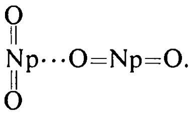
Les Figs. 1(*a*) et 1(*b*) sont deux projections partielles de la structure parallèlement à l'axe *c*. Les cations dont la cote est comprise entre 0 et $\frac{1}{4}$ sont représentés avec leur cortège d'anions sur la Fig. 1(*a*), ceux pour lesquels $\frac{1}{4} < z < \frac{1}{2}$ sur la Fig. 1(*b*). Les atomes de Np et de NH_4 se répartissent en couches séparées par les acides organiques. Les groupements neptunyles sont liés entre eux et constituent des chaînes infinies bidimensionnelles. La Fig. 2 est une projection des groupements neptunyles parallèlement à l'axe *a*; l'abscisse des atomes de Np est voisine de $\frac{1}{4}$. Les actinoïdes présentent la particularité de former des oxocations $(AnO_2)^{n+}$ et il fut montré que ces oxocations interagissent en solution. En particulier, Guillaume, Hahn & Narten (1983) ont évalué à environ 4,2 Å la distance entre atomes de Np dans un complexe 'cation–cation' en phase liquide. Ceci représente la somme des distances d'une liaison neptunyle et d'une liaison non neptunyle, la géométrie attendue pour ces complexes étant:



$$\begin{array}{l} O \\ \| \\ Np\cdots O{=}Np{=}O. \\ \| \\ O \end{array}$$

La structure décrite ici illustre ce phénomène d'interactions entre neptunyles dans un solide et la possibilité de former des polymères de Np ou complexes 'cation–cation' multiples.

Enfin, il faut remarquer que ce complexe a été obtenu sous pression, ce qui n'était pas le cas du composé au sodium. La formule chimique de ce dernier est d'ailleurs très différente puisque le rapport Np/Na = 2/6 alors que $Np/NH_4 = 5/3$ pour un même nombre de molécules organiques. Le rapprochement des cations neptunyles n'est donc peut-être pas dû seulement â la taille de l'élément monovalent et nous nous proposons, dans un premier temps, d'étudier un complexe d'ammonium préparé dans les mêmes conditions que celui de sodium, bien que l'obtention de monocristaux semble difficile. Nous remplacerons ensuite l'ammonium par le thallium de taille plus importante.

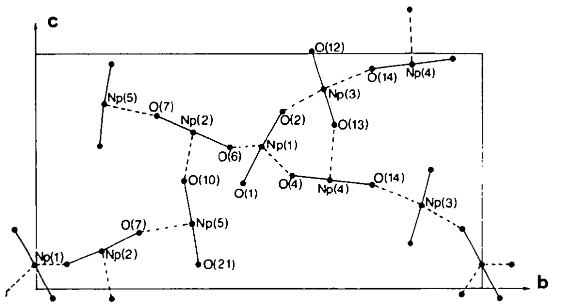


Fig. 2. Projection partielle parallèlement à **a**. Seuls les groupements neptunyles pour lesquels *x* est voision de $\frac{1}{4}$ sont représentés.

Je remercie Françoise Nectoux et Hicham Abazli pour la préparation des monocristaux.

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Dicarbonyl(η^5-cyclopentadienyl)(η^3-*cyclo*-triphosphorus)molybdenum

By Otto J. Scherer, Helmut Sitzmann and Gotthelf Wolmershäuser

Fachbereich Chemie der Universität Kaiserslautern, Erwin-Schrödinger-Strasse, D-6750 Kaiserslautern, Federal Republic of Germany

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Abstract. $[Mo(\eta^5\text{-}C_5H_5)(CO)_2(\eta^3\text{-}P_3)]$, $M_r = 309{\cdot}98$, monoclinic, $P2_1/c$, $a = 11{\cdot}459\ (2)$, $b = 14{\cdot}553\ (2)$, $c = 13{\cdot}603\ (1)$ Å, $\beta = 115{\cdot}462\ (9)°$, $V = 2048{\cdot}1\ (4)$ Å^3, $Z = 8$, $D_x = 2{\cdot}01$ Mg m^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 1{\cdot}548$ mm^{-1}, $F(000) = 1199{\cdot}65$, $T = 293$ K, $R = 0{\cdot}037$ for 2915 observed reflections. The asymmetric

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unit contains two crystallographically independent molecules (*A* and *B*), which do not significantly differ in their individual distances and angles. The three P atoms form an approximately equilateral triangle with P—P 2·127 (mean value for molecule *A*) and 2·134 Å (mean value for molecule *B*). *A* and *B* differ in the conformational arrangement of the C_5H_5 ring with respect to the P_3 unit and the CO groups.

Introduction. Reaction of (η^5-C_5H_5)$(CO)_2$-Mo≡Mo$(CO)_2$(η^5-C_5H_5) with P_4 gives the tetrahedral Mo complexes $Cp_2Mo_2(CO)_4(\mu\text{-}\eta^2\text{-}P_2)$ (1) and CpMo-$(CO)_2(\eta^3\text{-}P_3)$ (2). The structure of the P_2 complex has been elucidated by an X-ray structure analysis (Scherer, Sitzmann & Wolmershäuser, 1984). Temperature-dependent $^{31}P\{^1H\}$ NMR studies for (2) show that even at 173 K a singlet is observed for the P_3 ligand (Sitzmann & Scherer, 1984).

Experimental. Crystals obtained from *n*-pentane, mounted in capillaries; crystal size 0·41 × 0·58 × 0·11 mm. Enraf–Nonius CAD-4 diffractometer. Unit-cell parameters refined from θ values of 25 reflections ($19{\cdot}4 < \theta < 24{\cdot}9°$). Semi-empirical absorption corrections (*SDP* package) based on azimuthal scan data of 9 reflections; min. transmission 0·52, max. 0·63. θ–2θ scans, $5 < 2\theta < 50°$. 3762 measured reflections, 2915 observed independent reflections with $I > 2\sigma(I)$, index range h −13→13, k 0→17, l 0→16. Intensities of three standard reflections decreased by 8% in 58 h and were used to correct data. Mo positions from Patterson map, other non-H atoms from Fourier maps. Least-squares refinement (on *F*) of 275 parameters with *SHELX*76 (Sheldrick, 1976); anisotropic thermal parameters for non-H atoms; H atoms at calculated positions and refined with isotropic thermal parameters. $R = 0{\cdot}037$, $wR = 0{\cdot}045$, $w^{-1} = \sigma^2(F) + 0{\cdot}0005F^2$. Max. and min. electron density on final difference map 0·76 and 0·70 e Å^{-3}; max. Δ/σ (for non-H atoms) 0·11. No correction for secondary extinction. Neutral atomic scattering factors with anomalous-dispersion contributions from *International Tables for X-ray Crystallography* (1974).

Discussion. Atom coordinates are given in Table 1, selected bond lengths and angles in Table 2.*

The molecular structure of (2) (molecule *A*) is shown in Fig. 1. Fig. 2 shows the conformational differences of the two crystallographically independent molecules *A* and *B*. The Mo atom together with the three P atoms form a trigonal pyramid. The P_3 base is a nearly

Table 1. *Positional parameters* (×10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3)

	x	*y*	*z*	U_{eq}*
Molecule *A*				
Mo	−287 (1)	−939 (1)	2401 (1)	33 (1)
P(1)	1871 (2)	−1400 (1)	2487 (1)	65 (1)
P(2)	1209 (2)	−113 (1)	1730 (2)	63 (1)
P(3)	327 (2)	−1331 (1)	878 (1)	60 (1)
C(1)	618 (5)	−31 (4)	3589 (4)	45 (2)
O(1)	1100 (4)	473 (3)	4290 (3)	70 (2)
C(2)	−1348 (5)	91 (4)	1507 (4)	41 (2)
O(2)	−2005 (4)	677 (3)	1003 (3)	66 (2)
C(10)	−640 (8)	−1737 (5)	3701 (5)	72 (3)
C(11)	−359 (8)	−2397 (5)	3080 (7)	84 (3)
C(12)	−1362 (9)	−2353 (5)	2013 (6)	83 (3)
C(13)	−2206 (7)	−1683 (5)	1988 (6)	70 (3)
C(14)	−1774 (7)	−1308 (5)	3037 (6)	69 (3)
Molecule *B*				
Mo	−4711 (1)	1504 (1)	2633 (1)	28 (1)
P(1)	−5906 (2)	1058 (1)	3742 (1)	49 (1)
P(2)	−3852 (2)	1063 (1)	4654 (1)	54 (1)
P(3)	−4766 (2)	−5 (1)	3514 (1)	53 (1)
C(1)	−4386 (5)	2717 (4)	3378 (4)	42 (2)
O(1)	−4252 (5)	3430 (3)	3765 (3)	70 (2)
C(2)	−2796 (5)	1353 (4)	3175 (4)	44 (2)
O(2)	−1730 (4)	1279 (4)	3431 (4)	75 (2)
C(10)	−5955 (7)	2320 (4)	1073 (4)	63 (2)
C(11)	−6721 (5)	1595 (4)	1115 (4)	49 (2)
C(12)	−6138 (6)	790 (4)	1031 (4)	48 (2)
C(13)	−4994 (6)	1012 (5)	937 (4)	59 (2)
C(14)	−4888 (7)	1962 (6)	967 (4)	70 (3)

* The equivalent isotropic *U* is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

	Molecule *A*	Molecule *B*
Mo—P(1)	2·523 (2)	2·523 (1)
Mo—P(2)	2·567 (2)	2·565 (1)
Mo—P(3)	2·522 (2)	2·518 (1)
Mean value	2·538	2·535
P(1)—P(2)	2·117 (3)	2·140 (2)
P(1)—P(3)	2·141 (2)	2·137 (2)
P(2)—P(3)	2·123 (3)	2·126 (2)
Mean value	2·127	2·134
C(1)—Mo	1·997 (5)	1·991 (5)
C(2)—Mo	1·985 (5)	2·009 (5)
C(1)—O(1)	1·139 (6)	1·144 (6)
C(2)—O(2)	1·149 (6)	1·125 (6)
C(C_5H_5)—Mo	2·293 (6) −2·344 (6)	2·281 (5)–2·344 (5)
C—C(C_5H_5)	1·367 (10)–1·411 (11)	1·379 (8)–1·413 (8)
P(1)—Mo—P(2)	49·1 (1)	49·7 (1)
P(1)—Mo—P(3)	50·2 (1)	50·2 (1)
P(2)—Mo—P(3)	49·3 (1)	49·4 (1)
Mo—P(1)—P(3)	64·9 (1)	64·8 (1)
Mo—P(1)—P(2)	66·5 (1)	66·2 (1)
Mo—P(2)—P(3)	64·3 (1)	64·1 (1)
P(1)—P(3)—Mo	64·9 (1)	65·0 (1)
P(2)—P(3)—Mo	66·5 (1)	66·4 (1)
P(1)—P(2)—Mo	64·4 (1)	64·1 (1)
P(1)—P(2)—P(3)	60·7 (1)	60·1 (1)
P(1)—P(3)—P(2)	59·8 (1)	60·3 (1)
P(2)—P(1)—P(3)	59·8 (1)	59·6 (1)
C(1)—Mo—C(2)	87·8 (2)	88·6 (2)
O(1)—C(1)—Mo	176·9 (5)	176·7 (5)
O(2)—C(2)—Mo	177·0 (5)	176·7 (5)
C_5H_5(cent.)—Mo—P_3(cent.)	134·9 (6)	134·0 (5)
C(1)—Mo—C_5H_5(cent.)	114·7 (6)	115·5 (5)
C(2)—Mo—C_5H_5(cent.)	114·7 (6)	116·4 (5)
C(1)—Mo—P_3(cent.)	97·4 (2)	96·6 (2)
C(2)—Mo—P_3(cent.)	96·6 (2)	95·3 (2)

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* Lists of bond distances and angles, anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42470 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

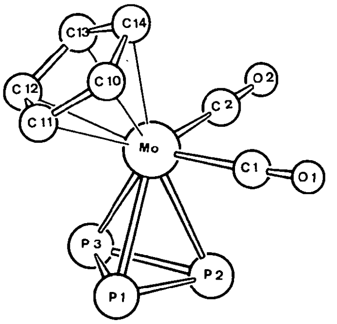

Fig. 1. Molecular structure of (2) (molecule *A*), showing the numbering scheme (H atoms omitted).

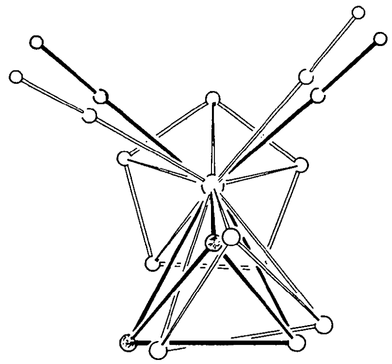

Fig. 2. Projection of molecules *A* (full bonds) and *B* on to the C_5H_5(Cp) plane.

equilateral triangle with mean P—P bond lengths of 2·127 (molecule *A*) and 2·134 Å (molecule *B*). These values are in good agreement with that of $[(\text{triphos})M(\eta^3\text{-}P_3)]$,[*] P—P 2·141 (2) Å, M = Co; 2·152 (2) Å, M = Rh; 2·159 (4) Å, M = Ir, as well as with that of $[(np_3)Co(\eta^3\text{-}P_3)]$, P—P 2·135 (6) Å (Di Vaira & Sacconi, 1982). Shorter P—P bond lengths are found for the P_2 complexes $[Cp_2Mo_2(CO)_4(\mu\text{-}\eta^2\text{-}P_2)]$ (1), P—P 2·079 (2) Å (Scherer, Sitzmann & Wolmershauser, 1984) and $[Co_2(CO)_5(PPh_3)(\mu\text{-}\eta^2\text{-}P_2)]$, P—P 2·019 (9) Å (Campana, Vizi-Orosz, Pályi, Markó & Dahl, 1979). With respect to the P_2 complex (1) (mean value P—Mo 2·508 Å) the mean P—Mo bond distance for (2) is longer (molecule *A* 2·538 Å, molecule *B* 2·535 Å).

In contrast to molecule *B*, molecule *A* has almost C_s symmetry (Fig. 2). For (2) the C_5H_5(cent.)—Mo—P_3(cent.) angle of 134·9 (6)° (molecule *A*) and 134·0 (5)° (molecule *B*) differs only slightly from that of $(\eta^5\text{-}C_5Me_5)Mo(CO)_2(\eta^3\text{-}As_3)$ with a value of 136 (2)° (Bernal, Brunner, Meier, Pfisterer, Wachter & Ziegler, 1984). A striking difference is found between the bond angles C_5H_5(cent.)—Mo—C(1,2) (mean value of molecules *A* and *B* 115·3°) and P_3(cent.)—Mo—C(1,2) (mean value of molecules *A* and *B* 96·5°) of the P_3 complex compared with those of the As_3 complex [C_5Me_5(cent.)—Mo—C(1) 96·4 (4)°, As_3(cent.)—Mo—C(1) 113·5 (2)°] (Bernal *et al.*, 1984).

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[*] Abbreviations: triphos = 1,1,1-tris(diphenylphosphinomethyl)-ethane; np_3 = tris(2-diphenylphosphinoethyl)amine; Cp = cyclopentadienyl.

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Structure of Bis(formamide)formatozinc(II) Formate

BY SUZANNE FORTIER

Department of Chemistry, Queen's University, Kingston, Canada K7L 3N6

AND KATHERINE A. M. CREBER

Department of Chemistry and Chemical Engineering, Royal Military College of Canada, Kingston, Canada K7L 2W3

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Abstract. $[Zn(CHO_2)(CH_3NO)_2]CHO_2$, M_r = 245·49, orthorhombic, *Pnn*2, a = 11·617 (10), b = 8·717 (3), c = 8·289 (2) Å, V = 839·4 Å^3, Z = 4, D_x = 1·942 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 3·00 mm^{-1}, $F(000)$ = 496, T = 292 K, final R = 0·036 for 1914 observed reflections. Bis(formamide)formato-