

atoms anisotropic, H atoms isotropic; 280 parameters [1 scale factor, 22 anisotropic atoms, 20 isotropic atoms, and a Rogers (1981) parameter η (an indicator of absolute configuration and twinning in non-centrosymmetric structures)]; $R = 0.021$ (non-zero-weighted data); $R(\text{all data}) = 0.026$; $wR = 0.025$; $S = 1.01$; $w = 4F^2/[\sigma^2(F^2) + (0.035F^2)^2]$; $\eta = 1.02$ (2) (theoretically, $\eta = 1.0$ for correct absolute configuration and no twinning); max. $\Delta/\sigma < 0.02$; max. empirical isotropic correction for extinction 12% of F ; max. and min. of ΔF synthesis 0.6 and $-0.5 \text{ e } \text{\AA}^{-3}$; scattering factors from *International Tables for X-ray Crystallography* (1974); local unpublished programs and ORTEP (Johnson, 1976). Atomic coordinates are listed in Table 1,* with the atomic numbering scheme shown in Fig. 1.

Related literature. The structure consists of [L-arginine]²⁺ cations and [AsO₂(OH)₂]⁻ anions interconnected by a network of hydrogen bonds that involve all of the H atoms bonded to the N and O atoms with the exception of H12. Distances and angles are given in Table 2. The optical properties of the title compound are reported elsewhere (Velsko, 1986; Monaco, Davis, Velsko, Wang, Eimerl & Zalkin, 1987).

*Lists of structure factors, anisotropic thermal parameters, H-atom parameters and interatomic distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51557 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

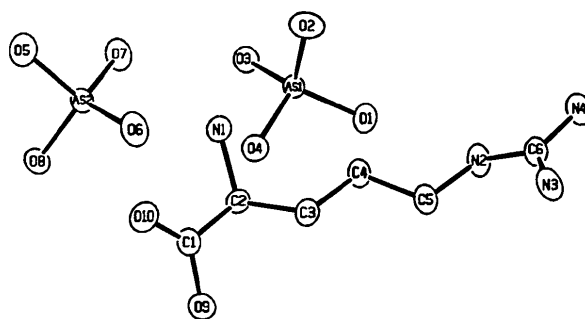


Fig. 1. ORTEP drawing showing the atomic numbering scheme; 50% probability ellipsoids are shown.

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Structure of the 1/1 Complex Between Chromium Tricarbonyl and (–)-Canadine

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Abstract. Tricarbonyl{8a–12a- η -5,6,13,13a-tetrahydro-9,10-dimethoxy-2,3-(methylenedioxy)-8H-dibenzo[*a,g*]quinolizine}chromium. [Cr(C₂₀H₂₁NO₄)(CO)₃], $M_r = 475.4$, orthorhombic, $P2_12_12_1$, $a = 8.061$ (2), $b = 14.658$ (1), $c = 18.001$ (1) Å, $V = 2127.06$ Å³, $Z = 4$, $D_x = 1.485$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.54148$ Å, $\mu = 4.88$ mm⁻¹, $F(000) = 984$, $T = 293$ K, $R = 0.045$ for 1681 observed reflections with $I > 3\sigma(I)$. The molecule is nearly planar with the two aromatic rings inclined at an angle of 150.1° to each other. The

chromium atom is 1.74 Å from the best plane calculated through the aromatic ring to which it is complexed.

Experimental. Columnar yellow crystals (0.20 × 0.20 × 0.58 mm); Enraf–Nonius CAD-4F diffractometer with graphite monochromator; $\omega/2\theta$ scans; lattice parameters from least-squares fits of 25 reflections ($25 < \theta < 35^\circ$); semi-empirical absorption correction (transmission factors 1.00–1.15); $\theta_{\text{max}} = 75^\circ$ ($h =$

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Table 1. Atomic coordinates and equivalent isotropic temperature factors for the non-hydrogen atoms with e.s.d.'s in parentheses (atomic labelling as in Fig. 1)

$$U_{eq} = (U_1 \times U_2 \times U_3)^{1/3}$$

	x	y	z	$U_{eq}(\text{\AA}^2)$
Cr(1)	-0.8190 (1)	0.04582 (6)	0.52406 (5)	0.0392
O(1)	-0.7459 (5)	-0.0895 (3)	0.6676 (2)	0.0482
O(2)	-0.9745 (6)	-0.1555 (3)	0.5720 (3)	0.0561
O(3)	-0.8507 (7)	0.5825 (3)	0.6054 (2)	0.0634
O(4)	-0.7795 (7)	0.6292 (3)	0.7275 (2)	0.0646
O(5)	-0.8215 (9)	0.2005 (4)	0.4162 (4)	0.0905
O(6)	-0.748 (1)	-0.0816 (6)	0.3991 (4)	0.0987
O(7)	-0.4530 (7)	0.0629 (7)	0.5461 (3)	0.0896
C(1)	-0.8525 (6)	-0.0299 (3)	0.6318 (3)	0.0368
C(2)	-0.9708 (7)	-0.0632 (3)	0.5814 (3)	0.0439
C(3)	-1.0785 (8)	-0.0017 (4)	0.5447 (3)	0.0439
C(4)	-1.0650 (8)	0.0917 (4)	0.5610 (3)	0.0442
C(5)	-0.9494 (7)	0.1265 (4)	0.6120 (3)	0.0395
C(6)	-0.9366 (8)	0.2276 (4)	0.6277 (3)	0.0452
C(7)	-0.7686 (7)	0.2527 (4)	0.6615 (3)	0.0419
C(8)	-0.7650 (7)	0.3630 (4)	0.6829 (3)	0.0431
C(9)	-0.8114 (9)	0.4174 (4)	0.6296 (3)	0.0502
C(10)	-0.814 (1)	0.5081 (4)	0.6503 (3)	0.0499
C(11)	-0.7712 (8)	0.5351 (4)	0.7205 (3)	0.0518
C(12)	-0.7178 (8)	0.4745 (4)	0.7732 (3)	0.0501
C(13)	-0.7132 (8)	0.3816 (4)	0.7532 (3)	0.0470
C(14)	-0.6542 (9)	0.3125 (4)	0.8089 (3)	0.0520
C(15)	-0.6054 (8)	0.2248 (4)	0.7717 (3)	0.0508
N(16)	-0.7434 (6)	0.1931 (3)	0.7255 (2)	0.0398
C(17)	-0.7146 (8)	0.0994 (4)	0.7024 (3)	0.0433
C(18)	-0.8410 (7)	0.0653 (3)	0.6472 (3)	0.0397
C(19)	-0.817 (1)	-0.1241 (4)	0.7353 (3)	0.0590
C(20)	-1.070 (1)	-0.1914 (4)	0.5122 (4)	0.0678
C(21)	-0.863 (1)	0.6552 (3)	0.6594 (3)	0.0664
C(22)	-0.819 (1)	0.1400 (5)	0.4579 (4)	0.0601
C(23)	-0.777 (1)	-0.0322 (6)	0.4480 (4)	0.0652
C(24)	-0.5980 (9)	0.0577 (6)	0.5359 (3)	0.0582

+10, $k = +18$, $l = +22$); four standard reflections used for scaling the data (0.99–1.03). 3275 reflections collected, merged to give 2492 unique ($R_m = 0.067$) of which 811 were considered to be unobserved [$I < 3\sigma(I)$]. Solved using direct methods (MULTAN80, Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), hydrogens placed in calculated positions. Full-matrix least-squares refinement (CRYSTALS, Watkin, Carruthers & Betteridge, 1985) on 290 parameters; all non-H atoms anisotropic, temperature factors of H atoms not refined and coordinates constrained to ride on those of the carbons to which they were attached. Modified Chebyshev weighting scheme (Prince, 1982) applied, parameters 3.025, 1.655 and 1.823. At the end of refinement the maximum least-squares shift to e.s.d. was 0.01, the minimum and maximum values of the residual electron density were -0.71 and 0.32 e \AA^{-3} , respectively, the secondary-extinction parameter (Larson, 1976) was 58 (6) and $R = 0.045$, $wR = 0.052$. The final atomic parameters are given in Table 1.* Bond lengths and

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, all bond angles, and details of selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51356 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (Å) and selected bond angles (°) for the non-hydrogen atoms with e.s.d.'s in parentheses

Cr(1) C(1)	2.251 (5)	Cr(1) C(2)	2.262 (5)
Cr(1) C(3)	2.236 (6)	Cr(1) C(4)	2.197 (6)
Cr(1) C(5)	2.238 (5)	Cr(1) C(18)	2.241 (5)
Cr(1) C(22)	1.824 (6)	Cr(1) C(23)	1.815 (7)
Cr(1) C(24)	1.803 (7)	O(1) C(1)	1.385 (6)
O(1) C(19)	1.438 (7)	O(2) C(2)	1.364 (6)
O(2) C(20)	1.425 (8)	O(3) C(10)	1.389 (7)
O(3) C(21)	1.445 (4)	O(4) C(11)	1.388 (7)
O(4) C(21)	1.450 (4)	O(5) C(22)	1.162 (7)
O(6) C(23)	1.165 (8)	O(7) C(24)	1.186 (9)
C(1) C(2)	1.405 (8)	C(1) C(18)	1.426 (7)
C(2) C(3)	1.415 (8)	C(3) C(4)	1.406 (8)
C(4) C(5)	1.403 (8)	C(5) C(6)	1.511 (7)
C(5) C(18)	1.404 (7)	C(6) C(7)	1.530 (8)
C(7) C(8)	1.520 (7)	C(7) N(16)	1.459 (6)
C(8) C(9)	1.397 (8)	C(8) C(13)	1.397 (7)
C(9) C(10)	1.381 (8)	C(10) C(11)	1.369 (8)
C(11) C(12)	1.368 (8)	C(12) C(13)	1.408 (8)
C(13) C(14)	1.504 (8)	C(14) C(15)	1.503 (9)
C(15) N(16)	1.464 (7)	N(16) C(17)	1.455 (7)
C(17) C(18)	1.508 (7)		
C(19) O(1)	C(1) 111.7 (5)	C(9) C(10)	O(3) 127.1 (5)
C(20) O(2)	C(2) 118.2 (5)	C(11) C(10)	O(3) 111.4 (5)
C(21) O(3)	C(10) 101.6 (3)	C(11) C(10)	C(9) 121.4 (6)
C(21) O(4)	C(11) 101.9 (3)	C(10) C(11)	O(4) 110.9 (5)
C(2) C(1)	O(1) 120.2 (4)	C(12) C(11)	O(4) 126.8 (5)
C(18) C(1)	O(1) 119.1 (5)	C(12) C(11)	C(10) 122.2 (5)
C(18) C(1)	C(2) 120.6 (5)	C(13) C(12)	C(11) 117.3 (5)
C(1) C(2)	O(2) 116.1 (5)	C(12) C(13)	C(8) 120.9 (6)
C(3) C(2)	O(2) 124.1 (5)	C(14) C(13)	C(8) 119.8 (5)
C(3) C(2)	C(1) 119.8 (5)	C(14) C(13)	C(12) 119.3 (5)
C(4) C(3)	C(2) 118.4 (5)	C(15) C(14)	C(13) 111.2 (5)
C(5) C(4)	C(3) 122.9 (6)	N(16) C(15)	C(14) 109.0 (5)
C(6) C(5)	C(4) 121.6 (5)	C(15) N(16)	C(7) 111.4 (4)
C(18) C(5)	C(4) 118.4 (5)	C(17) N(16)	C(7) 111.2 (4)
C(18) C(5)	C(6) 120.0 (5)	C(17) N(16)	C(15) 109.9 (5)
C(7) C(6)	C(5) 111.8 (5)	C(18) C(17)	N(16) 113.2 (5)
C(8) C(7)	C(6) 110.5 (5)	C(5) C(18)	C(1) 119.9 (5)
N(16) C(7)	C(6) 107.1 (5)	C(17) C(18)	C(1) 119.7 (5)
N(16) C(7)	C(8) 112.1 (4)	C(17) C(18)	C(5) 120.4 (5)
C(9) C(8)	C(7) 118.4 (5)	O(4) C(21)	O(3) 110.0 (3)
C(13) C(8)	C(7) 121.7 (5)	O(5) C(22)	Cr(1) 178.9 (7)
C(13) C(8)	C(9) 119.9 (5)	O(6) C(23)	Cr(1) 178.8 (8)
C(10) C(9)	C(8) 118.1 (5)	O(7) C(24)	Cr(1) 177.2 (7)

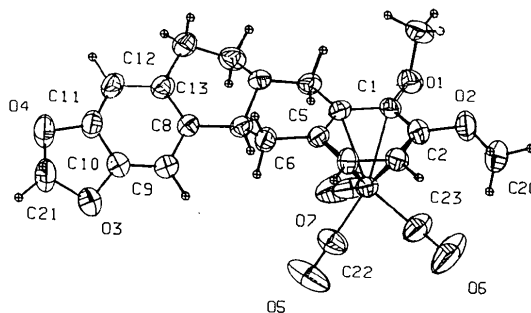


Fig. 1. Thermal ellipsoids drawn at the 50% probability level.

angles are given in Table 2. Fig. 1 shows the molecule and the numbering scheme adopted.

Related literature. The synthesis of this compound has been described by Blagg & Davies (1986).

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Bis(4,6-dimethyl-2-nitrosophenylamido)palladium(II) Monohydrate

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Abstract. $[\text{Pd}(\text{C}_8\text{H}_9\text{N}_2\text{O})_2]\cdot\text{H}_2\text{O}$, $M_r = 422.76$, monoclinic, $C2/c$, $a = 16.156(5)$, $b = 13.883(4)$, $c = 7.609(4)$ Å, $\beta = 105.15(5)^\circ$, $V = 1647.3$ Å³, $Z = 4$, $D_x = 1.70$ Mg m⁻³, $F(000) = 856$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.089$ mm⁻¹, $T = 293$ K, $R = 0.043$ for 1153 unique reflexions [$F \geq 3\sigma(F)$]. The water molecule lies on a crystallographic twofold axis and associates with adjacent centrosymmetric bis(4,6-dimethyl-2-nitrosophenylamido)palladium(II) molecules [$\text{Pd}-\text{N}$ 1.984(4), 2.023(4) Å] via two pairs of hydrogen bonds [$\text{H}_2\text{O}\cdots\text{HN}$ 2.38(8), $\text{O}\cdots\text{N}$ 3.02(1) Å, $\text{N}-\text{H}\cdots\text{O}$ 161(4)° and $\text{HOH}\cdots\text{O}=\text{N}$ 1.99(9), $\text{O}\cdots\text{O}$ 2.73(1) Å, $\text{O}-\text{H}\cdots\text{O}$ 165(4)°] to form infinite diagonal chains. An amine C–N bond order of approximately 2 [1.292(6) Å], considerable phenyl 3,5-diene character, and a shortened nitrosyl C–N [1.333(6) Å] indicate that excess amine negative charge, due to replacement of hydrogen by palladium, has been redistributed.

Experimental. The sample was prepared by adding 4,6-dimethyl-2-nitrosoaniline (1 mol) and sodium hydroxide (2 mol) to a methanolic solution of (dichlorodibenzonitrile)palladium(II) (1 mol) and stirring at room temperature for 5 h. The black precipitate thus formed was recrystallized from chloroform.

Crystal dimensions 0.30 × 0.15 × 0.08 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions ($11.1 \leq \theta \leq 16.8^\circ$), $\omega-2\theta$ scan mode, ω scan width of $(0.90 + 0.35 \tan \theta)^\circ$ and scan speed ranging from 0.5 to 5° min⁻¹ according to the intensity gathered in a pre-scan, $-18 \leq h \leq 18$, $0 \leq k \leq 16$, $0 \leq l \leq 9$, $0 \leq \theta \leq 25^\circ$, 3047 reflexions measured, 1315 unique, $R_{\text{int}} = 0.029$, 1153 observed [$F \geq 3\sigma(F)$], no drift in intensity standards (913, 751, 660) measured every 2.5 h, Lp and absorption corrections (transmission

factors max., min. 0.92, 0.80). Structure solved by normal heavy-atom techniques followed by full-matrix least squares based on F using *SHELX76* (Sheldrick, 1976), final $R = 0.043$, $wR = 0.050$, $w = 0.2607/[\sigma^2(F_o) + 0.0008F_o^2]$, anisotropic thermal parameters for heavier atoms, H from ΔF subjected to isotropic refinement. Maximum fluctuation in final ΔF map ± 1.42 e Å⁻³ near to Pd, ± 0.32 e Å⁻³ elsewhere, maximum Δ/σ 0.002. Scattering factors from *International Tables for X-ray Crystallography* (1974), computation carried out on the joint CDC7600/Amdahl 470 system of the University of Manchester Regional Computing Centre. Literature survey from the Cambridge Structural Database was performed using the Crystal Structural Search and Retrieval interactive system (CSSR, 1984). The molecule including labelling scheme is illustrated in Fig. 1. Final atomic coordinates and selected molecular geometry are presented in Tables 1* and 2 respectively.

* Lists of structure factors, H-atom coordinates, anisotropic vibrational parameters and complete molecular geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51592 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

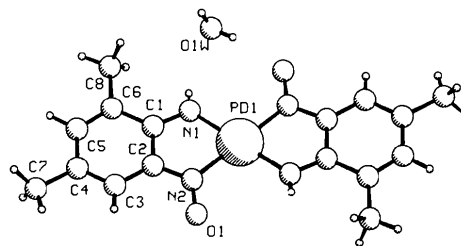


Fig. 1. The centrosymmetric title molecule and associated water drawn using *PLUTO* (Motherwell & Clegg, 1978).