

Journal of Organometallic Chemistry, 406 (1991) 363–369
 Elsevier Sequoia S.A., Lausanne
 JOM 21284

Organotransition-metal complexes of multidentate ligands

XII *. Crystal structure of allyl[*N,N*-bis(pyrazol-1-ylmethyl)aminomethane]dicarbonylchromium(II) hexafluorophosphate; the symmetric orientation of the η^3 -allyl group with respect to the pyrazole-derived tridentate ligand

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(Received September 23rd, 1989)

Abstract

The structure of $[\text{Cr}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$ (bpam = *N,N*-bis(pyrazol-1-ylmethyl)aminomethane) has been determined by X-ray diffraction methods. The crystals are orthorhombic, space group *Pbca*, with cell dimensions: $a = 13.430(3)$, $b = 18.530(4)$, $c = 15.565(3)$ Å and $Z = 8$. This structure was refined to give $R = 0.055$ and $R_w = 0.044$ using 1720 reflections with $I > 2\sigma(I)$ in the range of $2 \leq 2\theta \leq 50^\circ$ (Mo- K_α). As expected previously from NMR studies, the compound named in the title is now confirmed to contain, in the solid state, an η^3 -allyl group symmetric with respect to the nitrogen tridentate ligand, bpam. This symmetric structure is also compared with the unsymmetric structure reported for $[\text{Mo}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$ and a new five-stage process is proposed for the stereochemically nonrigid behaviour of this compound and of its tungsten analogue in solution at room temperature.

Introduction

Recently we have described the unusual behaviour in solution of the η^3 -allyldicarbonyl complexes of the Group VI metal atoms, $[\text{M}(\text{N}-\text{N}-\text{N})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$, where M is Cr, Mo, or W and N–N–N is *N,N*-bis(pyrazol-1-ylmethyl)aminomethane, bpam, or *N,N*-bis(3,5-dimethylpyrazol-1-ylmethyl)aminomethane, bdmpam [1g]. Following the assumption that the η^3 -allyl group occupies one coordination site in these pseudooctahedral molecules the allyl group can adopt either symmetric or

* For Parts I–XI, see ref. 1.

asymmetric orientation with respect to the N–N–N ligand. Since the structural characterization of the η^3 -allyl complexes containing tridentate ligands has so far only considered asymmetric systems, [Mo(bpam)(CO)₂(η^3 -allyl)]PF₆ [1g] and (η^3 -2-methylallyl)[hydroxymethylbis(3,5-dimethylpyrazol-1-yl)gallo]dicarbonylmolybdenum(II) [2], it seems unusual that [Cr(bpam)(CO)₂(η^3 -allyl)]PF₆ should take a symmetric structure though this is expected from the NMR data. Single crystals of this complex were therefore subjected to structural analysis. Whether the η^3 -allyl group is symmetric relative to the N–N–N ligand or not is probably an important factor in the subsequent regioselectivity of nucleophilic addition [3,4].

Experimental

Orange-red crystals of [Cr(bpam)(CO)₂(η^3 -allyl)]PF₆, suitable for X-ray diffraction studies, were grown from CH₂Cl₂/hexane at room temperature. They belong to the space group *Pbca*, and the refined cell constants and other crystallographic information are summarized in Table 1. The methods used have been presented elsewhere [1e]. All non-hydrogen atoms were refined anisotropically. The final refinement converged smoothly and no chemically significant peaks were found in the final difference map.

The fractional atomic coordinates of all the non-hydrogen atoms are listed in Table 2. Table 3 contains selected bond lengths and angles for the cation, [Cr(bpam)(CO)₂(η^3 -allyl)]⁺. Tables of fractional atomic coordinates of the hydrogen atoms, the anisotropic temperature factors of all the other atoms, bond lengths

Table 1
Crystallographic data for [Cr(bpam)(CO)₂(η^3 -allyl)]PF₆

| | | | |
|------------------------------------|--|---|-----------------------------------|
| <i>(a) Crystal data</i> | | | |
| Formula | C ₁₄ H ₁₈ CrF ₆ N ₅ O ₂ P | <i>Z</i> | 8 |
| Crystal system | orthorhombic | <i>D</i> _{calc.} , g/cm ³ | 1.66 |
| Space group | <i>Pbca</i> | Color | orange-red |
| <i>a</i> , Å | 13.430(3) | Size, mm | 0.4 × 0.4 × 0.5 |
| <i>b</i> , Å | 18.530(4) | Temp., K | 296 |
| <i>c</i> , Å | 15.565(3) | μ (Mo- <i>K</i> α), mm ⁻¹ | 0.82 |
| <i>V</i> , Å ³ | 3873.5 | <i>T</i> _{max} , <i>T</i> _{min} | 0.9989, 0.9824 |
| Orientation reflns | 24, 19° ≤ 2θ ≤ 31° | | |
| <i>(b) Data collection</i> | | | |
| Diffractometer | Enraf-Nonius CAD-4 | Radiation, λ | Mo- <i>K</i> α , 0.71069 Å |
| Monochromator | graphite | Reflns collected | 3399 |
| Scan type | θ/2θ | Obsd. reflection (2σ(<i>I</i>)) | 1720 |
| Scan range | 2° ≤ 2θ ≤ 50° | Std. reflections | 3 std/1 h |
| Scan speed, deg min ⁻¹ | variable, 2–8 | Std. variation, % | ± 3 |
| <i>(c) Refinement</i> | | | |
| <i>R</i> ^a | 0.055 | Δ/σ(final) | 0.09 |
| <i>R</i> _w ^b | 0.044 | Δ(ρ), e/Å ³ | 0.5 |
| GOF ^c | 2.241 | Number of variables | 263 |

^a $R = \sum ||F_o| - |F_c|| / \sum |F_o|$. ^b $R_w = [\sum w(|F_o| - |F_c|)^2 / \sum w|F_o|]^2$, $w = 1/\sigma^2(|F_o|)$. ^c GOF = $[\sum (w|F_o| - |F_c|)^2 / (NO - NV)]^{1/2}$, where NO = No. of obsd. reflections and NV = No. of variables.

Table 2

Fractional atomic coordinates for $[\text{Cr}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$

| Atom | <i>x</i> | <i>y</i> | <i>z</i> |
|-------|----------|----------|----------|
| Cr | 0.23233 | 0.36749 | 0.06011 |
| N(1) | 0.39101 | 0.36682 | 0.07107 |
| C(2) | 0.46375 | 0.41256 | 0.08477 |
| C(3) | 0.54751 | 0.37778 | 0.11535 |
| C(4) | 0.52404 | 0.30790 | 0.11779 |
| N(5) | 0.42889 | 0.30167 | 0.09241 |
| C(6) | 0.36859 | 0.23985 | 0.07548 |
| N(7) | 0.26478 | 0.25508 | 0.10144 |
| C(8) | 0.25244 | 0.24980 | 0.19549 |
| N(9) | 0.28307 | 0.31690 | 0.23312 |
| C(10) | 0.33288 | 0.32961 | 0.30584 |
| C(11) | 0.33400 | 0.40268 | 0.31753 |
| C(12) | 0.28159 | 0.43071 | 0.24887 |
| N(13) | 0.25085 | 0.37873 | 0.19638 |
| C(14) | 0.20275 | 0.19631 | 0.06269 |
| C(15) | 0.22539 | 0.33312 | -0.05033 |
| O(15) | 0.21952 | 0.30811 | -0.11746 |
| C(16) | 0.09900 | 0.34635 | 0.06055 |
| O(16) | 0.01635 | 0.33017 | 0.05562 |
| C(17) | 0.14160 | 0.47158 | 0.06995 |
| C(18) | 0.23813 | 0.47896 | 0.04314 |
| C(19) | 0.25969 | 0.45511 | -0.03882 |
| P | 0.49747 | 0.09916 | 0.26444 |
| F(1) | 0.50638 | 0.18208 | 0.26821 |
| F(2) | 0.61074 | 0.09508 | 0.28149 |
| F(3) | 0.49142 | 0.01701 | 0.25681 |
| F(4) | 0.38496 | 0.10473 | 0.24483 |
| F(5) | 0.51529 | 0.10326 | 0.16595 |
| F(6) | 0.48221 | 0.09452 | 0.36047 |

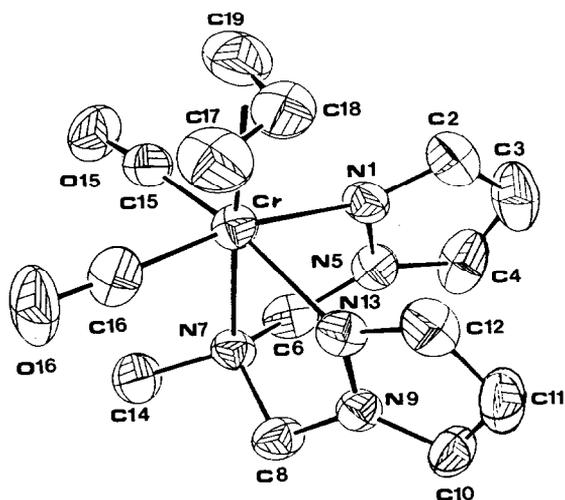


Fig. 1. Structure of the cation, $[\text{Cr}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]^+$ with the numbering scheme. The probability ellipsoids are drawn at the 50% level.

Table 3

Selected bond lengths (Å) and bond angles (deg)

| | | | | | |
|----------------|-----------|-------------------|-----------|-------------|-----------|
| Cr–N(1) | 2.138(5) | Cr–N(7) | 2.223(6) | Cr–N(13) | 2.146(5) |
| Cr–C(15) | 1.836(7) | Cr–C(16) | 1.833(7) | Cr–C(17) | 2.287(8) |
| Cr–C(18) | 2.084(7) | Cr–C(19) | 2.268(8) | N(1)–C(2) | 1.311(9) |
| N(1)–N(5) | 1.352(8) | C(2)–C(3) | 1.381(12) | C(3)–C(4) | 1.333(13) |
| C(4)–N(5) | 1.343(9) | N(5)–C(6) | 1.427(10) | C(6)–N(7) | 1.479(9) |
| N(7)–C(8) | 1.476(8) | N(7)–C(14) | 1.498(9) | C(8)–N(9) | 1.435(9) |
| N(9)–C(10) | 1.336(9) | N(9)–N(13) | 1.352(7) | C(10)–C(11) | 1.366(11) |
| C(11)–C(12) | 1.381(11) | C(12)–N(13) | 1.329(9) | C(15)–O(15) | 1.146(9) |
| C(16)–O(16) | 1.152(9) | C(17)–C(18) | 1.369(12) | C(18)–C(19) | 1.381(12) |
| N(1)–Cr–N(7) | 77.1(2) | C(2)–N(1)–N(5) | 104.9(5) | | |
| N(1)–Cr–N(13) | 78.8(2) | N(1)–C(2)–C(3) | 111.2(7) | | |
| N(1)–Cr–C(15) | 97.1(3) | C(2)–C(3)–C(4) | 105.7(7) | | |
| N(1)–Cr–C(16) | 166.5(3) | C(3)–C(4)–N(5) | 107.5(7) | | |
| N(1)–Cr–C(17) | 122.1(3) | N(1)–N(5)–C(4) | 110.7(6) | | |
| N(1)–Cr–C(18) | 88.8(3) | N(1)–N(5)–C(6) | 117.3(5) | | |
| N(1)–Cr–C(19) | 84.1(3) | C(4)–N(5)–C(6) | 131.6(6) | | |
| N(7)–Cr–N(13) | 77.4(2) | N(5)–C(6)–N(7) | 109.4(5) | | |
| N(7)–Cr–C(15) | 87.5(3) | Cr–N(7)–C(6) | 106.5(4) | | |
| N(7)–Cr–C(16) | 89.4(3) | Cr–N(7)–C(8) | 109.1(4) | | |
| N(7)–Cr–C(17) | 151.1(3) | Cr–N(7)–C(14) | 117.1(4) | | |
| N(7)–Cr–C(18) | 163.3(3) | C(6)–N(7)–C(8) | 111.4(5) | | |
| N(7)–Cr–C(19) | 146.7(3) | C(6)–N(7)–C(14) | 106.0(5) | | |
| N(13)–Cr–C(15) | 164.9(3) | C(8)–N(7)–C(14) | 106.8(5) | | |
| N(13)–Cr–C(16) | 97.5(3) | N(7)–C(8)–N(9) | 108.4(5) | | |
| N(13)–Cr–C(17) | 85.0(3) | C(8)–N(9)–C(10) | 130.0(6) | | |
| N(13)–Cr–C(18) | 91.4(3) | C(8)–N(9)–N(13) | 118.0(5) | | |
| N(13)–Cr–C(19) | 125.7(3) | C(10)–N(9)–N(13) | 111.7(5) | | |
| C(15)–Cr–C(16) | 83.1(3) | N(9)–C(10)–C(11) | 107.0(7) | | |
| C(15)–Cr–C(17) | 109.2(3) | C(10)–C(11)–C(12) | 105.3(6) | | |
| C(15)–Cr–C(18) | 103.1(3) | C(11)–C(12)–N(13) | 111.2(6) | | |
| C(15)–Cr–C(19) | 67.7(3) | Cr–N(13)–N(9) | 111.9(4) | | |
| C(16)–Cr–C(17) | 70.1(3) | Cr–N(13)–C(12) | 135.6(5) | | |
| C(16)–Cr–C(18) | 104.4(3) | N(9)–N(13)–C(12) | 104.8(5) | | |
| C(16)–Cr–C(19) | 108.3(3) | Cr–C(15)–O(15) | 176.3(6) | | |
| C(17)–Cr–C(18) | 36.1(3) | Cr–C(16)–O(16) | 175.1(7) | | |
| C(17)–Cr–C(19) | 61.8(3) | Cr–C(17)–C(18) | 63.8(4) | | |
| C(18)–Cr–C(19) | 36.7(3) | Cr–C(18)–C(17) | 80.0(5) | | |
| Cr–N(1)–C(2) | 138.8(5) | Cr–C(18)–C(19) | 78.9(4) | | |
| Cr–N(1)–N(5) | 113.6(4) | C(17)–C(18)–C(19) | 116.6(8) | | |
| | | Cr–C(19)–C(18) | 64.4(4) | | |

and angles for the anion, PF_6^- , and the listing of structure factor (F_o vs. F_c) are available from the authors. The ORTEP plot of the cation with the numbering scheme is shown in Figure 1.

Results and discussion

As shown in Fig. 1, the η^3 -allyl group is found to be approximately symmetric with respect to the nitrogen tridentate ligand, bpm. Within this approximation is a

mirror plane recognized to consist of the Cr atom, the central saturated nitrogen atom (N_{sat} or N(7)) of bpam, and the central carbon atom (C_c or C(18)) of the η^3 -allyl group ($\text{Cr}-N_{\text{sat}} = 2.223(6)$; $\text{Cr}-C_c = 2.084(7)$ Å). In fact, the two Cr–N distances between the Cr atom and the two terminal unsaturated nitrogen atoms (N_{unsat} or N(1), N(13)) of bpam, and the two Cr–C distances between Cr and the two terminal carbon atoms (C_t or C(17), C(19)) of the allyl group, do not differ significantly from each other, respectively ($\text{Cr}-N_{\text{unsat}} = 2.138(5)$, $2.146(5)$; $\text{Cr}-C_t = 2.287(8)$, $2.268(8)$ Å).

Why does the allyl group in this cation adopt symmetric rather than asymmetric orientation with respect to bpam? If both N_{sat} and N_{unsat} atoms of bpam can be viewed as pure σ -donors [5], the two N_{unsat} atoms of bpam donate apparently more electron density to the central metal atom than the N_{sat} atom as a result of the shorter Cr–N distances. Hence the two carbonyls, which are stronger π -acid ligands than the η^3 -allyl group in the cation, prefer to adopt the positions *trans* to the N_{unsat} atoms so that the negative charge accumulated on the Cr atom by this donation can be more effectively dissipated. Thus, if the above assumption is correct, the preference explains why the compound named in the title is in a symmetric geometry. If not correct, the simple comparison between the Cr^{II}–N distances in this compound and those in the complexes containing the pyrazole- and amine-derived multidentate ligands can reflect the relative electron-donating ability of the N_{sat} and N_{unsat} atoms of bpam. However, the structure is the only one, to the best of our knowledge, to contain both the Cr^{II} atom and the pyrazole-derived ligands. Thus, we need to compare the Cr–N distances with those of the complexes containing similar ligands and Cr atoms in other formal oxidation states. Accordingly, $[\text{Pt}(\text{PET}_3)_2(\mu\text{-pyrazolyl})_2\text{Cr}(\text{CO})_4]$ [6] is found to contain a Cr⁰ atom and “ $\text{Pt}(\text{PET}_3)_2(\text{pyrazolyl})_2$ ” which can be regarded as a metallobidentate ligand, following Luckehart’s definition [7], with $\text{Cr}-N_{\text{unsat}} = 2.14$, 2.16 Å. The distances of $2.183(6)$, $2.185(7)$ and $2.187(7)$ Å found in $[\text{Cr}(\text{dien})(\text{CO})_3]$ (dien = diethylenetriamine) [8] are then used for comparison. Apparently, these values are much smaller than the Cr– N_{sat} distance found in the compound named in the title while the Cr– N_{unsat} distances observed in this Cr^{II} compound are similar to those in the complex of Cr⁰ with the metallobidentate ligand. Whether the shrinking of the metal atom due to the higher oxidation state is considered or not, this comparison also supports the idea that the N_{unsat} atoms are better electron-donors than the N_{sat} atoms in the tridentate ligand, bpam, in $[\text{Cr}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$.

Since a Cr^{II} atom should be smaller than a Cr⁰ atom, the Cr–N distances in this compound being longer than they should be, probably reflects the weak bonding interaction between the metal atom and the ligating atoms, accounting for the heat sensitivity of the compound [1g]. This property may explain why only a few η^3 -allyldicarbonyl complexes of Cr^{II} are known in contrast to numerous examples of Mo^{II} and W^{II} [9,10].

Is there any characteristic difference between the symmetric structure for $[\text{Cr}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$ and the asymmetric structure reported for $[\text{Mo}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$ [1g]? We found some interesting and probably important differences, especially the C–C bond lengths and the inter-carbon angles of the allyl groups in both structures. Structural findings lead us to believe that our previous explanation of there being two different molecules, **A** and **B**, of the Mo compound found in each asymmetric unit of the single crystal of this compound

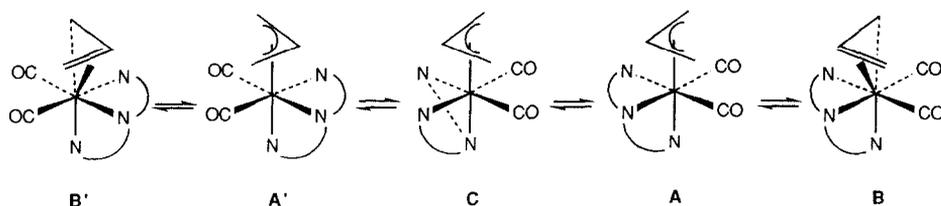


Fig. 2. The nondissociative trigonal twist rearrangement between two pairs of the asymmetric enantiomers, i.e., **A**, **A'** and **B**, **B'**, and the symmetric isomer, **C**, proposed for the cations, $[M(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]^+$ ($M = \text{Mo}, \text{W}$), in solution at room temperature. The bpam ligand is depicted formally as $\text{N}-\text{N}-\text{N}$ (i.e., $\text{N}_{\text{unsat}}-\text{N}_{\text{sat}}-\text{N}_{\text{unsat}}$).

may be erroneous. (Under constraints of the space group $P\bar{1}$ of this crystal, there are two pairs of enantiomers, i.e., **A**, **A'** and **B**, **B'**.) The C_c-C_t bond lengths are 1.369(12) and 1.381(12) in the symmetric structure of the compound named in the title; 1.439(8) and 1.446(8) in the asymmetric **A** structure and 1.405(13) and 1.561(12) Å in another asymmetric **B** structure [11]. Obviously, the allyl group in **B** is bound in a $\sigma-\pi$ mode as a result of the significant difference of 0.16(3) Å. The bond angle of $116.6(6(8))^\circ$ in the compound named in the title is about the average value of $110.3(5)^\circ$ in **A** and $123.2(6)^\circ$ in **B** [11]. Thus, if the co-occurrence of **A** and **B** in the Mo crystal is not due to crystal packing, the stereochemically nonrigid behaviour of $[M(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$ ($M = \text{Mo}, \text{W}$) in solution at room temperature on the NMR time scale is more complicated than that was reported previously [1g]. In brief, if the symmetric structure, **C**, of the Mo complex is similar to that of $[\text{Cr}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$, a new nondissociative-trigonal-twist rearrangement between **A**, **A'**, **B**, **B'** and **C** in solution can be proposed as $\text{B} \rightleftharpoons \text{A} \rightleftharpoons \text{C} \rightleftharpoons \text{A}' \rightleftharpoons \text{B}'$ (Fig. 2). In order to explain that there are two isomers identified by their corresponding ^1H and ^{13}C $\{^1\text{H}\}$ NMR resonances [1g], the rearrangement rate between **C** and **A** (or **A'**) should be much larger or smaller than that between **A** (or **A'**) and **B** (or **B'**).

Further, we believe that the similar scheme may also be applicable for the W complex. In 1980, Faller's group reported that the allyl group in $[\text{Mo}(\eta^5\text{-C}_5\text{H}_5)(\text{NO})\text{I}(\eta^3\text{-allyl})]$ is bound in a similar $\sigma-\pi$ mode but in a symmetric fashion in $[\text{Mo}(\eta^5\text{-C}_5\text{H}_5)(\text{CO})_2(\eta^3\text{-allyl})]$ [12]. It is quite obvious that the considerable electronic asymmetry of the NO and I ligands compared to two CO ligands induces the distorted conversion from the symmetric mode to the $\sigma-\pi$ mode, whereas the (probably) less marked electronic asymmetry of N_{sat} and N_{unsat} donors in $[\text{Mo}(\text{bpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$ [1g] affords a gradual conversion or a three-step process (Fig. 2), i.e., $\text{C} \rightleftharpoons \text{A} \rightleftharpoons \text{B}$ (or $\text{C} \rightleftharpoons \text{A}' \rightleftharpoons \text{B}'$).

Currently, we are trying to grow single crystals of $[\text{Mo}(\text{bdmpam})(\text{CO})_2(\eta^3\text{-allyl})]\text{PF}_6$, which is believed on the NMR data [1g] to be the asymmetric structure. If the solid-state structure of this complex can be obtained, the relative structural details, especially the binding mode of the allyl group, can help to clarify the nonrigid process involving the five stages like $\text{B} \rightleftharpoons \text{A} \rightleftharpoons \text{C} \rightleftharpoons \text{A}' \rightleftharpoons \text{B}'$ (Fig. 2) or the three stages as proposed previously [1g]. Nucleophilic addition experiments on this complex are also under way, and results will be reported in due course.

Acknowledgement

We thank the National Science Council of the Republic of China for financial support of this research.

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