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1,1′,3,3′-Tetraalkyl-2,2′-biperimidinylidenes: Unexpected Substituent Effects on the Reactivity of Carbon–Carbon Double Bond

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ABSTRACT: *C_2 deprotonation of 1,3-dibutylperimidinium bromide (***1a***) with sodium hydride and a catalytic amount of potassium* tert*-butoxide in dry THF led to the formation of the exceptionally inert tetraaminoalkene* **2a**. *In contrast, isostructural tetrakis(2-methoxyethyl)-tetraaminoalkene (***2b***) instantaneously reacted with O_2 to yield urea* **3b**, *and silver nitrate was readily reduced with* **2b** *to form a silver mirror. Compound* **2a** *has been characterized by X-ray diffraction studies; the naphtho-pyrimidine skeleton imposes structural constraints and some rigidity to the C=C bonding.* © 2003 Wiley Periodicals, Inc. Heteroatom Chem 14:82–87, 2003; Published online in Wiley InterScience (www.interscience.wiley.com). DOI 10.1002/hc.10088

INTRODUCTION

Exobicylic enetetramines of the type [:$\overline{CN(R)ANR}$]$_2$ (R = primary alkyl; A = CH_2CH_2, $CH_2CH_2CH_2$, or C_6H_4-*o*) are strongly nucleophilic and highly reactive [1–3]. Electrophiles such as dioxygen and HX induce scission of the formal C=C bond (e.g., chemiluminescent behavior) and is typical of *N*-alkyl enetetramines [4]. In this context, a number of carbene complexes have been prepared by the reaction of enetetramines with coordinatively unsaturated transition metal complexes by cleavage of the carbon–carbon bond [5]. The preparation of such carbene complexes from enetetramines has a close analogy with that of tertiary phosphine complexes from PR_3. Some of the carbene complexes, L_nM-$\overline{CN(R)ANR}$, have been found to be good catalysts for a variety of transformations [6–10]. Therefore, we were interested in synthesizing and exploring the chemistry of the hitherto unknown naphtho-fused alkenes **2**. The use of a naphtho-fused pyrimidine skeleton was expected to enhance the rigidity of the derived compounds.

EXPERIMENTAL

Reagents and Techniques

All manipulations of air- and/or moisture-sensitive compounds were carried out under an argon atmosphere by using standard Schlenk techniques. Solvents were dried and freshly distilled under argon before use. IR absorption spectra were obtained from

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a Mattson 1000-FTIR spectrometer in KBr discs, in the range of 400–4000 cm^{-1}. ^1H and ^{13}C NMR spectra were recorded on a Bruker DPX FT NMR spectrometer ($CDCl_3$ or C_6D_6 as internal standards).

*Synthesis of 1,3-Dialkylperimidinium Salts (**1**)*

To a solution of 1-*n*-butylperimidine (3.5 g, 15.6 mmol) in DMF (10 ml) *n*-butyl bromide (2.15 g, 15.68 mmol) was added and the resulting solution was stirred for 12 h at 25°C and then for 12 h at 105°C. Upon addition of diethyl ether (20 ml), crystals of **1a** were obtained. Yield: 4.14 g, 73%. Data for **1a**: mp 243–244°C (dec.), ^1H NMR ($CDCl_3$, 293 K): $\delta = 10.49$ (s, 1H, C2-*H*), 7.41–7.24 (m, 4H, Ar-*H*), 6.74 (d, 1H, $J = 1.3$ Hz, Ar2-*H*), 6.70 (d, 1H, $J = 1.3$ Hz, Ar-*H*), 4.17 (t, 4H, $J = 7.2$ Hz, $NCH_2CH_2CH_2CH_3$), 1.77 (m, 4H, $NCH_2CH_2CH_2CH_3$), 1.45 (quintet, 4H, $J = 7.3$ Hz, $NCH_2CH_2CH_2CH_3$), 0.87 (t, 6H, $J = 7.2$ Hz, $NCH_2CH_2CH_2CH_3$), ^{13}C {H}NMR ($CDCl_3$, 293 K): $\delta = 152.1$ (2-*C*H), 135.2, 131.3, 128.3, 124.5, 121.7 (C_{arene}), 51.3, 28.3, 19.5, 13.7 (C_4H_9). Anal calc for $C_{19}H_{25}N_2Br$: C, 63.16; H, 6.97; N, 7.75. Found: C, 62.95; H, 7.86; N, 7.66; IR ν(CN) 1658.5 cm^{-1}.

A mixture of *N*,*N*′-di(2-methoxyethyl)-1,8-diaminonaphthalene dihydrochloride (4.30 g, 12.47 mmol) and triethyl orthoformate (13.40 g, 90.2 mmol) was heated at 120°C for 30 h. The ethyl alcohol formed was distilled off. The remaining volatiles were removed in vacuo, and the oily residue was washed with Et_2O (20 ml) and crystallized from EtOH/Et_2O (10:20 ml). Yield: 3.17 g, 79%. Data for **1b**: mp 249–250°C (dec.), ^1H NMR ($CDCl_3$, 293 K): $\delta = 9.80$ (s, 1H, C2-*H*), 7.36–7.13 (m, 4H, Ar-*H*), 6.87 (d, $J = 7.3$ Hz, 2H, Ar2-*H*), 4.42 (t, 4H, $J = 4.75$ Hz, $NCH_2CH_2OCH_3$), 3.75 (t, 4H, $J = 7.1$ Hz, $NCH_2CH_2OCH_3$), 3.33 (s, 6H, $NCH_2CH_2OCH_3$), ^{13}C {H}NMR ($CDCl_3$, 293 K): $\delta = 154.7$ (2-*C*H), 135.5, 132.0, 128.5, 124.7, 121.9 (C_{arene}), 67.5 ($CH_2CH_2OCH_3$), 59.4 ($CH_2CH_2OCH_3$), 51.2 ($CH_2CH_2OCH_3$). IR υ(CN) 1662.7 cm^{-1}. Anal calc for $C_{17}H_{21}N_2O_2Cl$: C, 63.37; H, 6.60; N, 8.74. Found: C, 63.2; H, 6.4; N, 8.7.

*Synthesis of 1,1′,3,3′-tetrabutyl-2,2′-biperimidinylidene (**2a**)*

A suspension of **1a** (10 g, 27.7 mmol), NaH (1.25 g, 52 mmol), and $KOBu^t$ (0.4 g, 3.6 mmol) in THF (40 ml) was stirred at 25°C for 24 h and then at 90°C for 6 h. A vigorous H_2 evolution was observed. The resultant mixture was cooled to room temperature and the volatiles were removed in vacuo. The oily residue was treated with toluene (20 ml) and filtered. The volume of the filtrate was reduced to ca. 2 ml and *n*-hexane (10 ml) was added to effect precipitation of lemon–yellow crystals of **2a**. Yield: 4.2 g, 54%. Data for **2a**: mp: 207–208°C. ^1H NMR (C_6D_6, 293 K): $\delta =$ 7.37–7.18 (m, 8H, Ar-*H*), 6.61 (d, 4H, $J = 6.4$ Hz, Ar-*H*-2), 3.70–3.40 (m, 4H, $NCH_2CH_2CH_2CH_3$), 1.70–1.30 (m, 4H, $NCH_2CH_2CH_2CH_3$), 1.05 (m, app. quintet, $J = 7.1$ Hz, 4H, $NCH_2CH_2CH_2CH_3$), 0.47 (t, 6H, $J = 7.1$ Hz, $NCH_2CH_2CH_2CH_3$), ^{13}C {^1H}NMR (C_6D_6, 293 K): $\delta = 142.2$ (C_{alkene}), 136.2, 127.0, 126.4, 117.7, 117.5, 104.8 (C_{arene}), 49.8, 29.9, 20.5, 13.6 (C_4H_9) Anal calc for $C_{38}H_{48}N_4$: C, 81.38; H, 8.63; N, 9.99. Found: C, 80.57; H, 9.69; N, 9.86.

Using a similar procedure, 1,3-bis(2-methoxyethyl)perimidinium chloride (**1b**), (1.9 g, 5.9 mmol), NaH (0.17 g, 7.1 mmol), and $KOBu^t$ (0.1 g, 0.9 mmol) afforded 1,1′,3,3′-tetrakis(2-methoxyethyl)-2,2′-perimidinylidene (**2b**). Yield: 1.2 g, 71%. Data for **2b**: mp. 197–198°C (decom). ^1H NMR (C_6D_6, 293 K): $\delta =$ 7.30–6.98 (m, 8H, Ar-*H*), 6.84–6.65 (m, 4H, Ar-*H*), 4.30 (t, 8H, $J = 7.1$ Hz, $NCH_2CH_2OCH_3$), 3.62 (t, 8H, $J = 7.1$ Hz, $NCH_2CH_2OCH_3$), 3.18 (s, 6H, $NCH_2CH_2OCH_3$), ^{13}C\{^1H\} NMR (C_6D_6, 293 K): $\delta = 141.42$ (C_{alkene}), 135.45, 127.58, 126.16, 119.83, 119.75, 110.37 (C_{arene}), 65.47 ($CH_2CH_2OCH_3$), 58.35 ($CH_2CH_2OCH_3$), 53.62 ($CH_2CH_2OCH_3$).

*Synthesis of 1,3-bis(2-methoxyethyl)perimidine-2-one (**3b**)*

Dried O_2 gas was passed through a solution of **2b** (1.0 g, 1.76 mmol) in hexane (20 ml) and the mixture was stirred for 1 h at 25°C. A white–cream solid precipitated. The crude product recrystallized from *n*-hexane, yielding the product **3b**. Yield: 0.9 g, 85%. Data for **3b**: mp. 122–123°C. ^1H NMR ($CDCl_3$, 293 K): $\delta =$ 7.27–7.15 (m, 4H, Ar-*H*), 6.63 (d, $J = 7.5$ Hz, 2H, Ar2-*H*), 4.13 (t, 4H, $J = 6.25$ Hz, $NCH_2CH_2OCH_3$), 3.63 (t, 4H, $J = 8.25$ Hz, $NCH_2CH_2OCH_3$), 3.31 (s, 6H, $NCH_2CH_2OCH_3$), ^{13}C\{H\} NMR ($CDCl_3$, 293 K): $\delta =$ 177.7 (*C*=O), 137.6, 135.1, 128.2, 128.0, 121.0, 119.8 (C_{arene}), 69.3 ($CH_2CH_2OCH_3$), 59.5 ($CH_2CH_2OCH_3$), 43.7 ($CH_2CH_2OCH_3$), IR υ (C=O) 1660.39 cm^{-1}. Anal calc for $C_{17}H_{20}N_2O_3$: C, 67.98; H, 6.71; N, 9.33. Found: C, 67.6; H, 6.6; N, 9.2.

Crystallography

Suitable crystals for X-ray analyses were obtained by crystallization from *n*-hexane. Experimental data and methods and procedures used to elucidate the structure and other related parameters are given in Table 1. No absorption correction was applied due to the low absorption coefficient. The structure was solved by direct methods, SHELXS86 [11]. Since the

TABLE 1 Experimental Data of Structure Investigation

Compound	$C_{38}H_{48}N_4$
Colour/shape	Yellow/rod shaped
Formula wt.	560.83
Space group	–
T (K)	$P1$
Cell constants	293 $a = 11.426(1)$, $b = 11.710(1)$, $c = 14.379(1)$Å, $\alpha = 70.64(1)$, $\beta = 89.45(1)$, $\gamma = 68.12(1)^\circ$
Cell volume (Å^3)	1670.0(3)
Formula units/unit cell	2
D_{calc} (mg m^{-3})	1.12
μ_{calc} (mm^{-1})	0.06
Diffractometer/scan	Enraf-nonius CAD-4/ω-2θ
Radiation used, graphite monochromator	Mo Kα($\lambda = 0.71073$Å)
Max. Crystal dimen. (mm)	0.30 × 0.25 × 0.18 mm
Standard reflections	3
Decay of standard	<1%
Reflections measured	3808
θ(max) ($^\circ$)	21.1°
Range of h, k, l	$-11 < h < 11$; $-11 < k < 11$; $0 < l < 14$
Number of reflections with I > 2.5 (I)	1163
Corrections applied	Lorentz-Polarization
Computer programs	SHELXS86 [11] MOLEN [12] ORTEP [13]
Source of atomic scattering factor	Int. Table For X-ray Cryst. Vol. IV, 1974 [14]
Structure solution	Direct methods
Treatment of hydrogen atoms	Calculated geometrically and riding model was used
No. of parameters var.	364
Weight	$\omega = 1/(\sigma^2(I) + (0.04F^2)^2)^{1/2}$
GOF	0.86
$R = \|F_o\| - \|F_c\|/\|F_o\|$	0.065
R_w	0.068
$(\Delta\rho)_{max}$ (eÅ^{-3})	0.23
$(\Delta\rho)_{min}$ (eÅ^{-3})	−0.32

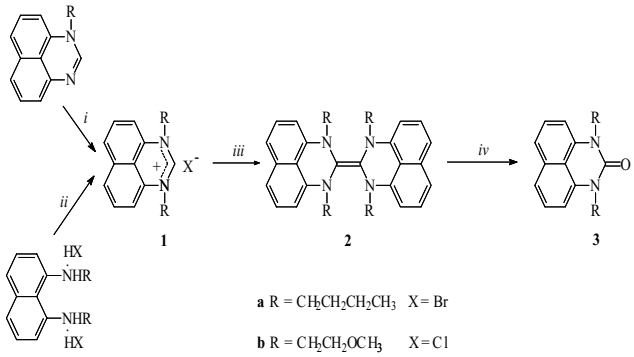
different syntheses did not clarify the positions of the H atoms they were placed in calculated positions at a distance of 0.95 Å from the corresponding C atoms, and a riding model was used in the refinement process of the geometrically calculated H positions.

Unfortunately, the *n*-Bu chains are highly flexible, which may explain the unusually large displacement parameters of the related atoms. As a result of this effect, the C12—C13, C13—C14, C16—C17, and C18—C19 bond lengths were unexpectedly deviated with respect to their conventional values (∼1.54 Å). (Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center, CCDC No. 185236. Copies of this information can be obtained from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1233-336033; e-mail: deposit@ccdc.ac.uk or www:http//www.ccdc.cam.ac.uk).)

RESULTS AND DISCUSSION

Imidazolidinium and benzimidazolinium salts are appropriate starting materials for the synthesis of enetetramines, [:$\overline{CN(R)ANR}$]$_2$, and of free *N*,*N*′-heterocarbenes by C_2 deprotonation with bases [10,15]. Therefore, 1,3-dialkylperimidinium salts were chosen as precursors of the biperimidine-2-ylidene systems. Two distinct routes were employed for the synthesis of salts **1a** and **1b** (Scheme 1): (i) *N*-alkylation of 1-alkylperimidine with alkyl halides and (ii) cyclization of the *N*,*N*′-dialkyldiamines dihydrochlorides with triethyl orthoformate. Although a few more steps are involved, in the case of **1b**, we found that method (ii) is superior to method (i). These salts are characterized by elemental analyses and ^{1}H and ^{13}C NMR spectroscopy (see Experimental section).

The perimidinium salts were subjected to deprotonation to produce the desired alkenes, as shown in Scheme 1. However, much to our surprise and disappointment, the enetetramine **2a** was resistant to oxidation by either dioxygen or silver nitrate in solution, and the starting material was recovered. Next, our attention was directed towards the analogue **2b**, which was exceedingly air sensitive. Thus, when **2b** was exposed to air, it immediately emited a brilliant yellow–green chemiluminescence, which eventually faded as the perimidine-2-one, and thus **3b** was formed. Consequently, identity of **2b** was established through its oxidation product, i.e., **3b** (see Experimental section). As expected, a solution of silver nitrate was instantly reduced by **2b** to give metallic silver. It appears that the variation from the *n*-Bu (**2a**) to on a methoxyethyl substituent **2b** has a dramatic



SCHEME 1 Reagents and reaction conditions: (i) *n*-BuBr, DMF, 110°C, 6 h; (ii) HC(OEt$_3$), 110°C, 24 h; (iii) THF, NaH, KOBu^t (catalytic amount), 25°C, 24 h, 65°C, 6 h; (iv) O_2, hexane, 25°C, 1 h.

impact on the enetetramine reactivity although the steric bulk of these two substituents is almost the same.

In the ^{1}H NMR spectrum of **2a**, the naphtho-protons exhibited a multiplet and a doublet centred at $\delta = 7.27$ and 6.61, respectively. The *n*-Bu methylene protons attached directly to the N atom showed a signal, which appeared to be an AB pair of doublets with additional fine couplings. However, the aforementioned protons in the corresponding salt **1a** appeared as a triplet. The above data collectively suggest that the methylene protons are diastereotopic and the pyrimidine nitrogen atoms are pyramidal, although not necessarily in the solid state (vide infra).

In the ^{13}C NMR spectrum the central C=C exhibited a singlet at $\delta = 142.2$. The corresponding resonance for the enetetramine containing the pyrimidine skeleton occurs at $\delta = 140.7$ [15]. Although elemental analysis and spectroscopic data were consistent with the proposed structure, unambiguous identification of the structure of the product required an X-ray crystallographic analysis.

Crystal Study

A single crystal X-ray structure of compound **2a** is reported to further corroborate the structure assignments. The molecular structure with the atom numbering scheme is shown in Fig. 1. The packing of the molecules in the unit cell is purely due to the van der Waals force of interaction. Selected bond lengths and angles with some torsion angles are given in Table 2. The asymmetric unit contains 2 half molecules, in which they are crystallographically centrosymmetric. The aromatic rings A, B, and D are planar, and E is nearly planar, while the pyrimidine rings C and F are not planar with maximum deviations at C11 [−0.241(10)] and C30 [−0.261(10)Å], from the best least squares planes, respectively. The dihedral angles between the best least squares planes are A/B = 6.3(2.6), A/C = 6.9(2.2), B/C = 7.1(2.2), D/E = 7.6(2.2), D/F = 8.4(1.8), and E/F = 8.9(1.6)°. In the C and F pyrimidine rings, the puckering parameters, i.e. the angles between the best planes [(N1—C1—C10—C9—N2) and (N1—C11—N2)] and [(N3—C20—C29—C28—N4) and (N3—C30—N4)] are 37.4(9) and 39.8(7)° respectively.

The ϕ_{CN} (C11—N1—C12—C13), ϕ_{CN} (C11—N2—C16—C17), ϕ_{CN} (C30—N4—C35—C36), and ϕ_{CN} (C30—N3—C31—C32) torsion angles are 94.2(1.1), −106.2 (1.3), 94.5(1.0), and −103.7(1.0)°, respectively, showing that the *n*-butyl groups attached to the perimidine rings are positioned away from the NNC=CNN plane with two symmetry-related groups pointing in the opposite directions. The central C=C bonds [C11=C11′ 1.35(2) and C30=C30′ 1.32(2)Å] are typical isolated double bonds, and the bonds from N to these central C atoms average 1.39(1) and 1.43(1)Å.

In the solid state, the sums of the bond angles around the N atoms [N1 358.5(9), N2 359.5(9), N3 359.6(7) and N4 358.8(8)°] are very close to 360°, which indicate that the N atoms' surroundings are planar. However, in solution, the ^{1}H NMR data show that the pyrimidine nitrogen atoms are pyramidal.

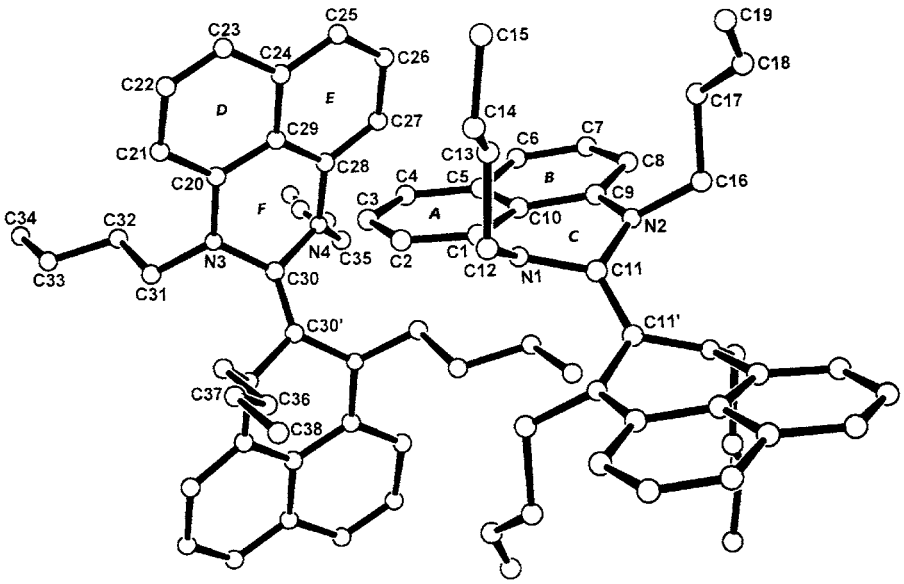


FIGURE 1 An ORTEP [13] drawing of (**2a**) with the atom numbering scheme.

TABLE 2 Selected Bond Lengths (Å) and Angles (°) with Some Torsion Angles (°)

N1—C12	1.46(2)	N3—C31	1.45(1)
N1—C11	1.39(1)	N3—C30	1.41(1)
N1—C1	1.40(1)	N4—C35	1.43(1)
N2—C9	1.38(1)	N4—C30	1.44(1)
N2—C11	1.39(1)	C28—N4	1.38(1)
N2—C16	1.44(2)	C29—C28	1.40(1)
N3—C20	1.40(1)	C29—C20	1.40(2)
C10—C9	1.43(2)	C10—C5	1.41(2)
C10—C1	1.41(2)	C12—C13	1.66(2)
N1—C12—C13	106(1)	C9—C8—C7	120(1)
N1—C1—C2	122(1)	C9—N2—C16	119(1)
N2—C11—N1	113.8(8)	C9—N2—C11	119.6(9)
N2—C9—C8	126(1)	C10—C9—N2	116(1)
N2—C16—C17	117(1)	C10—C9—C8	118(1)
N3—C20—C29	117.8(8)	C10—C1—N1	117(1)
N3—C30—N4	113.3(7)	C10—C1—C2	121(1)
N3—C20—C21	120(1)	C12—N1—C11	118.9(9)
N3—C31—C32	112.2(9)	C11—N2—C16	120.9(9)
N4—C28—C27	123.1(9)	C20—N3—C31	123.4(7)
N4—C35—C36	112.6(7)	C20—N3—C30	117.2(9)
C1—C10—C5	118(1)	C20—C29—C24	118(1)
C1—C2—C3	119(1)	C28—C29—C20	120.5(8)
C1—N1—C12	121.6(9)	C28—C29—C24	121(1)
C1—N1—C11	118(1)	C28—N4—C35	123.6(9)
C2—C3—C4	122(1)	C28—N4—C30	116.1(8)
C4—C5—C6	121(1)	C29—C28—N4	119(1)
C5—C6—C7	120(1)	C29—C28—C27	118(8)
C5—C4—C3	121(1)	C29—C20—C21	122.6(8)
C8—C7—C6	122(1)	C31—N3—C30	119.0(7)
C9—C10—C1	121(1)	C35—N4—C30	119.1(7)
C9—C10—C5	120(1)		
C30—N3—C20—C29	17.3(1.3)	C28—C29—C20—C21	−171.0(1.0)
C20—N3—C31—C32	69.3(1.3)	C24—C29—C20—N3	−177.8(0.9)
C30—N3—C31—C32	−103.7(1.0)	C28—C29—C24—C23	172.9(1.1)
C20—N3—C30—N4	−43.3(1.2)	C20—C29—C24—C25	−176.3(1.0)
C20—C29—C28—N4	−8.3(1.5)	C1—C10—C9—N2	7.7(1.5)
C20—C29—C28—C27	171.2(1.0)	C1—C10—C9—C8	−173.2(1.0)
C24—C29—C28—N4	179.0(1.0)	C5—C10—C9—N2	−178.6(1.0)
C28—C29—C20—N3	9.3(1.5)	C9—C10—C1—N1	−6.4(1.5)

The *n*-Bu groups are effective in determining the molecular geometry. With respect to the molecular structure of **2a** (Fig. 1), each pair of *n*-butyl groups that are attached to the same perimidine ring are almost parallel to each other and trans to the other pair of *n*-butyl groups on the other ring. Dangling of the *n*-Bu groups appears to protect the C=C double bond from electrophilic attack. However, at present, we cannot offer an exact explanation for the reactivity difference between **2a** and **2b**. Furthermore, our attempts to obtain crystals of **2b** suitable for X-ray structure determination have failed so far.

In conclusion, we have reported the synthesis and structure of the first examples of naphthofused enetetramines, one of which is unusually inert towards common electrophiles such as O_2 and $AgNO_3$. However, isostructural **2b** behaves as a typical electron-rich olefin. An investigation of the coordination chemistry of this class of enetetramines, e.g. **2b** is going on in our laboratory.

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