Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Ni	0.44444 (3)	0.18004 (9)	0.20866 (4)	2.73 (1)
S(1)	0.51054 (6)	0.0752 (2)	0.17695 (8)	3.41 (3)
S(2)	0.42082 (6)	0.2947 (2)	0.09404 (9)	4.32 (4)
P	0.37384 (6)	0.2690 (2)	0.23326 (8)	2.73 (3)
C(1)	0.5109 (3)	0.1706 (9)	0.0837 (3)	5.3 (2)
C(2)	0.4580 (3)	0.2010 (10)	0.0364 (4)	7.4 (2)
C(3)	0.4547 (3)	0.2910 (10)	−0.0411 (4)	7.1 (2)
C(11)	0.3103 (2)	0.2016 (7)	0.1686 (3)	3.1 (1)
C(12)	0.3075 (3)	0.1256 (7)	0.0985 (4)	4.0 (2)
C(13)	0.2572 (3)	0.0795 (9)	0.0480 (4)	5.6 (2)
C(14)	0.2119 (3)	0.1110 (10)	0.0689 (4)	6.1 (2)
C(15)	0.2150 (3)	0.1840 (10)	0.1406 (4)	6.1 (2)
C(16)	0.2642 (2)	0.2293 (8)	0.1919 (4)	4.8 (2)
C(21)	0.3665 (2)	0.2343 (6)	0.3327 (3)	2.9 (1)
C(22)	0.3556 (2)	0.0901 (7)	0.3501 (4)	4.0 (1)
C(23)	0.3527 (3)	0.0530 (8)	0.4280 (4)	4.8 (2)
C(24)	0.3624 (3)	0.1620 (8)	0.4870 (4)	5.5 (2)
C(25)	0.3738 (3)	0.3073 (8)	0.4694 (4)	5.5 (2)
C(26)	0.3749 (3)	0.3435 (7)	0.3914 (3)	4.3 (2)
C(31)	0.3721 (2)	0.4703 (7)	0.2220 (3)	3.3 (1)
C(32)	0.3252 (3)	0.5459 (8)	0.1813 (4)	5.0 (2)
C(33)	0.3277 (3)	0.7040 (8)	0.1727 (4)	6.2 (2)
C(34)	0.3757 (4)	0.7757 (8)	0.2045 (4)	6.6 (2)
C(35)	0.4212 (3)	0.7003 (8)	0.2446 (4)	6.0 (2)
C(36)	0.4198 (3)	0.5440 (8)	0.2529 (4)	5.0 (2)

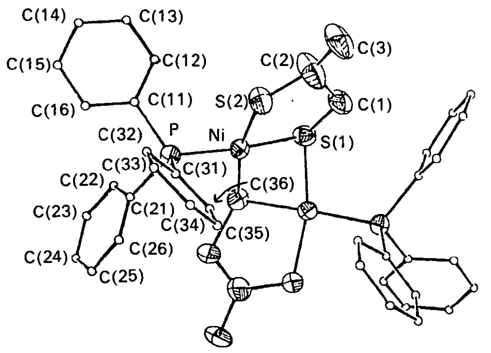

Fig. 1. The molecular structure of the title compound.

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

Ni—S(1)	2.201 (1)	P—C(11)	1.829 (4)
Ni—S(1′)	2.209 (2)	P—C(21)	1.827 (4)
Ni—S(2)	2.170 (1)	P—C(31)	1.828 (5)
Ni—P	2.193 (1)	C(1)—C(2)	1.424 (6)
S(1)—C(1)	1.841 (4)	C(2)—C(3)	1.559 (6)
S(2)—C(2)	1.812 (5)		
S(1)—Ni—S(1′)	77.61 (5)	C(11)—P—C(21)	100.9 (2)
S(1)—Ni—S(2)	90.44 (4)	C(11)—P—C(31)	106.0 (2)
S(1)—Ni—P	174.71 (4)	C(21)—P—C(31)	105.5 (2)
S(1′)—Ni—S(2)	164.93 (5)	S(1)—C(1)—C(2)	109.8 (4)
S(1′)—Ni—P	104.13 (4)	S(2)—C(2)—C(1)	111.9 (4)
S(2)—Ni—P	88.61 (4)	S(2)—C(2)—C(3)	109.7 (4)
Ni—S(1)—Ni′	81.11 (4)	C(1)—C(2)—C(3)	113.2 (5)
Ni—S(1)—C(1)	103.5 (2)	P—C(11)—C(12)	120.4 (3)
Ni—S(1′)—C(1′)	116.9 (2)	P—C(11)—C(16)	118.5 (4)
Ni—S(2)—C(2)	103.4 (2)	P—C(21)—C(22)	117.2 (3)
Ni—P—C(11)	116.1 (1)	P—C(21)—C(26)	122.4 (4)
Ni—P—C(21)	117.4 (1)	P—C(31)—C(32)	121.7 (3)
Ni—P—C(31)	109.8 (1)	P—C(31)—C(36)	116.4 (3)

Liu, 1992) and [$Ni_2(PPh_3)_2(SCH_2CH_2S)_2$] (Cao, Huang, Lei, Hong & Liu, 1992) have been reported.

This work was supported in part by a National Natural Science Foundation Grant and by the Provincial Science Fund of Fujian.

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Acta Cryst. (1992). C**48**, 1655–1657

Structure of the Condensation Product Formed by Pyrrolidine, Acetonitrile and Copper(I) Iodide

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(*Received* 28 *August* 1991; *accepted* 14 *January* 1992)

Abstract. Bis[1-aza-2-(1-pyrrolidinyl)propenyl]copper(I) iodide, [$Cu(C_6H_{12}N_2)_2$]I, $M_r = 414.80$, monoclinic, $A2/a$, $a = 16.149$ (1), $b = 7.205$ (5), $c = 15.358$ (5) Å, $\beta = 115.09$ (5)°, $V = 1618.4$ (2) Å^3, $Z = 4$, $D_x = 1.702$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 32.32$ cm^{-1}, $F(000) = 824$, $T = 294$ K, $R = 4.7\%$ for

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594 reflections $[I > 3\sigma(I)]$. The molecule crystallizes with copper(I) having twofold coordination to two $C_6H_{12}N_2$ molecules which were formed from the condensation of pyrrolidine and acetonitrile in the presence of CuI. Cu—N bond distances are 1.86 (1) Å. The N—Cu—N angle is 180° by symmetry. There is no interaction between Cu and I.

Experimental. Crystals were prepared by suspending copper(I) iodide (0.47 g, 2.5 mmol) in a solution of pyrrolidine (0.42 mL, 5.0 mmol) in acetonitrile (20 mL) under anhydrous, O_2-free conditions followed by stirring for 5 h. Filtration under N_2 and concentration yielded small needles overnight which were replaced by large cubic crystals after 2 d. Diffraction studies were carried out on the large cubic material (0.1 × 0.1 × 0.1 mm) using a Syntex *P*3 automated diffractometer with monochromated Mo $K\alpha$ radiation. Lattice parameters were determined from 15 reflections with $\theta = 19$–$26°$. Intensities were collected for $\theta \leq 50°$, h − 19 to 0, k − 8 to 0, l − 13 to 18, using θ–2θ scans, scan width of 1.2° below $K\alpha_1$ and 1.2° above $K\alpha_2$ and a variable scan rate (18–24° min^{-1}). Backgrounds were measured at each side of the scan for a combined time equal to the total scan time. Three standard reflections showed no significant variation. Lp corrections were applied. 1285 unique reflections were collected, 594 with $I \geq 3.0\sigma(I)$. The structure was solved using *MULTAN*80 (Main *et al.*, 1980) and refined by full-matrix least-squares procedures (*XRAY*; Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1978); H atoms were located from difference Fourier synthesis; refinement was on F with $w = 1/\sigma^2(F)$; scattering factors were obtained from Cromer & Mann (1968), anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974, Vol. IV). Final $R = 4.7$, $wR = 5.6\%$ for 82 parameters, $(\Delta/\sigma)_{max} = 0.023$, $(\Delta\rho)_{max} = 0.71$ e Å^{-3}. Positional parameters are reported in Table 1, bond lengths and angles in Table 2. A projection of the molecule is shown in Fig. 1.*

Table 1. *Positional parameters and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	U_{eq}
I1	0.2500	1.0748 (2)	0.0000	6.1
Cu1	0.5000	0.2500	0.2500	5.7
N1	0.441 (1)	0.372 (2)	0.133 (1)	5.6
N2	0.524 (1)	0.642 (1)	0.138 (1)	5.3
C1	0.452 (1)	0.532 (2)	0.096 (1)	5.6
C2	0.537 (1)	0.821 (2)	0.101 (1)	6.3
C3	0.621 (1)	0.901 (2)	0.181 (1)	9.7
C4	0.673 (1)	0.737 (3)	0.233 (1)	8.2
C5	0.603 (1)	0.595 (2)	0.229 (1)	6.5
C6	0.379 (1)	0.592 (2)	0.001 (1)	6.6

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

Cu1—N1	1.86 (1)	N1—Cu1—N1^i	180
N1—C1	1.32 (2)	Cu1—N1—C1	136 (1)
C1—N2	1.33 (2)	N1—C1—N2	124 (1)
C1—C6	1.50 (2)	N1—C1—C6	119 (1)
N2—C2	1.46 (2)	N2—C1—C6	117 (1)
N2—C5	1.47 (1)	C1—N2—C2	126 (1)
C2—C3	1.50 (2)	C1—N2—C5	123 (1)
C3—C4	1.47 (2)	C5—N2—C2	113 (1)
C4—C5	1.51 (3)	N2—C2—C3	104 (1)
		C2—C3—C4	104 (1)
		C3—C4—C5	106 (1)
		C4—C5—N2	102 (1)

Symmetry code: (i) $1 - x, \frac{1}{2} - y, \frac{1}{2} - z$.

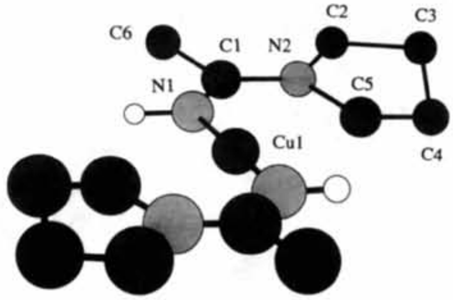
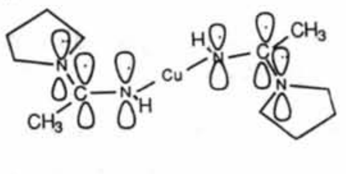


Fig. 1. Projection view of bis[1-aza-2-(1-pyrrolidinyl)propenyl]-copper(I).

Related literature. Twofold coordination of copper(I), Y—Cu—Y (Y = electron-pair donor ligand) has been observed with C, O, X, S and N donors [bicoordinate copper with triazine and benzimidazolyl derivatives (Brown & Dunitz, 1961; Schilstra, Birker, Verschoor & Reedijk, 1982; O'Conner, Janusonis & Corey, 1968; Hendriks, Birker, van Rijn, Verschoor & Reedijk, 1982, Birker, Hendriks & Reedijk, 1981; Agnus, Louis & Weiss, 1980)]. Commonly, a short non-bonded contact results in a Y—Cu—Y angle significantly less than 180°. In the title structure, as in bis(2,6-dimethylpyridine)copper(I) perchlorate (Engelhardt, Pakawatchai & White, 1985), placement of copper on an inversion center and the absence of non-bonded contacts results in a Y—Cu—Y angle of 180°. The equivalence of the C—N bonds at C1 and the angular totals at C1 and N2 suggest effective delocalization of the unshared pair on the N atom into the π system.



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* Lists of H-atom positions, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55042 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0580]

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Acta Cryst. (1992). C**48**, 1657–1658

Structure of Triphenyltin Glyoxalate *O*-Methyloxime

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(*Received* 9 *September* 1991; *accepted* 20 *January* 1992)

Abstract. *catena*-Poly[(triphenyltin)-μ-(methoxyiminoacetato-O:O')], $[Sn(C_3H_4NO_3)(C_6H_5)_3]_n$, $M_r = 452.08$, orthorhombic, $P2_12_12_1$, $a = 9.7484\,(7)$, $b = 10.3086\,(6)$, $c = 19.2656\,(8)$ Å, $V = 1936.0\,(2)$ Å^3, $Z = 4$, $D_x = 1.551$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0.71073$ Å, $\mu = 13.41$ cm^{-1}, $F(000) = 904$, $T = 298$ K, $R = 0.032$ for 2239 reflections [$I \geq 3\sigma(I)$]. The compound exists as a five-coordinate, *trans*-C_3SnO_2 trigonal bipyramidal carboxylate-bridged polymer.

Experimental. A stoichiometric amount of glyoxalic acid monohydrate was added to an ethanol solution of methoxyamine, prepared from equimolar amounts of sodium metal and methoxyamine hydrochloride in absolute ethanol. To this was added triphenyltin hydroxide, and the solution heated briefly. The solvent was then removed, and the solid obtained was purified by recrystallization from ethanol; m.p. 440–441 K. Analysis found: C 55.79, H 4.19, N 3.03%; calculated for $C_{21}H_{19}NO_3Sn$: C 55.79, H 4.23, N 3.09%.

A crystal measuring approximately 0.20 × 0.25 × 0.30 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. The cell dimensions were fixed from 25 reflections in the $17 \leq \theta \leq 19°$ thin shell. For data collection (ω–2θ-scan mode), the $2\theta_{\max}$ value was set at 54°, with the hkl ranges being h 0–12, k 0–13, l 0–24; 2404 reflections were measured, of which 2239 obeyed $I \geq 3\sigma(I)$. Three standard reflections (377, 6,0,12, 4,4,13) monitored hourly showed negligible intensity variation. Direct phase determination yielded the heavy atom and only one phenyl ring; the remaining non-H atoms were derived from successive difference Fourier syntheses. The non-H atoms were refined anisotropically; H atoms were generated (C—H = 0.95 Å, $B = 5$ Å^2) and included in the structure-factor calculations. The refinement was based on F. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). Computations were performed by using the *MolEN* structure determination system (Fair, 1990) on a DEC MicroVAX minicomputer. The final least-squares cycle was calculated with 235 variables; unit weights were used. The residuals were: $R = 0.032$ ($R = 0.038$ for all reflections) and $S = 1.96$; $\Delta/\sigma = 0.03$; $(\Delta\rho)_{\max} = 0.715$ e Å^{-3} about 1 Å from the Sn atom. Fractional coordinates are given in Table 1* and bond dimensions in Table 2. Fig. 1 shows the asymmetric unit.

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* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positional parameters, and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55060 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0552]

0108-2701/92/091657-02$06.00