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Hydrotris(triphenylphosphine)platinum(II) Hydrogenbis(trifluoroacetate)

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Abstract. $[PtH(PPh_3)_3](CF_3CO_2)_2H$, triclinic, $P\bar{1}$, $a = 13{\cdot}104\,(7)$, $b = 13{\cdot}134\,(7)$, $c = 16{\cdot}132\,(9)$ Å, $\alpha = 91{\cdot}07\,(2)$, $\beta = 100{\cdot}05\,(3)$, $\gamma = 102{\cdot}02\,(2)°$, $Z = 2$, $\rho_c = 1{\cdot}51$ g cm^{-3}. The distances P(1)—Pt, P(2)—Pt, and P(3)—Pt are 2·315 (7), 2·363 (7) and 2·309 (7) Å, and the bond angles P(3)—Pt—P(2) and P(1)—Pt—P(2) are 99·7 (2) and 100·6 (2)° respectively, while the angle between the *trans* P ligands, P(1)—Pt—P(3), is 159·6 (2)°. The proton was not located but its position was inferred from the geometry around the Pt atoms. The distorted square-planar cation does not interact with the hydrogenbis(trifluoroacetate) anion.

Introduction. The ^{1}H NMR spectra of salts of the $[PtH(PPh_3)_3]^+$ cation have been interpreted in terms of second-order effects causing the anticipated double triplet splitting to be observed as an apparent pair of double doublets (Thomas, Dumler, Renoe, Nyman & Roundhill, 1972; Dingle & Dixon, 1974). An alternative explanation has been offered, namely that ion pairing may occur, leading to a stereochemical distortion of the square-planar structure, thereby causing the mutually *trans* triphenylphosphines to become non-equivalent (Bird, Harrod & Than, 1974). The single-crystal structure determination of the title compound was carried out to investigate whether any of these triphenylphosphines showed a significant deviation from planarity in the solid state. A triclinic crystal with dimensions 0·47 × 0·30 × 0·06 mm was used. A total of 5549 reflections were collected with a θ–2θ step scan (20 steps per degree, 2·2° scan) on an automated four-circle Picker diffractometer out to $2\theta = 45°$, with Mo $K\alpha$ radiation and a Zr filter. The structure was solved with the heavy-atom technique and was refined by full-matrix least-squares calculations with anisotropic thermal parameters for the Pt and P atoms. During data collection, the intensities of standard reflections decreased exponentially with time to 88% of the initial intensities as a result of radiation damage. All data were corrected for this effect. Corrections were made for absorption ($\mu = 2{\cdot}91$ mm^{-1}) with transmission factors ranging from 0·84 to 0·92. Refinement was carried out for 5549 reflections. The R value decreased to 0·114 ($R = \Sigma|\Delta F|/\Sigma|F_{\rm obs.}|$). At this point, it was clear that the F atoms were disordered and undergoing large torsional oscillation, and it was decided that the cost of further structural analysis to sort out the disorder/thermal motion interactions was not justified. The final positional and thermal parameters are shown in Table 1.*

Discussion. Interatomic distances and bond angles are listed in Table 2, and an *ORTEP* (Johnson, 1965) drawing of the cation is shown in Fig. 1. This figure shows that the cation adopts a distorted square-planar geometrical arrangement with the mutually *trans* triphenylphosphines in equivalent positions. There is no interaction between cation and anion. The bond angles are 99·7 and 100·6° for P—Pt—P. The H atom on Pt has not been located but the third angle of 159·6° about the Pt center confirms the planarity of the cation. The large cone angle of triphenylphosphine causes considerable in-plane distortion from a square geometry; by comparison, the P—Pt—Cl and P—Pt—Br angles in $PtHCl(PPh_2Et)_2$ and $PtHBr(PEt_3)_2$ are close to 94° (Eisenberg & Ibers, 1965; Owston, Partridge & Rowe, 1960). Our structural data agree with the concept that the stereochemical influence of the hydride ligand causes the molecule to adopt a square-planar, albeit considerably distorted, geometry, rather than a trigonal

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32006 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final parameters for* $[PtH(PPh_3)_3](CF_3CO_2)_2H$

The standard deviation on the least significant digit is given in parentheses.

(*a*) Positional and anisotropic thermal parameters (× 10^4) for the Pt and P atoms

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Pt	889 (1)	2736 (1)	3171 (1)	29 (1)	53 (1)	26 (1)	12 (1)	1 (1)	2 (1)
P(1)	−539 (5)	1345 (5)	2840 (4)	53 (5)	56 (5)	24 (3)	25 (4)	4 (3)	15 (3)
P(2)	581 (5)	3763 (5)	2005 (4)	44 (5)	59 (5)	36 (4)	20 (4)	8 (4)	3 (3)
P(3)	2468 (5)	3757 (5)	3891 (4)	39 (5)	45 (5)	46 (4)	12 (4)	9 (4)	5 (4)

(*b*) Positional (× 10^3) and isotropic thermal parameters for the C, O, and F atoms

	x	y	z	B		x	y	z	B
C(11)	13 (2)	13 (2)	733 (2)	4·5 (6)	C(65)	160 (2)	429 (2)	866 (2)	4·9 (6)
C(12)	88 (2)	82 (2)	736 (2)	4·5 (6)	C(66)	75 (2)	486 (2)	867 (2)	5·1 (6)
C(13)	58 (2)	179 (2)	748 (2)	6·6 (7)	C(71)	237 (2)	487 (2)	443 (1)	2·5 (5)
C(14)	−51 (2)	175 (2)	753 (2)	4·3 (6)	C(72)	136 (2)	521 (2)	438 (2)	6·8 (7)
C(15)	−122 (2)	87 (2)	750 (1)	3·9 (6)	C(73)	124 (2)	617 (2)	474 (2)	6·5 (8)
C(16)	−95 (2)	14 (2)	739 (2)	5·0 (6)	C(74)	209 (2)	681 (2)	524 (2)	5·5 (7)
C(21)	163 (2)	875 (2)	805 (2)	3·3 (6)	C(75)	307 (3)	640 (2)	546 (2)	8·1 (8)
C(22)	228 (2)	795 (2)	799 (2)	5·5 (6)	C(76)	316 (2)	544 (2)	502 (1)	3·5 (5)
C(23)	314 (2)	797 (2)	867 (2)	5·5 (7)	C(81)	306 (2)	305 (2)	479 (2)	4·3 (6)
C(24)	328 (2)	857 (2)	937 (2)	4·7 (6)	C(82)	408 (2)	282 (2)	481 (2)	4·4 (6)
C(25)	261 (2)	927 (2)	951 (2)	6·6 (8)	C(83)	454 (2)	234 (2)	558 (2)	7·3 (8)
C(26)	177 (3)	945 (3)	874 (2)	4·9 (9)	C(84)	401 (2)	217 (2)	619 (2)	5·1 (6)
C(31)	119 (2)	890 (2)	626 (2)	5·2 (7)	C(85)	306 (3)	240 (2)	624 (2)	8·0 (8)
C(32)	231 (2)	940 (2)	636 (2)	5·1 (6)	C(86)	256 (2)	299 (2)	548 (2)	5·1 (6)
C(33)	278 (2)	963 (2)	564 (2)	5·1 (6)	C(91)	347 (2)	403 (2)	326 (1)	2·9 (5)
C(34)	214 (2)	951 (2)	489 (2)	6·6 (7)	C(92)	415 (2)	511 (2)	326 (2)	3·8 (6)
C(35)	114 (2)	912 (2)	479 (2)	5·5 (7)	C(93)	495 (2)	520 (2)	276 (2)	5·4 (6)
C(36)	62 (2)	890 (2)	544 (2)	5·3 (6)	C(94)	501 (2)	443 (2)	219 (2)	5·2 (6)
C(41)	162 (2)	483 (2)	183 (1)	3·2 (5)	C(95)	434 (2)	337 (2)	220 (2)	5·9 (7)
C(42)	215 (2)	471 (2)	112 (2)	7·0 (8)	C(96)	356 (2)	324 (2)	271 (2)	4·3 (6)
C(43)	291 (2)	565 (2)	91 (2)	5·4 (6)	C(10)	397 (2)	219 (2)	891 (2)	7·1 (7)
C(44)	306 (2)	648 (2)	146 (2)	5·8 (7)	C(1F)	402 (3)	272 (3)	970 (2)	12·1 (9)
C(45)	259 (2)	667 (2)	212 (2)	5·2 (6)	O(11)	339 (2)	232 (2)	828 (1)	10·5 (6)
C(46)	185 (2)	578 (2)	227 (2)	4·6 (6)	O(12)	458 (2)	154 (2)	892 (1)	7·7 (5)
C(51)	25 (2)	291 (2)	107 (2)	4·8 (6)	F(11)	395 (2)	388 (2)	948 (2)	9·0 (7)
C(52)	93 (2)	211 (2)	106 (1)	3·6 (5)	F(12)	321 (2)	283 (2)	991 (2)	16·2 (7)
C(53)	83 (2)	148 (2)	37 (2)	5·2 (6)	F(13)	492 (2)	315 (2)	1016 (1)	12·3 (6)
C(54)	−1 (3)	153 (2)	967 (2)	8·5 (8)	C(20)	553 (3)	27 (3)	761 (2)	11·9 (9)
C(55)	−71 (2)	299 (2)	959 (2)	8·2 (8)	C(2F)	557 (4)	−61 (4)	716 (3)	18·7 (12)
C(56)	−53 (2)	299 (2)	33 (2)	4·5 (6)	O(21)	633 (2)	68 (2)	821 (2)	16·8 (7)
C(61)	54 (2)	551 (2)	801 (1)	2·8 (5)	O(22)	470 (2)	48 (2)	777 (1)	9·5 (6)
C(62)	105 (2)	567 (2)	732 (2)	4·6 (6)	F(21)	476 (2)	−130 (2)	686 (1)	12·7 (6)
C(63)	188 (2)	508 (2)	732 (1)	4·1 (5)	F(22)	642 (2)	−84 (2)	710 (1)	14·7 (6)
C(64)	210 (2)	444 (2)	795 (2)	6·2 (7)	F(23)	558 (2)	4 (2)	634 (1)	14·2 (6)

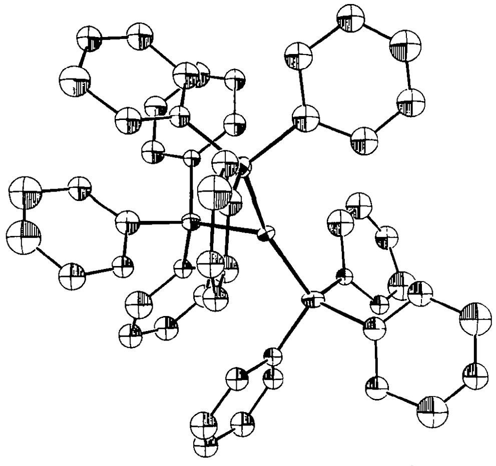

Fig. 1. *ORTEP* representation of the cation in $[PtH(PPh_3)_3]^+[(CF_3CO_2)_2H]^-$.

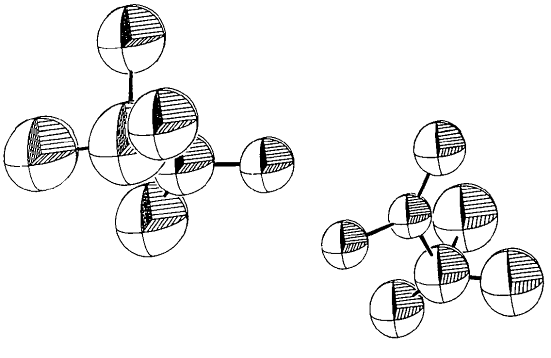

Fig. 2. *ORTEP* representation of the anion in $[PtH(PPh_3)_3]^+[(CF_3CO_2)_2H]^-$.

Table 2. *Bond distances* (Å) *and angles* (°) *in* $[PtH(PPh_3)_3](CF_3CO_2)_2H$

The standard deviation on the least significant digit is given in parentheses.

Pt coordination sphere			
Pt—P(1)	2·315 (6)	P(1)—Pt—P(2)	100·6 (2)
Pt—P(2)	2·363 (6)	P(2)—Pt—P(3)	99·6 (2)
Pt—P(3)	2·309 (6)	P(3)—Pt—P(1)	159·6 (2)
Phenyl ring 1			
C(11)—P(1)	1·80 (2)	C(11)—P(1)—Pt	111·8 (8)
C(12)—C(11)	1·41 (3)	C(11)—C(12)—C(13)	120 (2)
C(13)—C(12)	1·43 (3)	C(12)—C(13)—C(14)	117 (2)
C(14)—C(13)	1·43 (3)	C(13)—C(14)—C(15)	123 (2)
C(15)—C(14)	1·31 (2)	C(14)—C(15)—C(16)	122 (2)
C(16)—C(11)	1·44 (3)	C(15)—C(16)—C(11)	116 (2)
C(16)—C(15)	1·44 (3)	C(16)—C(11)—C(12)	120 (2)
Phenyl ring 2			
C(21)—P(1)	1·82 (2)	C(21)—P(1)—Pt	125·2 (8)
C(22)—C(21)	1·49 (3)	C(21)—C(22)—C(23)	116 (2)
C(23)—C(22)	1·42 (3)	C(22)—C(23)—C(24)	121 (2)
C(24)—C(23)	1·31 (3)	C(23)—C(24)—C(25)	124 (2)
C(25)—C(24)	1·43 (3)	C(24)—C(25)—C(26)	117 (2)
C(26)—C(21)	1·38 (3)	C(25)—C(26)—C(21)	114 (2)
C(26)—C(25)	1·56 (4)	C(26)—C(21)—C(22)	124 (2)
Phenyl ring 3			
C(31)—P(1)	1·81 (2)	C(31)—P(1)—Pt	109·8 (9)
C(32)—C(31)	1·44 (3)	C(31)—C(32)—C(33)	119 (2)
C(33)—C(32)	1·42 (3)	C(32)—C(33)—C(34)	117 (2)
C(34)—C(33)	1·33 (3)	C(33)—C(34)—C(35)	122 (2)
C(35)—C(34)	1·28 (3)	C(34)—C(35)—C(36)	123 (2)
C(36)—C(31)	1·40 (3)	C(35)—C(36)—C(31)	119 (2)
C(36)—C(35)	1·34 (3)	C(36)—C(31)—C(32)	114 (2)
Phenyl ring 4			
C(41)—P(2)	1·80 (2)	C(41)—P(2)—Pt	119·1 (7)
C(42)—C(41)	1·45 (3)	C(41)—C(42)—C(43)	117 (2)
C(43)—C(42)	1·50 (3)	C(42)—C(43)—C(44)	113 (2)
C(44)—C(43)	1·35 (3)	C(43)—C(44)—C(45)	132 (2)
C(45)—C(44)	1·36 (3)	C(44)—C(45)—C(46)	111 (2)
C(46)—C(41)	1·37 (2)	C(45)—C(46)—C(41)	125 (2)
C(46)—C(45)	1·41 (3)	C(46)—C(41)—C(42)	119 (2)
Phenyl ring 5			
C(51)—P(2)	1·79 (2)	C(51)—P(2)—Pt	107·3 (8)
C(52)—C(51)	1·50 (3)	C(51)—C(52)—C(53)	121 (2)
C(53)—C(52)	1·34 (3)	C(52)—C(53)—C(54)	117 (2)
C(54)—C(53)	1·45 (3)	C(53)—C(54)—C(55)	126 (2)
C(55)—C(54)	1·48 (3)	C(54)—C(55)—C(56)	113 (2)
C(56)—C(51)	1·45 (3)	C(55)—C(56)—C(51)	121 (2)
C(56)—C(55)	1·44 (3)	C(56)—C(51)—C(52)	119 (2)
Phenyl ring 6			
C(61)—P(2)	1·90 (2)	C(61)—P(2)—Pt	116·2 (7)
C(62)—C(61)	1·39 (3)	C(61)—C(62)—C(63)	113 (2)
C(63)—C(62)	1·46 (3)	C(62)—C(63)—C(64)	120 (2)
C(64)—C(63)	1·36 (3)	C(63)—C(64)—C(65)	125 (2)
C(65)—C(64)	1·40 (3)	C(64)—C(65)—C(66)	115 (2)
C(66)—C(61)	1·40 (3)	C(65)—C(66)—C(61)	118 (2)
C(66)—C(65)	1·46 (3)	C(66)—C(61)—C(62)	126 (2)
Phenyl ring 7			
C(71)—P(3)	1·72 (2)	C(71)—P(3)—Pt	116·4 (7)
C(72)—C(71)	1·46 (3)	C(71)—C(72)—C(73)	125 (2)
C(73)—C(72)	1·42 (3)	C(72)—C(73)—C)74)	119 (2)
C(74)—C(73)	1·37 (3)	C(73)—C(74)—C(75)	117 (2)
C(75)—C(74)	1·47 (3)	C(74)—C(75)—C(76)	119 (2)
C(76)—C(71)	1·35 (2)	C(75)—C(76)—C(71)	123 (2)
C(76)—C(75)	1·47 (3)	C(76)—C(71)—C(72)	112 (2)
Phenyl ring 8			
C(81)—P(3)	1·88 (2)	C(81)—P(3)—Pt	110·4 (8)
C(82)—C(81)	1·42 (3)	C(81)—C(82)—C(83)	116 (2)
C(83)—C(82)	1·50 (3)	C(82)—C(83)—C(84)	119 (2)
C(84)—C(83)	1·30 (3)	C(83)—C(84)—C(85)	127 (2)
C(85)—C(84)	1·34 (3)	C(84)—C(85)—C(86)	116 (2)
C(86)—C(81)	1·37 (3)	C(85)—C(86)—C(81)	115 (2)
C(86)—C(85)	1·57 (3)	C(86)—C(81)—C(82)	123 (2)
Phenyl ring 9			
C(91)—P(3)	1·77 (2)	C(91)—P(3)—Pt	112·7 (7)
C(92)—C(91)	1·50 (2)	C(91)—C(92)—C(93)	115 (2)
C(93)—C(92)	1·41 (3)	C(92)—C(93)—C(94)	123 (2)
C(94)—C(93)	1·37 (3)	C(93)—C(94)—C(95)	119 (2)
C(95)—C(94)	1·48 (3)	C(94)—C(95)—C(96)	117 (2)
C(96)—C(91)	1·39 (2)	C(95)—C(96)—C(91)	123 (2)
C(96)—C(95)	1·39 (3)	C(96)—C(91)—C(92)	119 (2)
Acetate ion 1			
C(1F)—C(10)	1·42 (3)	O(11)—C(10)—O(12)	122 (3)
O(11)—C(10)	1·19 (3)	O(12)—C(10)—C(1F)	114 (3)
O(12)—C(10)	1·28 (3)	C(1F)—C(10)—O(11)	122 (3)
F(11)—C(1F)	1·59 (3)	F(11)—C(1F)—F(12)	75 (2)
F(12)—C(1F)	1·20 (3)	F(12)—C(1F)—F(13)	119 (3)
F(13)—C(1F)	1·28 (3)	F(13)—C(1F)—C(10)	120 (3)
		C(10)—C(1F)—F(11)	104 (2)
Acetate ion 2			
C(2F)—C(20)	1·36 (4)	O(21)—C(20)—O(22)	109 (3)
O(21)—C(20)	1·31 (3)	O(21)—C(20)—C(2F)	119 (4)
O(22)—C(20)	1·25 (3)	O(22)—C(20)—C(2F)	124 (4)
F(21)—C(2F)	1·25 (4)	F(21)—C(2F)—F(22)	114 (4)
F(22)—C(2F)	1·23 (4)	F(22)—C(2F)—F(23)	92 (3)
F(23)—C(2F)	1·57 (4)	F(23)—C(2F)—C(20)	89 (3)
		C(20)—C(2F)—F(21)	123 (4)

one (Frenz & Ibers, 1971). The distances for P(1)—Pt, P(2)—Pt, and P(3)—Pt are 2·315, 2·363, and 2·309 Å respectively, indicative of equivalent positions for P(1) and P(3), and the high *trans* influence of the hydride ligand.

The structure of the anion is consistent with its formulation as a monohydrogen-bridged acetate dimer, $(CF_3CO_2)_2H$. The short O—O distance between the two halves of the dimer, O(12)—O(22), is 2·35 (3) Å, as expected for a strong O—H—O hydrogen bond. The trifluoro moieties of the anion are disordered and undergoing large torsional oscillations, and accurate positions cannot be assigned for the fluorines without further Fourier and least-squares analyses. Structures for the K, Rb and Cs salts of $(CF_3CO_2)_2H^-$ have been reported previously (Golič & Speakman, 1965), and there appear to be no significant differences in the structure of the anion in the $[PtH(PPh_3)_3]^+$ salt. A view of the anion is shown in Fig. 2.

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Bis(bis-η-cyclopentadienylmolybdenumdi-μ-oxo)phosphorus Hexafluorophosphate

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Abstract. $[(C_2H_5)_2MoO_2Mo(C_2H_5)_2](PF_6)_2$, $C_{20}H_{20}O_4F_{12}P_3Mo_2$, $M_r = 837{\cdot}2$. Monoclinic, $C2/c$, $a = 17{\cdot}079\,(9)$, $b = 23{\cdot}619\,(13)$, $c = 12{\cdot}399\,(7)$ Å, $\beta = 117{\cdot}02\,(2)°$, $U = 4455{\cdot}7$ Å^3, $D_c = 2{\cdot}50$ g cm^{-3} for $Z = 8$. Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 14$ cm^{-1}. The structure of the cation is as would be expected from the known structures of bent $d^2(\eta\text{-}C_5H_5)_2MX_2$ systems. The anions have near-spherical symmetry and show considerable apparent distortion.

Introduction. Needle crystals from the reaction of $(\eta\text{-}C_5H_5)_2MoCl_2$ and Na_2HPO_4 in an aqueous medium were supplied by Dr M. L. H. Green and Mr C. Ram̂ao. They were sensitive to air and moisture and were mounted for X-ray examination in nitrogen-filled capillary tubes. It was not possible to measure their density. After survey photography by oscillation and Weissenberg techniques, the selected crystal was set up on a Hilger & Watts *PDP8*-controlled four-circle diffractometer and cell parameters and orientation matrix were obtained by a least-squares fit to the setting angles of 20 reflexions.

The intensities of each independent reflexion with $\sin\theta/\lambda < 0{\cdot}59$ were measured by an $\omega/2\theta$ scan with ordinate analysis (Watson, Shotton, Cox & Muirhead, 1970). Mo $K\alpha$ radiation was used with a graphite monochromator. Reflexions with $I < 4\sigma(I)$, where $\sigma(I)$ is the standard deviation derived from counting statistics, or whose apparent centre was more than 0·14° from the predicted position, were not included in subsequent calculations. Lorentz and polarization corrections, and an empirical absorption correction (North, Phillips & Mathews, 1968), were applied, leading to a set of 3495 independent observed structure amplitudes.

The structure was solved by Patterson and Fourier techniques. Refinement was by least squares with a large-block approximation to the normal matrix: one block was calculated from the derivatives of the positional parameters, and one from those of the corresponding anisotropic temperature factors and the scale factor. Difference syntheses permitted the location of the H atoms in their expected positions. They were positioned geometrically (C–H = 1·0 Å; $U_{\rm iso} = 0{\cdot}05$) and included in the structure factor calculations, being readjusted after each cycle. Four low-angle reflexions, 150, $\bar{3}11$, 221, $\bar{1}32$, were given zero weight in the last cycles because of suspected extinction effects. Each reflexion was assigned a weight according to $\omega = 1/\Sigma_{r=1}^{n} A_r T_r^*(X)$ where n is the number of coefficients, A_r, for a Chebyshev series; T_r^* is the polynomial function and X is $F_o/F_o(\max)$. Five coefficients, A_r, were used with values 145·74, 193·82, 94·01, 40·75 and 18·23 (Rollett, 1965) and the final R was 0·041 for 3491 reflexions. All calculations were

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