Inorganic Chemistry
including bioinorganic chemistry
Inorg. Chem., 1996, 35(10), 3001-3006, DOI:10.1021/ic950438d

Terms & Conditions

Electronic Supporting Information files are available without a subscription to ACS Web Editions. The American Chemical Society holds a copyright ownership interest in any copyrightable Supporting Information. Files available from the ACS website may be downloaded for personal use only. Users are not otherwise permitted to reproduce, republish, redistribute, or sell any Supporting Information from the ACS website, either in whole or in part, in either machine-readable form or any other form without permission from the American Chemical Society. For permission to reproduce, republish and redistribute this material, requesters must process their own requests via the RightsLink permission system. Information about how to use the RightsLink permission system can be found at http://pubs.acs.org/page/copyright/permissions.html

ACS Publications
MOST TRUSTED. MOST CITED. MOST READ.

Copyright © 1996 American Chemical Society

P 3006 - 1

2

Table 1. Summary of Crystal Data, Details of Intensity Collection, and Least-Squares Refinement Parameters for **9**

empirical formula	$C_{49}H_{43}BF_4IrN_2P_2S_2 \cdot 0.5C_6H_6$
crystal color and size	yellow, 0.34 x 0.28 x 0.43
M_r	1064.92
crystal class	Monoclinic
space group	$P2_1/c$
a, Å	17.723(3)
b, Å	10.408(1)
c, Å	26.073(4)
β, deg	108.08(1)
V, Å^3	4572.0(11)
Z	4
D_{calc}, Mgm^{-3}	1.547
μ(MoKα), mm^{-1}	3.134
F(000)	2124
θ range for data collection, deg	2.56 to 27.00
T, K	173(2)
min., max. transmission[a]	0.5297, 0.7937
wavelength, Å	0.71073
absorption corrn.	SHELXA-90 in SHELXL-93[6]
limiting indices	$-18 \le h \le 22$, $-10 \le k \le 13$, $-33 \le l \le 31$
reflections collected	10290
independent reflections	9964
R_{int}	0.0453
no obsd. data [$I > 2\sigma(I)$]	6153
R1 [$I > 2\sigma(I)$][b]	0.0468
wR2 (all data)[c]	0.1156
weighting scheme	$w = 1/[\sigma^2(F_o^2)+(0.0503P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
goodness of fit on F^2	0.878
refinement method	full-matrix least-squares on F^2
parameters refined	556
largest diff. peak and hole, e/Å^3	1.472 and -0.952

a. Absorption correction using SHELXA-90 in SHELXL-93[6]

b. $R = \Sigma(F_o-F_c)/\Sigma F_o$. c. $wR2 = \{\Sigma[w(F_o^2-F_c^2)^2/\Sigma[w(F_o^2)^2]\}^{1/2}$.

P3006-2 3

Table 2. Bond lengths [Å] and angles [°] for **9**.

Ir-N(1)	2.080(6)	Ir-P(2)	2.291(2)
Ir-P(1)	2.304(2)	Ir-S(2)	2.394(2)
Ir-S(1)	2.535(2)	Ir-H(1Ir)	1.72(11)
S(1)-C(5)	1.741(7)	S(2)-C(10)	1.730(8)
P(1)-C(11)	1.828(7)	P(1)-C(21)	1.831(7)
P(1)-C(31)	1.843(7)	P(2)-C(41)	1.826(7)
P(2)-C(61)	1.827(7)	P(2)-C(51)	1.833(7)
N(1)-C(1)	1.320(9)	N(1)-C(5)	1.352(10)
N(2)-C(6)	1.349(10)	N(2)-C(10)	1.354(10)
N(2)...(F4)	2.778(8)	N(2)-H(2A)	0.94(8)
C(1)-C(2)	1.374(10)	C(2)-C(3)	1.393(11)
C(3)-C(4)	1.391(11)	C(4)-C(5)	1.412(10)
C(6)-C(7)	1.344(11)	C(7)-C(8)	1.370(11)
C(8)-C(9)	1.381(12)	C(9)-C(10)	1.383(10)
C(11)-C(12)	1.374(10)	C(11)-C(16)	1.411(9)
C(12)-C(13)	1.395(10)	C(13)-C(14)	1.408(11)
C(14)-C(15)	1.356(12)	C(15)-C(16)	1.387(10)
C(21)-C(22)	1.396(10)	C(21)-C(26)	1.401(10)
C(22)-C(23)	1.410(11)	C(23)-C(24)	1.367(11)
C(24)-C(25)	1.380(11)	C(25)-C(26)	1.405(11)
C(31)-C(32)	1.381(10)	C(31)-C(36)	1.390(10)
C(32)-C(33)	1.392(11)	C(33)-C(34)	1.358(12)
C(34)-C(35)	1.398(12)	C(35)-C(36)	1.366(12)
C(41)-C(46)	1.393(10)	C(41)-C(42)	1.395(10)
C(42)-C(43)	1.379(10)	C(43)-C(44)	1.377(11)
C(44)-C(45)	1.378(12)	C(45)-C(46)	1.399(11)
C(51)-C(56)	1.396(10)	C(51)-C(52)	1.398(10)
C(52)-C(53)	1.367(10)	C(53)-C(54)	1.405(11)
C(54)-C(55)	1.373(11)	C(55)-C(56)	1.392(10)
C(61)-C(66)	1.392(10)	C(61)-C(62)	1.395(9)
C(62)-C(63)	1.384(10)	C(63)-C(64)	1.361(11)
C(64)-C(65)	1.385(11)	C(65)-C(66)	1.376(11)
B(1)-F(4)	1.316(13)	B(1)-F(1)	1.326(14)
B(1)-F(3)	1.36(2)	B(1)-F(2)	1.48(2)
F(4)...H(1Ir)	2.30(11)	F(4)...H(2A)	1.96(8)
C(1S)-C(2S)	1.39	C(1S)-C(6S)	1.39
C(2S)-C(3S)	1.39	C(3S)-C(4S)	1.39
C(4S)-C(5S)	1.39	C(5S)-C(6S)	1.39
C(7S)-C(8S)	1.39	C(7S)-C(12S)	1.39
C(8S)-C(9S)	1.39	C(9S)-C(10S)	1.39
C(10S)-C(11S)	1.39	C(11S)-C(12S)	1.39

N(1)-Ir-P(2)	90.1(2)	N(1)-Ir-P(1)	170.7(2)
P(2)-Ir-P(1)	99.07(7)	N(1)-Ir-S(2)	85.2(2)
P(2)-Ir-S(2)	175.03(7)	P(1)-Ir-S(2)	85.74(7)
N(1)-Ir-S(1)	66.4(2)	P(2)-Ir-S(1)	93.87(7)
P(1)-Ir-S(1)	114.26(6)	S(2)-Ir-S(1)	82.95(7)
N(1)-Ir-H(1Ir)	69(4)	P(2)-Ir-H(1Ir)	82(4)
P(1)-Ir-H(1Ir)	111(4)	S(2)-Ir-H(1Ir)	98(4)
S(1)-Ir-H(1Ir)	135(4)	F(4)...H(1IR)-Ir	164(6)
C(5)-S(1)-Ir	77.7(3)	C(10)-S(2)-Ir	114.4(3)
C(11)-P(1)-C(21)	100.3(3)	C(11)-P(1)-C(31)	104.1(3)
C(21)-P(1)-C(31)	104.4(3)	C(11)-P(1)-Ir	116.5(2)
C(21)-P(1)-Ir	122.0(2)	C(31)-P(1)-Ir	107.7(2)
C(41)-P(2)-C(61)	100.6(3)	C(41)-P(2)-C(51)	103.2(3)
C(61)-P(2)-C(51)	105.0(3)	C(41)-P(2)-Ir	112.3(2)
C(61)-P(2)-Ir	119.0(2)	C(51)-P(2)-Ir	114.7(2)
C(1)-N(1)-C(5)	120.2(6)	C(1)-N(1)-Ir	134.9(5)
C(5)-N(1)-Ir	104.7(5)	C(6)-N(2)-C(10)	123.5(7)

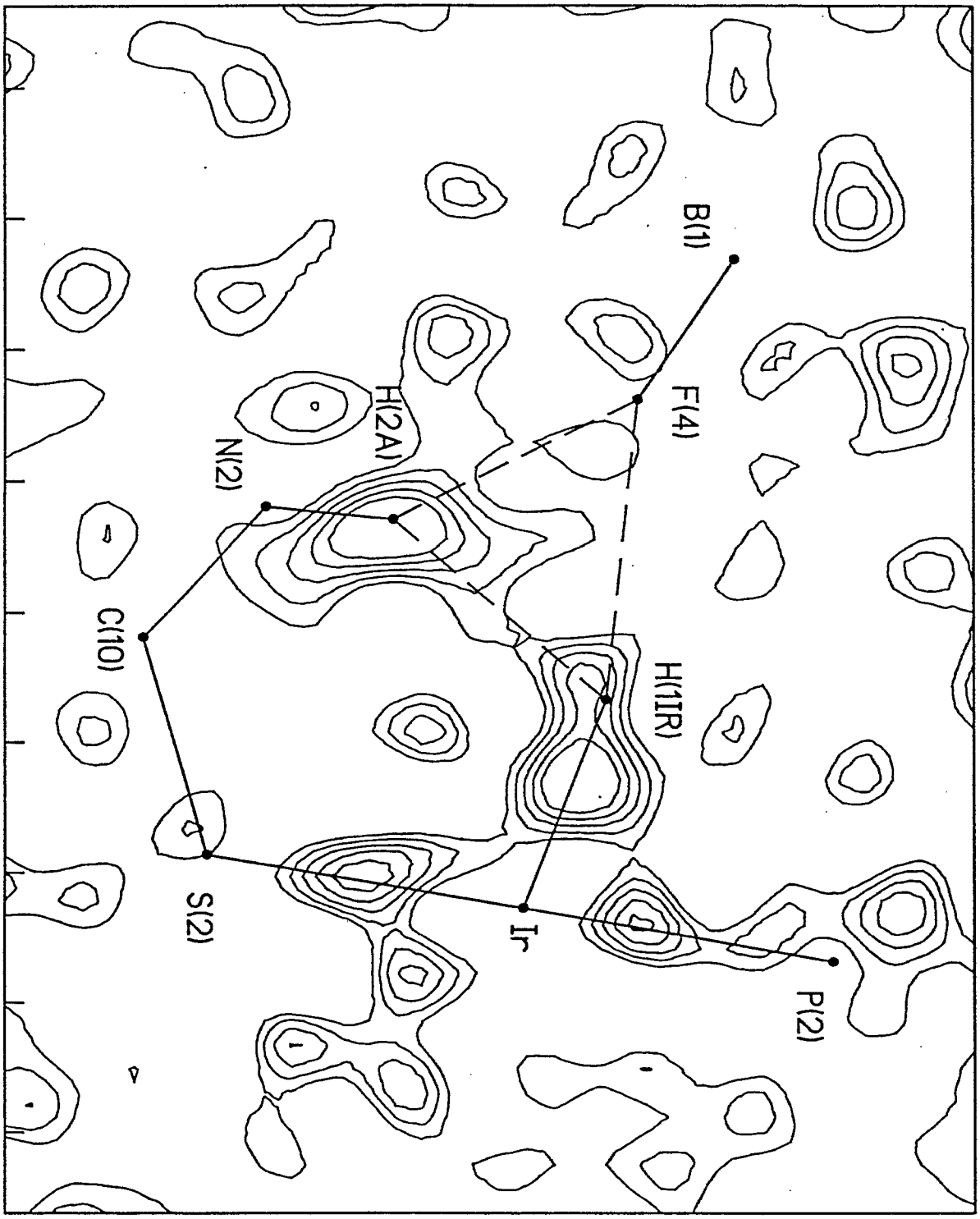
C(10)-N(2)-H(2A)	127(5)	C(6)-N(2)-H(2A)	110(5)
N(1)-C(1)-C(2)	122.6(7)	C(1)-C(2)-C(3)	117.8(7)
C(4)-C(3)-C(2)	121.5(7)	C(3)-C(4)-C(5)	116.1(8)
N(1)-C(5)-C(4)	121.8(7)	N(1)-C(5)-S(1)	110.9(5)
C(4)-C(5)-S(1)	127.3(6)	C(7)-C(6)-N(2)	120.1(8)
C(6)-C(7)-C(8)	119.5(8)	C(7)-C(8)-C(9)	119.4(8)
C(8)-C(9)-C(10)	121.2(8)	N(2)-C(10)-C(9)	116.0(8)
N(2)-C(10)-S(2)	122.7(6)	C(9)-C(10)-S(2)	121.3(6)
C(12)-C(11)-C(16)	118.9(7)	C(12)-C(11)-P(1)	121.6(6)
C(16)-C(11)-P(1)	119.4(6)	C(11)-C(12)-C(13)	122.3(8)
C(12)-C(13)-C(14)	117.6(8)	C(15)-C(14)-C(13)	120.7(8)
C(14)-C(15)-C(16)	121.6(7)	C(15)-C(16)-C(11)	118.9(7)
C(22)-C(21)-C(26)	118.3(7)	C(22)-C(21)-P(1)	118.5(6)
C(26)-C(21)-P(1)	123.1(6)	C(21)-C(22)-C(23)	120.4(7)
C(24)-C(23)-C(22)	121.2(8)	C(23)-C(24)-C(25)	118.6(7)
C(24)-C(25)-C(26)	121.8(8)	C(21)-C(26)-C(25)	119.6(7)
C(32)-C(31)-C(36)	117.8(7)	C(32)-C(31)-P(1)	119.5(6)
C(36)-C(31)-P(1)	122.3(6)	C(31)-C(32)-C(33)	121.1(8)
C(34)-C(33)-C(32)	120.5(9)	C(33)-C(34)-C(35)	118.8(8)
C(36)-C(35)-C(34)	120.5(8)	C(35)-C(36)-C(31)	121.2(8)
C(46)-C(41)-C(42)	118.5(7)	C(46)-C(41)-P(2)	123.5(6)
C(42)-C(41)-P(2)	118.0(5)	C(43)-C(42)-C(41)	121.1(7)
C(44)-C(43)-C(42)	119.7(8)	C(43)-C(44)-C(45)	120.7(8)
C(44)-C(45)-C(46)	119.6(8)	C(41)-C(46)-C(45)	120.4(8)
C(56)-C(51)-C(52)	118.2(7)	C(56)-C(51)-P(2)	123.9(6)
C(52)-C(51)-P(2)	117.9(5)	C(53)-C(52)-C(51)	120.9(7)
C(52)-C(53)-C(54)	120.2(8)	C(55)-C(54)-C(53)	120.1(7)
C(54)-C(55)-C(56)	119.3(8)	C(55)-C(56)-C(51)	121.3(7)
C(66)-C(61)-C(62)	119.0(7)	C(66)-C(61)-P(2)	120.0(5)
C(62)-C(61)-P(2)	120.9(5)	C(63)-C(62)-C(61)	120.1(7)
C(64)-C(63)-C(62)	120.0(7)	C(63)-C(64)-C(65)	120.9(7)
C(66)-C(65)-C(64)	119.7(7)	C(65)-C(66)-C(61)	120.3(7)
F(4)-B(1)-F(1)	118.9(12)	F(4)-B(1)-F(3)	114.1(11)
F(1)-B(1)-F(3)	116.4(10)	F(4)-B(1)-F(2)	99.6(10)
F(1)-B(1)-F(2)	102.7(11)	F(3)-B(1)-F(2)	100.4(11)
H(1Ir)...F(4)-B(1)	149(3)	H(1Ir)...F(4)...H(2A)	57(3)
F(4)...H(2A)-N(2)	144(7)	H(2A)...F(4)-B(1)	148.1(24)
C(2S)-C(1S)-C(6S)	120.0	C(3S)-C(2S)-C(1S)	120.0
C(4S)-C(3S)-C(2S)	120.0	C(3S)-C(4S)-C(5S)	120.0
C(6S)-C(5S)-C(4S)	120.0	C(5S)-C(6S)-C(1S)	120.0
C(8S)-C(7S)-C(12S)	120.0	C(9S)-C(8S)-C(7S)	120.0
C(10S)-C(9S)-C(8S)	120.0	C(11S)-C(10S)-C(9S)	120.0
C(10S)-C(11S)-C(12S)	120.0	C(11S)-C(12S)-C(7S)	120.0

Symmetry transformations used to generate equivalent atoms:

P3006-4
4

5

A difference electron density map calculated in the plane of H(2A), F(4) H(1IR) with contours every 0.1 eÅ^{-3}.



P3006-5

[Table 4] **Anisotropic displacement parameters [$Å^2$ x 10^3] for 9.**

The anisotropic displacement factor exponent takes the form:

$-2\pi^2 [(ha^*)^2 U_{11} + ... + 2hka^*b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
Ir	17(1)	16(1)	17(1)	-1(1)	5(1)	-2(1)
S(1)	24(1)	20(1)	26(1)	0(1)	5(1)	-3(1)
S(2)	21(1)	22(1)	27(1)	-1(1)	11(1)	-3(1)
P(1)	17(1)	19(1)	17(1)	0(1)	6(1)	0(1)
P(2)	20(1)	16(1)	16(1)	2(1)	7(1)	0(1)
N(1)	15(3)	24(3)	19(3)	-2(3)	5(3)	0(3)
N(2)	23(4)	29(4)	32(4)	2(3)	13(3)	5(3)
C(1)	26(4)	28(5)	22(4)	-3(3)	9(3)	0(4)
C(2)	31(5)	36(5)	21(4)	6(3)	10(3)	-2(4)
C(3)	23(4)	45(6)	27(4)	-13(4)	-2(4)	-12(4)
C(4)	30(5)	37(5)	28(4)	-7(4)	7(4)	-17(4)
C(5)	17(4)	33(5)	22(4)	0(3)	6(3)	0(3)
C(6)	29(5)	27(5)	32(4)	4(4)	8(4)	4(4)
C(7)	34(5)	23(5)	40(5)	-3(4)	10(4)	4(4)
C(8)	26(5)	41(5)	39(5)	-8(4)	16(4)	2(4)
C(9)	33(5)	40(5)	30(5)	1(4)	18(4)	7(4)
C(10)	21(4)	24(4)	24(4)	-5(4)	6(3)	-4(4)
C(11)	21(4)	22(4)	17(3)	2(3)	7(3)	0(3)
C(12)	35(5)	26(4)	23(4)	-2(3)	14(4)	-7(4)
C(13)	51(6)	31(5)	40(5)	0(4)	25(5)	-8(5)
C(14)	59(6)	34(5)	23(4)	8(4)	23(4)	9(5)
C(15)	39(5)	32(4)	14(4)	-3(3)	8(3)	12(4)
C(16)	20(4)	28(4)	21(4)	-1(4)	9(3)	4(4)
C(21)	15(4)	27(4)	15(4)	-1(3)	2(3)	-1(3)
C(22)	38(5)	21(4)	18(4)	4(3)	6(4)	2(4)
C(23)	31(5)	35(5)	20(4)	4(3)	6(4)	7(4)
C(24)	14(4)	49(5)	21(4)	1(4)	2(3)	-3(4)
C(25)	26(4)	39(6)	25(4)	-5(4)	8(3)	-9(4)
C(26)	24(4)	23(5)	24(4)	-2(3)	5(3)	-3(3)
C(31)	17(4)	16(4)	21(4)	-6(3)	0(3)	0(3)
C(32)	18(4)	28(5)	28(4)	-10(4)	1(3)	-2(3)
C(33)	36(5)	22(4)	49(6)	-1(4)	-2(4)	-4(4)
C(34)	47(6)	23(5)	37(5)	-10(4)	-4(4)	14(4)
C(35)	18(4)	57(6)	28(5)	-12(4)	0(4)	10(4)
C(36)	34(5)	34(5)	16(4)	-1(3)	1(3)	7(4)
C(41)	14(4)	21(4)	17(3)	-3(3)	6(3)	-5(3)
C(42)	25(4)	33(5)	17(4)	2(3)	6(3)	-2(4)
C(43)	46(6)	25(4)	25(5)	-6(3)	12(4)	-1(4)
C(44)	63(7)	46(6)	15(4)	-9(4)	9(4)	-13(5)
C(45)	61(7)	43(6)	23(4)	6(4)	11(4)	-25(5)
C(46)	41(5)	35(5)	26(4)	-6(4)	11(4)	-17(4)
C(51)	20(4)	18(4)	12(3)	-4(3)	6(3)	-2(3)
C(52)	18(4)	22(4)	21(4)	-3(3)	7(3)	-2(3)
C(53)	35(5)	22(4)	27(4)	0(4)	14(4)	-2(4)
C(54)	31(5)	29(5)	25(4)	-6(3)	15(4)	-14(4)
C(55)	24(4)	36(5)	33(5)	-12(4)	18(4)	-8(4)
C(56)	29(4)	17(4)	20(4)	-3(3)	8(3)	1(3)
C(61)	12(4)	16(4)	22(4)	0(3)	3(3)	-3(3)
C(62)	27(4)	20(4)	19(4)	-5(3)	10(3)	2(3)
C(63)	37(5)	23(4)	23(4)	7(3)	6(4)	0(4)
C(64)	25(4)	20(4)	33(5)	-3(4)	-6(4)	5(4)
C(65)	29(5)	27(4)	29(4)	-6(4)	10(4)	6(4)

P3006-6

7

C(66)	26(4)	32(5)	19(4)	3(3)	2(3)	1(4)
B(1)	72(10)	45(8)	65(9)	17(7)	28(8)	-5(7)
F(1)	34(3)	165(8)	62(4)	32(5)	21(3)	-14(4)
F(2)	132(8)	159(9)	101(6)	-11(6)	56(6)	-46(7)
F(3)	66(5)	77(5)	154(7)	74(5)	30(5)	17(4)
F(4)	71(4)	87(5)	63(4)	50(4)	46(3)	39(4)

P3006-7 8

[Table 5] Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$ x 10^3) for 9.

	x	y	z	U(eq)
H(1IR)	2367(64)	2675(108)	333(42)	94(39)
H(2A)	1682(45)	4188(74)	429(29)	22(21)
H(1A)	1531(4)	2464(8)	-459(3)	29(4)
H(2B)	559(5)	1610(8)	-1181(3)	29(4)
H(3A)	29(5)	-387(9)	-1082(3)	29(4)
H(4A)	500(5)	-1523(9)	-271(3)	29(4)
H(6A)	1264(5)	6096(8)	219(3)	29(4)
H(7A)	279(5)	6971(8)	495(3)	29(4)
H(8A)	-378(5)	5710(9)	948(3)	29(4)
H(9A)	20(5)	3613(9)	1157(3)	29(4)
H(12A)	2142(5)	-23(8)	1756(3)	29(4)
H(13A)	1890(6)	-1136(9)	2459(3)	29(4)
H(14A)	2560(6)	-496(8)	3348(3)	29(4)
H(15A)	3452(5)	1138(8)	3512(3)	29(4)
H(16A)	3701(4)	2249(8)	2813(3)	29(4)
H(22A)	4349(5)	292(7)	2092(3)	29(4)
H(23A)	5728(5)	256(8)	2328(3)	29(4)
H(24A)	6429(5)	2090(9)	2286(3)	29(4)
H(25A)	5750(5)	3990(8)	2019(3)	29(4)
H(26A)	4370(4)	4057(7)	1745(3)	29(4)
H(32A)	3229(5)	4564(8)	1184(3)	29(4)
H(33A)	2735(5)	6606(8)	1216(4)	29(4)
H(34A)	1965(5)	7032(8)	1759(3)	29(4)
H(35A)	1779(5)	5427(9)	2332(3)	29(4)
H(36A)	2286(5)	3417(8)	2312(3)	29(4)
H(42A)	2700(4)	-1332(8)	-132(3)	29(4)
H(43A)	2251(5)	-1881(8)	-1031(3)	29(4)
H(44A)	2420(6)	-481(9)	-1671(3)	29(4)
H(45A)	2964(6)	1525(9)	-1424(3)	29(4)
H(46A)	3435(5)	2079(8)	-514(3)	29(4)
H(52A)	3450(4)	3473(7)	287(3)	29(4)
H(53A)	4378(5)	5038(8)	337(3)	29(4)
H(54A)	5728(5)	4550(8)	665(3)	29(4)
H(55A)	6132(5)	2476(8)	920(3)	29(4)
H(56A)	5191(4)	898(7)	873(3)	29(4)
H(62A)	3513(4)	-894(7)	1394(3)	29(4)
H(63A)	4246(5)	-2696(8)	1763(3)	29(4)
H(64A)	5048(5)	-3642(8)	1335(3)	29(4)
H(65A)	5207(5)	-2736(8)	565(3)	29(4)
H(66A)	4521(5)	-892(8)	210(3)	29(4)