Journal of Molecular Structure 992 (2011) 33–38

Contents lists available at ScienceDirect

Journal of Molecular Structure

journal homepage: www.elsevier.com/locate/molstruc

Synthesis and X-ray crystallographic characterization of substituted aryl imines

James Kovach, Maria Peralta, William W. Brennessel, William D. Jones *

Department of Chemistry, University of Rochester, Rochester, NY 14627, USA

ARTICLE INFO

Article history:
Received 13 January 2011
Received in revised form 11 February 2011
Accepted 12 February 2011
Available online 17 February 2011

Keywords:
Schiff base
α-Diimine
α-Iminoketone
BIAN
Isomerization

ABSTRACT

We report the synthesis of α-diimine 1,4-bis(2,5-di-*tert*-butylphenyl)-2,3-dimethyl-1,4-diaza-1,3-butadiene, **1**, and α-iminoketones 2-[(3,5-xylyl)imino]acenaphthylen-1-one, **4**, and 2-[(4-chlorophenyl)-imino]acenaphthylen-1-one, **5**, all of which have been characterized by ^{1}H NMR, ^{13}C NMR, IR, and X-ray crystallography. Also, we report the previously unknown X-ray crystal structures of α-diimines Ar-BIAN (Ar-BIAN = bis(arylimino)acenaphthene; Ar = 3,5-xylyl, **2**; Ar = 4-chlorophenyl, **3**) and α-iminoketone 2-[(2,6-xylyl)imino]acenaphthylen-1-one, **6**. In solution, **4** and **5** show fluxional behavior observed with variable temperature ^{1}H NMR in DMSO-d_6 which is attributed to isomerization between the *E* and *Z* form of the imine.

1. Introduction

Imines (also known as Schiff bases) represent a large and rich area in chemistry [1–4]. When α-diimines are used as donor-ligands forming d^8 square planar coordination complexes, reactions including ethylene dimerization [5], ethylene oligomerization [6], ethylene polymerization [7–11], propylene polymerization [12], 2-butene polymerization [13], butadiene polymerization [14], CO and vinyl arene copolymerization generating atactic polyketones [15], and the C—H activation of methane [16] and benzene [17] are observed. Specifically, transition metal catalysts containing coordinated α-diimine **3** have been used for the synthesis of epoxides [18], *N*-arylpyrroles [19], hetero-Diels–Alder adducts [19], aniline from the reduction of nitrobenzene [20], and allylic amines [19,21]. Coordination of α-diimine **2** to nickel(II) gives a catalyst that can polymerize norbornene [22]. Additionally, complexes containing α-iminoketones have been shown to polymerize olefins [23] and phenylacetylene [24]. A schematic representation of compounds **1**–**6** investigated in this study is shown in Fig. 1.

2. Experimental

2.1. Materials and instrumentation

Acenaphthenequinone, 3,5-dimethylaniline (98%), 2,6-dimethylaniline (99%) and 2,5-di-*tert*-butylaniline (99%) were purchased from Aldrich and used as received. Methanol (Mallinckrodt), 4-chloroaniline (Eastman), 2,3-butanedione (Alfa Aesar, 99%), and formic acid (Baker, 90.5%) were used as received. $CDCl_3$ with 1% TMS (vol:vol) added was purchased from Cambridge Isotope Laboratories, Inc. and used as received. C, H, N analysis for **1** and **5** was obtained from the University of Illinois Microanalysis Laboratory. C, H, N analysis for **4** was obtained from the CENTC Elemental Analysis Facility at the University of Rochester. Infrared spectra were obtained on a Shimadzu 8400S FTIR using an ATR attachment. NMR data were obtained on a Bruker 400 or 500 MHz NMR spectrometer. Mass spectral data were obtained on a Shimadzu LCMS-2010 with an electrospray ion source.

2.2. 1,4-bis(2,5-di-tert-butylphenyl)-2,3-dimethyl-1,4-diaza-1,3-butadiene (*1*)

A 100 mL round bottom flask was loaded with 2,5-di-*tert*-butylaniline (1.451 g, 6.99 mmol), 2,3-butanedione (313 mg, 3.60 mmol), formic acid (0.06 mL, 1.44 mmol), and methanol (40 mL). The solution was vigorously stirred at room temperature for 22 h. The reaction mixture was filtered producing a yellow precipitate that was washed with methanol (3 × 10 mL) and dried under vacuum affording 705 mg (44%) of a yellow powder. Crystals suitable for structure determination were grown by evaporation of an acetone solution at 25 °C (mp 196–197 °C). IR (ATR) $\nu_{C=N}$ 1632 cm^{-1} Anal. Calcd for $C_{32}H_{48}N_2$: C, 83.42; H, 10.50; N, 6.08. Found: C, 83.45; H, 10.72; N, 6.26. ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 7.39 (d, J = 8 Hz, 2 H), 7.14 (dd, ^{3}J = 8 Hz, ^{4}J = 2 Hz, 2 H), 6.57 (d, ^{4}J = 2 Hz, 2 H), 2.27 (s, 6 H), 1.40 (s, 18 H), 1.37 (s, 18 H). ^{13}C NMR (125 MHz, $CDCl_3$, 25 °C): δ 166.9 (s, *C*), 149.2 (s, *C*), 148.9 (s, *C*), 136.5 (s, *C*), 126.1 (s, *C*H), 120.7 (s, *C*H), 116.6 (s, *C*H), 34.7 (s, *C*), 34.3 (s, *C*), 31.3 (s, C(*C*H$_3$)$_3$), 29.6 (s, C(*C*H$_3$)$_3$), 16.4 (s, *C*H$_3$). MS (ESI - MeCN): m/z = 461 [M]$^+$.

* Corresponding author. Tel.: +1 585 275 5493; fax: +1 585 276 0205.
E-mail address: jones@chem.rochester.edu (W.D. Jones).

0022-2860/$ - see front matter © 2011 Elsevier B.V. All rights reserved.
doi:10.1016/j.molstruc.2011.02.027

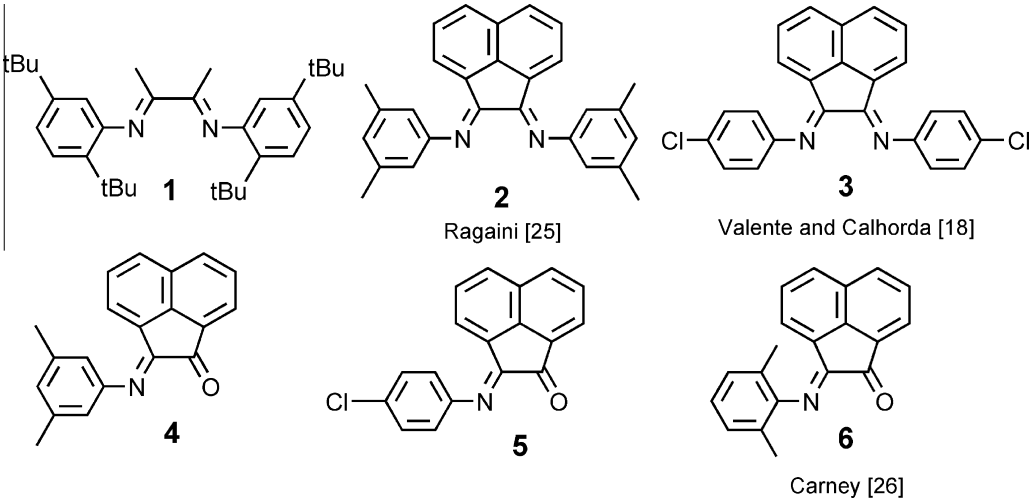

Fig. 1. α-diimines **1**–**3** and α-iminoketones **4**–**6**.

2.3. 3,5-xylyl-BIAN (*2*)

Synthesis of this compound was reported previously by Ragaini [25]. Synthesis of **2** was accomplished by refluxing 3,5-dimethylaniline and acenaphthenequinone in toluene with a catalytic amount of *p*-toluenesulfonic acid using a Dean–Stark trap. Crystals suitable for structure determination were grown by slow evaporation of a pentane and chloroform solution (5:1) at −20 °C.

2.4. 4-chlorophenyl-BIAN (*3*)

Synthesis of this compound was reported previously by Valenti and Calhorda [18] using Elsevier's method [27]. Synthesis of **3** was accomplished by refluxing 4-chloroaniline and acenaphthenequinone in toluene with a catalytic amount of glacial acetic acid using a Dean–Stark trap. Crystals suitable for structure determination were grown by evaporation of a $CDCl_3$ solution at 25 °C.

2.5. 2-[(3,5-xylyl)imino]acenaphthylen-1-one (*4*)

A 100 mL round bottom flask was loaded with acenaphthenequinone (100.6 mg, 0.552 mmol). A formic acid (10 μL, 0.24 mmol)/methanol (10 mL) solution was added and the contents stirred vigorously. A 3,5-dimethylaniline (71.6 mg, 0.579 mmol)/methanol (20 mL) solution was added and the contents were refluxed for 18 h. The red product solution precipitated an orange solid upon cooling to −20 °C. The precipitate was separated by filtration, washed with pentane (5 × 15 mL), and dried under vacuum at 112 °C for 21 h affording 36.3 mg (23%) of an orange powder. Crystals suitable for structure determination were grown by evaporation of an acetone solution at 25 °C (mp = 180 °C (decomp)). IR (ATR) $\nu_{C=O}$ 1718 cm^{-1}, $\nu_{C=N}$ 1652 cm^{-1}. Anal. Calcd for $C_{20}H_{15}NO$: C, 84.19; H, 5.30; N, 4.91. Found: C, 83.82; H, 5.19; N, 5.05. Major isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 8.17 (d, ^3J = 8 Hz, 1 H), 8.16 (d, ^3J = 7 Hz, 1 H), 8.00 (d, ^3J = 8 Hz, 1 H), 7.81 (t, ^3J = 8 Hz, 1 H), 7.46 (t, ^3J = 8 Hz, 1 H), 7.04 (d, J = 7 Hz, 1 H),

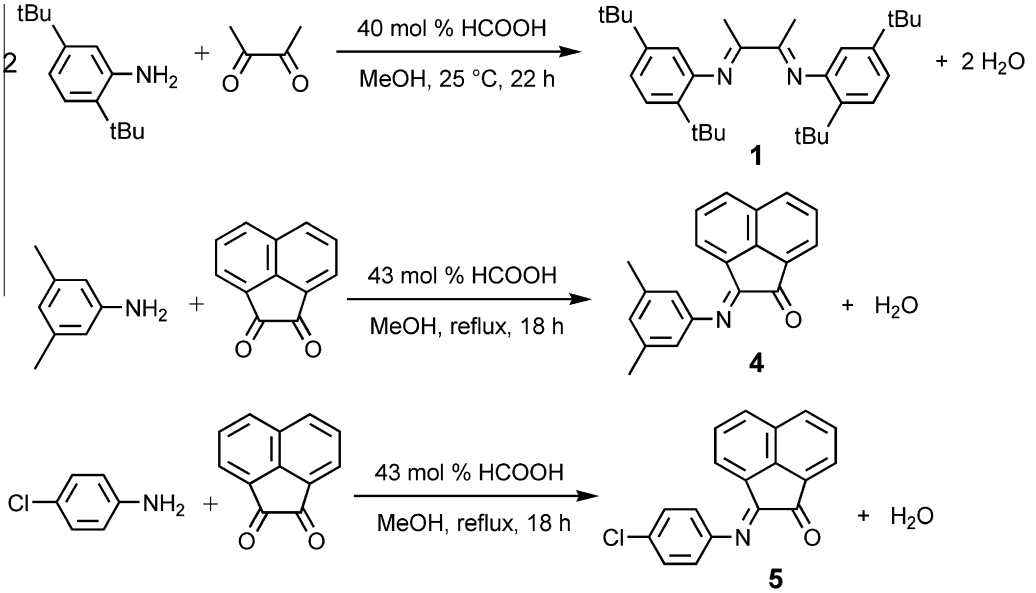

Fig. 2. Synthesis of **1**, **4**, and **5**.

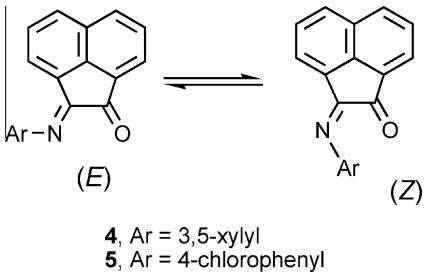

Fig. 3. Solution dynamics of **4** and **5**.

6.92 (s, 1 H), 6.70 (s, 2 H), 2.36 (s, 6 H). ^{13}C NMR (125 MHz, $CDCl_3$, 25 °C): δ 189.8 (s, *C*), 159.0 (s, *C*), 150.7 (s, *C*), 143.4 (s, *C*), 139.0 (s, *C*), 132.0 (s, *C*H), 131.0 (s, *C*), 130.6 (s, *C*), 129.1 (s, *C*H), 128.1 (s, *C*H), 127.9 (s, *C*H), 126.9 (s, *C*), 126.7 (s, *C*H), 123.7 (s, *C*H), 122.1 (s, *C*H), 115.5 (s, *C*H), 21.4 (s, *C*H_3). MS (ESI – MeCN): *m/z* = 286 $[M + H]^+$.

2.6. 2-[(4-chlorophenyl)imino]acenaphthylen-1-one (*5*)

A 100 mL round bottom flask was loaded with acenaphthenequinone (100.5 mg, 0.552 mmol). A formic acid (10 μL, 0.24 mmol)/methanol (10 mL) solution was added and the contents stirred vigorously. A 4-chloroaniline (77.0 mg, 0.604 mmol)/methanol (20 mL) solution was added and the contents were refluxed for 18 h, then cooled to 0 °C. The orange precipitate was separated by filtration, washed with pentane (5 × 15 mL), and dried under vacuum at 114 °C for 21 h affording 67.1 mg (42%) of an orange powder. Crystals suitable for structure determiniation were grown by evaporation of an acetone solution at 25 °C (mp = 230 °C (decomp)). IR (ATR) $\nu_{C=O}$ 1729 cm^{-1}, $\nu_{C=N}$ 1656 cm^{-1}. Anal. Calcd for $C_{18}H_{10}ClNO$: C, 74.11; H, 3.46; N, 4.80. Found: C, 74.01; H, 3.34; N, 4.77. Major isomer: ^{1}H NMR (400 MHz, $CDCl_3$, 25 °C): δ 8.20 (d, ^3J = 8 Hz, 1 H), 8.18 (d, ^3J = 7 Hz, 1 H), 8.04 (d, ^3J = 8 Hz, 1 H), 7.84 (t, ^3J = 8 Hz, 1 H), 7.49 (t, ^3J = 8 Hz, 1 H), 7.45 (d, ^3J = 8 Hz, 2 H), 7.08 (d, ^3J = 7 Hz, 1 H), 7.05 (d, ^3J = 8 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$, 25 °C): δ 189.4 (s, *C*), 159.7 (s, *C*), 149.0 (s, *C*), 143.6 (s, *C*), 132.2 (s, *C*H), 131.0 (s, *C*), 130.6 (s, *C*), 130.5 (s, *C*),

Table 1
Summary of crystallographic data for **1**–**6**.

Parameter	**1**	**2**·**2CHCl$_3$**	**3**·**1.5CDCl$_3$**	**4**	**5**	**6**
Empirical formula	$C_{32}H_{48}N_2$	$C_{30}H_{26}Cl_6N_2$	$C_{25.5}H_{14}Cl_{6.5}D_{1.5}N_2$	$C_{20}H_{15}NO$	$C_{18}H_{10}ClNO$	$C_{20}H_{15}NO$
Formula weight	460.72	627.23	581.83	285.33	291.72	285.33
Crystal system	Triclinic	Monoclinic	Monoclinic	Triclinic	Triclinic	Triclinic
Space group	*P*-1	$P2_1/n$	$P2_1/c$	*P*-1	*P*-1	*P*-1
a (Å)	6.3859(4)	9.7806(16)	26.661(5)	8.2804(6)	6.8246(13)	8.0450(9)
b (Å)	9.8772(7)	19.289(3)	8.9992(18)	8.5977(6)	9.0659(17)	8.4663(10)
c (Å)	12.1637(8)	15.827(3)	22.749(5)	11.0163(8)	10.721(2)	12.3511(14)
α (°)	89.846(1)	90	90	95.550(1)	92.818(3)	74.012(2)
β (°)	78.495(1)	93.150(2)	113.149(3)	94.390(1)	90.832(3)	84.002(2)
γ (°)	74.660(1)	90	90	111.317(1)	96.062(3)	61.948(2)
Volume (Å^3)	724.00(8)	2981.4(9)	5018.8(17)	721.90(9)	658.7(2)	713.39(14)
Z	1	4	8	2	2	2
Goodness-of-fit, F^2	1.037	1.095	1.066	1.031	1.079	1.044
Final R indices [$I > 2\sigma(I)$]	*R*1 = 0.0507	*R*1 = 0.0549	*R*1 = 0.0478	*R*1 = 0.0504	*R*1 = 0.0391	*R*1 = 0.0473
	*wR*2 = 0.1231	*wR*2 = 0.1233	*wR*2 = 0.1167	*wR*2 = 0.1383	*wR*2 = 0.1086	*wR*2 = 0.1320
R indices (all data)	*R*1 = 0.0799	*R*1 = 0.0922	*R*1 = 0.0623	*R*1 = 0.0628	*R*1 = 0.0505	*R*1 = 0.0578
	*wR*2 = 0.1386	*wR*2 = 0.1384	*wR*2 = 0.1226	*wR*2 = 0.1484	*wR*2 = 0.1134	*wR*2 = 0.1436

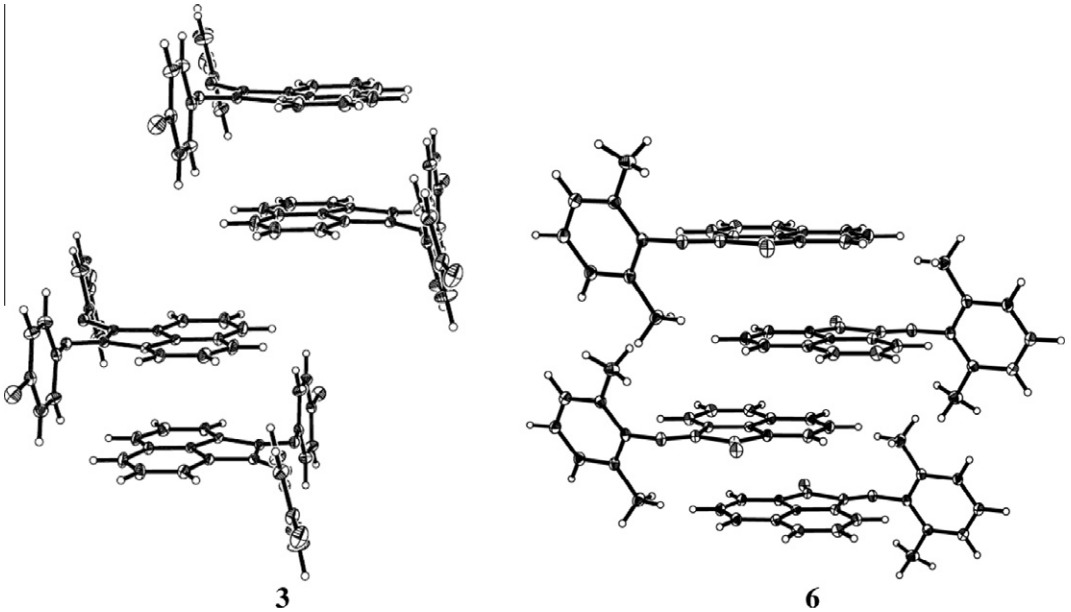

Fig. 4. Crystal packing diagram of **3** and **6** showing intermolecular π-stacking. Thermal ellipsoids were drawn at 50% probability level. Atom labels were removed for clarity.

129.6 (s, *C*H), 129.5 (s, *C*H), 128.3 (s, *C*H), 127.9 (s, *C*H), 126.5 (s, *C*), 123.6 (s, *C*H), 122.4 (s, *C*H), 119.7 (s, *C*H). MS (ESI - MeCN): *m/z* = 292 $[M]^+$.

2.7. 2-[(2,6-xylyl)imino]acenaphthylen-1-one (**6**)

Synthesis of this compound was reported previously by Carney [26]. Synthesis of **6** was accomplished by refluxing 2,6-dimethylaniline and acenaphthenequinone in toluene with a catalytic amount of *p*-toluenesulfonic acid using a Dean–Stark trap. Crystals suitable for structure determination were grown by slow evaporation of a pentane and chloroform solution (4:1) at −30 °C.

3. Results and discussion

3.1. Synthesis and NMR characterization

The syntheses of **1**, **4**, and **5** outlined in this paper lead to precipitates that can be easily separated by a filtration step in high

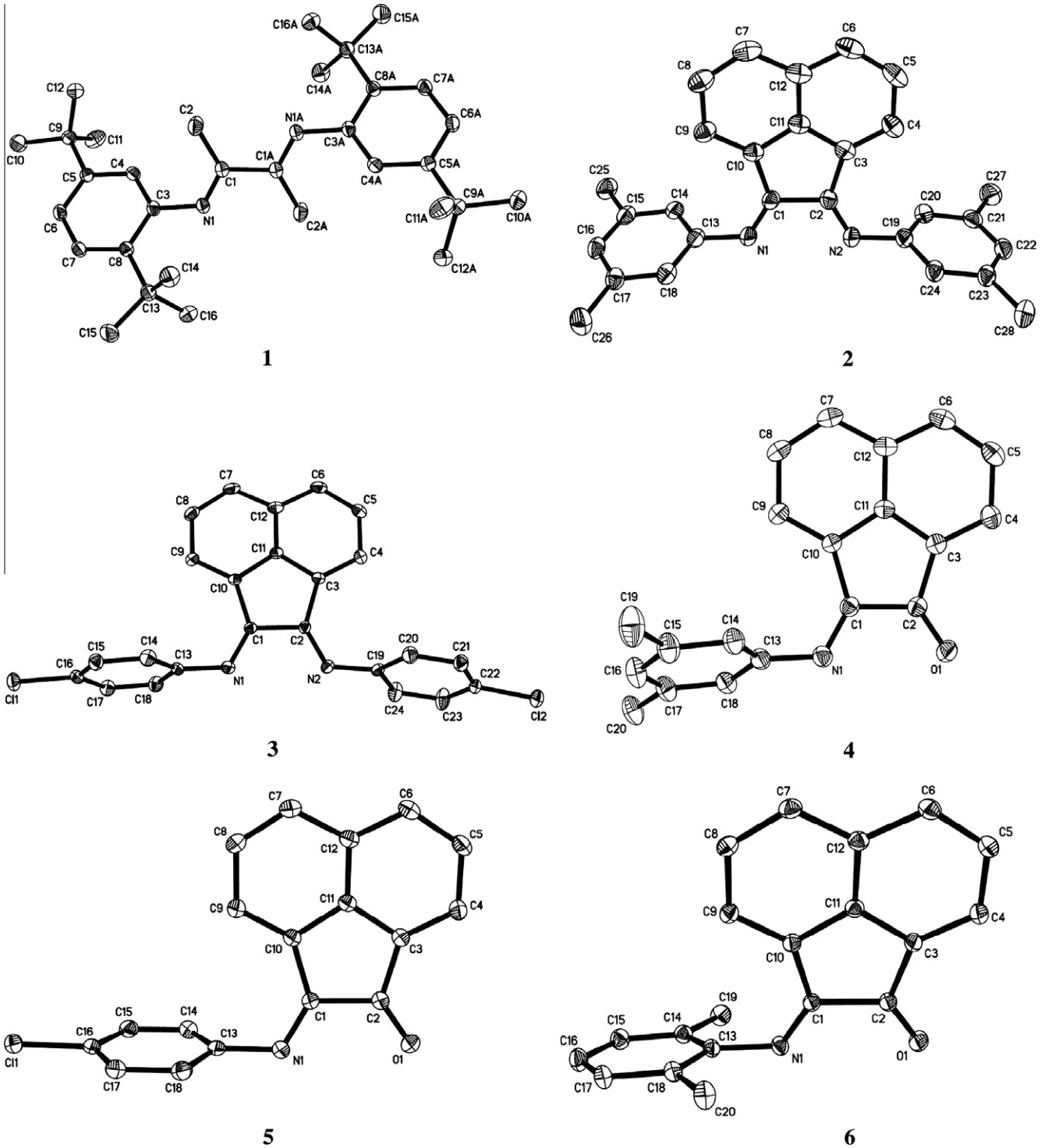

Fig. 5. Molecular structure of **1**–**6** with thermal ellipsoids drawn at 50%. Hydrogen atoms were omitted for clarity.

Table 2
Selected bond lengths of **1**–**6** (Å).

Cmpd	C(3)–N(1)	N(1)–C(1)	C(1)–C(2)	C(1)–C(1A)	C(1A)–C(2A)	C(1A)–N(1A)	N(1A)–C(3A)
1	1.4183(14)	1.2854(14)	1.4991(17)	1.498(2)	1.4991(17)	1.2854(14)	1.4183(14)
Cmpd	C(13)–N(1)	N(1)–C(1)	C(1)–C(10)	C(1)–C(2)	C(2)–C(3)	C(2)–N(2)	N(2)–C(19)
2	1.423(3)	1.274(3)	1.476(3)	1.526(3)	1.480(3)	1.273(3)	1.426(3)
3	1.412(3)	1.272(3)	1.478(3)	1.529(3)	1.478(3)	1.272(3)	1.414(3)
Cmpd	C(13)–N(1)	N(1)–C(1)	C(1)–C(10)	C(1)–C(2)	C(2)–C(3)	C(2)–O(1)	
4	1.4163(14)	1.2763(14)	1.4811(14)	1.5411(15)	1.4762(15)	1.2217(13)	
5	1.4215(15)	1.2742(15)	1.4825(16)	1.5463(16)	1.4817(16)	1.2131(14)	
6	1.4215(9)	1.2777(9)	1.4782(10)	1.5437(10)	1.4767(10)	1.2149(9)	

purity with no further purification necessary (Fig. 2). The synthesis procedures reported in the literature for α-diimines **2** and **3** involve the *in situ* complexation to $ZnCl_2$ forming $Zn(Ar\text{-}BIAN)Cl_2$, followed by the liberation of the Ar-BIAN ligand by addition of a zinc precipitating agent [18,25,27]. We were able to synthesize α-diimines **2** and **3** without the use of $ZnCl_2$ by simply refluxing a toluene solution of 2:1 aniline: acenaphthenequinone with an acid catalyst while removing water using a Dean–Stark trap. By changing to a lower boiling solvent (methanol), reducing the aniline: acenaphthenequinone ratio (1:1), and not removing any by-product water, the analogous α-iminoketones were isolated as **4** and **5**.

The ^1H NMR spectrum of **1** shows that the chemical shift of the backbone methyl groups occurs at δ 2.27 which is slightly upfield from that of 2,3-butanedione at δ 2.33 ppm [28]. Similarly, the chemical shifts of the *tert*-butyl groups of **1** (δ 1.40 and 1.37 ppm) were comparable to the resonances for the *tert*-butyl groups of 2,5-di-*tert*-butylaniline (δ 1.42 and 1.29 ppm) [29]. The room temperature ^1H NMR spectrum of both **4** and **5** show a major and a minor species present in $CDCl_3$; however, a variable temperature ^1H NMR experiment in $CDCl_3$ gave little insight into the solution state dynamics of these compounds. When compounds **4** and **5** were each dissolved in DMSO-d_6, coalescence was observed at approximately 115 °C and a single species was observed above this temperature. Cooling back to room temperature reproduced the original spectrum in both cases, suggesting the presence of an equilibrium in solution. We assign these two species as the *E* and *Z* isomers of the imine, which are present in a (*E*:*Z*) ratio of approximately 13:1 for **4** and 8:1 for **5** at room temperature in $CDCl_3$ (Fig. 3). This ratio is comparable to what was observed by Ragaini for asymmetric Ar,Ar′-BIAN ligands which had solution state (*E*,*E*:*E*,*Z*) ratios of 7:1 [30], 8:1 [15], and 10:1 [30]. A preference for the (*E*,*Z*) isomer was observed in the solid state for bis(1-naphthylimino)acenaphthene by Avilés [31], and R-BIAN (R = *tert*-butyl or 1-adamantyl) by Cowley [32].

3.2. X-ray crystallographic characterization

Compounds **1** and **4**–**6** crystallized in space group $P\bar{1}$ while α-diimines **2** and **3** both crystallized in monoclinic space groups with co-crystallized solvent molecules (Table 1). The crystal packing diagram of **6** shows π-stacking of the naphthalene moiety that extends throughout the lattice, while the crystal packing diagram of **3** shows π-stacking of the naphthalene moiety only between pairs of molecules (as also seen in a molybdenum complex of the ligand [18]) (Fig. 4). The molecular structure of **1** shows a torsion angle formed by N1–C1–C1A–N1A of 180.0° (Fig. 5) which is consistent with the *trans* geometry of other α-diimines derived from 2,3-butanedione (177.5–180.0°) [33–45]. The substituted phenyl groups of **2**–**6** show an orthogonal conformation with respect to the naphthalene moiety which has also been observed with similar α-diimines [11,46–48] and an α-iminoketone [49] derived from acenaphthenequinone (Fig. 5). The α-iminoketones **4**–**6** have an average C1-C2 bond length of 1.54 Å which is slightly longer than the corresponding α-dimines **2** and **3**, and all of which are longer than the C1—C1A bonds of **1** which measures 1.50 Å (Table 2). Conversely, the N1—C1 bond is slightly longer in **1** than the acenaphthene derived imines **2**–**6**. There is experimentally no difference in bond length between N1 and the *ipso* carbon of the aryl ring among the imines, with an average bond length of 1.42 Å.

4. Conclusion

We have shown the synthesis and characterization of new substituted aryl imines **1**, **4**, and **5** and compared structural characterization to previously known aryl imines **2**, **3**, and **6**. Iminoketones **4** and **5** showed dynamic solution state behavior that equilibrates to one isomer at elevated temperature.

Acknowledgements

This work was financially supported by the NSF through the Center for Enabling New Technologies through Catalysis (CENTC, CHE-0650456), the CENTC Elemental Analysis Facility at the University of Rochester, and NSF REU funding.

Appendix A. Supplementary material

NMR data for **1**, **4**, and **5**. CCDC 806882-806887 contain the supplementary crystallographic data for **1**–**6**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.molstruc.2011.02.027.

References

[1] S. Yamada, Coord. Chem. Rev. 190–192 (1999) 537.
[2] S. Patai (Ed.), The Chemistry of the Carbon–Nitrogen Double Bond, The Chemistry of Functional Groups, Wiley & Sons, New York, 1970.
[3] N.J. Hill, I. Vargas-Baca, A.H. Cowley, Dalton Trans. (2009) 240.
[4] P.G. Cozzi, Chem. Soc. Rev. 33 (2004) 410.
[5] M. Shiotsuki, P.S. White, M. Brookhart, J.L. Templeton, J. Am. Chem. Soc. 129 (2007) 4058.
[6] M.E. Bluhm, C. Folli, O. Walter, M. Döring, J. Mol. Catal. A: Chem. 229 (2005) 177.
[7] M. Jeon, S.Y. Kim, Polym. J. 40 (2008) 409.
[8] J. Liu, Y. Li, Y. Li, N. Hu, J. Appl. Polym. Sci. 109 (2008) 700.
[9] H.-K. Luo, H. Schumann, J. Mol. Catal. A: Chem. 227 (2005) 153.
[10] C.S. Popeney, Z. Guan, Macromolecules 43 (2010) 4091.
[11] H. Zou, S. Hu, H. Huang, F. Zhu, Q. Wu, Eur. Polym. J. 43 (2007) 3882.
[12] M. Jeon, C.J. Han, S.Y. Kim, Macromol. Res. 14 (2006) 306.
[13] M.D. Leatherman, M. Brookhart, Macromolecules 34 (2001) 2748.
[14] J.S. Kim, D. Chandran, D.-W. Park, C.-S. Ha, I. Kim, Stud. Surf. Sci. Catal. 172 (2007) 525.
[15] A. Scarel, M.R. Axet, F. Amoroso, F. Ragaini, C.J. Elsevier, A. Holuigue, C. Carfagna, L. Mosca, B. Milani, Organometallics 27 (2008) 1486.
[16] J.S. Owen, J.A. Labinger, J.E. Bercaw, J. Am. Chem. Soc. 128 (2006) 2005.
[17] H.A. Zhong, J.A. Labinger, J.E. Bercaw, J. Am. Chem. Soc. 124 (2002) 1378.

[18] J.C. Alonso, P. Neves, M.J.P. da Silva, S. Quintal, P.D. Vaz, C. Silva, A.A. Valente, P. Ferreira, M.J. Calhorda, V. Félix, M.G.B. Drew, Organometallics 26 (2007) 5548.
[19] F. Ragaini, S. Cenini, E. Borsani, M. Dompé, E. Gallo, M. Moret, Organometallics 20 (2001) 3390.
[20] F. Ragaini, S. Cenini, M. Gasperini, J. Mol. Catal. A: Chem. 174 (2001) 51.
[21] S. Cenini, F. Ragaini, S. Tollari, D. Paone, J. Am. Chem. Soc. 118 (1996) 11964.
[22] V. Appukuttan, J.H. Kim, C.S. Ha, I. Kim, Korean J. Chem. Eng. 25 (2008) 423.
[23] M.S. Brookhart, K. Kunitsky, W. Liu, J.M. Malinoski, L. Wang, Y. Wang, L.K. Johnson, K.A. Kreutzer, S.D. Ittel, E.I. Du Pont de Nemours & Co., USA and University of North Carolina at Chapel Hill. Polymerization of Olefinic Compounds, US Patent 6,710,007, March 23, 2004.
[24] B. Binotti, C. Carfagna, E. Foresti, A. Macchioni, P. Sabatino, C. Zuccaccia, D. Zuccaccia, J. Organomet. Chem. 689 (2004) 647.
[25] M. Gasperini, F. Ragaini, S. Cenini, Organometallics 21 (2002) 2950.
[26] B.M. Schmiege, M.J. Carney, B.L. Small, D.L. Gerlach, J.A. Halfen, Dalton Trans. (2007) 2547.
[27] R. van Asselt, C.J. Elsevier, W.J.J. Smeets, A.L. Spek, R. Benedix, Recl. Trav. Chim. Pays-Bas 113 (1994) 88.
[28] E. Zadok, S. Rubinraut, F. Frolow, Y. Mazur, J. Org. Chem. 50 (1985) 2647.
[29] D.P. Curran, S. Geib, N. DeMello, Tetrahedron 55 (1999) 5681.
[30] M. Gasperini, F. Ragaini, E. Gazzola, A. Caselli, P. Macchi, Dalton Trans. (2004) 3376.
[31] V. Rosa, T. Avilés, G. Aullon, B. Covelo, C. Lodeiro, Inorg. Chem. 47 (2008) 7734.
[32] J.A. Moore, K. Vasudevan, N.J. Hill, G. Reeske, A.H. Cowley, Chem. Commun. (2006) 2913.
[33] H.L. Aberdeen, R.E. Allan, J.D. Crane, Acta Crystallogr. E59 (2003) o1896.
[34] J.A.S. Bomfim, M.L. Dias, C.A.L. Filgueiras, L. do C. Visentin, J. Bordinhao, Acta Crystallogr. E63 (2007) o3150.
[35] H.M. Cho, J.S. Moore, S.R. Wilson, Acta Crystallogr. E61 (2005) o3767.
[36] H.M. Cho, J.S. Moore, S.R. Wilson, Acta Crystallogr. E61 (2005) o3771.
[37] E.K. Cope-Eatough, F.S. Mair, R.G. Pritchard, J.E. Warren, R.J. Woods, Polyhedron 22 (2003) 1447.
[38] L.C. Ferreira, C.A.L. Filgueiras, M. Hörner, L. do C. Visentin, J. Bordinhao, Acta Crystallogr. E62 (2006) o4282.
[39] J.C. Gordon, P. Shukla, A.H. Cowley, J.N. Jones, D.W. Keogh, B.L. Scott, Chem. Commun. (2002) 2710.
[40] A.S. Ionkin, W.J. Marshall, J. Organomet. Chem. 689 (2004) 1057.
[41] A.S. Ionkin, W.J. Marshall, Organometallics 23 (2004) 3276.
[42] N. Kuhn, M. Steimann, I. Walker, Z. Kristallogr. – New Cryst. Struct. 216 (2001) 319.
[43] R.S. Martins, C.A.L. Filgueiras, L. do C. Visentin, J. Bordinhao, L.C. Ferreira, Acta Crystallogr. E63 (2007) o4544.
[44] T. Schaub, U. Radius, Z. Anorg. Allg. Chem. 632 (2006) 807.
[45] H. Zou, Y. Hou, X. Yong, Y. Cen, F. Bao, Acta Crystallogr. E64 (2008) o567.
[46] U. El-Ayaan, F. Murata, S. El-Derby, Y. Fukuda, J. Mol. Struct. 692 (2004) 209.
[47] U. El-Ayaan, A. Paulovicova, Y. Fukuda, J. Mol. Struct. 645 (2003) 205.
[48] H.-R. Liu, P.T. Gomes, S.I. Costa, M.T. Duarte, R. Branquinho, A.C. Fernandes, J.C.W. Chien, R.P. Singh, M.M. Marques, J. Organomet. Chem. 690 (2005) 1314.
[49] A.C. Silvino, L.C. Ferreira, M.L. Dias, C.A.L. Filgueiras, M. Hörner, L. do C. Visentin, J. Bordinhao, Acta Crystallogr. E63 (2007) o999.