θ range for data collection/°: 6.618 to 146.804
Index ranges: -22 ≤ h ≤ 21, -26 ≤ k ≤ 26, -28 ≤ l ≤ 28
Reflections collected: 9870
Independent reflections: 9870 [R(int) = ?, R(sigma) = 0.0443]
Data/restraints/parameters: 9870/102/546
Goodness-of-fit on F²: 1.116

Final R indexes [I>=2σ (I)]: R₁ = 0.0659, wR₂ = 0.1888
Final R indexes [all data]: R₁ = 0.0831, wR₂ = 0.2030
Largest diff. peak/hole / e Å⁻³: 1.58/-2.33
References: