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Structure of (-)₅₈₉-Tris(3,3'-dimethyl-2,2'-bipyridine)rhodium(III) Triperchlorate Monohydrate

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Abstract. $[Rh(C_{12}H_{12}N_2)_3]^{3+}.3ClO_4^-.H_2O, M_r = 971.4$, orthorhombic, $P2_12_12_1$ (systematic absences $h00, h \neq 2n, 0k0, k \neq 2n, 00l, l \neq 2n$), a = 17.675 (2), b = 18.151 (2), c = 12.637 (4) Å, U = 4054 (1) Å³, $Z = 4, D_m = 1.57, D_x = 1.59$ Mg m⁻³, λ (Mo $K\alpha$) = 0.7107 Å, μ (Mo $K\alpha$) = 0.68 mm⁻¹. The structure was solved by Patterson-Fourier methods and refined to a final R value of 0.045 for the 4742 observed reflections. The absolute configuration of the complex ion can be designated as $\Lambda(\delta\lambda\lambda)$.

Introduction. The structures of two bis(diamine) Co^{III} complexes with 3,3'-dimethyl-2,2'-bipyridine (dmbpy) have been reported (Sato & Saito, 1978; Ohba, Sato & Saito, 1979). Recently, tris(dmbpy) complexes of Co^{III} and Rh^{III} were prepared and resolved into optical isomers (Suzuki, 1979). Because of its stability, the Rh complex was selected and subjected to crystal structure analysis in order to establish the absolute configuration and to gain the conformational details. Colorless platelike crystals of the title compound were kindly supplied by Dr Suzuki. The cell dimensions were refined by least squares on the basis of 15 2θ values of higher-order reflections $(31^{\circ} < 2\theta < 38^{\circ})$ measured on a diffractometer with Mo K_{α} radiation. Intensities were measured from a spherical crystal 0.5 mm in diameter on a Rigaku automated four-circle diffractometer. Data were collected by the θ -2 θ scan mode to a maximum 2θ value of 55° with Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) monochromated by a graphite plate. Of the 5153 measured unique reflections (the +h+k+l set), 4742 with $|F_{o}| > 3\sigma(|F_{o}|)$ were considered as observed. The intensities were corrected for Lorentz and polarization factors, but an absorption correction was not applied. Approximate positions of the Rh and three Cl atoms were deduced from three-dimensional Patterson maps. and the positions of all non-hydrogen atoms were derived by the Fourier method. They were refined by block-diagonal least squares with anisotropic thermal parameters. With R = 0.055 ($R = \sum |\Delta F| / \sum |F_o|$), all the H atoms were located from a difference synthesis except those of the water molecule. In the refinement

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the weight $w = [\sigma^2(|F_o|) + (0.010|F_o|)^2]^{-1}$ was assigned. The final R was 0.045 and $R_2 = 0.062 \{R_2 = [\sum w(\Delta F)^2 / \sum w(F_o)^2]^{1/2}\}$ for 4742 reflections. The atomic scattering factors were taken from *Inter*national Tables for X-ray Crystallography (1974). The calculations were carried out on a FACOM 230-48 computer of this Institute with a local version of UNICS (Ashida, 1967). The final atomic parameters are listed in Table 1.[†]

The absolute structure was determined by an anomalous-scattering technique. Eight hkl and $\bar{h}kl$ pairs, for which $|F_c(hkl)|$ and $|F_c(\bar{h}kl)|$ differed by more than 7%, were measured with Mo $K\alpha$ radiation. The observed and calculated intensity ratios are compared in Table 2. All the observed |F|'s were

[†] Lists of structure factors, thermal parameters and relevant interatomic distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34256 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.





$[Rh(C_{12}H_{12}N_2)_3]^{3+}.3ClO_4^-.H_2O$

Table 1. Positional parameters ($\times 10^4$; for Rh and Cl $\times 10^5$; for H $\times 10^3$)

$ \begin{array}{cccccccccccccccccccccccccccccccccccc$		x	У	Z		x	у	Z
	Rh	47723 (2)	42539 (2)	39490 (3)	O(3)	2955 (5)	3982 (8)	10140 (12)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	N(1)	4105 (3)	4865 (3)	4942 (4)	O(4)	2671 (9)	4066 (12)	8501 (12)
	N(2)	5533 (3)	5047 (3)	4356 (4)	O(5)	2904 (5)	6551 (5)	9549 (12)
	N(3)	3978 (3)	3465 (3)	3612 (4)	O(6)	4122 (3)	6961 (3)	9687 (5)
	N(4)	4915 (3)	3589 (3)	5234 (4)	O(7)	3400 (5)	7196 (7)	8217 (7)
$ N(6) \qquad 5469 (3) \qquad 3720 (3) \qquad 2951 (4) \qquad O(9) \qquad 7006 (9) \qquad 4437 (7) \qquad 7320 (12) \\ 3457 (4) \qquad 4422 (4) \qquad 5366 (6) \qquad O(10) \qquad 7248 (5) \qquad 5565 (6) \qquad 8028 (10) \\ C(2) \qquad 3176 (4) \qquad 4929 (4) \qquad 6274 (7) \qquad O(11) \qquad 6081 (6) \qquad 5110 (7) \qquad 7985 (11) \\ C(3) \qquad 3599 (5) \qquad 5423 (5) \qquad 6805 (6) \qquad O(12) \qquad 6540 (6) \qquad 5458 (8) \qquad 6519 (7) \\ C(4) \qquad 4270 (4) \qquad 5690 (4) \qquad 6414 (5) \qquad O(13) \qquad 6671 (8) \qquad 2826 (7) \qquad 8952 (11) \\ C(5) \qquad 4493 (4) \qquad 5427 (4) \qquad 5428 (5) \qquad H(C1) \qquad 310 (4) \qquad 417 (4) \qquad 526 (5) \\ C(7) \qquad 5119 (5) \qquad 5431 (4) \qquad 4790 (6) \qquad H(C3) \qquad 344 (6) \qquad 5533 (6) \qquad 726 (6) \\ C(7) \qquad 5519 (5) \qquad 5431 (4) \qquad 4790 (6) \qquad H(C3) \qquad 344 (6) \qquad 5533 (6) \qquad 726 (6) \\ C(8) \qquad 6632 (4) \qquad 5766 (5) \qquad 4029 (6) \qquad H(C9) \qquad 703 (4) \qquad 578 (4) \qquad 356 (6) \\ C(10) \qquad 6237 (4) \qquad 5166 (5) \qquad 4029 (6) \qquad H(C9) \qquad 703 (4) \qquad 578 (4) \qquad 356 (6) \\ C(10) \qquad 6237 (4) \qquad 516 (5) \qquad 4029 (6) \qquad H(C11) \\ 443 (5) \qquad 671 (5) \qquad 709 (7) \\ C(12) \qquad 5079 (6) \qquad 7029 (5) \qquad 4964 (9) \qquad H(C11) \\ H(C11) \qquad 443 (5) \qquad 671 (5) \qquad 709 (7) \\ C(13) \qquad 3361 (3) \qquad 4320 (4) \qquad 2695 (5) \qquad H(C11) \\ 4476 (5) \qquad 592 (5) \qquad 795 (7) \\ C(14) \qquad 3221 (5) \qquad 7272 (5) \qquad 3066 (7) \qquad H(C12) \\ 4489 (5) \qquad 6637 (6) \qquad 380 (5) \qquad 224 (8) \\ C(14) \qquad 3221 (5) \qquad 7272 (5) \qquad 3066 (7) \qquad H(C12) \\ 4480 (5) \qquad 6646 (5) \qquad 521 (7) \\ C(16) \qquad 3729 (4) \qquad 2206 (4) \qquad 931 (7) \qquad H(C13) \qquad 307 (4) \qquad 746 (4) \qquad 522 (5) \\ C(17) \qquad 4009 (4) \qquad 2891 (4) \qquad 4288 (5) \qquad H(C13) \qquad 307 (4) \qquad 746 (4) \qquad 522 (5) \\ C(17) \qquad 4009 (4) \qquad 2891 (4) \qquad 428 (5) \qquad H(C11) \qquad 307 (6) \qquad 380 (5) \qquad 224 (8) \\ C(21) \qquad 5181 (5) \qquad 3470 (4) \qquad 7039 (5) \qquad H(C21) \qquad 538 (3) \qquad 406 (3) \qquad 578 (4) \\ C(20) \qquad 448 (6) \qquad 748 (9) \\ C(21) \qquad 5181 (5) \qquad 3470 (4) \qquad 728 (5) \qquad H(C21) \qquad 336 (6) \qquad 145 (6) \qquad 788 (9) \\ C(21) \qquad 5181 (5) \qquad 3470 (4) \qquad 726 (5) \qquad H(C23) \qquad 360 (6) \qquad 144 (5) (6) \qquad 535 (5) \\ 745 (8) \\ C(22) \qquad 5458 (3) \ 7713 (6) \qquad H(C23) \qquad 360 (6) \qquad 144 (5) (6) \ 538 (6) \\ C(24) \qquad 346 (6) \ 538 (6) \qquad H(C23) \qquad 360 (6) \qquad 144 (6) \ 531 (6) \\ C(25) \qquad 467 (4) \qquad 3523 (4) \qquad 255 (6) \qquad H(C23) \qquad 316 (6) \ 144 (5) (6) \ 533 (6) \\ C(24) \qquad 346 (5) \ 522 (4) \qquad 1550 (6) \qquad H(C23) \ 316 (6) \ 4145 (4) \ 533 (6$	N(5)	4612 (3)	4859 (3)	2596 (4)	O(8)	3110 (4)	7782 (5)	9656 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	N(6)	5469 (3)	3720 (3)	2951 (4)	O(9)	7006 (9)	4437 (7)	7320 (12)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(1)	3457 (4)	4623 (4)	5366 (6)	O(10)	7248 (5)	5565 (6)	8028 (10)
	C(2)	3176 (4)	4929 (4)	6274 (7)	O(11)	6081 (6)	5110 (7)	7985 (11)
	C(3)	3599 (5)	5423 (5)	6805 (6)	O(12)	6540 (6)	5458 (8)	6519 (7)
$ C(5) 4493 (4) 5427 (4) 5428 (5) \\ C(6) 5192 (4) 5632 (3) 4861 (5) \\ C(7) 5519 (5) 6341 (4) 4790 (6) \\ H(C3) 265 (4) 470 (4) 663 (6) \\ C(8) 6250 (5) 6392 (4) 4436 (6) \\ H(C3) 344 (6) 553 (6) 726 (9) \\ C(8) 6632 (4) 576 (5) 4029 (6) \\ H(C9) 703 (4) 578 (4) 3356 (6) \\ C(10) 6237 (4) 5113 (4) 3970 (5) \\ C(11) 4768 (6) 6210 (6) 7067 (7) \\ H(C11)1 443 (5) 671 (5) 709 (7) \\ C(12) 5079 (6) 7029 (5) 4964 (9) \\ H(C11)2 476 (5) 592 (5) 795 (7) \\ C(13) 3611 (4) 3420 (4) 22695 (5) \\ H(C11)2 476 (5) 592 (5) 795 (7) \\ C(13) 3613 (4) 3420 (4) 22695 (5) \\ H(C11)2 476 (5) 592 (5) 795 (7) \\ C(13) 3613 (4) 3420 (4) 22695 (5) \\ H(C11)3 519 (6) 677 (5) 650 (8) \\ C(14) 3221 (5) 2792 (5) 2389 (7) \\ H(C12)1 510 (3) 735 (3) 442 (4) \\ C(15) 3327 (5) 2175 (5) 3066 (7) \\ H(C12)3 507 (4) 746 (4) 525 (5) \\ C(17) 4009 (4) 2891 (4) 4288 (5) \\ H(C13) 363 (6) 380 (5) 224 (8) \\ C(18) 4364 (4) 3063 (4) 5310 (5) \\ H(C14) 291 (3) 276 (3) 172 (5) \\ C(20) 4599 (5) 3028 (5) 7143 (6) \\ H(C21) 530 (3) 710 (3) 228 (5) \\ C(20) 4599 (5) 3028 (5) 7143 (6) \\ H(C23)1 436 (4) 155 (4) 470 (6) \\ C(24) 3911 (6) 1486 (4) 4491 (9) \\ H(C23)1 436 (4) 155 (4) 470 (6) \\ C(24) 3911 (6) 1486 (4) 4491 (9) \\ H(C23)1 436 (4) 155 (4) 470 (6) \\ C(24) 4599 (5) 3028 (5) 7143 (6) \\ H(C24)1 333 (0 (4) 145 (4) 503 (6) \\ C(24) 338 (4) 2350 (5) 745 (8) \\ C(24) 338 (4) 3510 (5) 675 (6) \\ H(C24)1 333 (0 (4) 145 (4) 503 (6) \\ C(25) 4075 (4) 5338 (4) 2510 (6) \\ H(C24)1 333 (0 (4) 145 (4) 503 (6) \\ C(25) 4075 (4) 5338 (4) 2510 (6) \\ H(C24)1 333 (6) (6) 544 (5) 578 (4) \\ C(25) 475 (4) 5338 (4) 2510 (6) \\ H(C24)1 333 (6) (6) 134 (5) 675 (6) \\ H(C24)1 333 (6) (6) 134 (5) 675 (6) \\ H(C24)1 333 (6) (6) 134 (6) 675 (6) \\ H(C33) 675 (6) 474 (6) 778 (4) \\ 326 (5) \\ C(33) 655 (5) 401 (6) \\ 4$	C(4)	4270 (4)	5690 (4)	6414 (5)	O(13)	6671 (8)	2826 (7)	8952 (11)
$ \begin{array}{cccccc} C(6) & 5192 (4) & 5632 (3) & 4861 (5) & H(C2) & 265 (4) & 470 (4) & 663 (6) \\ C(7) & 5519 (5) & 6334 (4) & 4790 (6) & H(C3) & 344 (6) & 553 (6) & 726 (9) \\ C(8) & 6250 (5) & 6392 (4) & 4436 (6) & H(C8) & 649 (5) & 682 (5) & 445 (7) \\ C(9) & 6632 (4) & 5766 (5) & 4029 (6) & H(C9) & 703 (4) & 578 (4) & 336 (6) \\ C(10) & 6237 (4) & 5113 (4) & 3970 (5) & H(C110) & 644 (3) & 464 (3) & 366 (5) \\ C(11) & 4768 (6) & 6210 (6) & 7067 (7) & H(C111)1 & 443 (5) & 671 (5) & 799 (7) \\ C(12) & 5079 (6) & 7029 (5) & 4964 (9) & H(C11)2 & 476 (5) & 592 (5) & 795 (7) \\ C(13) & 3613 (4) & 3420 (4) & 2695 (5) & H(C11)3 & 519 (6) & 637 (5) & 650 (8) \\ C(14) & 3221 (5) & 2792 (5) & 2389 (7) & H(C12)1 & 510 (3) & 735 (3) & 442 (4) \\ C(15) & 3327 (5) & 2175 (5) & 3066 (7) & H(C12)2 & 489 (5) & 686 (5) & 521 (7) \\ C(16) & 3729 (4) & 2206 (4) & 3931 (7) & H(C12)3 & 507 (4) & 746 (4) & 522 (5) \\ C(17) & 4009 (4) & 2891 (4) & 4288 (5) & H(C13) & 363 (6) & 380 (5) & 224 (8) \\ C(18) & 4364 (4) & 3063 (4) & 5310 (5) & H(C14) & 291 (3) & 276 (3) & 172 (5) \\ C(19) & 4161 (4) & 2782 (4) & 6312 (5) & H(C21) & 530 (5) & 355 (5) & 745 (8) \\ C(22) & 5338 (4) & 378 (4) & 6067 (5) & H(C22) & 585 (3) & 406 (3) 578 (5) \\ C(23) & 3911 (6) & 1486 (4) & 4491 (9) & H(C23)1 & 436 (4) & 155 (4) & 470 (6) \\ C(24) & 3469 (5) & 2356 (6) & 6510 (7) & H(C23)1 & 323 (4) & 260 (4) & 706 (5) \\ C(27) & 4137 (4) & 5301 (5) & 675 (6) & H(C24)1 & 323 (4) & 260 (4) & 706 (5) \\ C(27) & 4137 (4) & 5301 (5) & 675 (6) & H(C24)1 & 323 (4) & 260 (4) & 706 (5) \\ C(27) & 4457 (4) & 5334 (4) & 1367 (5) & H(C27) & 401 (5) & 542 (5) & 15 (8) \\ C(23) & 6485 (5) & 2901 (4) & 255 (6) & H(C23) & 391 (3) & 573 (4) & 328 (5) \\ C(29) & 4945 (3) & 4566 (3) & 1731 (5) & H(C27) & 401 (5) & 542 (5) & 15 (8) \\ C(33) & 6485 (5) & 2901 (4) & 255 (6) & H(C33) & 675 (6) & 247 (6) & 278 (9) \\ C(34) & 5934 (4) & 3167 (4) & 3232 (5) & H(C33) & 675 (6) & 247 (6) & 278 (9) \\ C(34) & 5934 (4) & 3167 (4) & 3232 (20) & H(C35) & 506 (4) & 379 (4) & 374 (4) & -97 (5) \\ C(11) & 26491 (12) & 35948 $	C(5)	4493 (4)	5427 (4)	5428 (5)	H(C1)	310 (4)	417 (4)	526 (5)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(6)	5192 (4)	5632 (3)	4861 (5)	H(C2)	265 (4)	470 (4)	663 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(7)	5519 (5)	6341 (4)	4790 (6)	H(C3)	344 (6)	553 (6)	726 (9)
	C(8)	6250 (5)	6392 (4)	4436 (6)	H(C8)	649 (5)	682 (5)	445 (7)
$ \begin{array}{cccccc} C(10) & 6237 (4) & 5113 (4) & 3970 (5) & H(C 10) & 644 (3) & 464 (3) & 366 (5) \\ C(11) & 4768 (6) & 6210 (6) & 7067 (7) & H(C 11)1 & 443 (5) & 671 (5) & 709 (7) \\ C(12) & 5079 (6) & 7029 (5) & 4964 (9) & H(C 11)3 & 519 (6) & 637 (5) & 650 (8) \\ C(14) & 3221 (5) & 2792 (5) & 2389 (7) & H(C 12)1 & 510 (3) & 735 (3) & 442 (4) \\ C(15) & 3327 (5) & 2175 (5) & 3066 (7) & H(C 12)1 & 510 (3) & 735 (3) & 442 (4) \\ C(16) & 3729 (4) & 2206 (4) & 3931 (7) & H(C 12)3 & 507 (4) & 746 (4) & 525 (5) \\ C(17) & 4009 (4) & 2891 (4) & 4288 (5) & H(C 13) & 363 (6) & 380 (5) & 224 (8) \\ C(18) & 4364 (4) & 3063 (4) & 5310 (5) & H(C 15) & 302 (3) & 170 (3) & 280 (5) \\ C(20) & 4599 (5) & 3028 (5) & 7143 (6) & H(C 20) & 448 (6) & 284 (6) & 788 (9) \\ C(21) & 5181 (5) & 3470 (4) & 7039 (5) & H(C 21) & 530 (5) & 355 (5) & 745 (8) \\ C(22) & 5338 (4) & 3785 (4) & 6067 (5) & H(C 23) & 350 (4) & 155 (4) & 470 (6) \\ C(24) & 3469 (5) & 2356 (6) & 6510 (7) & H(C 23) & 386 (4) & 155 (4) & 470 (6) \\ C(24) & 3469 (5) & 2356 (6) & 6510 (7) & H(C 23) & 360 (4) & 145 (4) & 503 (6) \\ C(25) & 4075 (4) & 5338 3 (4) & 2510 (6) & H(C 24) & 309 (6) & 233 (6) & 592 (9) \\ C(26) & 3876 (4) & 555 (4) & 1550 (6) & H(C 24) & 309 (6) & 233 (6) & 592 (9) \\ C(26) & 4672 (4) & 4753 (4) & 726 (5) & H(C 24) & 309 (6) & 233 (6) & 592 (9) \\ C(28) & 4672 (4) & 4753 (4) & 726 (5) & H(C 24) & 309 (6) & 233 (6) & 592 (9) \\ C(28) & 4672 (4) & 323 (4) & 1367 (5) & H(C 24) & 309 (6) & 233 (6) & 592 (9) \\ C(28) & 4672 (4) & 3292 (4) & 1682 (6) & H(C 32) & 716 (4) & 321 (4) & 129 (6) \\ C(31) & 6195 (4) & 3887 (4) & 1367 (5) & H(C 25) & 357 (4) & 590 (4) & 140 (6) \\ C(31) & 6195 (4) & 3887 (4) & 1367 (5) & H(C 23) & 503 (4) & 301 (4) & 394 (6) \\ C(33) & 6485 (5) & 2901 (4) & 2555 (7) & H(C 33) & 675 (6) & 247 (6) & 278 (9) \\ C(33) & 6485 (5) & 2901 (4) & 2555 (6) & H(C 33) & 675 (6) & 247 (6) & 278 (9) \\ C(34) & 5934 (4) & 3167 (4) & 3232 (20) & H(C 35) & 502 (4) & 478 (4) & -97 (5) \\ C(31) & 6195 (4) & 3887 (4) & 1367 (5) & H(C 23) & 503 (4) & 301 (4) & 394 (6) \\ C(33$	C(9)	6632 (4)	5766 (5)	4029 (6)	H(C9)	703 (4)	578 (4)	356 (6)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(10)	6237 (4)	5113 (4)	3970 (5)	H(C10)	644 (3)	464 (3)	366 (5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(11)	4768 (6)	6210 (6)	7067 (7)	H(C11)1	443 (5)	671 (5)	709 (7)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(12)	5079 (6)	7029 (5)	4964 (9)	H(C11)2	476 (5)	592 (5)	795 (7)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(13)	3613 (4)	3420 (4)	2695 (5)	H(C11)3	519 (6)	637 (5)	650 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(14)	3221 (5)	2792 (5)	2389 (7)	H(C12)1	510 (3)	735 (3)	442 (4)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(15)	3327 (5)	2175 (5)	3066 (7)	H(C12)2	489 (5)	686 (5)	521 (7)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(16)	3729 (4)	2206 (4)	3931 (7)	H(C12)3	507 (4)	746 (4)	525 (5)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(17)	4009 (4)	2891 (4)	4288 (5)	H(C13)	363 (6)	380 (5)	224 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(18)	4364 (4)	3063 (4)	5310 (5)	H(C14)	291 (3)	276 (3)	172 (5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(19)	4161 (4)	2782 (4)	6312 (5)	H(C15)	302 (3)	170 (3)	280 (5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(20)	4599 (5)	3028 (5)	7143 (6)	H(C20)	448 (6)	284 (6)	788 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(21)	5181 (5)	3470 (4)	7039 (5)	H(C21)	530 (5)	355 (5)	745 (8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(22)	5338 (4)	3785 (4)	6067 (5)	H(C22)	585 (3)	406 (3)	578 (5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(23)	3911 (6)	1486 (4)	4491 (9)	H(C23)1	436 (4)	155 (4)	470 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(24)	3469 (5)	2356 (6)	6510 (7)	H(C23)2	388 (4)	103 (4)	413 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(25)	4075 (4)	5383 (4)	2510 (6)	H(C23)3	360 (4)	145 (4)	503 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(26)	3876 (4)	5652 (4)	1550 (6)	H(C24)1	323 (4)	260 (4)	706 (5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(27)	4137 (4)	5301 (5)	675 (6)	H(C24)2	309 (6)	233 (6)	592 (9)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(28)	4672 (4)	4753 (4)	726 (5)	H(C24)3	360 (6)	184 (5)	669 (8)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(29)	4945 (3)	4566 (3)	1731 (5)	H(C25)	391 (3)	573 (4)	328 (5)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(30)	5553 (4)	4042 (3)	1962 (5)	H(C26)	357 (4)	590 (4)	140 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(31)	6195 (4)	3887 (4)	1367 (5)	H(C27)	401 (5)	542 (5)	15 (8)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(32)	6644 (4)	3292 (4)	1682 (6)	H(C32)	716 (4)	321 (4)	129 (6)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(33)	6485 (5)	2901 (4)	2565 (7)	H(C33)	675 (6)	247 (6)	278 (9)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(34)	5934 (4)	3167 (4)	3255 (6)	H(C34)	593 (4)	301 (4)	394 (6)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(35)	4855 (5)	4314 (5)	-254 (5)	H(C35)1	506 (4)	379 (4)	19 (5)
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	C(36)	6468 (4)	4325 (5)	458 (6)	H(C35)2	502 (4)	478 (4)	-97 (5)
$\begin{array}{c c c c c c c c c c c c c c c c c c c $	Cl(1)	 26491 (12) 	35948 (13)	93232 (20)	H(C35)3	445 (6)	424 (6)	-50 (9)
Cl(3) 67562 (14) 51275 (14) 74513 (18) H(C36)2 629 (6) 484 (5) 55 (8) O(1) 3075 (5) 2979 (6) 9231 (8) H(C36)3 702 (6) 427 (6) 38 (8) O(2) 1906 (4) 3471 (5) 9498 (11) 629 (6) 484 (5) 55 (8)	CI(2)	33815 (10)	70859 (10)	93122 (15)	H(C36)1	611 (4)	405 (4)	-18 (6)
O(1) 3075 (5) 2979 (6) 9231 (8) H(C36)3 702 (6) 427 (6) 38 (8) O(2) 1906 (4) 3471 (5) 9498 (11) 702 (6) 427 (6) 38 (8)	Cl(3)	67562 (14)	51275 (14)	74513 (18)	H(C36)2	629 (6)	484 (5)	55 (8)
O(2) 1906 (4) 3471 (5) 9498 (11)	O(1)	3075 (5)	2979 (6)	9231 (8)	H(C36)3	702 (6)	427 (6)	38 (8)
	O(2)	1906 (4)	3471 (5)	9498 (11)				

averaged for the two equivalent reflections (*hkl* and $h\bar{k}l$). The concordance in Table 2 indicated that $(-)_{589}$ -[Rh(dmbpy)₃]³⁺ has the absolute configuration Λ (IUPAC, 1970) in agreement with the assignment based on the sign of the circular dichroism of the π - π * exciton bands ($\nu = 29.85 \times 10^2$ mm⁻¹, $\Delta \varepsilon = +19.7$ and $\nu = 32.68 \times 10^2$ mm⁻¹, $\Delta \varepsilon = -35.6$: Suzuki, 1979).

Discussion. Fig. 1 shows a projection of the structure along c. A perspective drawing of the complex cation is given in Fig. 2, where the ion is viewed along the normal to the plane through the midpoints of the line

joining the two N atoms of each ligand. Hereafter this line will be called the axis of the complex ion. The absolute configuration of the present complex ion is $\Lambda(\delta\lambda\lambda)$. Two of the C-C bonds in the chelate rings are inclined at a mean angle of 54.8° to the axis of the complex ion, while the remaining bond is at an angle of 25.2°. Thus the complex ion takes the 'ob₂ lel' conformation. The inclination of the C-C bond in the lel ring is greater by about 10° than those of [Co(diamine)₂(dmbpy)]³⁺ complexes (Sato & Saito, 1978; Ohba, Sato & Saito, 1979). The ion has an approximate twofold symmetry axis through the Rh and the midpoint of the C-C bond in the δ chelate ring.

					$ F_o(hkl) $	$ F_c(hkl) $
h	k	l	$ F_o $	$ F_c $	$ F_o(\bar{h}kl) $	$\overline{F_c(hkl)}$
5	4	1	12.9	12.8	1.48	1.51
5	4	1	8.7	8.5		
12	6	1	37.9	36.7	1.08	1.08
12	6	1	35.0	34.1		
1	7	1	38.0	36.1	1.09	1.09
ĩ	7	1	34.9	33.1		
8	14	1	18.0	17.6	1.08	1.07
8	14	1	16.6	16.4		
5	14	3	20.8	20.6	1.11	1.09
5	14	3	18.7	18.9		-
7	2	4	14.3	14.7	0.85	0.86
Ż	2	4	16.9	17.1		
5	1	10	13.8	14.7	0.85	0.87
5	1	10	16-2	16.9		
6	2	11	16.2	17.5	1.11	1.11
ō	2	11	14.6	15.8		



Fig. 3. Mean bond lengths (Å) and angles (°) within the complex ion. The upper values refer to the δ chelate ring and the lower to the λ ring.



Fig. 4. Conformations of the chelate rings. Figures next to each C atom indicate the deviations (Å) from the coordination plane; the average e.s.d. is 0.01 Å.

do not occur in the complex ion, except for those between the methyl groups.

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Fig. 2. A perspective drawing of the complex ion, $(-)_{589}[Rh(dmbpy)_3]^{3+}$.

Mean values of the interatomic distances and bond angles within the chelate rings are indicated in Fig. 3. There are no significant differences between the δ and λ chelate rings. The average values are: Rh–N 2.039 (5), N–C 1.368 (8), C–C 1.470 (9) Å; N–Rh–N 80.4 (2), Rh–N–C 113.1 (4), N–C–C 113.3 (6)°. Each chelate ring takes an unsymmetrical skew conformation as shown in Fig. 4. The dihedral angles about the C–C bond of the chelate rings are 25.0 (7) and 31.9 (8)° for the δ and λ rings respectively.

Two pyridine moieties of dmbpy are twisted owing to the interaction between bulky methyl groups, and are inclined at mean angles of 15.8 and 21.0° to the coordination plane, for δ -dmbpy and λ -dmbpy respectively. The twist angles of both pyridine rings for the δ and λ chelate rings are 29.0 and 36.7° respectively. Accordingly, the C...C distance between the two methyl groups is 0.11 Å longer in the λ ring than the value of 2.989 (11) Å in the δ ring. The mean tilt angle of the C--CH₃ bond to the appropriate pyridine ring is 10.5°. Non-bonded H...H contacts less than 3.00 Å