# A Practical and Efficient Synthesis of 5'-Substituted *m*-Terphenyls

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**Abstract:** Outlined is a simple, convenient, scalable procedure for the synthesis of a variety of 5'-substituted *m*-terphenyl compounds from commercially available materials. A series of novel compounds were generated based on the *m*-terphenyl scaffold. In addition we present a full and complete characterization, including the crystal structure, of 3,5-diphenylbenzaldehyde (1,1':3',1'')-terphenyl-5'-carbaldehyde).

Key words: imine, Kumada, pyridine, heterocycle, biaryl

The *m*-terphenyl unit exists in numerous natural products,<sup>1</sup> pharmaceuticals,<sup>2</sup> and plays an important role as a sterically crowded protecting group,<sup>3</sup> a ligand for asymmetric catalysis,<sup>4</sup> and as a building block in material science<sup>5</sup> due to its unique molecular architecture and potential for interesting electronics. Here, we outline an expeditious synthetic route to an unexplored class of new *m*terphenyl compounds. The synthetic work described in Scheme 1 can be realized using 2,4,6-tribromoaniline as the starting material. Sandmeyer chemistry converts 2,4,6-tribromoaniline into 1,3,5-tribromo-2-iodobenzene (1). When treated with phenylmagnesium bromide, 1 readily forms a reactive benzyne species that affords 1-bromo-3,5-diphenylbenzene (2) as outlined by Hart.<sup>6</sup> The brominated 1-position of the 3,5-diphenylbenzene is left available for conversion to 3,5-diphenylphenylmagnesium bromide (3), which can then be used in a variety of reactions to afford the desired products 4. Grignard 3 is readily prepared by refluxing 2 in tetrahydrofuran with a slight excess of magnesium metal initiated by dibromoethane. With 3 in hand, a series of reactions were employed to give formyl- (5), acetyl- (6), pyridyl- (7), and pyrimidyl- (8) substituted products at the 1-position (Figure 1). In addition, a number of Schiff bases 9-13 were prepared from 5 and their characterization is reported.



Scheme 1 Reaction conditions: (i) NaNO<sub>2</sub>, concd HCl, 0 °C, 30 min; (ii) KI, r.t., 1 h; (iii) PhMgBr, THF, reflux, 1 h, then r.t. 12 h; (iv) Mg, THF, reflux, 1 h; (v) various reagents.



#### Figure 1

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Olah and co-workers<sup>7</sup> have outlined a series of formylation reactions using *N*-formylmorpholine as a formylating agent. We employed this chemistry in, what is to date, the most expeditious route to compound **5**. A prior reported synthesis of **5** employed a lithium aluminum hydride reduction of the corresponding carboxylic acid, followed by manganese dioxide oxidation to the aldehyde, which gave modest yields (43% from the carboxylic acid).<sup>8</sup> The formylation outlined here gives **5** (Scheme 2) in one step from **2** in good yield (73%). In addition full characterization of the aldehyde is described and we report <sup>1</sup>H NMR data different from that previously reported by Ohemeng and Nguyen. The X-ray diffraction analysis of the aldehyde has confirmed its structure and our new assignments.





X-ray quality crystals of 3,5-diphenylbenzaldehyde (5) were grown from a concentrated dichloromethane solution by slow diffusion of hexane. The crystal structure is composed of discrete molecules separated by van der Waals forces only. There is no apparent  $\pi$ -stacking of any phenyl rings. A view of the molecule showing the labeling scheme is seen in Figure 2. The molecule adopts a nonplanar arrangement of the meta-substituted phenyl rings and the angles of phenyl groups to the benzaldehyde ring are 37.5(3)° (C8-C13 ring) and 37.8(3)° (C14-C19 ring). A DFT study using Gaussian 03<sup>9</sup> was performed on 3,5diphenylbenzaldehyde with geometry optimization and no constraints. The phenyl rings were shown to be nonplanar at 37.1° (C8-C13 ring) and 38.0° (C14-C19 ring). An energy profile was determined using the Spartan  $04^{10}$ semi-empirical molecular modeling package for rotation about one phenyl-benzaldehyde bond; a minimum was



Figure 2 ORTEP of 3,5-diphenylbenzaldehyde

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seen at approximately 38°. Both results are in agreement with the X-ray diffraction results and indicate that the nonplanarity is not solely a result of crystal packing. The structure is consistent with the IR spectrum and with our new NMR peak assignments. Selected bonds and angles are given in Table 1.

 Table 1
 Selected Bond Lengths and Angles for Compound 5

Bonds	Value <sup>a</sup>
01–C1	1.204(2) Å
C1–C2	1.483(3) Å
C6–C8	1.479(6) Å
C4–C14	1.498(6) Å
C <sub>aryl</sub> -C <sub>aryl</sub> <sup>b</sup>	1.39(2) Å
01–C1–C2	124.8(2)°
C1C2C3	118.6(2)°
C1C2C7	120.3(3)°
$C_{aryl} - C_{aryl} - C_{aryl}^{b}$	120(1)°

<sup>a</sup> Estimated standard deviation in the least significant figure are given in parentheses.

<sup>b</sup> Average.

3,5-Diphenylacetophenone (6) (Scheme 3) was prepared by the reaction of 3 with acetic anhydride. This reaction gives modest yields (59%) but provides a quick and easy route to the acetylated terphenyl.





We employed an inverse addition technique outlined by Leazer et al. whereby **3** was slowly added to a large excess of acetic anhydride.<sup>11</sup> When these measures are taken, the bisubstituted product expected from the direct addition of acetic anhydride to **3** is not produced.

A Kumada-like nickel-catalyzed coupling (Scheme 4) was used to prepare 2-(3,5-diphenylphenyl)pyridine (7). Herrmann and co-workers reported the success of several high yield coupling reactions of halogenated aromatic heterocycles to aryl Grignards using imidizolium ligands in the presence of bis(acetylacetonato)nickel(II).<sup>12</sup> Here, **3** is coupled to 2-chloropyridine in the presence of bis(acetyl-acetonato)nickel(II) and 1,3-bis(2,4,6-trimethylphenyl)imidizolium to afford **7** in 82% yield. The *m*-terphenylpyrimidine **8** was prepared by an analogous route.



#### Scheme 4

We have outlined a convenient, scalable procedure for the generation of a new set of compounds based on the terphenyl scaffold. We have used the terphenyl Grignard as a useful intermediate, coupling the scaffold to a variety of electrophiles, demonstrating the utility of the approach. Molecules possessing a terphenyl scaffold possess unique architecture and may find use in a variety of applications including catalysis and material science.

<sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were obtained using a Varian Gemini 2020 200 MHz spectrometer or Varian Mercury 300 MHz with CDCl<sub>3</sub> as the solvent and TMS as an internal standard unless otherwise noted. GC-MS analysis was performed on an Agilent 6890N gas chromatograph equipped with an N10149 autosampler coupled to an Agilent 5973 mass spectrometer. UV-Visible absorption studies were completed using an HP 8453 spectrophotometer. All reactions were run under an atmosphere of dry N<sub>2</sub> except in the preparation of 1,3,5-tribromo-2-iodobenzene (1). Anhydrous MgSO<sub>4</sub> or Na<sub>2</sub>SO<sub>4</sub> was used as the drying agent in all reactions. All reagents and solvents were purchased from Sigma-Aldrich and were used without additional purification. Elemental analyses were performed by Atlantic Microlab Inc. (Norcross, GA).

# 1,3,5-Tribromo-2-iodobenzene (1)

Prepared similar to the method described by Hart and co-workers.<sup>6</sup> A soln of NaNO<sub>2</sub> (3.28 g, 47.5 mmol) in H<sub>2</sub>O (15 mL) was added dropwise to a mechanically stirred slurry of 2,4,6-tribromoaniline (15 g, 45.5 mmol) in concd HCl (23 mL) at 0 °C. Stirring was continued for 30 min after complete addition of NaNO<sub>2</sub>. The diazonium salt was slowly transferred through a glass wool filter to a soln of KI (75.53 g, 0.455 mol) in H<sub>2</sub>O (114 mL). The soln was stirred vigorously with both magnetic and mechanical stirring at r.t. for 1 h. CH<sub>2</sub>Cl<sub>2</sub> (200 mL) and 0.5 M Na<sub>2</sub>SO<sub>3</sub> (20 mL) were added successively. The aqueous layer was separated and washed with CH<sub>2</sub>Cl<sub>2</sub>. The combined organic layers were washed with 10% NaOH and sat. NaCl and dried. A red solid was isolated upon solvent removal. Recrystallization (25% hexane–CH<sub>2</sub>Cl<sub>2</sub>) afforded pure product (14.2 g, 71%); mp 101–103 °C (Lit.<sup>13</sup> 104–105);  $R_f = 0.61$  (10% CH<sub>2</sub>Cl<sub>2</sub>–hexane).

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.71 (s, 2 H).

# 1-Bromo-3,5-diphenylbenzene (2)

Prepared by a modification of the procedure described by Hart and co-workers.<sup>6</sup> A soln of **1** (10.0 g, 22.7 mmol) in anhyd (200 mL) was added dropwise over 1 h to a stirred, refluxing soln of 1 M PhMgBr in THF (227 mL). Reflux was continued for 1 h after complete addition of **1**. Stirring was continued at r.t. for 12 h. Excess Ph-MgBr was quenched with sat. NH<sub>4</sub>Cl. The aqueous layer was washed with Et<sub>2</sub>O. The combined organic layers were washed with sat. NaCl and dried. A white solid was isolated after solvent removal. Recrystallization (40% CH<sub>2</sub>Cl<sub>2</sub>–hexane) gave pure product (5.41 g, 77%); mp 103–105 °C (Lit.<sup>13</sup> 107.5–109 °C);  $R_f = 0.57$  (30% CH<sub>2</sub>Cl<sub>2</sub>–hexane).

# 3,5-Diphenylphenylmagnesium Bromide (3)

A soln of 2 (3.09 g, 10 mmol) in anhyd THF (10 mL) was added dropwise to a stirred, refluxing mixture of Mg turnings (0.27 g, 11 mmol) in anhyd THF (10 mL). 1,2-Dibromoethane (2 drops) was added to initiate the reaction. Reflux was continued for 1 h after complete addition of 2.

### **3,5-Diphenylbenzaldehyde** (5)

A soln of *N*-formylmorpholine (10 mmol) in anhyd THF (10 mL) was slowly transferred via cannula to the stirring soln of **3** and the mixture was stirred at r.t. for 30 min. Excess Grignard was quenched with 3 M HCl to a pH of 2. The aqueous layer was washed with Et<sub>2</sub>O and the combined organic layers were washed with sat. NaHCO<sub>3</sub> and sat. NaCl. The product was purified by flash chromatography (silica gel 200–425 mesh, 30% CH<sub>2</sub>Cl<sub>2</sub>–hexane) and recrystallized (30% CH<sub>2</sub>Cl<sub>2</sub>–hexane) to give pure product (1.88 g, 73%); mp 101–102 °C;  $R_f = 0.36$  (50% CH<sub>2</sub>Cl<sub>2</sub>–hexane).

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>,): δ = 7.42–7.55 (m, 6 H), 7.66–7.71 (m, 4 H), 8.01 (s, 3 H), 10.15 (s, 1 H).

 $^{13}\text{C}$  NMR (50 MHz, CDCl<sub>3</sub>):  $\delta$  = 192.52, 142.97, 139.93, 137.65, 132.04, 129.26, 128.35, 127.46, 127.38.

UV (MeOH):  $\lambda_{max}$  ( $\epsilon$ ) = 202 (sh, 39200), 205 (41600), 248 nm (37200).

Anal. Calcd for  $C_{19}H_{14}O$ : C, 88.34; H, 5.46. Found: C, 88.05; H, 5.47.

# 3',5'-Diphenylacetophenone (6)

The *m*-terphenyl Grignard **3** (3.24 mmol) was slowly transferred via cannula to a stirred soln of Ac<sub>2</sub>O (6 mL, 63.5 mmol) at 0 °C. The mixture was stirred at 0 °C for 2 h and the reaction was quenched with H<sub>2</sub>O. The aqueous layer was washed with Et<sub>2</sub>O and the combined organic layers were washed with 10% NaOH and sat. NaCl and dried. The product was purified by flash chromatography (silica gel, 200–425 mesh, 20% CH<sub>2</sub>Cl<sub>2</sub>–hexane) and recrystallized (20% CH<sub>2</sub>Cl<sub>2</sub>–hexane) to give pure product (0.524 g, 59%); mp 99–101 °C;  $R_f = 0.16$  (50% CH<sub>2</sub>Cl<sub>2</sub>–hexanes).

IR (neat): 1678 cm<sup>-1</sup> (C=O).

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>): δ = 2.71 (s, 3 H), 7.41–7.54 (m, 6 H), 7.66–7.70 (m, 4 H), 7.99–8.01 (t, 1 H), 8.15–8.16 (d, 2 H).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 27.12, 126.14, 127.52, 128.16, 129.19, 130.78, 138.40, 140.43, 142.55, 198.26.

UV (MeOH):  $\lambda_{max}$  ( $\epsilon$ ) = 204 (946), 249 nm (794).

Anal. Calcd for  $C_{20}H_{16}O$ : C, 88.20; H, 5.92. Found: C, 87.94; H, 5.98.

# 2-(3,5-Diphenylphenyl)pyridine (7)

The *m*-terphenyl Grignard (4.17 mmol) was added dropwise by cannula transfer to a stirred soln of 2-chloropyridine (2.84 mmol), Ni(acac)<sub>2</sub> (0.085 mmol, 3 mol%), and 1,3-bis(2,4,6-trimethylphenyl)imidizolium (0.085 mmol, 3 mol%) in anhyd THF (3 mL) at r.t. and the mixture was allowed to stir. After 18 h the reaction was quenched with 0.5 M HCl. The aqueous layer was washed with Et<sub>2</sub>O and the combined organic layers were washed with sat. NaCl and dried. The product was purified by flash chromatography (silica gel, 200–425 mesh, 25% CH<sub>2</sub>Cl<sub>2</sub>–hexane) and recrystallized (40% CH<sub>2</sub>Cl<sub>2</sub>–hexane) to give pure product (0.7150 g, 82%); mp 137–139 °C;  $R_f = 0.46$  (100% CH<sub>2</sub>Cl<sub>2</sub>).

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.25–7.32 (m, 1 H), 7.39–7.53 (m, 6 H), 7.70–7.87 (m, 7 H), 8.20–8.21 (d, 2 H), 8.74–8.77 (m, 1 H).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 121.06, 122.59, 125.08, 126.97, 127.61, 127.78, 129.03, 137.05, 140.69, 141.26, 142.56, 149.99, 157.57.

UV (MeOH):  $\lambda_{max}$  ( $\epsilon$ ) = 200 (40800), 202 (sh, 37400), 250 nm (35200).

Anal. Calcd for  $C_{23}H_{17}N$ : C, 89.87; H, 5.57; N, 4.56. Found: C, 89.90; H, 5.43; N, 4.43.

#### 2-(3,5-Diphenylphenyl)pyrimidine (8)

The *m*-terphenyl Grignard **3** (4.17 mmol) was added dropwise to a stirred soln of 2-chloropyrimidine (2.84 mmol), Ni(acac)<sub>2</sub>, (0.085 mmol, 3 mol%), and 1,3-bis(2,4,6-trimethylphenyl)imidizolium (0.085 mmol, 3 mol%) in anhyd THF (3 mL) at r.t. and the mixture was allowed to stir for 18 h. The reaction was quenched with 0.5 M HCl. The aqueous layer was washed with Et<sub>2</sub>O and the combined organic layers were washed with sat. NaCl and dried. The product was purified by flash chromatography (silica gel, 200–425 mesh, 25% CH<sub>2</sub>Cl<sub>2</sub>–hexane) and recrystallized (40% CH<sub>2</sub>Cl<sub>2</sub>–hexane) to give pure product (0.4291 g, 49%); mp 145–147 °C;  $R_f = 0.34$  (80% CH<sub>2</sub>Cl<sub>2</sub>–hexanes).

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.21–7.24 (m, 1 H), 7.35–7.54 (m, 6 H), 7.75–7.80 (m, 4 H), 7.94–7.97 (m, 1 H), 8.70–8.71 (m, 2 H), 8.84–8.87 (m, 2 H).

<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>): δ = 119.55, 126.14, 127.58, 127.78, 128.59, 129.01, 138.76, 141.08, 142.39, 157.51, 164.77.

UV/Vis (MeOH):  $\lambda_{max}$  ( $\epsilon$ ) = 202 (48400), 252 (57900), 310 nm (1450).

Anal. Calcd for  $C_{22}H_{16}N_2$ : C, 85.69; H, 5.23; N, 9.08. Found: C, 85.36; H, 5.22; N, 8.98.

#### **Imines; General Procedure**

Imine syntheses were performed by two methods: (1) Reactions between a volatile amine (1.2-1.6 equiv) and **5** (0.500 g, 1.93 mmol)in CH<sub>2</sub>Cl<sub>2</sub>. Mixtures were magnetically stirred and brought to reflux. Molecular sieves were added for removal of H<sub>2</sub>O. Removal of the excess amine was accomplished under vacuum and products isolated in excellent yields and purities. (2) A 1:1 molar ratio of amine and **5** (0.500 g) were mixed in CH<sub>2</sub>Cl<sub>2</sub> at r.t. for several hours. Na<sub>2</sub>SO<sub>4</sub> was added to remove H<sub>2</sub>O. The mixture was filtered and the solvent removed by evaporation. Products were isolated in excellent yields and purity.

# **3,5-Diphenylbenzaldehyde** *N***-Phenylimine** (9)

Yield: 98%.

IR (neat): 1627 cm<sup>-1</sup> (C=N).

<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.24–7.31 (m, 3 H), 7.35–7.53 (m, 8 H), 7.69–7.76 (m, 4 H), 7.94 (t, 1 H), 8.12 (d, 2 H), 8.59 (s, 1 H). HRMS-ESI: *m*/*z* [M + H]<sup>+</sup> calcd for C<sub>25</sub>H<sub>20</sub>N: 334.1590; found:

34.1585.  $m_{\ell}$  [M + H] calcu for  $C_{25}H_{20}N$ . 554.1590, found 34.1585.

# **3,5-Diphenylbenzaldehyde** *N***-Benzylimine (10)** Yield: 97%.

IR (neat): 1645 cm<sup>-1</sup> (C=N).

<sup>1</sup>H NMR (200 MHz,  $CDCl_3$ ):  $\delta = 4.89$  (s, 2 H), 7.26–7.53 (m, 11 H), 7.65–7.74 (m, 4 H), 7.88 (t, 1 H), 8.00 (d, 2 H), 8.53 (s, 1 H).

HRMS-ESI:  $m/z [M + H]^+$  calcd for C<sub>26</sub>H<sub>22</sub>N: 348.1749; found: 348.1743.

#### 3,5-Diphenylbenzaldehyde N-2-Aminoethylimine (11)

The monoimine was generated by using excess ethylenediamine and by allowing the reaction to reflux overnight; yield: 85%.

IR (neat): 1654 cm<sup>-1</sup> (C=N).

<sup>1</sup>H NMR (200 MHz,  $CDCl_3$ ):  $\delta = 1.33$  (s, 2 H), 3.07 (t, 2 H), 3.74 (t, 2 H), 7.35–7.52 (m, 6 H), 7.62–7.84 (m, 4 H), 7.87 (t, 1 H), 7.95 (d, 2 H), 8.45 (s, 1 H).

HRMS-ESI:  $m/z [M + H]^+$  calcd for  $C_{21}H_{21}N_2$ : 301.1699; found: 301.1692.

#### Bis(3,5-Diphenylbenzaldehyde) N-Ethylenediimine (13)

Relatively pure diimine was generated by placing the monoimine under vacuum for 24 h. Alternatively a 2:1 amine/aldehyde mixture was refluxed for 48 h; yield: 25%.

IR (neat):  $1645 \text{ cm}^{-1}$  (C=N).

<sup>1</sup>H NMR (200 MHz,  $CDCl_3$ ):  $\delta = 4.06 (s, 4 H), 7.36-7.50 (m, 12 H), 7.60-7.81 (m, 8 H), 7.83 (t, 2 H), 7.91 (d, 4 H), 8.41 (s, 2 H).$ 

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 163.03, 142.39, 140.74, 137.35, 128.04, 128.53, 127.87, 127.52, 126.03, 61.75.

HRMS-ESI:  $m/z \ [M + H]^+$  calcd for  $C_{40}H_{33}N_2$ : 541.2638; found: 541.2619.

# 3,5-Diphenylbenzaldehyde *N*-2-(Dimethylamino)ethylimine (12)

Yield: 85%.

IR (neat):  $1647 \text{ cm}^{-1}$  (C=N).

<sup>1</sup>H NMR (300 MHz, acetone- $d_6$ ):  $\delta = 2.33$  (s, 6 H), 2.68 (t, 2 H), 3.81 (t, 2 H), 7.34–7.51 (m, 6 H), 7.65–7.71 (m, 4 H), 7.86 (t, 1 H), 7.94 (d, 2 H), 8.46 (s, 1 H).

<sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>): δ = 162.10, 142.41, 140.78, 137.39, 129.08, 128.55, 127.92, 127.54, 126.07, 60.29, 60.15, 46.09.

HRMS-ESI:  $m/z \ [M + H]^+$  calcd for  $C_{23}H_{25}N_2$ : 329.2012; found: 329.2011.

#### X-ray Structure Determination of 5

X-ray diffraction data were collected on a Bruker APEX 2 CCD platform diffractometer [Mo-K $\alpha$  ( $\lambda = 0.71073$  Å)] at 115 K.<sup>14</sup> A suitable crystal was mounted in a nylon loop with Paratone-N cryoprotectant oil. The structure was solved using direct methods and standard difference map techniques, and was refined by full-matrix least-squares procedures on  $F^2$  with SHELXTL (Version 6.14).<sup>15</sup> All nonhydrogen atoms were refined anisotropically. Hydrogen atoms were included in calculated positions and were refined using a riding model, except for the aldehyde hydrogen, which was included in a calculated position and refined semi-freely with the help of a distance restraint. The structure exhibits a twofold whole molecule disorder about the crystallographic mirror plane; each atom of every disordered atom pair was assigned an occupancy of 0.5. The disorder was refined with the help of similarity restraints on 1-2 and 1-3 distances and displacement parameters and rigid bond restraints for anisotropic displacement parameters. Selected data is found in Table 2.

#### **Computational Details**

A molecular model of 3,5-diphenylbenzaldehyde was created using Gaussian 03's GaussView building tools.<sup>9</sup> This model was then minimized using Density Functional Theory at the B3LYP level with a 6-31G basis set using the Gaussian 03 package.<sup>9</sup> The subsequent checkpoint output file was then obtained to view the optimized parameters. Evaluation of rotational energy barriers (semiempirical calculations): A molecular model of 3,5-diphenylbenzaldehyde was created using Spartan 04 entry model kit (Wavefunction Inc., Irvine, CA.).<sup>10</sup> The dihedral angle containing the aldehyde constituent was constrained to 5° and the dihedral angle of the phenyl-benzaldehyde bond [C(phenyl)-C(benzaldehyde)] furthest from the oxygen atom in the molecule was constrained to 37.8°. These constraints were placed to mimic the X-ray crystallographic data. The other phenyl-benzaldehyde bond [C(benzaldehyde)–C(phenyl)] closest to the oxygen atom was constrained at  $0^{\circ}$ and set to rotate 180° during the subsequent job. An energy profile  $(\Delta rel E)$  was then created as Hartree–Fock molecular orbital calcu-

Table 2	Selected	Crystal	Data for	Compound 5
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Property	Value
formula	C <sub>19</sub> H <sub>14</sub> O
habit, color	parallelepiped, colorless
lattice type	orthorhombic
space group	$Cmc2_1$
a	23.647(3) Å
b	6.9588(8) Å
с	8.3389(9) Å
V	1372.2(3) Å <sup>3</sup>
Z	4

lations using a  $6-31G^*$  basis set resulting in relative energies in kcal/mol<sup>-1</sup> calculated for successive 20° rotation about the specified bond. These relative energies were then plotted against their defined angle of rotation. Within this energy profile, energy maxima occur near the planar 0° and 180° rotamers and energy minima occur approximately at 38° and 137°.

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