

methyl) suggests, at least, a near isotropy of crystal forces around the center of the molecule, with the sum of intermolecular forces being of a magnitude estimated to be about 2 kcal mol⁻¹ (Bürgi & Dunitz, 1971), sufficient to compensate for the preference of the non-planar conformation over the planar one.

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A Cobalt (III) Complex with 1-(2-Thiazolylazo)-2-naphthol, [Co^{III}(C₁₃H₈N₃OS)₂](Cl, I) · 2CHCl₃

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Abstract. [Co^{III}(C₁₃H₈N₃OS)₂](Cl_{0.7}I_{0.3}) · 2CHCl₃, monoclinic, *P*2₁/*a*, *a* = 16.487 (4), *b* = 21.750 (3), *c* = 10.045 (3) Å, β = 108.89 (3)°, *U* = 3408.1 Å³; *Z* = 4, *D_m* = 1.69, *D_c* = 1.69 g cm⁻³; μ = 9.9 cm⁻¹ (for Mo *K*α); *R* = 0.087 for 1125 reflexions collected by diffractometry. The cobalt atom is surrounded octahedrally by two terdentate ligands in the *mer* configuration. The 1-(2-thiazolylazo)-2-naphtholato group is coordinated to the cobalt atom through the phenolic oxygen atom, the azo nitrogen atom adjacent to the naphthol ring and the thiazole nitrogen atom, forming two five-membered chelate rings.

Introduction. As a part of a series of studies on the structures of metal chelates with 1-(2-thiazolylazo)-2-naphthol ('Htan'), the X-ray determination of the title compound was undertaken. A powder sample of [Co^{III}(tan)₂](Cl, I) was kindly supplied by Dr A. Kawase. Small green crystals of the complex were obtained from a chloroform solution by slow evaporation. The crystal used for the X-ray study had dimensions of 0.06 × 0.06 × 0.25 mm. Both the determination of cell constants and the collection of intensity data were carried out on a Rigaku automated four-circle diffractometer using Mo *K*α radiation monochromated with a graphite plate. The ω–2θ scan technique was

employed at a scan rate of 4° min⁻¹ in ω. The scan ranges were determined according to the formula 1.5° + 0.6° × tan θ. The background countings were taken at both sides of each peak for 10 s. Intensities were converted to the structure amplitudes in the usual manner, but an absorption correction was not applied. Thus 1125 independent reflexions with |*F*| > 3σ(|*F*|) were obtained.

The structure was solved by the heavy-atom method. Initially it was assumed that the crystal contained only chloride ions. On this supposition the structure was refined by the least-squares technique to give

Table 1. *Positional* (× 10⁴) and *thermal parameters*
(a) Fractional atomic coordinates and isotropic temperature factors for the non-hydrogen atoms

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i>
O	1002 (14)	2670 (10)	1924 (22)	4.6 (5)
Co	639 (3)	2099 (2)	407 (5)	
Cl, I	3499 (4)	4797 (3)	4552 (7)	
S	1409 (7)	679 (5)	–1845 (11)	
S'	–1011 (7)	1102 (5)	2167 (11)	
Cl(1)	1354 (10)	5436 (7)	569 (15)	
Cl(2)	1490 (10)	4117 (7)	624 (16)	
Cl(3)	869 (11)	4748 (8)	2551 (22)	
Cl(1')	2234 (14)	3917 (8)	7013 (21)	
Cl(2')	1688 (21)	5125 (15)	6759 (28)	
Cl(3')	3214 (21)	4825 (12)	8502 (28)	

Table 1 (cont.)

N(1)	1816 (15)	1888 (12)	1052 (27)	4.1 (6)
N(2)	2071 (15)	1444 (12)	373 (28)	4.3 (6)
N(3)	576 (15)	1459 (13)	-988 (27)	4.4 (6)
C(1)	2262 (21)	2233 (16)	2084 (33)	4.5 (8)
C(2)	1791 (21)	2675 (15)	2566 (34)	4.5 (8)
C(3)	2183 (21)	3059 (15)	3650 (34)	4.4 (8)
C(4)	3038 (22)	3031 (17)	4295 (37)	5.7 (9)
C(5)	3600 (22)	2603 (16)	3959 (36)	4.9 (8)
C(6)	4424 (23)	2575 (14)	4502 (39)	6.1 (9)
C(7)	4925 (22)	2169 (18)	4172 (36)	6.0 (9)
C(8)	4627 (22)	1784 (16)	3108 (35)	5.0 (8)
C(9)	3774 (22)	1753 (17)	2488 (35)	5.1 (9)
C(10)	3210 (22)	2169 (17)	2823 (35)	5.1 (8)
C(11)	1358 (22)	1232 (16)	-683 (34)	4.6 (8)
C(12)	369 (22)	779 (16)	-2720 (36)	4.8 (8)
C(13)	-14 (23)	1202 (16)	-2130 (38)	5.7 (9)
O'	759 (14)	2719 (11)	-820 (22)	4.9 (5)
N(1')	-513 (15)	2281 (12)	-246 (28)	4.8 (7)
N(2')	-1402 (15)	1971 (13)	232 (28)	4.8 (7)
N(3')	255 (15)	1555 (13)	1545 (29)	5.3 (7)
C(1')	-724 (22)	2707 (15)	-1275 (33)	4.3 (8)
C(2')	-15 (22)	2948 (18)	-1529 (35)	5.1 (8)
C(3')	-79 (22)	3433 (16)	-2576 (36)	4.9 (9)
C(4')	-885 (23)	3624 (18)	-3357 (38)	6.2 (9)
C(5')	-1665 (22)	3343 (16)	-3253 (37)	5.0 (8)
C(6')	-2466 (22)	3514 (14)	-4130 (38)	5.9 (9)
C(7')	-3225 (22)	3275 (14)	-4035 (37)	5.5 (9)
C(8')	-3175 (22)	2825 (19)	-3047 (38)	6.5 (9)
C(9')	-2368 (22)	2621 (17)	-2073 (37)	5.3 (9)
C(10')	-1599 (22)	2876 (17)	-2213 (35)	5.0 (8)
C(11')	-588 (22)	1575 (16)	1217 (34)	4.2 (8)
C(12')	7 (22)	830 (16)	3065 (35)	4.6 (8)
C(13')	582 (22)	1096 (16)	2586 (36)	4.8 (8)
CH	1626 (29)	4748 (21)	1687 (43)	8.6 (11)
CH'	2427 (29)	4633 (21)	6752 (44)	8.8 (12)

(b) Positional parameters for the hydrogen atoms

	x	y	z
H(3)	1796	3414	3972
H(4)	3326	3372	5227
H(6)	4734	2911	5336
H(7)	5617	2146	4858
H(8)	5081	1468	2788
H(9)	3496	1392	1716
H(12)	7	526	-3706
H(13)	-706	1327	-2497
H(3')	516	3615	-2768
H(4')	-970	4003	-4136
H(6')	-2506	3861	-4930
H(7')	-3867	3419	-4786
H(8')	-3772	2603	-3028
H(9')	-2357	2256	-1275
H(12')	178	488	3951
H(13')	1283	972	2869

(c) Anisotropic thermal parameters ($\times 10^4$)

The anisotropic thermal parameters are in the form $\exp \{-(h^2 B_{11} + k^2 B_{22} + l^2 B_{33} + hk B_{12} + hl B_{13} + kl B_{23})\}$.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Co	37 (2)	18 (1)	109 (7)	-1 (3)	32 (6)	-21 (6)
Cl, I	15 (3)	8 (2)	24 (9)	1 (4)	-21 (8)	-3 (7)
S	66 (6)	27 (3)	120 (15)	9 (7)	19 (16)	-51 (12)
S'	53 (6)	25 (3)	141 (15)	-15 (7)	50 (15)	14 (12)
Cl(1)	144 (11)	43 (5)	264 (26)	-18 (12)	39 (27)	27 (17)
Cl(2)	145 (12)	50 (5)	304 (30)	6 (13)	-53 (30)	-60 (20)
Cl(3)	151 (14)	66 (6)	606 (50)	-36 (16)	346 (44)	-90 (30)
Cl(1')	257 (19)	44 (5)	569 (49)	-51 (16)	502 (54)	-17 (26)
Cl(2')	484 (41)	181 (17)	640 (66)	458 (46)	766 (91)	406 (55)
Cl(3')	506 (43)	99 (11)	646 (69)	-184 (36)	807 (96)	-169 (45)

an R value of 0.11. The temperature factor for the chloride ions converged at a very small value, and the Fourier and difference peaks for the ion calculated at this stage were unusually high. These facts undoubtedly indicate that part of the chloride is replaced by iodide ions, inclusion of which was previously detected by the X-ray fluorescent spectra. Therefore the structure factor calculations were performed varying the Cl-to-I ratio, and the minimum R value was obtained when the Cl-to-I ratio was 7 to 3.

At the final stage of the refinement, the hydrogen atoms for the complex cation were included in the structure factor calculations with fixed thermal par-

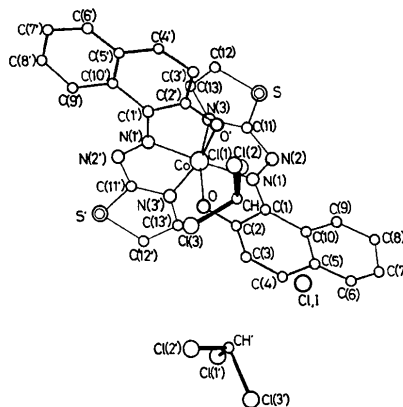


Fig. 1. Molecular geometry and numbering of atoms.

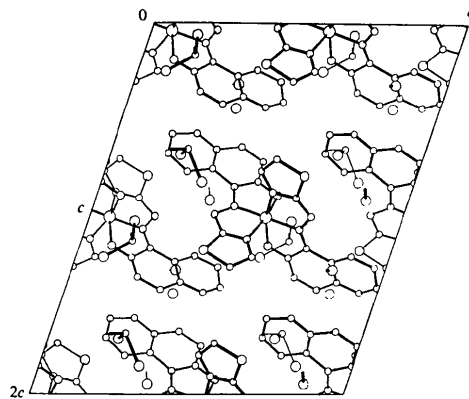
Fig. 2. The crystal structure viewed down the b axis.

Table 1 (cont.)

Table 2. Bond lengths and angles

(a) Bond lengths (Å)

	Unprimed atoms	Primed atoms
Co—O	1.91 (2)	1.88 (2)
Co—N(1)	1.90 (3)	1.84 (3)
Co—N(3)	1.96 (3)	1.90 (3)
S—C(11)	1.70 (4)	1.69 (4)
S—C(12)	1.67 (4)	1.73 (4)
O—C(2)	1.25 (4)	1.34 (4)
N(1)—N(2)	1.32 (4)	1.31 (4)
N(1)—C(1)	1.29 (4)	1.35 (4)
N(2)—C(11)	1.38 (4)	1.35 (4)
N(3)—C(11)	1.32 (4)	1.32 (4)
N(3)—C(13)	1.36 (5)	1.41 (5)
C(1)—C(2)	1.43 (5)	1.38 (5)
C(1)—C(10)	1.51 (5)	1.50 (5)
C(2)—C(3)	1.35 (5)	1.46 (5)
C(3)—C(4)	1.35 (5)	1.38 (5)
C(4)—C(5)	1.43 (5)	1.45 (5)
C(5)—C(6)	1.29 (5)	1.38 (5)
C(5)—C(10)	1.47 (5)	1.44 (5)
C(6)—C(7)	1.32 (5)	1.39 (5)
C(7)—C(8)	1.33 (5)	1.38 (5)
C(8)—C(9)	1.35 (5)	1.44 (5)
C(9)—C(10)	1.41 (5)	1.43 (5)
C(12)—C(13)	1.35 (5)	1.33 (5)
CH—Cl(1)	1.84 (5)	1.62 (5)
CH—Cl(2)	1.70 (5)	1.63 (6)
CH—Cl(3)	1.74 (5)	1.87 (6)

(b) Bond angles (°)

	Unprimed atoms	Primed atoms
O—Co—N(1)	82 (1)	86 (1)
N(1)—Co—N(3)	83 (1)	81 (1)
C(11)—S—C(12)	90 (2)	90 (2)
Co—O—C(2)	115 (2)	110 (2)
Co—N(1)—N(2)	113 (2)	119 (2)
Co—N(1)—C(1)	117 (2)	114 (2)
N(2)—N(1)—C(1)	130 (3)	127 (3)
N(1)—N(2)—C(11)	108 (3)	109 (3)
Co—N(3)—C(11)	106 (2)	111 (2)
Co—N(3)—C(13)	139 (2)	140 (2)
C(11)—N(3)—C(13)	115 (3)	109 (3)
N(1)—C(1)—C(2)	116 (3)	113 (3)
N(1)—C(1)—C(10)	125 (3)	128 (3)
C(2)—C(1)—C(10)	119 (3)	119 (3)
O—C(2)—C(1)	113 (3)	118 (3)
O—C(2)—C(3)	125 (3)	119 (3)
C(1)—C(2)—C(3)	122 (3)	123 (3)
C(2)—C(3)—C(4)	121 (3)	118 (3)
C(3)—C(4)—C(5)	125 (3)	123 (3)
C(4)—C(5)—C(6)	128 (4)	122 (3)
C(4)—C(5)—C(10)	117 (3)	119 (3)
C(6)—C(5)—C(10)	115 (3)	119 (3)
C(5)—C(6)—C(7)	127 (4)	124 (4)
C(6)—C(7)—C(8)	121 (4)	118 (3)
C(7)—C(8)—C(9)	117 (4)	123 (3)
C(8)—C(9)—C(10)	122 (3)	118 (3)
C(1)—C(10)—C(5)	117 (3)	118 (3)
C(1)—C(10)—C(9)	127 (3)	123 (3)
C(5)—C(10)—C(9)	116 (3)	119 (3)
S—C(11)—N(2)	124 (3)	125 (3)
S—C(11)—N(3)	111 (2)	116 (3)
N(2)—C(11)—N(3)	125 (3)	120 (3)
S—C(12)—C(13)	113 (3)	110 (3)
N(3)—C(13)—C(12)	110 (3)	115 (3)
Cl(1)—CH—Cl(2)	108 (2)	117 (3)
Cl(1)—CH—Cl(3)	103 (2)	101 (3)
Cl(2)—CH—Cl(3)	111 (3)	97 (3)

ameters (5.0 \AA^2) and fixed positional parameters deduced from the geometry of the molecule. The R value became 0.087 for 1125 observed reflexions.* Unit weight was given to all reflexions. The atomic scattering factors for the non-hydrogen atoms were taken from *International Tables for X-ray Crystallography* (1962), and for hydrogen from Stewart, Davidson & Simpson (1965). Table 1 gives the final atomic parameters of all the atoms with their e.s.d.'s.

Discussion. The asymmetric unit consists of a positively charged complex cation $[\text{Co}^{\text{III}}(\text{tan})_2]^+$, a negatively charged halogen anion $[\text{Cl}^- (70\%) \text{ and } \text{I}^- (30\%)]$, and two chloroform molecules as a crystal solvent. The structure viewed down the b axis is illustrated in Fig. 1 together with the atom numbering. The $[\text{Co}^{\text{III}}(\text{tan})_2]^+$ complex has a twofold axis approximately through Co and the midpoint of O and O'. The cobalt atom is surrounded octahedrally by two ligand anions in the *mer* configuration. The 1-(2-thiazolylazo)-2-naphtholato group (tan) acts as a terdentate ligand; the thiazole nitrogen atom, the azo nitrogen atom adjacent to the naphthol ring and the phenolic oxygen atom are involved in chelate ring formation. The structural features are very similar to those observed in $[\text{Co}^{\text{III}}(\text{tan})_2]\text{ClO}_4$ (Kurahashi, 1974).

The bond lengths and angles are listed in Table 2, with their e.s.d.'s. The geometries of the two moieties agree well within the limit of three times their corresponding e.s.d.'s. The bond angles around the metal, O—Co—N(1), O'—Co—N(1'), N(1)—Co—N(3) and N(1')—Co—N(3') are compressed to 82, 83, 86 and 81°, respectively. This is the characteristic feature commonly observed in the metal-tan chelates. The mean planes through Co and the unprimed tan and through Co and the primed tan are both roughly planar within the deviations of ± 0.13 and $\pm 0.14 \text{ \AA}$ respectively, and they make an angle of 89°.

A projection of the structure along the b axis is presented in Fig. 2. There are no unusually short intermolecular contacts in the structure. The chemical implications are discussed elsewhere.

The author wishes to express his sincere thanks to Dr A. Kawase for supplying the specimen and for his valuable discussions. He is also grateful to Dr K. Ohno for his kind help in the X-ray fluorescent spectra measurement. Calculations were performed on the HITAC 5020F computer at the Computer Centre of the National Aerospace Laboratory and on the NEAC 3100 computer at this Institute. Some UNICS programs (Sakurai, 1965) were used. The figures were

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31672 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

drawn with the *DEAM-4* program written by Takenaka (1972).

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Structure and Absolute Configuration of the Sesquiterpene Lemnacarnol

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Abstract. Lemnacarnol, $C_{15}H_{24}O_3$, tetragonal, $a=b=9.208$ (1), $c=16.918$ (3) Å, $M=252.3$, $Z=4$, $D_m=1.169$ g cm⁻³, $\mu(\text{Cu } K\alpha)=6.4$ cm⁻¹. The compound was extracted from the scft coral *Lemnalia Carnosa* and its structure and absolute configuration have been determined by single-crystal X-ray diffraction.

Introduction. Chemical and spectroscopic investigations of lemnacarnol have been summarized by Tursch, Colin, Dalozé, Losman & Karlsson (1975). Crystals were grown from ether. Weissenberg photographs indicated a tetragonal cell with systematic extinctions characteristic for space group $P4_1$ and its enantiomer $P4_3$. A crystal of $0.25 \times 0.30 \times 0.30$ mm, cut from a larger crystal, was fixed to a thin glass fibre (0.025 mm). Data for half the reciprocal sphere and $\theta < 60^\circ$ were collected by ω - 2θ scan on a Philips PW 1100 diffractom-

eter with graphite monochromatized Cu $K\alpha$ radiation. Lp-corrected data were reduced to 1103 unique reflexions. The structure was solved by direct methods (*MULTAN*). The 24 H atoms were located by Fourier difference synthesis. The positional and thermal parameters for all C and O atoms and the thermal parameters for all H atoms were refined by full-matrix least squares to a conventional R value of 0.05, calculated from all data with unit weights.* Final parameters ($P4_3$) are given for C and O in Table 1 and for H in Table 2.

The correct space group, $P4_3$, was determined from

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31675 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Atomic coordinates ($\times 10^4$) and vibrational components ($\times 10^3$)

$$T = \exp \{ -2\pi^2 (U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^* \dots) \}.$$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	3585 (6)	301 (7)	3816 (0)	34 (3)	48 (4)	22 (3)	-2 (3)	0 (3)	1 (3)
C(2)	2224 (6)	688 (7)	4259 (5)	29 (3)	46 (4)	38 (4)	10 (3)	-5 (3)	0 (3)
C(3)	2550 (6)	1217 (7)	5088 (5)	33 (3)	47 (4)	43 (4)	10 (3)	2 (3)	-11 (3)
C(4)	3720 (6)	296 (7)	5493 (5)	27 (3)	44 (4)	27 (3)	2 (3)	3 (3)	-6 (3)
C(5)	5179 (6)	320 (6)	5027 (5)	24 (3)	36 (3)	26 (3)	-1 (2)	3 (2)	-2 (3)
C(6)	6214 (6)	-901 (7)	5328 (5)	24 (3)	46 (3)	24 (3)	3 (2)	4 (2)	2 (3)
C(7)	7583 (6)	-1099 (6)	4804 (5)	22 (3)	36 (3)	32 (4)	-2 (2)	4 (2)	-3 (3)
C(8)	7596 (6)	-335 (8)	4015 (5)	27 (3)	70 (5)	28 (3)	3 (3)	8 (3)	0 (3)
C(9)	6132 (7)	-406 (9)	3623 (5)	29 (3)	112 (7)	23 (3)	2 (4)	4 (3)	-10 (4)
C(10)	4872 (6)	104 (7)	4138 (5)	23 (3)	42 (3)	23 (3)	-4 (2)	0 (2)	0 (3)
C(11)	5561 (7)	-2447 (7)	5381 (6)	35 (3)	49 (4)	63 (5)	-5 (3)	6 (3)	22 (4)
C(12)	6857 (8)	-3413 (8)	5184 (7)	50 (4)	48 (4)	90 (7)	-1 (3)	11 (5)	10 (4)
C(13)	4916 (9)	-2909 (10)	6179 (7)	67 (5)	97 (7)	95 (8)	15 (5)	38 (5)	59 (6)
C(14)	3865 (7)	758 (10)	6362 (5)	35 (4)	101 (3)	24 (4)	2 (3)	3 (3)	-15 (3)
C(15)	5922 (7)	1827 (7)	5145 (5)	44 (4)	41 (6)	46 (4)	-13 (4)	-5 (3)	-6 (4)
O(1)	7697 (5)	-2621 (4)	4637 (5)	52 (3)	40 (2)	67 (3)	-5 (2)	18 (3)	-11 (3)
O(2)	1288 (4)	-573 (5)	4332 (4)	24 (2)	70 (3)	36 (3)	-11 (2)	2 (2)	-10 (2)
O(3)	8812 (4)	-683 (5)	5266 (4)	24 (2)	58 (3)	30 (2)	0 (2)	0 (2)	0 (2)