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### EtOH/Ba(OH)<sub>2</sub> Triggers Self-Condensation of (E)-1,4-Diaryl-2-Butene-1,4-Diones to Cyclopentanol Derivatives

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## EtOH/Ba(OH)<sub>2</sub> Triggers Self-Condensation of (*E*)-1,4-Diaryl-2-Butene-1,4-Diones to Cyclopentanol Derivatives

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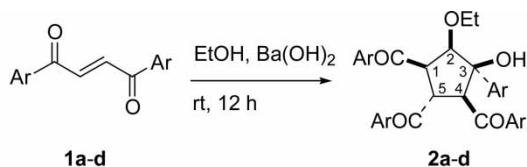
**Abstract:** Several stereo-defined penta-substituted cyclopentanols were synthesized from EtOH/Ba(OH)<sub>2</sub>-induced self-condensation of (*E*)-1,4-diaryl-2-butene-1,4-diones formed via domino pathways.

**Keywords:** Barium hydroxide, 1,4-diaryl-2-butene-1,4-diones, domino reactions

The  $\alpha,\beta$ -unsaturated carbonyl compounds have emerged as test systems to study the multicomponent condensation reactions involving the domino process.<sup>[1]</sup> In the domino reaction, the starting material undergoes a series of transformations in which the functional group generated in the first step serves to form the next bond in the subsequent step.<sup>[2]</sup> We have earlier reported that in the presence of barium hydroxide the enolate anion from cyclopentanone triggers multicomponent condensation of (*E*)-1,3-diaryl-2-propene-1-one (*trans*-chalcone) and phenyl vinyl ketone to furnish polycyclic products.<sup>[3]</sup> In continuation, EtOH/Ba(OH)<sub>2</sub>-mediated self-condensation of (*E*)-1,4-diphenyl-2-butene-1,4-dione (*trans*-dibenzoyl ethylene, *trans*-DBE) to the cyclopentanol derivative has been presented here (Scheme 1). Previously, Cabrera and coworkers have reported the formation of some

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**1a, 2a:** Ar = C<sub>6</sub>H<sub>5</sub>; **1b, 2b:** Ar = 4-Cl-C<sub>6</sub>H<sub>4</sub>; **1c, 2c:** Ar = 4-Br-C<sub>6</sub>H<sub>4</sub>; **1d, 2d:** Ar = 4-CH<sub>3</sub>-C<sub>6</sub>H<sub>4</sub>

*Scheme 1.*

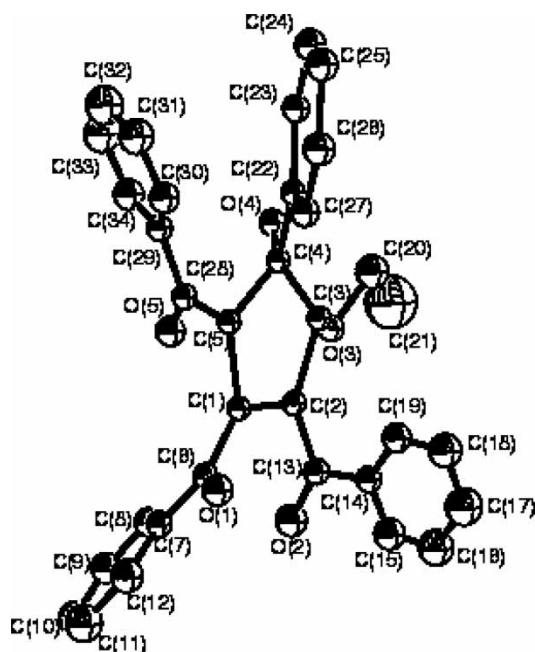
tetrasubstituted cyclopentanol derivatives from *trans*-DBE in the presence of the one-electron reducing agent samarium (II) iodide.<sup>[4]</sup> Similarly, Al-Arab and co-workers reported the formation of cyclohexanol derivatives from the base-mediated reaction of acetophenone with DBE.<sup>[5]</sup>

While attempting the condensation of cyclohexanone and *trans*-DBE in the presence of activated Ba(OH)<sub>2</sub>, we noticed the formation of a polar product (6%) apart from the expected conjugate addition product (63%).<sup>[6]</sup> When the condensation reaction was conducted in the absence of cyclohexanone, the polar product was formed in 67% yield (Scheme 1). Its <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra revealed the presence of least three diastereomeric cyclopentanols in the ratio of 61:28:11. The ratio of the diastereomers was determined on the integration of the cluster of signals located at  $\delta$  5.7 ppm in the <sup>1</sup>H NMR spectrum assignable to 5-H and the cluster of signals located at  $\delta$  90 ppm assignable to C-3. The major diastereomer **2a** was isolated in the pure form through fractional crystallization from the column fractions during column chromatography.

The structure and stereochemistry of the cyclopentanol derivative **2a**, obtained as a white crystalline solid (mp 184–186°C) was deduced on the basis of analytical and spectral data (IR, <sup>1</sup>H NMR, <sup>13</sup>C NMR, DEPT, COSY, and NOESY). Final confirmation of the proposed structure of **2a** was obtained by X-ray analysis (Figure 1) on the sample recrystallized from 10% EtOAc in hexanes. (See the Appendix.)

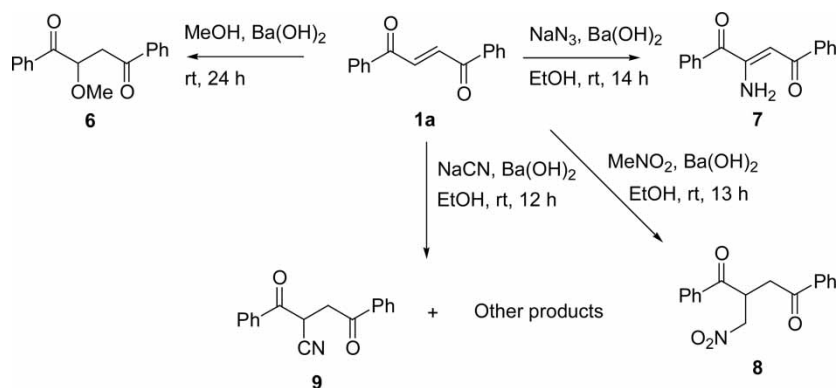
Having established the structure of the cyclopentanol derivative **2a**, the generality of the reaction was next studied by subjecting different *trans*-DBE derivatives with electron-withdrawing (Cl, **1b**, Br, **1c**) and electron-donating (Me, **1d**) groups located on the *para* position of the aryl rings to self-condensation. The reactions furnished cyclopentanol products **2b–d** in 64–72% yield. Although pure cyclopentanols **2b** and **2c** could be isolated from the fractional crystallization their respective mixture of isomers, **2d** could not be obtained as a single product by different chromatographic or recrystallization techniques.

Surprisingly, the present three-component condensation reaction involving two units of *trans*-DBE **1a** and the alkoxide anion did not take place with MeOH/Ba(OH)<sub>2</sub>. Only conjugate addition product **6** was isolated from the reaction in moderate yield (34%; Scheme 2).<sup>[7]</sup>



**Figure 1.** Crystal structure of [(1*R*,2*S*,3*S*,4*S*,5*R*)-4,5-dibenzoyl-2-ethoxy-3-hydroxy-3-phenylcyclopentyl](phenyl)methanone (**2a**, with crystal structure numbering; hydrogens were omitted for clarity).

The Ba(OH)<sub>2</sub>-mediated three-component domino reaction of **1a** was attempted with azide, cyanide, and nitromethane anions. Although the nitromethane added to *trans*-DBE **1a** in conjugate mode to furnish known diketone **8**<sup>[8]</sup> (54%, Scheme 2), the product from the conjugate addition of



**Scheme 2.**

azide anion underwent further reduction to yield known 2-amino-1,4-diphenylbut-2-ene-1,4-dione **7**<sup>[9]</sup> (58%, Scheme 2). We did not notice any cyclopentanol products from these reactions. The reaction of **1a** with cyanide anion in the presence of activated Ba(OH)<sub>2</sub> furnished a complex mixture along with conjugate addition product **9**.<sup>[10]</sup> Surprisingly there was no Ba(OH)<sub>2</sub>-mediated reaction of *trans*-DBE **1a** with thiophenol or heptanethiol.

In short, we have shown that EtOH/Ba(OH)<sub>2</sub> induces self-condensation of *trans*-DBE and its derivatives to furnish penta-substituted cyclopentanol derivatives with defined stereochemistry. It appears that the cyclopentanol formation is induced only in the presence of EtOH/Ba(OH)<sub>2</sub>.

## EXPERIMENTAL

### General

The progress of all the reactions was monitored by TLC (TLC silica gel; Qualigens or TLC alumina: SRL, India) using hexanes/ethyl acetate mixture as an eluent. Column chromatography was accomplished on silica gel (100–200 mesh, Acme Synthetic Chemicals) using hexanes/ethyl acetate mixture as an eluent. The IR spectra were recorded as a solution in KBr or neat using ABB Bomem MB-104 FT-IR spectrometer. The <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded in CDCl<sub>3</sub> or CDCl<sub>3</sub>·CCl<sub>4</sub> (1:1) using JEOL 400-MHz or Varian 300-MHz NMR spectrometer. The mass spectra were recorded on Finnigan MAT 8230 or JEOL DX-303 mass spectrometer. The elemental analysis was carried out on a Elementar vario EL (Germany) apparatus. The X-ray diffraction data was generated on SMART (Siemens) diffractometer. The *trans*-DBE and derivatives (**1a–d**) were prepared according to literature procedure.<sup>[11]</sup> The activated Ba(OH)<sub>2</sub> was prepared by heating commercial Ba(OH)<sub>2</sub>·8H<sub>2</sub>O at 200°C for 3 h in a furnace and then stored in a desiccator.<sup>[12]</sup>

*Synthesis of (±) [(1R,2S,3S,4S,5R)-4,5-dibenzoyl-2-ethoxy-3-hydroxy-3-phenylcyclopentyl](phenyl)methanone 2a; representative procedure:* To a stirred mixture of activated Ba(OH)<sub>2</sub> (73 mg, 0.43 mmol) in 10 mL of absolute ethanol, *trans*-DBE **1a** (500 mg, 2.12 mmol) was added in four equal portions and allowed to stir at room temperature for 12 h. After complete disappearance of **1a** (TLC), the suspended solid particles were filtered through a celite pad. The clear filtrate was concentrated to 3 mL and was diluted with 30 mL of dichloromethane and then poured over ice-cooled water. The organic layer was separated, washed again with water (3 × 20 mL) and brine solution (2 × 10 mL), dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, and concentrated. The crude product was subjected to column

chromatography (silica gel, 100–200 mesh) and eluted with 10% EtOAc–hexanes to give **2a** (63 mg, 12%) as a crystalline solid. Mp 184–186°C;  $R_f$  = 0.43 (10% EtOAc–hexanes); IR (KBr) 3466, 1683, 1596, 1449, 1258, 709, 691 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  0.68 (t,  $J$  = 7.2 Hz, 3H), 2.85 (m, 1H), 3.09 (m, 1H), 4.20 (s, 1H), 4.49 (d,  $J$  = 7.8 Hz, 1H), 4.55 (d,  $J$  = 10.5 Hz, 1H), 4.78 (dd,  $J$  = 7.8, 10.5 Hz, 1H), 5.76 (t,  $J$  = 10.2 Hz, 1H), 7.05 (t,  $J$  = 7.8 Hz, 2H), 7.21–7.54 (m, 14H), 7.89 (d,  $J$  = 8.7 Hz, 2H), 8.26 (d,  $J$  = 8.7 Hz, 2H) ppm; <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  14.6, 46.1, 54.8, 62.3, 68.9, 82.7, 89.7, 124.8, 127.3, 127.8, 128.0, 128.3, 128.5 (2C), 128.6, 129.4, 132.4, 133.1, 133.6, 136.2, 137.2, 137.3, 144.7, 196.9, 197.5, 202.5 ppm; FAB-MS 519 (M<sup>+</sup> + 1); Anal. calcd. for C<sub>34</sub>H<sub>30</sub>O<sub>5</sub>: C, 78.74; H, 5.83. Found: C, 78.77; H, 5.87.

(±) (4-Chlorophenyl)[(1*R*,2*S*,3*S*,4*S*,5*R*)-4,5-di(4-chlorobenzoyl)-3-(4-chlorophenyl)-2-ethoxy-3-hydroxycyclopentyl]methanone **2b**: Mp 168–170°C;  $R_f$  = 0.36 (10% EtOAc–hexanes); IR (KBr) 3466, 2975, 2929, 1684, 1588, 1489, 1401, 1247, 1093, 1006, 840 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>/CCl<sub>4</sub>)  $\delta$  0.76 (t,  $J$  = 6.9 Hz, 3H), 2.81–2.91 (m, 1H), 3.06–3.16 (m, 1H), 4.04 (s, 1H), 4.30 (d,  $J$  = 10.8 Hz, 1H), 4.35 (d,  $J$  = 9.9 Hz, 1H), 4.60 (t,  $J$  = 7.5 Hz, 1H), 5.56 (t,  $J$  = 10.2 Hz, 1H), 7.07 (d,  $J$  = 8.4 Hz, 2H), 7.24 (d,  $J$  = 8.1 Hz, 2H), 7.31 (d,  $J$  = 8.1 Hz, 2H), 7.39–7.50 (m, 6H), 7.81 (d,  $J$  = 8.4 Hz, 2H), 8.16 (d,  $J$  = 8.4 Hz, 2H) ppm; <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>/CCl<sub>4</sub>)  $\delta$  14.98, 45.86, 54.60, 62.44, 69.04, 82.53, 89.73, 126.38, 128.29, 129.00, 129.15, 129.51, 129.77, 130.21, 131.15, 133.87, 134.29, 135.50, 135.61, 139.33, 139.87, 140.63, 143.37, 194.65, 195.09, 200.72 ppm; LRMS M<sup>+</sup> did not appear, 639 (7), 517 (19), 515 (30), 497 (18), 468 (29), 447 (15), 445 (35), 443 (27), 428 (20), 364 (31), 348 (24), 348 (18), 307 (69), 305 (100), 304 (74), 181 (12), 183 (7), 113 (27), 111 (78), 89 (19), 75 (55), 69 (18); Anal. calcd. for C<sub>34</sub>H<sub>24</sub>Cl<sub>4</sub>O<sub>5</sub>: C, 62.24; H, 3.99. Found: C, 62.21; H, 4.02.

(±) (4-Bromophenyl)[(1*R*,2*S*,3*S*,4*S*,5*R*)-4,5-di(4-bromobenzoyl)-3-(4-bromophenyl)-2-ethoxy-3-hydroxycyclopentyl]methanone **2c**: Mp 176–178°C;  $R_f$  = 0.38 (10% EtOAc–hexanes); IR (KBr) 3496, 2971, 2927, 1671, 1585, 1486, 1398, 1234, 1096, 1073, 1003, 980, 834 cm<sup>-1</sup>; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>/CCl<sub>4</sub>)  $\delta$  0.71 (t,  $J$  = 14.1 Hz, 3H), 2.82–2.92 (m, 1H), 3.03–3.13 (m, 1H), 4.07 (s, 1H), 4.36 (d,  $J$  = 10.8 Hz, 1H), 4.41 (d,  $J$  = 7.8 Hz, 1H), 4.62 (t,  $J$  = 18.0 Hz, 1H), 5.61 (t,  $J$  = 20.4 Hz, 1H), 7.17 (d,  $J$  = 7.5 Hz, 2H), 7.25 (d,  $J$  = 8.1 Hz, 2H), 7.38 (d,  $J$  = 8.1 Hz, 2H), 7.46 (d,  $J$  = 8.7 Hz, 2H), 7.60 (dd,  $J$  = 6.3, 4.8 Hz, 4H), 7.74 (d,  $J$  = 8.4 Hz, 2H), 8.11 (d,  $J$  = 8.4 Hz, 2H) ppm; <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>/CCl<sub>4</sub>)  $\delta$  14.64, 45.81, 54.56, 62.29, 69.14, 82.40, 89.32, 121.74, 126.60, 127.94 (2C), 128.55 (2C), 129.44, 129.64, 130.97, 131.23, 131.84, 131.96, 132.11, 134.71, 135.91, 143.41, 195.42, 196.10, 201.24 ppm; LRMS M<sup>+</sup> peak did not

appear, 338 (20), 228 (100), 216 (12), 117 (10), 117 (38), 90 (40), 57 (42); Anal. calcd. for  $C_{34}H_{24}Br_4O_5$ : C, 48.94; H, 3.14. Found: C, 48.91; H, 3.19.

( $\pm$ ) [2-Ethoxy-3-hydroxy-4,5-di(4-methylbenzoyl)-3-(4-methylphenyl)cyclopentyl](4-methylphenyl)methanone **2d**: Mp 200–202°C;  $R_f$  = 0.40 (10% EtOAc–hexanes); IR (KBr) 3463, 2923, 2853, 1670, 1606, 1573, 1463, 1377, 1240, 1182, 1104, 1006, 828, 721  $cm^{-1}$ ;  $^1H$  NMR (300 MHz,  $CDCl_3/CCl_4$ , 1:1)  $\delta$  0.76 (t,  $J$  = 7.5 Hz, 3H), 2.28 (s, 3H), 2.31 (s, 3H), 2.37 (s, 3H), 2.44 (s, 3H), 2.86–2.91 (m, 1H), 3.09–3.17 (m, 1H), 4.15 (s, 1H), 4.31 (d,  $J$  = 7.2 Hz, 1H), 4.40 (d,  $J$  = 7.8 Hz, 1H), 4.58 (dd,  $J$  = 5.7, 13.8 Hz, 1H), 5.59 (t,  $J$  = 10.2 Hz, 1H), 6.81–6.86 (m, 2H), 7.07–7.29 (m, 4H), 7.42–7.53 (m, 2H), 7.75–7.85 (m, 4H), 7.95 (d,  $J$  = 8.40 Hz, 2H), 8.08 (d,  $J$  = 8.40 Hz, 2H) ppm;  $^{13}C$  NMR (75 MHz,  $CDCl_3/CCl_4$ , 1:1)  $\delta$  15.38, 21.09, 21.25 (2C), 21.79, 47.05, 56.24, 62.71, 68.66, 83.00, 90.42, 125.03, 126.02, 128.34, 128.47, 128.95, 129.16, 129.29, 129.84, 132.98, 134.08, 135.07, 136.20, 138.28, 139.90, 142.85, 145.10, 196.01, 199.98, 201.71 ppm; FAB-MS 575 ( $M^+$  + 1); Anal. calcd for  $C_{38}H_{38}O_5$ : C, 79.44; H, 6.66. Found: C, 79.42; H, 6.69.

2-Methoxy-1,4-diphenyl-1,4-butanedione **6**:  $R_f$  = 0.38 (10% EtOAc–hexanes); IR (KBr) 763, 841, 1005, 1250, 1401, 1489, 1588, 1683, 2929, 2977, 3070  $cm^{-1}$ ;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  1.83 (t,  $J$  = 6.35 Hz, 1H), 2.31 (t,  $J$  = 6.35 Hz, 1H), 3.41 (s, 3H), 5.39 (dd,  $J$  = 4.39, 11.72 Hz, 1H), 7.41–7.59 (m, 6H), 7.95 (d,  $J$  = 7.81 Hz, 2H), 8.04 (d,  $J$  = 7.81 Hz, 2H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  40.62, 57.80, 78.59, 127.58, 128.21, 128.32, 128.35, 133.42, 133.53, 135.06, 136.53, 196.72, 199.60 ppm.

(Z)-2-Amino-1,4-diphenyl-2-butene-1,4-dione **7**: Mp 132–134°C (lit.<sup>[9]</sup> 133–134°C);  $R_f$  = 0.48 (10% EtOAc–hexanes); IR (KBr) 3378, 3269, 1664, 1613, 1593, 1527, 1445, 1275, 1239, 1172, 768, 725  $cm^{-1}$ ;  $^1H$  NMR (300 MHz,  $CDCl_3$ )  $\delta$  6.23 (s, 1H), 7.39–7.56 (m, 4H), 7.66 (t,  $J$  = 14.86 Hz, 2H), 7.82 (t,  $J$  = 8.61 Hz, 2H), 7.88 (t,  $J$  = 8.66 Hz, 2H), 9.5 (br s, 2H) ppm;  $^{13}C$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  97.63, 127.37, 128.51, 128.58, 129.81, 131.96, 133.41, 135.42, 139.28, 152.57, 191.75, 193.63 ppm.

2-(Nitromethyl)-1,4-diphenyl-1,4-butanedione **8**: Mp 93–94°C (lit.<sup>[8]</sup> 95°C);  $R_f$  = 0.30 (10% EtOAc–hexanes); IR (KBr) 3066, 2978, 1679, 1552, 1447, 1416, 1386, 1253, 1216, 984, 961, 757, 691  $cm^{-1}$ ;  $^1H$  NMR (300 MHz,  $CDCl_3$ )  $\delta$  3.25 (dd,  $J$  = 6.91, 7.40 Hz, 1H), 3.55 (dd,  $J$  = 4.76, 1.58 Hz, 1H), 4.60–4.68 (m, 1H), 4.88–4.96 (m, 2H), 7.44–7.63 (m, 6H), 7.91 (d,  $J$  = 8.69 Hz, 2H), 8.04 (d,  $J$  = 8.74 Hz, 2H) ppm;  $^{13}C$  NMR (75 MHz,  $CDCl_3$ )  $\delta$  37.99, 39.5, 74.62, 128.12, 128.66, 128.84, 129.06, 133.95, 134.0, 134.91, 135.69, 195.81, 198.46 ppm.



## APPENDIX

Crystal structure analysis was carried out by Professor H.-K. Fun, Universiti Sains Malaysia, Malaysia. Crystal data and experimental crystallographic details for **2a**; Cambridge Crystallographic Data Center, deposition no. CCDC 226255.

|  |  |
|--|--|
| Empirical formula                            | C <sub>34</sub> H <sub>30</sub> O <sub>5</sub>   |
| Formula weight                               | 518.58   |
| Temperature                                  | 293(2) K   |
| Wavelength                                   | 0.71073  |
| Crystal system, space group                  | Triclinic, P-1   |
| Unit cell dimensions                         | a = 11.0603(4) Å    α = 108.079(1) deg.<br>b = 11.2631(4) Å    β = 91.207(1) deg.<br>c = 12.4962(4) Å    γ = 106.913(1) deg. |
| Volume                                       | 1405.18(8) Å <sup>3</sup>  |
| z, calculated density                        | 2, 1.226 Mg/m <sup>3</sup>   |
| Absorption coefficient                       | 0.081 mm <sup>-1</sup>   |
| F(000)                                       | 548  |
| Crystal size                                 | 0.40 × 0.32 × 0.24 mm  |
| Theta range for data collection              | 1.73 to 29.37 deg.   |
| Limiting indices                             | -13 ≤ h ≤ 15, -9 ≤ k ≤ 15,<br>-17 ≤ l ≤ 16   |
| Reflection collected/unique                  | 10071/6641 [R(int) = 0.0499]   |
| Completeness to theta = 29.37                | 85.7%  |
| Absorption correction                        | None   |
| Refinement method                            | Full-matrix least-squares on F <sup>2</sup>  |
| Data/restraints/parameters                   | 6641/0/352   |
| Goodness of fit                              | 0.882  |
| Final R indices                              | R1 = 0.0657, wR2 = 0.1516  |
| [I > 2 sigma(I)]                             |  |
| R indices (all data)                         | R1 = 0.1400, wR2 = 0.1803  |
| Largest diff. peak and hole/eA <sup>-3</sup> | 0.272 and -0.287   |

## ACKNOWLEDGMENT

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