$PR_3 = PMe_3$ (Jones, Kelly, Warrens, Williams & Woollins, 1986) continuous stacks also exist [interplanar (PtS_2N_2) separation 3·90 Å]. In (3) ($PR_3 = PMe_2Ph$) the cations stack as 'dimer pairs' (intradimer separation 3·58 Å, interdimer separation 5·00 Å). In (1) an intermediate situation is observed with an intradimer separation of 4·06 (2) Å [shortest contact N(2)···N(1′) 4·11 Å] and an interdimer separation of 4·22 (2) Å [shortest contact N(2)···S(2″) 4·38 Å]. The vector normal to the stacking planes is inclined by 18° to the crystallographic **a** direction (Fig. 2*a*). As expected, the separation between adjacent cations is dependent, in part, upon the relative bulk of the phosphine ligand. We believe a significant factor determining the stacking geometries in these systems is the presence of counterions, *i.e.* ionic species favour stacking (Jones *et al.*, 1988).

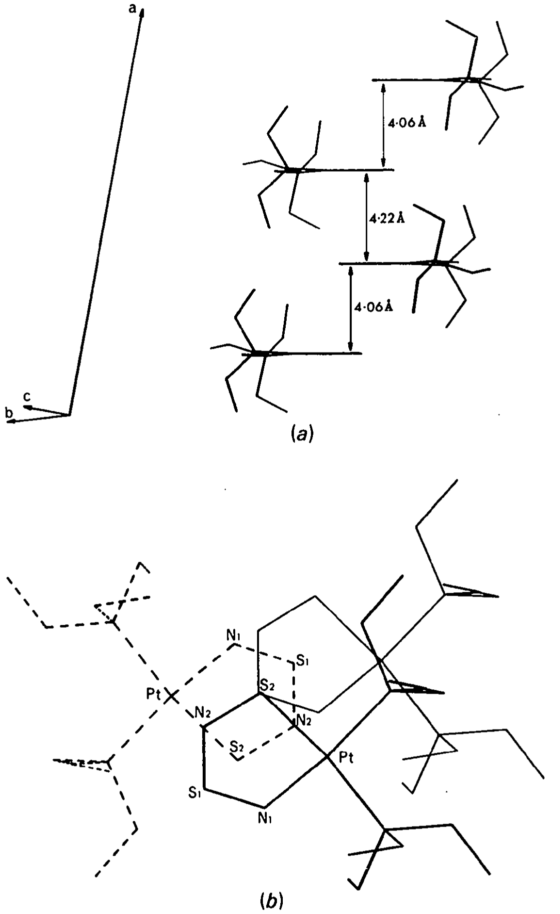

Fig. 2. View of the packing of the stacks of the cations in (1) (*a*) normal to the stacking direction and (*b*) viewed down the stacking axis (normal to the PtS_2N_2 planes).

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Structure of $[Ir(2,3,5\text{-}\eta\text{-}C_{10}H_{13})(\eta\text{-}C_5Me_5)]PF_6$ at 135 K: a Complex Containing a Strong Ir—H—C Interaction

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Abstract. (η-Pentamethylcyclopentadienyl)[(4–5-η, κC^8)-tricyclo[5.2.1.0^{2,6}]dec-4-en-8-yl)iridium(II) hexafluorophosphate, $C_{20}H_{28}Ir^+.PF_6^-$, $M_r = 605·6$, orthorhombic, $Pna2_1$, $a = 16·674$ (5), $b = 10·084$ (3), $c = 12·109$ (3) Å, $U = 2036·0$ (10) Å^3, $Z = 4$, $D_x = 1·98$ g cm^{-3} [D_m, D_x at 294 K 1·87 (1), 1·90 g cm^{-3}], Mo $K\bar{\alpha}$, $\lambda = 0·71069$ Å, $\mu = 66·3$ cm^{-1}, $F(000) = 1176$, $T = 135$ (2) K, $R = 0·030$, $wR = 0·034$ for 4032 unique reflections [$I \geq 3\sigma(I)$]. The cation is comprised of an (η^5-pentamethylcyclopentadienyl)iridium(III) fragment bound to mono-protonated dicyclopentadiene by an η^2 olefin bond to the cyclopentene fragment [Ir—C 2·11 (2), 2·16 (1) Å], a σ bond to one norbornane C atom [Ir—C 2·12 (1) Å] and a two-electron-

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three-centre C—H—*M* bond to the second norbornane C atom [Ir—C 2·30 (1) Å]. For C—H = 0·95 Å and tetrahedral carbon the calculated Ir—H distance is 1·79 Å [1·75 Å for C—H = 1·08 Å].

Introduction. The structure of the title complex was determined in the course of a series of studies of transition-metal complexes containing C—H···*M* bridges (Bennett, McMahon, Pelling, Robertson & Wickramasinghe, 1985). Complexes of this type constitute model intermediates in C—H activation reactions (Muetterties, 1982) and have been reviewed recently by Brookhart & Green (1983) and by Crabtree, Holt, Lavin & Morehouse (1985). Because crystals showed promise of being suitable for neutron diffraction analysis, and with a view to improving H-atom definition, data were collected at 135 K.

Experimental. Yellow crystals from dichloromethane/ether. D_m by flotation in aq. $ZnBr_2$. Sample crystal 0·18 × 0·07 × 0·08 mm perpendicular to bounding forms {001}, {110} and {$\bar{1}$10}; Philips PW 1100/20 diffractometer, θ–2θ continuous-scan mode [2° min^{-1} 2θ, 2 × 10 s background counts at extremes, $4 < 2\theta < 60°$, Mo $K\bar{\alpha}$, graphite-crystal monochromator, forms recorded $hk\pm l$, $23 \geq h \geq 0$, $14 \geq k \geq 0$, $17 \geq l \geq -17$, 6769 reflections including standards (3 every 100 min, 10,0,0, 060, 008, 6% max. degradation), $T = 135$ (2) K]; data corrected for absorption (de Meulenaer & Tompa, 1965; Alcock, 1969) [transmission factors: 0·68 max., 0·53 min.] and for crystal degradation (rate = $0\cdot72 \times 10^{-5}$ per measured reflection; three standards average); sorting and averaging (point group *mm*2) yielded 4044 reflections with $I \geq 3\sigma(I)$; R_s [$= \sum\sigma(F_o)/\sum|F_o|$] for this data set = 0·029; $R_{\rm int}$ for 196 pairs = 0·023; cell dimensions from 25 well dispersed reflections with $20 < 2\theta < 28°$ [Mo $K\alpha_1$ radiation, $T = 135$ (2) K]; structure solved by conventional Patterson and Fourier techniques; full-matrix least-squares refinement on F with weights $w = [\sigma^2(F_o) + 0\cdot0005|F_o|^2]^{-1}$ (Busing & Levy, 1957; Corfield, Doedens & Ibers, 1967); initial anisotropic refinement (253 parameters, H by calculation, Me H atoms omitted) gave unacceptable ligand and anion geometry consistent with a false minimum (Rae, 1974); subsequent refinement with *RAELS*80 (Rae, 1976) and firm and slack geometric constraints [firm constraints: Cp ring planar, PF_6 regular octahedral (P—F refineable), C—H 0·95 Å, Me H atoms perpendicular away from metal atom; slack constraints: C_5Me_5 C—C(ring) 1·42, C—C(Me) 1·51 Å, C—C—C(ring) 108, C—C—C(Me) 126°; $C_{10}H_{13}$ C—C 1·52, C=C 1·37 Å, no angle constraints; $K(\partial s/\partial p_i)^2/a_{ii}$ initial 10, final 2]; all H atoms by calculation ($B_H = B_C$ or *TLX*); individual anisotropic thermal parameters for Ir and $C_{10}H_{13}$ C atoms, rigid-body (*TLX*) parameters for C_5Me_5 and PF_6; no extinction correction, 167 parameters, $R = 0\cdot030$, $wR = 0\cdot034$,* $S = 1\cdot0$, $-0\cdot6 < (\rho_o - \rho_c) < 1\cdot3$ e Å^{-3} (near Ir atom), max. $\Delta/\sigma = 0\cdot32$; scattering factors with dispersion corrections for all non-H atoms from *International Tables for X-ray Crystallography* (1974); calculations performed with *RAELS*80 (Rae, 1976) and *ANUCRYS* programs (McLaughlin, Taylor & Whimp, 1977) and the Australian National University Univac 1100/82 computer.

Discussion. Atomic coordinates are listed in Table 1† and selected bond lengths and bond angles in Table 2. The atom nomenclature is defined in Fig. 1(*ORTEP*II; Johnson, 1976).

As shown in Fig. 1, the cation consists of an Ir atom bound to η^5-pentamethylcyclopentadienyl and also to a protonated dicyclopentadiene fragment attached *via* an η^2 olefin bond from C(1)—C(2), a σ bond from C(5) and a three-centre-two-electron bond involving C(6) and H(6*B*). Dimensions of the dicp–Ir$^{\rm III}$ fragment are very similar to those found in the osmium(II) analogue $[Os(2,3,5\text{-}\eta\text{-}C_{10}H_{13})(\eta\text{-}C_6H_3Me_3)]PF_6$ (Bennett *et al.*, 1985). Metal–arene distances in both complexes are also very similar [Ir—C_5 = 2·15 (1)–2·26 (1) Å, Os—C_6 = 2·19 (1)–2·29 (2) Å]. All methyl groups in the C_5Me_5 group are bent away from the Ir atom. Perpendicular distances from the C_5 ring plane are C(111) 0·15 (2), C(121) 0·07 (1), C(131) 0·08 (2), C(141) 0·03 (1) and C(151) 0·25 (1) Å.

The packing arrangement is strongly pseudo-centrosymmetric (*Pna*2$_1$ pseudo *Pnam*) and initial unconstrained refinement yielded unacceptable bond distances and angles. The pseudo-mirror plane 'contains' atoms Ir and C(3) of the Ir–dicp fragment, C(15) and C(151) of the C_5Me_5 group and P, F(2) and F(4) of the anion. It relates, pairwise, C(1) to C(5), all other (non-H) atoms of the C_5Me_5 group [*e.g.* C(12) to C(13)], and the remaining F atoms of the anion [F(1) to F(5), F(3) to F(6)] with correlation coefficients (magnitudes) in the range 0·7–0·9. In all, twenty-one of the twenty-eight non-H atoms are affected by the pseudo-symmetry.

Unconstrained refinement (253 parameters) with initial atom parameters from the converged constrained model (167 refined parameters) yields a formally superior scattering model [$R(wR)^{253} = 0\cdot029$ (0·033), $R(wR)^{167} = 0\cdot030$ (0·034); wR ratio = 1·022, $\mathcal{R}_{86,3875,0\cdot005} = 1\cdot015$ (Hamilton, 1965)] but appreciably higher standard error estimates [(2–5)×] both for correlated and for $z \simeq 0$ coordinates. Only two coordi-

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* The enantiomeric packing array has $wR = 0\cdot036$ and can be rejected (Hamilton, 1965) at the 99·5% confidence level.

† Lists of structure factor amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44955 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}(Å^2)
Ir	0·35315 (1)	0·55790 (2)	0·00000	0·017
C(1)	0·3799 (6)	0·6714 (12)	0·1408 (13)	0·045
C(2)	0·3295 (4)	0·7472 (7)	0·0773 (6)	0·029
C(3)	0·3748 (3)	0·8545 (6)	0·0155 (8)	0·030
C(4)	0·3721 (4)	0·8369 (7)	−0·1087 (7)	0·032
C(5)	0·3746 (5)	0·6889 (8)	−0·1338 (9)	0·022
C(6)	0·4583 (4)	0·6494 (7)	−0·0960 (7)	0·034
C(7)	0·4966 (5)	0·7809 (7)	−0·0624 (7)	0·045
C(8)	0·4573 (4)	0·8759 (9)	−0·1441 (8)	0·045
C(9)	0·4635 (5)	0·7338 (9)	0·1437 (8)	0·052
C(10)	0·4630 (4)	0·8309 (8)	0·0463 (7)	0·043
P	0·1529 (1)	0·0803 (1)	0·0025 (3)	0·026
F(1)	0·0881 (3)	0·0629 (3)	−0·0932 (4)	0·041
F(2)	0·1698 (1)	−0·0753 (2)	0·0004 (4)	0·051
F(3)	0·2177 (3)	0·0976 (3)	0·0982 (4)	0·056
F(4)	0·1360 (1)	0·2358 (2)	0·0046 (4)	0·050
F(5)	0·0847 (3)	0·0582 (3)	0·0929 (5)	0·045
F(6)	0·2211 (3)	0·1023 (3)	−0·0878 (4)	0·043
C(11)	0·3181 (3)	0·3772 (6)	−0·0927 (6)	0·029
C(12)	0·3574 (2)	0·3337 (3)	0·0047 (7)	0·027
C(13)	0·3138 (3)	0·3817 (5)	0·0967 (6)	0·025
C(14)	0·2472 (2)	0·4552 (4)	0·0563 (7)	0·024
C(15)	0·2498 (2)	0·4524 (4)	−0·0609 (7)	0·026
C(111)	0·3397 (8)	0·3375 (16)	−0·2091 (7)	0·058
C(121)	0·4303 (3)	0·2453 (6)	0·0095 (11)	0·051
C(131)	0·3311 (7)	0·3506 (15)	0·2162 (7)	0·046
C(141)	0·1835 (5)	0·5212 (9)	0·1256 (10)	0·045
C(151)	0·1837 (5)	0·4980 (10)	−0·1369 (9)	0·049

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Ir—C(1)	2·101 (16)	C(8)—C(4)	1·536 (7)
Ir—C(2)	2·163 (8)	C(9)—C(1)	1·530 (8)
Ir—C(5)	2·121 (10)	C(10)—C(3)	1·536 (6)
Ir—C(6)	2·296 (7)	C(10)—C(7)	1·516 (7)
Ir—C(11)	2·219 (6)	C(10)—C(9)	1·532 (7)
Ir—C(12)	2·263 (3)	C(11)—C(12)	1·419 (2)
Ir—C(13)	2·227 (6)	C(12)—C(13)	1·416 (2)
Ir—C(14)	2·158 (4)	C(13)—C(14)	1·421 (2)
Ir—C(15)	2·154 (4)	C(14)—C(15)	1·420 (2)
C(1)—C(2)	1·372 (7)	C(15)—C(11)	1·421 (6)
C(2)—C(3)	1·518 (6)	C(11)—C(111)	1·509 (3)
C(3)—C(4)	1·515 (6)	C(12)—C(121)	1·508 (3)
C(4)—C(5)	1·523 (6)	C(13)—C(131)	1·508 (3)
C(5)—C(6)	1·522 (6)	C(14)—C(141)	1·508 (3)
C(6)—C(7)	1·528 (6)	C(15)—C(151)	1·508 (3)
C(7)—C(8)	1·525 (7)	P—F	1·594 (1)
C(1)—Ir—C(2)	37·5 (2)	C(6)—C(7)—C(10)	111·4 (7)
C(1)—Ir—C(6)	91·7 (4)	C(10)—C(7)—C(8)	101·3 (6)
C(2)—Ir—C(5)	79·1 (3)	C(4)—C(8)—C(7)	93·2 (6)
C(5)—Ir—C(6)	40·1 (2)	C(1)—C(9)—C(10)	103·9 (7)
Ir—C(1)—C(2)	73·7 (7)	C(3)—C(10)—C(7)	101·2 (6)
Ir—C(1)—C(9)	115·8 (10)	C(3)—C(10)—C(9)	106·9 (6)
C(9)—C(1)—C(2)	110·0 (8)	C(9)—C(10)—C(7)	117·0 (7)
Ir—C(2)—C(1)	68·8 (7)	C(12)—C(11)—C(15)	108·0 (1)
Ir—C(2)—C(3)	108·9 (5)	C(12)—C(11)—C(111)	125·8 (1)
C(3)—C(2)—C(1)	111·6 (7)	C(15)—C(11)—C(111)	125·9 (1)
C(2)—C(3)—C(4)	113·0 (6)	C(11)—C(12)—C(13)	108·1 (1)
C(2)—C(3)—C(10)	104·3 (6)	C(11)—C(12)—C(121)	125·9 (1)
C(10)—C(3)—C(4)	104·6 (6)	C(13)—C(12)—C(121)	125·9 (1)
C(3)—C(4)—C(5)	108·2 (7)	C(12)—C(13)—C(14)	108·0 (1)
C(8)—C(4)—C(5)	99·8 (6)	C(14)—C(13)—C(131)	126·0 (1)
Ir—C(5)—C(4)	117·0 (6)	C(12)—C(13)—C(131)	125·8 (1)
Ir—C(5)—C(6)	76·2 (5)	C(13)—C(14)—C(15)	108·1 (1)
C(6)—C(5)—C(4)	102·8 (6)	C(13)—C(14)—C(141)	126·1 (1)
Ir—C(6)—C(5)	63·7 (5)	C(15)—C(14)—C(141)	125·9 (1)
Ir—C(6)—C(7)	122·3 (6)	C(14)—C(15)—C(11)	107·8 (1)
C(7)—C(6)—C(5)	103·7 (6)	C(11)—C(15)—C(151)	125·7 (1)
C(6)—C(7)—C(8)	101·1 (6)	C(14)—C(15)—C(151)	125·6 (1)

nates [C(3)z, F(1)x] and one bond length [C(3)—C(4)] are formally different ($\Delta/\sigma > 3$) from the constrained refinement values. A half-normal probability plot of coordinate differences (Abrahams & Keve, 1971) is systematically non-linear. Points with $\Delta/\sigma < 0{\cdot}5$ have near-unit slope whereas those with $\Delta/\sigma > 0{\cdot}5$ have a slope of *ca* 1·7. Points in the first category derive substantially from atoms not conforming to the pseudo-symmetry and those in the second category to 'conforming' atoms. For conforming atoms (principally PF_6, C_5Me_5), the standard error estimates for the coordinate differences are about 40% low. For 'non-conforming' atoms the standard error estimates are about right (and separate coordinate e.s.d.'s from constrained and unconstrained refinements are very similar). There is no clear evidence as to the source of the 40% underestimate for conforming atoms but the very much lower e.s.d.'s from constrained refinement must make that procedure suspect.

^{1}H and ^{13}C NMR evidence for the presence of the C—H—M bridge has been described elsewhere (Bennett *et al.*, 1985) together with the structure of [Os(2,3,5-η-$C_{10}H_{13}$)(η-$C_6H_3Me_3$-1,3,5)]PF_6, an osmium analogue of the title complex. As for the osmium complex, the approximate location of the bridging H atom can be inferred with some certainty, both from the bonding geometry about C(5), C(6) and from R-factor analysis. In summary, (i) C—C distances require both C(5) and C(6) to be sp^3 hybridized; (ii) the Ir—C(6) distance [2·29 (1) Å] is too great for a direct Ir—C σ bond but quite compatible with a C—H—Ir bridge; (iii)

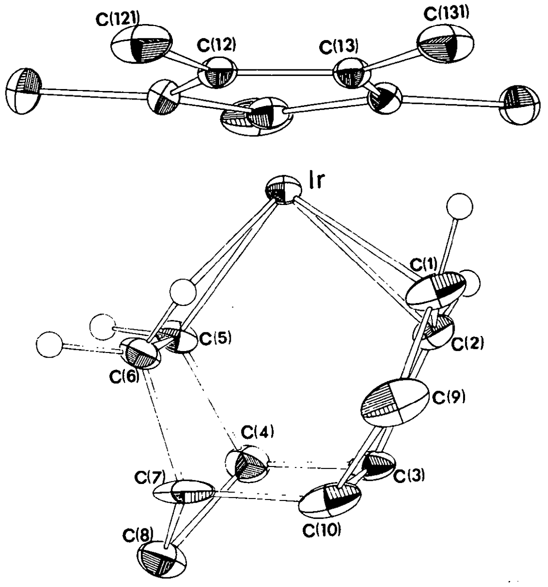

Fig. 1. Molecular structure and atom nomenclature for [Ir(2,3,5-η-$C_{10}H_{13}$)(η-C_5Me_5)]$^+$. 30% ellipsoids; H-atom numbering follows that of attached C atoms.

the Ir—H distance [*ca* 1·8 Å] to a tetrahedrally disposed *endo* H atom on C(6) [C—H = 0·95 Å] is directly comparable with those in the C—H—*M* bridges in [Fe(η^3-C_8H_{13}){P(OMe)$_3$}$_3$]BF_4 (Brown, Williams, Schultz, Stucky, Ittel & Harlow, 1980), in [Mn(η^3-C_6H_8Me)(CO)$_3$] (Schultz, Teller, Beno, Williams, Brookhart, Lamanna & Humphrey, 1983) and in [Fe_4(η^2-CH)(H)(CO)$_{12}$] (Beno, Williams, Tachikawa & Muetterties, 1980) [1·80–1·87 Å; all neutron diffraction studies]; and (iv) omission of the bridging H atom [H(6*B*)] from the scattering model (followed by reconvergence) results in a significant increase in *wR* [0·0339 to 0·0342; ratio significant at better than 99·5% confidence level (Hamilton, 1965)]. Omission of H(6*B*) from the scattering model also generates a well defined difference Fourier peak [$\Delta\rho \simeq 0\cdot5$ e Å^{-3}] 1·7 Å from Ir, 1·4 Å from C(6) and 0·5 Å from H(6*B*). The peak persists [$\Delta\rho \simeq 0\cdot4$ e Å^{-3}] to become the dominant feature of a $(\sin\theta)/\lambda < 0\cdot3$ Å^{-1} map, but is further displaced from the H(6*B*) site [$\Delta = 0\cdot6$ Å]. The displacement results largely from the persistence (in the difference maps) of the pseudo-mirror plane at $z = 0$. The calculated H(6*B*) sites lies just 0·42 Å from the $z = 0$ plane and the residual peak lies on that plane.

Although the exact location of the bridging H atom is unknown, the known geometry of C(6) dictates a strong interaction. Crabtree *et al.* (1985) have suggested $r_{bp} = d_{bp} - r_M$ as a measure of the interaction strength [d_{bp} is the metal to C—H bond-pair centroid distance and r_M is the metal-atom covalent radius]. For the title complex (with C—H = 1·08 Å), $d_{bp} \simeq 1\cdot9$ Å and $r_{bp} \simeq 0\cdot6$ Å. Both values are at the 'strong' end of the Crabtree *et al.* (1985) compilation. The C—H—*M* angle ($\simeq 106°$) is similar to those [96, 99°] in [Mo_2(η-C_5H_5)$_2$($C_{16}H_{25}$)]$^+$ (Green, Norman & Orpen, 1981) and [Fe_2(η-C_5H_5)$_2$(CO)(CH_3)($Ph_2PCH_2PPh_2$)]$^+$ (Dawkins, Green, Orpen & Stone, 1982), both of which exhibit similarly strong agostic interactions to β C—H groups [$r_{bp} = 0\cdot61$, 0·65 Å; Crabtree *et al.* (1985)] to that in the title complex.

Except for F(1)···H(6*A*) [2·5 Å] all ion···ion contacts are close to or exceed the normal van der Waals separations.

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Structure of Tris(diethyldithiophosphinato)chromium(III)

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Abstract. [Cr{$S_2P(C_2H_5)_2$}$_3$], $M_r = 511\cdot67$, triclinic, $P\bar{1}$, $a = 9\cdot638$ (1), $b = 10\cdot017$ (3), $c = 13\cdot983$ (3) Å, $\alpha = 86\cdot25$ (2), $\beta = 75\cdot15$ (1), $\gamma = 66\cdot95$ (1)°, $V = 1199\cdot8$ (2) Å^3, $Z = 2$, $D_m = 1\cdot39$ g cm^{-3}, $D_x = 1\cdot416$ (1) g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 11\cdot86$ cm^{-1}, $F(000) = 534$, $T = 298$ K, 5795 X-ray

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