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## COMMUNICATION

# P=O functional group-containing cryptands: from supramolecular complexes to poly[2]pseudorotaxanes

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Two kinds of cryptand-based host-guest complexes were constructed successfully, in which P=O functional groups were located at the different positions of the third arms. Consequently, supramolecular poly[2]pseudorotaxanes with 10 almost linear and zigzag shapes were formed, respectively in the solid states.

Host-guest system has undoubtedly played key roles in selfassemblies due to its self-selectivity, high efficiency, and stimuliresponsiveness in the host-guest chemistry,<sup>1</sup> and thus, it is highly <sup>15</sup> desirable to design and synthesize novel host molecules which are one of the major driving forces to accelerate the development of supramolecular chemistry. Since the "cryptand" which was firstly introduced by Lehn *et al.* in the late 1960's opened the path to use cryptand as the host molecule,<sup>2</sup> numerous valuable <sup>20</sup> contributions had been made in synthesizing and utilizing different kinds of cryptands.<sup>3</sup> As one of the representative compounds, crown ether-based cryptands have attracted more attentions not only because of their 3D spatial architectures but also due to the possibility of their noteworthy application in

- <sup>25</sup> molecular recognition and self-assembly based on non-covalent interactions.<sup>4</sup> For example, Gibson *et al.* synthesized a bis(*m*phenylene)-32-crown-10 (BMP32C10)-based cryptand which exhibited excellent binding affinity with dimethyl paraquat (100fold greater than BMP32C10),<sup>5</sup> after that, a series of BMP32C10-
- <sup>30</sup> based cryptands with different functional groups in the third arms were designed and synthesized during the past decades,<sup>6</sup> and accordingly, their applications in the construction of supramolecular assemblies, such as supramolecular dimers and polymers were explored as well.<sup>7</sup>
- Typically, the first pseudocryptand-type poly[2]pseudorotaxanes based on BMP32C10 were reported by Gibson *et al.*, where a paraquat molecule lay parallelly between the carbazole rings generating a "sandwich" structure which was stabilized by the hydrogen bonds among the host, guest, and the central  $PF_6^-$ , and
- <sup>40</sup> as a result, a pseudocryptand-type poly[2]pseudorotaxane was formed in the solid state.<sup>8</sup> Soon after, supramolecular poly[2]pseudorotaxane formed from a BMP32C10-based cryptand and vinylogous viologen was reported by Huang *et al.*, in which two ester carbonyl oxygen atoms of the cryptand formed
- <sup>45</sup> two hydrogen bonds with one aromatic hydrogen and one *O*-methylene hydrogen of the cryptand in the adjacent [2]pseudorotaxane, affording a linear poly[2]pseudorotaxane packing structure in the solid state.<sup>7e</sup> As shown above, hydrogen

bonds as one of the most crucial intermolecular forces played the <sup>50</sup> key, even the decisive roles in the self-assembly and the fabrication of supramolecular polymers.

- On the other hand, phosphine oxide functional group, as an important unit in coordination chemistry and organometallic catalysis<sup>9</sup> has been widely employed to construct supramolecular
- <sup>55</sup> architectures due to its strong binding affinity with cationic guests, and particularly, unique properties as excellent hydrogen bonding acceptor.<sup>10</sup> Recently, our group has successfully realized a switchable three-station molecular shuttle using phosphine oxide as a potential recognition binding unit *via* the formation of
   <sup>60</sup> new hydrogen bonds between the phosphine oxide unit of the
- thread and NH groups of the macrocycle, moreover intermolecular hydrogen bonds between phosphine oxide unit and urea protons of a neighboring molecule were observed as well.<sup>10e</sup> Also inspired by our recent researches on the host-guest systems
- <sup>65</sup> on a BMP32C10-based cryptand,<sup>11</sup> herein, we report two novel BMP32C10-based cryptands **1** and **2** bridging by the P=O functional groups, which were located at the different positions of the third arms of the cryptands (Fig. 1), and such new cryptands **1** and **2** were capable of forming [2]pseudorotaxanes with paraquat
- <sup>70</sup> guest **3** both in solution and in the solid state. More interestingly, the generated [2]pseudorotaxanes could further self-assemble into supramolecular poly[2]pseudorotaxanes in the solid state driven by phosphine oxide-based hydrogen bonding interactions and C-H… $\pi$  interactions. It was found that two different types of <sup>75</sup> supramolecular poly[2]pseudorotaxanes were formed in almost linear and zigzag shapes, respectively in the solid states.



Fig. 1 Chemical structures of two cryptands 1, 2 and guest 3.

Cryptands 1 and 2 were obtained in moderate yields using

commercially available starting materials according to the reported methods (Scheme S1, ESI<sup>†</sup>).<sup>6i</sup> The chemical structure of cryptands 1 and 2 were fully confirmed by <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>31</sup>P NMR, and HR-ESI-MS, respectively. The complexation 5 behaviors of the two cryptands 1 and 2 with guest 3 were investigated then. The acetonitrile solutions of 1, 2, and 3 are colorless, while their equimolar mixtures  $1 \supset 3$  and  $2 \supset 3$  became yellow due to the possible charge-transfer interactions between the electron-rich aromatic rings of the cryptands 1, 2 and the 10 electron-poor pyridinium rings of 3, which were further investigated in details by UV-vis spectroscopy (Fig. S27, ESI<sup>+</sup>), and it is an obvious evidence for the possible complexation between cryptands and paraquat.<sup>12</sup> Job plots based on <sup>1</sup>H NMR data demonstrated that the above host-guest complexes  $1 \supset 3$  and 15  $2 \supset 3$  were both of 1:1 stoichiometry in solution (Fig. S28, ESI<sup>+</sup>), which were also confirmed by low-resolution electrospray ionization mass spectrometry (LR-ESI-MS): m/z 1229.50 (100 %) for  $[1 \supset 3 - PF_6]^+$  (Fig. S29, ESI<sup>†</sup>) and m/z 1229.45 (100 %) for  $[2 \supset 3 - PF_6]^+$  (Fig. S30, ESI<sup>+</sup>). The association constant ( $K_a$ )

<sup>20</sup> between  $1 \supset 3$  and  $2 \supset 3$  in CD<sub>3</sub>CN were calculated to be  $(1.62 \pm 0.04) \times 10^3$  and  $917 \pm 19 \text{ M}^{-1}$ , respectively, by using a nonlinear curve-fitting analysis based on the <sup>1</sup>H NMR titration experiments (Fig. S31–S34, ESI†). The binding affinity of complex  $1 \supset 3$  is about 1.8 times higher than that of  $2 \supset 3$ .

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Then, the self-assembly of 1 and 3 was studied by <sup>1</sup>H NMR spectroscopy. As shown in Fig. 2, only one set of peaks in the spectrum of the equimolar mixture of 1 and 3 in CD<sub>3</sub>CN solution could be observed, indicating the formation of supramolecular complex  $1 \supset 3$  is a fast exchange process on the NMR 30 spectroscopy timescale. After mixing 1 and 3, obvious upfield chemical shift changes were observed for H<sub>3b</sub> of the paraquat derivative 3 ( $\Delta\delta H_{3b} = -0.27$  ppm). With respect to cryptand host 1, peaks corresponding to protons  $H_{1a}$ ,  $H_{1b}$ , and  $H_{1c}$  shifted upfield ( $\Delta\delta H_{1a} = -0.31$  ppm,  $\Delta\delta H_{1b} = -0.35$  ppm, and  $\Delta\delta H_{1c} =$  $_{35}$  –0.15 ppm), while peaks for protons H<sub>1e</sub> and H<sub>1h</sub> moved downfield ( $\Delta\delta H_{1e} = 0.36$  ppm and  $\Delta\delta H_{1h} = 0.20$  ppm) (Fig. 2, spectra a-c), indicating that protons  $H_{1a}$ ,  $H_{1b}$ , and  $H_{1c}$  of host 1 were located in the shielding zone of the two pyridinium rings of 3, while protons  $H_{1e}$  and  $H_{1h}$  were in the deshielding zone. <sup>40</sup> Similar chemical shift changes could be observed in the <sup>1</sup>H NMR spectra for the case of supramolecular complex  $2 \supset 3$  (Fig. 2, spectra c-e). Moreover, 2D NOESY was also carried out to study



**Fig. 2** Partial <sup>1</sup>H NMR spectra (400 MHz, CD<sub>3</sub>CN, 298 K): (a) **1**; (b) 3.00 <sup>45</sup> mM **1** and 3.00 mM **3**; (c) **3**; (d) 3.00 mM **2** and 3.00 mM **3**; (e) **2**.

the self-assembly of **1** and **3** in CD<sub>3</sub>CN solution. Correlation signals were observed between protons  $H_{3a}$  and  $H_{3b}$  of guest **3** and protons on the ether chains ( $H_{1j}$ ) and phenyl rings ( $H_{1a}$ ,  $H_{1b}$ , and  $H_{1e}$ ) of host **1**, indicating the formation of host-guest <sup>50</sup> inclusion complex between **1** and **3** (Fig. S37, ESI†). Similar correlations were observed for the inclusion complex between **2** and **3** as well (Fig. S38, ESI†).

Fortunately, the host-guest complexation of  $1 \supset 3$  and  $2 \supset 3$  were both observed in their single crystals, which were obtained by 55 slow diffusion of isopropyl ether into an equimolar acetone solution of  $1 \supset 3$  or  $2 \supset 3$ . The colors of the crystal of  $1 \supset 3$  and  $2 \supset 3$ were yellow due to the charge-transfer interactions, which were consistent with the phenomena in solution. The crystal structures of  $1 \supset 3$  and  $2 \supset 3$  demonstrate that guest 3 threads through the 60 cavity of host 1 or 2, forming a [2]pseudorotaxane-type threaded structure (Fig. 3), which are stabilized cooperatively by: (i) hydrogen bonds marked by green dashed line; (ii) charge-transfer interactions between the electron-rich aromatic rings of cryptands 1 or 2 and the electron-poor pyridinium rings of paraquat 3, and 65 face-to-face  $\pi$ -stacking interactions could also be recognized as an important factor to stable the crystal. Three hydrogen bonds (**b**, **h**, **i**) are formed between three oxygen atoms on host 1 and three hydrogen atoms of guest 3 in the crystal structure of  $1 \supset 3$ (Fig. 3a). In addition, eleven hydrogen bonds are formed by two  $_{70}$  PF<sub>6</sub> ions, which act as hydrogen bonding bridges interacting with host 1 and guest  $3^{13}$ . In the crystal structure of  $2\supset 3$ , five hydrogen bonds (o, p, q, r, s) are formed, involving four oxygen atoms on host 2 and three hydrogen atoms of guest 3 (Fig. 3b). However, only two hydrogen bonds are formed by one  $PF_6^-$  ion 75 as the hydrogen bonging bridge. In the solid state of  $1 \supset 3$ , the number of hydrogen bonds is much more than that of  $2 \supset 3$ , which

is probably the reason why the binding constant of  $1 \supset 3$  is higher than that of  $2 \supset 3$ . Moreover, in the crystal structure of  $1 \supset 3$ , the



80 Fig. 3 Ball-stick views of the X-ray crystal structure of (a) 1⊃3; (b) 2⊃3. Host is red, guest is blue, hydrogens are gray, oxygens are green, nitrogens are lime, fluorines are plum and phosphorus is pink. Solvent molecules and hydrogen atoms except those involved in hydrogen bonding between 1 or 2 and 3 were omitted for clarity. Hydrogen bond
85 parameters are as follows: H…O (F) distances (Å), C…O (F) distances (Å), C—H…O (F) angles (deg): (a) 2.44, 3.03, 118; (b) 2.43, 3.33, 152; (c) 2.39, 2.93, 114; (d) 2.36, 3.34, 172; (e) 2.52, 3.44, 163; (f) 2.54, 3.29, 136; (g) 2.12, 3.03, 158; (h) 2.05, 3.00, 174; (i) 2.62, 3.43, 141; (j) 2.37, 3.09, 129; (k) 2.48, 3.04, 116; (l) 2.27, 2.98, 131; (m) 2.55, 3.39, 147; (n) 90 2.10, 2.96, 150; (o) 2.56, 3.25, 129; (p) 2.61, 3.41, 144; (q) 2.62, 3.27, 128; (r) 2.59, 3.28, 131; (s) 2.46, 3.31, 152; (t) 2.46, 3.39, 172; (u) 2.49, 3.03, 118. Face to face π-stacking parameters: centroid–centroid distance (Å) 1⊃3, 4.33; 4.16; 2⊃3, 4.52; 3.62; 3.76; 4.62; ring plane–ring plane inclination (deg): 1⊃3, 5.37; 4.66; 2⊃3, 7.97; 4.57; 6.48; 10.6.

phenylene rings of the 32-crown-10 motif are face-to-face  $\pi$ -stacked with only one pyridinium ring of the guest, while in the crystal structure of  $2\supset 3$ , the phenylene rings are face-to-face  $\pi$ -stacked with both pyridinium rings of the guest **3** (Fig. S39–S40,

- s ESI<sup>†</sup>). The dihedral angles between the two pyridinium rings of **3** are  $35.59^{\circ}$  and  $5.16^{\circ}$  in the solid states of  $1 \supset 3$  and  $2 \supset 3$ , respectively. Presumably, these differences might result from the maximization of the non-covalent bonding interactions between the cryptand (1 or 2) and paraquat guest **3**.
- Interestingly, as shown in Fig. 4, in the solid state of  $1 \supset 3$ , one 10  $C-H\cdots\pi$  interaction (a) and two intermolecular hydrogen bonds (b, c) between phosphine oxide and two pyridinium hydrogen atoms of the guest 3 in the adjacent [2]pseudorotaxane are observed, which connect the individual [2]pseudorotaxane into an 15 almost linear supramolecular poly[2]pseudorotaxane in the solid state. But in the solid state of  $2 \supset 3$  (Fig. 5), supramolecular poly[2]pseudorotaxane with zigzag packed structure is formed, which is driven by two kinds of C-H $\cdots\pi$  interactions (d, f) and one intermolecular hydrogen bond (e) between phosphine oxide 20 and one pyridinium hydrogen atom of 3 in the adjacent [2]pseudorotaxane. That is, only by changing the locations of the P=O functional group in the benzene rings of the third arms of cryptands 1 and 2, two supramolecular poly[2]pseudorotaxane with different shapes could be obtained, reflecting the complexity 25 and diversity of self-assembly processes based on crown etherbased cryptands.



Fig. 4 Linear-shape supramolecular poly[2]pseudorotaxane packing structure of 1⊃3 in the solid state. PF<sub>6</sub><sup>-</sup> and hydrogen atoms except those <sup>30</sup> involved in hydrogen bonding between 1 and 3 were omitted for clarity. Hydrogen bond parameters are as follows: H…O distances (Å), C…O distances (Å), C–H…O angles (deg): b, 2.71, 3.44, 135; c, 2.16, 3.09, 168. C–H…π parameters: H…ring centre distances (Å), C–H…ring angles (deg): a, 2.98, 142.



Fig. 5 Zigzag-shape supramolecular poly[2]pseudorotaxane packing structure of 2⊃3 in the solid state. PF<sub>6</sub><sup>-</sup> and hydrogen atoms except those involved in hydrogen bonding between 2 and 3 were omitted for clarity. Hydrogen bond parameters are as follows: H…O distances (Å), C…O 40 distances (Å), C–H…O angles (deg): e, 2.21, 3.09, 158. C–H…π parameters: H…ring centre distances (Å), C–H…ring angles (deg): d, 3.18, 142; f, 3.24, 143.

### Conclusions

In summary, [2]pseudorotaxanes were formed in solution and in 45 the solid state via the self-assembly of phosphine oxide functional group-containing cryptands 1 and 2 with paraquat guest 3. Moreover, in the solid states of  $1 \supset 3$  and  $2 \supset 3$ , it was found that two different supramolecular poly[2]pseudorotaxanes were formed, which were driven by  $C-H\cdots\pi$  interactions and 50 phosphine oxide-based hydrogen bonding interactions. Interestingly, due to the tiny difference between the two cryptands, the shape of supramolecular poly[2]pseudorotaxanes  $1 \supset 3$  and  $2 \supset 3$  was almost linear and zigzag, respectively in the solid states. Our future work will focus on: (i) utilizing P=O 55 functional group as the hydrogen bonding acceptor to construct more complicated BMP32C10-based molecular shuttles and machines; and (ii) applying P=O functional group as the active site in organometallic chemistry, especially in catalytic chemistry, and BMP32C10-based cryptands could provide the unique 60 cavities, which are necessary in mimicking enzyme catalysis in order to control the catalytic process.

#### Notes and references

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 † Electronic Supplementary Information (ESI) available: Experimental

details, <sup>1</sup>H NMR, <sup>13</sup>C NMR, <sup>31</sup>P NMR, NOESY, Job plots for the host-<sup>5</sup> guest complexes, LR-ESI-MS of the host-guest complexes, determination of the association constants, X-ray crystal data (CIF) of 1⊃3 (CCDC 1004105) and 2⊃3 (CCDC 1011981). For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/b000000x/

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- (a) J. D. Badjić, V. Balzani, A. Credi, S. Silvi and J. F. Stoddart, Science, 2004, 303, 1845; (b) Q. C. Wang, D. H. Qu, J. Ren, K. Chen and H. Tian, Angew. Chem., Int. Ed., 2004, 43, 2661; (c) J. V.
- Hernández, E. R. Kay and D. A. Leigh, *Science*, 2004, **306**, 1532; (d)
   D. S. Guo, K. Wang, Y. X. Wang and Y. Liu, *J. Am. Chem. Soc.*, 2012,
   **134**, 10244; (e) S. L. Li, T. Xiao, C. Lin and L. Wang, *Chem. Soc. Rev.*, 2012, **41**, 5950; (f) K. Zhu, V. N. Vukotic, N. Noujeim and S. J. Loeb, *Chem. Sci.*, 2012, **3**, 3265; (g) J. d. Barrio, P. N. Horton, D.
- Lairez, G. O. Lloyd, C. Toprakcioglu and O. A. Scherman, J. Am. Chem. Soc., 2013, 135, 11760; (h) C. Talotta, C. Gaeta, Z. Qi, C. A. Schalley and P. Neri, Angew. Chem., Int. Ed., 2013, 52, 7437; (i) Q. Zhou, P. Wei, Y. Zhang, Y. Yu and X. Yan, Org. Lett., 2013, 15, 5350.
   (j) H. Yang, B. Yuan, X. Zhang and O. A. Scherman, Acc. Chem. Res., 2014, 47, 2106; (k) Y. Han, Z. Meng, Y. X. Ma and C. F. Chen, Acc.
- Chem. Res., 2014, 47, 2026.
   B. Dietrich, J. M. Lehn and J. P. Sauvage, *Tetrahedron Lett.*, 1969, 10,
- 2. B. Dietrich, J. M. Lehn and J. P. Sauvage, *Tetrahedron Lett.*, 1969, **10**, 2889.
- (a) A.-M. Caminade and J. P. Majoral, *Chem. Rev.*, 1994, 94, 1183; (b)
   H.-J. Schneider and A. K. Yatsimirsky, *Chem. Soc. Rev.*, 2008, 37, 263; (c) J.-C. Chambron and M. Meyer, *Chem. Soc. Rev.*, 2009, 38, 1663; (d) S. O. Kang, J. M. Llinares, V. W. Day and K. Bowman-James, *Chem. Soc. Rev.*, 2010, 39, 3980; (e) X. Yan, P. Wei, B. Xia, F. Huang and Q. Zhou, *Chem. Commun.*, 2012, 48, 4968.
- 35 4. (a) S. Li, M. Liu, B. Zheng, K. Zhu, F. Wang, N. Li, X.-L. Zhao and F. Huang, Org. Lett., 2009, 11, 3350; (b) M. Liu, S. Li, M. Zhang, Q. Zhou, F. Wang, M. Hu, F. R. Fronczek, N. Li and F. Huang, Org. Biomol. Chem., 2009, 7, 1288; (c) Z. Niu, C. Slebodnick, D. Schoonover, H. Azurmendi, K. Harich and H. W. Gibson, Org. Lett.,
- 2011, 13, 3992; (d) M. Zhang, X. Yan, F. Huang, Z. Niu and H. W. Gibson, *Acc. Chem. Res.*, 2014, 47, 1995; (e) J.-F. Xu, Y.-Z. Chen, L.-Z. Wu, C.-H. Tung and Q.-Z. Yang, *Org. Lett.*, 2014, 16, 684; (f) P. Wei, B. Xia, Y. Zhang, Y. Yu and X. Yan, *Chem. Commun.*, 2014, 50, 3973.
- 45 5. W. S. Bryant, J. W. Jones, P. E. Mason, I. Guzei, A. L. Rheingold, F. R. Fronczek, D. S. Nagvekar and H. W. Gibson, *Org. Lett.*, 1999, 1, 1001.
- (a) F. Huang, F. R. Fronczek and H. W. Gibson, J. Am. Chem. Soc., 2003, 125, 9272;
   (b) M. Zhang, K. Zhu and F. Huang, Chem.
- Commun., 2010, 46, 8131; (c) K. Zhu, L. Wu, X. Yan, B. Zheng, M. Zhang and F. Huang, *Chem.-Eur. J.*, 2010, 16, 6088; (d) M. Liu, X. Yan, M. Hu, X. Chen, M. Zhang, B. Zheng, X. Hu, S. Shao and F. Huang, *Org. Lett.*, 2010, 12, 2558; (e) F. Wang, J. Zhang, X. Ding, S. Dong, M. Liu, B. Zheng, S. Li, L. Wu, Y. Yu, H. W. Gibson and F.
- Huang, Angew. Chem., Int. Ed., 2010, 49, 1090; (f) Z. Niu, F. Huang and H. W. Gibson, J. Am. Chem. Soc., 2011, 133, 2836; (g) M. Zhang, B. Zheng and F. Huang, Chem. Commun., 2011, 47, 10103; (h) J. Li, P. Wei, X. Wu, M. Xue and X. Yan, Org. Lett., 2013, 15, 4984; (i) X. Ji, M. Zhang, X. Yan, J. Li and F. Huang, Chem. Commun., 2013, 49, 1178.
- 1178
- (a) F. Huang, L. Zhou, J. W. Jones, H. W. Gibson and M. Ashraf-Khorassani, *Chem. Commun.*, 2004, 2670; (b) F. Huang, K. A. Switek, L. N. Zakharov, F. R. Fronczek, C. Slebodnick, M. Lam, J. A. Golen, W. S. Bryant, P. E. Mason, A. L. Rheingold, M. Ashraf-Khorassani
- and H. W. Gibson, J. Org. Chem., 2005, 70, 3231; (c) F. Wang, Q. Zhou, K. Zhu, S. Li, C. Wang, M. Liu, N. Li, F. R. Fronczek and F. Huang, *Tetrahedron*, 2009, 65, 1488; (d) M. Liu, S. Li, M. Hu, F. Wang and F. Huang, Org. Lett., 2010, 12, 760; (e) X. Yan, P. Wei, M. Zhang, X. Chi, J. Liu and F. Huang, Org. Lett., 2011, 13, 6370; (f) X. Yan, X. Chi, P. Wei, M. Zhang and F. Huang, Eur. J. Org. Chem.,
- 2012, **2012**, 6351; (g) J. Li, P. Wei, X. Wu, M. Xue, X. Yan and Q.

Zhou, RSC Adv., 2013, 3, 21289.

- Z. Niu, C. Slebodnick, K. Bonrad, F. Huang and H. W. Gibson, Org. Lett., 2011, 13, 2872.
- 75 9. (a) V. V. Grushin, *Chem. Rev.*, 2004, **104**, 1629; (b) F. Däbritz, G. Theumer, M. Gruner and I. Bauer, *Tetrahedron*, 2009, **65**, 2995.
  - (a) I. Bauer, O. Rademacher, M. Gruner and W. D. Habicher, *Chem.-Eur. J.*, 2000, 6, 3043; (b) A. Theil, J. Hitce, P. Retailleau and A. Marinetti, *Eur. J. Org. Chem.*, 2006, 2006, 154; (c) A. Theil, C.
- Mauve, M.-T. Adeline, A. Marinetti and J.-P. Sauvage, Angew. Chem., Int. Ed., 2006, 45, 2104; (d) R. Ahmed, A. Altieri, D. M. D'Souza, D. A. Leigh, K. M. Mullen, M. Papmeyer, A. M. Z. Slawin, J. K. Wong and J. D. Woollins, J. Am. Chem. Soc., 2011, 133, 12304; (e) L. Liu, Y. Liu, P. Liu, J. Wu, Y. Guan, X. Hu, C. Lin, Y. Yang, X. Sun, J. Ma and L. Wang, Chem. Sci., 2013, 4, 1701.
  - Q. Wang, M. Cheng, Y. Zhao, Z. Yang, J. Jiang, L. Wang and Y. Pan, Chem. Commun., 2014, 50, 15585.
  - (a) X. Yan, M. Zhang, P. Wei, B. Zheng, X. Chi, X. Ji and F. Huang, *Chem. Commun.*, 2011, **47**, 9840; (b) Y. Guan, M. Ni, X. Hu, T. Xiao, S. Xiong, C. Lin and L. Wang, *Chem. Commun.*, 2012, **48**, 8529.
- 13. Z. Niu, C. Slebodnick and H. W. Gibson, Org. Lett., 2011, 13, 4616.

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