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# Specific formation of isocyanide iron complexes by reaction of primary carbamoyl ferrates with oxalylchloride

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Dedicated in honor of Professor Pierre Braunstein

## Abstract

Reaction of primary carbamoyl ferrates  $\{(CO)_4Fe[C(O)NHR]\}^-$  (R = Me, Et, allyl, decyl, cyclohexyl, t-butyl, benzyl, phenyl) with 1/2 equiv. of oxalylchloride affords *cis*-bis-carbamoyl intermediates:  $(CO)_4Fe[C(O)NHR]_2$  which thermally give rise, in good yields, to the mono-isocyanide complexes  $(CO)_4Fe(CNR)$ . The mechanism of the reaction is discussed. Via a similar process, an alkoxycarbamoyl intermediate  $(CO)_4Fe[C(O)NHR](CO_2Me)$  affords  $Fe(CO)_5$  and 1,3-dialkylurea. © 2003 Elsevier Science B.V. All rights reserved.

Keywords: Iron; Isocyanide; Carbamoyl; Dialkylurea

## 1. Introduction

Bis-alkoxycarbonyl organometallic species have been proposed as intermediates in catalytic processes affording oxalates by oxidative carbonylation of alcohols. For this reason, numerous complexes bearing two alkoxycarbonyl ligands have been prepared and their reactivity widely investigated [1]. Conversely, the chemistry of their bis-carbamoyl homologues is scarcely documented. To date, only a few  $\eta^1$ -bis-carbamoyl complexes of Hg [2a], Ru [2b], Pt [2c] or Pd [2d] stabilized by strong electron donor ancillary ligands and  $\eta^2$ -bis-carbamoyl of U and Th [2e] have been characterized. {W[C(O)Ni- $Pr_{2}_{2}(CO)_{4}^{2-}$  [3a] {(CO)<sub>3</sub>Fe[C(O)NR<sub>2</sub>]<sub>2</sub>}{Sn(CH<sub>3</sub>)<sub>2</sub>} [3b] and  $\{(CO)_3Fe[C(O)NR_2]_2\}_2\{Ti\}$  [3c,3d] are better described as carbene complexes. For our part, we recently reported the rapid thermal transformation of cis-bis-secondary carbamoyl iron complexes  $(CO)_4 Fe[\eta^1 - C(O)NR_2]_2$  (R = Me, Et, n-Pr) into cyclometallacarbenes  $(CO)_3$  Fe[= $C(NR_2)OC(NR_2)O$ ] by a C-O coupling performed between the two carbamoyl ligands of  $(CO)_3Fe[\eta^1-C(O)NR_2][\eta^2-C(O)NR_2]$  inter-

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mediates [4]. This paper describes (1) our attempts to obtain complexes of the *cis*-(CO)<sub>4</sub>Fe[C(O)NHR]<sub>2</sub> series displaying primary carbamoyl ligands with labile N–H bonds by reaction of the appropriate ferrate:  $\{(CO)_4Fe[C(O)NHR]\}^-$  with 1/2 equiv. of oxalylchloride and (2) the transformation of these complexes into mono-isocyanide compounds (CO)<sub>4</sub>Fe(CNR).

#### 2. Experimental

#### 2.1. Material and methods

Fe(CO)<sub>5</sub>, Bu–Li and the primary amines were purchased commercially and used without further purification. (CO)<sub>4</sub>Fe(CO<sub>2</sub>Me)<sub>2</sub> was prepared as described elsewhere [5]. Infrared spectra were obtained in solution on a Nicolet Nexus spectrometer. <sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on Brucker AC 300 and AMX 3-400 spectrometers. Chemical shifts are reported in  $\delta$ units (parts per million) downfield from TMS in CH<sub>2</sub>Cl<sub>2</sub> (external reference) (<sup>1</sup>H) or from the solvent resonances as external references (<sup>13</sup>C). Gas chromatography studies were performed on a Hewlett-Packard 5890 instrument using a CP Sil 25 m capillary column. Elemental analyses were performed by the Centre de Microana-

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lyses du CNRS at Lyon-Solaize, France. All reactions were carried out under an atmosphere of argon using standard Schlenk techniques, and all solvents were purified by distillation under an inert atmosphere from an appropriate drying agent. Yields of reactions are evaluated from recrystallized products and relative to ClC(O)C(O)Cl.

#### 2.2. Syntheses of $(CO)_4Fe(CNR)$ complexes 4

To 8 mmol of primary amine solution in 30 ml of THF at -80 °C, was added 3.2 ml of a 2.5 M solution of n-butyllithium. After 30 min, the temperature of the solution was raised to -10 °C and 1.57 ml (12 mmol) of Fe(CO)<sub>5</sub> solution in 5 ml of THF was introduced dropwise. After 30 min, the temperature of the solution was lowered to -30 °C and 4 mmol (0.35 ml) of oxalylchloride was added. The orange-brown solution rapidly turned yellow. After 1 h, the solvent was evaporated to dryness (for MeNH<sub>2</sub>, EtNH<sub>2</sub>, AllylNH<sub>2</sub>, and t-BuNH<sub>2</sub>, the solvent was trapped with liquid nitrogen). For C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub>, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>NH<sub>2</sub>, C<sub>10</sub>H<sub>21</sub>NH<sub>2</sub> and C<sub>6</sub>H<sub>11</sub>NH<sub>2</sub>, the formed amine was extracted from the residue by two portions of 5 ml of hexane at -40 °C. The presence of amines was evidenced by gas chromatography and <sup>13</sup>C NMR. Complexes 4 were extracted from the residue by two portions of 20 ml of hexane at 0 °C. Crystals were obtained from these solutions after 1 day at -30 °C.

Complex **4a**: R = Me, yield 68% (570 mg). Anal. calc. for FeC<sub>6</sub>H<sub>3</sub>NO<sub>4</sub>: C, 34.49; H, 1.45. Found: C, 34.42; H, 1.51%. IR: 2192, m (*v*CN); 2058, m, 1998, m, 1969 cm<sup>-1</sup>, s (*v*CO). <sup>1</sup>H NMR 3.43 ppm (s). <sup>13</sup>C NMR 213.0, 151.8, 31.0 ppm.

Complex **4b**: R = Et, yield 65% (580 mg). Anal. calc. for FeC<sub>7</sub>H<sub>5</sub>NO<sub>4</sub>: C, 37.70; H, 2.26. Found: C, 37.81; H, 2.32%. IR: 2186, m ( $\nu$ CN); 2059, m, 1995, m, 1968 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 3.72 (2H, q, J = 7.2 Hz), 1.43 ppm (3H, t, J = 7.2 Hz). <sup>13</sup>C NMR 212.0, 154.6, 40.4, 18.9 ppm.

Complex 4c: R = allyl, yield 62% (583 mg). Anal. calc. for FeC<sub>8</sub>H<sub>5</sub>NO<sub>4</sub>: C, 40.89; H, 2.15. Found: C, 40.96; H, 2.17%. IR: 2180, m ( $\nu$ CN); 2060, m, 1997, m,1972 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 5.87 (1H, m), 5.30 (1H, m), 5.25 (1H, m), 3.56 ppm (2H, d, J = 2.5 Hz). <sup>13</sup>C NMR 213.4, 157.8, 128.6, 118.0, 47.8 ppm.

Complex **4d**: R = t-Bu, yield 75% (753 mg). Anal. calc. for FeC<sub>9</sub>H<sub>9</sub>NO<sub>4</sub>: C, 43.05; H, 3.62. Found: C, 43.12; H, 3.58%. IR: 2171, m ( $\nu$ CN); 2056, m, 1994, m, 1967 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 1.48 ppm s. <sup>13</sup>C NMR 213.4, 152.2 (t, *J* = 18 Hz), 59.2, 30.1 ppm.

Complex 4e: R = cyclohexyl, yield 70% (775 mg). Anal. calc. for FeC<sub>11</sub>H<sub>11</sub>NO<sub>4</sub>: C, 47.68; H, 4.01. Found: C, 47.55; H, 4.15%. IR: 2178, m ( $\nu$ CN); 2057, m, 1994, m,1966 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 1.45–2.10 (10H, m); 3.92 ppm (1H, m). <sup>13</sup>C NMR 213.5, 153.9, 56.0, 32.4, 25.1, 22.8 ppm.

Complex **4f**: R = decyl, yield 60% (807 mg). Anal. calc. for FeC<sub>15</sub>H<sub>21</sub>NO<sub>4</sub>: C, 53.60; H, 6.31. Found: C, 53.82; H, 6.38%. IR: 2187, w ( $\nu$ CN); 2059, m, 1994, m, 1967 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 3.66 (2H, m); 0.60–1.75 ppm (19H, m). <sup>13</sup>C NMR 213.4, 154.3, 45.9, 32.0, 29.6, 29.5, 29.4, 29.1, 28.8, 26.4, 22.9, 14.1 ppm.

Complex **4g**: R = benzyl, yield 65% (740 mg). Anal. calc. for FeC<sub>12</sub>H<sub>7</sub>NO<sub>4</sub>: C, 50.55; H, 2.48. Found: C, 50.49; H, 2.39%. IR: 2182, m ( $\nu$ CN); 2061, m, 1999, m, 1972 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 7.4 (5H, m); 4.95 ppm (2H, s). <sup>13</sup>C NMR 213.3, 157.7, 132.4, 129.4, 128.9, 126.8, 49.2 ppm.

Complex **4h**: R = phenyl, yield 63% (683 mg). Anal. calc. for FeC<sub>11</sub>H<sub>5</sub>NO<sub>4</sub>: C, 48.75; H, 1.86. Found: C, 48.65; H, 1.89%. IR: 2155, w ( $\nu$ CN); 2061, m, 1999, m, 1972 cm<sup>-1</sup>, s ( $\nu$ CO). <sup>1</sup>H NMR 7.5 ppm (5H, m). <sup>13</sup>C NMR 212.9, 166.8, 129.8, 129.7, 128.1, 125.7 ppm.

#### 2.3. Exchange reactions from $(CO)_4Fe(CO_2Me)_2$ 1

To a stirred solution of 3 mmol of 1 (858 mg) in THF, 12 mmol of primary amine (900  $\mu$ l of allylamine or 6 ml of a 2 M solution of methyl or ethylamine in THF) was added at 25 °C. After 1 h, the solvent was evaporated to dryness and the residue extracted with two portions of a 1/5 dichloromethane/hexane mixture. The formed 1,3dialkylurea was identified either by gas chromatography or by <sup>13</sup>C by comparison with authentic samples.

## 3. Results and discussion

3.1. Reactions of  $(CO)_4 Fe[C(O)NHR]^-$  with oxalylchloride

We already reported the synthesis of the bis-alkoxycarbonyl complexes  $(CO)_4Fe(CO_2R)_2$  **1** [5] and the achievement of the very unstable bis-carbamoyl  $(CO)_4Fe[C(O)NR_2]_2$  **2** intermediates [4] by reaction of 1/2 equiv. of oxalylchloride with the appropriate alkoxycarbonyl or carbamoyl ferrates { $(CO)_4Fe[C(O)R]$ }<sup>-</sup> R = OR', NR'<sub>2</sub>, themselves obtained by nucleophilic addition of RNa or Rli on a terminal carbonyl of Fe(CO)<sub>5</sub>. As mentioned above, complexes **2** were found to give, at low temperature, metallacarbenes **3** (Scheme 1).

The same reaction performed from  $\{(CO)_4Fe[C-(O)N(H)R]\}^-$  bearing a primary carbamoyl was then supposed to afford *cis*-(CO)\_4Fe[C(O)N(H)R]\_2 **2'** which only differ from **2** by the presence of mobile hydrogen on nitrogen of carbamoyl ligands. Surprisingly, the process was found to afford specifically and in good yield (from 60 to 80%) the iron isocyanide complexes (CO)\_4Fe(CNR) (R = Me, Et, phenyl, benzyl, allyl, t-



Scheme 1.

butyl, decyl and cyclohexyl (4)) together with the corresponding amine (Scheme 2).

It has been known for a long time that isocyanide complexes **4** can be prepared from  $Fe(CO)_5$  and isocyanides or with isocyanide precursors such as isocyanates, phosphine-imines, isocyanide dichlorides or metal trimethylsilyl amides; these reactions often present a low selectivity and then very low yields. However, a specific method of preparation of  $Fe(CO)_{5-n}(CNR)_n$  (n = 1-5), catalyzed by  $CoCl_2$ ·  $H_2O$  has been reported from  $Fe(CO)_5$  and RCN [6]. The transformation of the carbamoyl ligand of  $CpFe(CO)_2\{C(O)N(H)R\}$  into an isocyanide ligand has been performed by phosgene in the presence of an excess of tertiary amines [7] and, more recently, the phosphoramidate conversion of terminal carbonyl ligands into isocyanide ligands has been achieved [8].

The new formation of iron isocyanide complexes (4) by the reaction of carbamoyl ferrates with oxalylchloride seems general as it has been performed from primary amines of different electronic or steric effects (MeNH<sub>2</sub>, EtNH<sub>2</sub>, CH<sub>2</sub>=CH-CH<sub>2</sub>NH<sub>2</sub>, t-BuNH<sub>2</sub>, C<sub>6</sub>H<sub>11</sub>NH<sub>2</sub>, C<sub>6</sub>H<sub>5</sub>NH<sub>2</sub>, C<sub>6</sub>H<sub>5</sub>CH<sub>2</sub>NH<sub>2</sub>). Complexes 4 display spec-



troscopic characteristics identical to those already reported for analogous compounds [6]. In IR, in addition to vC=N stretching frequency between 2155 and 2192 cm<sup>-1</sup>, three  $vC \equiv O$  bands are observed in the vC=0 frequencies area ( $C_3v$  symmetry). <sup>1</sup>H NMR spectra of 4 are also in agreement with the proposed structures. It can be noted that the observed resonances of the ligands are found downfield relative to the free isonitriles. In <sup>13</sup>C NMR, the resonance of the terminal carbonyls is observed as a single signal near 213 ppm. Variable-temperature NMR studies performed on these complexes have shown the occurrence, even at -70 °C, of rapid exchanges between these terminal carbonyls. The isonitrile ligating carbon resonance, generally observed as a broad signal, is found between 151.8 and 166.8 ppm. On the spectrum of the t-Bu isocyanide complex this signal, which should appear as a 1.1.1 triplet from  ${}^{13}C-{}^{14}N$  spin coupling, is observed as a triplet  $(J({}^{13}C - {}^{14}N) = 18 \text{ Hz})$  with a more intense central peak probably resulting from quadrupolar relaxation of the coupling.

# 3.2. Alkoxylamine exchanges from cis-(CO)<sub>4</sub>Fe(CO<sub>2</sub>Me)<sub>2</sub> 1

Another method which was supposed suitable for the preparation of bis-carbamoyl complexes 2' was the alkoxy/amine exchange performed from bis-alkoxycarbonyl complexes (CO)<sub>4</sub>Fe(CO<sub>2</sub>R)<sub>2</sub> (1). This type of exchanges affording carbamoyl ligands has been observed for numerous alkoxycarbonyl complexes [9]. For example, this reaction has been used for the preparation of the bis-carbamoyl compound: Ru(dppe)(CO)<sub>2</sub>-[C(O)Ni-Pr<sub>2</sub>]<sub>2</sub> [2b] and, performed from 1 and secondary amines, the process is another suitable method of synthesis of the unstable *cis*-(CO)<sub>4</sub>Fe[C(O)NR<sub>2</sub>]<sub>2</sub> intermediates [4].

When the exchange was carried out by reacting 1 with an excess (4 equiv.) of primary amine (methyl, ethyl or allylamine), the reaction was found to afford quantitatively Fe(CO)<sub>5</sub> and, respectively, 1,3-dimethyl, diethyl or diallylurea identified by gas chromatography or <sup>13</sup>C NMR by comparison with authentic samples (Scheme 3).

The formation of isocyanide complexes by reaction of a carbamoyl ferrate with oxalylchloride and that of dialkylurea by alkoxy/amine exchanges from bis-alkox-

R' = Me, Et, Allyl

Scheme 2.

Scheme 3.

ycarbonyl complex 1 may be explained by the following considerations.

# 3.3. Mechanisms of the reactions

Similar to the processes performed with  $\{(CO)_4-Fe[CO_2R]\}^-$  and  $\{(CO)_4Fe[C(O)NR_2]\}^-$ , the reaction of the primary carbamoyl ferrates  $\{(CO)_4Fe[C(O)NHR]\}^-$  with 1/2 equiv. of oxalylchloride very likely affords bis-carbamoyl complexes *cis*- $(CO)_4Fe[C(O)N(H)R]_2$  **2**′. These unstable entities would be responsible for the formation of the isocyanide complexes **4** (Scheme 2). A mechanism that could account for the formation of **4** from **2**′ is displayed in Scheme 4.

An intramolecular migration of the labile proton of the amino group of a first carbamoyl ligand of 2'towards oxygen of the second carbamoyl would give rise to an hydroxy amino carbene **5** and to isocyanate. Further elimination of water from **5** would afford the isocyanide complexes **4** and a last reaction between water and isocyanate affords carbamic acid which by decarboxylation gives the amine. Though different (formation of Ru(dppe)(CO)<sub>3</sub> and 1,3-diisopropylurea), the thermal transformation of the close complex *cis*-(dppe)(CO)<sub>2</sub>Ru[C(O)N(H)i-Pr]<sub>2</sub> has been reported to follow a similar mechanism. However, a specific elimination of i-propylamine from an intermediate analogous to 5 affords Ru(dppe)(CO)<sub>3</sub> and not an isocyanide complex formed by elimination of water [10].

The migration of an hydrogen between the two carbamoyl ligands of 2' is not surprising as such an interaction between the proton of a first carbamoyl and the nitrogen of a second carbamoyl has been observed in the solid state and in solution for cis-(dppe)(CO)<sub>2</sub>Ru[C(O)N(H)i-Pr]<sub>2</sub> [2b]. The transformation, involving an intermolecular proton migration, of a carbamoyl ligand inducing the formation of isocyanate is also documented: for example, this reaction has been performed by reaction of CpW(CO)<sub>3</sub>[C(O)N(H)CH<sub>3</sub>] with triethylamine which here plays the part of proton acceptor. Methylisocyanate was then formed together with  $CpW(CO)_3^-NH(C_2H_5)_3^+$ . However, under the same conditions, no reaction was observed for CpFe(CO)<sub>2</sub>[C(O)N(H)R] [11]. Amino mercapto carbenes analogous to 5 have already been suggested as



intermediates in the reaction of formation of isocyanide complexes by the well-known nucleophilic addition of primary amines to thiocarbonyl ligand of Mo [12a], W [12a,12b,12c], Ru [12d] or Fe [12e,12f] complexes. The preparation and the study of these carbenes have allowed to confirm their transformation into isocyanide complexes thus  $CpFe(CO)_2[=C(NHR)(NHR')]^+$  was found to give rise to  $CpFe(CO)_2[(CNR')]^+$  [13]. These reactions involving elimination of  $H_2S$ ,  $H_2O$  or  $RNH_2$  and affording isocyanide ligands are the reverse of the classic reaction of isocyanide complexes with pronucleophile reagents.

In conclusion, it can be noted that if the two steps of the mechanism described in Scheme 4 have already been observed separately, their simultaneous occurrence and then the transformation of two carbamoyl ligands into an isocyanide complex and an amine has never been reported.

As reported above, though supposed to also give rise to the bis-carbamovl intermediates 2', the alkoxy/amine exchanges performed from 1 (Scheme 3) do not afford the same results than the reaction of carbamoyl ferrates with oxalylchloride. We have already shown that, on complex 1, the second alkoxy/secondary amine exchange giving 2 occurs more slowly than the first one. It then becomes credible that the intermediate cis- $(CO)_4Fe(CO_2Me)[C(O)NR_2]$  [4] could be stable enough to present, as observed for 2, a proton exchange, between its two organic ligands. Such a reaction has already been observed for a ruthenium homologue [10]. As shown in Scheme 5, this proton migration would afford an isocyanate and an hydroxy alkoxycarbene whose transformation could give rise to alcohol and Fe(CO)<sub>5</sub>. A further reaction between isocyanate and amine in excess in solution gives 1,3-dialkylurea.

#### 4. Conclusion

This work reports the first description of the thermal evolution of *cis*-bis-primary carbamoyl intermediates into isocyanide compounds. The reaction is a specific and convenient method for the preparation of  $(CO)_4$ Fe(CNR) complexes starting from a large variety



Scheme 5.

of primary amines which are more easily available than the corresponding isocyanides required by other methods. Furthermore, this one-pot process does not require dangerous and air- or moisture-sensitive reagents.

#### References

- J.Y. Salaün, P. Laurent, H. des Abbayes, Coord. Chem. Rev. 178–180 (1998) 353 (and references cited therein).
- [2] (a) U. Schollkopf, F. Gerhart, Angew. Chem. Int. Ed. Engl. 5 (1966) 664;

(b) J.D. Gargulak, W.D. Gladfelter, Inorg. Chem. 33 (1994) 253;
(c) U. Abram, D. Belli Dell'Amico, F. Calderazzo, L. Marchetti, J. Strähle, J. Chem. Soc., Dalton Trans. (1999) 4093;

(d) M. Aresta, P. Giannoccaro, I. Tommasi, A. Dibenedetto, A.M. Manotti Lanfredi, F. Ugozzoli, Organometallics 19 (2000) 3879;

- (e) P.J. Fagan, J.M. Manriquez, S.H. Vollmer, C. Secaur Day, V.W. Day, T.J. Marks, J. Am. Chem. Soc. 103 (1981) 2206.
- [3] (a) E.O. Fischer, R. Reitmeir, K. Ackermann, Z. Naturforsch. B 39 (1984) 668;
  - (b) W. Petz, A. Jonas, J. Organomet. Chem. 120 (1976) 423;
  - (c) W. Petz, J. Organomet. Chem. 456 (1993) 85;

(d) M. Galakhov, A. Martin, M. Mena, F. Palacios, C. Yélamos, Organometallics 14 (1995) 131.

- [4] N. Le Gall, D. Luart, J.Y. Salaün, H. des Abbayes, L. Toupet, J. Organomet. Chem. 617–618 (2001) 483.
- [5] J.Y. Salaün, G. Le Gall, P. Laurent, H. des Abbayes, J. Organomet. Chem. 441 (1992) 99.
- [6] M.O. Albers, N.J. Coville, J. Chem. Soc., Dalton Trans. (1982) 1069.
- [7] W.P. Fehlhammer, A. Mayr, Angew. Chem. Int. Ed. 14 (1975) 757.
- [8] S.E. Gibson, H. Ibrahim, C. Pasquier, M.A. Peplow, J.M. Rushton, J.W. Steed, S. Sur, Chem. Eur. J. 8 (2002) 269.
- [9] (a) R.J. Angelici, Acc. Chem. Res. 5 (1972) 335;
  (b) P.C. Ford, A. Rokicki, Adv. Organomet. Chem. 28 (1988) 139.
- [10] J.D. Gargulak, W.L. Gladfelter, J. Am. Chem. Soc. 116 (1994) 3792.
- [11] W. Jetz, R.J. Angelici, J. Am. Chem. Soc. 94 (1972) 3799.
- [12] (a) B.D. Dombeck, R.J. Angelici, J. Am. Chem. Soc. 95 (1973) 7516;
  - (b) B.D. Dombeck, R.J. Angelici, Inorg. Chem. 15 (1976) 2403;
    (c) W.W. Greaves, R.J. Angelici, Inorg. Chem. 20 (1981) 2983;
    (d) F. Faraone, P. Piraino, V. Marsala, S. Sergi, J. Chem. Soc., Dalton Trans. (1977) 859;
    (e) M.H. Quick, R.J. Angelici, J. Organomet. Chem. 160 (1978) 231;
  - (f) L. Busetto, A. Palazzi, Inorg. Chim. Acta 19 (1976) 233.
- [13] (a) W.P. Fehlhammer, U. Plaia, Z. Naturfosch. 41b (1986) 1005;
  (b) W.P. Fehlhammer, A. Mayr, G. Christian, J. Organomet. Chem. 209 (1981) 57.