GREENWOOD, N. N. & EARNSHAW, A. (1984). *Chemistry of the Elements.* New York: Pergamon Press.

JOHNSON, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MOKUOLU, J. A. A., PAYNE., D. S. & SPEAKMAN, J. C. (1973). *J. Chem. Soc. Dalton Trans.* pp. 1443–1445.

NOTH, H. & FLUCH, E. (1984). *Z. Naturforsch. Teil B,* **39,** 744–753.

SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination and refinement. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS86.* Program for crystal structure determination. Univ. of Göttingen, Germany.

STEFFEN, W. L. & PALENIK, G. J. (1976). *Inorg. Chem.* **15,** 2432–2438.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A39, 158–166.

An Organometallic Cyanine: μ-1,3,5-Heptatriene-1,7-diylidene-bis[μ^2-carbonyldicarbonylbis(η^5-cyclopentadienyl)diiron] Tetrafluoroborate

BY WILLIAM P. SCHAEFER

Division of Chemistry and Chemical Engineering* and The Beckman Institute, Mail Code 139-74, California Institute of Technology, Pasadena, California 91125, USA

AND JAMES M. SPOTTS AND SETH R. MARDER

Jet Propulsion Laboratory, California Institute of Technology, Pasadena, California 91109, USA and Molecular Materials Resource Center, The Beckman Institute, Mail Code 139-74, California Institute of Technology, Pasadena, California 91125, USA

Abstract. $C_{33}H_{25}Fe_4O_6^+.BF_4^-$, $M_r = 827.75$, monoclinic, $C2/c$, $a = 32.289$ (10), $b = 8.984$ (3), $c = 28.024$ (10) Å, $\beta = 126.84$ (2)°, $V = 6506$ (4) Å^3, $Z = 8$, $D_x = 1.69$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71073$ Å, $\mu = 18.2$ cm^{-1}, $F(000) = 3328$, $T = 297$ K, $R = 0.047$ for 2619 reflections with $F_o^2 > 0$. This compound has an intense low-energy visible absorption band ($\varepsilon = 165\,500$ at 630 nm); the material is likely to be useful in non-linear optical devices. The Fe$_2$—C$_7$—Fe$_2$ group is planar within $\pm$ 0.14 Å and all the C—C bonds in the bridge are essentially the same length, 1.379 (11) Å.

Introduction. Organometallic compounds are potentially useful as second-order and third-order non-linear optical materials (Jones, 1989; Marder, Sohn & Stucky, 1991). The most useful materials will have large extinction coefficients, obtained by optimizing the electronic coupling between the metal center(s) and the organic ligand. Because organic cyanines have among the largest extinction coefficients known, we have synthesized organometallic cyanines and examined them for useful properties, reasoning that these organometallic analogs would have low-energy transitions with large transition dipole moments. The title compound was synthesized by the reaction of {(η-C$_5$H$_5$)$_2$Fe$_2$(CO)$_2$(μ-CO)(μ-C-CH$_3$)}$^+$.BF$_4^-$ with 1,1′,3,3′-tetramethoxypropane,

with added water in THF at 333 K. The compound precipitates as essentially pure blue microcrystals from the reaction mixture in 60% yield. The ultraviolet–visible spectrum of the compound, in methylene chloride, shows an intense band centered at 630 nm with an extinction coefficient of 160 000 LM^{-1}cm^{-1} and an oscillator strength of 1.12. The compound thus fulfills our expectations for a highly absorbing material with a low-energy transition. This low-energy absorption is most likely a consequence of high-energy orbitals of the electropositive iron centers mixing efficiently with the frontier orbitals of the carbon skeleton. This highly delocalized organometallic cyanine is a prototype for a class of materials which may be of interest for electron transfer and non-linear optical studies that are currently under way.

Experimental. Material synthesized as described by Spotts, Schaefer & Marder (1991); crystal a rectangular plate, $0.077 \times 0.250 \times 0.378$ mm; CAD-4 diffractometer; cell dimensions from 25 reflections with $30 < 2\theta < 33°$; a cell with a smaller β angle, but with symmetry $C2/n$, can be obtained by transformation with the matrix ($\bar{1}$00; 0$\bar{1}$0; 101) ($c = 27.255$ Å, $\beta = 124.62°$), or one with $I2/a$ symmetry with the matrix ($\bar{1}$0$\bar{1}$; 0$\bar{1}$0; 001) ($a = 27.255$, $b = 8.984$, $c = 28.024$ Å, $\beta = 108.54°$); we chose to use the standard $C2/c$ setting; analytical absorption correction by Gaussian integration over an $8 \times 8 \times 8$ grid, maximum trans-

---

* Contribution No. 8234.

mission 0.874, minimum 0.640 (an empirical correction gave similar results); $(\sin\theta/\lambda)_{max} = 0.48 \text{ Å}^{-1}$; h from -23 to 23, k from -6 to 6, l from 0 to 20; standard reflections $51\bar{8}$, $91\bar{3}$ and $\bar{4}20$ showed a small (0.3%) linear increase in intensities which was corrected for; 8560 reflections measured, 2803 independent; goodness of fit for merging, 0.963, R_{merge} for 2237 reflections with exactly two measurements 0.023; all reflections used in solution and refinement; structure solved by *MULTAN* (the Fe atoms) and successive structure factor–Fourier calculations for the rest of the heavy atoms; F_o^2 magnitudes used in refinement minimizing $\sum w(F_o^2 - F_c^2)^2$; H atoms placed at calculated positions 0.95 Å from C atoms; H positions not refined, but adjusted once near the conclusion of the refinement; 432 positional and anisotropic thermal parameters of all non-hydrogen atoms plus a scale factor refined in one full matrix; $R = 0.047$ for 2619 reflections with $F_o^2 > 0$, 0.037 for 2216 reflections with $F_o^2 > 3\sigma(F_o^2)$; wR on $F^2 = 0.0065$, goodness of fit = 2.02. Variances $[\sigma^2(F_o^2)]$ derived from counting statistics plus an additional term, $(0.014\,I)^2$; variances of the merged data by propagation of error plus another additional term $(0.014\,I)^2$; factor 0.014 derived from our previous experience [see, for example, Marsh & Schaefer (1968)]; ratio of maximum least-squares shift to e.s.d., 0.16 for B atom, 0.05 for other atoms; largest peaks in final difference Fourier map, $+0.63$ and -0.57 e Å^{-3}; computer programs were those of the *CRYRM Crystallographic Computing System* (Duchamp, 1964), *MULTAN* (Debaerdemaeker *et al.*, 1988) and *ORTEP* (Johnson, 1976). Atomic scattering factors were from *International Tables* (Cromer & Waber, 1974). The final refined parameters are given in Table 1 and selected distances and angles in the cation are listed in Table 2.*

Discussion. Our interests in the structure of this organometallic cyanine were twofold: to confirm that the bridging chain had equivalent C—C bonds, and to see whether the Fe—C bonds to the bridge showed evidence of double-bond character. The entire chromophore is planar within ± 0.14 Å – all four Fe atoms and the seven bridging C atoms. The C—C bond lengths range from 1.366 (11) to 1.391 (11) Å, with average 1.379 (8) Å: clearly they are all the same within experimental error. The angles C—C—C in the bridge are not quite as regular, ranging from 122.4 (7) to 127.3 (7)°, with average

Table 1. *Final refined parameters ($\times 10^4$) for bis-Cp tricarbonyl diiron dimer*

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{eq}(\text{Å}^2)$
C1	3606 (3)	5089 (8)	-2784 (3)	573 (22)
O1	4017 (2)	5563 (7)	-2554 (3)	1007 (21)
C2	2942 (2)	3990 (8)	-3848 (3)	500 (23)
O2	2872 (2)	4648 (6)	-4255 (2)	746 (17)
C3	3702 (3)	2119 (8)	-3295 (3)	482 (20)
O3	4124 (2)	2193 (6)	-3122 (2)	780 (18)
Fe1	2984 (0.3)	4384 (1)	-3144 (0.4)	418 (3)
Fe2	3056 (0.3)	1998 (1)	-3555 (0.4)	431 (3)
C4	2184 (3)	4885 (13)	-3687 (4)	752 (30)
C5	2322 (4)	4184 (10)	-3169 (7)	884 (31)
C6	2685 (5)	5041 (17)	-2689 (4)	928 (35)
C7	2768 (3)	6288 (11)	-2921 (5)	814 (32)
C8	2458 (4)	6181 (10)	-3523 (5)	744 (30)
C9	2985 (4)	-291 (9)	-3648 (6)	938 (39)
C10	2614 (5)	245 (12)	-3577 (5)	766 (28)
C11	2291 (3)	1178 (11)	-4050 (6)	792 (30)
C12	2436 (5)	1214 (12)	-4410 (4)	908 (35)
C13	2850 (6)	367 (15)	-4190 (7)	962 (34)
C14	3262 (2)	2537 (7)	-2794 (3)	421 (18)
C15	3487 (3)	1782 (9)	-2270 (4)	434 (19)
C16	3616 (2)	2367 (7)	-1737 (4)	426 (19)
C17	3814 (2)	1534 (7)	-1230 (3)	394 (18)
C18	3940 (2)	2061 (7)	-695 (3)	398 (18)
C19	4149 (2)	1157 (7)	-200 (3)	383 (18)
C20	4254 (2)	1533 (6)	338 (3)	338 (17)
Fe3	4538 (0.3)	460 (1)	1042 (0.4)	381 (3)
Fe4	4168 (0.3)	3035 (1)	728 (0.4)	402 (3)
C24	5224 (4)	206 (1)	1136 (7)	962 (43)
C25	5338 (4)	869 (10)	1630 (9)	993 (49)
C26	5205 (4)	-67 (22)	1898 (4)	879 (42)
C27	5001 (4)	-1321 (13)	1560 (8)	862 (36)
C28	5015 (4)	-1145 (16)	1080 (6)	878 (38)
C21	3968 (3)	-601 (8)	649 (3)	462 (19)
O21	3599 (2)	-1302 (5)	373 (2)	691 (16)
C22	4280 (3)	1730 (8)	1356 (4)	508 (22)
O22	4227 (2)	1787 (6)	1736 (3)	801 (17)
C23	3505 (3)	2649 (12)	287 (3)	508 (21)
O23	3071 (2)	2407 (6)	-9 (3)	851 (20)
C29	4836 (3)	4380 (9)	1179 (6)	889 (41)
C30	4563 (5)	4654 (9)	590 (6)	749 (28)
C31	4094 (4)	5157 (8)	383 (4)	747 (34)
C32	4060 (4)	5240 (9)	855 (8)	906 (36)
C33	4532 (8)	4738 (12)	1368 (5)	1083 (46)
B	1076 (5)	1684 (16)	6048 (7)	908 (39)
F1	1453 (2)	753 (6)	6187 (3)	1384 (20)
F2	1214 (3)	2385 (8)	6549 (3)	1734 (28)
F3	1018 (4)	2644 (7)	5684 (3)	2408 (45)
F4	667 (3)	936 (12)	5830 (5)	2697 (45)

125.1 (20)°, but there is no question that the bridge has equivalent, rather than alternating C—C bonds. A drawing of the cation is shown in Fig. 1. The packing is illustrated in Fig. 2.

The question of the bond order of the Fe—C bonds is less easy to answer. All four bonds are equivalent, averaging 1.866 (8) Å [1.858 (7) to 1.877 (8) Å]. These are significantly shorter than the Fe—C bonds to the bridging carbonyl ligands [average 1.927 (24) Å, compared to 1.930 Å expected], and also significantly shorter than bonds that are presumably single to μ-C—CR_2 groups [Carty, Mott, Taylor, Ferguson, Khan & Roberts (1978), 1.963 (7) Å] or the Fe—C bonds in (η-$C_5H_5)_2Fe_2(CO)_2(\mu$-CO)(μ-CHCH$_3$), 1.986 (3) Å (Orpen, 1983). They are about the same as Fe—C

Table 2. *Distances (Å) and angles (°) not involving hydrogen in the bis-Cp tricarbonyl diiron dimer cation*

Fe1—Cp1	1.744 (1)	C14—C15	1.366 (11)
Fe2—Cp2	1.740 (1)	C15—C16	1.390 (11)
Fe3—Cp3	1.736 (1)	C16—C17	1.378 (11)
Fe4—Cp4	1.741 (1)	C17—C18	1.380 (11)
Fe1—Fe2	2.509 (1)	C18—C19	1.384 (11)
Fe3—Fe4	2.507 (1)	C19—C20	1.376 (10)
C1—O1	1.153 (1)	C20—Fe3	1.868 (7)
C1—Fe1	1.737 (9)	C20—Fe4	1.858 (7)
C2—O2	1.180 (10)	Fe3—C21	1.754 (8)
C2—Fe1	1.927 (8)	Fe3—C22	1.909 (8)
C2—Fe2	1.910 (8)	Fe4—C22	1.959 (8)
C3—O3	1.144 (10)	Fe4—C23	1.750 (8)
C3—Fe2	1.750 (8)	C24—C25	1.34 (2)
Fe1—C14	1.861 (7)	C24—C28	1.35 (2)
Fe2—C14	1.877 (7)	C25—C26	1.36 (2)
C4—C5	1.388 (18)	C26—C27	1.36 (2)
C4—C8	1.364 (15)	C27—C28	1.38 (2)
C5—C6	1.37 (2)	C21—O21	1.143 (10)
C6—C7	1.399 (18)	C22—O22	1.175 (10)
C7—C8	1.354 (16)	C23—O23	1.143 (10)
C9—C10	1.412 (18)	C29—C30	1.347 (18)
C9—C13	1.43 (2)	C29—C33	1.40 (2)
C10—C11	1.374 (18)	C30—C31	1.335 (17)
C11—C12	1.347 (18)	C31—C32	1.393 (18)
C12—C13	1.32 (2)	C32—C33	1.40 (2)

Cp1—Fe1—C1	124.3	C15—C14—Fe2	133.8 (6)
Cp1—Fe1—C2	120.3	C16—C15—C14	126.2 (7)
Cp1—Fe1—C14	127.1	C17—C16—C15	123.9 (7)
Cp2—Fe2—C3	125.7	C18—C17—C16	125.9 (7)
Cp2—Fe2—C2	122.8	C19—C18—C17	122.4 (7)
Cp2—Fe2—C14	124.2	C20—C19—C18	127.3 (7)
Cp3—Fe3—C21	124.1	Fe3—C20—C19	132.3 (6)
Cp3—Fe3—C20	123.6	Fe4—C20—C19	142.9 (6)
Cp3—Fe3—C22	124.3	Fe4—C20—Fe3	84.6 (3)
Cp4—Fe4—C23	123.1	C21—Fe3—C20	87.2 (3)
Cp4—Fe4—C20	127.0	C22—Fe3—C20	96.0 (3)
Cp4—Fe4—C22	122.3	C22—Fe3—C21	91.8 (4)
C2—Fe1—C1	90.5 (4)	C22—Fe4—C20	94.7 (3)
C14—Fe1—C1	89.7 (4)	C23—Fe4—C20	89.4 (3)
C14—Fe1—C2	95.3 (3)	C23—Fe4—C22	90.3 (4)
C3—Fe1—C2	89.8 (4)	C28—C24—C25	109.2 (15)
C14—Fe2—C2	95.3 (3)	C26—C25—C24	108.4 (14)
C14—Fe2—C3	89.3 (4)	C27—C26—C25	107.9 (13)
Fe1—C1—O1	178.9 (8)	C28—C27—C26	107.3 (13)
Fe1—C2—O2	138.8 (7)	C27—C28—C24	107.2 (14)
Fe2—C2—O2	139.5 (7)	O21—C21—Fe3	176.9 (7)
Fe2—C2—Fe1	81.7 (3)	Fe4—C22—Fe3	80.8 (3)
Fe2—C3—O3	179.6 (7)	O22—C22—Fe3	141.6 (7)
C8—C4—C5	107.3 (10)	O22—C22—Fe4	137.5 (7)
C6—C5—C4	108.7 (12)	O23—C23—Fe4	178.9 (8)
C7—C6—C5	106.4 (12)	C30—C29—Cp4	54.2
C8—C7—C6	108.5 (11)	C33—C29—Cp4	55.0
C7—C8—C4	109.1 (10)	C30—C29—Cp4	54.2
C13—C9—C10	105.3 (12)	C33—C29—Cp4	55.0
C11—C10—C9	106.9 (11)	C33—C29—C30	109.2 (12)
C12—C11—C10	109.4 (12)	C31—C30—C29	109.4 (11)
C13—C12—C11	110.1 (13)	C32—C31—C30	108.7 (11)
C12—C13—C9	108.3 (14)	C33—C32—C31	107.5 (13)
Fe2—C14—Fe1	84.3 (3)	C32—C33—C29	105.3 (13)
C15—C14—Fe1	141.9 (6)		

Fig. 1. An *ORTEP* drawing of the cation showing the numbering system with 50% probability ellipsoids. H atoms are not shown.

Fig. 2. An *ORTEP* drawing of the structure projected down the *b* axis showing the packing, with a unit cell outlined.

bonds to a μ-C—C*R* group [Smith, Yule, Taylor, Park & Carty (1977), 1.890 Å], but this example is not a perfect comparison. Simple Fe—C single bonds (non-bridging C atoms) appear to be 1.95–2.05 Å (many examples), however, so those in this compound are 0.15 Å or so shorter, indicative of metal—carbon multiple bonding, especially considering that the C atom is bonded to two metal atoms. The pairs of Fe atoms 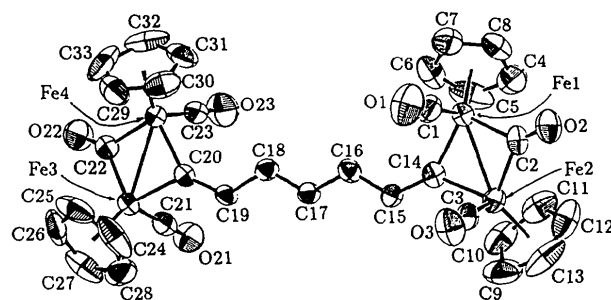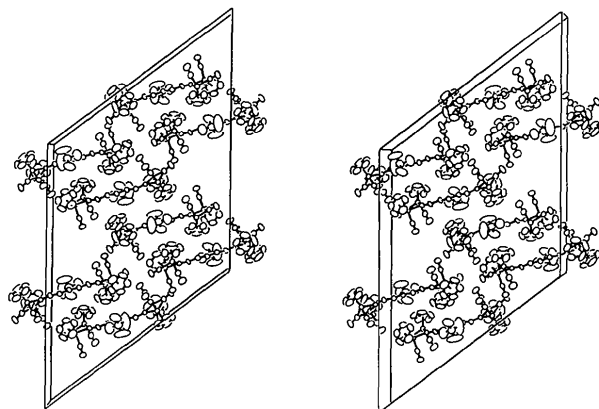at the ends of the chromophore are joined by a moderately strong metal–metal bond, 2.508 (1) Å, compared to 2.482 Å in α-iron metal (eight coordinate). This compound is structurally analogous to $\{[(\eta\text{-}C_5H_5)_2Fe_2(CO)_2[\mu\text{-}CO)]_2[\mu\text{-}C\text{—}(CH\text{=}CH)\text{—}CH\text{=}C]\}^+.BF_4^-$ (Casey, Konings & Haller, 1986; Casey, Konings & Marder, 1988), in which Fe—C bond lengths average 1.845 Å and the C—C bonds in the hydrocarbon bridge average 1.37 (1) Å.

Other aspects of the geometry of the complex are normal. The cyclopentadienyl rings are fairly regular and Fe—C distances to those C atoms are only very slightly longer than expected [2.095 (17) *vs* 2.080 (35) Å expected (Orpen, Brammer, Allen, Kennard, Watson & Taylor, 1989, p. S19)]. The carbonyl ligands are also normal. The tetrafluoroborate anion shows high apparent thermal motion with a concomitant shortening of the B—F bonds, especially for F3 and F4. Such a motion (or disorder) is not unexpected for this ion.

References

CARTY, A. J., MOTT, G. N., TAYLOR, N. J., FERGUSON, G., KHAN, M. A. & ROBERTS, P. J. (1978). *J. Organomet Chem.* **149**, 345–354.

CASEY, C. P., KONINGS, M. S. & HALLER, K. J. (1986). *J. Organomet. Chem.* **301**, C55–C58.

CASEY, C. P., KONINGS, M. S. & MARDER, S. R. (1988). *Polyhedron*, 7, 881–902.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 149–151. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 99–101. (Present distributor Kluwer Academic Publishers, Dordrecht.)

DEBAERDEMAEKER, T., GERMAIN, G., MAIN, P., REFAAT, L. S., TATE, C. & WOOLFSON, M. M. (1988). *MULTAN88. Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univ. of York, England.

DUCHAMP, D. J. (1964). ACA Meeting, Bozeman, Montana, paper B-14, p. 29.

JOHNSON, C. K. (1976). *ORTEPII*. Report ORNL-3794, third revision. Oak Ridge National Laboratory, Tennessee, USA.

JONES, R. J. (1989). *Organic Materials for Non-linear Optics*, edited by R. A. HANN & D. BLOOR, pp. 225–231. Royal Society of Chemistry, London, England.

MARDER, S. R., SOHN, J. E. & STUCKY, G. D. (1991). *Materials for Non-linear Optics: Chemical Perspectives. ACS Symp. Ser.* Vol. 455. American Chemical Society, Washington, USA.

MARSH, R. E. & SCHAEFER, W. P. (1968). *Acta Cryst.* B24, 246–251.

ORPEN, A. G. (1983). *J. Chem. Soc. Dalton Trans.* p. 1427.

ORPEN, A. G., BRAMMER, L., ALLEN, F. H., KENNARD, O., WATSON, D. G. & TAYLOR, R. (1989). *J. Chem. Soc. Dalton Trans.* S1.

SMITH, W. F., YULE, J., TAYLOR, N. J., PARK, H. N. & CARTY, A. J. (1977). *Inorg. Chem.* 16, 1593–1600.

SPOTTS, J. M., SCHAEFER, W. P. & MARDER, S. R. (1991). *J. Am. Chem. Soc.* Submitted.

Structure of Bromodicyano(2,2':6',2''-terpyridine)gold(III)

BY VALERIA FERRETTI, PAOLA GILLI AND VALERIO BERTOLASI

Dipartimento di Chimica e Centro di Strutturistica Diffrattometrica, Università di Ferrara, Via L. Borsari 46, 44100 Ferrara, Italy

AND GIAMPAOLO MARANGONI, BRUNO PITTERI AND GAVINO CHESSA

Dipartimento di Chimica, Facoltà di Chimica Industriale, Università di Venezia, Calle Larga S. Marta 2137, 30123 Venezia, Italy

Abstract. $C_{17}H_{11}AuBrN_5$, $M_r = 562.19$, monoclinic, $P2_1/c$, $a = 13.229$ (2), $b = 11.069$ (2), $c = 12.849$ (1) Å, $\beta = 117.92$ (9)°, $V = 1661.3$ (4) Å^3, $Z = 4$, $D_x = 2.25$ g cm^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 112.3$ cm^{-1}, $F(000) = 1048$, $T = 298$ K, $R = 0.030$ for 2186 observed independent reflections. The coordination is distorted square-pyramidal and terpyridine acts as a bidentate ligand with one N atom in the basal square [Au—N = 2.078 (6) Å] and the other in the apical position at a distance of 2.839 (5) Å which is intermediate between the sum of the covalent and van der Waals radii. The geometry of the coordination polyhedron is discussed by comparing the present structure with other square-pyramidal or square-planar Au^{III} complexes of known molecular structure.

Introduction. The only Au complexes of 2,2':6',2''-terpyridine (terpy) reported and structurally characterized are [Au(terpy)Cl]Cl$_2$.3H$_2$O and the mixed-valence compound [Au(terpy)Cl]$_2$[AuCl$_2$]$_3$[AuCl$_4$] (Hollis & Lippard, 1983), in both of which terpy acts as a terdentate ligand and the [Au(terpy)Cl]$^{2+}$ cation displays a square-planar geometry. We report here the crystal structure of the neutral complex bromodicyano(terpy)gold(III) which, to our knowledge, is an uncommon example of a coordination compound where terpy acts as a bidentate ligand in a distorted square-pyramidal coordination environment.

Experimental. Bromodicyano(terpy)gold(III) was prepared by adding 2,2':6',2''-terpyridine (0.233 g, 1 mmol) dissolved in ethanol (2 cm^3) to a solution of *trans*-K[Au(CN)$_2$Br$_2$].3H$_2$O (0.502 g, 1 mmol) in water (20 cm^3) at 273 K with stirring. The pale yellow product that precipitated immediately was filtered off, washed with cold water, and dried under reduced pressure (0.534 g, 95%). It was crystallized from a concentrated solution of the crude product in dimethylformamide–diethyl ether (1:4, v:v) at 263 K (found: C, 36.2; H, 1.91; N, 12.3; Br 14.7%; C$_{17}$H$_{11}$AuBrN$_5$ requires C, 36.3; H, 1.97; N, 12.5; Br, 14.2%).

Single crystal of dimensions 0.19 × 0.21 × 0.31 mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\omega/2\theta$ scan ($2 \leq \theta \leq 27°$); cell parameters from 25 reflections in the range $8 \leq \theta \leq 14°$; 3614 independent