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The crystal and molecular structure of tris(ethylthioxanthato)cobalt(III), $Co(C_2H_5SCS_2)_3$. An emendation.

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After re-investigation by Ting-i Li & S. J. Lippard (personal communication) it became clear that the previously published crystal structure of the title compound [Chiesi Villa, A., Gaetani Manfredotti, A., Guastini, C. & Nardelli, M. *Acta Cryst.* B**28**, 2231–2236] should have been that of the isostructural chromium(III) derivative: $Cr(C_2H_5SCS_2)_3$. New crystal-structure analyses for both Co(III) and Cr(III) complexes have been carried out using new sets of diffractometer data. The results of these analyses are compared.

The crystal structure of tris(ethylthioxanthato)cobalt(III), which was previously determined by Chiesi Villa, Gaetani Manfredotti, Guastini & Nardelli (1972) (hereafter indicated by VMGN), has been recently reinvestigated by Li & Lippard (1973) (hereafter indicated by TLL) who find that the results given by VMGN would be more consistent

Table 1. *Comparison of crystallographic data for different crystal analyses of* Cr(III) *and* Co(III) *tris*(*ethylthioxanthato*) *complexes*

	$Cr(C_2H_5CS_3)_3$			$Co(C_2H_5CS_3)_3$	
	VMGN	TLL	Present work	TLL	Present work
a	10·23 (1) Å	10·228 (2) Å	10·24 (1) Å	10·134 (2) Å	10·15 (1) Å
b	17·56 (1)	17·562 (4)	17·54 (1)	17·328 (9)	17·37 (1)
c	11·71 (1)	11·711 (2)	11·77 (1)	11·613 (2)	11·66 (1)
α	91·1 (1)°	91·08 (2)°	91·0 (1)°	90·99 (3)°	91·1 (1)°
β	115·5 (1)	115·51 (3)	115·7 (1)	115·09 (9)	115·1 (1)
γ	94·6 (1)	94·59 (2)	94·6 (1)	94·42 (3)	94·5 (1)
Z	4	4	4	4	4
R	0·046		0·038	0·037	0·041
Observed reflexions	5257		5408	6514	4780

Table 2. *Final atomic fractional coordinates* (× 10⁴), *thermal parameters* (× 10²) *and e.s.d.'s for non-hydrogen atoms*

The anisotropic temperature factor is: $\exp[-0{\cdot}25\,(B_{11}h^2a^{*2}+\cdots+2B_{23}klb^*c^*)]$.

$Cr(C_2H_5SCS_2)_3$

	x	*y*	*z*	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cr(1)	532 (1)	2314 (1)	1640 (1)	246 (2)	240 (2)	237 (2)	25 (2)	115 (2)	−2 (2)
Cr(2)	487 (1)	2767 (1)	6652 (1)	219 (2)	244 (2)	246 (2)	44 (2)	103 (2)	46 (2)
S(1)	2328 (1)	2747 (1)	915 (1)	275 (4)	323 (4)	325 (4)	39 (3)	136 (4)	62 (3)
S(2)	2711 (1)	1761 (1)	2954 (1)	343 (5)	355 (5)	280 (4)	66 (4)	131 (4)	67 (3)
S(3)	5254 (1)	2159 (1)	2482 (1)	245 (4)	529 (6)	462 (6)	57 (4)	101 (4)	85 (5)
S(4)	−952 (1)	1578 (1)	2416 (1)	381 (5)	344 (5)	317 (4)	20 (4)	215 (4)	0 (4)
S(5)	−587 (1)	1227 (1)	154 (1)	305 (4)	304 (4)	313 (4)	−24 (3)	185 (4)	−50 (3)
S(6)	−2511 (1)	169 (1)	925 (1)	411 (5)	339 (5)	441 (6)	−62 (4)	222 (5)	23 (4)
S(7)	−1259 (1)	3126 (1)	389 (1)	291 (4)	303 (4)	240 (4)	42 (3)	90 (3)	−11 (3)
S(8)	883 (1)	3427 (1)	2989 (1)	389 (5)	295 (4)	243 (4)	75 (4)	68 (4)	−28 (3)
S(9)	−1238 (1)	4547 (1)	1904 (1)	511 (6)	343 (5)	375 (5)	165 (5)	109 (5)	−37 (4)
S(11)	2128 (1)	2429 (1)	5808 (1)	227 (4)	326 (4)	294 (4)	12 (3)	112 (3)	−18 (3)
S(12)	2761 (1)	3517 (1)	7904 (1)	311 (4)	307 (4)	346 (5)	15 (3)	135 (4)	−42 (4)
S(13)	5102 (1)	3313 (1)	7146 (1)	252 (4)	380 (5)	421 (5)	−37 (4)	123 (4)	−33 (4)
S(14)	−879 (1)	3421 (1)	7509 (1)	322 (4)	344 (4)	287 (4)	74 (4)	170 (4)	66 (3)
S(15)	−623 (1)	3727 (1)	5190 (1)	344 (5)	370 (5)	333 (5)	119 (4)	197 (4)	133 (4)
S(16)	−2554 (1)	4601 (1)	5991 (1)	404 (5)	343 (5)	473 (6)	147 (4)	219 (5)	73 (4)
S(17)	−1381 (1)	1781 (1)	5408 (1)	255 (4)	325 (4)	243 (4)	29 (3)	60 (3)	53 (3)
S(18)	833 (1)	1699 (1)	7963 (1)	311 (4)	289 (4)	241 (4)	5 (3)	47 (3)	49 (3)
S(19)	−1224 (1)	337 (1)	6817 (1)	457 (6)	312 (5)	336 (5)	−65 (4)	97 (4)	51 (4)
C(1)	3436 (4)	2232 (2)	2077 (4)	249 (15)	268 (16)	296 (16)	5 (12)	88 (13)	−28 (13)
C(2)	5571 (5)	2623 (3)	1250 (5)	312 (20)	470 (24)	623 (29)	47 (18)	228 (20)	102 (21)
C(3)	5181 (6)	2104 (3)	108 (5)	385 (23)	728 (33)	528 (27)	156 (22)	233 (21)	149 (24)
C(4)	−1367 (4)	981 (2)	1129 (4)	240 (15)	267 (15)	277 (16)	55 (12)	115 (12)	26 (12)
C(5)	−2769 (5)	−285 (2)	−556 (5)	364 (20)	284 (18)	469 (23)	−22 (15)	162 (17)	−27 (16)
C(6)	−3960 (6)	15 (3)	−1692 (5)	393 (23)	560 (28)	510 (27)	94 (20)	138 (20)	88 (22)
C(7)	−563 (4)	3708 (2)	1718 (4)	290 (16)	270 (15)	272 (16)	48 (13)	125 (13)	32 (12)
C(8)	−2670 (6)	4671 (3)	340 (5)	509 (26)	428 (24)	476 (25)	211 (20)	85 (20)	17 (19)

Table 2 (*cont.*)

C(9)	−2114 (7)	5032 (4)	−520 (6)	740 (37)	539 (31)	556 (32)	146 (27)	79 (27)	161 (25)
C(11)	3348 (4)	3073 (2)	6940 (4)	248 (15)	291 (16)	254 (15)	54 (12)	102 (13)	54 (12)
C(12)	5315 (5)	2651 (3)	6061 (5)	280 (18)	443 (22)	477 (23)	−31 (16)	206 (17)	−41 (18)
C(13)	5701 (6)	1885 (3)	6599 (7)	423 (25)	548 (29)	932 (41)	77 (21)	384 (28)	−2 (27)
C(14)	−1368 (4)	3921 (2)	6184 (4)	246 (15)	225 (15)	322 (17)	−6 (12)	125 (13)	−1 (12)
C(15)	−2923 (5)	4968 (3)	4462 (5)	436 (23)	354 (20)	526 (25)	190 (18)	159 (19)	163 (18)
C(16)	−4096 (6)	4490 (4)	3374 (6)	477 (28)	701 (35)	604 (33)	125 (25)	89 (24)	16 (27)
C(17)	−625 (4)	1263 (2)	6690 (3)	262 (15)	288 (16)	244 (15)	32 (12)	121 (13)	22 (12)
C(18)	−2663 (5)	57 (3)	5250 (5)	379 (21)	385 (22)	449 (23)	−100 (17)	77 (18)	−23 (18)
C(19)	−2099 (6)	−226 (3)	4350 (5)	559 (29)	497 (27)	489 (27)	−49 (22)	115 (22)	−176 (21)
$Co(C_2H_5SCS_2)_3$									
Co(1)	495 (1)	2317 (1)	1634 (1)	231 (2)	238 (2)	229 (2)	19 (2)	109 (2)	−5 (2)
Co(2)	456 (1)	2755 (1)	6640 (1)	205 (2)	237 (2)	242 (2)	33 (2)	97 (2)	36 (2)
S(1)	2192 (1)	2737 (1)	927 (1)	256 (4)	308 (5)	301 (5)	25 (4)	118 (4)	45 (4)
S(2)	2525 (1)	1766 (1)	2909 (1)	303 (5)	328 (5)	295 (5)	54 (4)	125 (4)	51 (4)
S(3)	5187 (1)	2165 (1)	2508 (1)	228 (5)	526 (7)	453 (7)	49 (5)	88 (5)	71 (6)
S(4)	−939 (1)	1641 (1)	2397 (1)	341 (5)	346 (5)	308 (5)	23 (4)	195 (4)	11 (4)
S(5)	−521 (1)	1279 (1)	223 (1)	281 (5)	283 (5)	306 (5)	−21 (4)	169 (4)	−43 (4)
S(6)	−2491 (2)	172 (1)	955 (1)	389 (6)	351 (6)	451 (7)	−71 (5)	217 (5)	26 (5)
S(7)	−1225 (1)	3082 (1)	438 (1)	273 (5)	293 (5)	253 (5)	26 (4)	94 (4)	−13 (4)
S(8)	889 (1)	3382 (1)	2939 (1)	350 (5)	306 (5)	241 (5)	65 (4)	62 (4)	−21 (4)
S(9)	−1239 (2)	4557 (1)	1907 (1)	473 (7)	352 (6)	368 (6)	169 (5)	86 (5)	−38 (5)
S(11)	2017 (1)	2430 (1)	5824 (1)	221 (4)	290 (5)	286 (5)	15 (4)	105 (4)	−16 (4)
S(12)	2579 (1)	3487 (1)	7861 (1)	303 (5)	283 (5)	340 (5)	14 (4)	139 (4)	−31 (4)
S(13)	5030 (1)	3322 (1)	7136 (1)	240 (5)	369 (6)	423 (6)	−52 (4)	126 (4)	−30 (5)
S(14)	−876 (1)	3343 (1)	7478 (1)	312 (5)	329 (5)	301 (5)	55 (4)	163 (4)	54 (4)
S(15)	−558 (1)	3684 (1)	5256 (1)	293 (5)	335 (5)	335 (5)	91 (4)	177 (4)	106 (4)
S(16)	−2523 (2)	4608 (1)	6035 (1)	388 (6)	342 (6)	471 (7)	148 (5)	220 (5)	63 (5)
S(17)	−1328 (1)	1828 (1)	5453 (1)	238 (4)	311 (5)	258 (5)	19 (4)	69 (4)	47 (4)
S(18)	851 (1)	1739 (1)	7913 (1)	284 (5)	291 (5)	229 (4)	−3 (4)	42 (4)	39 (4)
S(19)	−1231 (2)	335 (1)	6831 (1)	421 (6)	303 (5)	345 (6)	−68 (5)	102 (5)	55 (4)
C(1)	3341 (5)	2232 (3)	2090 (4)	255 (18)	312 (20)	270 (19)	10 (15)	111 (15)	−39 (15)
C(2)	5492 (5)	2634 (3)	1250 (6)	268 (21)	449 (27)	685 (35)	4 (19)	233 (22)	79 (24)
C(3)	5127 (6)	2101 (4)	102 (6)	333 (24)	688 (37)	559 (33)	110 (24)	200 (24)	116 (28)
C(4)	−1362 (5)	1003 (3)	1154 (4)	265 (18)	241 (18)	326 (20)	50 (14)	134 (16)	17 (15)
C(5)	−2725 (6)	−265 (3)	−548 (5)	349 (22)	295 (21)	451 (26)	2 (17)	146 (19)	−55 (18)
C(6)	−3933 (6)	31 (4)	−1670 (6)	440 (28)	634 (35)	425 (28)	17 (25)	146 (23)	20 (25)
C(7)	−556 (5)	3710 (3)	1725 (4)	298 (19)	308 (19)	239 (18)	25 (15)	121 (15)	26 (15)
C(8)	−2684 (6)	4657 (3)	338 (5)	475 (28)	406 (26)	396 (25)	134 (22)	41 (22)	−32 (21)
C(9)	−2140 (8)	5007 (4)	−557 (7)	687 (40)	537 (36)	665 (41)	136 (30)	88 (32)	163 (30)
C(11)	3264 (5)	3069 (3)	6933 (4)	237 (17)	266 (18)	292 (19)	31 (14)	83 (15)	58 (15)
C(12)	5233 (6)	2660 (3)	6020 (6)	311 (21)	488 (28)	520 (29)	2 (19)	226 (21)	−15 (22)
C(13)	5606 (7)	1869 (4)	6542 (8)	419 (29)	512 (33)	1095 (54)	39 (24)	432 (34)	−24 (33)
C(14)	−1370 (5)	3900 (3)	6216 (5)	255 (17)	248 (18)	353 (21)	27 (14)	147 (16)	39 (15)
C(15)	−2864 (6)	4968 (3)	4500 (6)	422 (25)	367 (24)	488 (28)	144 (20)	160 (22)	106 (21)
C(16)	−4027 (7)	4501 (4)	3421 (6)	496 (32)	718 (41)	515 (34)	137 (28)	89 (26)	−49 (29)
C(17)	−615 (5)	1267 (3)	6701 (4)	252 (17)	291 (19)	251 (18)	8 (14)	128 (15)	−1 (14)
C(18)	−2673 (6)	84 (3)	5250 (5)	364 (23)	382 (25)	445 (27)	−76 (19)	123 (20)	0 (20)
C(19)	−2134 (7)	−196 (4)	4315 (6)	557 (32)	506 (31)	515 (32)	−12 (25)	158 (26)	−153 (25)

Table 3. *Coordinates* ($\times 10^3$), *thermal parameters and e.s.d.'s for hydrogen atoms*

$Cr(C_2H_5SCS_2)_3$

	x	*y*	*z*	*B*		*x*	*y*	*z*	*B*
H(2,1)	648 (6)	277 (3)	168 (5)	8·8 (1·5)	H(18,1)	−330 (5)	−33 (3)	534 (4)	5·5 (1·2)
H(2,2)	514 (5)	308 (3)	113 (4)	5·2 (1·1)	H(18,2)	−324 (5)	55 (2)	495 (4)	4·4 (1·1)
H(3,1)	426 (6)	203 (3)	−25 (5)	6·9 (1·3)	H(19,1)	−148 (6)	27 (3)	423 (6)	9·4 (1·6)
H(3,2)	544 (5)	236 (3)	−43 (4)	5·2 (1·2)	H(19,2)	−150 (6)	−70 (3)	467 (5)	8·2 (1·5)
H(3,3)	564 (6)	164 (3)	42 (5)	7·5 (1·5)	H(19,3)	−293 (5)	−48 (3)	361 (5)	5·7 (1·2)
H(12,1)	617 (5)	295 (3)	601 (4)	5·1 (1·1)	H(8,1)	−333 (6)	499 (3)	47 (5)	7·2 (1·4)
H(12,2)	437 (5)	261 (3)	518 (5)	6·7 (1·3)	H(8,2)	−309 (5)	418 (3)	6 (5)	6·1 (1·3)
H(13,1)	655 (6)	201 (3)	728 (5)	7·8 (1·5)	H(9,1)	−150 (6)	549 (3)	−9 (5)	8·4 (1·5)
H(13,2)	578 (5)	162 (3)	590 (5)	6·4 (1·3)	H(9,2)	−283 (7)	523 (4)	−115 (6)	10·3 (1·8)
H(13,3)	489 (5)	166 (3)	668 (5)	6·1 (1·3)	H(9,3)	−162 (6)	461 (3)	−80 (5)	8·9 (1·6)
H(5,1)	−182 (5)	−21 (3)	−55 (5)	6·3 (1·3)	H(15,1)	−328 (6)	540 (3)	447 (5)	7·0 (1·4)
H(5,2)	−306 (5)	−84 (3)	−45 (4)	5·1 (1·1)	H(15,2)	−199 (6)	496 (3)	441 (5)	7·1 (1·4)
H(6,1)	−388 (6)	54 (3)	−173 (5)	7·0 (1·4)	H(16,1)	−381 (6)	403 (3)	327 (5)	8·5 (1·6)
H(6,2)	−407 (7)	−27 (3)	−250 (6)	10·3 (1·8)	H(16,2)	−415 (6)	483 (3)	266 (5)	8·1 (1·5)
H(6,3)	−470 (7)	−17 (4)	−152 (6)	10·2 (1·8)	H(16,3)	−495 (6)	446 (3)	341 (6)	9·0 (1·7)

Table 3 (*cont.*)

$Co(C_2H_5SCS_2)_3$

	x	*y*	*z*	*B*
H(2,1)	645 (6)	275 (3)	167 (6)	7·0 (1·6)
H(2,2)	506 (6)	308 (3)	111 (6)	6·9 (1·6)
H(3,1)	423 (7)	200 (3)	−23 (6)	7·6 (1·6)
H(3,2)	541 (7)	236 (4)	−46 (6)	8·0 (1·7)
H(3,3)	560 (6)	161 (3)	38 (6)	7·0 (1·6)
H(12,1)	610 (6)	292 (3)	595 (6)	6·7 (1·5)
H(12,2)	432 (6)	260 (3)	519 (5)	5·8 (1·4)
H(13,1)	648 (7)	199 (4)	727 (6)	8·0 (1·7)
H(13,2)	575 (7)	160 (4)	589 (6)	8·5 (1·7)
H(13,3)	485 (6)	163 (3)	670 (5)	6·1 (1·5)
H(5,1)	−188 (6)	−19 (3)	−57 (6)	7·3 (1·6)
H(5,2)	−305 (6)	−84 (3)	−45 (6)	7·3 (1·6)
H(6,1)	−385 (6)	57 (3)	−172 (5)	5·7 (1·4)
H(6,2)	−407 (6)	−27 (3)	−251 (5)	6·4 (1·5)
H(6,3)	−472 (7)	−15 (4)	−154 (6)	9·1 (1·8)
H(18,1)	−329 (6)	−33 (3)	538 (5)	6·3 (1·5)
H(18,2)	−324 (6)	57 (3)	495 (5)	4·9 (1·3)
H(19,1)	−154 (6)	28 (3)	424 (6)	7·1 (1·6)
H(19,2)	−153 (6)	−69 (3)	467 (6)	6·8 (1·5)
H(19,3)	−299 (7)	−47 (4)	357 (6)	8·1 (1·7)
H(8,1)	−336 (6)	499 (3)	45 (6)	7·0 (1·6)
H(8,2)	−314 (6)	418 (3)	2 (5)	5·3 (1·4)
H(9,1)	−155 (6)	549 (3)	−10 (6)	7·0 (1·6)
H(9,2)	−293 (7)	518 (4)	−116 (6)	9·7 (1·9)
H(9,3)	−162 (7)	459 (4)	−76 (6)	8·1 (1·7)
H(15,1)	−325 (6)	541 (3)	453 (6)	6·9 (1·6)
H(15,2)	−200 (6)	495 (3)	443 (5)	6·6 (1·5)
H(16,1)	−380 (6)	403 (3)	330 (6)	6·6 (1·5)
H(16,2)	−417 (6)	481 (3)	266 (5)	6·0 (1·4)
H(16,3)	−490 (6)	447 (3)	347 (5)	5·9 (1·4)

if assigned to the isostructural chromium-(III) complex. New crystal-structure analyses of both Co(III) and Cr(III) derivatives have therefore been carried out in our laboratory to test the suggestion of TLL and the results of these analyses confirm their hypothesis, indicating that a wrong assignment was made by VMGN. In the present short communication a comparison of the results of the new analyses is briefly given.

In Table 1 crystallographic data from the different analyses are compared and in Tables 2 and 3 final atomic fractional coordinates with thermal parameters from the present analyses are given. These results have been obtained using the same techniques used in the previous paper. In Table 4 the most relevant bond distances and angles, found from the two new analyses, are quoted and compared with the data obtained previously, showing that the changes are only a few units in the third decimal place for bond lengths and in the first decimal figure for the angles. These results remove all the discrepancies between Co–S distances and spectroscopic properties considered in the previous paper, as pointed out by Li & Lippard.

Table 4. *Most relevant bond distances and angles*

The formulae used in averaging are:

$$l_{av}=\sum_i \frac{l_i}{\sigma_i^2} \Big/ \sum_i \frac{1}{\sigma_i^2} \text{ and } \sigma_{av}=\left(1 \Big/ \sum_i \frac{1}{\sigma_i^2}\right)^{1/2}$$

where l_i are the individual distances or angles and σ_i are the corresponding e.s.d.'s.

	$Co(C_2H_5CS_3)_3$ Present work	VMGN	$Cr(C_2H_5CS_3)_3$ Present work
M(1)–S(1)	2·282 (3) Å	2·410 (3) Å	2·418 (3) Å
M(1)–S(2)	2·267 (4)	2·389 (4)	2·391 (4)
M(1)–S(4)	2·271 (3)	2·395 (3)	2·396 (3)
M(1)–S(5)	2·275 (4)	2·404 (4)	2·409 (4)
M(1)–S(7)	2·265 (4)	2·396 (4)	2·390 (4)
M(1)–S(8)	2·271 (3)	2·395 (3)	2·397 (4)
M(2)–S(11)	2·266 (3)	2·391 (3)	2·398 (3)
M(2)–S(12)	2·277 (4)	2·409 (4)	2·406 (4)
M(2)–S(14)	2·264 (3)	2·386 (3)	2·394 (3)
M(2)–S(15)	2·285 (4)	2·408 (4)	2·408 (4)
M(2)–S(17)	2·262 (4)	2·394 (4)	2·397 (4)
M(2)–S(18)	2·277 (3)	2·400 (3)	2·398 (3)
Average	2·272 (1)	2·398 (1)	2·400 (1)
S(1)M(1)S(2)	76·2 (1)°	73·9 (1)°	73·8 (1)°
S(4)M(1)S(5)	76·3 (1)	74·2 (1)	74·5 (1)
S(7)M(1)S(8)	76·2 (1)	74·3 (1)	74·5 (1)
S(11)M(2)S(12)	76·2 (1)	74·0 (1)	74·2 (1)
S(14)M(2)S(15)	76·4 (1)	74·3 (1)	74·5 (1)
S(17)M(2)S(18)	76·1 (1)	74·1 (1)	74·2 (1)
Average	76·23 (4)	74·13 (4)	74·28 (4)

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