Zhang & Zhang, 1983), and of a neutral derivative $C_5H_5FeC_5H_4CH_2N(CH_3)_2BH_3$ (Fu, Chen, Cai, Pang, Zhang & Zhu, 1985) are consistent with those of ferrocene derivatives and their corresponding borohydride cage anion fragments.

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Structure of (2,2′-Bipyrimidine)tetracarbonylchromium(0)–2,2′-Bipyrimidine

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Abstract. $[Cr(C_8H_6N_4)(CO)_4].C_8H_6N_4$, $M_r = 480{\cdot}36$, triclinic, $P\bar{1}$, $a = 11{\cdot}331\,(3)$, $b = 13{\cdot}701\,(4)$, $c = 7{\cdot}178\,(5)$ Å, $\alpha = 91{\cdot}51\,(4)$, $\beta = 105{\cdot}76\,(4)$, $\gamma = 105{\cdot}34\,(4)^\circ$, $V = 1028{\cdot}41\,(2)$ Å^3, $Z = 2$, $D_x = 1{\cdot}55$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu_1 = 5{\cdot}86$ cm^{-1}, $F(000) = 488$, $T = 294$ K, final $R = 0{\cdot}042$ for 2609 reflections with $I > \sigma(I)$. The tetracarbonyl complex has approximate C_{2v} symmetry and the bipyrimidine molecule is nearly planar. Both the 2,2′-bipyrimidine and the metal complex showing stacking along the crystallographic c axis. The C—O, C—C and C—N bond lengths within the complex and 2,2′-bipyrimidine molecules are normal. Both diimine molecules are essentially planar, with dihedral angles of 9·64 and 3·74° for 2,2′-bipyrimidine and the Cr complex, respectively.

Experimental. The complex was formed by refluxing equal molar equivalents of 2,2′-bipyrimidine and $Cr(CO)_6$ in THF for 12 h. Upon cooling, dark crystals deposited from solution. Black prism, 0·25 × 0·25 × 0 × 15 mm, Enraf–Nonius CAD-4 diffractometer, graphite monochromator, Mo $K\alpha$ radiation, lattice parameters from least-squares refinement using 25 accurately centered reflections with $14 < 2\theta < 35^\circ$, space group $P\bar{1}$ determined from intensity data and successful solution and refinement of structure; 3611 unique reflections collected of which 2609 were considered observed at greater than one $\sigma(I)$ level [$\sigma(I)$ from counting statistics] and used in refinement of structure; $\theta_{\max} = 25^\circ$, $\omega/2\theta$ scans with scan range $0{\cdot}6^\circ + 0{\cdot}34^\circ\tan\theta$ and variable scan speeds of 1–5° min^{-1}, three standard reflections $(25\bar{1}, \bar{1}40, 0\bar{1}3)$ were measured every 180 min of X-ray exposure, no significant deviation noted (less than 1%); collected data: h, $\pm k$, $\pm l$, to max. indices of 13, 16, 18. Data corrected for Lp and background but not for absorption, intensities of equivalent reflections were averaged, agreement factor for averaging of 522 observed reflections was 1·4% based on intensity. Structure solved by direct methods (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and

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Table 1. *Coordinates and B_{eq} for the non-*H *atoms for (2,2′-bipyrimidine)*$Cr(CO)_4$.*2,2′-bipyrimidine*

B_{eq} is defined as $\frac{1}{3}$ the trace of the B_{ij} matrix.

	x	y	z	B_{eq}(Å^2)
Cr	0·17652 (4)	0·35239 (3)	0·12480 (7)	4·02 (1)
O1	0·0827 (3)	0·4038 (2)	−0·2884 (4)	8·31 (8)
O2	0·1942 (2)	0·2577 (2)	0·5049 (4)	7·00 (7)
O3	−0·0816 (2)	0·3603 (2)	0·1404 (4)	8·06 (8)
O4	0·0546 (2)	0·1358 (2)	−0·0417 (5)	9·57 (9)
N1	0·2862 (2)	0·5008 (2)	0·2471 (3)	3·63 (5)
N2	0·3633 (2)	0·3577 (2)	0·1198 (3)	3·32 (5)
N3	0·4973 (3)	0·6129 (2)	0·3494 (4)	4·87 (7)
N4	0·5809 (2)	0·4583 (2)	0·2219 (4)	4·34 (6)
N5	0·4565 (2)	0·0860 (2)	0·3134 (4)	4·17 (6)
N6	0·7101 (2)	0·1294 (2)	0·3537 (4)	4·70 (7)
N7	0·4260 (2)	−0·0805 (2)	0·1694 (4)	4·40 (6)
N8	0·6833 (2)	−0·0451 (2)	0·2632 (4)	4·81 (6)
C1	0·1278 (3)	0·3895 (3)	−0·1315 (5)	5·36 (8)
C2	0·1964 (3)	0·2995 (2)	0·3697 (5)	4·80 (8)
C3	0·0203 (3)	0·3602 (3)	0·1384 (5)	5·26 (8)
C4	0·1025 (3)	0·2213 (3)	0·0216 (6)	5·99 (9)
C5	0·4120 (3)	0·5247 (2)	0·2728 (4)	3·45 (6)
C6	0·2416 (3)	0·5753 (2)	0·3059 (5)	5·23 (8)
C7	0·3243 (4)	0·6703 (2)	0·3844 (5)	5·90 (9)
C8	0·4505 (4)	0·6859 (2)	0·4021 (5)	6·0 (1)
C9	0·4570 (2)	0·4424 (2)	0·2020 (4)	3·25 (6)
C10	0·3998 (3)	0·2817 (2)	0·0475 (4)	4·04 (7)
C11	0·5254 (3)	0·2909 (2)	0·0645 (5)	4·68 (7)
C12	0·6138 (3)	0·3802 (3)	0·1536 (5)	4·89 (8)
C13	0·4983 (3)	0·0117 (2)	0·2560 (4)	3·50 (6)
C14	0·3310 (3)	0·0653 (2)	0·2798 (5)	4·85 (8)
C15	0·2469 (3)	−0·0262 (3)	0·1910 (5)	5·01 (8)
C16	0·3009 (3)	−0·0975 (3)	0·1393 (5)	5·09 (9)
C17	0·6392 (3)	0·0334 (2)	0·2920 (4)	3·62 (6)
C18	0·8353 (3)	0·1467 (3)	0·3866 (5)	5·79 (9)
C19	0·8917 (3)	0·0717 (3)	0·3603 (6)	5·9 (1)
C20	0·8089 (3)	−0·0241 (3)	0·2990 (5)	5·67 (9)

Table 2. *Bond lengths* (Å) *and angles* (°)

Cr	N1	2·104 (2)	N5	C13	1·330 (5)
Cr	N2	2·108 (2)	N5	C14	1·326 (4)
Cr	C1	1·896 (4)	N6	C17	1·338 (3)
Cr	C2	1·898 (4)	N6	C18	1·327 (4)
Cr	C3	1·830 (4)	N7	C13	1·336 (4)
Cr	C4	1·810 (3)	N7	C16	1·329 (4)
O1	C1	1·149 (4)	N8	C17	1·334 (4)
O2	C2	1·143 (5)	N8	C20	1·324 (4)
O3	C3	1·160 (4)	C5	C9	1·485 (4)
O4	C4	1·173 (5)	C6	C7	1·386 (4)
N1	C5	1·334 (4)	C7	C8	1·359 (6)
N1	C6	1·357 (4)	C10	C11	1·367 (4)
N2	C9	1·340 (3)	C11	C12	1·368 (4)
N2	C10	1·356 (5)	C13	C17	1·491 (4)
N3	C5	1·325 (4)	C14	C15	1·372 (4)
N3	C8	1·339 (5)	C15	C16	1·373 (6)
N4	C9	1·329 (4)	C18	C19	1·379 (6)
N4	C12	1·344 (5)	C19	C20	1·376 (5)

N1	Cr	N2	75·75 (9)	N3	C5	C9	118·3 (3)
N1	Cr	C1	94·3 (1)	N1	C6	C7	120·5 (3)
N1	Cr	C2	93·7 (2)	C6	C7	C8	118·1 (3)
N1	Cr	C3	98·9 (1)	N3	C8	C7	122·6 (3)
N1	Cr	C4	172·3 (1)	N2	C9	N4	126·6 (3)
N2	Cr	C1	94·1 (1)	N2	C9	C5	114·0 (2)
N2	Cr	C2	93·6 (1)	N4	C9	C5	119·4 (2)
N2	Cr	C3	174·7 (1)	N2	C10	C11	121·4 (2)
N2	Cr	C4	96·6 (1)	N4	C12	C11	122·4 (3)
C1	Cr	C2	170·1 (1)	N5	C14	C15	123·3 (3)
C1	Cr	C3	86·0 (2)	C14	C15	C16	115·6 (4)
C1	Cr	C4	87·4 (2)	N7	C16	C15	123·4 (3)
C2	Cr	C3	87·0 (2)	C9	N4	C12	115·7 (2)
C2	Cr	C4	85·5 (2)	C17	N6	C18	116·1 (3)
C3	Cr	C4	88·7 (2)	C17	N8	C20	115·8 (2)
Cr	N1	C5	117·7 (2)	C13	N5	C14	115·9 (2)
Cr	N1	C6	126·3 (2)	C13	N7	C16	115·6 (3)
C5	N1	C6	116·1 (2)	C10	C11	C12	118·0 (3)
Cr	N2	C9	117·7 (2)	N5	C13	N7	126·1 (3)
Cr	N2	C10	126·4 (2)	N5	C13	C17	116·8 (2)
C9	N2	C10	116·0 (2)	N7	C13	C17	117·0 (3)
C5	N3	C8	115·7 (3)	N6	C17	N8	125·9 (3)
Cr	C1	O1	171·1 (3)	N6	C17	C13	117·1 (3)
Cr	C2	O2	171·0 (2)	N8	C17	C13	117·0 (2)
Cr	C3	O3	176·4 (3)	N6	C18	C19	123·2 (3)
Cr	C4	O4	178·7 (4)	C18	C19	C20	115·2 (3)
N1	C5	N3	127·0 (3)	N8	C20	C19	123·8 (4)
N1	C5	C9	114·6 (2)				

successive difference Fourier maps, model refined by full-matrix least squares based on F, function minimized was $\sum w(|F_o| - |F_c|)^2$; w is defined as $4F_o^2/\sigma^2(F_o^2)$ and $(\sigma F_o)^2 = [S(C + RB + pF_o^2)]/\mathrm{Lp}$ (S = scan rate, C = total integrated peak count, R = scan time/background counting time, B = total background count, and p = factor to downweight intense reflections, set to 0·040). All calculations performed on VAX 11/750 using *SDP* (Frenz, 1978) programs; H atoms included in calculated positions with isotropic thermal parameters to $1·3 \times B_{eq}$ of the atom to which it is bonded and added to structure-factor calculations, all non-H atoms were refined with anisotropic thermal parameters. Model converged with 2609 observations, 298 variables, $R = 0·042$, $wR = 0·051$, max. $\Delta/\sigma = 0·060$, $S = 1·54$, max. residual electron density 0·22 e Å^{-3} associated with Cr. Scattering factors for non-H atoms were taken from Cromer & Waber (1974). Anomalous-dispersion corrections were included in F_c (Ibers & Hamilton, 1964) using values of f' and f'' from Cromer (1974).

The structure showed stacking of both the 2,2′-bipyrimidine and the (2,2′-bipyrimidine)$Cr(CO)_4$ molecules along the crystallographic **c** direction. Metal—ligand and intraligand bond lengths are normal. Closest interionic contact distance is 3·256 Å between atoms N(3) and C(11). A view of the tetracarbonyl complex and bipyrimidine molecule with chemical numbering schemes is presented in Fig. 1. Atomic coordinates are given in Table 1 and bond lengths and angles are given in Table 2.*

Related literature. A number of diimine tetracarbonyl complexes of Group 6 metals are known [Stoddard, 1962; Cambridge Structural Database, 1980 (128 well determined bipyridine complexes)]. Several reports of mono- and binuclear d^6-metal—carbonyl complexes of 2,2′-bipyrimidine (Overton & Connor, 1982; Kain, 1984) have appeared; however, no structural data were reported. Binuclear μ-2,2′-bipyrimidine-bridged ruthenium complexes have been of interest (Hunziker & Ludi, 1977; Ruminski & Petersen, 1982).

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, the positional and isotropic thermal parameters for the H atoms, r.m.s. amplitudes of thermal vibrations, torsional angles, least-squares planes, and short non-bonded distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51404 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

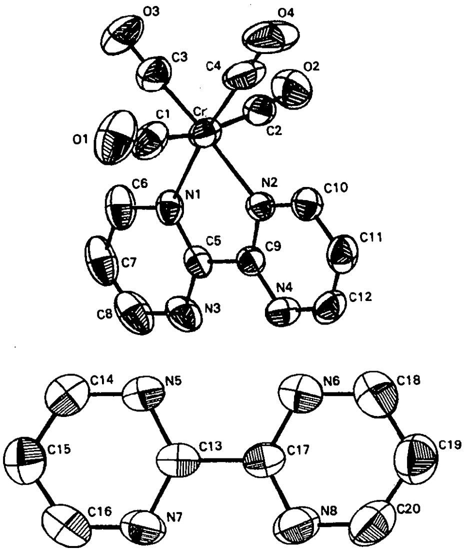

Fig. 1. A view of the (2,2'-bipyrimidine)Cr(CO)$_4$ and 2,2'-bipyrimidine complex molecules.

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Structure of α-*trans*-Cinnamic Acid

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Abstract. 3-Phenyl-2-propenoic-acid, $C_9H_8O_2$, M_r = 148·2, monoclinic, $P2_1/n$, a = 5·582 (2), b = 17·671 (4), c = 7·735 (2) Å, β = 96·49 (2)°, V = 758·0 (3) Å^3, Z = 4, D_x = 1·298 g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ = 0·46 cm^{-1}, $F(000)$ = 312, T = 193 (1) K, final R = 0·041 for 1724 unique reflections. The molecule is planar with a slight decrease of the C—C single bonds [C(1)—C(2) = 1·469 (2), C(3)—C(4) = 1·467 (2) Å] and an increase of the C=C double bond [C(2)—C(3) = 1·334 (2) Å] as a result of conjugation. The structural units are dimers formed by the hydrogen bonding of carboxyl groups across centres of symmetry, the O···O distance being 2·634 (2) Å.

Experimental. Cinnamic acid was recrystallized from ethanol. A crystal of size 0·2 × 0·3 × 0·3 mm was used for diffraction measurement. Unit-cell parameters were obtained from a least-squares refinement of 25 reflections. Nicolet $R3m/V$ diffractometer, equipped with an LT-1 low temperature device, and graphite-monochromated radiation. The intensities of 1982 reflections were measured in the 2θ range 4–45°, with the $\theta/2\theta$ scan mode. Index ranges $0 < h < 7$, $0 < k < 22$, $-10 < l < 10$. Three intensity control reflections were monitored every 60 reflections of data collected; no crystal decay was observed. 1724 unique reflections ($R_{\rm int}$ = 0·026) of which 1503 were considered observed, the criterion $F_o > 4\sigma(F_o)$, were used in the refinement. Lorentz–polarization and empirical absorption corrections (ψ scans) were applied. The structure was

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