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Stereochemistry of Bissalicylaldiminatometal(II) Compounds. Part III*. Crystal and Molecular Structure of One Form of Bis-[N-(2,6-dimethylphenyl)salicylaldiminato] cobalt(II)

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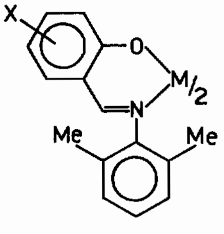
Abstract

Crystals of the β-form of the title compound are tetragonal, a = 10.630(5), c = 22.76(2) Å, space group $P4_1$. A three-dimensional X-ray analysis of the structure was made with counter data (1777 independent reflections). The structure was solved by Patterson and Fourier methods, and refined by block-diagonal least-squares to R = 0.059. The $[Co(N_2O_2)]$ polyhedron is tetrahedral, distorted necessarily by the chelate ligand constraints, but also by significant distortions in the chelate rings. The differences between the Co–O bond lengths [1.879(7) and 1.873(8) Å] and the Co–N bond lengths [2.035(8) and 2.003(8) Å] are greater than those in other salicylaldiminates because of the bulky nitrogen substituents here.

Introduction

The title compound (I for M = Co, X = H) was first reported by Yamada and coworkers [2], who used the steric crowding (B-strain) [11] in the molecule to produce a five-co-ordinate cobalt(II) species $[CoL_2py]$.

For this reason, we became interested in the whole series of compounds of type I, and have made an intensive study, especially of the nickel(II) compounds [3, 4]. As with mono-, ortho-substituted N-aryl salicylaldiminates [5], these nickel compounds do not readily adopt tetrahedral structures [11].



I

Hence, to help in the definition of any possible steric problems, which might favour planar over tetrahedral coordination, we have chosen one of two known [4] crystalline forms of the cobalt(II) compound I (X = H) for an X-ray analysis.

Experimental

Red-brown crystals were obtained from the slow reaction of 2,6-dimethylaniline with bissalicylaldehydato–cobalt(II) in ethanol at ~20 °C.

The data crystal had approximate dimensions 0.4 × 0.2 × 0.2 mm, and was mounted up the c axis. Sampling relied on microscopic examination of the apparently homogeneous solid.

Crystal Data

$C_{30}H_{28}N_2O_2Co$, M = 507.5, Tetragonal, a = 10.630(5), c = 22.76(2) Å. D_m (flotation) = 1.30 g cm^{-3}, Z = 4, D_c = 1.31 g cm^{-3}, U = 2572 Å^3, $F(000)$ = 1060, MoKα radiation, λ = 0.71069 Å, μ(MoKα) = 6.92 cm^{-1}. Space group $P4_1$ ($C_4{}^2$, No. 76) from systematic absences and Laue symmetry (photography).

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*Part II, see ref. [1].

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TABLE I. Atomic Positions and Thermal Vibrational Parameters with e.s.d.s in Parentheses.

	x/*a*	*y*/*b*	*z*/*c*
(a) Atomic positions ($\times 10^4$) of non-hydrogen atoms			
Co	3760(1)	327(1)	–5(1)
O(1)	2061(7)	–20(7)	–164(3)
O(2)	4585(8)	1092(7)	–638(3)
N(1)	3371(7)	1334(7)	733(3)
N(2)	5009(7)	–1066(7)	96(4)
C(1)	1089(8)	606(8)	26(5)
C(2)	–66(11)	478(10)	–255(5)
C(3)	–1152(9)	1099(12)	–70(6)
C(4)	–1074(11)	1868(14)	432(6)
C(5)	8(10)	1980(13)	718(5)
C(6)	1128(9)	1389(10)	534(4)
C(7)	2235(9)	1633(10)	855(4)
C(8)	4344(9)	1657(10)	1141(4)
C(9)	4405(11)	883(12)	1666(4)
C(10)	5390(13)	1172(14)	2044(5)
C(11)	6244(12)	2160(14)	1923(6)
C(12)	6135(11)	2823(12)	1441(6)
C(13)	5192(10)	2609(10)	1019(5)
C(14)	5153(14)	3331(12)	453(6)
C(15)	3463(13)	–171(14)	1778(6)
C(16)	5528(11)	659(10)	–922(5)
C(17)	5964(14)	1330(13)	–1425(6)
C(18)	6956(15)	908(14)	–1732(6)
C(19)	7578(14)	–181(13)	–1602(6)
C(20)	7195(14)	–843(12)	–1131(6)
C(21)	6171(11)	–458(10)	–793(4)
C(22)	5893(11)	–1263(9)	–280(5)
C(23)	4995(10)	–1916(10)	594(5)
C(24)	5889(11)	–1707(11)	1036(5)
C(25)	5853(13)	–2509(14)	1515(6)
C(26)	4960(15)	–3455(15)	1545(7)
C(27)	4108(13)	–3672(12)	1111(7)
C(28)	4102(12)	–2889(11)	613(6)
C(29)	6885(11)	–656(11)	997(6)
C(30)	3199(12)	–3129(12)	120(6)

	x/*a*	*y*/*b*	*z*/*c*	*B*
(b) Hydrogen atom calculated positions ($\times 10^3$) and vibration parameters				
H(2)	–10	–7	–60	5.64
H(3)	–193	99	–29	6.11
H(4)	–183	230	57	6.84
H(5)	3	253	106	6.99
H(7)	213	211	121	4.69
H(10)	545	67	240	8.01
H(11)	691	232	219	7.67
H(12)	673	349	137	6.72
H(17)	552	211	–153	7.34
H(18)	773	139	–207	9.27
H(19)	827	–48	–184	7.95
H(20)	763	–163	–103	7.55
H(25)	646	–238	183	8.28
H(26)	497	–400	188	8.86
H(27)	350	–436	113	7.24

(continued on facing page)

TABLE I. *(continued)*

	b_{11}	b_{22}	b_{33}	b_{23}	b_{13}	b_{12}
(c) Anisotropic thermal parameters ($\times 10^4$)*						
Co	80(1)	105(2)	15(0.2)	−11(1)	−2(1)	37(3)
O(1)	98(8)	133(9)	20(2)	−27(6)	−12(6)	39(13)
O(2)	145(10)	128(9)	19(2)	3(7)	19(7)	66(16)
N(1)	81(8)	84(8)	15(2)	2(6)	−4(6)	10(13)
N(2)	91(8)	90(9)	18(2)	−15(7)	3(6)	1(13)
C(1)	72(8)	75(9)	18(2)	7(8)	−7(8)	−24(14)
C(2)	103(12)	111(13)	24(3)	−2(10)	−19(9)	−41(21)
C(3)	62(10)	159(15)	33(3)	31(13)	−23(10)	2(19)
C(4)	93(13)	197(20)	27(3)	−27(13)	21(10)	57(25)
C(5)	78(11)	192(19)	22(3)	−21(12)	7(9)	21(23)
C(6)	71(10)	102(11)	18(2)	15(8)	4(7)	−20(17)
C(7)	87(11)	103(11)	14(2)	−6(8)	9(7)	30(18)
C(8)	72(10)	123(13)	16(2)	−29(8)	−8(7)	29(18)
C(9)	103(12)	156(15)	15(2)	−33(9)	0(8)	85(22)
C(10)	170(18)	190(19)	17(2)	−15(11)	−24(11)	141(30)
C(11)	124(15)	216(21)	24(3)	−58(13)	−36(11)	77(28)
C(12)	99(13)	144(16)	37(4)	−67(13)	−2(12)	29(23)
C(13)	78(11)	110(13)	26(3)	−28(10)	2(9)	1(18)
C(14)	202(21)	101(14)	33(4)	24(12)	−22(15)	−86(27)
C(15)	134(15)	180(18)	22(3)	31(12)	22(11)	−26(27)
C(16)	119(13)	94(11)	18(2)	−5(8)	26(9)	9(19)
C(17)	188(19)	149(16)	20(3)	16(11)	9(12)	82(29)
C(18)	233(23)	164(18)	20(3)	28(12)	14(13)	160(32)
C(19)	191(20)	146(17)	26(3)	46(12)	52(13)	66(28)
C(20)	196(19)	98(13)	31(3)	15(11)	64(14)	−1(27)
C(21)	130(13)	96(11)	15(2)	10(8)	29(9)	34(20)
C(22)	126(13)	79(11)	23(3)	−23(8)	52(10)	−45(18)
C(23)	92(11)	83(11)	26(3)	−12(9)	−18(9)	57(18)
C(24)	121(14)	116(13)	22(3)	−6(10)	−1(10)	56(22)
C(25)	128(15)	177(19)	28(3)	3(13)	−19(12)	31(27)
C(26)	195(21)	175(20)	32(4)	46(14)	4(15)	89(33)
C(27)	147(17)	109(14)	44(4)	37(13)	13(14)	−24(25)
C(28)	135(14)	92(12)	28(3)	−15(10)	4(11)	23(22)
C(29)	92(12)	116(14)	30(3)	−24(11)	−29(10)	0(20)
C(30)	127(14)	147(16)	35(4)	−44(13)	−38(12)	−33(23)

*The expression for the temperature factor is $\exp[-(h^2b_{11} + k^2b_{22} + l^2b_{33} + klb_{23} + hlb_{13} + hkb_{12})]$.

Data were collected on a Stöe Stadi-2 diffractometer with stationary counter and moving crystal, for layers $hk0 \rightarrow hk27$ ($2\theta_{max} = 50°$), using graphite-monochromated MoKα radiation. Angular step-scan ranges for the reflections were systematically varied to allow for variations in peak-width. A counting time of 1.0 s was used for each 0.01° increment of scan. Background counts were accumulated for 30 s at each extremity of the scan.

Reflections with intensity $I < 3\sigma(I)$ were ignored, as were those with background differences $\Delta > 3\sigma_B$. Corrections were applied for Lorentz and polarisation factors, but not for absorption or extinction. The final data set consisted of 1777 unique reflections.

The structure was solved by conventional Patterson and Fourier techniques; and refined by block-diagonal least-squares, using anisotropic temperature factors for all non-hydrogen atoms.

Hydrogen atoms, except for those attached to methyl carbons, were observed in a difference Fourier; and they were included (C–H = 0.98 Å), but not refined. Fixed isotropic temperature factors were assigned at values 2.0 greater than the latest refined isotropic values for the atoms to which they are attached.

Anomalous dispersion corrections [6] (both $\Delta f'$ and $\Delta f''$) were applied to all non-hydrogen atom scattering factors [7]. A non-unit weighting scheme was used in which $w = 1$ if $|F_o| < 25$ and $\sqrt{w} = 25/|F_o|$ if $|F_o| > 25$.

Final refinement, when all shifts were less than 0.1σ, reduced R to 0.059. The final difference Fourier showed no significant features.

Scattering factors were taken from International Tables [7]. Programmes used were from the Sheffield X-ray system on the Sheffield University ICL 1907 computer.

Fractional co-ordinates for atomic positions, and the thermal parameters, with e.s.d.s, are given in Table I. Hydrogen atoms take the same number as the carbon to which they are attached.

A list of observed and calculated structure factors has been deposited with the Editor.

Results and Discussion

The observed molecular structure is represented in Fig. 1, together with the atom numbering scheme. Details of the geometry of the molecule are listed in Table II; and equations to some pertinent least-squares planes, with atom deviations and interplanar angles, are listed in Table III.

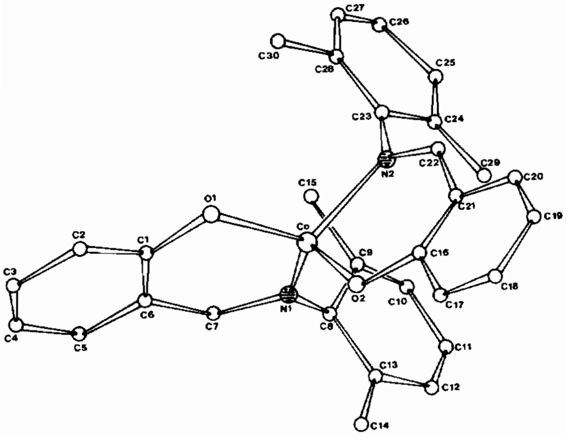

Fig. 1. Molecular structure of Bis-[N-(2,6-dimethylphenyl)-salicylaldiminato]cobalt(II).

The cobalt(II) polyhedron has the expected tetrahedral arrangement of the N_2O_2 donor set.

We were surprised to find only a few structures of bissalicylaldiminato–cobalt(II) species with which to compare this one. Of the three known [8–10], two [8, 9] are but poorly refined, and the third [10] has the complication of a 1,3,5-trinitrobenzene in co-crystallisation. The geometries of the $[CoN_2O_2]$ tetrahedra, however, are compared with that of the present structure in Table IV.

Here the bulkiness of the 2,6-dimethylphenyl substituent is reflected in greater than normal differences between the Co–N and Co–O bond lengths. Such differences are greater even than those in the t-Bu substituted molecule. By contrast, the latter shows a much greater N–Co–N angle of 125.3°, against the present 115.4°, reflecting the different shapes of the bulky N-substituents.

The interatom bond lengths and angles for the ligands (Table II) are generally within expected limits, and the phenyl nitrogen substituents are perpendicular to the rest of the ligand molecule – a conformation which is necessitated by the methyl substituents. This is emphasised by a pair of close intramolecular contacts C(7)–C(15) = 3.13 Å and C(22)–C(29) = 3.16 Å, both of which clearly prevent any further rotation of the 2,6-dimethylphenyl moieties.

The least-squares planes calculations (Table III) do, however, show quite distinct distortions

TABLE II. Interatomic Distances (Å) and Angles (°) with e.s.d.s. in Parentheses.

Bond distances (Å)

Co–O(1)	1.879(7)	C(9)–C(15)	1.52(2)
Co–O(2)	1.873(8)	C(10)–C(11)	1.42(2)
Co–N(1)	2.035(8)	C(11)–C(12)	1.31(2)
Co–N(2)	2.003(8)	C(12)–C(13)	1.41(2)
O(1)–C(1)	1.30(1)	C(13)–C(14)	1.50(2)
O(2)–C(16)	1.28(1)	C(16)–C(17)	1.43(2)
N(1)–C(7)	1.28(1)	C(16)–C(21)	1.40(2)
N(1)–C(8)	1.43(1)	C(17)–C(18)	1.34(2)
N(2)–C(22)	1.29(1)	C(18)–C(19)	1.37(2)
N(2)–C(23)	1.45(1)	C(19)–C(20)	1.35(2)
C(1)–C(2)	1.39(2)	C(20)–C(21)	1.39(2)
C(1)–C(6)	1.43(1)	C(21)–C(22)	1.48(2)
C(2)–C(3)	1.40(2)	C(23)–C(24)	1.40(2)
C(3)–C(4)	1.41(2)	C(23)–C(28)	1.40(2)
C(4)–C(5)	1.33(2)	C(24)–C(25)	1.39(2)
C(5)–C(6)	1.41(2)	C(24)–C(29)	1.54(2)
C(6)–C(7)	1.41(1)	C(25)–C(26)	1.38(2)
C(8)–C(9)	1.45(2)	C(26)–C(27)	1.36(2)
C(8)–C(13)	1.38(2)	C(27)–C(28)	1.41(2)
C(9)–C(10)	1.39(2)	C(28)–C(30)	1.50(2)

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TABLE II. *(continued)*

Angles (°)

O(1)–Co–O(2)	112.8(3)	C(10)–C(9)–C(15)	124(1)
O(1)–Co–N(1)	93.8(3)	C(9)–C(10)–C(11)	122(1)
O(1)–Co–N(2)	121.0(3)	C(10)–C(11)–C(12)	120(1)
O(2)–Co–N(1)	120.2(3)	C(11)–C(12)–C(13)	123(1)
O(2)–Co–N(2)	95.6(3)	C(8)–C(13)–C(12)	117(1)
N(1)–Co–N(2)	115.4(3)	C(8)–C(13)–C(14)	122(1)
Co–O(1)–C(1)	126.8(6)	C(12)–C(13)–C(14)	122(1)
Co–O(2)–C(16)	126.9(7)	O(2)–C(16)–C(17)	119(1)
Co–N(1)–C(7)	120.0(7)	O(2)–C(16)–C(21)	126(1)
Co–N(1)–C(8)	121.0(6)	C(17)–C(16)–C(21)	116(1)
C(7)–N(1)–C(8)	119(1)	C(16)–C(17)–C(18)	120(1)
Co–N(2)–C(22)	121.9(7)	C(17)–C(18)–C(19)	124(1)
Co–N(2)–C(23)	122.9(6)	C(18)–C(19)–C(20)	118(1)
C(22)–N(2)–C(23)	115(1)	C(19)–C(20)–C(21)	122(1)
O(1)–C(1)–C(2)	120(1)	C(16)–C(21)–C(20)	121(1)
O(1)–C(1)–C(6)	123(1)	C(16)–C(21)–C(22)	124(1)
C(2)–C(1)–C(6)	117(1)	C(20)–C(21)–C(22)	115(1)
C(1)–C(2)–C(3)	123(1)	N(2)–C(22)–C(21)	125(1)
C(2)–C(3)–C(4)	118(1)	N(2)–C(23)–C(24)	117(1)
C(3)–C(4)–C(5)	120(1)	N(2)–C(23)–C(28)	119(1)
C(4)–C(5)–C(6)	123(1)	C(24)–C(23)–C(28)	124(1)
C(1)–C(6)–C(5)	119(1)	C(23)–C(24)–C(25)	117(1)
C(1)–C(6)–C(7)	124(1)	C(23)–C(24)–C(29)	123(1)
C(5)–C(6)–C(7)	118(1)	C(25)–C(24)–C(29)	121(1)
C(6)–C(7)–N(1)	129(1)	C(24)–C(25)–C(26)	120(1)
N(1)–C(8)–C(9)	116(1)	C(25)–C(26)–C(27)	123(1)
N(1)–C(8)–C(13)	121(1)	C(26)–C(27)–C(28)	119(1)
C(9)–C(8)–C(13)	123(1)	C(23)–C(28)–C(27)	117(1)
C(8)–C(9)–C(10)	115(1)	C(23)–C(28)–C(30)	122(1)
C(8)–C(9)–C(15)	122(1)	C(27)–C(28)–C(30)	120(1)

Non-bonding distances within the molecule (Å)

O(1)–O(2)	3.13(1)	O(2)–N(1)	3.39(1)
O(1)–N(1)	2.86(1)	O(2)–N(2)	2.87(1)
O(1)–N(2)	3.38(1)	N(1)–N(2)	3.41(1)

TABLE III. Some Least-Squares Planes.[a]

	l	*m*	*n*	*d*
Ligand A:				
Plane (1): C(1)–C(6)	0.2270	0.7859	–0.5752	0.7281
	[O(1) –0.033(7), N(1) 0.239(8), C(1) 0.006(9), C(2) –0.011(11), C(3) 0.004(13), C(4) 0.007(14), C(5) –0.012(13), C(6) 0.005(10), C(7) 0.056(10)]			
Plane (2): C(1)–C(7), N(1), O(1)	0.1998	0.7759	–0.5984	0.7042
	[C(1) –0.01(1), C(2) 0.02(1), C(3) 0.05(1), C(4) 0.02(1), C(5) –0.05(1), C(6) –0.05(1), C(7) –0.05(1), N(1) 0.113(8), O(1) –0.060(7), Co 0.371(2), C(8) 0.03(1)]			
Plane (3): C(8)–C(13)	0.5931	–0.6480	–0.4779	0.3605
	[C(8) –0.004(10), C(9) –0.003(11), C(10) 0.007(14), C(11) –0.027(14), C(12) –0.005(13), C(13) 0.008(11), C(14) 0.101(14), C(15) 0.007(14), N(1) 0.049(8)]			

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TABLE III. *(continued)*

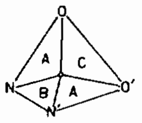
Ligand B:				
Plane (4): C(16)–C(21)	−0.6229	−0.5167	−0.5874	−2.7819
	[C(16) −0.008(11), C(17) 0.008(14), C(18) −0.007(14), C(19) 0.006(14), C(20) −0.007(14), C(21) 0.008(11), C(22) −0.052(11), N(2) −0.077(8), O(2) −0.001(8)]			
Plane (5): C(16)–C(22), N(2), O(2)	−0.6251	−0.5266	−0.5761	−2.8348
	[C(16) 0.00(1), C(17) −0.00(1), C(18) −0.02(1), C(19) 0.00(1), C(20) 0.01(1), C(21) 0.03(1), C(22) −0.01(1), N(2) −0.022(8), O(2) 0.014(8), Co 0.160(2), C(23) −0.19(1)]			
Plane (6): C(23)–C(28)	0.6395	−0.6182	−0.4571	4.0442
	[C(23) −0.008(11), C(24) 0.002(12), C(25) 0.007(14), C(26) −0.010(16), C(27) 0.005(14), C(28) 0.004(12), C(29) 0.030(12), C(30) 0.061(13), N(2) −0.038(8)]			
Cobalt Tetrahedron:				
Plane (7): Co, N(1), O(1)	−0.0617	0.8351	−0.5467	0.0507
Plane (8): Co, N(2), O(2)	−0.5220	−0.5667	−0.6375	−2.2760
Plane (9): Co, N(1), N(2)	0.7429	0.6322	−0.2199	3.1918
Plane (10): Co, O(1), O(2)	0.2546	−0.9004	−0.3527	0.7082
Plane (11): Co, N(2), O(1)	0.1917	0.0202	−0.9812	0.7850
Plane (12): Co, N(1), O(2)	−0.8826	0.2672	−0.3868	−3.4297

Angles between planes (°)

1–2	2.1	7–8	95.3	8–12	56.2
2–3	95.6	7–9	53.0	9–10	107.6
2–7	15.7	7–10	125.1	9–11	68.2
2–5	100.9	7–11	57.2	9–12	113.7
4–5	0.9	7–12	60.7	10–11	67.9
5–6	79.1	8–9	127.3	10–12	109.2
5–8	7.2	8–10	53.0	11–12	77.5
3–6	3.4	8–11	59.1		

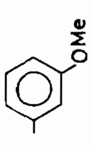
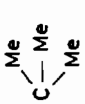
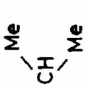
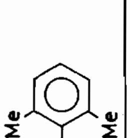
[a] The equations to the planes are in the form $lX + mY + nZ - d = 0$ (where X, Y, and Z are co-ordinates in Å referred to *a*, *b* and *c*). The deviations (Å) from these planes of various atoms are given, together with e.s.d.s, in square brackets.

of the ligand frameworks. Ligand A has a significant twist in the chelate ring: the atoms have much larger deviations from the least-squares plane 2 than do those of ligand B [plane 5]. This is especially marked for N(1), which is 0.11 Å out-of-plane and Co (0.37 Å). Comparable values for plane 5 are N(2) 0.02 Å and Co 0.160 Å. On the other hand, ligand B has a different distortion. Here the N-substituent is showing strain, with C(23) 0.19 Å out of the plane 5.



Scheme 1.

TABLE IV. A. Comparison of the Known Bissalicylaldimatocobalt(II) Tetrahedra.

Reference	N-substituent	Co–O	Co–N	O–Co–N	O(1)–Co–O(2)	N(1)–Co–N(2)	O(2)–Co–N(1) O(1)–Co–N(2)	Angle between planes A[a]	Angle between planes B and C[a]
8		1.88 Å 1.91	1.95 Å 1.97	96° 96	120°	122°	113° 111	84°	78°
10		1.902(4) 1.901(5)	1.994(4) 1.988(4)	97.8(2) 98.0(2)	116.9(2)	125.3(2)	109.3(2) 110.9(2)	81.6	82.2
9		1.912	1.975	95.9	118.1	119.9	114.4	86.7	78.5
		1.879(7) 1.873(8)	2.035(8) 2.003(8)	93.8(3) 95.6(3)	112.8(3)	115.4(3)	121.0(3) 120.2(3)	85	73

[a] Refer to Scheme 1.

Both of these distortions have the effect of bringing the 2,6-dimethylphenyl moieties closer together and they are parallel one to another (angle between planes only 3.4°). This latter feature is evident in both of the figures. However, we do not distinguish between the possibilities of *either* an attractive force between the two phenyl moieties, *or* intermolecular interactions in the crystal (crystal-packing forces').

The arrangement of the molecules about the tetrad axis is shown in Fig. 2. There are no intermolecular C···C contacts <3.5 Å.

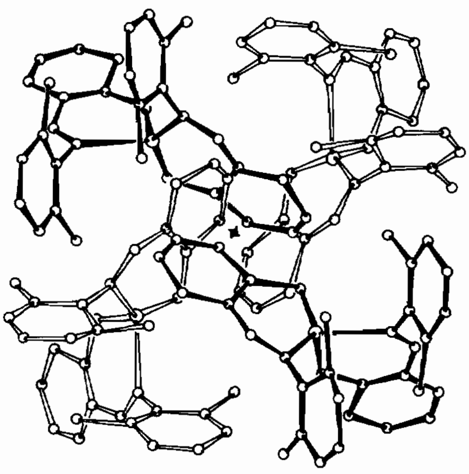


Fig. 2. Projection down the *c*-axis (along the tetrad axis).

We did also attempt the structure of the second crystalline form of this molecule. However, the data set was obtained at a time of difficulties with the diffractometer; there were clear problems with this data; and we have not proceeded.

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