

# Acetylenic NIR-chromophores: 2,3-dialkynyl-1,4-diazabuta-1,3-diene complexes of Ni(0) and Ni(II)

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## Abstract

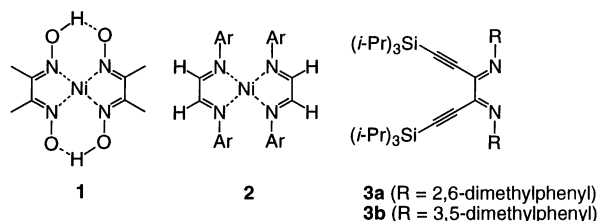
Acetylenic diazabutadienes (DADs) were used to modulate the electron absorption properties of Ni(0) and Ni(II) chelate complexes with respect to those of non-acetylenic DAD metal coordination compounds. Alkynylated bis(*N*-aryl) DAD ligands are shown to induce efficient metal-to-ligand charge-transfer in coordination compounds of the type (DAD)<sub>2</sub>Ni(0). These metal–ligand interactions manifest themselves as intense absorptions in the near infrared (NIR) around 800 nm and are significantly shifted batho- and hyperchromic compared to charge-transfer bands of non-acetylenic DAD metal complexes. Likewise, alkynyl substitution of bis(glyoximate)nickel(II) complexes results in a sizeable absorption band in the visible region of the electromagnetic spectrum. © 1999 Elsevier Science S.A. All rights reserved.

**Keywords:** Alkynes; Charge-transfer; Diazabutadienes; Nickel complexes; UV–vis/NIR

## 1. Introduction

Vicinal diimines (1,4-diazabuta-1,3-dienes, DADs), long known for their multifaceted coordination behaviour to transition metal centers [1,2], have recently re-emerged into the limelight due to their prominent role as ligands in efficient catalyst systems for olefin polymerization reactions [3–6]. A second intriguing aspect of DAD metal complexes, particularly those of nickel, can be found in their electron absorption properties. A well-known example is the intense and diagnostic red colour of solid bis(dimethylglyoximate)-nickel(II) **1** used for gravimetric determinations of nickel(II) [7]. Similarly, tom Dieck et al. [8] have observed that *N*-aryl substituted (DAD)<sub>2</sub>Ni(0) complexes like **2** exhibit sizable metal-to-ligand charge-transfer bands

that extend into the near infrared (NIR). Substances that absorb in the NIR region of the electromagnetic spectrum are currently receiving much attention [9,10], mainly emanating from two directions. On one hand, NIR chromophores can interact with cheap laser diodes (for example, in CD players) that emit between 750 and 830 nm and are hence promising materials for use in optical data storage devices. Secondly, organic tissue is relatively translucent in the NIR region, suggesting applications of NIR chromophores as photosensitizers in photodynamic tumour therapy [11–13].



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In order to modulate the electronic properties of DAD transition metal complexes in general, and in an effort to shift the electron absorptions of DAD–nickel



3c

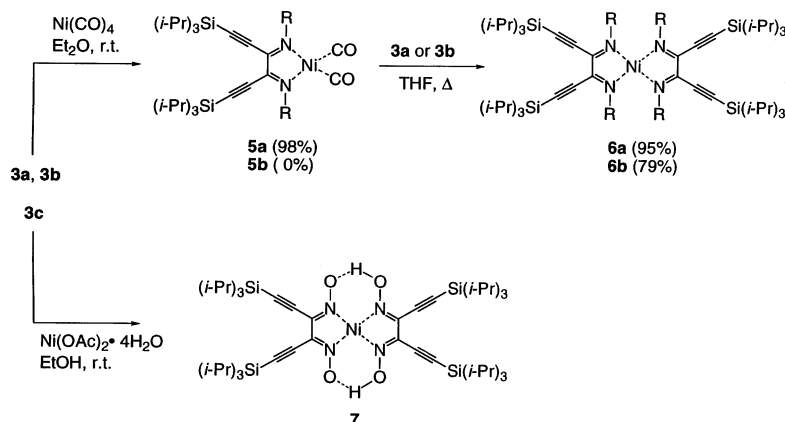
## 2. Results and discussion

Using a smaller hydroxylamine/**4** ratio (e.g. a 3-fold excess) led only to mono-imine formation, to the corresponding  $\alpha$ -hydroximinoketone. The dialkynyl dioxime **3c** was obtained as a colourless solid that is stable to the ambient and that melts without decomposition at 213°C.

shows diagnostic  $^{13}\text{C}$ -NMR resonances in THF at 198.6 (CO), 103.8 and 100.5 ( $\text{C}\equiv\text{C}$ ) ppm. Upon refluxing **5a** with one equivalent of the chelating ligand **3a** in THF for 5 h the initially violet solution turned cherry-red. Removal of the volatiles in vacuo and washing the solid residue with acetone furnished the bis-chelated complex **6a** as a red-brown, microcrystalline solid. In contrast to the mono-DAD complex **5a**, the  $(\text{DAD})_2\text{Ni}(0)$  derivative **6a** can be handled in air for short periods of time and melts under decomposition at  $285^\circ\text{C}$ .

The bis(diethynylglyoximate)nickel(II) derivative **7** was readily obtained in 85% as a red–brown precipitate formed by stirring a solution of **3c** and Ni(OAc)<sub>2</sub> · 4H<sub>2</sub>O in EtOH at r.t. for 10 min (Scheme 2). Analytically pure **7** is an air stable solid that melts under decomposition at 217°C. The presence of hydrogen bridges between the two chelating diimine ligands of **7** is revealed in its <sup>1</sup>H-NMR spectrum by very deshielded hydroxy hydrogen nuclei whose resonance is observed at 17.51 ppm in CDCl<sub>3</sub>.

As stated in the introduction, the influence alkynyl substitution of the DAD ligand system might have on the electron absorption properties of the new nickel complexes was of particular interest. Inspection of the pertinent UV-vis data reveals that acetylenic diimine ligands indeed lead to sizable and bathochromically shifted transitions in all cases. For example, CH<sub>2</sub>Cl<sub>2</sub> solutions of bis(dialkynylglyoximato)nickel(II) complex **7** feature two dominant absorption maxima at wave-

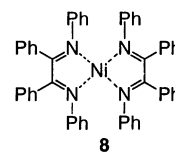


Scheme 2. Preparation of bis(2,3-dialkynyl-1,4-diazabuta-1,3-diene) complexes of Ni(0) and Ni(II).

lengths of 276 and 422 nm with extinction coefficients of 45 300 and 14 700  $\text{M}^{-1} \text{cm}^{-1}$ , respectively. The non-acetylenic nickel(II)dioxime **1**, on the other hand, has just one pronounced absorption maximum at 262 nm ( $\epsilon = 18\,100 \text{ M}^{-1} \text{cm}^{-1}$ ) and an additional, very broad, low-intensity absorption centering around 370 nm ( $\epsilon = 8900 \text{ M}^{-1} \text{cm}^{-1}$ ) in  $\text{CH}_2\text{Cl}_2$ . It should be noted that the characteristic red colour of solid **1** arises from Ni–Ni interactions ( $\lambda = 554 \text{ \AA}$  [18]) in the solid state. The presence of the bulky triisopropylsilyl groups in **7** apparently preclude close metal–metal contacts in the solid and hence no bands beyond 422 nm are observed.

The effect of alkynylation of the DAD ligand becomes even more apparent upon examination of the electron absorption properties of the Ni(0) complexes **6a** and **6b**. Thus, the UV–vis/NIR spectrum of **6a** in  $\text{Et}_2\text{O}$  is dominated by two low-energy absorptions at 531 and 810 nm with extinction coefficients of 18 000 and 16 700  $\text{M}^{-1} \text{cm}^{-1}$ , respectively. Reducing the electron density around the nickel center by moving arylimino methyl groups from positions 2,6 to positions 3,5 leads to a hypso- and hypochromically shifted

longest wavelength absorption of 792 nm ( $\epsilon = 9300 \text{ M}^{-1} \text{cm}^{-1}$ ) for **6b**.



In line with our expectations, the lowest energy charge-transfer band of  $(\text{DAD})_2\text{Ni}(0)$  complexes is drastically shifted into the NIR and considerably enhanced when positions 2 and 3 of the DAD backbone are substituted by alkynyl groups. Comparing the electron absorptions of **6a** with those of bis(*N,N'*-diphenyl-2,3-diphenylethane-1,2-diimine)nickel(0) **8** [19] (Fig. 1) in  $\text{Et}_2\text{O}$  reveals that the latter has only a broad, low-intensity absorption at 744 nm ( $\epsilon = 2400 \text{ M}^{-1} \text{cm}^{-1}$ ). Similar data were reported for non-acetylenic bis [*N,N'*-(2,6-dimethylphenyl)butane-2,3-diimine]nickel(0) ( $\lambda_{\text{max}} = 764 \text{ nm}$ ,  $\epsilon = 13\,100 \text{ M}^{-1} \text{cm}^{-1}$  in  $\text{C}_6\text{H}_6$ ) by tom Dieck et al. [8]. These authors have also provided spectroscopic and structural evidence [8] for the tendency of *N*-aryl substituted  $(\text{DAD})_2\text{Ni}(0)$  complexes to adopt a more planarized coordination sphere around

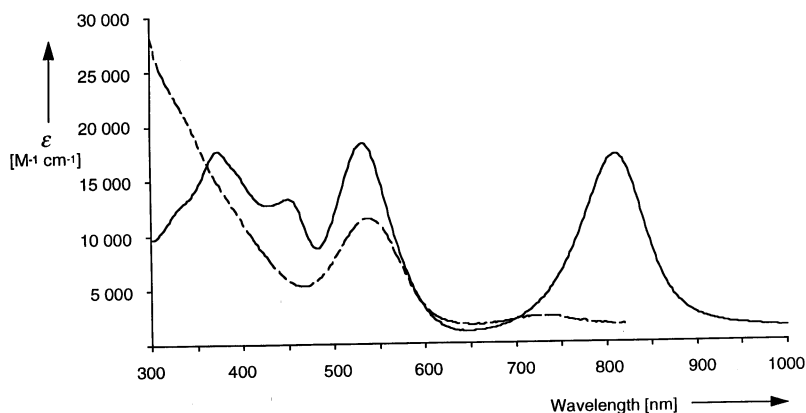


Fig. 1. Electron absorption spectra of  $(\text{DAD})_2\text{Ni}(0)$  complexes **6a** (solid line) and **8** (dashed line) in  $\text{Et}_2\text{O}$  at r.t.

the nickel in which efficient electronic delocalization occurs across the transition metal center. At a qualitative level, it is likely that the expansion of the ligand's  $\pi$  system by 2,3-dialkynyl substitution of the DAD skeleton induces the bathochromic shift and leads to the intense NIR absorptions observed for **6a** and **6b**. Compounds like **8**, on the other hand, do not benefit from an extended  $\pi$  system, since the phenyl groups on positions 2 and 3 cannot be coplanar with the DAD backbone [20] and hence nickel(0) complexes with these ligands do not show pronounced transitions in the NIR.

This work clearly demonstrates that the beneficial properties of alkynyl groups (i.e. small steric demand, electronic interactions with adjacent  $\pi$  systems) can be exploited for the rapid assembly of efficient NIR chromophores from small acetylenic building blocks. Evidently, an orbital-based theoretical investigation is warranted to understand the interaction between transition metal centers and the  $\pi$  system of the 2,3-dialkynyl DADs. Work along those lines is underway as are investigations pertaining to the catalytic properties of coordination compounds with this new ligand system.

### 3. Experimental

General: melting points were determined on a Reichert hotstage and are uncorrected. UV–vis spectra were recorded on Hewlett Packard HP 8452 or HP 8453 UV–vis ChemStation spectrometers. Infrared spectra were obtained as KBr pellets or films on a Perkin–Elmer PE 1600 FT-IR spectrometer.  $^1\text{H}$ -NMR spectra were recorded at 300 MHz on a Varian XL 300 or at 250 MHz on a Bruker WM-250.  $^{13}\text{C}$ -NMR spectra were measured at 62.9 MHz on a Bruker WM-250 spectrometer, at 90.6 MHz on a Bruker AM 360, or at 75.4 MHz on the Varian spectrometer described above. The degree of carbon substitution was determined by  $J$ -modulated spin echo experiments.  $\delta$  values are ppm downfield from internal  $\text{Me}_4\text{Si}$ . Mass spectra were obtained on a Varian MAT-311 A mass spectrometer at 70 eV (EI) or on a JEOL MS-700 (FAB, FD). Elemental analyses were performed on a Foss–Heraeus Vario EL.

Solvents were purified and dried according to standard procedures [21]. Tetrahydrofuran was distilled from sodium benzophenone immediately prior to use. Silica gel (60–200 mesh) for column chromatography was kindly provided by Merck KGaA, Darmstadt. All reactions were performed in predried glass vessels in an inert atmosphere under argon.

#### 3.1. 1,6-Bis(triisopropylsilyl)hexa-1,5-diyne-3,4-dionedioxime (**3c**)

1,6-Bis(triisopropylsilyl)hexa-1,5-diyne-3,4-dione **4** [17] (418 mg, 1 mmol),  $\text{NH}_2\text{OH} \cdot \text{HCl}$  (1.4 g, 20 mmol)

and NaOAc (1.6 g, 20 mmol) were heated to 50°C in acetic acid (5 ml) for 30 min. After cooling, the mixture was poured into water and extracted with  $\text{Et}_2\text{O}$  ( $3 \times 100$  ml). After drying the organic layer over  $\text{Na}_2\text{SO}_4$ , the solvent was evaporated and the residue was chromatographed on silica gel (hexane:ethyl acetate 10:1) to yield, after recrystallization from  $\text{CHCl}_3$ , **3c** (156 mg, 33%) as a colourless solid: m.p. 213°C. —IR (KBr):  $\nu = 3276\text{ cm}^{-1}$  (s, O–H),  $2944\text{ cm}^{-1}$  (s, C–H),  $2866\text{ cm}^{-1}$  (s, C–H),  $2164\text{ cm}^{-1}$  (w,  $\text{C}\equiv\text{C}$ ),  $1591\text{ cm}^{-1}$  (w,  $\text{C}=\text{N}$ ). —UV ( $\text{CH}_2\text{Cl}_2$ ):  $\lambda_{\text{max}}$  ( $\epsilon$ ) = 232 nm (16 400), 256 nm (sh, 15 600). — $^1\text{H}$ -NMR (360 MHz,  $\text{CDCl}_3$ ):  $\delta = 11.66$  (s, 2 H, OH), 1.13 (pseudo-s, 42 H, *i*-Pr). — $^{13}\text{C}$ -NMR (90.56 MHz,  $\text{CDCl}_3$ ):  $\delta = 139.3$  ( $\text{C}=\text{N}$ ), 105.9 ( $\text{C}\equiv\text{C}$ ), 94.7 ( $\text{C}\equiv\text{C}$ ), 18.9 ( $\text{CH}_3$ ), 11.9 (CH). —MS (EI),  $m/z$  (%): 448 (16) [ $M^+$ ], 405 (100) [ $M^+ - i\text{-Pr}$ ], 377 (18), 349 (10). — $\text{C}_{24}\text{H}_{44}\text{N}_2\text{O}_2\text{Si}_2$  (448.29): calcd. C 64.24, H 9.89, N 6.25; found C 64.15, H 9.81, N 6.15.

#### 3.2. *N,N'*-Bis(2,6-dimethylphenyl)-1,6-bis(triisopropylsilyl)hexa-1,5-diyne-3,4-diimine)nickel(0)-dicarbonyl (**5a**)

To a solution of **3a** [16] (310 mg, 0.5 mmol) in diethyl ether (10 ml) was added  $\text{Ni}(\text{CO})_4$  (170 mg, 1.0 mmol). After stirring for 3 h at r.t., the volatiles were removed in vacuo, leaving behind **5a** (370 mg, quant.) as an analytically pure, violet solid: m.p. 232°C (decomp.). —IR (KBr):  $\nu = 2943\text{ cm}^{-1}$  (m, CH), 2865 (m, CH), 2146 (w,  $\text{C}\equiv\text{C}$ ), 2012 (s,  $\text{C}=\text{O}$ ), 1963 (s,  $\text{C}=\text{O}$ ), 1591 (w,  $\text{C}=\text{N}$ ). —UV ( $\text{Et}_2\text{O}$ ):  $\lambda_{\text{max}}$  ( $\epsilon$ ) = 262 nm (15 200), 346 nm (7600), 562 nm (9800). — $^1\text{H}$ -NMR (299.95 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.2$ –7.0 (m, 6H, Aryl-H), 2.36 (s, 12H,  $\text{CH}_3$ ), 1.2–0.9 (m, 42H, *i*-Pr). — $^{13}\text{C}$ -NMR (62.89 MHz,  $\text{THF}/\text{D}_2\text{O}$ ):  $\delta = 198.6$  (CO), 151.4 (C), 143.9 (C), 128.4 (CH), 127.4 (C), 125.2 (CH), 103.8 ( $\text{C}\equiv\text{C}$ ), 100.5 ( $\text{C}\equiv\text{C}$ ), 18.1 ( $\text{CH}_3$ ), 17.2 ( $\text{CH}_3$ ), 11.0 (CH). —MS (FAB)  $m/z$  (%): 710.6 (50), 711.6 (43), 712.6 (64), 713.6 (50) [ $M^+ - \text{CO}$ ], 682.6 (100) [ $M^+ - 2\text{CO}$ ], 625.6 (60) [**3a**], 609.6 (53) [**3a**- $\text{CH}_3$ ]. — $\text{C}_{42}\text{H}_{60}\text{N}_2\text{O}_2\text{Si}_2\text{Ni}$  (739.83): calcd. C 68.19, H 8.17, N 3.79; found C 67.89, H 8.34, N 3.53.

#### 3.3. Bis[*N,N'*-bis(2,6-dimethylphenyl)-1,6-bis(triisopropylsilyl)hexa-1,5-diyne-3,4-diimine]nickel(0) (**6a**)

To a solution of **5a** (370 mg, 0.50 mmol) in THF (10 ml) was added **3a** (310 mg, 0.55 mmol). The reaction mixture was heated to reflux for 5 h, cooled to r.t., and all volatiles were removed in vacuo. The residue was washed with acetone ( $5 \times 10$  ml) and dried in vacuo to furnish **6a** as a red–brown, microcrystalline solid (615 mg, 95%): m.p. 285°C (decomp.). —IR (KBr):  $\nu = 3065\text{ cm}^{-1}$  (w, CH), 2941 (s, CH), 2864 (s, CH), 2131 (s,  $\text{C}\equiv\text{C}$ ), 1583 (w,  $\text{C}=\text{N}$ ). —UV ( $\text{Et}_2\text{O}$ ):  $\lambda_{\text{max}}$  ( $\epsilon$ ) = 371 nm (17 300), 449 nm (13 000), 531 nm (18 000), 810 nm (16 700). — $^1\text{H}$ -NMR (250.13 MHz,  $\text{C}_6\text{D}_6$ ):  $\delta = 7.0$ –6.6

(m, 12H, Aryl-H), 1.99 (s, 24H, CH<sub>3</sub>), 0.8–0.5 (m, 84H, *i*-Pr). —<sup>13</sup>C-NMR (62.89 MHz, THF/D<sub>2</sub>O):  $\delta$  = 156.6 (C), 132.9 (C), 129.0 (C), 128.1 (CH), 125.2 (CH), 111.5 (C≡C), 95.6 (C≡C), 18.3 (CH<sub>3</sub>), 18.1 (CH<sub>3</sub>), 10.3 (CH). —MS (EI),  $m/z$  (%): 1306.7 (8), 1307.7 (10), 1308.7 (9), 1309.7 (7) [ $M^+$ ], 609.4 (100) [ $3a$ -Me<sup>+</sup>], 312.2 (80) [ $3a/2^+$ ]. —C<sub>80</sub>H<sub>120</sub>N<sub>4</sub>Si<sub>4</sub>Ni (1308.92): calcd. C 73.41, H 9.24, N 4.28; found C 73.29, H 9.26, N 4.23.

### 3.4. Bis[N,N'-bis(3,5-dimethylphenyl)-1,6-bis(triisopropylsilyl)hexa-1,5-diyne-3,4-diimine]nickel(0) (**6b**)

To a solution of **3b** [16] (310 mg, 0.5 mmol) in THF (10 ml) was added Ni(CO)<sub>4</sub> (170 mg, 1.0 mmol) and the reaction mixture was heated to reflux for 8 h. During this time, the mixture changed colour from an initial yellow, over violet to cherry-red. After cooling to r.t., the volatiles were removed in vacuo. The residue was washed with acetone (5 × 10 ml) and dried in vacuo to furnish **6b** as a red–brown microcrystalline solid (260 mg, 79%): m.p. 205°C (decomp.). —IR (KBr):  $\nu$  = 3072 cm<sup>-1</sup> (w, CH), 2942 (s, CH), 2864 (s, CH), 2134 (s, C≡C), 1607 (m, C=N), 1590 (m, C=N). —UV (Et<sub>2</sub>O):  $\lambda_{\max}$  ( $\epsilon$ ) = 364 nm (20 000), 406 nm (20 300), 548 nm (22 400), 792 nm (9300). —<sup>1</sup>H-NMR (250.13 MHz, C<sub>6</sub>D<sub>6</sub>):  $\delta$  = 6.89 (s, 4H, Aryl-H), 6.60 (s, 8H, Aryl-H), 2.03 (s, 24H, CH<sub>3</sub>), 0.9–0.5 (m, 84H, *i*-Pr). —<sup>13</sup>C-NMR (62.89 MHz, C<sub>6</sub>D<sub>6</sub>):  $\delta$  = 158.9 (C), 137.8 (C), 131.3 (C), 126.7 (CH), 120.5 (CH), 111.5 (C≡C), 96.8 (C≡C), 21.1 (CH<sub>3</sub>), 18.7 (CH<sub>3</sub>), 10.7 (CH). —MS (FAB),  $m/z$  (%): 1306.1 (52), 1307.1 (54), 1308.1 (77), 1309.1 (82) [ $M^+$ ], 684.6 (73) [ $M^+ - 3b$ ], 626.6 (100) [ $3b^+$ ], 312.2 [ $3b/2^+$ ]. —C<sub>80</sub>H<sub>120</sub>N<sub>4</sub>Si<sub>4</sub>Ni (1308.92): calcd. C 73.41, H 9.24, N 4.28; found C 73.03, H 9.26, N 4.22.

### 3.5. Bis[1,6-bis(triisopropylsilyl)hexa-1,5-diyne-3,4-dioximato]nickel(II) (**7**)

Ni(OAc)<sub>2</sub> · 4H<sub>2</sub>O (10 mg, 0.039 mmol) was dissolved in refluxing ethanol (1 ml). The solution was cooled to r.t., then mixed with a solution of **3c** (35 mg, 0.078 mmol) in ethanol (1 ml). The mixture turned brown and within 10 min a red–brown precipitate of **7** formed, which was collected on a filter, dried in vacuo at 50°C, and recrystallized from Et<sub>2</sub>O (32 mg, 85%): m.p. 217°C (decomp.). —IR (KBr):  $\nu$  = 2944 cm<sup>-1</sup> (s, C–H), 2866 cm<sup>-1</sup> (s, C–H), 2150 cm<sup>-1</sup> (w, C≡C), 1600 cm<sup>-1</sup> (w, C=N). —UV (CH<sub>2</sub>Cl<sub>2</sub>):  $\lambda_{\max}$  ( $\epsilon$ ) = 276 nm (45 300), 376 nm (broad, 8900), 422 nm (14 700). —<sup>1</sup>H-NMR (250.13 MHz, CDCl<sub>3</sub>):  $\delta$  = 17.51 (s, 2 H, OHO),

1.2–0.9 (m, 42 H, *i*-Pr). —<sup>13</sup>C-NMR (62.89 MHz, CDCl<sub>3</sub>):  $\delta$  = 133.6 (C=N), 111.9 (C≡C), 93.4 (C≡C), 18.6 (CH<sub>3</sub>), 11.1 (CH). —MS (FD),  $m/z$  (%): 952 (100) [ $M^+$ ]. —C<sub>48</sub>H<sub>86</sub>N<sub>4</sub>NiO<sub>4</sub>Si<sub>4</sub> (952.51): calcd. C 60.47, H 9.10, N 5.88; found C 60.47, H 9.08, N 5.58.

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