ring are very close to 90°, in accord with the phosphorus analog. The N atom and the C skeleton of the cyclobutane ring lie in a plane (see Fig. 1), the deviations being: N, 0·003; C(1), 0·002; C(2), –0·007; C(3), 0·009; C(4), –0·007 Å. C(9) also lies close to the plane of the cyclobutane ring, the deviation from the plane being only 0·04 Å.

The N ylide described herein is the first example of a stable halogenated ylide – capable of $\beta$-elimination of halide – in which the negative charge on the ylide C atom has not been delocalized either by a resonance interaction or by overlap with the $d$ orbitals of an adjacent atom, as in the phosphorus analog. $\beta$-stabilization of the negative charge by the adjacent difluoromethyl groups [note that the C(3)–F(3) distances are shorter] presumably accounts for the stability of the ylide, since if other halogens (Cl, Br) are incorporated at the site $\alpha$ to the ylide C, $\beta$-elimination readily occurs.

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# The Adduct Zinc Dicyanide–2,9-Dimethyl-1,10-phenanthroline

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**Abstract.** $Zn(CN)_2.C_{14}H_{12}N_2$, $M_r = 325{\cdot}7$, monoclinic, space group $P2_1/c$, $a = 10{\cdot}989$ (1), $b = 10{\cdot}028$ (1), $c = 13{\cdot}897$ (2) Å, $\beta = 106{\cdot}54$ (1)°, $U = 1468{\cdot}1$ (2) Å$^3$, $Z = 4$, $D_c = 1{\cdot}47$ g cm$^{-3}$, $\mu$(Mo $K\alpha$) = 17·1 cm$^{-1}$. The atomic parameters have been refined by least-squares analysis including 2436 observed reflexions to an $R$ value of 0·036. The Zn atom presents distorted tetrahedral coordination. The bidentate phenanthroline group forms an angle of 85° with the plane defined by the Zn atom and the two cyanide ligands.

**Introduction.** A clear colourless, prismatic crystal prepared as described by Cano Esquivel (1975) was mounted on a computer-controlled four-circle PW 1100 diffractometer. The cell dimensions were refined by least-squares fitting of the $\theta$ values of 36 reflexions. The intensities of 3193 reflexions within $2 \leq \theta \leq 27°$ were collected at 22 °C with monochromatic Mo $K\alpha$ radiation and an $\omega/2\theta$ scan technique. Two reflexions were monitored periodically during the data collection and showed no crystal decomposition. An absorption correction was applied with the program *ORABS* (Schwarzenbach, 1972). The intensities were corrected for the Lorentz and polarization effects and 757 of these were considered as unobserved by the criterion $I < 2\sigma(I)$.

Scattering factors for neutral atoms and the

Table 1. *Coefficients for the weighting scheme*

| | $a$ | $b$ | | $c$ | $d$ |
|---|---|---|---|---|---|
| $\lvert F_o\rvert \leq 10$ | 2·08 | –0·11 | $\sin\theta/\lambda \leq 0{\cdot}43$ Å$^{-1}$ | 2·74 | –4·80 |
| $10 < \lvert F_o\rvert \leq 18$ | 1·02 | –0·01 | $\sin\theta/\lambda > 0{\cdot}43$ | –0·02 | 1·71 |
| $18 < \lvert F_o\rvert$ | 0·14 | 0·04 | | | |

anomalous-dispersion corrections for Zn were taken from *International Tables for X-ray Crystallography* (1974). Phases of the 150 highest normalized structure factors ($E > 1{\cdot}9$) were calculated by the multisolution tangent formula using the program *MULTAN* (Main, Woolfson, Declercq & Germain, 1974). The best phased $E$ map revealed all the non-hydrogen atoms. After several cycles of anisotropic full-matrix least-squares refinement the conventional $R$ index for the observed reflexions was 0·052. A difference synthesis calculated with those reflexions within $\sin\theta/\lambda < 0{\cdot}5$ Å$^{-1}$ showed all H atoms as the highest peaks of the

Table 2. *Positional parameters* ($\times 10^5$, *for* H $\times 10^3$) *for* $\mathrm{Zn(CN)_2.C_{14}H_{12}N_2}$

Standard deviations are given in parentheses.

| | *x* | *y* | *z* |
|---|---|---|---|
| Zn | 74854 (3) | 22395 (3) | 2637 (2) |
| N(1) | 83148 (20) | 41052 (23) | 5426 (15) |
| N(2) | 59965 (20) | 33649 (21) | −6030 (14) |
| N(3) | 84769 (33) | 4382 (35) | −11840 (27) |
| N(4) | 70250 (29) | 11635 (31) | 22354 (21) |
| C(1) | 94451 (27) | 44486 (34) | 11522 (19) |
| C(2) | 98725 (30) | 57821 (40) | 11785 (24) |
| C(3) | 91454 (33) | 67234 (36) | 6013 (25) |
| C(4) | 79346 (28) | 63964 (28) | −279 (21) |
| C(5) | 70850 (35) | 73373 (30) | −6459 (25) |
| C(6) | 59191 (32) | 69679 (29) | −11982 (23) |
| C(7) | 54976 (26) | 56277 (27) | −11972 (17) |
| C(8) | 63226 (23) | 46674 (25) | −6274 (16) |
| C(9) | 75633 (24) | 50606 (27) | −263 (17) |
| C(10) | 42782 (28) | 51883 (32) | −17511 (19) |
| C(11) | 39665 (27) | 38834 (34) | −17137 (21) |
| C(12) | 48461 (26) | 29724 (28) | −11377 (19) |
| C(13) | 45284 (33) | 15245 (35) | −11027 (25) |
| C(14) | 102050 (32) | 33980 (49) | 18029 (25) |
| C(15) | 81568 (30) | 10939 (31) | −6396 (25) |
| C(16) | 71855 (27) | 15254 (30) | 15128 (22) |
| H(2) | 1065 (4) | 597 (4) | 159 (3) |
| H(3) | 944 (4) | 763 (4) | 62 (3) |
| H(5) | 738 (3) | 818 (3) | −63 (2) |
| H(6) | 541 (3) | 758 (3) | −156 (2) |
| H(10) | 369 (3) | 583 (4) | −213 (2) |
| H(11) | 320 (3) | 356 (3) | −205 (2) |
| H(131) | 473 (4) | 124 (5) | −48 (3) |
| H(132) | 503 (5) | 102 (5) | −134 (4) |
| H(133) | 368 (6) | 139 (6) | −140 (4) |
| H(141) | 1012 (4) | 253 (4) | 150 (3) |
| H(142) | 987 (4) | 324 (5) | 228 (3) |
| H(143) | 1100 (4) | 359 (5) | 203 (3) |

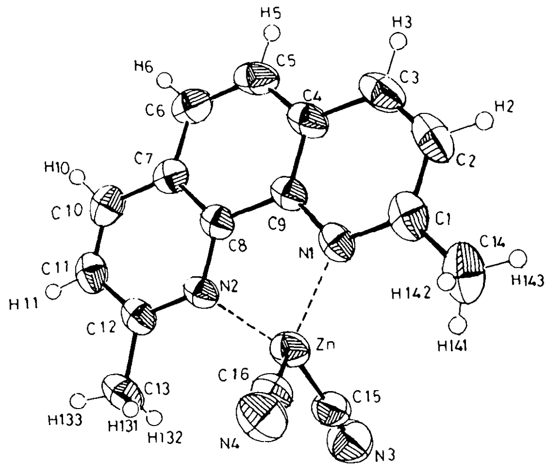

Fig. 1. *ORTEP* drawing of $\mathrm{Zn(CN)_2.C_{14}H_{12}N_2}$ (Johnson, 1965). Thermal ellipsoids for non-hydrogen atoms are scaled to 50% probability.

Table 3. *Bond lengths* (Å) *and bond angles* (°) *in* $\mathrm{Zn(CN)_2.C_{14}H_{12}N_2}$

Standard deviations

| | σ(Å) | σ(°) |
|---|---|---|
| Involving H atoms | 0·040 | 4·0 |
| Involving Zn atoms | 0·002 | 0·1 |
| Involving other atoms | 0·004 | 0·3 |

| | | | |
|---|---|---|---|
| Zn—C(15) | 1·992 | C(7)—C(10) | 1·413 |
| —C(16) | 1·990 | C(8)—C(9) | 1·437 |
| —N(1) | 2·069 | —N(2) | 1·357 |
| —N(2) | 2·069 | C(9)—N(1) | 1·361 |
| C(1)—C(2) | 1·414 | C(10)—C(11) | 1·357 |
| —C(14) | 1·482 | —H(10) | 0·96 |
| —N(1) | 1·334 | C(11)—C(12) | 1·403 |
| C(2)—C(3) | 1·345 | —H(11) | 0·90 |
| —H(2) | 0·90 | C(12)—C(13) | 1·497 |
| C(3)—C(4) | 1·407 | —N(2) | 1·331 |
| —H(3) | 0·96 | C(13)—H(131) | 0·87 |
| C(4)—C(5) | 1·429 | —H(132) | 0·88 |
| —C(9) | 1·401 | —H(133) | 0·92 |
| C(5)—C(6) | 1·345 | C(14)—H(141) | 0·96 |
| —H(5) | 0·91 | —H(142) | 0·86 |
| C(6)—C(7) | 1·422 | —H(143) | 0·86 |
| —H(6) | 0·89 | C(15)—N(3) | 1·131 |
| C(7)—C(8) | 1·403 | C(16)—N(4) | 1·128 |

| | | | |
|---|---|---|---|
| C(15)—Zn—C(16) | 121·7 | C(7)—C(8)—N(2) | 122·8 |
| C(15)—Zn—N(2) | 108·7 | C(9)—C(8)—N(2) | 117·6 |
| C(15)—Zn—N(1) | 114·7 | C(4)—C(9)—C(8) | 119·4 |
| C(16)—Zn—N(2) | 113·8 | C(4)—C(9)—N(1) | 123·0 |
| C(16)—Zn—N(1) | 109·6 | C(8)—C(9)—N(1) | 117·6 |
| N(1)—Zn—N(2) | 81·3 | C(7)—C(10)—C(11) | 119·4 |
| N(1)—C(1)—C(14) | 117·7 | C(7)—C(10)—H(10) | 118 |
| C(2)—C(1)—C(14) | 122·1 | C(11)—C(10)—H(10) | 122 |
| C(2)—C(1)—N(1) | 120·2 | C(10)—C(11)—C(12) | 120·6 |
| C(1)—C(2)—C(3) | 120·8 | C(10)—C(11)—H(11) | 122 |
| C(1)—C(2)—H(2) | 117 | C(12)—C(11)—H(11) | 117 |
| C(3)—C(2)—H(2) | 122 | C(11)—C(12)—N(2) | 121·1 |
| C(2)—C(3)—C(4) | 120·2 | C(11)—C(12)—C(13) | 121·4 |
| C(2)—C(3)—H(3) | 121 | C(13)—C(12)—N(2) | 117·5 |
| C(4)—C(3)—H(3) | 119 | C(12)—C(13)—H(131) | 111 |
| C(3)—C(4)—C(5) | 124·1 | C(12)—C(13)—H(132) | 112 |
| C(3)—C(4)—C(9) | 116·5 | C(12)—C(13)—H(133) | 110 |
| C(5)—C(4)—C(9) | 119·4 | H(131)—C(13)—H(132) | 100 |
| C(4)—C(5)—C(6) | 121·0 | H(131)—C(13)—H(133) | 109 |
| C(4)—C(5)—H(5) | 116 | H(132)—C(13)—H(133) | 115 |
| C(6)—C(5)—H(5) | 123 | C(1)—C(14)—H(141) | 114 |
| C(5)—C(6)—C(7) | 121·2 | C(1)—C(14)—H(142) | 108 |
| C(5)—C(6)—H(6) | 119 | C(1)—C(14)—H(143) | 114 |
| C(7)—C(6)—H(6) | 120 | H(141)—C(14)—H(142) | 99 |
| C(6)—C(7)—C(8) | 119·4 | H(141)—C(14)—H(143) | 109 |
| C(6)—C(7)—C(10) | 123·6 | H(142)—C(14)—H(143) | 112 |
| C(8)—C(7)—C(10) | 117·0 | N(3)—C(15)—Zn | 176·5 |
| C(7)—C(8)—C(9) | 119·6 | N(4)—C(16)—Zn | 177·6 |

map, with electron densities ranging from 0·8 to 0·5 e Å$^{-3}$. A good weighting scheme was $w = w_1 \times w_2$, where $w_1 = 1/\sigma_1^2$, $w_2 = 1/\sigma_2^2$, $\sigma_1 = a + b|F_o|$ and $\sigma_2 = c + d\sin\theta/\lambda$, with coefficients calculated by the program *PESOS* (Martínez-Ripoll & Cano, 1975) (Table 1). After two cycles of least-squares refinement with isotropic temperature factors for the H atoms, the final unweighted and weighted discrepancy indices were $R = 0{\cdot}036$ and $R_w = 0{\cdot}042$, where $R_w = (\Sigma w\Delta^2F/\Sigma w|F_o|^2)^{1/2}$. A final difference synthesis had no electron density greater than 0·25 e Å$^{-3}$. The final positional parameters are given in Table 2.*

**Discussion.** From infrared spectroscopy it was suggested (Cano Esquivel, 1975) that $Zn(CN)_2.C_{14}H_{12}N_2$ contained six-coordinated Zn. The present investigation was undertaken to establish unequivocally the structure of this compound.

Fig. 1 is a computer-generated perspective drawing of the molecule. Table 3 shows a list of bond angles and bond lengths together with their standard deviations. The Zn atom presents distorted tetrahedral coordination. The bidentate phenanthroline group is almost planar with an average deviation of 0·035 Å and a maximum deviation of 0·086 Å, and forms an angle of 85° with the plane defined by the Zn atom and the two cyanide ligands.

A glance at the bond lengths on both sides of a hypothetical line passing through Zn and the mid-point of the C(5)–C(6) bond suggests the existence of a noncrystallographic twofold axis. This has been tested in terms of a $\delta_p$ half-normal probability plot (Abrahams & Keve, 1971), calculated from a comparison of all intramolecular distances less than 3·5 Å on both sides of the above-mentioned line; this shows all the $\delta_p$ terms lying on a linear array with a slope of 1·22 (3) and a zero intercept at 0·14 (3). The value of the zero intercept cannot be interpreted as significant for the existence of any bias because the data are not independent. The same can apply to the slope, but in any case the linearity of the plot supports the above-mentioned symmetry of the molecule.

Most of the calculations were carried out using the XRAY 70 system (Stewart, Kundell & Baldwin, 1970). Thanks are due to the staff of the Centro de Proceso de Datos del Ministerio de Educación y Ciencia, Madrid, for the facilities provided on the 1108 Univac computer.

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* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32612 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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# Bis($\mu$-diethylimmoniocarbene)-hexacarbonyldiiron(−I)

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**Abstract.** $[(C_2H_5)_2NCFe(CO)_3]_2$, FW 448·04, orthorhombic, *Pbca*, $a = 10{\cdot}732\ (4)$, $b = 14{\cdot}922\ (4)$, $c = 25{\cdot}721\ (7)$ Å, $Z = 8$, $D_m = 1{\cdot}43$, $D_c = 1{\cdot}45$ g cm$^{-3}$, $\mu = 14{\cdot}8$ cm$^{-1}$ (Mo $K\alpha$ radiation), crystal size 0·95 × 0·35 × 0·60 mm. Diffractometer data, 3631 reflections measured, 2449 with $I \geq 3\sigma_I$, $R = 0{\cdot}044$. The molecule consists of two $Fe(CO)_3$ moieties bridged by two $>C{=}N(C_2H_5)_2$ units. The Fe–Fe distance is 2·482 (1) Å, and the coordination about each Fe atom is distorted octahedral.