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## [N-(Benzylidene)aniline-N]penta- carbonylchromium(0)

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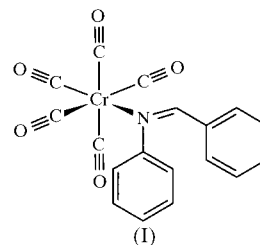
In the title compound, [Cr(C<sub>13</sub>H<sub>11</sub>N)(CO)<sub>5</sub>], the imino ligand displays weak  $\pi$ -acceptor capabilities and, in contrast to Fischer-type carbene complexes, is not positioned on the bisecting line of the angle between two CO ligands.

### Comment

Aziridines are valuable target molecules and versatile reagents for stereoselective organic synthesis (Tanner, 1994). One strategic pathway leading to this class of compounds is the activation of an imine by complexation to a Lewis acid and the addition of a cryptocarbanionic component (Wulff & Antilla, 1999).

We report here the crystal structure of such an activated imine, (I). Reaction of the transfer reagent ( $\eta^2$ -*cis*-cyclooctene)pentacarbonylchromium(0) (Grevels & Skibbe, 1984) with *N*-(benzylidene)aniline and ethyl diazoacetate did not lead to aziridines. Instead, we were able to isolate the title compound. This complex exhibits a slight deviation from ideal octahedral symmetry due to steric interaction of the bulky imino ligand. To minimize these repulsions, the imine adopts an *E* configuration of the C2=N1 double bond and the phenyl ring bonded to the N1 atom arranges itself nearly parallel to the (CO)<sub>4</sub>Cr plane. This is also reflected in the torsion angle at C2–N1–C11–C12 of 89.6 (3)°. In contrast to Fischer-type carbene complexes (Schubert, 1983), the ligand is not positioned on the bisecting line of the angle between two carbonyl ligands: C2–N1–Cr–C1e 15.86 (9)° and C2–N–Cr–C1c 73.40 (19)°. The activation of the imine by complexation to the metal centre is reflected by the C2=N1 distance of 1.285 (3) Å as compared to 1.27 Å, the sum of the covalent radii of doubly bonded C and N atoms (Holleman & Wiberg, 1995). When compared to Cr–N distances of pentacarbonylchromium(0) complexes with ligands of little or no  $\pi$ -acidity such as piperidine (2.204 Å) or pyridine (2.194 Å) which were reported by Cotton *et al.* (1981), the shortening of the bond length between these two atoms to 2.1659 (18) Å

indicates a slight  $\pi$ -back bonding into ligand orbitals. As a result of the weaker  $\pi$ -acceptor capability of the imine with respect to CO, the *trans*-Cr–C1a bond is shortened to 1.845 (2) Å versus the average (Cr–C1b–e) of 1.912 Å and the *trans*-C1a–O1a bond is lengthened to 1.157 (3) Å versus the average (C1b–e–O1b–e) of 1.139 Å. In summary, the interaction between the pentacarbonylchromium(0) fragment and the imino ligand does lead to visible differences in its bonding situation, but the effects are not strong enough to enable a nucleophilic attack of ethyl diazoacetate onto the C2 atom.



### Experimental

Ethyl diazoacetate was added to a solution of ( $\eta^2$ -*cis*-cyclooctene)-pentacarbonylchromium(0) and *N*-(benzylidene)aniline in dichloromethane (Liptau, 2000). The title compound was purified by column chromatography. Crystals were obtained from *n*-pentane in 70% yield.

#### Crystal data

[Cr(C<sub>13</sub>H<sub>11</sub>N)(CO)<sub>5</sub>]  
*M<sub>r</sub>* = 373.28  
Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 13.0959 (12) Å  
*b* = 15.3703 (13) Å  
*c* = 8.7107 (3) Å  
 $\beta$  = 108.860 (4)°  
*V* = 1659.2 (2) Å<sup>3</sup>  
*Z* = 4

*D<sub>x</sub>* = 1.494 Mg m<sup>-3</sup>  
Mo *K* $\alpha$  radiation  
Cell parameters from 20447 reflections  
 $\theta$  = 2.5–28.3°  
 $\mu$  = 0.717 mm<sup>-1</sup>  
*T* = 123 (2) K  
Plates, yellow  
0.25 × 0.10 × 0.10 mm

#### Data collection

Nonius KappaCCD diffractometer  
Rotation in  $\varphi$  and  $\omega$  (1°) scans  
Absorption correction: multi-scan  
(*MULABS* in *PLATON*98 (Spek, 1990; cf. Blessing, 1995)  
*T*<sub>min</sub> = 0.822, *T*<sub>max</sub> = 0.922  
17 681 measured reflections

2915 independent reflections  
2344 reflections with *I* > 2 $\sigma$ (*I*)  
*R*<sub>int</sub> = 0.0616  
 $\theta$ <sub>max</sub> = 25.00°  
*h* = –15 → 15  
*k* = –18 → 18  
*l* = –10 → 10

#### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.037  
*wR*(*F*<sup>2</sup>) = 0.095  
*S* = 1.024  
2915 reflections  
226 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.3439P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
( $\Delta/\sigma$ )<sub>max</sub> < 0.001  
 $\Delta\rho$ <sub>max</sub> = 0.33 e Å<sup>-3</sup>  
 $\Delta\rho$ <sub>min</sub> = –0.56 e Å<sup>-3</sup>

All H atoms were located by a difference Fourier synthesis and refined with fixed individual displacement parameters [*U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(C)], using a riding model with C–H = 0.95 Å.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97*

(Sheldrick, 1997); software used to prepare material for publication: *SHELXL97*.

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