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The influence of the cation on the conformation of the anion μ-hydridobis[pentacarbonylchromium(0)]: crystal and molecular structure of $[(phenH)_2Cl][Cr_2(CO)_{10}(\mu\text{-}H)]$ and $[K(phen)_2][Cr_2(CO)_{10}(\mu\text{-}H)]$

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Abstract

The crystal and molecular structures of $[(phenH)_2Cl][Cr_2(CO)_{10}(\mu\text{-}H)]$ (**1**) and $[K(phen)_2][Cr_2(CO)_{10}(\mu\text{-}H)]$ (**2**) have been determined by single crystal X-ray diffraction. The availability of the potassium cation for the carbonyl oxygens coordination influences the conformation of the anion in **2** as demonstrated by the comparison with the non-coordinating $[(phenH)_2Cl]$ cation in **1** and with literature data. © 1999 Elsevier Science S.A. All rights reserved.

Keywords: Chromium carbonyls; Conformation; Crystal structure

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1. Introduction

In our previous studies on the reactivity of the $[Cr_2(CO)_{10}(\mu\text{-}H)]^-$ complex species, we have isolated various derivatives of this anion, which provided infrared and Raman evidence for the ability of the latter to undergo in the solid state distortion of its three-center electron-deficient Cr–H–Cr system to a variable extent, even significant, depending on the nature of the associated counter ion [1,2] as confirmed by the X-ray structural characterization of the K^+ and of the $[K(phen)_3]^+$ derivatives [3,4].

The $[(phenH)_2Cl][(Cr_2(CO)_{10}(\mu\text{-}H)]$ derivative (**1**) isolated unexpectedly by the $[K(phen)_3]Cr_2(CO)_{10}(\mu\text{-}H)]$/HCl interaction ([1]b), exhibited a solid state IR spectrum in the ν(CO) region comparable with that of the chlorine bridged species, $[Cr_2(CO)_{10}(\mu\text{-}Cl)]^-$, in its PPN^+ derivatives [5] but different from that of $[K(phen)_3][(Cr(CO)_5)_2(\mu\text{-}H)]$ ([1]a).

$[K(phen)_2][(Cr_2(CO)_{10}(\mu\text{-}H)]$ (**2**) obtained by partial dissociation of $[K(phen)_3][(Cr_2(CO)_{10}(\mu\text{-}H)]$ ([1]a) has a solid state IR spectrum still different, indicating other possible conformation changes in the anion probably induced by the different cation $[K(phen)_2]^+$.

The crystal and molecular structures of (**1**) and (**2**) have been determined in order to clarify the anion geometry in presence of possible perturbing cation species.

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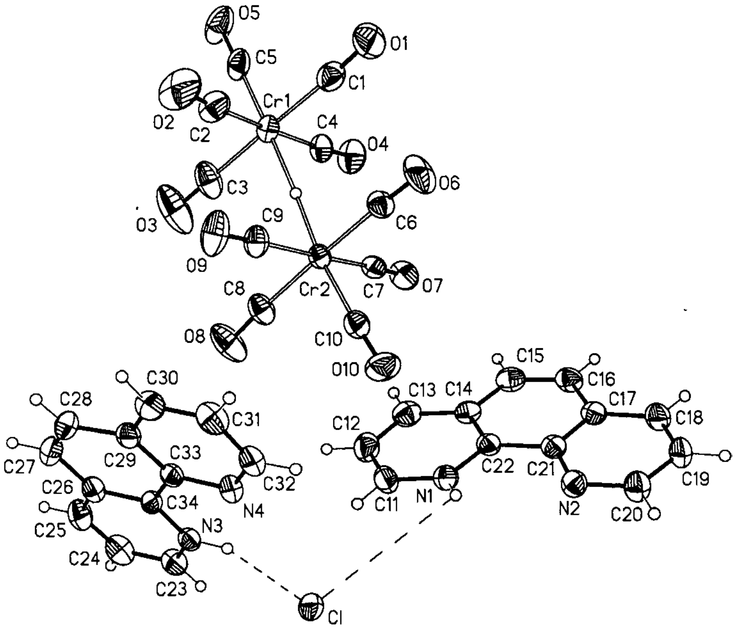

Fig. 1. Molecular structure of the cation and anion in [(phenH)$_2$Cl][Cr$_2$(CO)$_{10}$(μ-H)].

Table 1
Selected bond lengths (Å) and angles (°) for [(phenH)$_2$Cl][Cr$_2$(CO)$_{10}$(μ-H)] (**1**)

Bond lengths (Å)			
Cr(1)···Cr(2)	3.371(1)	Cr(1)–C(2)	1.881(4)
Cr(1)–C(1)	1.886(5)	Cr(1)–C(4)	1.895(4)
Cr(1)–C(3)	1.880(5)	Cr(1)–H	1.72(4)
Cr(1)–C(5)$_{\mathrm{ax.}}$	1.818(3)	Cr(2)–C(7)	1.907(4)
Cr(2)–C(6)	1.885(4)	Cr(2)–C(9)	1.882(4)
Cr(2)–C(8)	1.888(5)	Cr(2)–H	1.66(4)
Cr(2)–C(10)$_{\mathrm{ax.}}$	1.842(3)	O(2)–C(2)	1.141(6)
O(1)–C(1)	1.141(6)	O(4)–C(4)	1.136(5)
O(3)–C(3)	1.139(7)	O(6)–C(6)	1.132(6)
O(5)–C(5)	1.168(4)	O(8)–C(8)	1.141(6)
O(7)–C(7)	1.137(5)	O(10)–C(10)	1.151(4)
O(9)–C(9)	1.140(5)	N(1)–C(22)	1.366(5)
N(1)–C(11)	1.318(5)	C(20)–N(2)	1.322(5)
C(21)–C(22)	1.426(5)	N(2)–C(21)	1.356(3)
N(3)–C(34)	1.352(4)	N(3)–C(23)	1.321(5)
C(32)–N(4)	1.325(5)	C(33)–C(34)	1.441(5)
N(4)–C(33)	1.349(4)		
Bond angles (°)			
Cr(1)–H–Cr(2)	174(3)	Cr(1)–C(1)–O(1)	175.8(4)
Cr(1)–C(2)–O(2)	176.5(4)	Cr(1)–C(3)–O(3)	178.4(4)
Cr(1)–C(4)–O(4)	177.0(4)	Cr(1)–C(5)–O(5)	178.7(4)
Cr(2)–C(6)–O(6)	176.2(4)	Cr(2)–C(7)–O(7)	177.5(4)
Cr(2)–C(8)–O(8)	176.0(4)	Cr(2)–C(9)–O(9)	178.3(4)
Cr(2)–C(10)–O(10)	177.4(4)	C(11)–N(1)–C(22)	123.7(4)
C(20)–N(2)–C(21)	116.4(3)	N(2)–C(21)–C(22)	118.4(3)
N(1)–C(22)–C(21)	120.4(3)	C(23)–N(3)–C(34)	123.0(4)
C(32)–N(4)–C(33)	116.2(4)	N(4)–C(33)–C(34)	117.6(4)
N(3)–C(34)–C(33)	120.1(4)		

2. Results and discussion

The molecular structure of [(phenH)$_2$Cl][Cr$_2$(CO)$_{10}$(μ-H)] (**1**) is shown in Fig. 1 and in Table 1 are reported its significant geometrical parameters.

The compound consists of discrete cation [(phenH)$_2$Cl]$^+$ and [Cr$_2$(CO)$_{10}$(μ-H)]$^-$ anion units. The anion presents an approximately eclipsed conformation (the twisting angle is 4°) of the carbonyl groups of the two chromium coordination spheres. The bridging hydrogen is (in the limit of the estimated S.D.) linearly connected to the two chromium ions (the Cr–H–Cr angle is 174(3)°) with a Cr···Cr separation of 3.371(1) Å. The Cr–C$_{\mathrm{eq.}}$ are on average 1.888(4) Å longer than Cr–C$_{\mathrm{ax.}}$ 1.830(4) Å as expected for derivatives having the same anion conformation (see Table 3) attributed to a greater electron back-donation from the metal centers to the axial C atoms.

The complex cation [(phenH)$_2$Cl]$^+$ has strong hydrogen bonds interactions between the chlorine and the hydrogens of the two protonated phenantrolines (Cl···H(1) 2.41(3) and Cl···H(3) 2.24(4) Å).

These results appear to exclude the preliminary assumption of a possible acid–base Cl$^-$/Cr–H–Cr interaction ([1]b). Cl$^-$ is inserted in the crystal lattice of the anion producing the [(phenH)$_2$Cl]$^+$ entity which does not interact with the anion.

Compound **2** [K(phen)$_2$][Cr$_2$(CO)$_{10}$(μ-H)] is constituted by [K(phen)$_2$]$^+$ cations and [Cr$_2$(CO)$_{10}$(μ-H)]$^-$

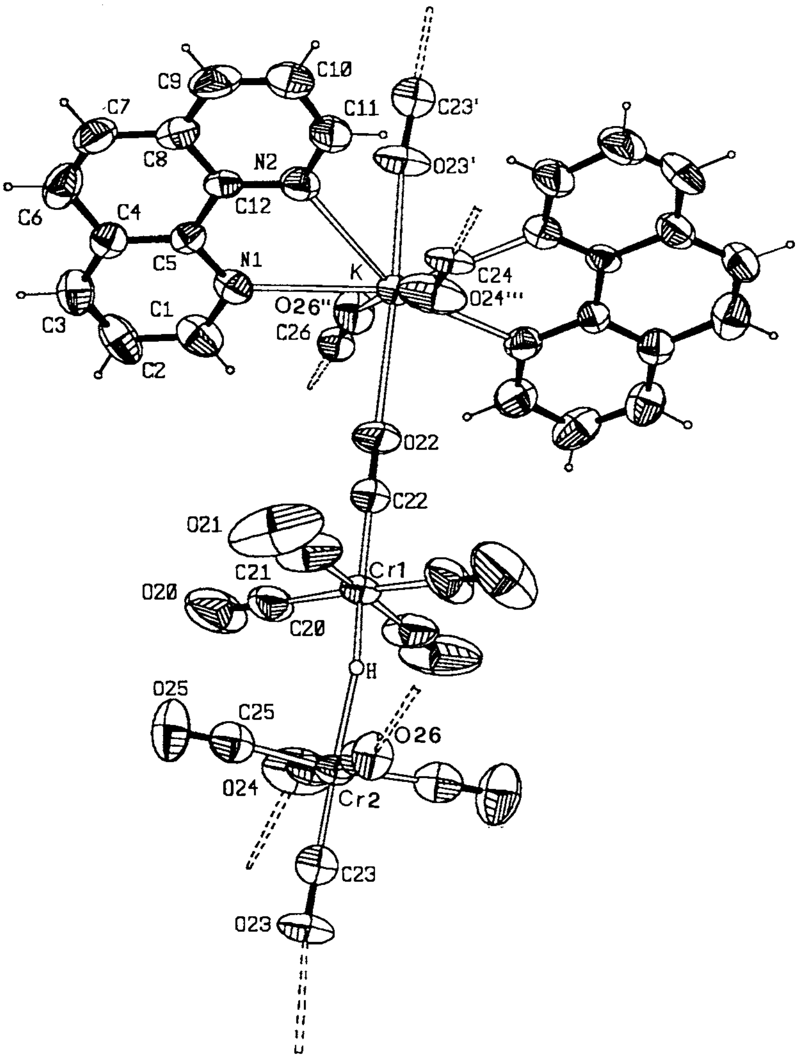

Fig. 2. Molecular structure of $[K(phen)_2][Cr_2(CO)_{10}(\mu\text{-H})]$.

anions. The molecular structure is highly symmetric with a crystallographic imposed mirror plane where K, Cr(1), Cr(2) together with four carbonyl groups are located as is shown in Fig. 2. The anion presents a bent staggered geometry with relatively strong interaction between the K^+ ion, the four nitrogens of two phenantroline molecules (symmetrically related) and four oxygens of the carbonyl groups of adjacent $[(Cr_2(CO)_{10}(\mu\text{-H})]^-$ moieties. In Table 2 are reported selected bond distances and angles.

The $Cr-C_{ax.}$ bond distances does not differ significantly from the $Cr-C_{eq.}$ (see Table 2) in this staggered conformation as it has been already observed in the $[KCr_2(CO)_{10}(\mu\text{-H})]$ structure for the staggered $[(Cr_2(CO)_{10}(\mu\text{-H})]^-$ moiety. In particular the axial distances show a lengthening attributable to the bonding interaction of the relevant oxygens to the potassium ion where the cation-anion interactions perturb the anion electron density to a greater extent.

In the complex cation $[K(phen)_2]^+$ the four nitrogens of the two phen units (related by mirror symmetry) are 2.884(4) and 2.877(5) Å apart from K which in turn has the following distances from the carbonyl oxygens: K⋯O(22) 2.870(5), K⋯O(23)′ 2.799(6), K⋯O(26)″ 3.083(5) and K⋯O(24)‴ 3.202(5) Å (′x, y, $1+z$; ″$0.5-x$, y, $0.5-z$; ‴$0.5+x$, y, $0.5-z$). The resulting coordination polyhedron can be assimilated to a square antiprism distorted towards dodecahedron. It was square-antiprismatic in the $[K(phen)_3]_2^{2+}$ centrosymmetric dimers of $[K(phen)_3][BPh_4]$ [6] and cubic in the centrosymmetric dimers $[K(phen)_3]^{2+}$ of $[K(phen)_3]$-$[Cr_2(CO)_{10}(\mu\text{-H})]$ [4].

The whole structure is polymeric being formed by double columns of $[(Cr_2(CO)_{10}(\mu\text{-H})]$ anions interca-

Table 2
Selected bond lengths (Å) and angles(°) for $[K(phenH)_2][Cr_2(CO)_{10}(\mu\text{-}H)]$ (**2**)

Bond lengths (Å)			
Cr(1)···Cr(2)	3.295(2)	Cr(1)–C(20)	1.860(6)
Cr(1)–C(21)	1.824(7)	$Cr(1)–C(22)_{ax.}$	1.847(6)
$Cr(2)–C(23)_{ax.}$	1.860(6)	Cr(2)–C(24)	1.892(5)
Cr(2)–C(25)	1.913(6)	Cr(2)–C(26)	1.877(7)
Cr(1)–H	1.69(7)	Cr(2)–H	1.81(7)
C(20)–O(20)	1.119(9)	C(21)–O(21)	1.121(9)
C(22)–O(22)	1.147(7)	C(23)–O(23)	1.161(8)
C(24)–O(24)	1.148(7)	C(25)–O(25)	1.121(8)
C(26)–O(26)	1.137(7)	N(1)–C(1)	1.321(6)
N(1)–C(5)	1.372(7)	N(2)–C(11)	1.317(6)
N(2)–C(12)	1.365(8)	C(1)–C(2)	1.396(8)
C(2)–C(3)	1.335(9)	C(3)–C(4)	1.360(6)
C(4)–C(5)	1.397(8)	C(4)–C(6)	1.465(9)
C(5)–C(12)	1.417(6)	C(6)–C(7)	1.329(6)
C(7)–C(8)	1.452(9)	C(8)–C(9)	1.400(6)
C(8)–C(12)	1.398(8)	C(9)–C(10)	1.330(9)
C(10)–C(11)	1.384(8)	K–N(1)	2.884(4)
K–N(2)	2.877(5)	K–O(22)	2.870(5)
K–O(23)′	2.799(6)	K–O(24)‴	3.202(5)
K–O(26)″	3.083(5)		
Bond angles (°)			
Cr(1)–C(20)–O(2O)	179.2(6)	Cr(1)–C(21)–O(21)	178.9(6)
Cr(1)–C(22)–O(22)	179.3(6)	Cr(2)–C(23)–O(23)	178.9(5)
Cr(2)–C(24)–O(24)	177.1(5)	Cr(2)–C(25)–O(25)	178.3(5)
Cr(2)–C(26)–O(26)	178.5(6)	Cr(1)–H–Cr(2)	140(3)
N(1)–C(1)–C(2)	122.4(5)	C(1)–C(2)–C(3)	119.9(6)
C(2)–C(3)–C(4)	119.4(5)	C(3)–C(4)–C(5)	119.8(5)
C(4)–C(5)–N(1)	120.6(5)	C(5)–N(1)–C(1)	117.8(4)
C(5)–C(4)–C(6)	118.3(6)	C(4)–C(6)–C(7)	121.8(5)
C(6)–C(7)–C(8)	119.6(5)	C(7)–C(8)–C(12)	120.1(5)
C(8)–C(12)–C(5)	119.7(5)	C(12)–C(5)–C(4)	120.5(4)
N(2)–C(12)–C(8)	121.0(5)	C(12)–C(8)–C(9)	117.7(5)
C(8)–C(9)–C(10)	121.1(5)	C(9)–C(10)–C(11)	117.9(6)
C(10)–C(11)–N(2)	124.4(5)	C(11)–N(2)–C(12)	117.9(4)
N(1)–C(5)–C(12)	118.8(4)	N(2)–C(12)–C(5)	119.3(4)
K–N(1)–C(5)	122.3(3)	K–N(2)–C(12)	122.6(3)
N(1)–K–N(2)	56.9(2)		

(′*x*, *y*, 1+*z*; ″0.5−*x*, *y*, 0.5−*z*; ‴0.5+*x*, *y*, 0.5−*z*)

lated by the potassium coordination spheres. It is worth noticing that in the corresponding $[K(phen)_3]^+$ salt the presence of three phen molecules satisfy the coordinative needs of the potassium without any interaction with the carbonyl oxygens and the anion conformation is bent-eclipsed. A comparison of the structural parameters of potassium and $[(phenH)_2Cl]^+$ salts of $[Cr_2(CO)_{10}(\mu\text{-}H)]$ is reported in Table 3. In $[K(crypt\text{-}222)]^+$ beside the insertion of the potassium in the cryptate ligand the potassium ion shows appreciable cation–anion interactions. The close K···O contact 2.966(3) Å with one oxygen of the anion is sufficient to affect the anion geometry which is bent staggered.

$K[Cr_2(CO)_{10}(\mu\text{-}H)]$ is a unique example of the coexistence in the same crystal of two centrosymmetric anions, for sake of crystallographic symmetry, belonging to the linear eclipsed configuration while a third anion with no-crystallographic constrains is highly bent and staggered and the shortest K···O contacts (2.72(1) and 2.77(1) Å) involve its axial carbonyl oxygen atoms.

3. Conclusion

The presence of cation–anion interactions determines the $[(Cr_2(CO)_{10}(\mu\text{-}H)]$ geometry as demonstrated by the structural analysis of the compounds here reported and by the comparison with literature data. In absence of such interactions the preferred anion geometry seems to be eclipsed with a relatively long Cr···Cr distance. When the cation involves in its coordination sphere the carbonyl oxygens, the tendency is for a staggered geometry. The twisting of the two carbonyl planes is related to the number of carbonyl oxygens interacting with the cation. The trend of the

Table 3
Structural parameters for the chromium pentacarbonyl derivatives

Compound	Cr···Cr (Å)	Cr–H (Å)	Cr–H–Cr (°)	Carbonyl conformation	$Cr–C_{eq\cdot av}$	$Cr–C_{ax\cdot av}$
$K[Cr_2(CO)_{10}(\mu\text{-}H)]$ [3]	3.350(3)		Linear	Eclipsed		
	3.260(3)	1.65(7) 1.90(7)	132(3) (bent)	Staggered 36°	1.88(2)	1.88(1)
$[K(phen)_3][Cr_2(CO)_{10}(\mu\text{-}H)]$ [4]	3.394(3)	1.74(7)	159(3) (bent)	Eclipsed	1.88(1)	1.82(1)
$[K(phen)_2][Cr_2(CO)_{10}(\mu\text{-}H)]$[a]	3.295(1)	1.69(7) 1.81(7)	140(3) (bent)	Staggered 45°	1.873(7)	1.853(6)
$[K(crypt\text{-}222)][Cr_2(CO)_{10}(\mu\text{-}H)]$ [7]	3.300(4)[b]	1.735(5)	145.2(3) (bent)	Staggered 19°	1.900(3)	1.848(3)
$[Cl(phenH)_2][Cr_2(CO)_{10}(\mu\text{-}H)]$[a]	3.371(1) 1.66(4)	1.72(4)	174(3) (linear)	Eclipsed	1.888(4)	1.830(4)

[a] Present work.

[b] (neutron 20 K).

Table 4
Crystallographic data

Compound	[(phenH)$_2$Cl][Cr$_2$(CO)$_{10}$(μ-H)]	[K(phen)$_2$][Cr$_2$(CO)$_{10}$(μ-H)]
Formula	$C_{34}H_{19}N_4O_{10}ClCr_2$	$C_{34}H_{17}N_4O_{10}KCr_2$
Molecular weight	783.0	784.6
Crystal system	Triclinic	Orthorhombic
Space group	$P\bar{1}$	*Pnma*
a (Å)	15.090(3)	11.954(2)
b (Å)	11.749(2)	19.877(4)
c (Å)	10.514(2)	14.789(3)
α (°)	105.42(4)	
β (°)	94.05(4)	
γ (°)	107.92(4)	
V (Å^3)	1686(1)	3514(2)
Formula units (unit cell)	2	4
D$_{calc.}$ (mg m^{-3})	1.542	1.483
Crystal size (mm)	$0.36\times0.16\times0.18$	$0.20\times0.20\times0.15$
μ(Mo–K$_\alpha$) (cm^{-1})	7.70	7.80
Diffractometer/scan	Philips PW1100 ($\theta/2\theta$)	Enraf-Nonius CAD-4 ($\omega/2\theta$)
Scan width	1.2	$1.2+0.35\tan\theta$
θ range (°)	3–25	3–25
Reflections measured	6289	4389
Reflections observed [$I\geq3\sigma(I)$]	3963	1762
Weighting scheme w	$[\sigma^2(F_o)+0.00057(F_o)^2]^{-1}$	$4F_o^2/[\sigma^2(F_o^2)+(0.04F_o^2)^2]$
$R=\Sigma\,\lvert F_o\rvert-\lvert F_c\rvert/\Sigma\,\lvert F_o\rvert$	0.035	0.055
$R\text{w}=[\Sigma\,w(\lvert F_o\rvert-\lvert F_c\rvert)^2/\Sigma\,w(\lvert F_o\rvert)^2]^{1/2}$	0.036	0.073
Goodness-of-fit	0.99	1.37

bending of the bridging hydride does not seem be influenced by these interactions as bent-eclipsed and bent-staggered structures have been observed.

4. Experimental

4.1. Preparation of (**1**)

Ruby red crystals of [(phenH)$_2$Cl][Cr$_2$(CO)$_{10}$(μ-H)] have been unexpectedly obtained when the yellow [K(phen)$_3$][Cr$_2$(CO)$_{10}$(μ-H)] was reacted with HCl in MeOH at room temperature and at 1:2 molar ratios. It was, however, the main reactions product in the case of 1:2.5–1:2.7 molar ratios. It crystallizes from $CHCl_3$ + toluene or CH_2Cl_2 + toluene mixtures cooled from 5 to − 30°C depending on the experimental conditions. Details on all operations carried out under oxygen-free nitrogen with carefully deaerated solvents, have been reported already ([1b]).

4.2. Preparation of (**2**)

Pale yellow crystals of [K(phen)$_2$][Cr$_2$(CO)$_{10}$(μ-H) were obtained by partial dissociation of [K(phen)$_3$][Cr$_2$(CO)$_{10}$(μ-H)] in petroleum ether ([1]a)

4.3. X-ray measurements and structure determination

Crystal data, collected reflections and parameters of the final refinement for compounds **1** and **2**, respectively, are reported in Table 4. Reflections were collected using an Enraf-Nonius CAD-4 diffractometer for **1**, and a Philips PW1100 diffractometer for **2**, using graphite-monochromated Mo–K$_\alpha$ radiation (λ = 0.71073 Å). The orientation matrix and cell dimensions were determined by least squares refinement of the angular positions of 25 reflections. Intensities data were collected at room temperature. Three standard reflections were monitored every 2 h. The diffracted intensities were corrected for Lorentz polarization and absorption [8]. Scattering factors and anomalous scattering corrections of non-hydrogen were taken from ref. [9]. The structures were solved by Patterson and Fourier methods. The structures were refined by full-matrix least squares using anisotropic temperature factors for all non-hydrogen atoms. All the hydrogen atoms were introduced at calculated positions in their described geometries (d$_{C-H}$ 0.95 Å) and during refinement were allowed to ride on the attached carbon atoms with unique isotropic thermal parameters, except for the hydride hydrogen of compounds **1** and **2** which were detected in Fourier differences and refined isotrop-

Table 5
Atomic coordinates for non-hydrogen atoms and equivalent isotropic thermal parameters (Å^2) with the estimated S.D. in parentheses for [(phenH)$_2$Cl][Cr$_2$(CO)$_{10}$(μ-H)] (**1**)

Atom	x	y	z	B_{eq}
Cr(1)	−0.16522(4)	0.16307(5)	0.01504(5)	3.28(2)
Cr(2)	0.04395(4)	0.35471(4)	0.20505(5)	2.95(2)
Cl(1)	0.52980(6)	0.29553(8)	0.42434(8)	4.10(3)
O(1)	−0.2129(2)	0.3730(3)	−0.0560(4)	7.6(2)
O(2)	−0.2640(2)	0.2062(4)	0.2544(3)	8.9(2)
O(3)	−0.1186(3)	−0.0518(3)	0.0738(4)	9.8(2)
O(4)	−0.0732(2)	0.1295(3)	−0.2329(3)	5.5(1)
O(5)	−0.3487(2)	−0.0178(3)	−0.1491(3)	6.9(1)
O(6)	−0.0036(2)	0.5764(3)	0.1683(3)	6.4(2)
O(7)	0.1333(2)	0.3212(2)	−0.0467(3)	4.8(1)
O(8)	0.1062(3)	0.1493(3)	0.2625(3)	7.2(1)
O(9)	−0.0538(2)	0.3606(2)	0.4465(3)	6.1(1)
O(10)	0.2247(2)	0.5507(2)	0.3627(3)	5.6(1)
C(1)	−0.1921(3)	0.2971(4)	−0.0258(4)	4.7(2)
C(2)	−0.2240(3)	0.1911(4)	0.1667(4)	5.1(2)
C(3)	−0.1359(3)	0.0302(4)	0.0536(4)	5.5(2)
C(4)	−0.1053(2)	0.1429(3)	−0.1379(4)	3.7(1)
C(5)	−0.2772(3)	0.0541(4)	−0.0851(3)	4.5(1)
C(6)	0.0118(2)	0.4906(3)	0.1786(3)	3.7(1)
C(7)	0.0982(2)	0.3338(3)	0.0457(3)	3.3(1)
C(8)	0.0800(3)	0.2234(3)	0.2377(3)	4.2(1)
C(9)	−0.0178(3)	0.3593(3)	0.3548(4)	3.9(1)
C(10)	0.1556(3)	0.4733(3)	0.3033(3)	3.8(1)
N(1)	0.4277(2)	0.5477(3)	0.2766(3)	3.4(1)
C(11)	0.3954(2)	0.4398(3)	0.3024(4)	4.1(2)
C(12)	0.3483(3)	0.3318(4)	0.1994(4)	4.7(2)
C(13)	0.3345(2)	0.3377(3)	0.0722(4)	4.3(2)
C(14)	0.3705(2)	0.4525(3)	0.0448(3)	3.4(2)
C(15)	0.3601(2)	0.4642(3)	−0.0880(3)	4.2(2)
C(16)	0.3975(2)	0.5754(3)	−0.1089(3)	4.1(2)
C(17)	0.4486(2)	0.6857(3)	−0.0013(3)	3.4(2)
C(18)	0.4917(2)	0.8024(3)	−0.0204(3)	4.1(2)
C(19)	0.5409(3)	0.9020(3)	0.0870(4)	4.3(2)
C(20)	0.5481(3)	0.8860(3)	0.2138(3)	4.2(1)
N(2)	0.5086(2)	0.7784(3)	0.2379(3)	3.7(1)
C(21)	0.4594(2)	0.6788(3)	0.1306(3)	3.1(1)
C(22)	0.4181(2)	0.5598(3)	0.1512(3)	3.1(1)
N(3)	0.3563(2)	0.0991(3)	0.4605(2)	3.1(1)
C(23)	0.3882(3)	0.0047(3)	0.4209(3)	3.9(1)
C(24)	0.3371(3)	−0.1130(3)	0.4267(4)	4.5(1)
C(25)	0.2542(3)	−0.1308(3)	0.4745(4)	4.5(1)
C(26)	0.2204(2)	−0.0303(3)	0.5181(3)	3.5(1)
C(27)	0.1350(3)	−0.0411(3)	0.5726(4)	4.4(1)
C(28)	0.1060(2)	0.0570(3)	0.6111(3)	4.1(1)
C(29)	0.1578(2)	0.1770(3)	0.5980(3)	3.3(1)
C(30)	0.1276(3)	0.2809(3)	0.6327(3)	4.1(1)
C(31)	0.1809(3)	0.3897(4)	0.6159(4)	4.5(1)
C(32)	0.2653(3)	0.3978(3)	0.5663(3)	3.8(1)
N(4)	0.2972(2)	0.3026(2)	0.5325(2)	3.3(1)
C(33)	0.2428(2)	0.1936(3)	0.5476(3)	2.8(1)
C(34)	0.2742(2)	0.0871(3)	0.5084(3)	2.8(1)

ically. The calculations were performed using the SDP-Plus Structure Determination Package [10] for **1**, and SHELX-76 program package [11] for **2**. The atomic coordinates for the **1** and **2** complexes are reported in Tables 5 and 6, drawings were produced using ORTEP II [12].

Table 6
Atomic coordinates for non-hydrogen atoms and equivalent isotropic thermal parameters (Å^2) with the estimated S.D. in parentheses for [K(phen)$_2$][Cr$_2$(CO)$_{10}$(μ-H)] (**2**)

Atom	x	y	z	B_{eq}
Cr(1)	0.9774(1)	0.2500	0.17116(9)	4.56(3)
Cr(2)	0.9537(1)	0.2500	−0.05079(9)	4.27(3)
K	0.9531(2)	0.2500	0.5671(1)	4.44(4)
O(20)	0.8004(4)	0.1450(4)	0.1584(4)	11.3(2)
O(21)	1.1486(4)	0.1436(4)	0.1776(4)	12.3(3)
O(22)	0.9589(3)	0.2500	0.3731(3)	7.6(2)
O(23)	0.8952(4)	0.2500	−0.2495(4)	7.4(2)
O(24)	0.7075(3)	0.2500	0.0006(4)	9.3(3)
O(25)	0.9570(2)	0.4026(3)	−0.0490(3)	10.1(2)
O(26)	1.1984(3)	0.2500	−0.0997(3)	6.3(2)
N(1)	0.8682(3)	0.3855(3)	−0.4451(3)	5.0(1)
N(2)	1.0639(3)	0.3637(3)	−0.3524(3)	5.5(1)
C(1)	0.7746(4)	0.3975(3)	−0.4898(4)	6.7(2)
C(2)	0.7325(3)	0.4624(3)	−0.5019(3)	7.1(2)
C(3)	0.7877(3)	0.5151(4)	−0.4682(3)	6.3(2)
C(4)	0.8835(3)	0.5048(3)	−0.4206(4)	4.8(1)
C(5)	0.9242(3)	0.4395(3)	−0.4091(4)	3.9(1)
C(6)	0.9468(3)	0.5605(4)	−0.3807(4)	6.8(2)
C(7)	1.0416(2)	0.5503(4)	−0.3358(3)	6.5(2)
C(8)	1.0826(3)	0.4822(3)	−0.3232(3)	5.7(2)
C(9)	1.1821(3)	0.4684(4)	−0.2769(3)	8.3(2)
C(10)	1.2185(2)	0.4056(3)	−0.2668(3)	8.1(2)
C(11)	1.1581(3)	0.3546(4)	−0.3078(3)	7.3(2)
C(12)	1.0247(4)	0.4279(3)	−0.3608(3)	4.1(1)
C(20)	0.8665(3)	0.1847(4)	0.1627(4)	8.3(2)
C(21)	1.0841(4)	0.1845(4)	0.1756(4)	9.8(2)
C(22)	0.9653(4)	0.2500	0.2957(4)	5.5(2)
C(23)	0.9166(4)	0.2500	−0.1729(4)	5.3(2)
C(24)	0.7997(4)	0.2500	−0.0212(5)	5.9(3)
C(25)	0.9547(3)	0.3462(3)	−0.0485(3)	6.4(2)
C(26)	1.1065(3)	0.2500	−0.0800(4)	4.6(2)
H	1.016(4)	0.2500	0.061(5)	8(2)

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