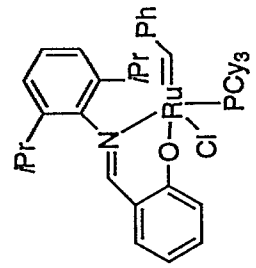
ORGANOMETALLICS
Organometallics, 1998, 17(16), 3460-3465, DOI:10.1021/om970910y



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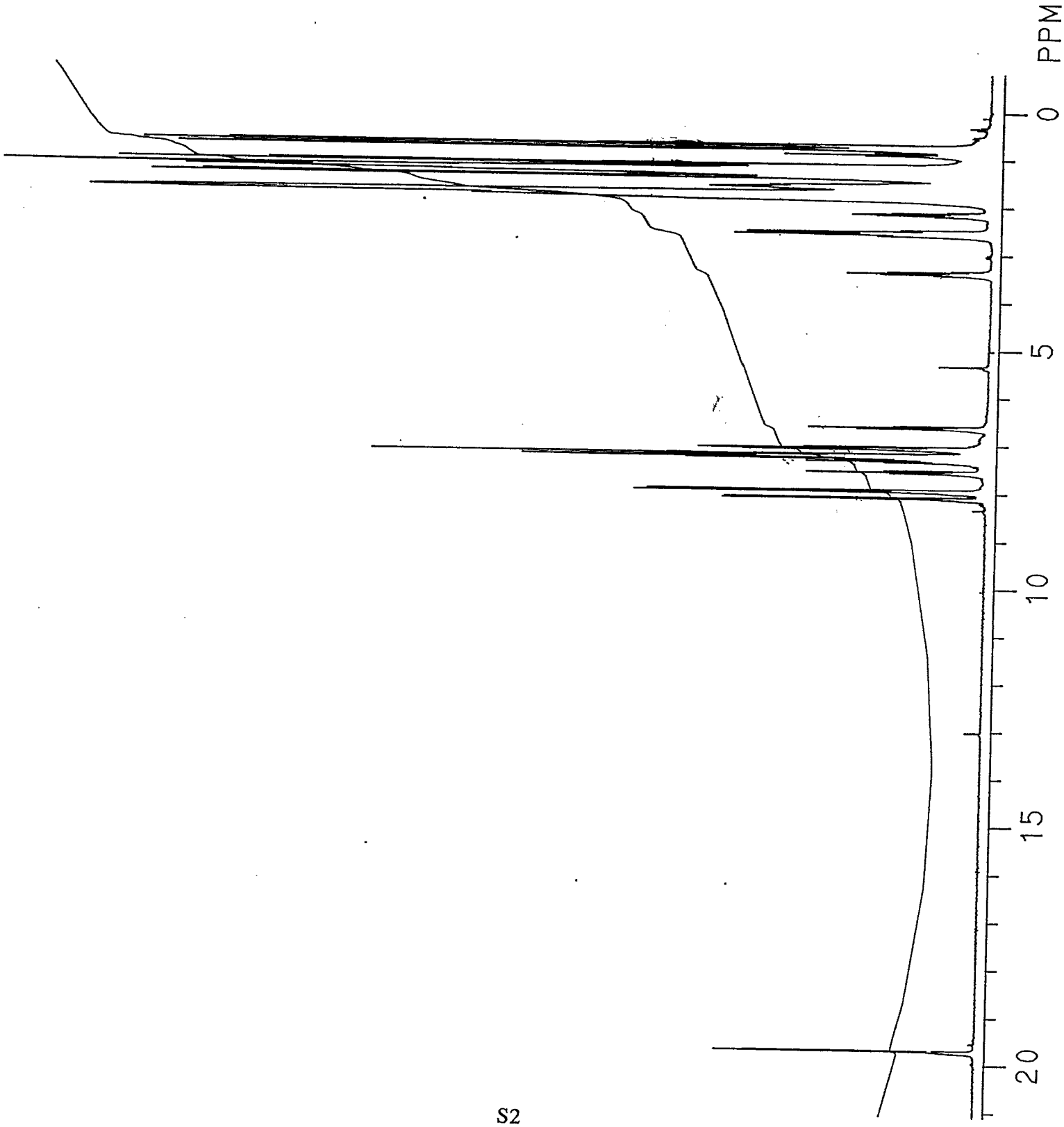

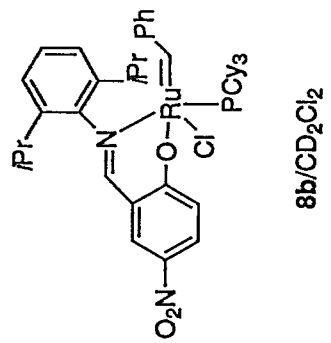

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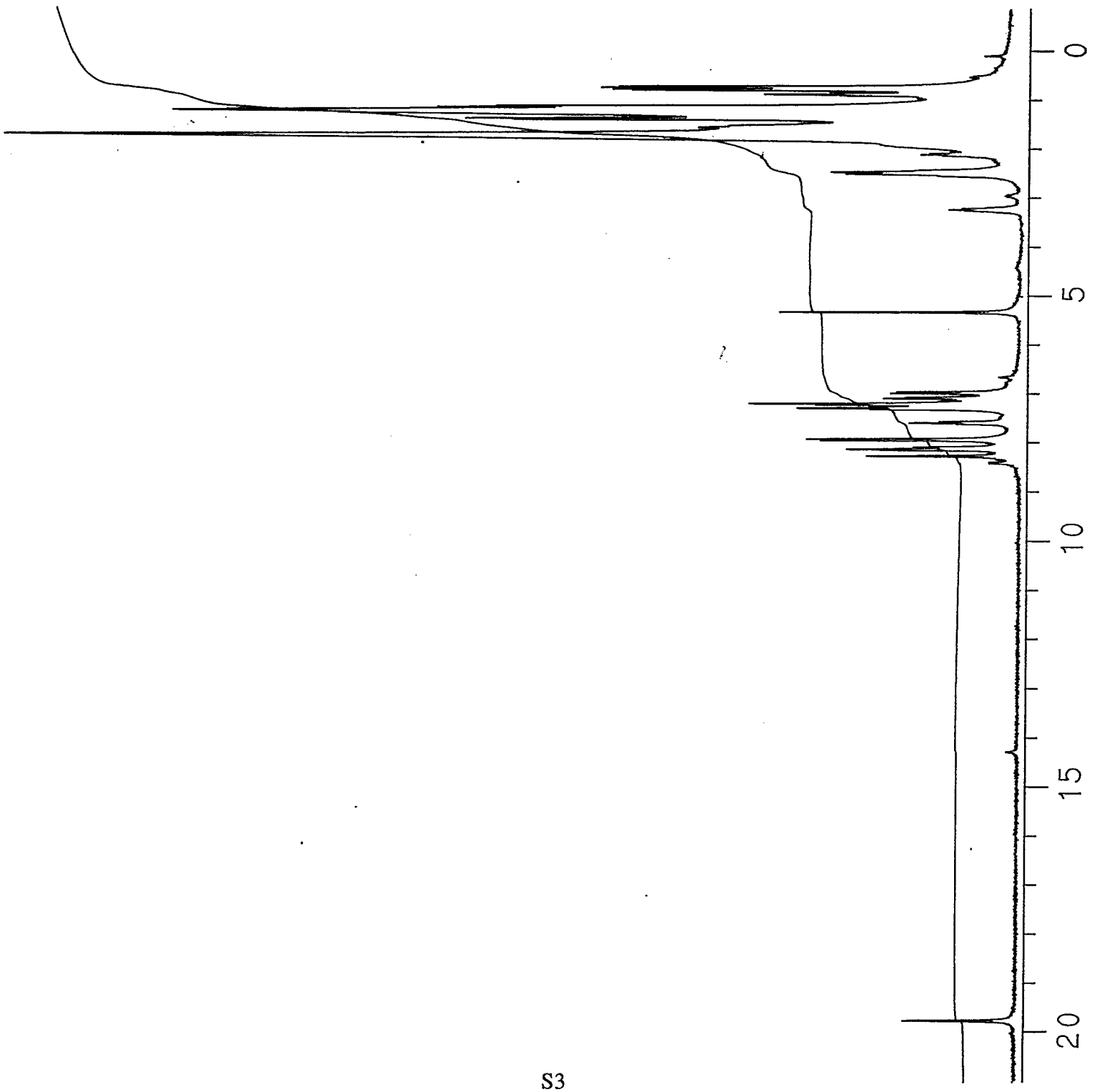

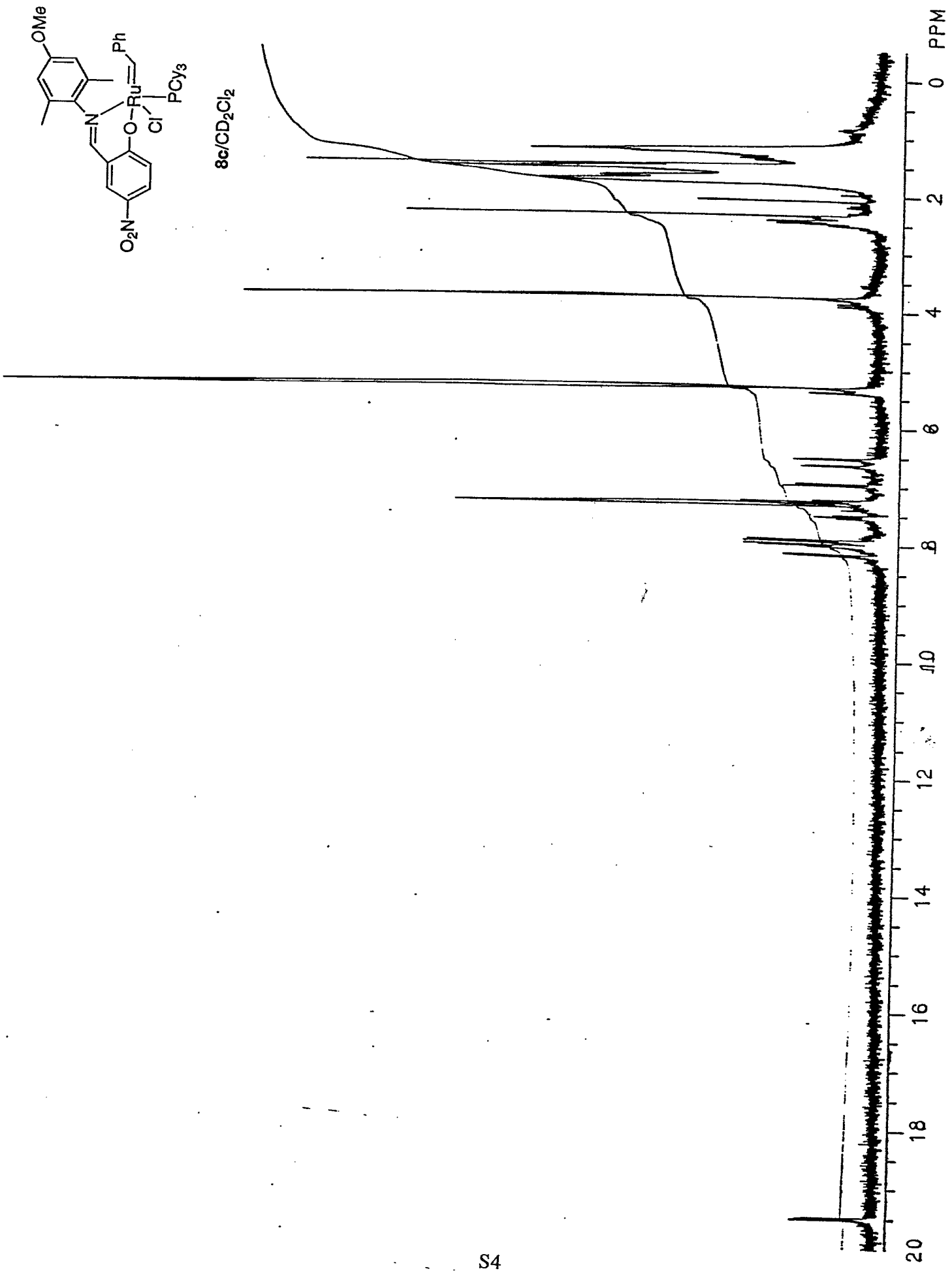

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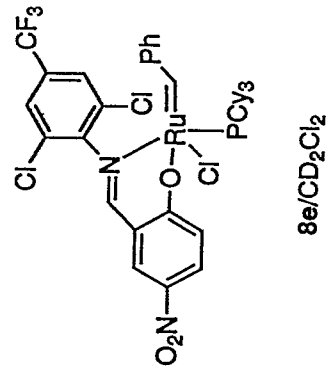

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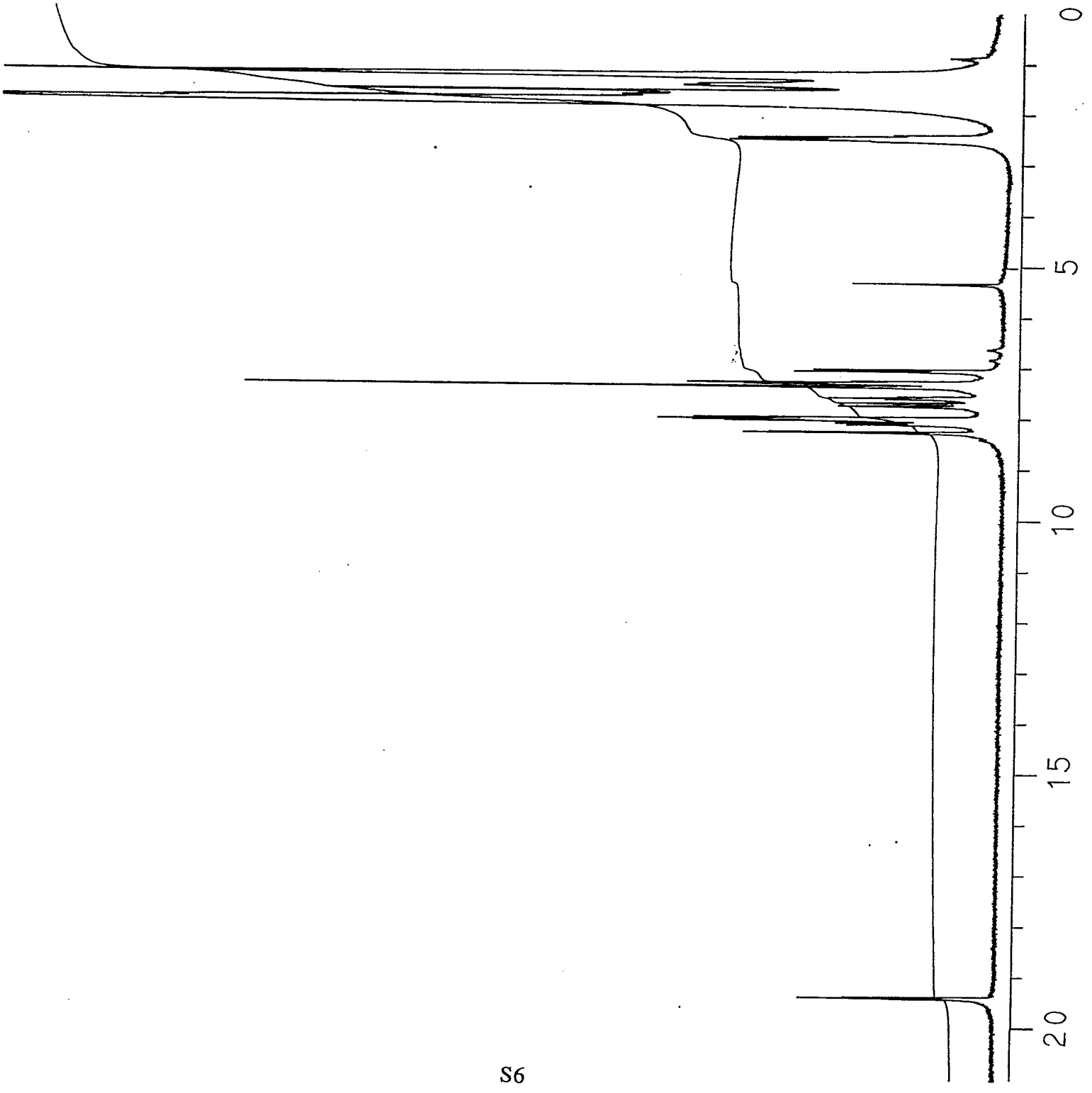

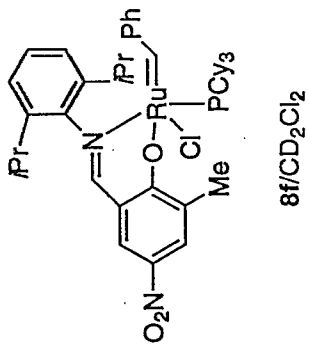

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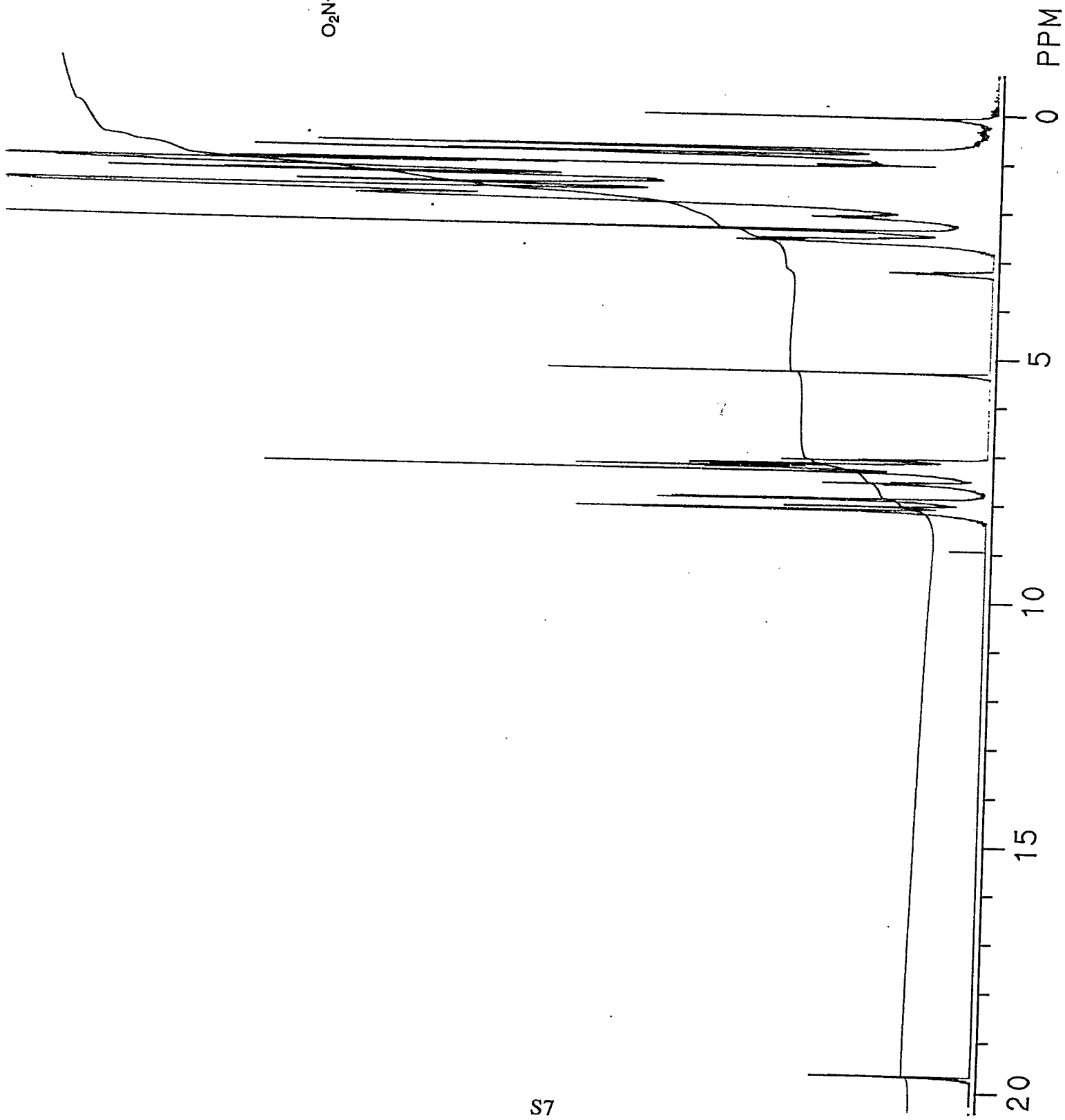

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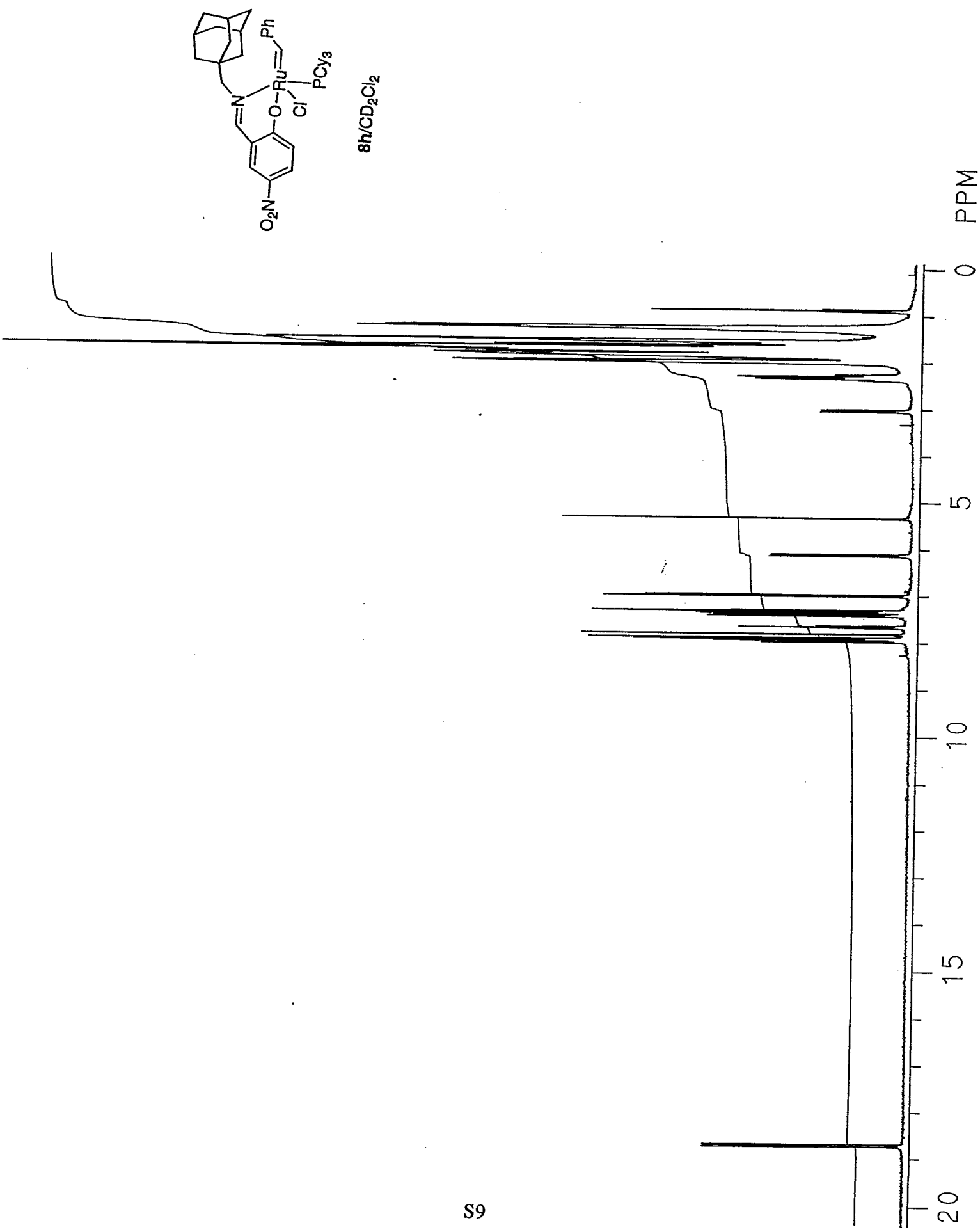
8S

20 15 10 5 0 PPM


Ph
PCy3
Ru
Cl
O
N
O2N
8h/CD2Cl2
PPM
0
5
10
15
20

$C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

Benzylidene-chloro-(tricyclohexyl-phosphine)
(N-2,6-diisopropyl-5-nitro-salicylideniminato)ruthenium

Data Collection

A crystal was cut from a small cluster, mounted with Paratone on a glass fiber, and placed in the cold stream of the CAD-4. The unit cell was determined from the setting angles of 25 reflections. The peaks were broad, up to 1.8° in the centering. Two equivalents sets of data were collected.

Solution and Refinement

No decay was observed. No absorption corrections were made as the ψ-scans of 6 reflections showed only noise. Individual backgrounds were replaced with a background function of 2θ derived from the backgrounds of reflections with$I < 3\sigma(\mathrm{I})$. Lorentz and polarization factors were applied and the data then merged in point group $2/m$ with CRYM programs. Weights w were calculated as $1/\sigma^2(\mathrm{F_o^2})$; variances $(\sigma^2(\mathrm{F_o^2}))$ were derived from counting statistics plus an additional term, $(0.014\mathrm{I})^2$; variances of the merged data were obtained by propagation of error plus another additional term, $(0.014\bar{\mathrm{I}})^2$.

The structure was solved with SHELXS-86 using direct methods.

The hydrogen atoms were originally placed at calculated positions. Eventually the coordinates of all but two (H38a and H38b, see below) were refined, with U_{iso}'s fixed at 1.2 times the U_{eq} of the attached atom. Refinement was full-matrix least-squares using SHELXL-93.

Definitions

$$R = \frac{\Sigma||F_o| - |F_c||}{\Sigma|F_o|}; \quad R_w = \left\{\frac{\Sigma w(F_o^2 - F_c^2)^2}{\Sigma w(F_o^2)^2}\right\}^{\frac{1}{2}}$$

$$S = \left\{\frac{\Sigma w(F_o^2 - F_c^2)^2}{n-p}\right\}^{\frac{1}{2}}$$

where n = number of data,

p = number of parameters refined.

Comment

In the final difference map, there was one fairly large peak (1.27 $eÅ^{-3}$) 1.31 Å from the phosphorus atom. This peak may be due to absorption. (The next largest peak is 0.81 $eÅ^{-3}$.) Because the hydrogen atoms on C38 wanted to migrate to this area, H38a and H38b were fixed. (Normally, all similar hydrogen atoms are treated the same, but here it is clear why the atoms move to chemically unreasonable positions.

The crystal contains solvent around a center of symmetry. There is no unambiguous interpretation of this region and the model finally chosen may not correspond to reality. The presence of the center and the distance to the closest peaks make isopropyl ether an unreasonable choice. This area was modelled with a water and a dichloromethane. These molecules were refined isotropically (with riding hydrogen atoms on the dichloromethane); with a fractional population parameter for each molecule also refined. The chlorine (Cl98) closest to the center has the largest displacement parameter; this is consistent with disorder of the site dependent on the occupation of neighboring solvent sites.

References

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XP/PC

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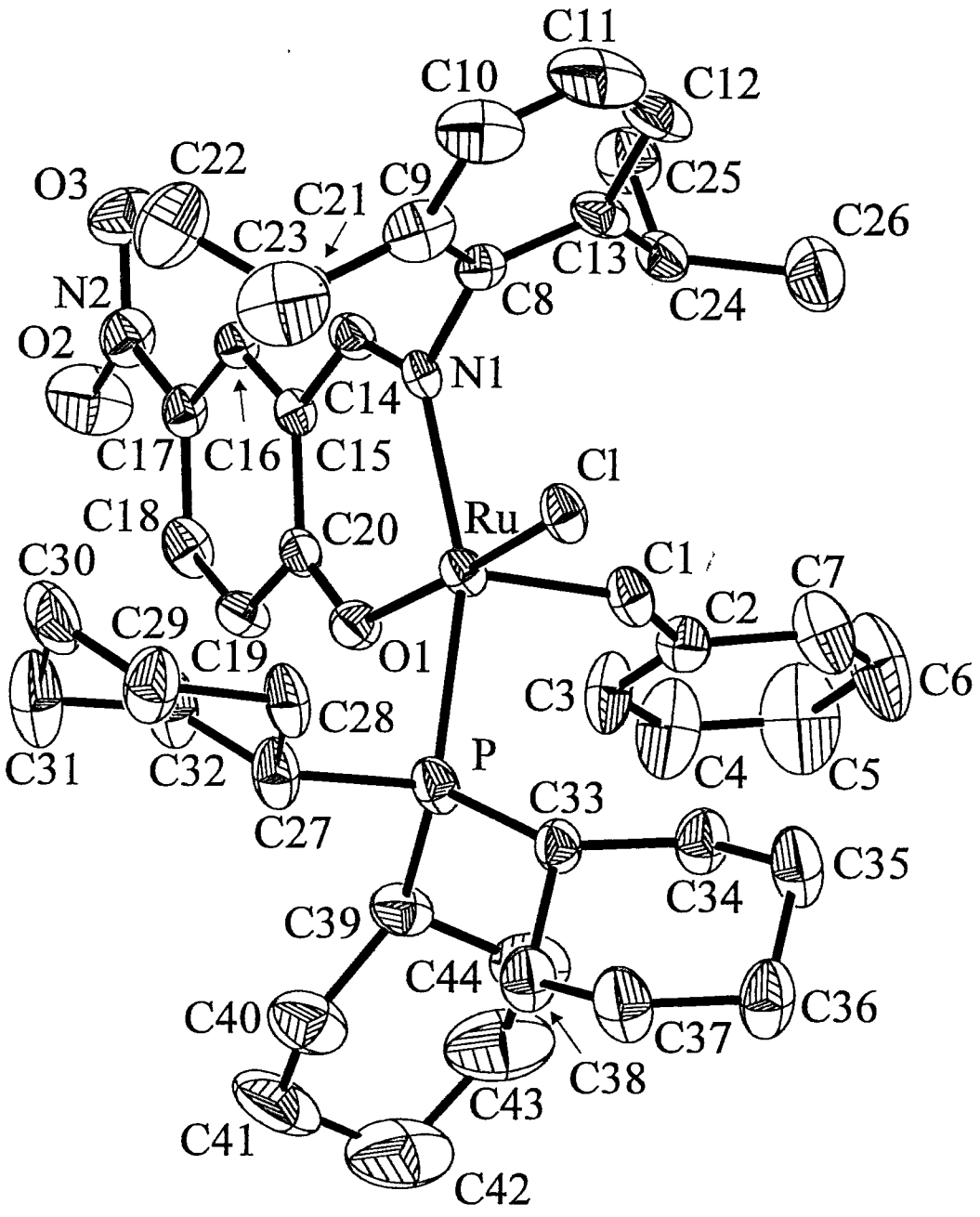
C11
C10
C12
C22
O3
C9
C25
C21
C23
C26
C13
N2
C8
C24
O2
N1
C14
C17
C16
C15
Cl
C18
C20
Ru
C30
C1
C7
C29
C2
C19
O1
C6
C3
C28
C31
C32
P
C4
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C35
C34
C39
C44
C36
C40
C37
C38
C43
C41
C42

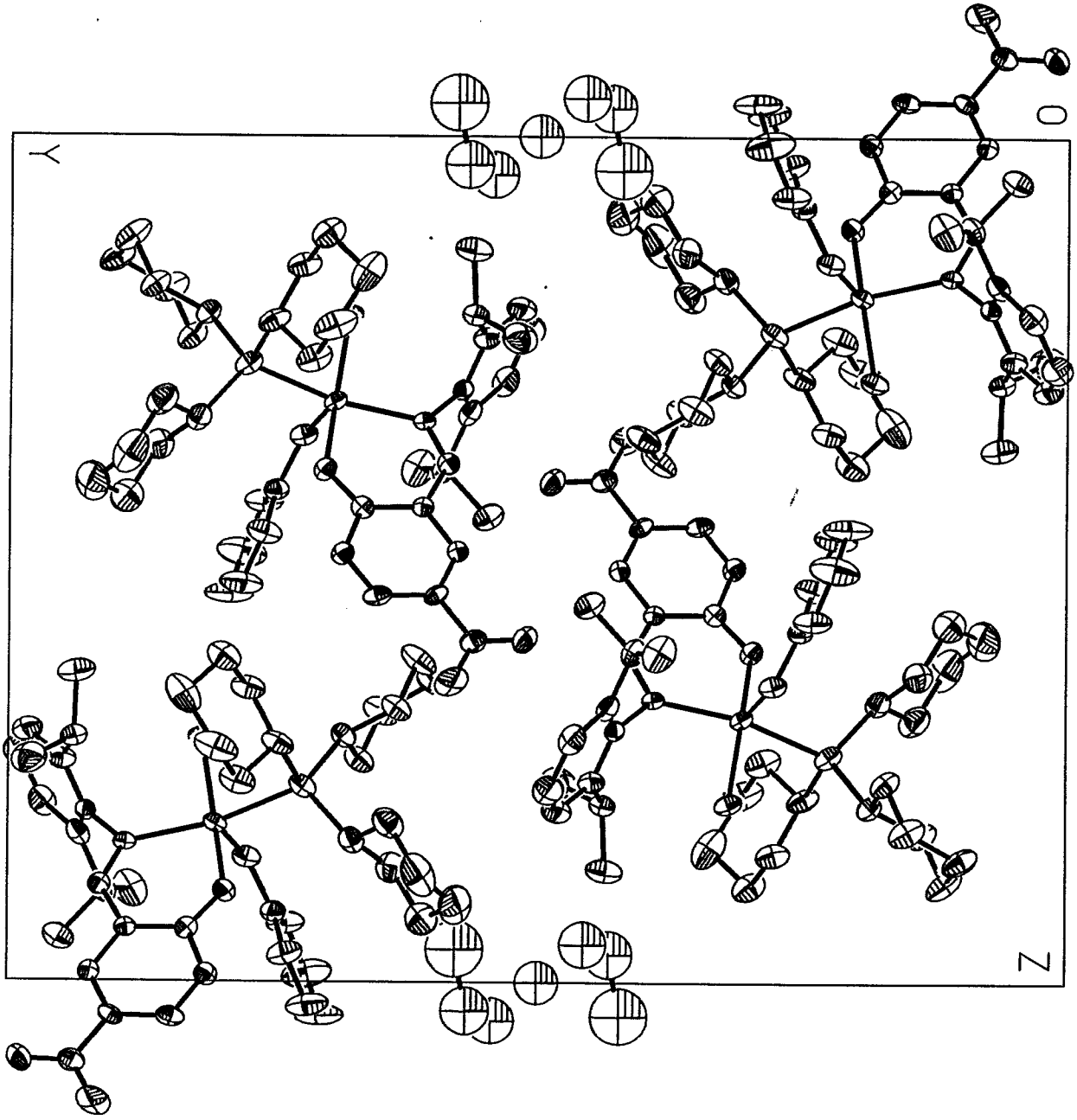
Y
O
Z

Table 1. Crystal and Intensity Collection Data for $C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

Formula: $C_{44.31}H_{61.16}ClN_2O_{3.27}PRu$	Formula (compound) weight: 863.53 (832.47)
Crystal color: Dark brown	Habit: Prismatic
Crystal size: $0.10 \times 0.13 \times 0.44$ mm	$\rho_{calc} = 1.302$ g cm^{-3}
Crystal System: Monoclinic	Space group: $P2_1/c$ (#14)
$a = 9.123(4)$Å	
$b = 24.320(7)$Å	$\beta = 91.67(3)°$
$c = 19.863(5)$Å	
V = 4405(3)Å^3	$Z = 4$
Lattice parameters: 25 reflections,	$12° \leq \theta \leq 16°$
$\mu = 5.30$ cm^{-1} ($\mu r_{max} = 0.13$)	Absorption correction: None
CAD-4 diffractometer	ω scan
MoKα, $\lambda = 0.7107$Å	Graphite monochromator
2θ range: 3°–50°	$0 \leq h \leq 10, -28 \leq k \leq 28, -23 \leq l \leq 23$
T = 160K	$F_{000} = 1815$
Number of reflections measured: 17106	Number of independent reflections: 7741
Number with $F_o^2 > 0$: 7095	Number with $F_o^2 > 2\sigma(F_o^2)$: 5735
Standard reflections: 3 every 1.25 hrs	Variation: Within counting statistics
GOF_{merge} : 0.98 for 7549 multiples	R_{merge} : 0.055 for 5987 duplicates
Number used in refinement: 7741	Criterion: All reflections used

Final R(F_o): 0.079 for 5735 reflections with $F_o^2 > 2\sigma(F_o^2)$

Final R(F_o): 0.114 for 7741 reflections

Final weighted R(F_o^2) : 0.121 for 7741 reflections

Final goodness of fit: 1.64 for 658 parameters and 7741 reflections

$(\Delta/\sigma)_{max}$ in final least squares cycle: -0.001

$\Delta\rho_{max}$: 1.27 eÅ^{-3}, $\Delta\rho_{min}$: -0.55 eÅ^{-3} in final difference map

Table 2. Final Heavy Atom Parameters for

$C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

x, y, z and $U_{eq}{}^a \times 10^4$

Atom	x	y	z	U_{eq} or B
Ru	32828(6)	30567(2)	68735(2)	236(1)
Cl	4727(2)	3182(1)	7880(1)	291(4)
C1	4912(7)	2760(3)	6475(3)	296(15)
C2	5197(7)	2504(2)	5832(3)	300(15)
C3	6691(8)	2388(3)	5703(4)	480(20)
C4	7044(9)	2181(4)	5096(4)	720(30)
C5	5955(10)	2061(4)	4604(4)	740(30)
C6	4563(9)	2174(4)	4726(4)	610(20)
C7	4161(8)	2391(3)	5338(4)	430(20)
N1	3423(5)	3900(2)	6646(2)	201(11)
O1	1817(4)	2966(2)	6076(2)	278(10)
C8	4457(6)	4266(2)	7013(3)	241(14)
C9	4031(8)	4487(3)	7626(3)	410(20)
C10	5058(10)	4817(3)	7972(4)	530(20)
C11	6419(11)	4898(3)	7720(4)	620(30)
C12	6824(8)	4670(3)	7117(4)	440(20)
C13	5843(7)	4335(2)	6745(3)	320(20)
C14	2712(7)	4134(2)	6145(3)	248(14)
C15	1774(6)	3892(2)	5635(3)	215(13)
C16	1270(6)	4227(2)	5110(3)	232(13)

Table 2. (Cont.)

Atom	x	y	z	U_{eq} or B
C17	418(6)	4021(2)	4590(3)	250(14)
C18	15(7)	3464(3)	4578(3)	300(20)
C19	476(7)	3135(3)	5082(3)	280(15)
C20	1383(6)	3319(2)	5630(3)	240(13)
C21	2556(9)	4381(3)	7898(3)	420(20)
C22	1430(11)	4802(4)	7629(5)	640(30)
C23	2556(12)	4360(4)	8671(4)	670(30)
C24	6247(7)	4093(3)	6083(3)	330(20)
C25	6083(9)	4514(3)	5521(4)	450(20)
C26	7802(10)	3845(4)	6092(5)	590(30)
N2	−51(6)	4381(2)	4044(3)	332(13)
O2	−736(6)	4172(2)	3564(2)	590(20)
O3	293(5)	4872(2)	4067(2)	422(12)
P	2394(2)	2232(1)	7316(1)	306(4)
C27	834(7)	2471(3)	7826(3)	370(20)
C28	1308(7)	2754(3)	8484(3)	390(20)
C29	0(8)	2980(3)	8852(4)	490(20)
C30	−905(9)	3362(4)	8403(5)	610(30)
C31	−1451(8)	3051(4)	7776(4)	620(20)
C32	−132(8)	2851(4)	7384(4)	480(20)

Table 2. (Cont.)

Atom	x	y	z	U_{eq} or B
C33	1520(7)	1767(3)	6678(3)	370(20)
C34	194(10)	1423(3)	6881(4)	540(20)
C35	−500(11)	1133(4)	6271(6)	770(30)
C36	569(14)	781(4)	5921(6)	870(40)
C37	1952(15)	1101(4)	5748(5)	820(40)
C38	2646(9)	1393(3)	6356(4)	540(20)
C39	3618(6)	1847(3)	7917(3)	287(15)
C40	2936(8)	1350(3)	8245(4)	400(20)
C41	3967(8)	1139(4)	8819(4)	520(20)
C42	5455(8)	993(3)	8551(4)	480(20)
C43	6129(8)	1490(4)	8209(4)	490(20)
C44	5101(8)	1699(3)	7635(4)	390(20)
O99	5000	5000	0	8.5(8) *
C99	3782(44)	5683(16)	335(19)	10.3(12)*
Cl98	4303(17)	5786(6)	−397(8)	14.6(6) *
Cl99	2207(13)	5437(4)	453(5)	9.8(4) *

[a] $U_{eq} = \frac{1}{3}\sum_i \sum_j [U_{ij}(a_i^* a_j^*)(\vec{a}_i \cdot \vec{a}_j)]$

* Isotropic displacement parameter, B

Table 3. Selected Distances and Angles for

$C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

	Distance(Å)		Distance(Å)
Ru –C1	1.850(6)	C11 –C12	1.378(11)
Ru –O1	2.055(4)	C12 –C13	1.406(8)
Ru –N1	2.106(4)	C13 –C24	1.497(9)
Ru –P	2.345(2)	C14 –C15	1.433(8)
Ru –Cl	2.382(2)	C15 –C16	1.391(7)
C1 –C2	1.451(8)	C15 –C20	1.439(8)
C2 –C7	1.371(9)	C16 –C17	1.370(8)
C2 –C3	1.423(9)	C17 –C18	1.404(8)
C3 –C4	1.354(10)	C17 –N2	1.448(7)
C4 –C5	1.404(11)	C18 –C19	1.340(8)
C5 –C6	1.329(11)	C19 –C20	1.419(8)
C6 –C7	1.384(10)	C21 –C22	1.531(11)
N1 –C14	1.301(7)	C21 –C23	1.535(10)
N1 –C8	1.473(7)	C24 –C25	1.520(9)
O1 –C20	1.288(6)	C24 –C26	1.540(10)
C8 –C13	1.396(8)	N2 –O2	1.234(6)
C8 –C9	1.398(8)	N2 –O3	1.235(6)
C9 –C10	1.399(10)	P –C33	1.860(6)
C9 –C21	1.492(10)	P –C39	1.862(6)
C10 –C11	1.367(11)	P –C27	1.864(7)

Table 3. (Cont.)

	Distance(Å)				Angle(°)
C27	–C28	1.528(9)	C1	–Ru –O1	98.1(2)
C27	–C32	1.536(9)	C1	–Ru –N1	103.5(2)
C28	–C29	1.521(9)	O1	–Ru –N1	88.9(2)
C29	–C30	1.516(10)	C1	–Ru –P	96.8(2)
C30	–C31	1.528(12)	O1	–Ru –P	88.4(1)
C31	–C32	1.530(10)	N1	–Ru –P	159.8(1)
C33	–C38	1.527(9)	C1	–Ru –Cl	88.7(2)
C33	–C34	1.535(10)	O1	–Ru –Cl	173.0(1)
C34	–C35	1.524(12)	N1	–Ru –Cl	91.2(1)
C35	–C36	1.484(15)	P	–Ru –Cl	89.0(1)
C36	–C37	1.53(2)	C2	–C1 –Ru	135.3(5)
C37	–C38	1.522(12)	C7	–C2 –C3	118.4(6)
C39	–C40	1.516(8)	C7	–C2 –C1	125.4(6)
C39	–C44	1.523(9)	C3	–C2 –C1	116.2(6)
C40	–C41	1.542(9)	C4	–C3 –C2	119.2(7)
C41	–C42	1.515(10)	C3	–C4 –C5	121.0(8)
C42	–C43	1.523(10)	C6	–C5 –C4	119.4(8)
C43	–C44	1.542(9)	C5	–C6 –C7	121.2(8)
			C2	–C7 –C6	120.7(7)
			C14	–N1 –C8	114.4(5)

Table 3. (Cont.)

	Angle(°)		Angle(°)
C14 –N1 –Ru	123.8(4)	C16 –C17 –C18	120.5(5)
C8 –N1 –Ru	121.5(3)	C16 –C17 –N2	119.5(5)
C20 –O1 –Ru	129.7(4)	C18 –C17 –N2	120.0(5)
C13 –C8 –C9	124.4(6)	C19 –C18 –C17	119.2(6)
C13 –C8 –N1	117.2(5)	C18 –C19 –C20	123.2(6)
C9 –C8 –N1	118.2(6)	O1 –C20 –C19	118.6(5)
C8 –C9 –C10	116.8(7)	O1 –C20 –C15	124.7(5)
C8 –C9 –C21	121.9(6)	C19 –C20 –C15	116.7(5)
C10 –C9 –C21	121.4(7)	C9 –C21 –C22	111.1(6)
C11 –C10 –C9	120.2(7)	C9 –C21 –C23	113.0(7)
C10 –C11 –C12	122.1(7)	C22 –C21 –C23	110.8(7)
C11 –C12 –C13	120.4(7)	C13 –C24 –C25	111.0(6)
C8 –C13 –C12	116.1(6)	C13 –C24 –C26	113.2(6)
C8 –C13 –C24	122.5(5)	C25 –C24 –C26	110.1(6)
C12 –C13 –C24	121.4(6)	O2 –N2 –O3	123.3(5)
N1 –C14 –C15	129.4(5)	O2 –N2 –C17	117.5(5)
C16 –C15 –C14	117.8(5)	O3 –N2 –C17	119.2(5)
C16 –C15 –C20	119.1(5)	C33 –P –C39	111.7(3)
C14 –C15 –C20	123.1(5)	C33 –P –C27	103.9(3)
C17 –C16 –C15	121.3(5)	C39 –P –C27	105.2(3)

Table 3. (Cont.)

	Angle(°)		Angle(°)
C33 –P	–Ru	114.2(2)	C40 –C39 –P	115.5(4)
C39 –P	–Ru	117.5(2)	C44 –C39 –P	113.9(4)
C27 –P	–Ru	102.4(2)	C39 –C40 –C41	109.5(6)
C28 –C27	–C32	111.1(6)	C42 –C41 –C40	110.6(6)
C28 –C27	–P	113.8(5)	C41 –C42 –C43	110.3(6)
C32 –C27	–P	108.2(4)	C42 –C43 –C44	110.3(6)
C29 –C28	–C27	111.6(6)	C39 –C44 –C43	109.5(5)
C30 –C29	–C28	111.0(6)		
C29 –C30	–C31	109.7(7)		
C30 –C31	–C32	109.2(7)		
C31 –C32	–C27	110.3(6)		
C38 –C33	–C34	109.3(6)		
C38 –C33	–P	111.6(5)		
C34 –C33	–P	118.6(5)		
C35 –C34	–C33	110.9(7)		
C36 –C35	–C34	112.0(8)		
C35 –C36	–C37	111.7(8)		
C38 –C37	–C36	112.7(9)		
C37 –C38	–C33	109.9(7)		
C40 –C39	–C44	110.4(5)		

Table 4. Anisotropic Displacement Parameters for $C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ru	21.4(2)	28.6(3)	20.9(2)	−2.9(3)	−0.5(2)	5.8(2)
Cl	20.1(8)	42.3(10)	24.6(8)	−1.1(7)	−2.3(6)	7.3(7)
C1	21(3)	43(4)	25(3)	−2(3)	−1(3)	5(3)
C2	32(4)	35(4)	24(3)	1(3)	5(3)	0(3)
C3	35(4)	81(6)	27(4)	−4(4)	4(3)	−2(4)
C4	34(5)	136(9)	47(5)	−2(5)	11(4)	−19(5)
C5	70(6)	132(9)	22(4)	15(6)	2(4)	−3(5)
C6	52(6)	101(7)	28(4)	24(5)	−7(4)	−11(4)
C7	29(4)	67(5)	32(4)	17(4)	−6(3)	−11(4)
N1	15(3)	28(3)	17(2)	−2(2)	0(2)	1(2)
O1	27(2)	27(2)	29(2)	−2(2)	−2(2)	4(2)
C8	27(3)	22(3)	23(3)	1(3)	−10(3)	2(3)
C9	57(5)	39(4)	26(4)	1(4)	−10(4)	1(3)
C10	78(7)	49(5)	31(4)	−13(5)	−9(4)	−4(4)
C11	83(7)	53(5)	48(5)	−29(5)	−37(5)	3(4)
C12	38(5)	41(4)	53(5)	−19(4)	−17(4)	9(4)
C13	28(4)	29(4)	37(4)	−10(3)	−14(3)	11(3)
C14	22(3)	26(3)	27(3)	−4(3)	6(3)	−1(3)
C15	18(3)	25(3)	21(3)	−1(3)	3(3)	0(3)
C16	20(3)	24(3)	26(3)	−1(3)	3(3)	0(3)
C17	21(3)	31(3)	23(3)	3(3)	−3(3)	9(3)
C18	23(4)	44(4)	22(3)	−3(3)	−9(3)	4(3)
C19	26(3)	28(4)	30(3)	−7(3)	−2(3)	2(3)
C20	17(3)	28(3)	27(3)	−2(3)	2(3)	−2(3)
C21	62(5)	38(4)	25(4)	4(4)	6(4)	−3(3)
C22	70(7)	69(6)	54(5)	21(5)	8(5)	−7(5)
C23	94(8)	77(7)	31(5)	0(6)	13(5)	−8(4)
C24	25(4)	32(4)	42(4)	−5(3)	5(3)	8(3)
C25	43(5)	52(5)	39(4)	−2(4)	6(4)	11(4)
C26	37(5)	62(6)	78(7)	3(4)	12(5)	14(5)
N2	31(3)	38(3)	30(3)	3(3)	−6(3)	5(3)
O2	64(4)	62(3)	48(3)	−14(3)	−41(3)	14(3)
O3	53(3)	33(3)	39(3)	2(2)	−9(2)	8(2)
P	21.4(9)	39.2(1)	31.2(9)	−1.4(8)	−1.4(7)	10.1(8)
C27	25(4)	57(5)	31(4)	7(3)	6(3)	15(3)
C28	21(4)	63(5)	32(4)	3(3)	8(3)	7(3)
C29	39(4)	60(6)	48(5)	14(4)	15(4)	14(4)
C30	28(5)	66(6)	88(7)	−3(4)	19(5)	32(5)
C31	31(4)	103(7)	53(5)	17(5)	5(4)	26(5)

Table 4. (Cont.)

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C32	28(4)	70(6)	46(5)	8(4)	1(4)	13(4)
C33	37(4)	34(4)	38(4)	−7(3)	−15(3)	6(3)
C34	57(6)	53(6)	53(5)	−15(4)	−6(4)	15(4)
C35	55(6)	68(7)	105(8)	−34(5)	−37(6)	26(6)
C36	111(9)	60(7)	88(8)	−20(7)	−53(7)	−5(6)
C37	128(11)	57(7)	60(7)	−12(7)	−17(7)	2(5)
C38	65(6)	48(5)	50(5)	−10(4)	−3(4)	1(4)
C39	22(3)	32(4)	31(3)	2(3)	−1(3)	7(3)
C40	35(4)	47(5)	38(4)	5(4)	4(3)	15(3)
C41	36(5)	67(6)	52(5)	3(4)	6(4)	33(4)
C42	37(4)	65(6)	41(5)	16(4)	3(4)	23(4)
C43	28(4)	72(6)	48(5)	7(4)	7(4)	19(4)
C44	33(4)	45(4)	40(4)	8(3)	6(3)	15(3)

$U_{i,j}$ values have been multiplied by 10^3

The form of the displacement factor is:

$$\exp -2\pi^2(U_{11}h^2a^{*^2} + U_{22}k^2b^{*^2} + U_{33}\ell^2c^{*^2} + 2U_{12}hka^*b^* + 2U_{13}h\ell a^*c^* + 2U_{23}k\ell b^*c^*)$$

Table 5. Final Hydrogen Parameters for $C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

	x, y and $z \times 10^4$			
Atom	x	y	z	B
H1	5760(65)	2771(22)	6756(28)	2.8
H3	7312(75)	2446(25)	6101(34)	4.5
H4	8077(88)	2116(31)	5078(38)	6.9
H5	6171(81)	1905(30)	4001(37)	7.0
H6	3844(78)	2068(28)	4365(35)	5.8
H7	3387(76)	2448(29)	5401(35)	4.0
H10	4828(78)	4944(27)	8411(34)	5.0
H11	7207(84)	5083(29)	7905(36)	5.8
H12	7673(72)	4737(28)	6978(33)	4.2
H14	2832(62)	4485(22)	6157(27)	2.4
H16	1406(60)	4599(21)	5125(26)	2.2
H18	−474(63)	3351(22)	4192(28)	2.8
H19	269(65)	2790(23)	5113(28)	2.7
H21	2260(73)	4042(26)	7779(31)	4.0
H22a	1250(79)	4761(28)	7072(37)	6.1
H22b	507(86)	4734(32)	7804(38)	6.1
H22c	1784(83)	5190(30)	7693(36)	6.1
H23a	3097(88)	4028(31)	8849(38)	6.4
H23b	2913(86)	4767(31)	8831(36)	6.4
H23c	1711(87)	4235(33)	8860(39)	6.4
H24	5655(66)	3788(24)	5965(29)	3.2
H25a	4992(76)	4667(25)	5511(30)	4.3
H25b	6774(76)	4777(27)	5598(32)	4.3
H25c	6450(75)	4340(26)	5150(33)	4.3
H26a	7909(88)	3696(31)	6479(37)	5.6
H26b	7905(92)	3660(31)	5750(40)	5.6
H26c	8520(82)	4154(29)	6149(35)	5.6
H27	277(66)	2115(24)	7966(29)	3.5
H28a	2100(65)	3110(25)	8387(28)	3.6
H28b	1862(70)	2456(24)	8794(31)	3.6
H29a	685(73)	3231(25)	9216(32)	4.7
H29b	−639(78)	2709(27)	9010(34)	4.7
H30a	−315(83)	3693(28)	8313(35)	5.8
H30b	−1872(85)	3341(28)	8622(36)	5.8
H31a	−2054(81)	3281(28)	7448(37)	5.9
H31b	−2112(82)	2715(29)	7966(36)	5.9
H32a	−416(77)	2630(26)	6999(35)	4.6
H32b	576(74)	3167(26)	7337(32)	4.6
H33	1086(64)	2032(24)	6313(29)	3.5

Table 5. (Cont.)

Atom	*x*	*y*	*z*	*B*
H34a	−346(86)	1629(30)	7095(38)	5.1
H34b	551(75)	1153(27)	7272(34)	5.1
H35a	−846(87)	1466(31)	5909(40)	7.3
H35b	−1210(94)	929(35)	6399(42)	7.3
H36a	222(100)	601(34)	5509(43)	8.3
H36b	848(95)	459(34)	6275(42)	8.3
H37a	1609(99)	1390(33)	5453(42)	7.7
H37b	2490(112)	937(40)	5512(47)	7.7
H38a	3476	1610	6216	5.1
H38b	3000	1124	6682	5.1
H39	3755(63)	2155(22)	8257(27)	2.7
H40a	2723(68)	1044(24)	7859(31)	3.8
H40b	2160(70)	1520(25)	8492(31)	3.8
H41a	3565(75)	819(28)	9024(33)	4.9
H41b	4112(75)	1500(27)	9173(33)	4.9
H42a	5507(78)	749(27)	8195(34)	4.5
H42b	6076(74)	892(26)	8941(33)	4.5
H43a	6809(79)	1340(28)	7981(35)	4.7
H43b	6296(74)	1800(27)	8538(32)	4.7
H44a	5425(71)	1975(26)	7429(31)	3.7
H44b	4990(66)	1305(24)	7294(29)	3.7
H99a	3842	6031	573	12.4
H99b	4489	5438	553	12.4

Table 6. Complete Distances and Angles for $C_{44}H_{60}ClN_2O_3PRu \cdot 0.31CH_2Cl_2 \cdot 0.27H_2O$

	Distance(Å)		Distance(Å)
Ru –C1	1.850(6)	C17 –C18	1.404(8)
Ru –O1	2.055(4)	C17 –N2	1.448(7)
Ru –N1	2.106(4)	C18 –C19	1.340(8)
Ru –P	2.345(2)	C18 –H18	0.92(5)
Ru –Cl	2.382(2)	C19 –C20	1.419(8)
C1 –C2	1.451(8)	C19 –H19	0.86(5)
C1 –H1	0.94(6)	C21 –C22	1.531(11)
C2 –C7	1.371(9)	C21 –C23	1.535(10)
C2 –C3	1.423(9)	C21 –H21	0.90(6)
C3 –C4	1.354(10)	C22 –H22a	1.12(7)
C3 –H3	0.97(7)	C22 –H22b	0.93(8)
C4 –C5	1.404(11)	C22 –H22c	1.01(7)
C4 –H4	0.96(8)	C23 –H23a	1.01(7)
C5 –C6	1.329(11)	C23 –H23b	1.09(7)
C5 –H5	1.28(7)	C23 –H23c	0.92(8)
C6 –C7	1.384(10)	C24 –C25	1.520(9)
C6 –H6	0.99(7)	C24 –C26	1.540(10)
C7 –H7	0.73(7)	C24 –H24	0.94(6)
N1 –C14	1.301(7)	C25 –H25a	1.06(7)
N1 –C8	1.473(7)	C25 –H25b	0.91(7)
O1 –C20	1.288(6)	C25 –H25c	0.92(6)
C8 –C13	1.396(8)	C26 –H26a	0.85(7)
C8 –C9	1.398(8)	C26 –H26b	0.82(7)
C9 –C10	1.399(10)	C26 –H26c	1.00(7)
C9 –C21	1.492(10)	N2 –O2	1.234(6)
C10 –C11	1.367(11)	N2 –O3	1.235(6)
C10 –H10	0.95(7)	P –C33	1.860(6)
C11 –C12	1.378(11)	P –C39	1.862(6)
C11 –H11	0.92(7)	P –C27	1.864(7)
C12 –C13	1.406(8)	C27 –C28	1.528(9)
C12 –H12	0.85(6)	C27 –C32	1.536(9)
C13 –C24	1.497(9)	C27 –H27	1.05(6)
C14 –C15	1.433(8)	C28 –C29	1.521(9)
C14 –H14	0.86(5)	C28 –H28a	1.15(6)
C15 –C16	1.391(7)	C28 –H28b	1.07(6)
C15 –C20	1.439(8)	C29 –C30	1.516(10)
C16 –C17	1.370(8)	C29 –H29a	1.12(6)
C16 –H16	0.91(5)	C29 –H29b	0.94(7)

Table 6. (Cont.)

	Distance(Å)		Distance(Å)
C30 –C31	1.528(12)	C43 –H43a	0.86(7)
C30 –H30a	0.99(7)	C43 –H43b	1.01(6)
C30 –H30b	1.00(8)	C44 –H44a	0.84(6)
C31 –C32	1.530(10)	C44 –H44b	1.17(6)
C31 –H31a	1.01(7)	C99 –Cl98	1.56(4)
C31 –H31b	1.09(7)	C99 –Cl99	1.58(4)
C32 –H32a	0.96(6)	C99 –H99a	0.970
C32 –H32b	1.01(7)	C99 –H99b	0.970
C33 –C38	1.527(9)		
C33 –C34	1.535(10)		
C33 –H33	1.04(6)		
C34 –C35	1.524(12)		
C34 –H34a	0.83(7)		
C34 –H34b	1.06(7)		
C35 –C36	1.484(15)		
C35 –H35a	1.12(8)		
C35 –H35b	0.86(8)		
C36 –C37	1.53(2)		
C36 –H36a	0.97(8)		
C36 –H36b	1.08(8)		
C37 –C38	1.522(12)		
C37 –H37a	0.96(8)		
C37 –H37b	0.80(9)		
C38 –H38a	0.970		
C38 –H38b	0.970		
C39 –C40	1.516(8)		
C39 –C44	1.523(9)		
C39 –H39	1.01(5)		
C40 –C41	1.542(9)		
C40 –H40a	1.08(6)		
C40 –H40b	0.97(6)		
C41 –C42	1.515(10)		
C41 –H41a	0.96(7)		
C41 –H41b	1.13(6)		
C42 –C43	1.523(10)		
C42 –H42a	0.92(6)		
C42 –H42b	0.98(6)		
C43 –C44	1.542(9)		

Table 6. (Cont.)

	Angle(°)		Angle(°)
C1 –Ru –O1	98.1(2)	C8 –C9 –C10	116.8(7)
C1 –Ru –N1	103.5(2)	C8 –C9 –C21	121.9(6)
O1 –Ru –N1	88.9(2)	C10 –C9 –C21	121.4(7)
C1 –Ru –P	96.8(2)	C11 –C10 –C9	120.2(7)
O1 –Ru –P	88.4(1)	C11 –C10 –H10	121.0(45)
N1 –Ru –P	159.8(1)	C9 –C10 –H10	118.2(45)
C1 –Ru –Cl	88.7(2)	C10 –C11 –C12	122.1(7)
O1 –Ru –Cl	173.0(1)	C10 –C11 –H11	129.3(48)
N1 –Ru –Cl	91.2(1)	C12 –C11 –H11	108.6(48)
P –Ru –Cl	89.0(1)	C11 –C12 –C13	120.4(7)
C2 –C1 –Ru	135.3(5)	C11 –C12 –H12	118.6(49)
C2 –C1 –H1	111.6(36)	C13 –C12 –H12	121.0(50)
Ru –C1 –H1	113.1(36)	C8 –C13 –C12	116.1(6)
C7 –C2 –C3	118.4(6)	C8 –C13 –C24	122.5(5)
C7 –C2 –C1	125.4(6)	C12 –C13 –C24	121.4(6)
C3 –C2 –C1	116.2(6)	N1 –C14 –C15	129.4(5)
C4 –C3 –C2	119.2(7)	N1 –C14 –H14	110.6(39)
C4 –C3 –H3	129.2(42)	C15 –C14 –H14	119.9(39)
C2 –C3 –H3	111.4(41)	C16 –C15 –C14	117.8(5)
C3 –C4 –C5	121.0(8)	C16 –C15 –C20	119.1(5)
C3 –C4 –H4	110.8(48)	C14 –C15 –C20	123.1(5)
C5 –C4 –H4	128.0(49)	C17 –C16 –C15	121.3(5)
C6 –C5 –C4	119.4(8)	C17 –C16 –H16	117.4(34)
C6 –C5 –H5	114.0(34)	C15 –C16 –H16	120.9(34)
C4 –C5 –H5	126.1(35)	C16 –C17 –C18	120.5(5)
C5 –C6 –C7	121.2(8)	C16 –C17 –N2	119.5(5)
C5 –C6 –H6	115.4(42)	C18 –C17 –N2	120.0(5)
C7 –C6 –H6	123.2(42)	C19 –C18 –C17	119.2(6)
C2 –C7 –C6	120.7(7)	C19 –C18 –H18	125.5(36)
C2 –C7 –H7	119.1(59)	C17 –C18 –H18	115.0(35)
C6 –C7 –H7	120.2(59)	C18 –C19 –C20	123.2(6)
C14 –N1 –C8	114.4(5)	C18 –C19 –H19	124.7(40)
C14 –N1 –Ru	123.8(4)	C20 –C19 –H19	112.1(39)
C8 –N1 –Ru	121.5(3)	O1 –C20 –C19	118.6(5)
C20 –O1 –Ru	129.7(4)	O1 –C20 –C15	124.7(5)
C13 –C8 –C9	124.4(6)	C19 –C20 –C15	116.7(5)
C13 –C8 –N1	117.2(5)	C9 –C21 –C22	111.1(6)
C9 –C8 –N1	118.2(6)	C9 –C21 –C23	113.0(7)

Table 6. (Cont.)

			Angle(°)				Angle(°)
C22	–C21	–C23	110.8(7)	C33	–P	–C27	103.9(3)
C9	–C21	–H21	109.0(44)	C39	–P	–C27	105.2(3)
C22	–C21	–H21	109.5(44)	C33	–P	–Ru	114.2(2)
C23	–C21	–H21	103.1(41)	C39	–P	–Ru	117.5(2)
C21	–C22	–H22a	111.3(37)	C27	–P	–Ru	102.4(2)
C21	–C22	–H22b	110.6(51)	C28	–C27	–C32	111.1(6)
H22a	–C22	–H22b	104.1(63)	C28	–C27	–P	113.8(5)
C21	–C22	–H22c	112.0(46)	C32	–C27	–P	108.2(4)
H22a	–C22	–H22c	104.2(56)	C28	–C27	–H27	105.8(32)
H22b	–C22	–H22c	114.1(67)	C32	–C27	–H27	112.1(33)
C21	–C23	–H23a	111.5(45)	P	–C27	–H27	105.7(33)
C21	–C23	–H23b	104.8(39)	C29	–C28	–C27	111.6(6)
H23a	–C23	–H23b	119.3(64)	C29	–C28	–H28a	108.4(30)
C21	–C23	–H23c	116.2(53)	C27	–C28	–H28a	111.2(28)
H23a	–C23	–H23c	89.9(64)	C29	–C28	–H28b	109.4(34)
H23b	–C23	–H23c	115.4(66)	C27	–C28	–H28b	107.7(33)
C13	–C24	–C25	111.0(6)	H28a	–C28	–H28b	108.6(44)
C13	–C24	–C26	113.2(6)	C30	–C29	–C28	111.0(6)
C25	–C24	–C26	110.1(6)	C30	–C29	–H29a	109.3(33)
C13	–C24	–H24	111.9(37)	C28	–C29	–H29a	94.3(34)
C25	–C24	–H24	107.7(36)	C30	–C29	–H29b	107.1(44)
C26	–C24	–H24	102.5(37)	C28	–C29	–H29b	114.2(43)
C24	–C25	–H25a	108.8(34)	H29a	–C29	–H29b	120.4(54)
C24	–C25	–H25b	107.3(43)	C29	–C30	–C31	109.7(7)
H25a	–C25	–H25b	113.8(55)	C29	–C30	–H30a	108.4(43)
C24	–C25	–H25c	104.3(42)	C31	–C30	–H30a	115.0(43)
H25a	–C25	–H25c	120.6(56)	C29	–C30	–H30b	100.7(42)
H25b	–C25	–H25c	101.0(58)	C31	–C30	–H30b	93.4(43)
C24	–C26	–H26a	105.0(56)	H30a	–C30	–H30b	127.9(60)
C24	–C26	–H26b	109.4(62)	C30	–C31	–C32	109.2(7)
H26a	–C26	–H26b	120.0(81)	C30	–C31	–H31a	114.2(42)
C24	–C26	–H26c	107.9(42)	C32	–C31	–H31a	105.6(42)
H26a	–C26	–H26c	99.2(67)	C30	–C31	–H31b	105.0(39)
H26b	–C26	–H26c	114.4(73)	C32	–C31	–H31b	112.9(40)
O2	–N2	–O3	123.3(5)	H31a	–C31	–H31b	110.1(56)
O2	–N2	–C17	117.5(5)	C31	–C32	–C27	110.3(6)
O3	–N2	–C17	119.2(5)	C31	–C32	–H32a	112.5(44)
C33	–P	–C39	111.7(3)	C27	–C32	–H32a	104.7(41)

Table 6. (Cont.)

			Angle(°)				Angle(°)
C31	–C32	–H32b	108.7(38)	H38a	–C38	–H38b	108.2
C27	–C32	–H32b	98.8(37)	C40	–C39	–C44	110.4(5)
H32a	–C32	–H32b	120.5(57)	C40	–C39	–P	115.5(4)
C38	–C33	–C34	109.3(6)	C44	–C39	–P	113.9(4)
C38	–C33	–P	111.6(5)	C40	–C39	–H39	110.3(32)
C34	–C33	–P	118.6(5)	C44	–C39	–H39	109.1(34)
C38	–C33	–H33	108.9(33)	P	–C39	–H39	96.7(31)
C34	–C33	–H33	103.5(33)	C39	–C40	–C41	109.5(6)
P	–C33	–H33	104.3(32)	C39	–C40	–H40a	108.1(33)
C35	–C34	–C33	110.9(7)	C41	–C40	–H40a	112.9(33)
C35	–C34	–H34a	116.6(59)	C39	–C40	–H40b	101.1(38)
C33	–C34	–H34a	106.9(58)	C41	–C40	–H40b	102.0(37)
C35	–C34	–H34b	114.0(37)	H40a	–C40	–H40b	122.2(52)
C33	–C34	–H34b	107.6(39)	C42	–C41	–C40	110.6(6)
H34a	–C34	–H34b	99.9(64)	C42	–C41	–H41a	108.5(42)
C36	–C35	–C34	112.0(8)	C40	–C41	–H41a	110.6(42)
C36	–C35	–H35a	107.0(42)	C42	–C41	–H41b	108.1(36)
C34	–C35	–H35a	106.1(40)	C40	–C41	–H41b	105.1(34)
C36	–C35	–H35b	108.6(61)	H41a	–C41	–H41b	113.9(53)
C34	–C35	–H35b	109.3(60)	C41	–C42	–C43	110.3(6)
H35a	–C35	–H35b	114.0(71)	C41	–C42	–H42a	119.0(47)
C35	–C36	–C37	111.7(8)	C43	–C42	–H42a	97.9(44)
C35	–C36	–H36a	116.8(56)	C41	–C42	–H42b	106.6(40)
C37	–C36	–H36a	106.9(56)	C43	–C42	–H42b	108.7(40)
C35	–C36	–H36b	105.1(47)	H42a	–C42	–H42b	113.8(58)
C37	–C36	–H36b	109.6(48)	C42	–C43	–C44	110.3(6)
H36a	–C36	–H36b	106.5(65)	C42	–C43	–H43a	102.0(49)
C38	–C37	–C36	112.7(9)	C44	–C43	–H43a	100.4(49)
C38	–C37	–H37a	105.2(53)	C42	–C43	–H43b	111.1(39)
C36	–C37	–H37a	104.7(58)	C44	–C43	–H43b	108.1(39)
C38	–C37	–H37b	116.9(81)	H43a	–C43	–H43b	124.2(66)
C36	–C37	–H37b	113.8(80)	C39	–C44	–C43	109.5(5)
H37a	–C37	–H37b	101.6(89)	C39	–C44	–H44a	108.5(45)
C37	–C38	–C33	109.9(7)	C43	–C44	–H44a	114.0(46)
C37	–C38	–H38a	109.7	C39	–C44	–H44b	110.0(31)
C33	–C38	–H38a	109.7	C43	–C44	–H44b	101.4(29)
C37	–C38	–H38b	109.7	H44a	–C44	–H44b	113.2(53)
C33	–C38	–H38b	109.7	Cl98	–C99	–Cl99	120.0(26)

Table 6. (Cont.)

			Angle(°)
Cl98	–C99	–H99a	107.3
Cl99	–C99	–H99a	107.3
Cl98	–C99	–H99b	107.3
Cl99	–C99	–H99b	107.3
H99a	–C99	–H99b	106.9