# Chemistry of Platinum Sulphido-complexes Part 5.[1] Synthesis and Crystal and Molecular Structure† of 3-(η-Cyclo-octa-1,5-diene)bis($\mu_3$-sulphido)-1,1,2,2-tetrakis(triphenylphosphine)diplatinum(II)rhodium(I) Hexafluorophosphate–Dichloromethane (1/1), $[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(\eta\text{-}C_8H_{12})]PF_6\cdot CH_2Cl_2$

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The reaction between $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ and $[\{RhCl(cod)\}_2]$ (cod = cyclo-octa-1,5-diene) gives $[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]Y$ (Y = Cl, $PF_6$, or $BPh_4$). The molecular structure of $[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]PF_6\cdot CH_2Cl_2$ has been determined by single-crystal *X*-ray techniques using diffractometer data. The compound crystallises in the monoclinic space group $P2_1/c$ with four formula units in a cell of dimensions $a$ = 16.681(2), $b$ = 17.181(5), $c$ = 27.476(2) Å, and $\beta$ = 103.58(4)°. Least-squares refinement of the structure has led to a final $R$ value of 0.043 using 6 349 observed intensities [$I \geqslant 3\sigma(I)$]. The structure shows an approximately equilateral triangular arrangement of non-bonded metal atoms. The geometries at individual metal sites are approximately square planar.

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In recent years considerable interest has been shown in the synthesis of heterometallic complexes of the transition metals, because of their potential in catalytic processes requiring multiple metal sites.[2] Rational syntheses of cluster compounds of this type have been developed by Vahrenkamp,[3] Pringle and Shaw,[4] and Stone.[5] However, many of these compounds are co-ordinatively saturated by virtue of the metal–metal bonds which they form. We have defined an alternative synthetic goal, namely the preparation of aggregates of co-ordinatively unsaturated complexes which have several metal atoms in close proximity, but do not have strong metal–metal bonds and thus retain their unsaturated character. The term aggregate has been specifically chosen to distinguish these compounds from heterometallic clusters where the metal–metal bonding is an important feature.

The compound $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ (**1**) appeared to be a particularly useful precursor for the synthesis of such compounds because it has two 16-electron square-planar platinum(II) moieties linked in a hinged fashion through sulphido-bridges.[6,7] The lone pairs on the sulphur atoms are sufficiently basic to form dative bonds towards other metal centres. In addition, the hinge angle between the square planes of *ca.* 125°[7,8] gives (**1**) a viable cone angle when it functions as a ligand. In preliminary communications[9,10] the manner in which (**1**) co-ordinates to $Ag^+$, $Hg^{2+}$, $Pd^{2+}$, and $Ni^{2+}$ to form aggregates of up to six metal atoms has been briefly described and the results are summarised in the Scheme.

In this paper a more detailed description of the synthesis and structure of the complex formed between (**1**) and $[\{RhCl(cod)\}_2]$ (cod = cyclo-octa-1,5-diene) is given. The formation of a rhodium–platinum aggregate was of interest because of the well established catalytic activity of rhodium in hydrogenation and hydroformylation reactions.

## Results and Discussion

When a solution of $[\{RhCl(cod)\}_2]$ in thf is added to a suspension of $[Pt_2S_2(PPh_3)_4]$ (**1**) in the same solvent the solution darkens and reduction of its volume gives $[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]Cl$ (**2a**) as a yellow microcrystalline solid. The corresponding $PF_6^-$ (**2b**) and $BPh_4^-$ (**2c**) salts were obtained by metathetical reactions in MeOH and recrystallised from dichloromethane–cyclohexane. Proton, $^{13}C\text{-}\{^1H\}$, $^{31}P\text{-}\{^1H\}$, and $^{195}Pt\text{-}\{^1H\}$ n.m.r. data for the salts (**2**) were consistent with the trinuclear aggregate structure shown (Y = Cl, $PF_6$, or $BPh_4$).

The absence of significant Rh–Pt metal–metal bonding in (**2**) was suggested by the non-observation of Rh–P coupling in the $^{31}P\text{-}\{^1H\}$ spectra, and Rh–Pt coupling in the $^{195}Pt\text{-}\{^1H\}$ spectra. The $^{195}Pt$ resonance was observed as a triplet at −4 467 p.p.m. with $^1J(\text{Pt–P})$ = 3 182 Hz, *i.e.* in the range anticipated for a conventional square-planar platinum(II) complex.[1,9,11] The $^{31}P\text{-}\{^1H\}$ spectrum consists of a single resonance at 15.1 p.p.m., flanked by satellites arising from the isotopomers with single $^{195}Pt$ (44.6% abundance $A_2A_2'X$ spin system) and double $^{195}Pt$ (11.3% abundance $A_2A_2'XX'$ spin system) nuclei. The computer simulation of the spectrum yielded the following parameters: $^1J(P_A\text{–Pt})$ = 3 151, $^3J(P_A\text{–Pt})$ = 19.5, $^4J(P_A\text{–}P_{A'})$ = 10.5 Hz. Satisfactory computer simulations[12–14] were only obtained when $^3J(P_A\text{–Pt})$ and $^3J(P_{A'}\text{–Pt})$ coupling constants were assigned negative signs relative to $^1J(\text{Pt–P})$. The data also suggested that Pt–Pt metal–metal bonding was not occurring to a significant extent.

The structure of (**2b**) was confirmed by a single-crystal *X*-ray diffraction study. Selected intramolecular bond lengths and angles are given in Table 1. The fractional co-ordinates of the non-hydrogen atoms are given in Table 2. The structure of the cation in (**2b**) is illustrated in the Figure and consists of three essentially square-planar metal moieties arranged in an approximately equilateral triangle. The intermetallic bond distances lie in the range 3.040(1)—3.255(1) Å and confirm that the bonding in the complex can be satisfactorily described in terms of square-planar 16-electron platinum and rhodium centres. No formal metal–metal bonds need to be invoked.

Around both platinum atoms the deviations from square-planarity are small and all atoms lie within 0.07 Å of the best $PtS_2P_2$ least-squares planes. The 'hinge angle' between these planes is 131.9°, and the 'hinge angle' between the $RhS_2$ plane and the two $PtS_2P_2$ units is 114.0(5)°. The rhodium atom also exhibits an approximately square-planar geometry, with S(1)–Rh(1)–S(2) 79.6(1)°. The Rh(1)–S(1) and Rh(1)–S(2) bond distances of 2.344(3) Å and 2.349(3) Å are similar to those in

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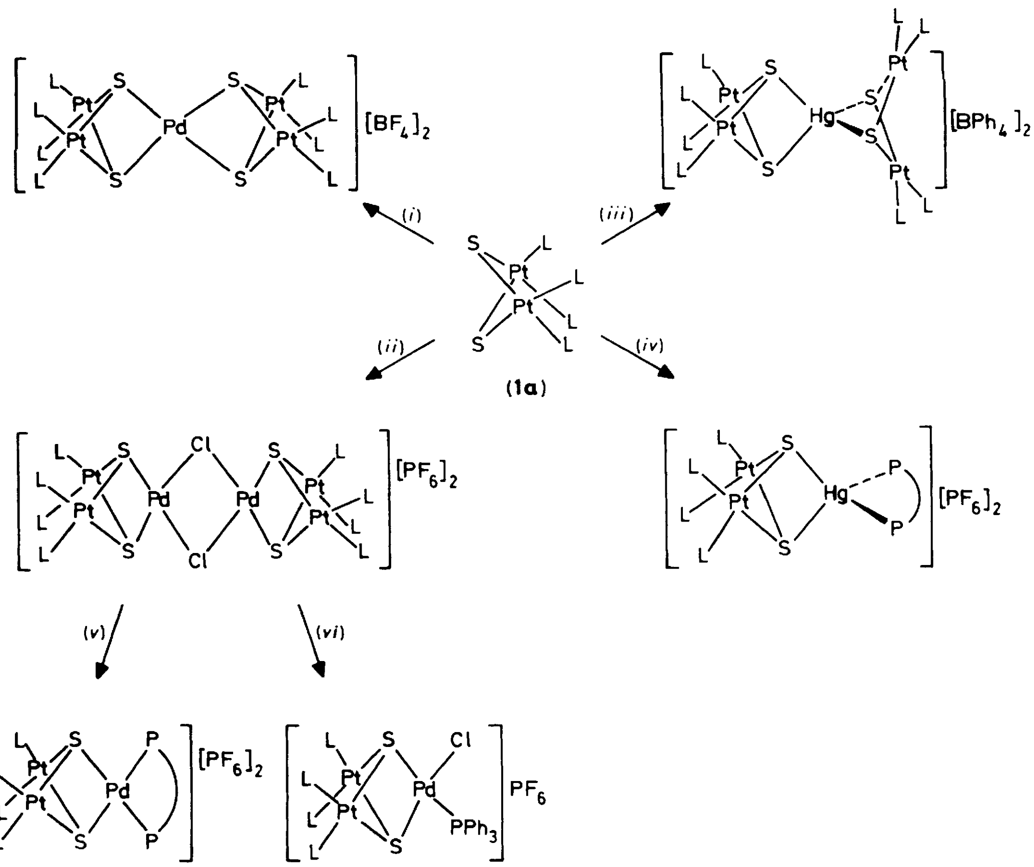
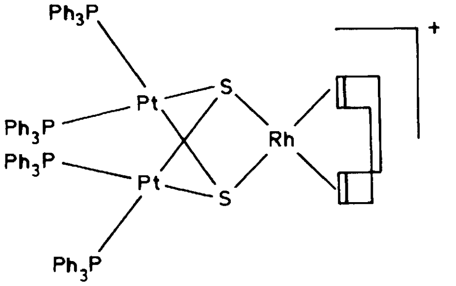
† *Supplementary data available* (No. SUP 56157, 106 pp.): bond lengths and angles involving all atoms, thermal parameters. See Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1985, Issue 1, pp. xvii—xix. Structure factors are available from the editorial office.



**Scheme.** L = PPh$_3$. (*i*) [Pd(dmf)$_4$][BF$_4$]$_2$ (dmf = dimethylformamide), tetrahydrofuran (thf); (*ii*) [PdCl$_2$(PhCN)$_2$], thf; MeOH, NH$_4$PF$_6$; (*iii*) [HgCl$_2$(PPh$_3$)$_2$], MeOH; NaBPh$_4$; (*iv*) [HgCl$_2$(dppe)] [dppe = 1,2-bis(diphenylphosphino)ethane], thf; NH$_4$PF$_6$, MeOH; (*v*) dppe, MeOH; (*vi*) PPh$_3$, MeOH



**(2)**

related rhodium–sulphido-complexes.[15] The bond lengths and angles in the co-ordinated cod ligand are comparable with those reported previously.[16]

The compounds **(2)** were synthesised with cod co-ordinated to rhodium with the aim that the cod would be readily displaced by substrates of interest in catalytic processes, *e.g.* CO, H$_2$, ethene, *etc.* This ease of replacement in related Rh$^I$ cod complexes has been illustrated by reactions with both unidentate and bidentate phosphines under mild conditions.[17–19] Surprisingly the complexes **(2)** showed no tendency for substitution of the co-ordinated cod with H$_2$, CO, or SO$_2$ under mild conditions. Reaction with dppe was very slow and then the only product to be isolated containing dppe was [Rh(dppe)$_2$]$^+$. This type of behaviour has been previously noted when the rhodium centre has a large bulky ligand in addition to co-ordinated cod.[16] The complex proved to be remarkably stable to replacement reactions under ambient conditions and forcing conditions resulted only in the breakdown of the Pt$_2$Rh unit. Studies are currently being made on the comparable ethene complexes with a view to synthesising more labile derivatives.

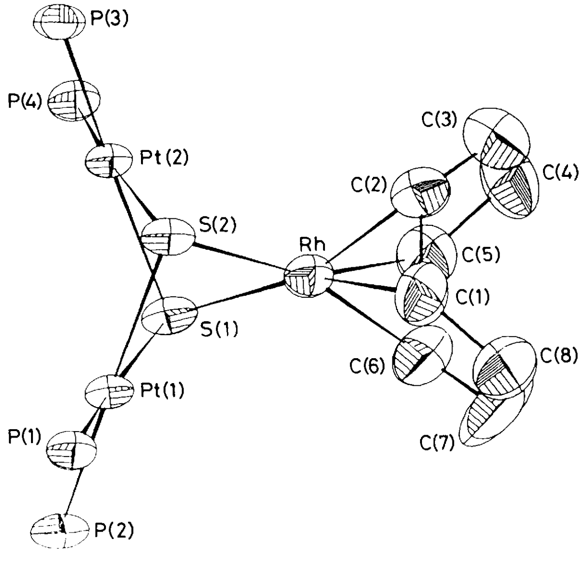


**Figure.** The molecular structure of the [Pt$_2$Rh($\mu_3$-S)$_2$(PPh$_3$)$_4$(cod)]$^+$ cation. The phenyl rings have been omitted for clarity

**Table 1.** Selected molecular dimensions for $[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]^+$ (**2b**) with estimated standard deviations in parentheses

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| (*a*) Intramolecular distances (Å) | | | | | | | |
| Pt(1)–S(1) | 2.355(3) | Rh(1)–S(1) | 2.344(3) | Pt(1)–P(3) | 2.274(3) | Rh(1)–C(1) | 2.151(16) |
| Pt(1)–S(2) | 2.343(3) | Rh(1)–S(2) | 2.349(3) | Pt(1)–P(4) | 2.285(3) | Rh(1)–C(2) | 2.167(15) |
| Pt(2)–S(1) | 2.335(3) | Rh(1)–Pt(1) | 3.063(1) | Pt(2)–P(1) | 2.274(3) | Rh(1)–C(5) | 2.149(15) |
| Pt(2)–S(2) | 2.357(3) | Rh(1)–Pt(2) | 3.040(1) | Pt(2)–P(2) | 2.289(3) | Rh(1)–C(6) | 2.153(16) |
| S(1)–S(2) | 3.004(4) | Pt(1)–Pt(2) | 3.255(1) | | | | |
| Average P–C 1.82(1), average C–C (phenyl) 1.38(2), average P–F (hexafluorophosphate) 1.46(3) | | | | | | | |
| (*b*) Bond distances (Å) in the cod ligand | | | | | | | |
| C(1)–C(2) | 1.38(2) | C(3)–C(4) | 1.44(3) | C(5)–C(6) | 1.36(2) | C(7)–C(8) | 1.46(2) |
| C(2)–C(3) | 1.50(2) | C(4)–C(5) | 1.50(2) | C(6)–C(7) | 1.51(2) | C(8)–C(1) | 1.46(2) |
| (*c*) Bond angles (°) | | | | | | | |
| P(3)–Pt(1)–S(1) | 170.37(1) | P(2)–Pt(2)–S(1) | 169.6(1) | P(3)–Pt(1)–P(4) | 99.1(1) | Pt(1)–S(1)–Rh(1) | 81.4(1) |
| P(3)–Pt(1)–S(2) | 92.47(1) | P(2)–Pt(2)–S(2) | 90.00(1) | P(2)–Pt(2)–P(1) | 98.3(1) | Pt(1)–S(2)–Rh(1) | 81.5(1) |
| P(4)–Pt(1)–S(1) | 89.0(1) | P(1)–Pt(2)–S(1) | 92.1(1) | Pt(1)–S(1)–Pt(2) | 87.9(1) | Pt(2)–S(1)–Rh(1) | 81.00(1) |
| P(4)–Pt(1)–S(2) | 168.5(1) | P(1)–Pt(2)–S(2) | 170.5(1) | Pt(1)–S(2)–Pt(2) | 87.6(1) | Pt(2)–S(2)–Rh(1) | 80.4(1) |
| | | | | | | S(1)–Rh(1)–S(2) | 79.6(1) |
| Average Pt–P–C (phosphine) 114.1(4) | | | | | | | |
| (*d*) Bond angles (°) in cod | | | | | | | |
| C(8)–C(1)–C(2) | 127.8(2) | C(2)–C(3)–C(4) | 115.6(2) | C(4)–C(5)–C(6) | 124.7(2) | C(6)–C(7)–C(8) | 115.9(2) |
| C(1)–C(2)–C(3) | 123.9(2) | C(3)–C(4)–C(5) | 119.4(2) | C(5)–C(6)–C(7) | 124.0(2) | C(7)–C(8)–C(1) | 116.2(2) |

## Experimental

Reactions were routinely carried out, using standard Schlenk-line procedures, under an atmosphere of pure, dry $N_2$ and using dry dioxygen-free solvents. Microanalyses (C,H,N,Cl, and Pt) were carried out by Mr. M. Gascoyne and his staff of this laboratory. Infrared spectra were recorded, both as Nujol and hexachlorobutadiene (hcbd) mulls, using a Pye-Unicam SP2000 spectrometer. Melting points were measured using an Electrothermal apparatus and conductivities were recorded on a Philips 9505 bridge using a standard conductivity cell with a cell constant of 0.7 $cm^{-1}$.

Hydrogen-1 n.m.r. data were recorded using a Bruker WH-300 spectrometer; $^{13}C\text{-}\{^1H\}$, $^{13}C$, $^{31}P\text{-}\{^1H\}$, and $^{195}Pt\text{-}\{^1H\}$ n.m.r. were recorded using a Bruker AM-250 spectrometer. The samples were referenced as follows: $^1H$ to $SiMe_4$, $^{31}P$ to $P(OMe)_3O$ in $D_2O$, $^{13}C$ to internal $CD_2Cl_2$, and $^{195}Pt$ to $Na_2[PtCl_6]$ in $D_2O$. The machine operating frequencies were as follows: $^1H$, 300; $^{31}P$, 101.26; $^{31}C$, 62.90; $^{195}Pt$, 53.55 MHz. All samples were run in deuteriated solvents. Proton-decoupled $^{31}P$ n.m.r. computer simulations were carried out using the Oxford University VAX/VMS computer system utilising a program developed by Professor R. K. Harris of the University of East Anglia and adapted for use at Oxford by Dr. A. E. Derome. $[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ was synthesised by standard literature methods, either from $[Pt(PPh_3)_3]$ and elemental sulphur,[7] or $[PtCl_2(PPh_3)_2]$ and $Na_2S{\cdot}9H_2O$.[6]

*Synthesis of* $[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]Cl$ (**2a**).—$[Pt_2(\mu\text{-}S)_2(PPh_3)_4]$ (0.3 g, 0.2 mmol) was suspended in thf (30 $cm^3$). To this was added, with stirring, a solution of $[\{RhCl(cod)\}_2]$ (0.05 g, 0.1 mmol) in thf (30 $cm^3$). The orange suspension soon became a bright yellow solution and this was stirred for 1 h to ensure complete reaction. The resulting solution was evaporated to dryness and the residue recrystallised from dichloromethane–cyclohexane to yield (**2a**) (0.33 g, 94%) as a bright yellow microcrystalline product, m.p. 210 °C (decomp.) (Found: C, 54.7; H, 4.6; Cl, 2.1. $C_{80}H_{72}ClP_4Pt_2RhS_2$ requires C, 54.9; H, 4.1; Cl, 2.0%). $\delta_H(CD_2Cl_2)$ 7.4 and 7.2 (60 H, m, Ph), 3.7 (4 H, br s, =C–H), 2.36 (4 H, br m, $-CH_2-$), and 2.10 (4 H, br m, $-CH_2-$); $\delta_C(CD_2Cl_2)$ 134.9 (s, Ph), 77.73 [4 C, d, $^1J$(C–Rh) = 11.1 Hz, =C–H], 31.9 (4 C, s, $-CH_2-$); $\delta_P(CDCl_3)$ 15.1 [t, $^1J$(P–Pt) = 3 151, $^3J$(P–Pt) = 19.5, $^4J(P_A\text{–}P_{A'})$ 10.5 Hz]; $\delta_{Pt}(CD_2Cl_2)$ −4 467 [t, $^1J$(Pt–P) = 3 182 Hz].

The corresponding $PF_6^-$ and $BPh_4^-$ salts were synthesised by adding methanolic solutions of $NH_4PF_6$ and $NaBPh_4$ to $CH_2Cl_2$–MeOH solutions of (**2a**).

$[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]PF_6{\cdot}CH_2Cl_2$ (**2b**) crystallises from dichloromethane–cyclohexane (yield 90%), m.p. 305—310 °C (decomp.) (Found: C, 49.7; H, 3.8. $C_{81}H_{74}Cl_2F_6P_5Pt_2RhS_2$ requires C, 50.0; H, 3.8%). Conductivity ($CH_3NO_2$): $\Lambda_M$ = 69.9 × $10^{-6}$ S $cm^2$ $mol^{-1}$ ($10^{-3}$ mol $dm^{-3}$).

$[Pt_2Rh(\mu_3\text{-}S)_2(PPh_3)_4(cod)]BPh_4$ (**2c**): yield 90%, m.p. 205 °C (decomp.) (Found: C, 61.2; H, 4.5. $C_{104}H_{92}BP_4Pt_2RhS_2$ requires C, 61.4; H, 4.5%).

*Crystal Data for* (**2b**).—$C_{80}H_{72}F_6P_5Pt_2RhS_2{\cdot}CH_2Cl_2$, $M$ = 1 944.3, monoclinic, $a$ = 16.681(2), $b$ = 17.181(5), $c$ = 27.476(2) Å, $\beta$ = 103.58(4)°, $U$ = 7 646 Å$^3$ (by least-squares refinement on diffractometer angles for 15 automatically centred reflections, $\lambda$ = 0.710 69 Å), space group $P2_1/c$, $D_m$ = 1.65, $Z$ = 4, $D_c$ = 1.69 g $cm^{-3}$. Orange-yellow crystals grown from dichloromethane–cyclohexane and sealed in a 0.7 mm Lindemann capillary. Crystal dimension: 0.15 × 0.1 × 0.05 mm, $\mu$(Mo-$K_\alpha$) = 43.3 $cm^{-1}$.

*Data Collection and Processing.*—A CAD4 diffractometer, in $\omega/2\theta$ mode with $\omega$-scan width = 0.9 was used, with graphite-monochromated Mo-$K_\alpha$ radiation; 12 227 reflections were measured ($1 \leqslant \theta \leqslant 20$, $\pm h$, $\pm k$, $l$), with 6 349 unique (merging $R$ = 0.020 6). Absorption profiles for several reflections showed only small differences in the transmission coefficients and therefore no correction was applied, giving 6 349 reflections with $I \geqslant 3\sigma(I)$.

*Structure Analysis and Refinement.*—Direct methods were used (Pt, Rh atoms) followed by normal heavy-atom procedures. Blocked-matrix least-squares refinement was carried out with the Pt, Rh, F, P, and S atoms and carbons of

**Table 2.** Final fractional co-ordinates ($\times 10^4$) for non-hydrogen atoms with estimated standard deviations in parentheses

| Atom | *X*/*a* | *Y*/*b* | *Z*/*c* |
|---|---|---|---|
| Pt(1) | 3 033.4(3) | 2 267.8(3) | 3 300.3(2) |
| Pt(2) | 1 864.5(3) | 2 293.1(3) | 4 096.4(2) |
| Rh(1) | 2 355.7(7) | 780.6(6) | 3 671.6(4) |
| S(1) | 3 168(2) | 1 779(2) | 4 116(1) |
| S(2) | 1 674(2) | 1 877(2) | 3 259(1) |
| P(1) | 2 201(2) | 2 528(2) | 4 934(1) |
| P(2) | 536(2) | 2 743(2) | 3 925(1) |
| P(3) | 2 741(2) | 2 578(2) | 2 473(1) |
| P(4) | 4 406(2) | 2 568(2) | 3 503(1) |
| C(1) | 3 256(10) | −119(9) | 3 919(6) |
| C(2) | 2 719(10) | −118(9) | 4 234(6) |
| C(3) | 2 037(14) | −700(11) | 4 203(8) |
| C(4) | 1 327(17) | −590(14) | 3 797(10) |
| C(5) | 1 370(10) | −23(10) | 3 387(6) |
| C(6) | 1 920(11) | −65(10) | 3 092(6) |
| C(7) | 2 532(15) | −718(11) | 3 115(8) |
| C(8) | 3 279(13) | −666(11) | 3 514(8) |
| C(111) | 2 118(5) | 3 527(7) | 5 133(4) |
| C(112) | 2 055(5) | 4 103(7) | 4 782(4) |
| C(113) | 2 030(5) | 4 879(8) | 4 925(5) |
| C(114) | 2 072(5) | 5 065(8) | 5 411(6) |
| C(115) | 2 141(5) | 4 494(9) | 5 761(5) |
| C(116) | 2 170(5) | 3 715(8) | 5 624(5) |
| C(121) | 3 264(7) | 2 352(6) | 5 250(3) |
| C(122) | 3 857(8) | 2 858(6) | 5 156(4) |
| C(123) | 4 678(8) | 2 750(7) | 5 390(4) |
| C(124) | 4 900(10) | 2 147(9) | 5 716(5) |
| C(125) | 4 331(11) | 1 654(9) | 5 816(5) |
| C(126) | 3 491(9) | 1 744(7) | 5 581(4) |
| C(131) | 1 612(6) | 1 855(6) | 5 223(4) |
| C(132) | 1 196(6) | 2 043(7) | 5 586(4) |
| C(133) | 764(7) | 1 477(9) | 5 773(4) |
| C(134) | 739(7) | 729(9) | 5 609(5) |
| C(135) | 1 149(7) | 520(9) | 5 240(5) |
| C(136) | 1 580(7) | 1 095(8) | 5 054(4) |
| C(211) | 425(7) | 3 672(6) | 3 608(3) |
| C(212) | 1 074(7) | 3 977(6) | 3 435(3) |
| C(213) | 990(8) | 4 674(7) | 3 175(4) |
| C(214) | 243(9) | 5 069(8) | 3 078(4) |
| C(215) | −408(9) | 4 771(8) | 3 249(4) |
| C(216) | −329(8) | 4 073(7) | 3 512(4) |
| C(221) | 8(6) | 2 907(6) | 4 428(4) |
| C(222) | 97(6) | 3 606(7) | 4 677(4) |
| C(223) | −284(7) | 3 743(8) | 5 079(5) |
| C(224) | −745(7) | 3 158(9) | 5 207(5) |
| C(225) | 847(7) | 2 458(9) | 4 965(5) |
| C(226) | −463(7) | 2 324(8) | 4 567(5) |
| C(231) | −144(6) | 2 088(7) | 3 497(4) |
| C(232) | −625(7) | 2 344(8) | 3 051(5) |
| C(233) | −1 159(8) | 1 826(10) | 2 731(6) |
| C(234) | −1 190(7) | 1 077(9) | 2 866(5) |
| C(235) | −724(8) | 813(9) | 3 298(6) |
| C(236) | −184(7) | 1 329(8) | 3 620(5) |
| C(311) | 2 926(8) | 3 573(8) | 2 299(5) |
| C(312) | 2 789(9) | 3 798(9) | 1 804(5) |
| C(313) | 2 910(10) | 4 569(10) | 1 693(6) |
| C(314) | 3 128(10) | 5 097(10) | 2 055(6) |
| C(315) | 3 243(10) | 4 913(10) | 2 553(6) |
| C(316) | 3 124(8) | 4 130(8) | 2 669(5) |
| C(321) | 1 652(7) | 2 491(6) | 2 148(3) |
| C(322) | 1 354(8) | 1 890(7) | 1 819(4) |
| C(323) | 498(9) | 1 858(7) | 1 599(4) |
| C(324) | −19(8) | 2 401(8) | 1 713(4) |
| C(325) | 264(8) | 2 991(7) | 2 033(4) |
| C(326) | 1 111(8) | 3 043(7) | 2 252(4) |
| C(331) | 3 275(5) | 1 905(7) | 2 152(3) |
| C(332) | 3 722(6) | 2 100(7) | 1 808(4) |
| C(333) | 4 147(6) | 1 530(9) | 1 609(4) |
| C(334) | 4 123(6) | 779(9) | 1 754(4) |
| C(335) | 3 677(7) | 567(8) | 2 098(5) |
| C(336) | 3 256(6) | 1 134(8) | 2 295(4) |
| C(411) | 4 972(6) | 2 699(7) | 3 009(4) |
| C(412) | 4 985(6) | 3 415(7) | 2 738(4) |
| C(413) | 5 390(7) | 3 525(8) | 2 400(4) |
| C(414) | 5 779(7) | 2 912(9) | 2 242(4) |
| C(415) | 5 781(7) | 2 194(9) | 2 459(5) |
| C(416) | 5 377(6) | 2 080(8) | 2 841(4) |
| C(421) | 4 620(7) | 3 454(6) | 3 858(3) |
| C(422) | 3 998(7) | 3 866(7) | 4 001(3) |
| C(423) | 4 157(9) | 4 571(8) | 4 255(4) |
| C(424) | 4 937(10) | 4 860(8) | 4 366(4) |
| C(425) | 5 564(9) | 4 475(9) | 4 237(4) |
| C(426) | 5 422(9) | 3 771(8) | 3 984(4) |
| C(431) | 4 988(6) | 1 812(8) | 3 900(5) |
| C(432) | 4 920(7) | 1 064(9) | 3 717(5) |
| C(433) | 5 351(9) | 455(10) | 4 006(7) |
| C(434) | 5 825(8) | 624(11) | 4 468(7) |
| C(435) | 5 897(8) | 1 348(11) | 4 656(6) |
| C(436) | 5 477(7) | 1 958(8) | 4 371(5) |
| P(5) | 7 253(3) | 1 656(3) | 1 181(2) |
| F(1) | 6 387(10) | 1 512(14) | 1 255(8) |
| F(2) | 6 970(14) | 2 396(10) | 912(10) |
| F(3) | 6 902(15) | 1 283(13) | 706(7) |
| F(4) | 7 510(18) | 2 112(16) | 1 625(8) |
| F(5) | 7 463(13) | 937(15) | 1 453(13) |
| F(6) | 8 056(11) | 1 730(18) | 1 105(11) |
| Cl(1) | 7 421(6) | 3 525(7) | 3 471(4) |
| Cl(2) | 7 283(8) | 1 932(9) | 3 896(5) |
| C(12) | 7 597(24) | 2 682(26) | 3 631(15) |

cod anisotropic, and the remainder isotropic. Ring hydrogens were in calculated positions (C–H 0.99 Å), with $U_{\text{iso.}} = 0.05$ Å$^2$. A Chebyshev weighting scheme with coefficients of 202.1, 270.0, and 89.0 gave satisfactory agreement analyses. Final *R* and *R'* values are 0.043 and 0.054. Programs and computers used and sources of scattering factor data are given in refs. 20—22.

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