ANEXO

Procesos de oxidación selectiva catalizados por oxidoperoxidocomplejos de molibdeno usando criterios de química sostenible

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Tabla de contenidos

Anexo 1. Datos cristalográficos de los complejos $[C_4mim]_4[Mo_8O_{26}]$ y $[Htmpy]_2[\{MoO(O_2)_2\}_2(\mu\text{-}O)]$.

	$[C_4mim]_4[Mo_8O_{26}]$	$[Htmpy]_2[\{MoO(O_2)_2\}_2(\mu\text{-}O)]$
Fórmula	$C_{32}H_{60}Mo_8N_8O_{26}$	$C_{16}H_{24}Mo_2N_2O_{11}$
Masa molecular	1740.40	612.25
Sistema cristalino	Triclínico	Triclínico
Grupo espacial	$P\bar{1}$	$P\bar{1}$
a, (Å)	10.8826(4)	8.653(3)
b, (Å)	12.5912(5)	9.350(4)
c, (Å)	19.6589(8)	13.886(5)
α, (º)	84.6960(10)	90.639(8)
β, (º)	84.2860(10)	90.689(9)
γ, (º)	84.5940(10)	97.866(8)
V, ($Å^3$)	2659.06(18)	1112.7(7)
Z, F(000)	2, 1704	2, 608
D_{calc}, ($Mg\cdot m^{-3}$)	2.174	1.821
μ, (mm^{-1})	1.911	1.185
θ_{max}, (º)	25.0	30.77
Nº de reflexiones recogidos	67371	27302
Nº de refrexiones usadas	14924	6818
Nº de parámetros.	712	288
$R_1(F)$ $[F^2>2\sigma(F^2)]$[a]	0.0270	0.0750
$wR_2(F^2)$[b](todos los datos incluídos)	0.0648	0.2223
S[c] (todos los datos incluídos)	1.042	1.068

[a] $R_1(F) = \sum(|F_o|-|F_c|)/\sum|F_o|$ para las reflexiones observadas $[F^2>2\sigma(F^2)]$.

[b] $wR_2(F^2) = \{\sum [w(F_o^2- F_c^2)^2]/ \sum w(F_o^2)^2\}^{1/2}$.

[c] $S = \{\sum [w(F_o^2- F_c^2)^2]/(n\text{-}p)\}^{1/2}$; (n = número de reflexiones, p = número de parámetros)

Anexo 2. Distancias [Å] y ángulos [°] de enlace seleccionados del compuesto $[C_4mim]_4[Mo_8O_{26}]$.

Mo(1)-O(1)	1.7585(18)	Mo(5)-O(14)	1.9192(17)
Mo(1)-O(2)	1.9515(16)	Mo(5)-O(15)	1.7138(18)
Mo(1)-O(3)	1.6928(19)	Mo(5)-O(16)	1.707(2)
Mo(1)-O(4)	2.1308(17)	Mo(5)-O(17)	1.9073(18)
Mo(1)-O(4)#1	2.3922(17)	Mo(5)-O(18)	2.4934(18)
Mo(1)-O(5)	1.9420(16)	Mo(5)-O(19)	2.2730(18)
Mo(2)-O(2)#1	2.3540(18)	Mo(6)-O(18)	2.3785(16)
Mo(2)-O(4)	2.2971(16)	Mo(6)-O(18)#2	2.1565(17)
Mo(2)-O(5)	1.9989(18)	Mo(6)-O(19)	1.7555(19)
Mo(2)-O(6)	1.705(2)	Mo(6)-O(20)	1.9443(17)
Mo(2)-O(7)	1.7115(18)	Mo(6)-O(21)	1.6921(18)
Mo(2)-O(13)#1	1.8972(18)	Mo(6)-O(24)#2	1.9517(16)
Mo(3)-O(2)#1	2.0061(17)	Mo(7)-O(17)#2	1.9129(19)
Mo(3)-O(4)#1	2.3684(16)	Mo(7)-O(18)#2	2.3207(16)
Mo(3)-O(5)	2.3429(18)	Mo(7)-O(20)	1.9907(18)
Mo(3)-O(8)	1.6995(18)	Mo(7)-O(22)	1.7006(19)
Mo(3)-O(9)	1.698(2)	Mo(7)-O(23)	1.7006(18)
Mo(3)-O(10)	1.8944(18)	Mo(7)-O(24)	2.3696(17)
Mo(4)-O(1)	2.2612(19)	Mo(8)-O(14)	1.9085(19)
Mo(4)-O(4)#1	2.4694(17)	Mo(8)-O(18)	2.2848(16)
Mo(4)-O(10)	1.9125(17)	Mo(8)-O(20)	2.3677(18)
Mo(4)-O(11)	1.710(2)	Mo(8)-O(24)	2.0009(17)
Mo(4)-O(12)	1.703(2)	Mo(8)-O(25)	1.7072(18)
Mo(4)-O(13)	1.9298(17)	Mo(8)-O(26)	1.7005(19)
O(3)-Mo(1)-O(1)	103.76(9)	O(9)-Mo(3)-O(10)	101.41(9)
O(3)-Mo(1)-O(5)	103.17(8)	O(8)-Mo(3)-O(10)	102.37(8)
O(1)-Mo(1)-O(5)	96.35(8)	O(9)-Mo(3)-O(2)#1	96.65(8)
O(3)-Mo(1)-O(2)	100.16(8)	O(8)-Mo(3)-O(2)#1	100.70(8)
O(1)-Mo(1)-O(2)	96.12(8)	O(10)-Mo(3)-O(2)#1	145.70(7)
O(5)-Mo(1)-O(2)	150.02(7)	O(9)-Mo(3)-O(5)	163.77(7)
O(3)-Mo(1)-O(4)	100.60(8)	O(8)-Mo(3)-O(5)	88.46(8)
O(1)-Mo(1)-O(4)	155.64(8)	O(10)-Mo(3)-O(5)	83.91(7)
O(5)-Mo(1)-O(4)	78.51(7)	O(2)#1-Mo(3)-O(5)	71.66(7)
O(2)-Mo(1)-O(4)	78.91(7)	O(9)-Mo(3)-O(4)#1	94.81(8)
O(3)-Mo(1)-O(4)#1	176.30(7)	O(8)-Mo(3)-O(4)#1	159.64(8)
O(1)-Mo(1)-O(4)#1	79.62(7)	O(10)-Mo(3)-O(4)#1	77.27(6)
O(5)-Mo(1)-O(4)#1	77.76(6)	O(2)#1-Mo(3)-O(4)#1	72.33(6)
O(2)-Mo(1)-O(4)#1	77.84(7)	O(5)-Mo(3)-O(4)#1	71.21(6)
O(4)-Mo(1)-O(4)#1	76.02(7)	O(12)-Mo(4)-O(11)	104.58(11)
O(6)-Mo(2)-O(7)	104.69(10)	O(12)-Mo(4)-O(10)	102.73(8)
O(6)-Mo(2)-O(13)#1	102.20(9)	O(11)-Mo(4)-O(10)	98.87(9)
O(7)-Mo(2)-O(13)#1	100.11(8)	O(12)-Mo(4)-O(13)	102.71(8)
O(6)-Mo(2)-O(5)	96.58(8)	O(11)-Mo(4)-O(13)	97.67(9)
O(7)-Mo(2)-O(5)	100.57(8)	O(10)-Mo(4)-O(13)	144.91(8)
O(13)#1-Mo(2)-O(5)	147.41(7)	O(12)-Mo(4)-O(1)	91.20(9)
O(6)-Mo(2)-O(4)	95.36(8)	O(11)-Mo(4)-O(1)	164.19(9)
O(7)-Mo(2)-O(4)	159.69(8)	O(10)-Mo(4)-O(1)	78.22(7)
O(13)#1-Mo(2)-O(4)	78.39(7)	O(13)-Mo(4)-O(1)	77.49(7)
O(5)-Mo(2)-O(4)	73.49(6)	O(12)-Mo(4)-O(4)#1	160.70(9)
O(6)-Mo(2)-O(2)#1	164.79(8)	O(11)-Mo(4)-O(4)#1	94.71(8)
O(7)-Mo(2)-O(2)#1	87.20(8)	O(10)-Mo(4)-O(4)#1	74.43(6)
O(13)#1-Mo(2)-O(2)#1	84.64(7)	O(13)-Mo(4)-O(4)#1	73.54(6)
O(5)-Mo(2)-O(2)#1	71.53(7)	O(1)-Mo(4)-O(4)#1	69.50(6)
O(4)-Mo(2)-O(2)#1	72.49(6)	O(16)-Mo(5)-O(15)	104.59(10)
O(9)-Mo(3)-O(8)	105.11(10)	O(16)-Mo(5)-O(17)	103.27(9)

O(15)-Mo(5)-O(17)	98.10(8)	O(23)-Mo(7)-O(17)#2	99.83(9)
O(16)-Mo(5)-O(14)	103.74(9)	O(22)-Mo(7)-O(17)#2	101.34(9)
O(15)-Mo(5)-O(14)	98.05(8)	O(23)-Mo(7)-O(20)	100.83(9)
O(17)-Mo(5)-O(14)	143.71(8)	O(22)-Mo(7)-O(20)	98.96(9)
O(16)-Mo(5)-O(19)	90.11(8)	O(17)#2-Mo(7)-O(20)	145.89(7)
O(15)-Mo(5)-O(19)	165.30(8)	O(23)-Mo(7)-O(18)#2	160.14(8)
O(17)-Mo(5)-O(19)	78.02(7)	O(22)-Mo(7)-O(18)#2	94.40(8)
O(14)-Mo(5)-O(19)	78.16(7)	O(17)#2-Mo(7)-O(18)#2	77.72(7)
O(16)-Mo(5)-O(18)	160.06(8)	O(20)-Mo(7)-O(18)#2	73.64(6)
O(15)-Mo(5)-O(18)	95.35(8)	O(23)-Mo(7)-O(24)	88.40(8)
O(17)-Mo(5)-O(18)	73.52(7)	O(22)-Mo(7)-O(24)	164.83(8)
O(14)-Mo(5)-O(18)	72.79(7)	O(17)#2-Mo(7)-O(24)	82.10(7)
O(19)-Mo(5)-O(18)	69.95(6)	O(20)-Mo(7)-O(24)	71.69(7)
O(21)-Mo(6)-O(19)	104.23(9)	O(18)#2-Mo(7)-O(24)	71.73(6)
O(21)-Mo(6)-O(20)	101.45(8)	O(26)-Mo(8)-O(25)	104.57(9)
O(19)-Mo(6)-O(20)	96.85(8)	O(26)-Mo(8)-O(14)	102.29(9)
O(21)-Mo(6)-O(24)#2	101.00(8)	O(25)-Mo(8)-O(14)	100.59(9)
O(19)-Mo(6)-O(24)#2	97.07(8)	O(26)-Mo(8)-O(24)	96.98(9)
O(20)-Mo(6)-O(24)#2	149.70(7)	O(25)-Mo(8)-O(24)	99.46(8)
O(21)-Mo(6)-O(18)#2	98.88(8)	O(14)-Mo(8)-O(24)	147.56(7)
O(19)-Mo(6)-O(18)#2	156.89(7)	O(26)-Mo(8)-O(18)	95.36(7)
O(20)-Mo(6)-O(18)#2	78.40(7)	O(25)-Mo(8)-O(18)	159.77(8)
O(24)#2-Mo(6)-O(18)#2	78.25(7)	O(14)-Mo(8)-O(18)	78.13(7)
O(21)-Mo(6)-O(18)	174.32(8)	O(24)-Mo(8)-O(18)	74.27(6)
O(19)-Mo(6)-O(18)	81.45(7)	O(26)-Mo(8)-O(20)	164.47(8)
O(20)-Mo(6)-O(18)	77.53(6)	O(25)-Mo(8)-O(20)	88.00(8)
O(24)#2-Mo(6)-O(18)	78.11(6)	O(14)-Mo(8)-O(20)	83.91(7)
O(18)#2-Mo(6)-O(18)	75.44(7)	O(24)-Mo(8)-O(20)	71.57(7)
O(23)-Mo(7)-O(22)	105.37(9)	O(18)-Mo(8)-O(20)	71.77(6)

Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1.

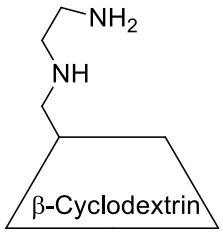
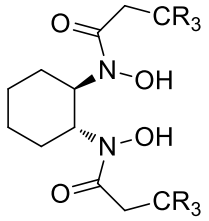
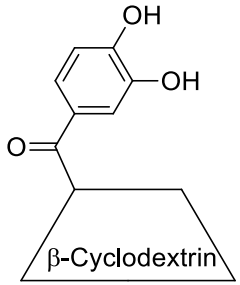
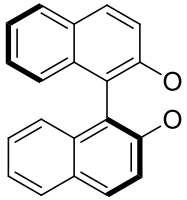
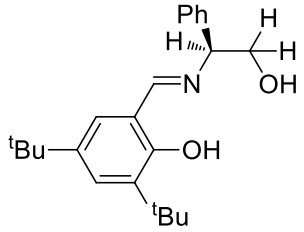
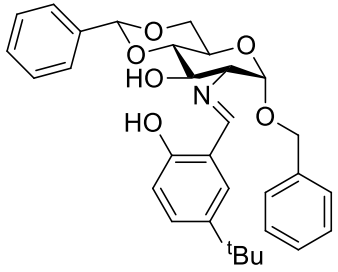
Anexo 3. Longitudes [Å] y ángulos [º] de enlace seleccionados del complejo $[Htmpy]_2[\{MoO(O_2)_2\}_2(\mu\text{-}O)]$.

Mo(1)-O(1)	1.676(12)	Mo(2)-O(8)	1.966(11)
Mo(1)-O(2)	1.966(12)	Mo(2)-O(9)	1.913(14)
Mo(1)-O(3)	1.960(15)	Mo(2)-O(10)	1.972(14)
Mo(1)-O(4)	1.963(12)	Mo(2)-O(11)	1.694(12)
Mo(1)-O(5)	1.917(13)	O(2)-O(3)	1.495(17)
Mo(1)-O(6)	1.949(10)	O(4)-O(5)	1.475(17)
Mo(2)-O(6)	1.927(10)	O(7)-O(8)	1.465(17)
Mo(2)-O(7)	1.970(12)	O(9)-O(10)	1.494(17)
O(1)-Mo(1)-O(5)	104.6(6)	O(6)-Mo(2)-O(8)	123.5(5)
O(1)-Mo(1)-O(6)	102.9(6)	O(11)-Mo(2)-O(7)	105.3(6)
O(5)-Mo(1)-O(6)	82.2(5)	O(9)-Mo(2)-O(7)	149.7(6)
O(1)-Mo(1)-O(3)	105.1(6)	O(6)-Mo(2)-O(7)	83.3(5)
O(5)-Mo(1)-O(3)	149.9(6)	O(8)-Mo(2)-O(7)	43.7(5)
O(6)-Mo(1)-O(3)	87.0(5)	O(11)-Mo(2)-O(10)	103.1(6)
O(1)-Mo(1)-O(4)	107.7(6)	O(9)-Mo(2)-O(10)	45.2(5)
O(5)-Mo(1)-O(4)	44.7(5)	O(6)-Mo(2)-O(10)	129.0(5)
O(6)-Mo(1)-O(4)	123.4(5)	O(8)-Mo(2)-O(10)	87.9(6)
O(3)-Mo(1)-O(4)	127.1(6)	O(7)-Mo(2)-O(10)	129.5(5)
O(1)-Mo(1)-O(2)	103.7(6)	O(11)-Mo(2)-Mo(1)	132.4(5)
O(5)-Mo(1)-O(2)	129.9(5)	O(9)-Mo(2)-Mo(1)	98.3(4)
O(6)-Mo(1)-O(2)	129.5(5)	O(6)-Mo(2)-Mo(1)	37.6(3)
O(3)-Mo(1)-O(2)	44.8(5)	O(8)-Mo(2)-Mo(1)	88.5(3)
O(4)-Mo(1)-O(2)	87.4(5)	O(7)-Mo(2)-Mo(1)	57.4(3)
O(1)-Mo(1)-Mo(2)	131.5(4)	O(10)-Mo(2)-Mo(1)	122.3(4)
O(5)-Mo(1)-Mo(2)	56.6(3)	O(3)-O(2)-Mo(1)	67.4(7)
O(6)-Mo(1)-Mo(2)	37.1(3)	O(2)-O(3)-Mo(1)	67.8(7)
O(3)-Mo(1)-Mo(2)	99.2(4)	O(5)-O(4)-Mo(1)	66.0(7)
O(4)-Mo(1)-Mo(2)	88.8(4)	O(4)-O(5)-Mo(1)	69.3(7)
O(2)-Mo(1)-Mo(2)	122.8(4)	Mo(2)-O(6)-Mo(1)	105.3(5)
O(11)-Mo(2)-O(9)	104.7(7)	O(8)-O(7)-Mo(2)	68.0(7)
O(11)-Mo(2)-O(6)	103.0(6)	O(7)-O(8)-Mo(2)	68.3(6)
O(9)-Mo(2)-O(6)	85.9(5)	O(10)-O(9)-Mo(2)	69.5(8)
O(11)-Mo(2)-O(8)	108.1(6)	O(9)-O(10)-Mo(2)	65.3(7)
O(9)-Mo(2)-O(8)	127.7(6)		

Anexo 4. Resumen de los resultados publicados para la oxidación asimétrica de PhMeS catalizada por complejos de molibdeno y de la comparación con el sistema [Mo]/[(*S*,*S*)-L^{iPr}].

Complejo de Molibdeno	Condiciones de Reacción				Rendimiento a sulfóxido (%)	*ee* (%)[a]	Ref.
	Disolvente	Oxidante	T (ºC)	t (h)			
$[Mo(O)(O_2)_2(H_2O)_n]$ y (*S*,*S*)-HL^{iPr} [b]	Cl_3CH	H_2O_2	0	1	89	40 (*R*)	Esta memoria
$Na\{[Mo(O)(O_2)_2(H_2O)]_2(\mu\text{-}L^{iPr})\}$ [b]	Cl_3CH	H_2O_2	0	1	86	42 (*R*)	Esta memoria
$[Mo(O)(O_2)_2(H_2O)_n]$ y (*S*,*S*)-HL^{iPr} [b]	Cl_3CH	H_2O_2	0	1	39	83 (*R*) [c]	Esta memoria
Na_2MoO_4 y **L1**	H_2O/CH_3OH	H_2O_2	20	2	98	60 (*R*)	4
$[MoO_2(acac)_2]$ y **L2**	CH_2Cl_2	THP	0	16	81	79 (*S*)	5
$MoCl_5$ y **L3**	H_2O	H_2O_2	0	2	99	45 (*R*)	6
$[MoO_2$(**L4**)$(THF)_2]$	Cl_3CH	TBHP	-30	6 (15 min)	87 (71)	< 5	7
$[MoO_2$(**L5**)(MeOH)]	Cl_3CH	H_2O_2	0	13	83	55 (*S*)	8
$[MoO_2(acac)_2]$ y **L6**	CH_2Cl_2	H_2O_2	0	5	82	13 (*S*)	9

THP: Tritilhidroperóxido anhidro; TBHP: tercbutilhidroperóxido. [a] Las referencias sin *ee* publicados no se han incluido. [b] Bajo las condiciones de reacción optimizadas de la Tabla 2.8 de la memoria. [c] Por resolución cinética.

 **L1**	 **L2,** R = 4-isopropylphenyl	 **L3**
 **L4**	 **L5**	 **L6**

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Anexo 5. Datos cristalográficos de los compuestos $[H(3\text{-}Mepz)]_4[Mo_8O_{26}(3\text{-}Mepz)_2]\cdot 2H_2O$, $[H(3,5\text{-}Me_2pz)]_4[Mo_8O_{26}(3,5\text{-}Me_2pz)_2]\cdot 2(3,5\text{-}Me_2pz)$ y $[Hpz]_4[Mo_8O_{22}(O_2)_4(pz)_2]\cdot 3H_2O$, respectivamente.

Fórmula empírica	$C_{24}H_{40}Mo_8N_{12}O_{28}$	$C_{40}H_{70}Mo_8N_{16}O_{26}$	$C_{18}H_{34}Mo_8N_{12}O_{33}$
Peso Molecular	1716.23	1956.62	1714.09
Sistema cristalográfico	Monoclínico	Monoclínico	Monoclínico
Grupo espacial simet.	*C c*	*P 2₁/c*	*C 2/c*
a (Å)	19.1767(5)	11.7737(5)	20.1800(14)
b (Å)	11.3151(4)	13.2676(6)	10.0629(7)
c (Å)	23.0147(7)	21.7757(11)	22.8645(16)
α (º)	90	90	90
β (º)	98.482(2)	102.197(2)	105.041(2)
γ (º)	90	90	90
V (Å^3)	4939.3(3)	3324.8(3)	4484.0(5)
Z	4	2	4
ρ (calculado, Mg·m^{-3})	2.308	1.954	2.539
coef abs (mm^{-1})	2.061	1.544	2.278
F(000)	3328	1928	3304
Tamaño del cristal (mm^3)	0.50 x 0.45 x 0.45	0.50 x 0.45 x 0.40	0.50 x 0.45 x 0.40
Reflexiones recogidas	25228	25601	41624
Reflexiones independientes	6510	5894	4061
Número de parámetros	692	414	321
$R_1(F)$ [$F^2>2\sigma(F^2)$] [a]	0.0173	0.0647	0.0323
$wR_2(F^2)$ [b] (todos los datos incluídos)	0.0453	0.1926	0.0769
S [c] (todos los datos incluídos)	1.035	1.057	1.146
CCDC refcodes	1004213	1004214	1004215

[a] $R_1(F) = \sum(\left|Fo\right| - \left|Fc\right|)/\sum\left|Fo\right|$ para las reflexiones observadas [$F^2>2\sigma(F^2)$]. [b] $wR_2(F^2) = \{\sum [w(F_o^2 - F_c^2)^2]/ \sum w(F_o^2)^2\}^{1/2}$. [c] $S = \{\sum [w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$; (n = número de reflexiones, p = número de parámetros).

Anexo 6. Distancias [Å] y ángulos [°] de enlace seleccionados del compuesto $[H(3\text{-Mepz}]_4[Mo_8O_{26}(3\text{-Mepz})_2]\cdot 2H_2O$.

Mo(1)-O(2)	1.693(3)	Mo(5)-O(18)	1.701(3)
Mo(1)-O(1)	1.702(3)	Mo(5)-O(17)	1.719(3)
Mo(1)-O(3)	1.948(3)	Mo(5)-O(4)	1.899(3)
Mo(1)-O(4)	1.975(3)	Mo(5)-O(21)	2.063(3)
Mo(1)-O(7)	2.235(3)	Mo(5)-N(3)	2.209(4)
Mo(1)-O(19)	2.298(3)	Mo(5)-O(19)	2.215(3)
Mo(2)-O(5)	1.698(3)	Mo(6)-O(20)	1.681(3)
Mo(2)-O(6)	1.714(3)	Mo(6)-O(8)	1.758(3)
Mo(2)-O(3)	1.946(3)	Mo(6)-O(19)	1.904(3)
Mo(2)-O(10)	1.949(3)	Mo(6)-O(12)	1.960(3)
Mo(2)-O(7)	2.178(3)	Mo(6)-O(21)	2.157(3)
Mo(2)-O(8)	2.393(3)	Mo(6)-O(7)	2.454(3)
Mo(3)-O(9)	1.698(3)	Mo(7)-O(22)	1.700(3)
Mo(3)-O(11)	1.748(3)	Mo(7)-O(23)	1.719(3)
Mo(3)-O(15)	1.869(3)	Mo(7)-O(24)	1.938(3)
Mo(3)-O(7)	1.973(3)	Mo(7)-O(21)	1.939(3)
Mo(3)-O(10)	2.168(3)	Mo(7)-O(12)	2.189(3)
Mo(3)-O(12)	2.453(3)	Mo(7)-O(11)	2.380(3)
Mo(4)-O(13)	1.699(3)	Mo(8)-O(26)	1.686(3)
Mo(4)-O(14)	1.723(3)	Mo(8)-O(25)	1.704(3)
Mo(4)-O(16)	1.906(3)	Mo(8)-O(16)	1.964(3)
Mo(4)-O(10)	2.056(3)	Mo(8)-O(24)	1.966(3)
Mo(4)-N(1)	2.216(4)	Mo(8)-O(12)	2.209(3)
Mo(4)-O(15)	2.261(3)	Mo(8)-O(15)	2.282(3)
O(2)-Mo(1)-O(1)	105.07(16)	O(5)-Mo(2)-O(8)	174.21(14)
O(2)-Mo(1)-O(3)	101.18(14)	O(6)-Mo(2)-O(8)	80.79(14)
O(1)-Mo(1)-O(3)	96.13(14)	O(3)-Mo(2)-O(8)	81.28(12)
O(2)-Mo(1)-O(4)	96.49(14)	O(10)-Mo(2)-O(8)	83.37(11)
O(1)-Mo(1)-O(4)	100.27(14)	O(7)-Mo(2)-O(8)	71.64(11)
O(3)-Mo(1)-O(4)	151.83(14)	O(9)-Mo(3)-O(11)	104.10(15)
O(2)-Mo(1)-O(7)	93.05(14)	O(9)-Mo(3)-O(15)	103.54(15)
O(1)-Mo(1)-O(7)	160.49(14)	O(11)-Mo(3)-O(15)	103.98(14)
O(3)-Mo(1)-O(7)	72.89(12)	O(9)-Mo(3)-O(7)	103.90(15)
O(4)-Mo(1)-O(7)	84.49(12)	O(11)-Mo(3)-O(7)	97.33(13)
O(2)-Mo(1)-O(19)	163.39(13)	O(15)-Mo(3)-O(7)	139.65(13)
O(1)-Mo(1)-O(19)	89.62(13)	O(9)-Mo(3)-O(10)	93.75(13)
O(3)-Mo(1)-O(19)	84.55(12)	O(11)-Mo(3)-O(10)	161.52(12)
O(4)-Mo(1)-O(19)	72.88(12)	O(15)-Mo(3)-O(10)	75.83(12)
O(7)-Mo(1)-O(19)	73.59(11)	O(7)-Mo(3)-O(10)	73.40(11)
O(5)-Mo(2)-O(6)	104.89(16)	O(9)-Mo(3)-O(12)	178.92(14)
O(5)-Mo(2)-O(3)	96.20(14)	O(11)-Mo(3)-O(12)	76.60(12)
O(6)-Mo(2)-O(3)	102.47(14)	O(15)-Mo(3)-O(12)	75.46(12)
O(5)-Mo(2)-O(10)	96.25(13)	O(7)-Mo(3)-O(12)	76.76(11)
O(6)-Mo(2)-O(10)	103.27(14)	O(10)-Mo(3)-O(12)	85.61(10)
O(3)-Mo(2)-O(10)	147.35(13)	O(13)-Mo(4)-O(14)	105.05(16)
O(5)-Mo(2)-O(7)	102.68(14)	O(13)-Mo(4)-O(16)	100.62(14)
O(6)-Mo(2)-O(7)	152.43(14)	O(14)-Mo(4)-O(16)	98.94(14)
O(3)-Mo(2)-O(7)	74.25(12)	O(13)-Mo(4)-O(10)	96.75(15)
O(10)-Mo(2)-O(7)	73.61(12)	O(14)-Mo(4)-O(10)	153.29(15)

O(16)-Mo(4)-O(10)	91.88(12)	O(20)-Mo(6)-O(7)	179.07(13)
O(13)-Mo(4)-N(1)	93.40(14)	O(8)-Mo(6)-O(7)	77.10(12)
O(14)-Mo(4)-N(1)	83.40(14)	O(19)-Mo(6)-O(7)	75.93(11)
O(16)-Mo(4)-N(1)	164.58(14)	O(12)-Mo(6)-O(7)	76.94(11)
O(10)-Mo(4)-N(1)	80.03(12)	O(21)-Mo(6)-O(7)	84.22(10)
O(13)-Mo(4)-O(15)	166.22(14)	O(22)-Mo(7)-O(23)	104.35(15)
O(14)-Mo(4)-O(15)	88.69(14)	O(22)-Mo(7)-O(24)	96.95(14)
O(16)-Mo(4)-O(15)	75.77(12)	O(23)-Mo(7)-O(24)	103.64(13)
O(10)-Mo(4)-O(15)	70.33(12)	O(22)-Mo(7)-O(21)	96.88(14)
N(1)-Mo(4)-O(15)	89.09(12)	O(23)-Mo(7)-O(21)	103.18(14)
O(18)-Mo(5)-O(17)	104.91(15)	O(24)-Mo(7)-O(21)	145.59(13)
O(18)-Mo(5)-O(4)	101.06(14)	O(22)-Mo(7)-O(12)	103.50(14)
O(17)-Mo(5)-O(4)	100.04(14)	O(23)-Mo(7)-O(12)	152.13(13)
O(18)-Mo(5)-O(21)	92.54(14)	O(24)-Mo(7)-O(12)	73.81(12)
O(17)-Mo(5)-O(21)	157.60(13)	O(21)-Mo(7)-O(12)	72.37(12)
O(4)-Mo(5)-O(21)	90.03(12)	O(22)-Mo(7)-O(11)	174.60(14)
O(18)-Mo(5)-N(3)	96.17(14)	O(23)-Mo(7)-O(11)	81.04(13)
O(17)-Mo(5)-N(3)	83.20(15)	O(24)-Mo(7)-O(11)	81.37(11)
O(4)-Mo(5)-N(3)	160.93(14)	O(21)-Mo(7)-O(11)	81.93(11)
O(21)-Mo(5)-N(3)	81.03(13)	O(12)-Mo(7)-O(11)	71.12(11)
O(18)-Mo(5)-O(19)	163.11(13)	O(26)-Mo(8)-O(25)	105.22(17)
O(17)-Mo(5)-O(19)	91.96(13)	O(26)-Mo(8)-O(16)	95.77(14)
O(4)-Mo(5)-O(19)	76.22(12)	O(25)-Mo(8)-O(16)	100.69(14)
O(21)-Mo(5)-O(19)	70.92(12)	O(26)-Mo(8)-O(24)	101.61(14)
N(3)-Mo(5)-O(19)	84.91(12)	O(25)-Mo(8)-O(24)	95.41(14)
O(20)-Mo(6)-O(8)	103.82(15)	O(16)-Mo(8)-O(24)	152.28(14)
O(20)-Mo(6)-O(19)	103.94(15)	O(26)-Mo(8)-O(12)	93.13(14)
O(8)-Mo(6)-O(19)	102.93(14)	O(25)-Mo(8)-O(12)	160.05(14)
O(20)-Mo(6)-O(12)	102.70(15)	O(16)-Mo(8)-O(12)	84.88(12)
O(8)-Mo(6)-O(12)	99.60(13)	O(24)-Mo(8)-O(12)	72.85(12)
O(19)-Mo(6)-O(12)	139.53(13)	O(26)-Mo(8)-O(15)	163.64(14)
O(20)-Mo(6)-O(21)	94.86(13)	O(25)-Mo(8)-O(15)	89.56(13)
O(8)-Mo(6)-O(21)	161.05(13)	O(16)-Mo(8)-O(15)	74.20(12)
O(19)-Mo(6)-O(21)	75.18(12)	O(24)-Mo(8)-O(15)	83.58(12)
O(12)-Mo(6)-O(21)	72.70(12)	O(12)-Mo(8)-O(15)	73.41(11)

Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1.

Anexo 7. Distancias [Å] y ángulos [°] de enlace seleccionados del compuesto $[H(3,5\text{-}Me_2pz)]_4[Mo_8O_{26}(3,5\text{-}Me_2pz)_2]\cdot 2(3,5\text{-}Me_2pz)$.

Mo(1)-O(1)	1.708(17)	Mo(3)-O(10)	1.711(15)
Mo(1)-O(2)	1.654(17)	Mo(3)-O(11)	1.797(17)
Mo(1)-O(3)	1.883(16)	Mo(3)-O(4)	1.904(15)
Mo(1)-O(4)	2.243(16)	Mo(3)-O(9)#1	1.915(16)
Mo(1)-O(5)	2.142(16)	Mo(3)-O(5)	2.157(16)
Mo(1)-N(1)	2.23(2)	Mo(3)-O(9)	2.446(14)
Mo(2)-O(3)	1.983(17)	Mo(4)-O(12)	1.690(16)
Mo(2)-O(4)	2.235(15)	Mo(4)-O(13)	1.724(16)
Mo(2)-O(6)	1.658(17)	Mo(4)-O(5)#1	1.923(16)
Mo(2)-O(7)	1.707(16)	Mo(4)-O(8)	1.968(16)
Mo(2)-O(8)	1.955(16)	Mo(4)-O(9)	2.242(16)
Mo(2)-O(9)	2.264(15)	Mo(4)-O(11)	2.293(16)
O(2)-Mo(1)-O(1)	105.4(8)	O(10)-Mo(3)-O(11)	100.6(7)
O(2)-Mo(1)-O(3)	99.6(8)	O(10)-Mo(3)-O(4)	106.8(7)
O(1)-Mo(1)-O(3)	100.7(8)	O(11)-Mo(3)-O(4)	101.7(7)
O(2)-Mo(1)-O(5)	161.6(7)	O(10)-Mo(3)-O(9)#1	102.1(7)
O(1)-Mo(1)-O(5)	88.8(7)	O(11)-Mo(3)-O(9)#1	96.0(7)
O(3)-Mo(1)-O(5)	88.8(6)	O(4)-Mo(3)-O(9)#1	142.4(6)
O(2)-Mo(1)-N(1)	87.5(8)	O(10)-Mo(3)-O(5)	98.8(7)
O(1)-Mo(1)-N(1)	96.4(8)	O(11)-Mo(3)-O(5)	159.8(6)
O(3)-Mo(1)-N(1)	158.9(8)	O(4)-Mo(3)-O(5)	77.7(6)
O(5)-Mo(1)-N(1)	79.3(7)	O(9)#1-Mo(3)-O(5)	74.5(6)
O(2)-Mo(1)-O(4)	95.3(7)	O(10)-Mo(3)-O(9)	176.7(7)
O(1)-Mo(1)-O(4)	159.3(7)	O(11)-Mo(3)-O(9)	76.3(6)
O(3)-Mo(1)-O(4)	74.2(6)	O(4)-Mo(3)-O(9)	75.2(6)
O(5)-Mo(1)-O(4)	71.2(6)	O(9)#1-Mo(3)-O(9)	77.3(6)
N(1)-Mo(1)-O(4)	85.4(7)	O(5)-Mo(3)-O(9)	84.2(5)
O(6)-Mo(2)-O(7)	108.2(8)	O(12)-Mo(4)-O(13)	103.7(8)
O(6)-Mo(2)-O(8)	93.3(8)	O(12)-Mo(4)-O(5)#1	95.6(8)
O(7)-Mo(2)-O(8)	101.8(7)	O(13)-Mo(4)-O(5)#1	103.5(8)
O(6)-Mo(2)-O(3)	102.0(8)	O(12)-Mo(4)-O(8)	97.4(7)
O(7)-Mo(2)-O(3)	97.3(7)	O(13)-Mo(4)-O(8)	103.5(8)
O(8)-Mo(2)-O(3)	150.4(7)	O(5)#1-Mo(4)-O(8)	146.2(7)
O(6)-Mo(2)-O(4)	90.9(7)	O(12)-Mo(4)-O(9)	99.9(7)
O(7)-Mo(2)-O(4)	160.2(7)	O(13)-Mo(4)-O(9)	156.4(7)
O(8)-Mo(2)-O(4)	82.1(6)	O(5)#1-Mo(4)-O(9)	72.4(6)
O(3)-Mo(2)-O(4)	72.6(6)	O(8)-Mo(4)-O(9)	74.7(6)
O(6)-Mo(2)-O(9)	161.0(6)	O(12)-Mo(4)-O(11)	172.0(7)
O(7)-Mo(2)-O(9)	88.8(7)	O(13)-Mo(4)-O(11)	84.3(7)
O(8)-Mo(2)-O(9)	74.4(6)	O(5)#1-Mo(4)-O(11)	81.7(6)
O(3)-Mo(2)-O(9)	83.7(6)	O(8)-Mo(4)-O(11)	81.2(6)
O(4)-Mo(2)-O(9)	73.3(5)	O(9)-Mo(4)-O(11)	72.1(5)

Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1.

Anexo 8. Distancias [Å] y ángulos [°] de enlace seleccionados del compuesto $[Hpz]_4[Mo_8O_{22}(O_2)_4(pz)_2]\cdot 3H_2O$.

Mo(1)-O(1)	1.783(6)	Mo(3)-O(11)	1.703(4)
Mo(1)-O(2)	1.875(7)	Mo(3)-O(12)	1.908(3)
Mo(1)-O(3)	1.699(4)	Mo(3)-O(5)	1.957(3)
Mo(1)-O(4)	2.064(3)	Mo(3)-O(6)	2.285(3)
Mo(1)-O(5)	1.928(3)	Mo(3)-O(9)	2.347(3)
Mo(1)-O(6)	2.250(4)	Mo(4)-O(13)	1.695(4)
Mo(1)-N(1)	2.220(4)	Mo(4)-O(14)	1.867(5)
Mo(2)-O(6)	1.871(3)	Mo(4)-O(15)	1.893(5)
Mo(2)-O(7)	1.689(3)	Mo(4)-O(12)	1.970(3)
Mo(2)-O(8)	1.774(3)	Mo(4)-O(4)#1	1.978(3)
Mo(2)-O(9)#1	1.966(3)	Mo(4)-O(9)	2.149(3)
Mo(2)-O(4)	2.103(3)	Mo(4)-O(8)	2.273(3)
Mo(2)-O(9)	2.444(3)	O(1)-O(2)	1.099(9)
Mo(3)-O(10)	1.700(4)	O(14)-O(15)	1.317(7)

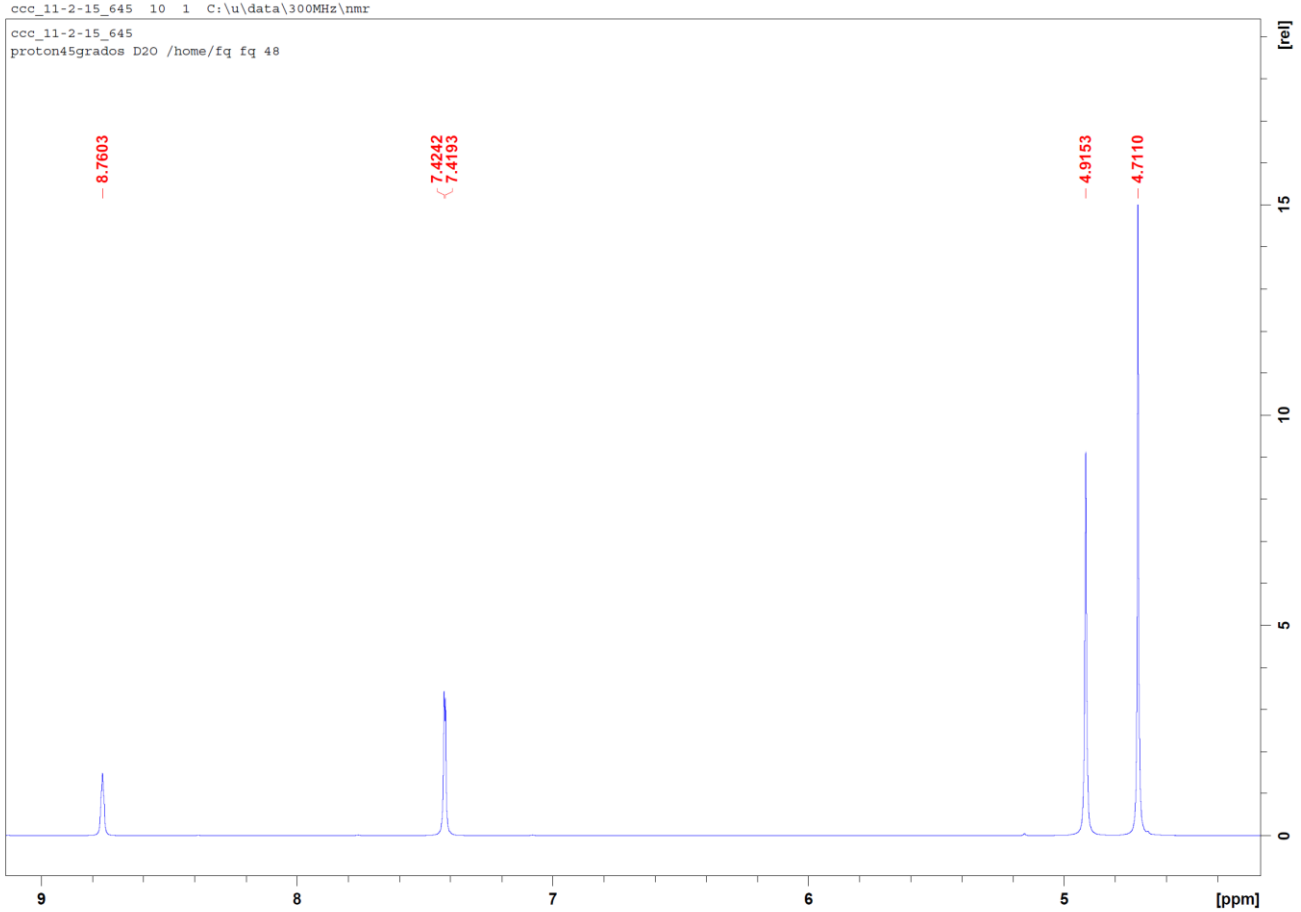
O(3)-Mo(1)-O(1)	105.4(2)	O(6)-Mo(2)-O(4)	76.16(14)
O(3)-Mo(1)-O(2)	104.5(3)	O(9)#1-Mo(2)-O(4)	74.04(13)
O(1)-Mo(1)-O(2)	34.8(3)	O(7)-Mo(2)-O(9)	176.70(14)
O(3)-Mo(1)-O(5)	99.33(16)	O(8)-Mo(2)-O(9)	74.53(14)
O(1)-Mo(1)-O(5)	88.5(3)	O(6)-Mo(2)-O(9)	77.39(13)
O(2)-Mo(1)-O(5)	122.4(3)	O(9)#1-Mo(2)-O(9)	77.04(14)
O(3)-Mo(1)-O(4)	154.39(16)	O(4)-Mo(2)-O(9)	85.23(12)
O(1)-Mo(1)-O(4)	98.2(2)	O(10)-Mo(3)-O(11)	105.66(18)
O(2)-Mo(1)-O(4)	89.2(2)	O(10)-Mo(3)-O(12)	99.16(17)
O(5)-Mo(1)-O(4)	90.98(14)	O(11)-Mo(3)-O(12)	102.14(17)
O(3)-Mo(1)-N(1)	82.52(17)	O(10)-Mo(3)-O(5)	100.72(16)
O(1)-Mo(1)-N(1)	109.1(3)	O(11)-Mo(3)-O(5)	94.45(16)
O(2)-Mo(1)-N(1)	74.4(3)	O(12)-Mo(3)-O(5)	149.52(14)
O(5)-Mo(1)-N(1)	161.33(16)	O(10)-Mo(3)-O(6)	91.11(16)
O(4)-Mo(1)-N(1)	80.48(15)	O(11)-Mo(3)-O(6)	160.88(16)
O(3)-Mo(1)-O(6)	90.87(15)	O(12)-Mo(3)-O(6)	83.73(14)
O(1)-Mo(1)-O(6)	158.2(2)	O(5)-Mo(3)-O(6)	73.05(13)
O(2)-Mo(1)-O(6)	153.8(2)	O(10)-Mo(3)-O(9)	161.47(16)
O(5)-Mo(1)-O(6)	74.39(13)	O(11)-Mo(3)-O(9)	92.10(15)
O(4)-Mo(1)-O(6)	69.30(13)	O(12)-Mo(3)-O(9)	71.37(13)
N(1)-Mo(1)-O(6)	87.04(15)	O(5)-Mo(3)-O(9)	82.75(13)
O(7)-Mo(2)-O(8)	102.20(17)	O(6)-Mo(3)-O(9)	72.34(12)
O(7)-Mo(2)-O(6)	103.98(16)	O(13)-Mo(4)-O(14)	105.3(2)
O(8)-Mo(2)-O(6)	102.51(15)	O(13)-Mo(4)-O(15)	105.8(2)
O(7)-Mo(2)-O(9)#1	103.14(15)	O(14)-Mo(4)-O(15)	41.0(2)
O(8)-Mo(2)-O(9)#1	97.28(15)	O(13)-Mo(4)-O(12)	96.89(17)
O(6)-Mo(2)-O(9)#1	141.93(15)	O(14)-Mo(4)-O(12)	83.9(2)
O(7)-Mo(2)-O(4)	98.00(15)	O(15)-Mo(4)-O(12)	123.9(2)
O(8)-Mo(2)-O(4)	159.40(15)	O(13)-Mo(4)-O(4)#1	93.71(16)

O(14)-Mo(4)-O(4)#1	123.0(2)	O(12)-Mo(4)-O(9)	74.85(13)
O(15)-Mo(4)-O(4)#1	82.4(2)	O(4)#1-Mo(4)-O(9)	72.77(13)
O(12)-Mo(4)-O(4)#1	147.06(14)	O(13)-Mo(4)-O(8)	170.80(16)
O(13)-Mo(4)-O(9)	98.56(16)	O(14)-Mo(4)-O(8)	83.58(19)
O(14)-Mo(4)-O(9)	149.72(19)	O(15)-Mo(4)-O(8)	82.44(19)
O(15)-Mo(4)-O(9)	146.11(19)	O(12)-Mo(4)-O(8)	81.36(13)
O(4)#1-Mo(4)-O(8)	83.25(13)	O(9)-Mo(4)-O(8)	72.25(12)

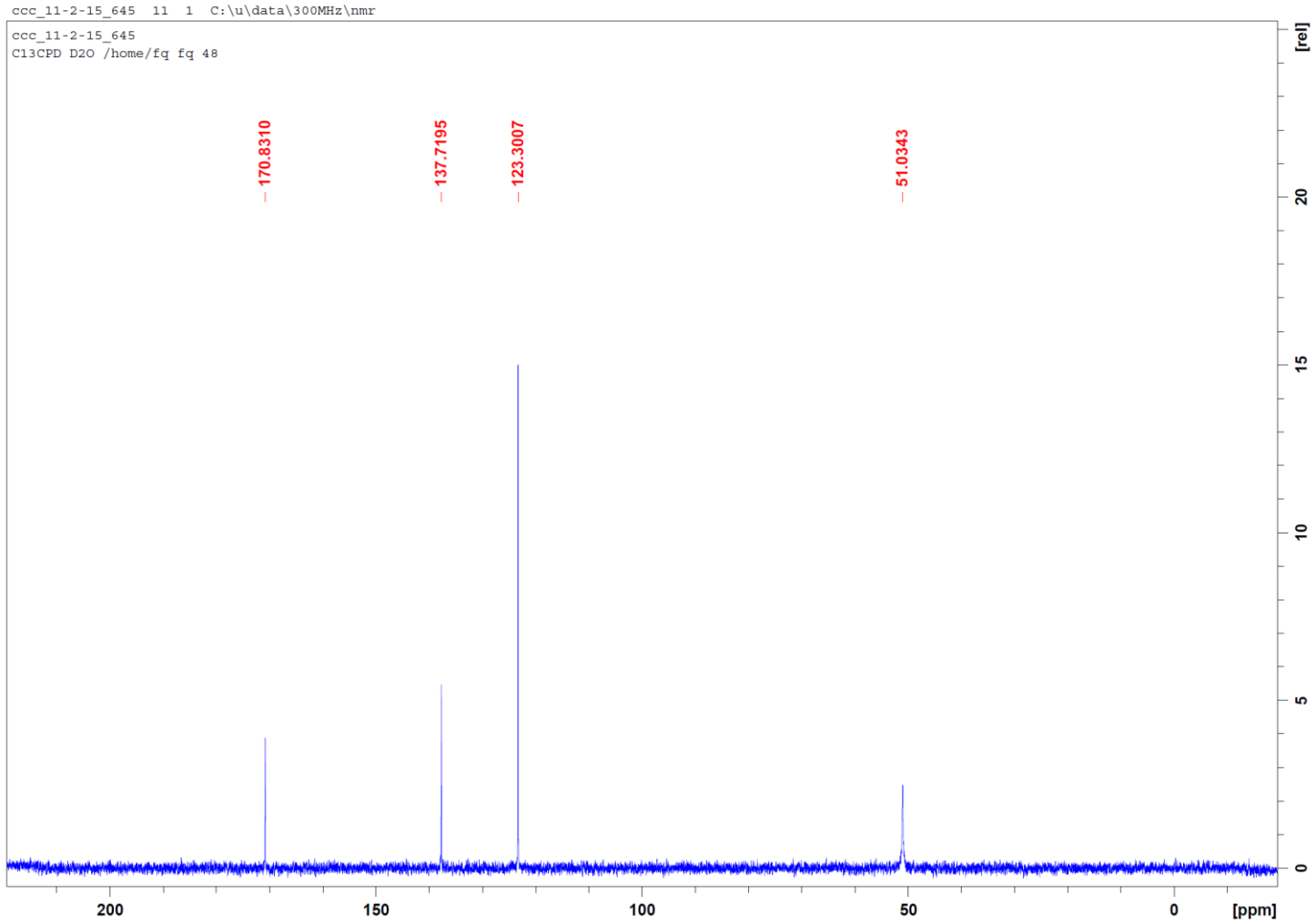
Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1.

Anexo 9. Espectros de RMN de ^{1}H y $^{13}C\{^{1}H\}$ y espectros de masas de los compuestos HL^{H} y H[(*S*,*S*)-L^{R}] donde R = Me, ^{i}Pr, CH_2Ph, ^{i}Bu y (*S*)-*sec*-Bu.

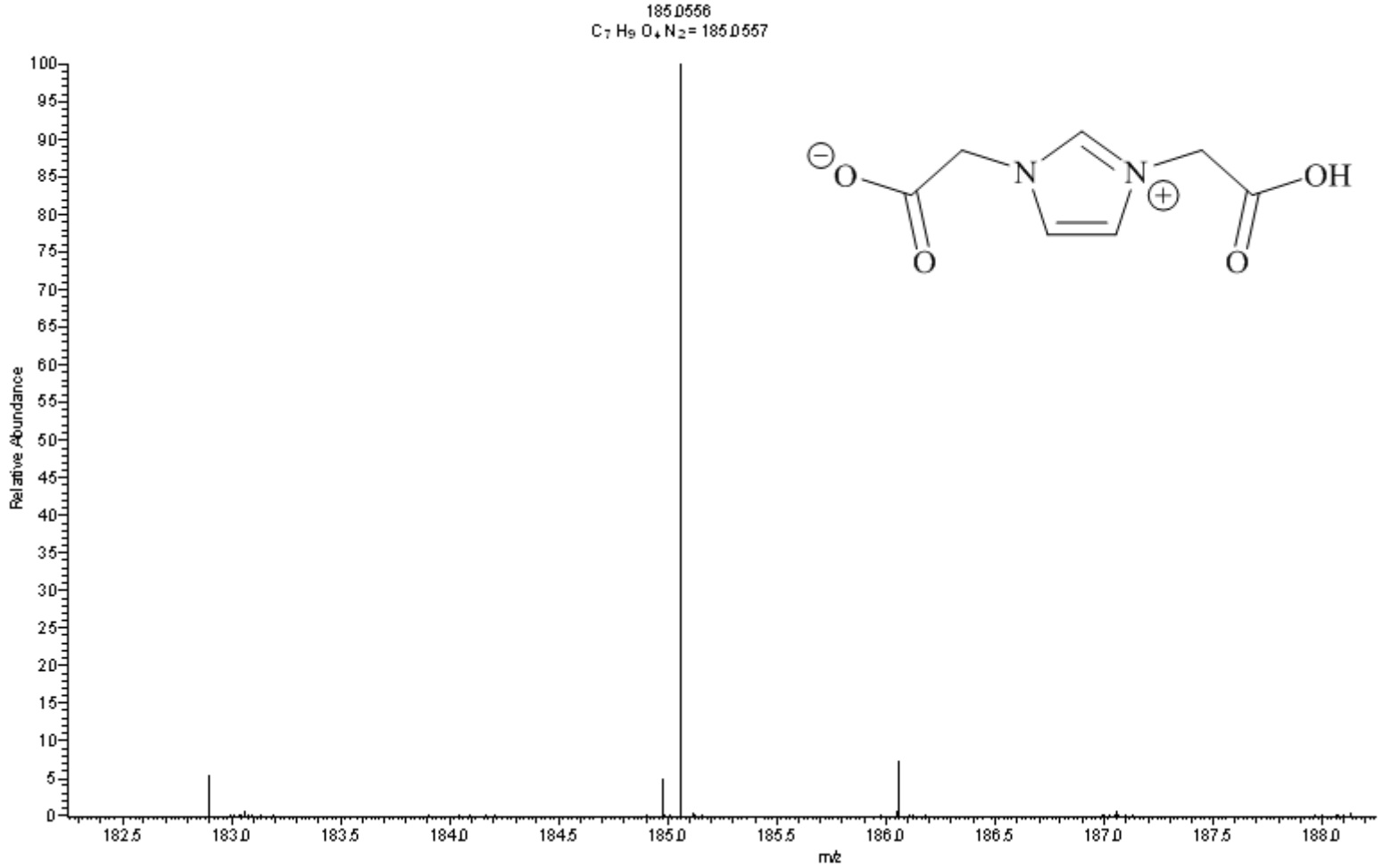
Espectro de RMN de ^{1}H de HL^{H}.



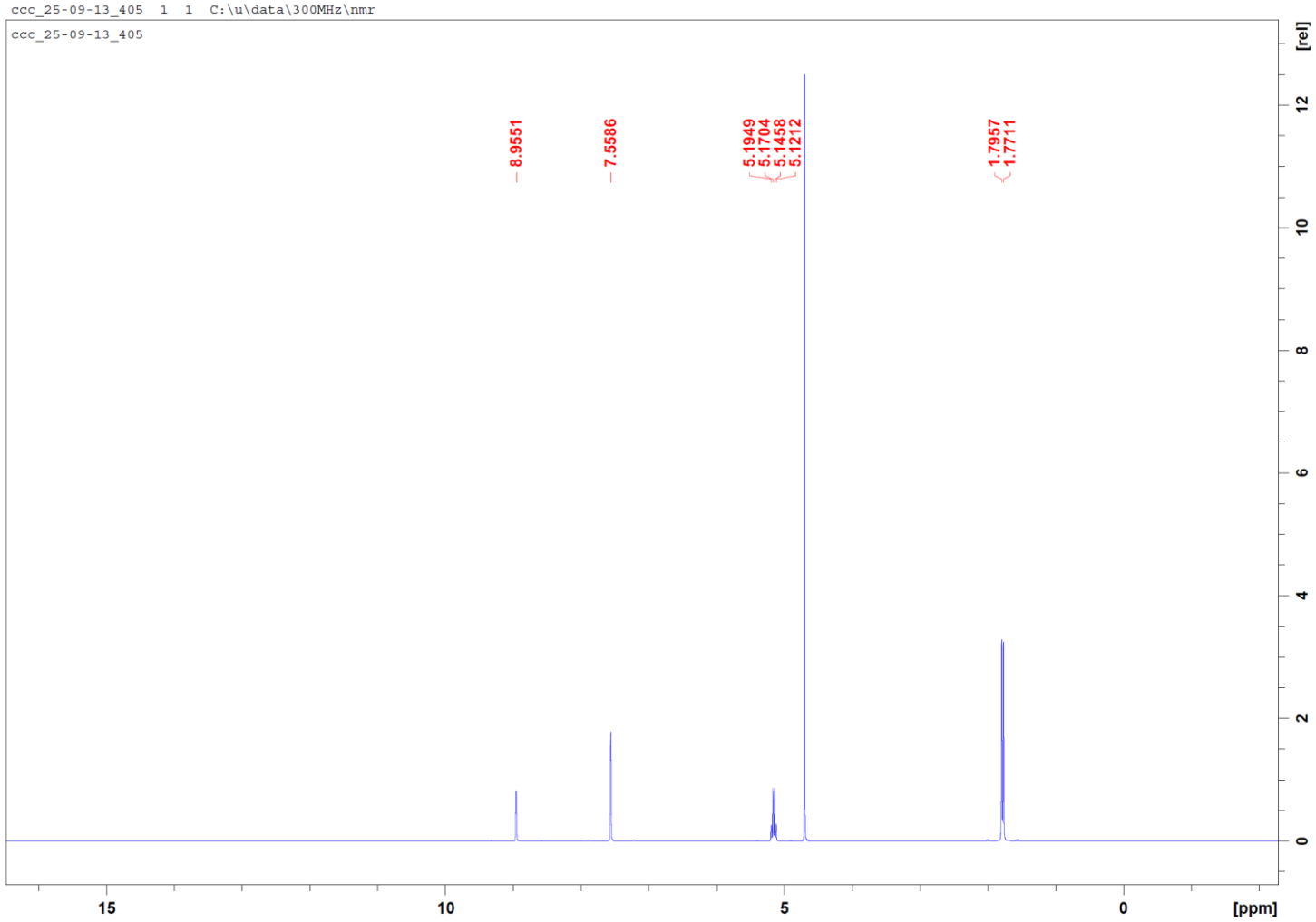
Espectro de RMN de $^{13}C\{^{1}H\}$ de HL^{H}.



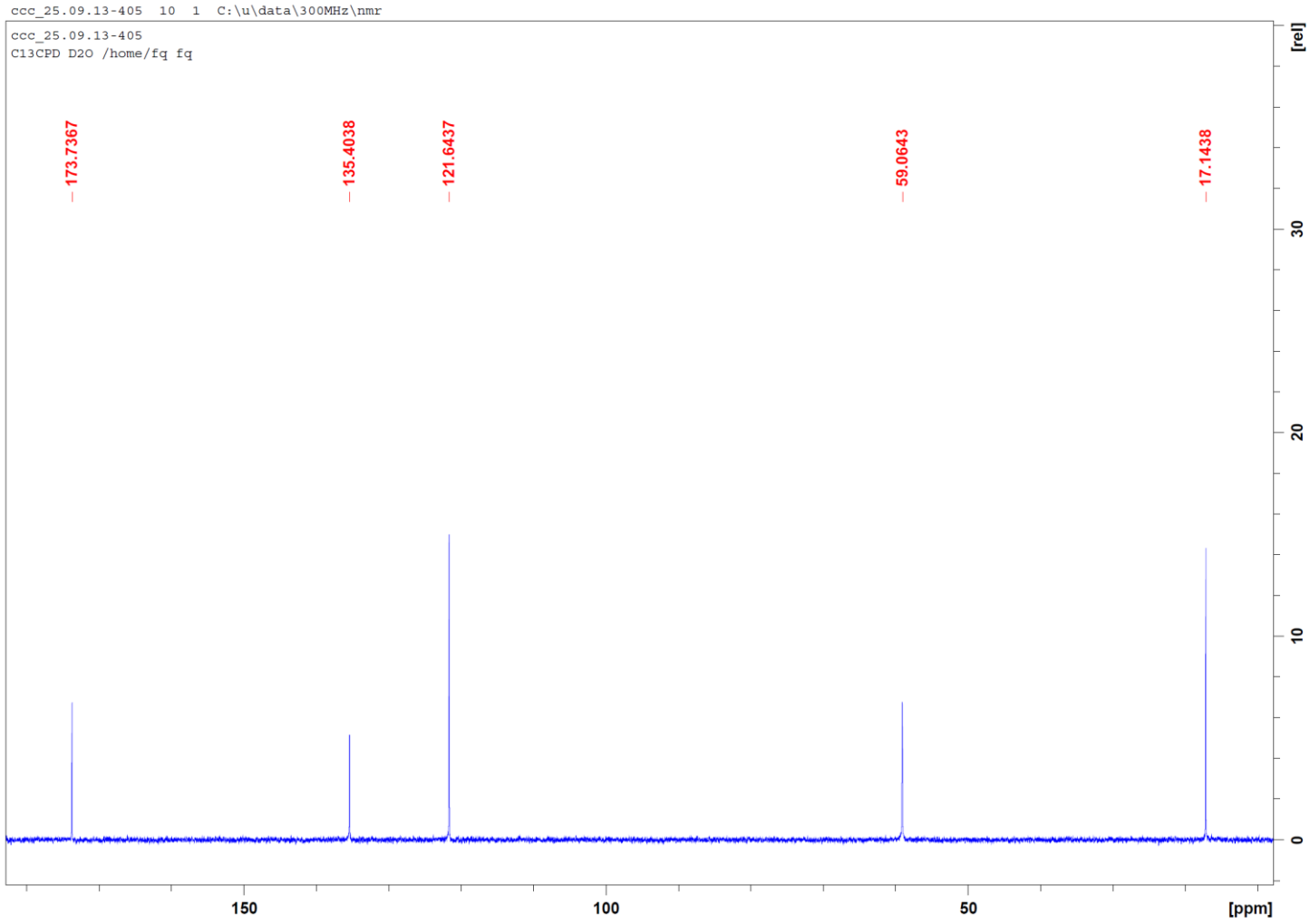
Espectro de masas de HL^{H}.



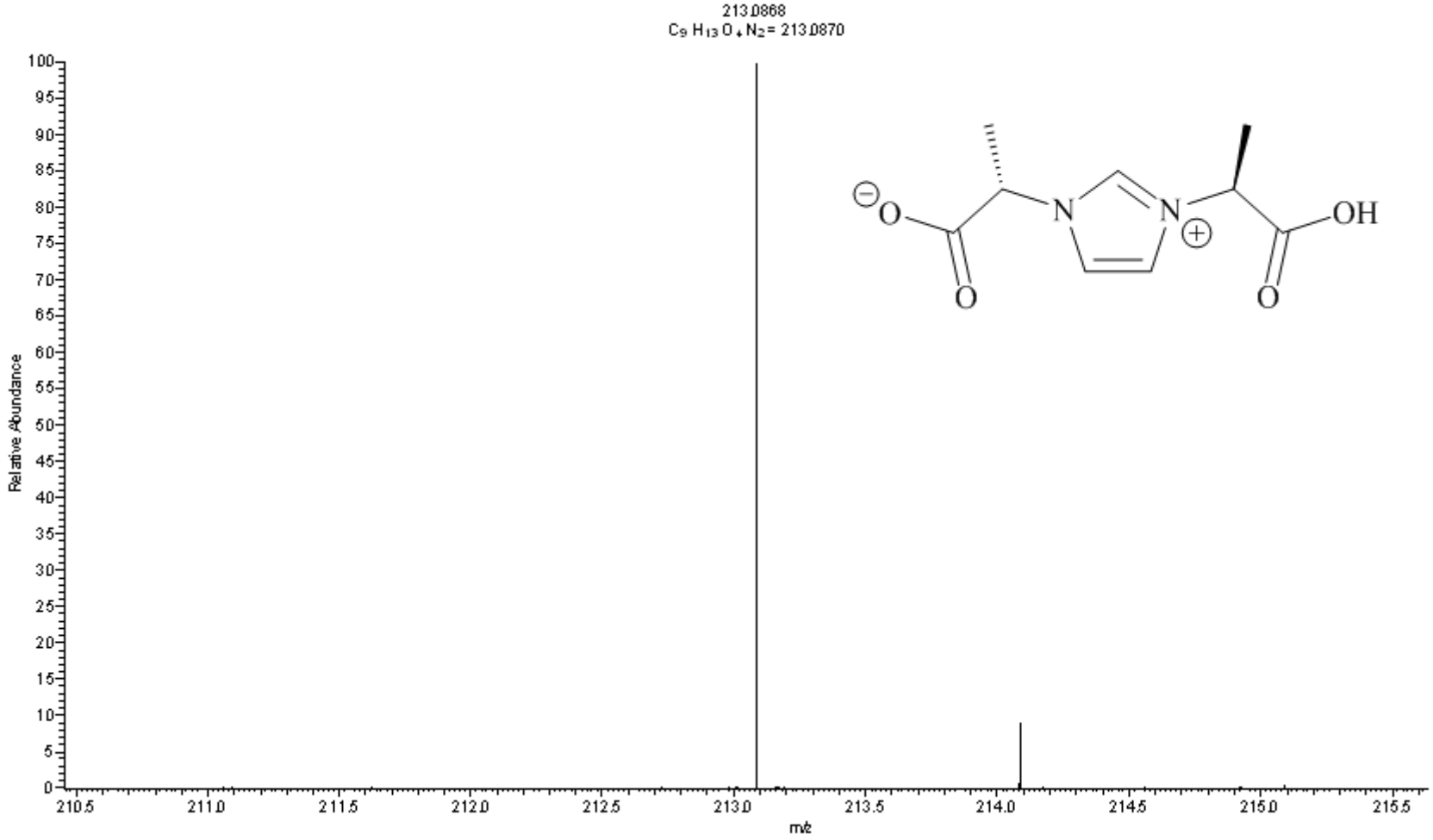
Espectro de RMN de ^{1}H de H[(*S*,*S*)-L^{Me}].



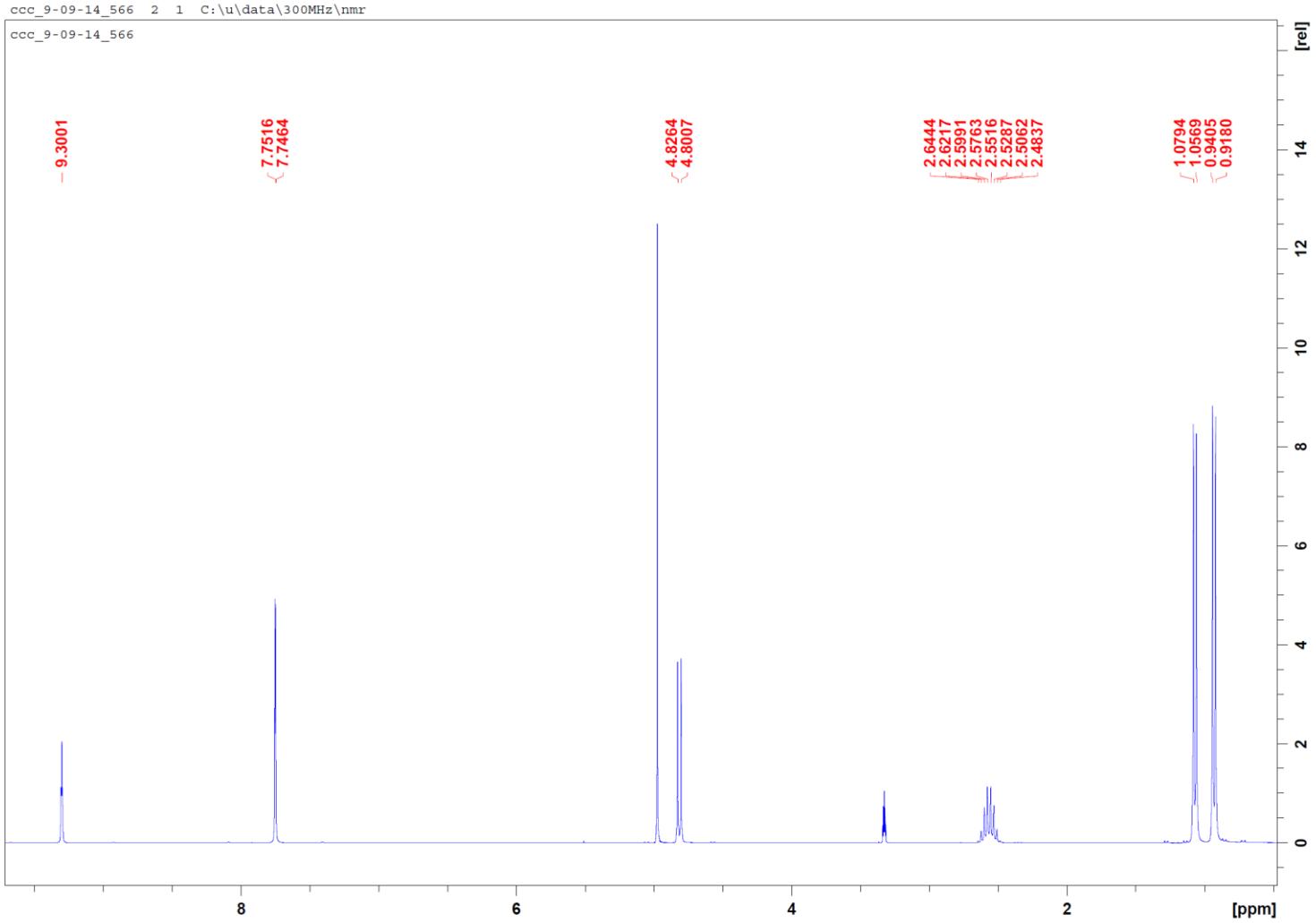
Espectro de RMN de $^{13}C\{^{1}H\}$ de H[(*S*,*S*)-L^{Me}].



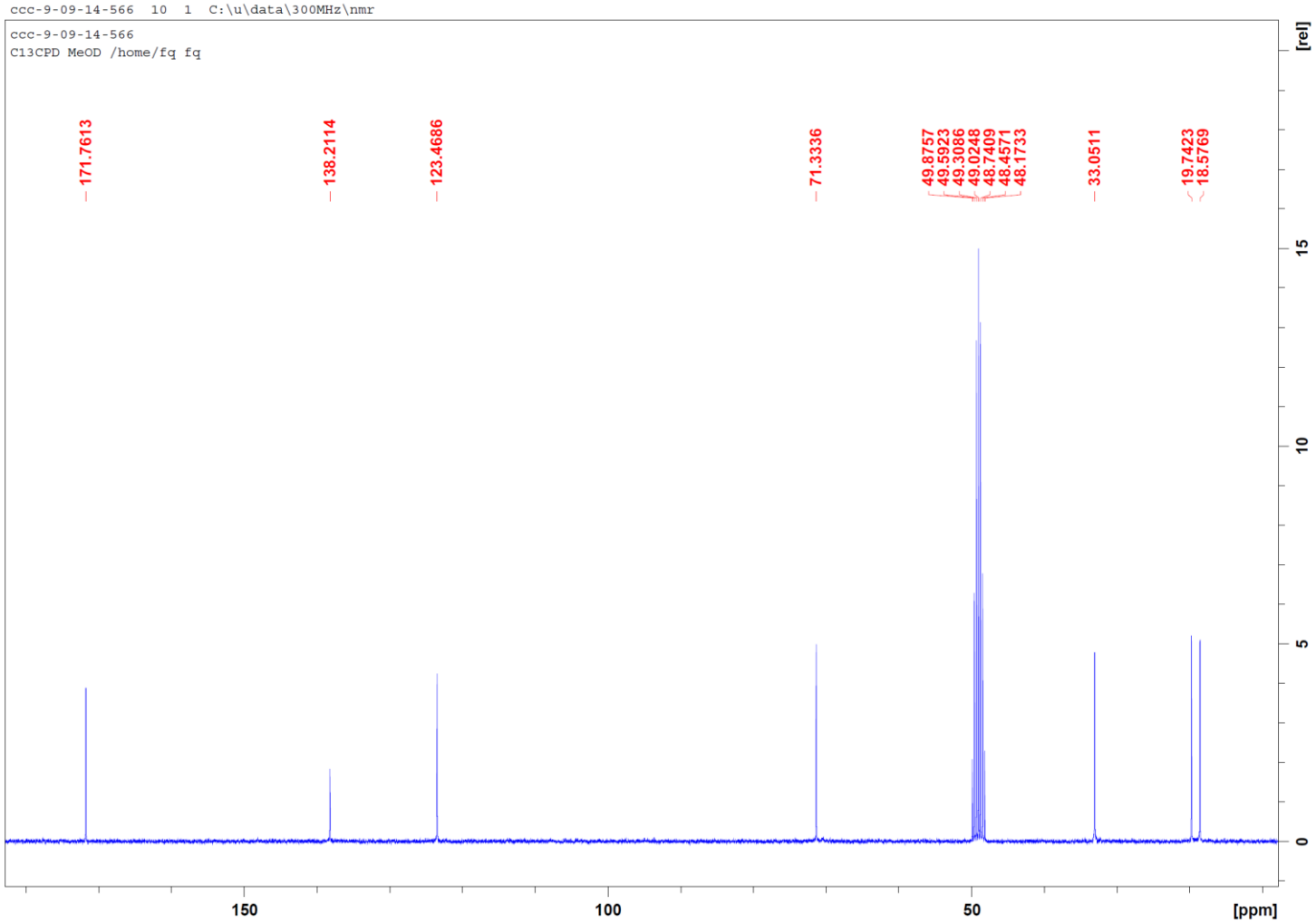
Espectro de masas de H[(*S*,*S*)-L^{Me}].



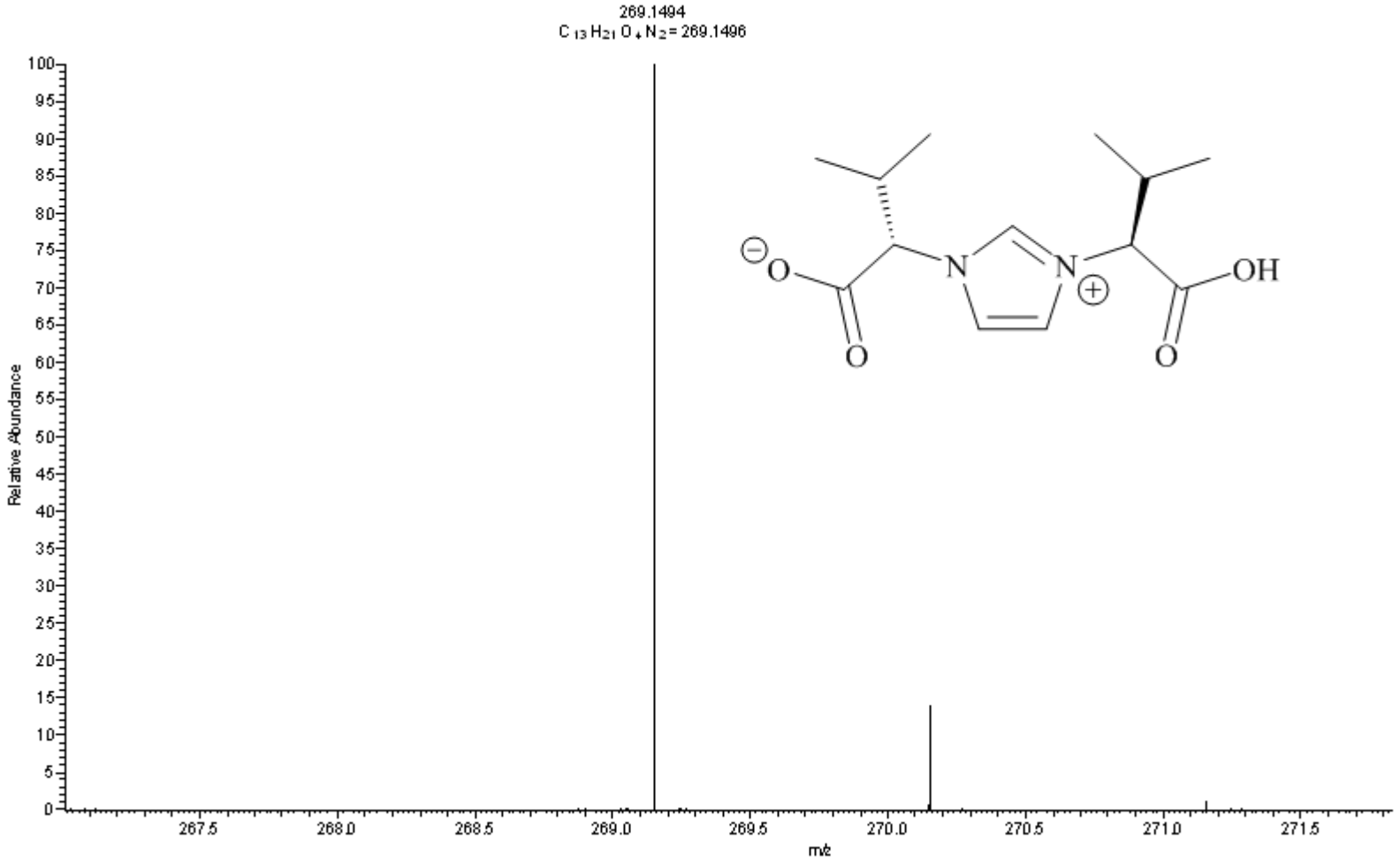
Espectro de RMN de ^{1}H de H[(*S*,*S*)-L^{iPr}].



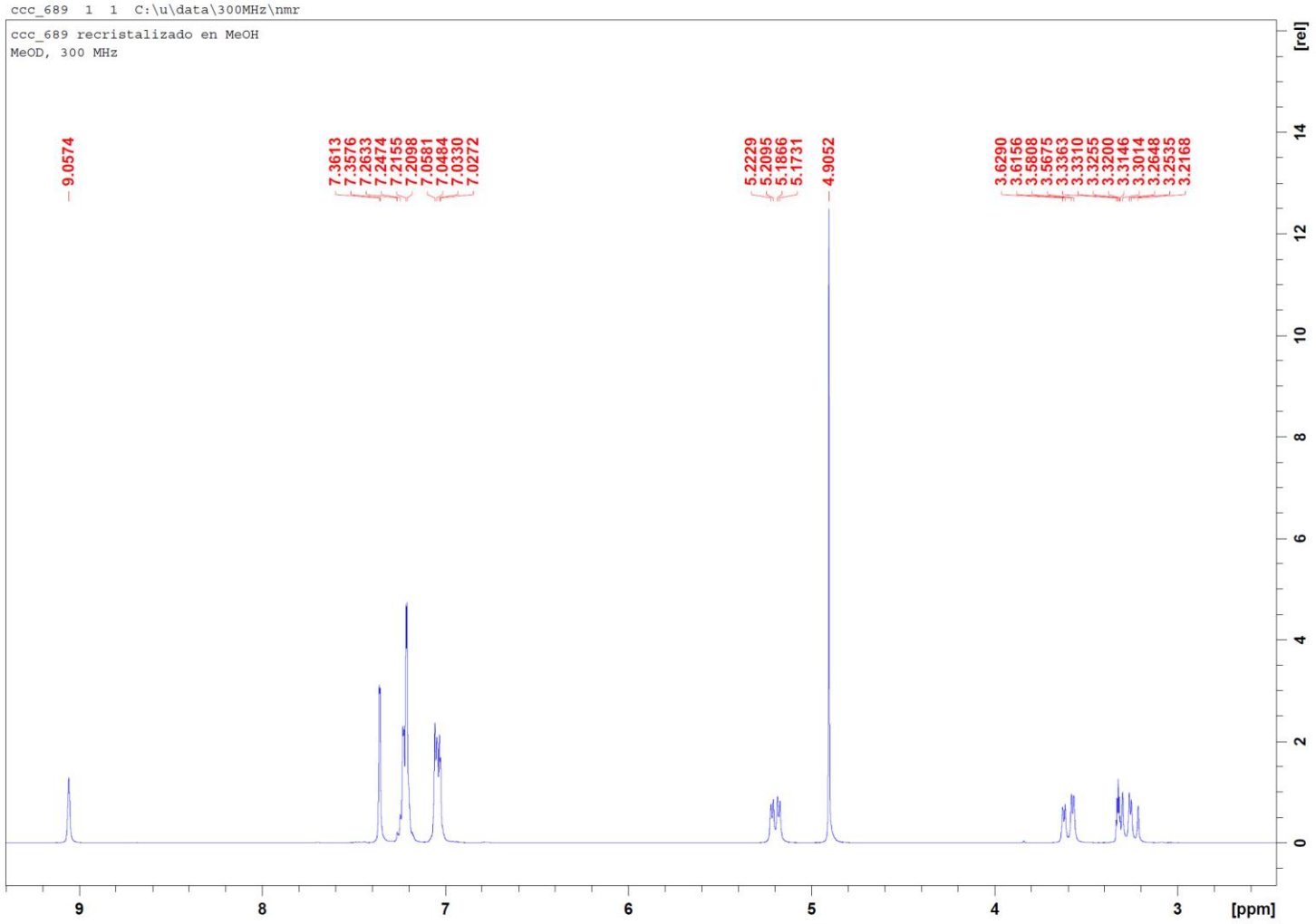
Espectro de RMN de $^{13}C\{^{1}H\}$ de H[(*S*,*S*)-L^{iPr}].



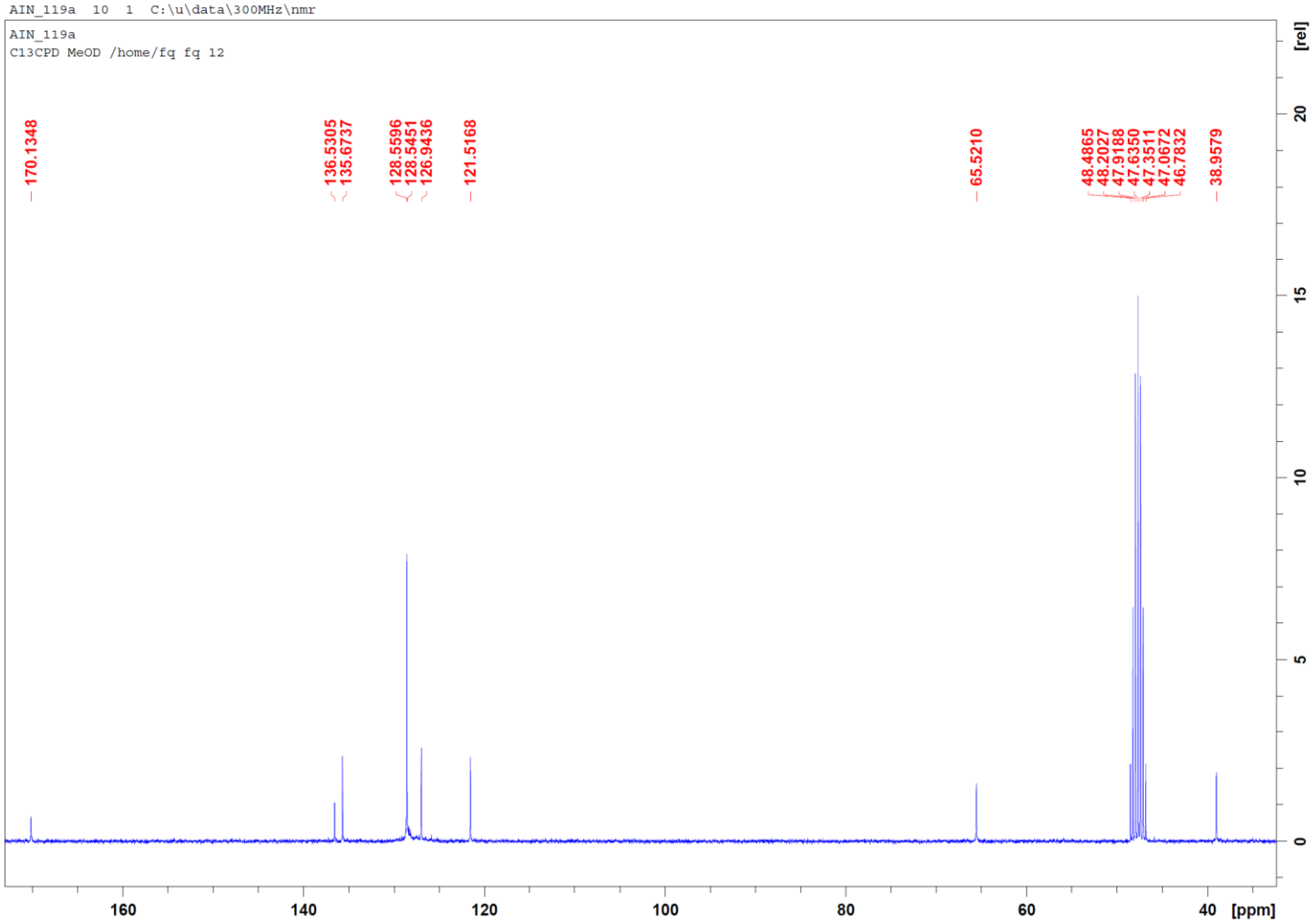
Espectro de masas de H[(*S*,*S*)-L^{iPr}].



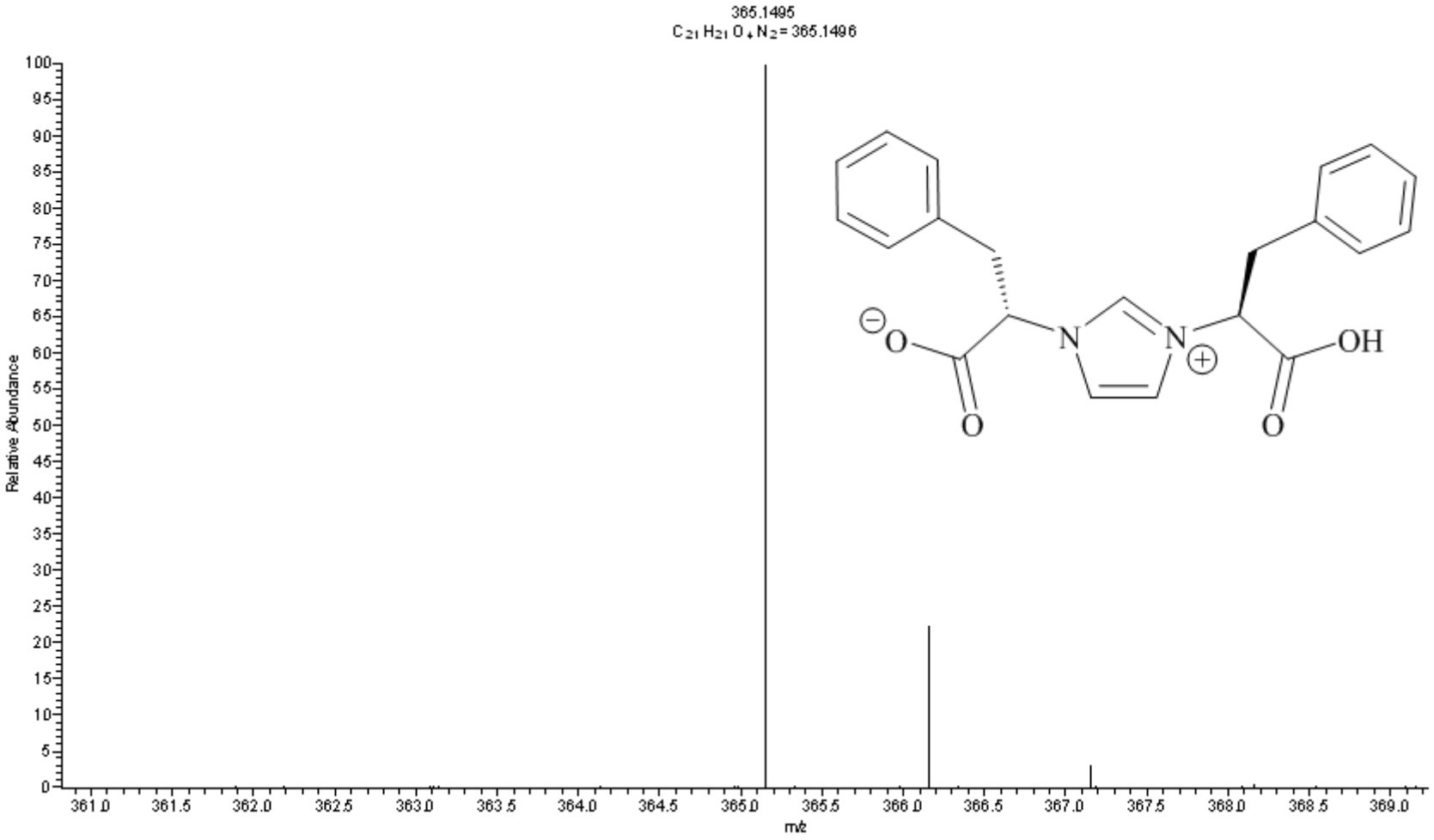
Espectro de RMN de ^{1}H de H[(*S*,*S*)-L^{CH2Ph}].



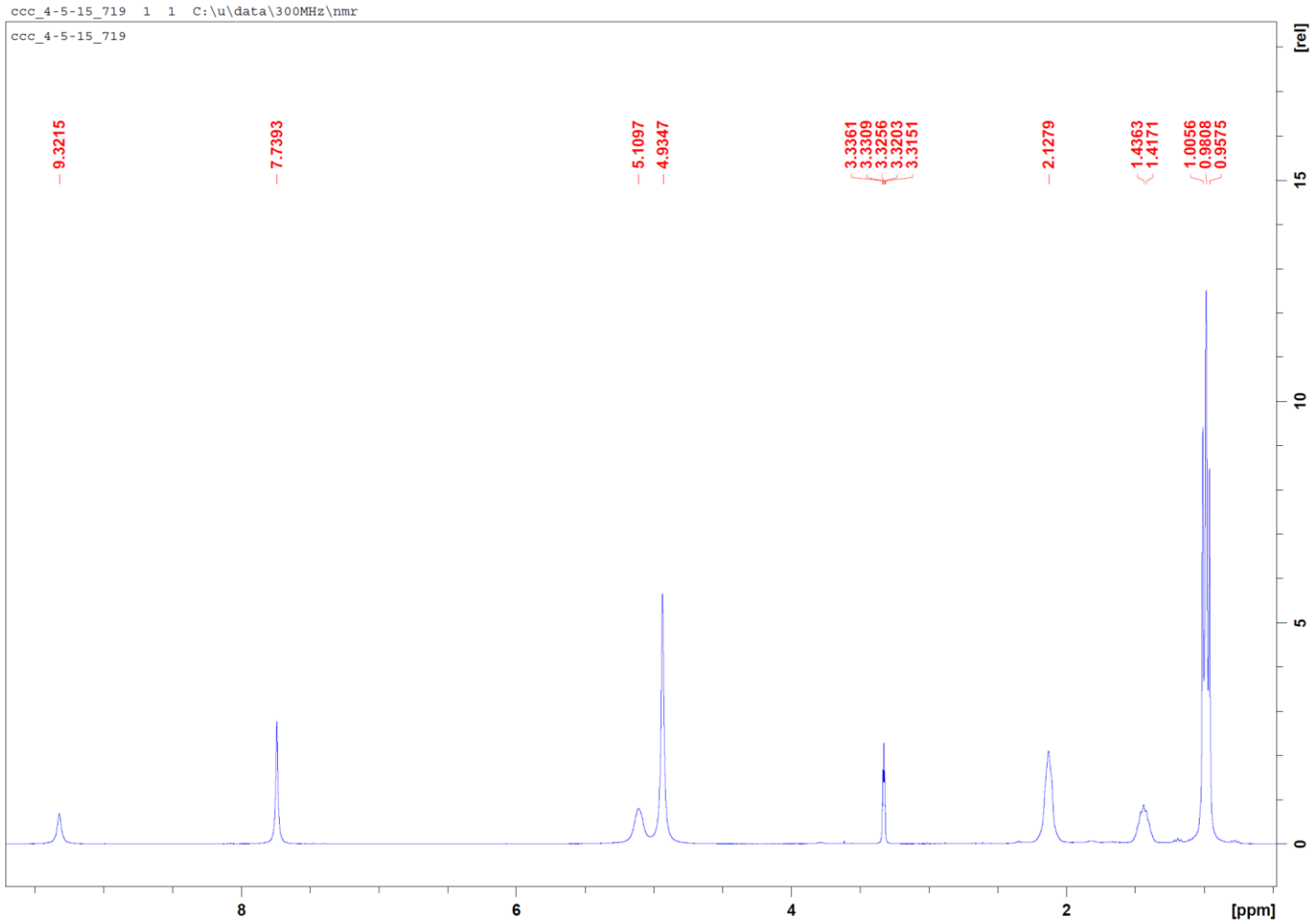
Espectro de RMN de $^{13}C\{^{1}H\}$ de H[(*S*,*S*)-L^{CH2Ph}].



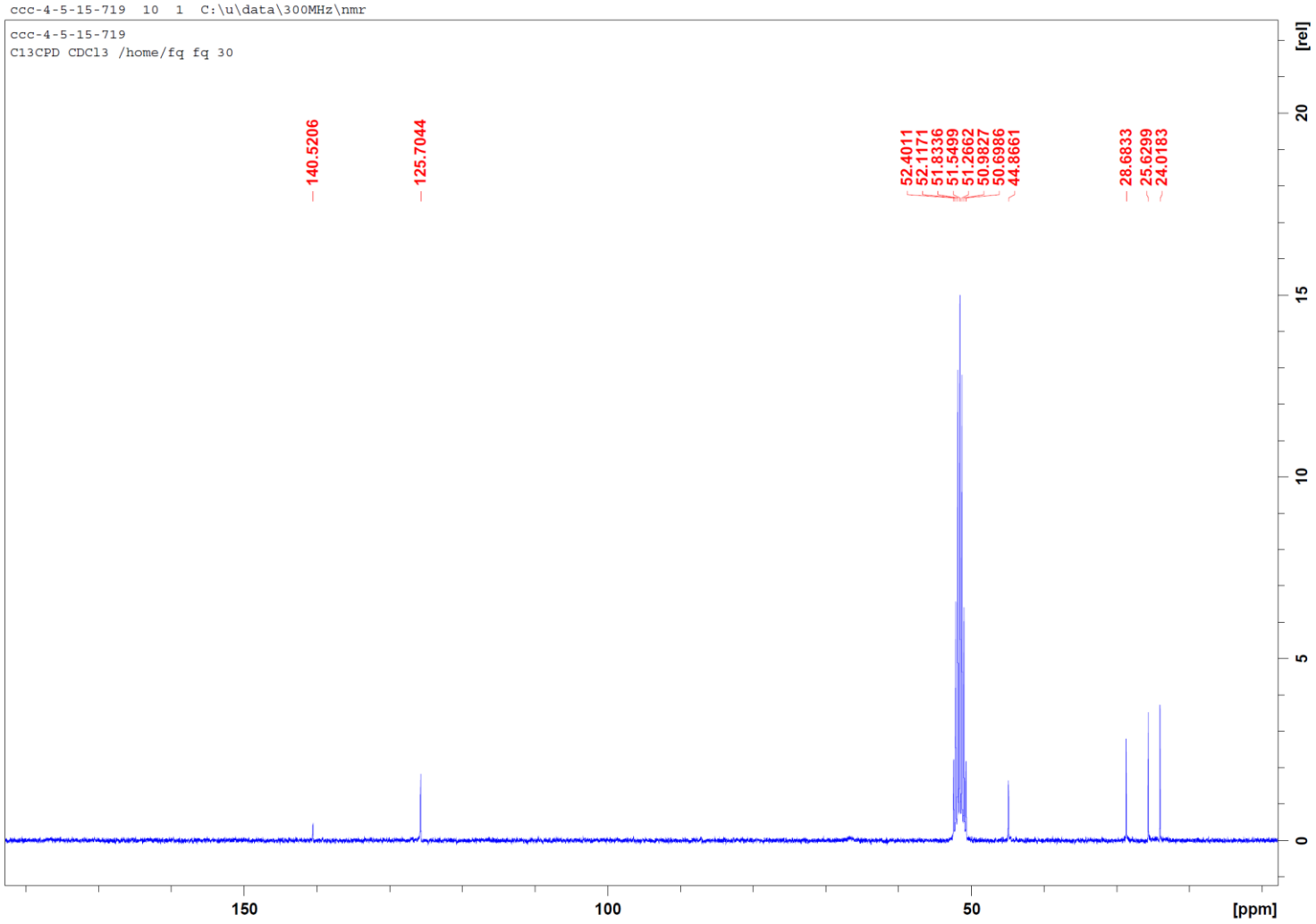
Espectro de masas de H[(*S*,*S*)-L^{CH2Ph}].



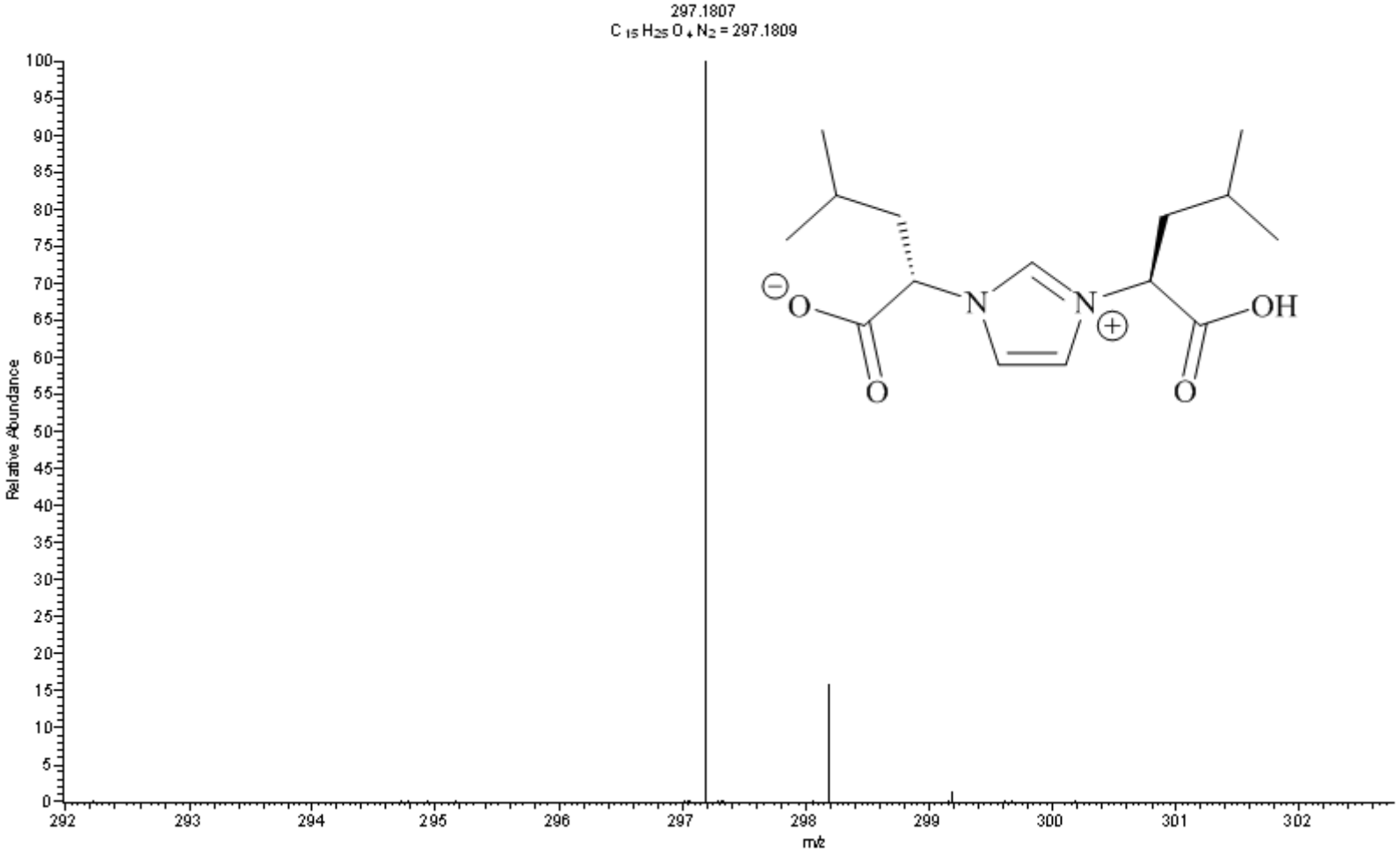
Espectro de RMN de ^{1}H de H[(*S*,*S*)-L^{iBu}].



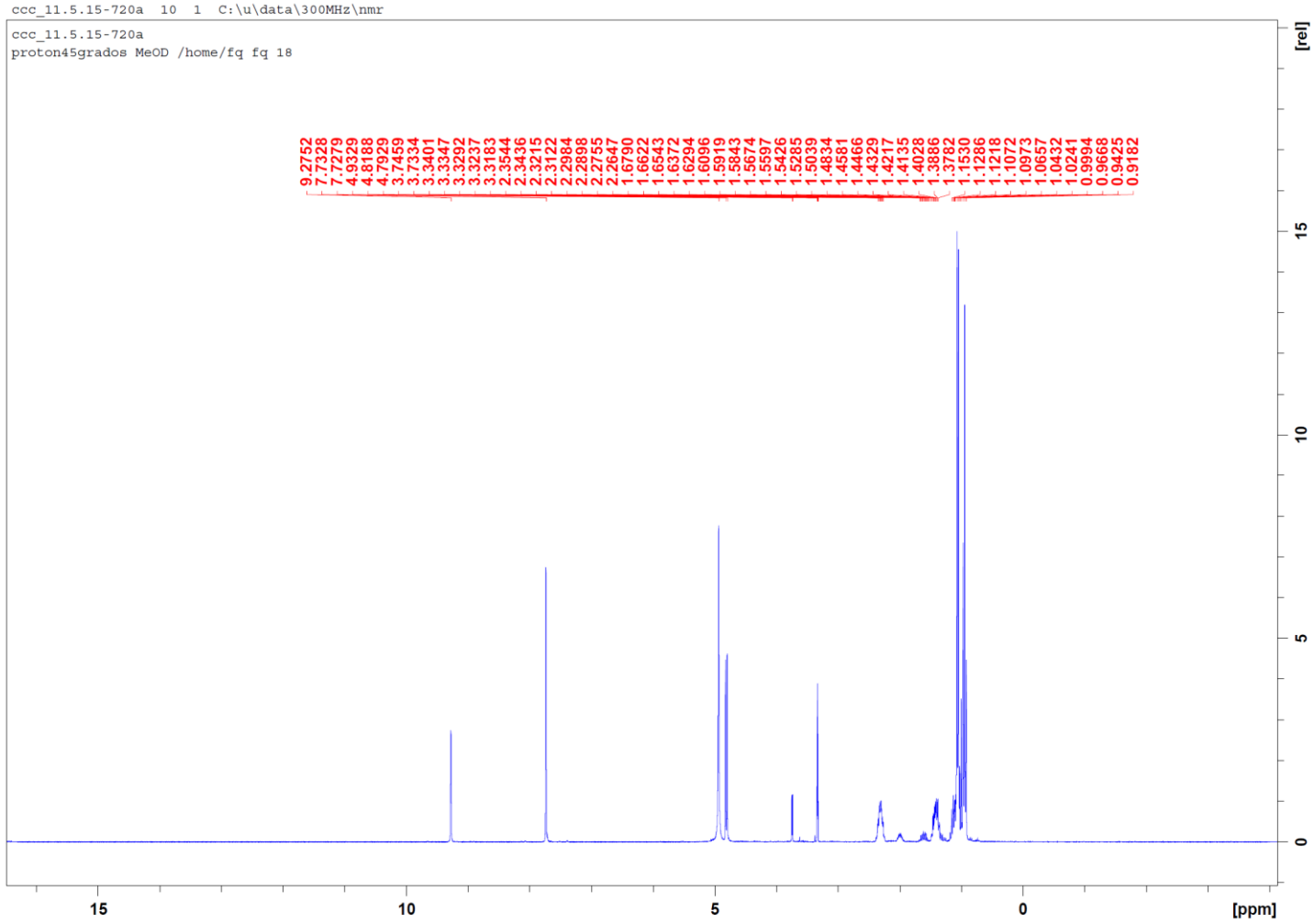
Espectro de RMN de $^{13}C\{^{1}H\}$ de H[(*S*,*S*)-L^{iBu}].



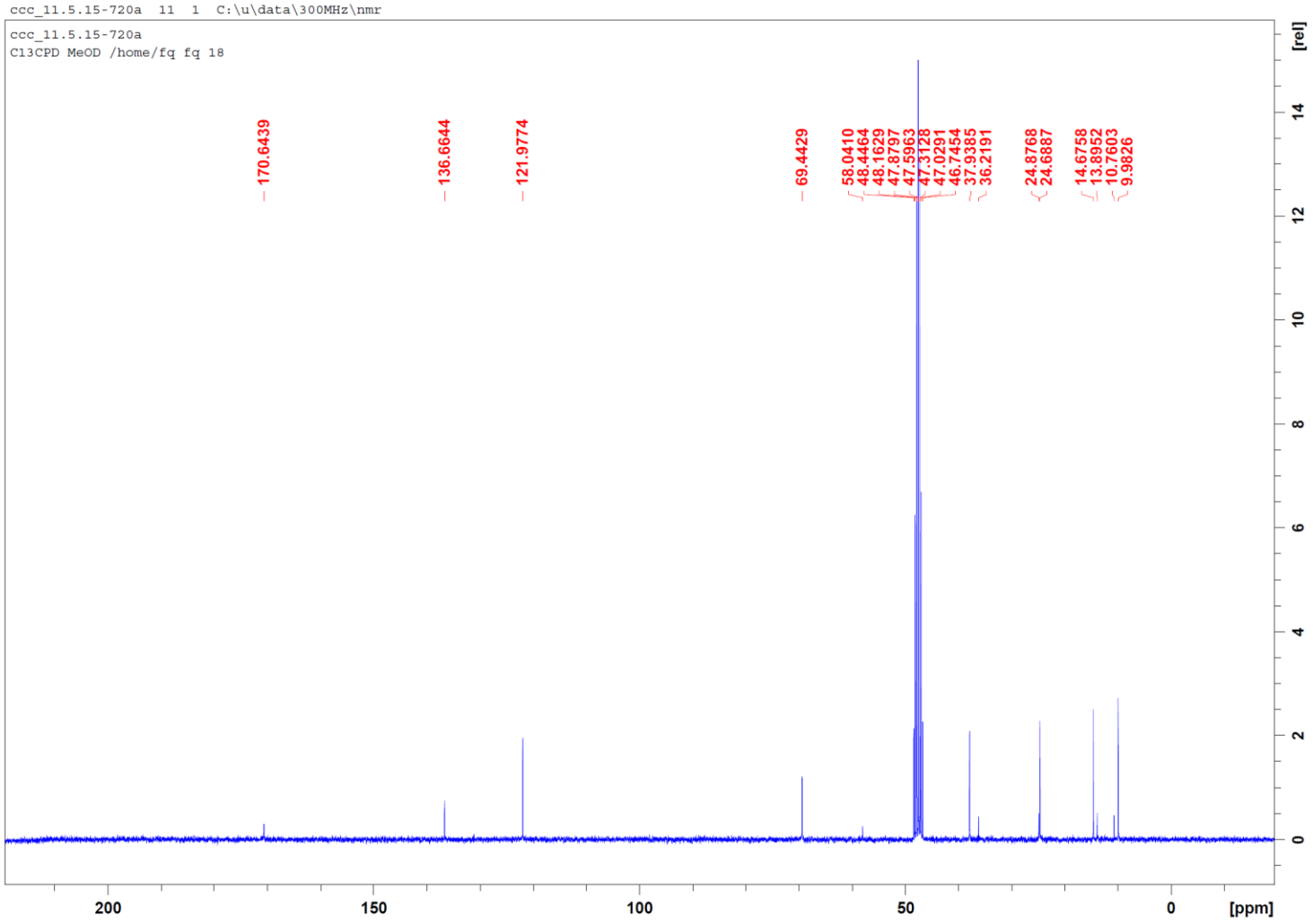
Espectro de masas de H[(*S*,*S*)-L^{iBu}].



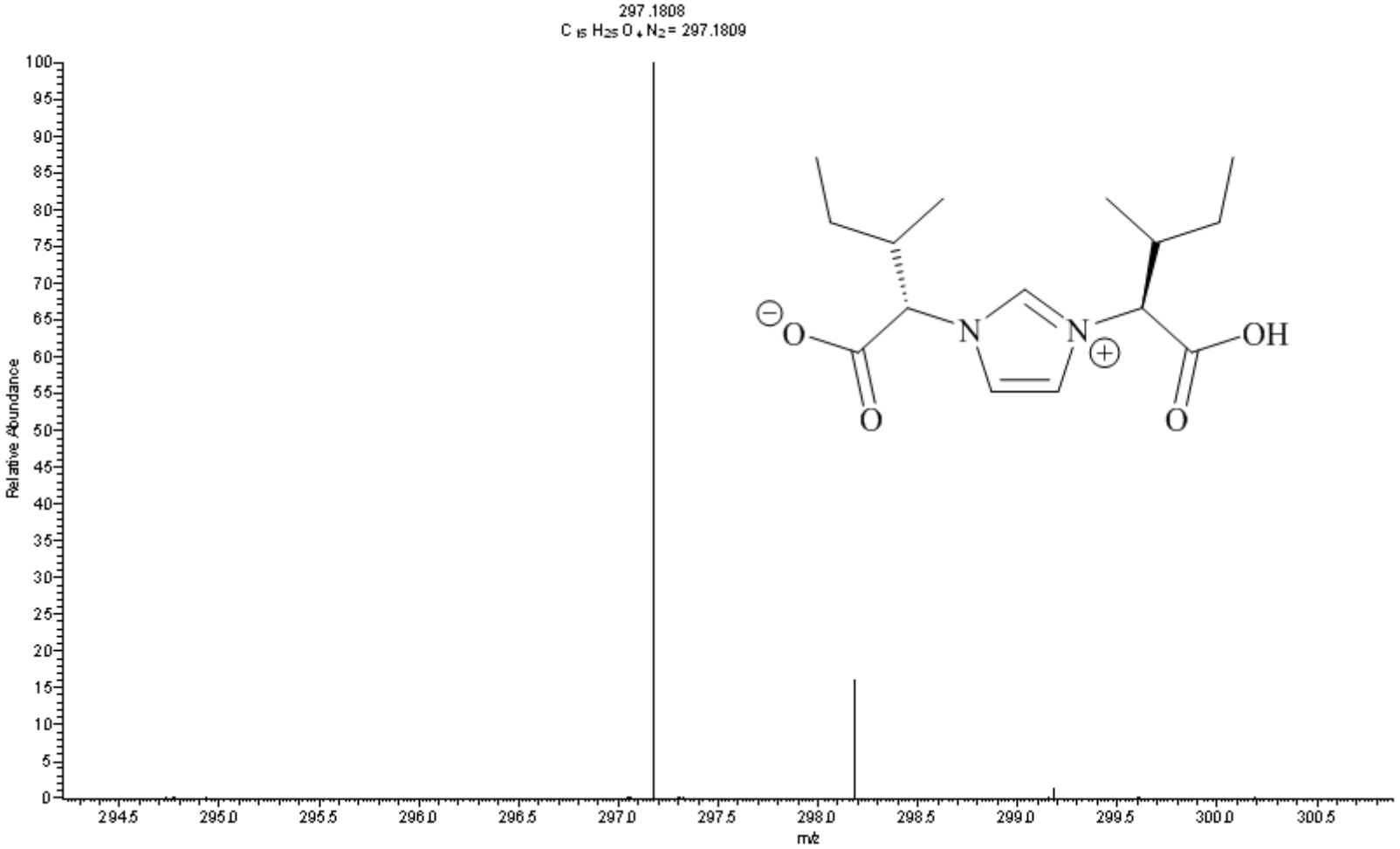
Espectro de RMN de ^{1}H de H[(*S*,*S*)-L$^{sec\text{-}Bu}$].



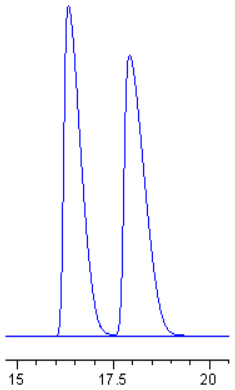
Espectro de RMN de $^{13}C\{^{1}H\}$ de H[(*S*,*S*)-$L^{sec\text{-}Bu}$].



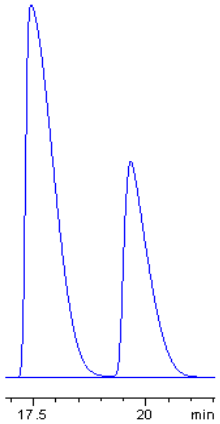
Espectro de masas de H[(*S*,*S*)-L$^{sec\text{-}Bu}$].



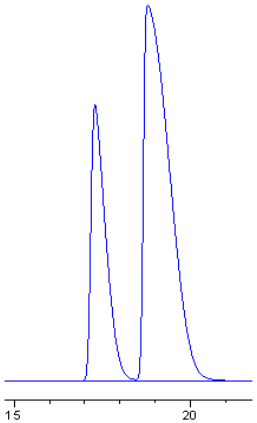
Anexo 10. Selección de espectros de HPLC de sulfóxidos racémicos y ópticamente activos con diferentes *ee*.[*]



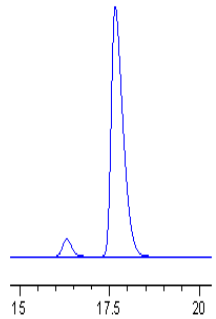
PhMeSO racémico



(*S*)-PhMeSO (33 % *ee*)



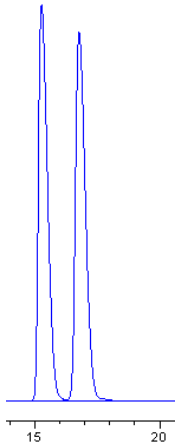
(*R*)-PhMeSO (40 % *ee*)



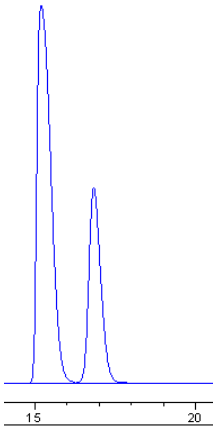
(*R*)-PhMeSO (89 % *ee*)

Poder Rotatorio seleccionado: $[\alpha]_D^{25}$ (MeOH) data:	+55.2	(*R*)-PhMeSO (40 % *ee*)
	+47.9	(*R*)-PhMeSO (34 % *ee*)
	-47.5	(*S*)-PhMeSO (33 % *ee*)

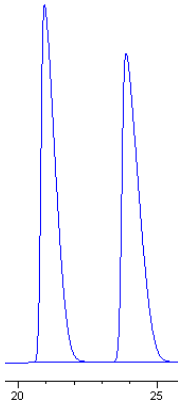
[*] Los análisis de HPLC se llevaron a cabo con una columna ChiralPak IA, con flujo de 1ml/min, una mezcla de eluyente de AcOEt/Heptano de 6/4 (v/v) y un detector de UV de 254 nm.



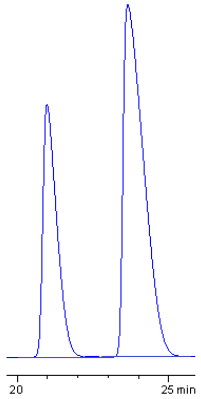
Ph(PhCH$_2$)SO racémico



(*R*)-Ph(PhCH$_2$)SO (38 % *ee*)



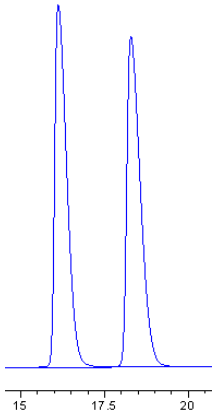
(*p*-Me-C$_6$H$_4$)MeSO racémico



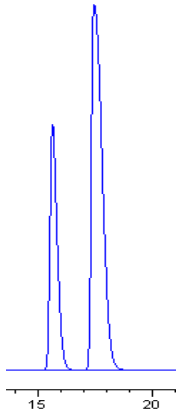
(*R*)-(*p*-Me-C$_6$H$_4$)MeSO (34 % *ee*)

Poder Rotatorio seleccionado: $[\alpha]_D^{25}$ (MeOH) data:

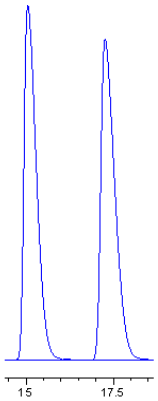
+69.6	(*R*)-Ph(PhCH$_2$)SO (38 % *ee*)
+34.5	(*R*)-(*p*-Me-C$_6$H$_4$)MeSO (34 % *ee*)



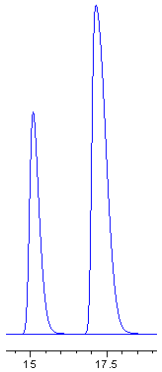
(*p*-Br-C_6H_4)MeSO racémico



(*R*)-(*p*-Br-C_6H_4)MeSO (38 % *ee*)



(*p*-Cl-C_6H_4)MeSO racémico

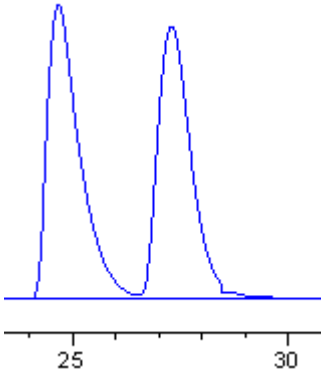


(*R*)-(*p*-Cl-C_6H_4)MeSO (36 % *ee*)

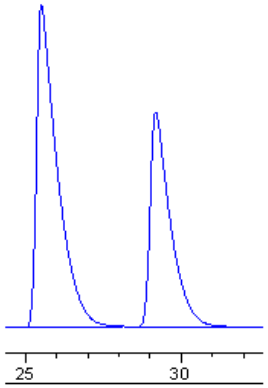
Poder Rotatorio seleccionado $[\alpha]_D^{25}$ (MeOH) data:

+32.8 (*R*)-(*p*-Br-C_6H_4)MeSO (38 % *ee*)

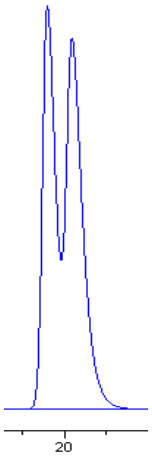
+36.5 (*R*)-(*p*-Cl-C_6H_4)MeSO (36 % *ee*)



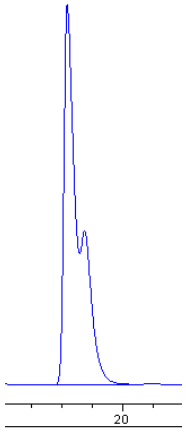
Ph(HOCH$_2$CH$_2$)SO racémico



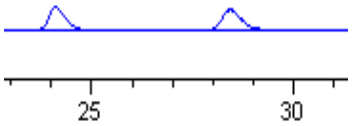
(*S*)-Ph(HOCH$_2$CH$_2$)SO (21 % *ee*)



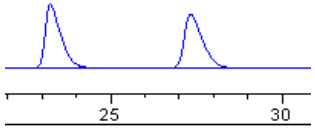
PhEtSO racémico



(*R*)-PhEtSO
(21 % *ee*, ver **DESARROLLO 1**)



Sulfóxido de benzotiofeno racémico

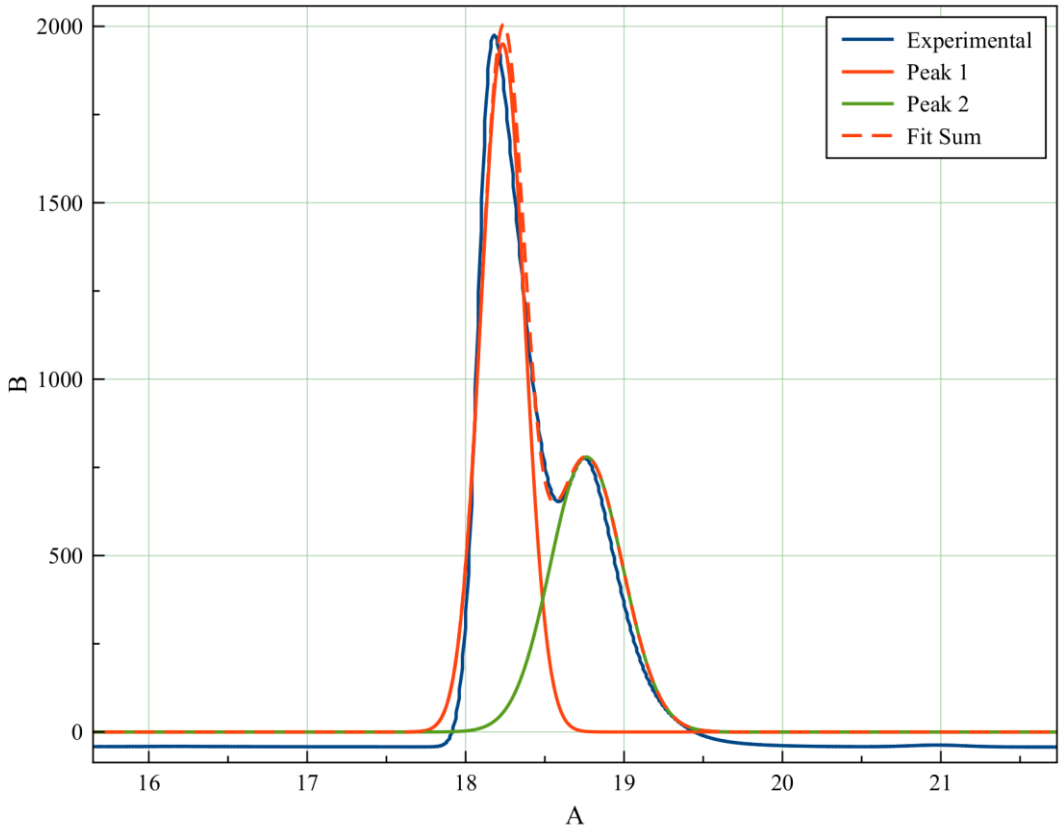


(*S*)-Sulfóxido de benzotiofeno (BTFO) (1 % *ee*)

Poder Rotatorio seleccionado $[\alpha]_D^{25}$ (MeOH) data:

+31.2	(*S*)-Ph(HOCH$_2$CH$_2$)SO (21 % *ee*)
+76.5	(*R*)-PhEtSO (21 % *ee*)
-0.05	(S)-BTFO (1 % *ee*)

Determinación del área de los picos en el espectro de HPLC del sulfóxido (*R*)-PhEtSO:



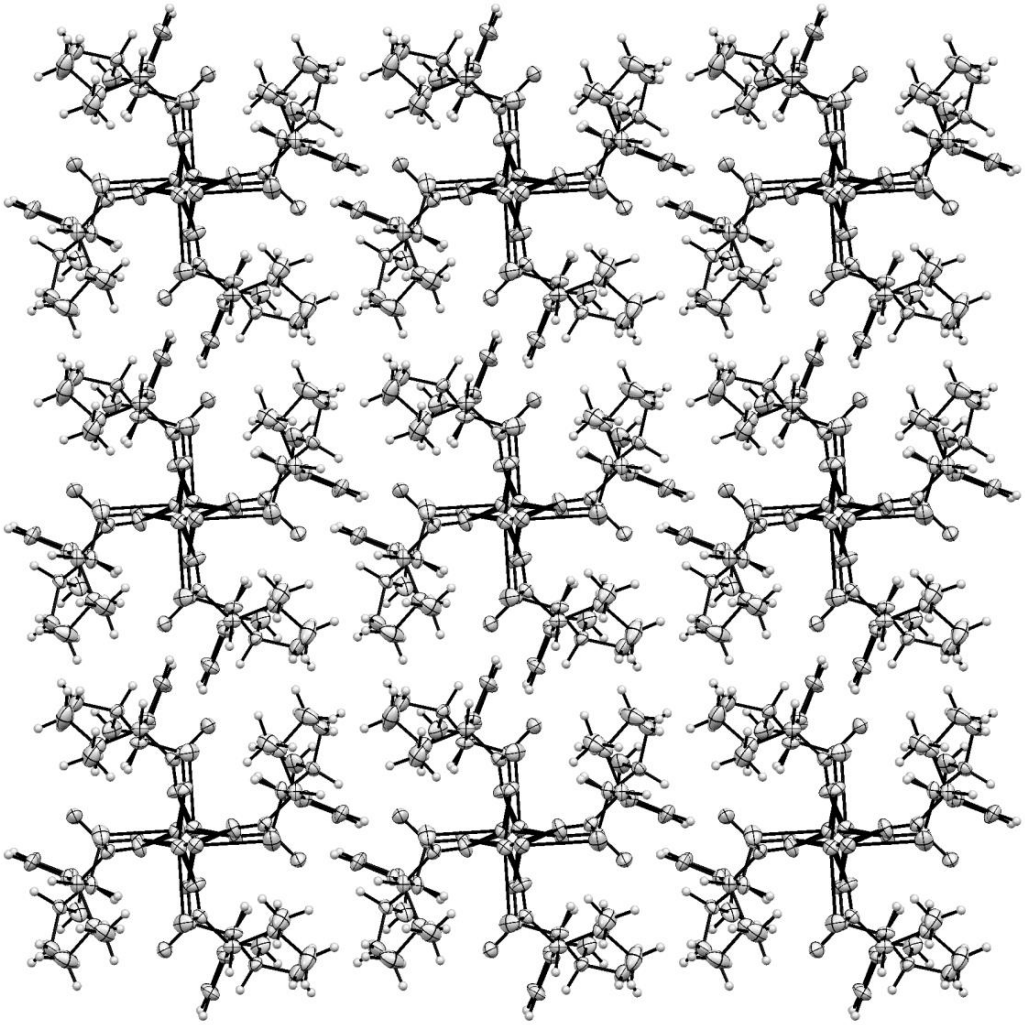
La búsqueda del pico apropiado se realizó usando el software MagicPlot Pro, asumiendo que el pico mantiene una distribución Gaussiana.

Área del pico 1 = 678.68

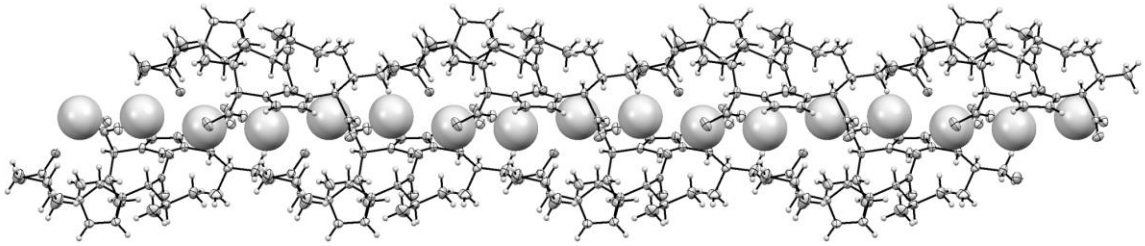
Área del pico 2 = 446.37

ee calculado = 21 %.

Anexo 11. (a) Empaquetamiento cristalino del compuesto Na[(*S*,*S*)-L^{iPr}]$_n$ a lo largo del eje *c* (3x3). (b) Cadena de iones sodio (al 50% del radio para mayor claridad) a lo largo del eje *c* en el compuesto Na[(*S*,*S*)-L^{iPr}]$_n$ (vista desde el eje *b*).



(a)



(b)

Anexo 12. Datos cristalográficos del compuesto Na[(*S*,*S*)-L^{iPr}]$_n$.

	Na[(*S*,*S*)-L^{iPr}]$_n$
Fórmula	$C_{13}H_{19}N_2NaO_4$
Masa molecular	290.29
Sistema cristalino	Tetragonal
Grupo espacial	$P\,4_1$
a, (Å)	10.7327(6)
b, (Å)	10.7327(6)
c, (Å)	12.6277(10)
α, (º)	90
β, (º)	90
γ, (º)	90
V, (Å^3)	1454.60(16)
Z, F(000)	4, 616
D_{calc}, (Mg·m^{-3})	1.326
μ, (mm^{-1})	0.123
θ_{max}, (º)	25.2
Nº de reflexiones recogidos	10394
Nº de refrexiones usadas	2534
Nº de parámetros.	187
$R_1(F)$ [$F^2>2\sigma(F^2)$][a]	0.0288
$wR_2(F^2)$[b](todos los datos incluídos)	0.0778
S[c] (todos los datos incluídos)	1.099

[a] $R_1(F) = \sum(|F_o|-|F_c|)/\sum|F_o|$ para las reflexiones observadas [$F^2>2\sigma(F^2)$].

[b] $wR_2(F^2) = \{\sum [w(F_o^2- F_c^2)^2]/ \sum w(F_o^2)^2\}^{1/2}$.

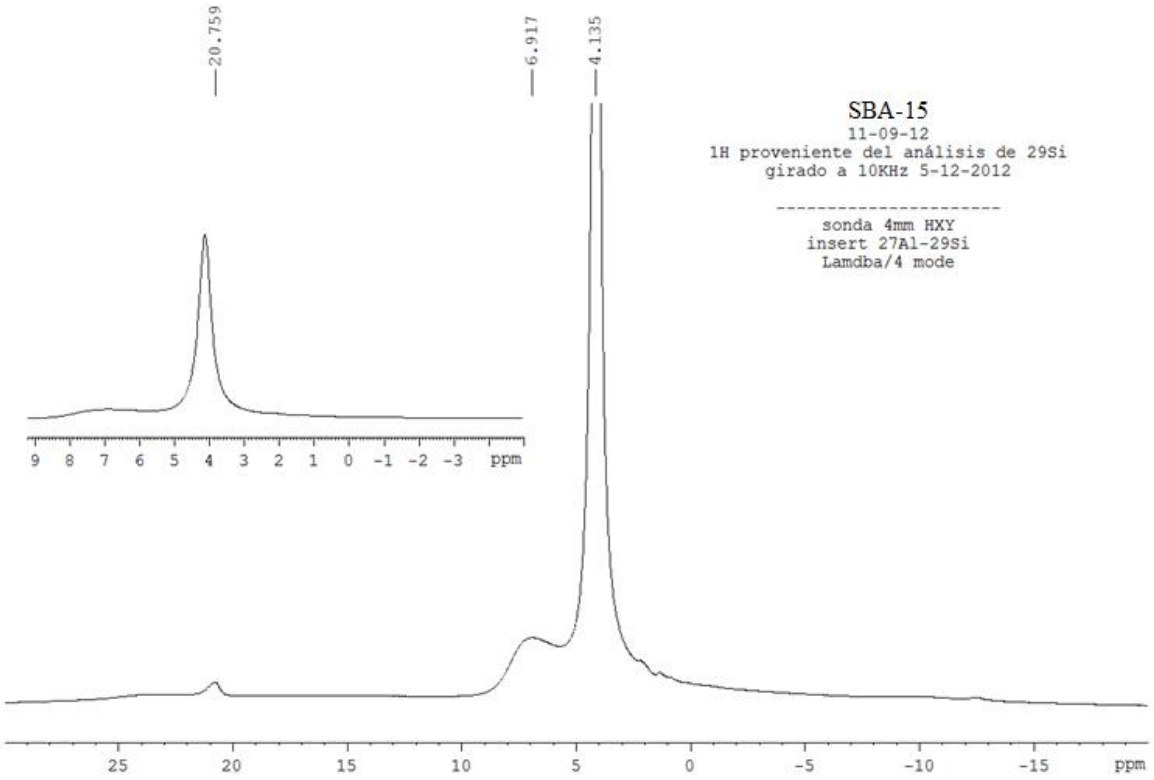
[c] $S = \{\sum [w(F_o^2- F_c^2)^2]/(n-p)\}^{1/2}$; (n = número de reflexiones, p = número de parámetros)

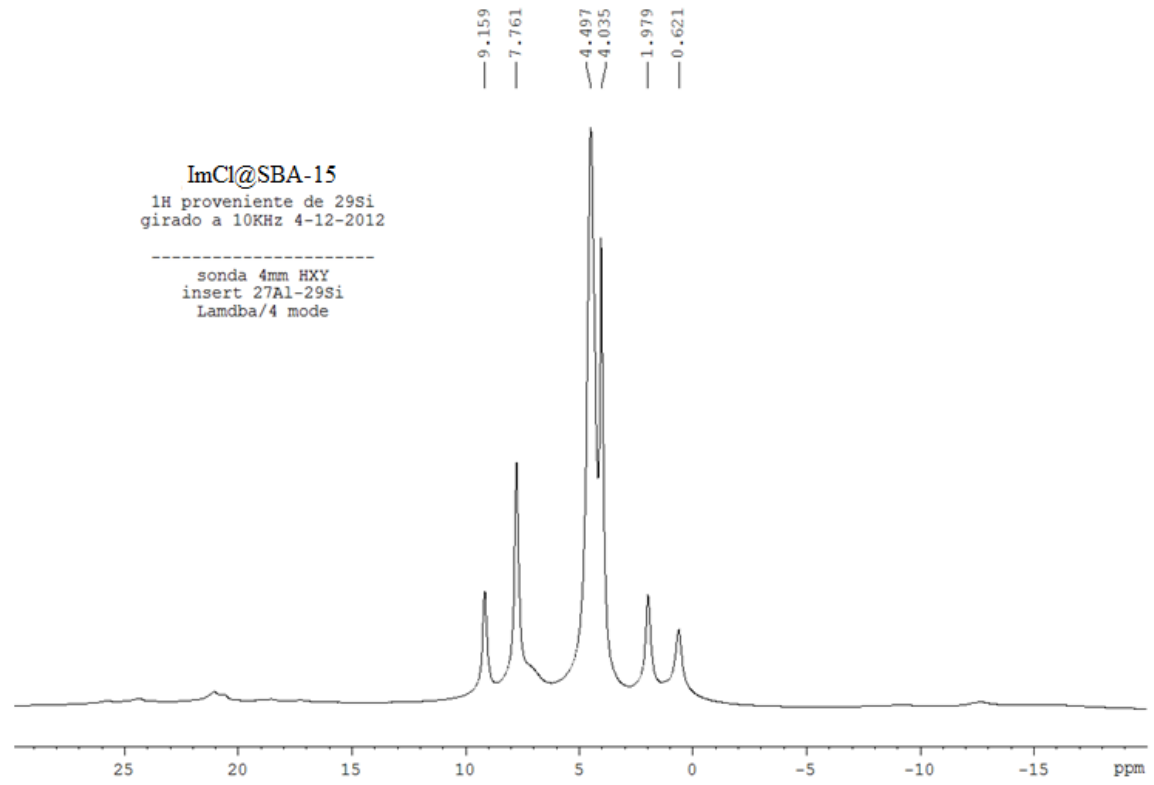
Anexo 13. Longitudes [Å] y ángulos [°] de enlace seleccionados del compuesto Na[(*S*,*S*)-L^{iPr}]$_n$.

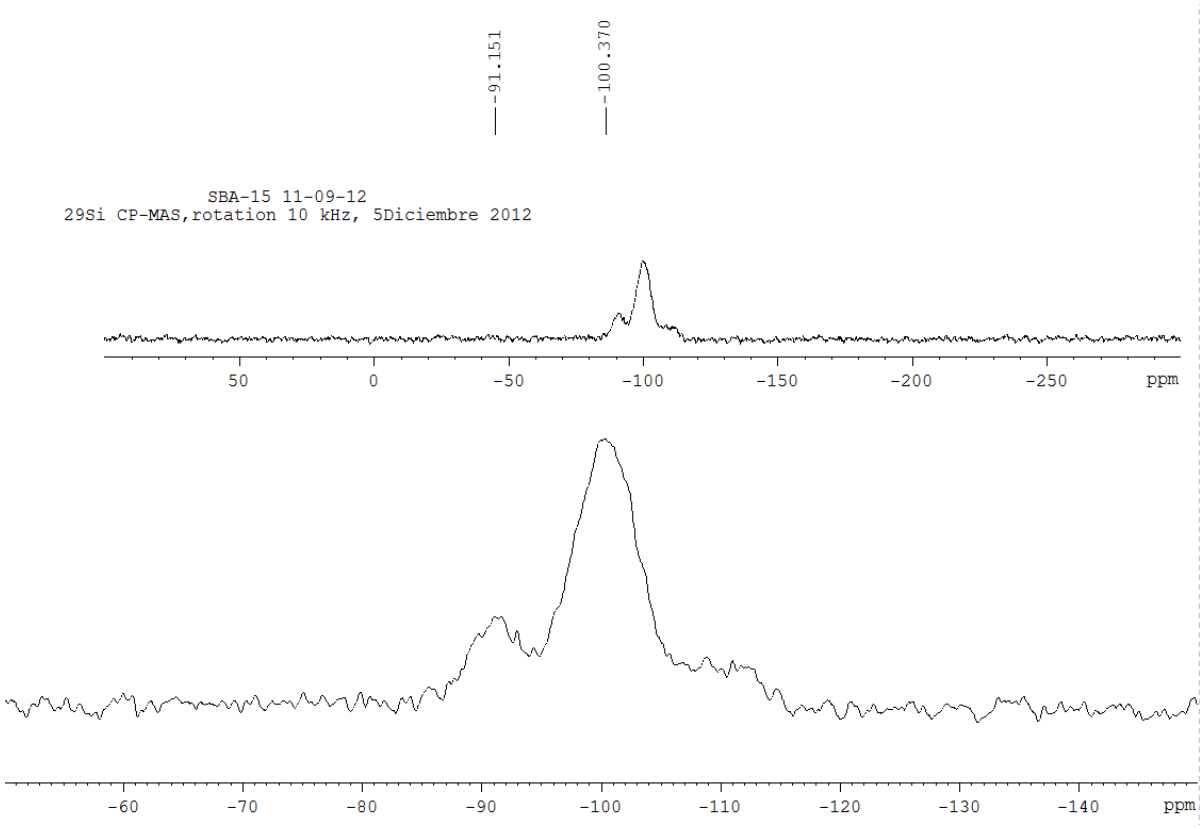
Na(1)-O(1)#1	2.258(2)	N(1)-C(1)	1.325(3)
Na(1)-O(3)#2	2.260(2)	N(1)-C(3)	1.381(3)
Na(1)-O(1)	2.364(2)	N(1)-C(4)	1.481(3)
Na(1)-O(3)#3	2.372(2)	N(2)-C(1)	1.332(3)
Na(1)-O(4)#3	2.516(2)	N(2)-C(2)	1.375(3)
Na(1)-C(10)#3	2.688(3)	N(2)-C(9)	1.478(3)
Na(1)-Na(1)#1	3.206(1)	C(2)-C(3)	1.347(4)
O(1)-C(5)	1.259(3)	C(4)-C(6)	1.533(3)
O(1)-Na(1)#2	2.258(2)	C(4)-C(5)	1.547(3)
O(2)-C(5)	1.231(3)	C(6)-C(7)	1.520(4)
O(3)-C(10)	1.252(3)	C(6)-C(8)	1.527(4)
O(3)-Na(1)#1	2.260(2)	C(9)-C(11)	1.532(4)
O(3)-Na(1)#4	2.372(2)	C(9)-C(10)	1.539(4)
O(4)-C(10)	1.236(3)	C(11)-C(12)	1.519(4)
O(4)-Na(1)#4	2.516(2)	C(11)-C(13)	1.523(4)
O(1)#1-Na(1)-O(3)#2	91.31(7)	Na(1)#2-O(1)-Na(1)	87.80(7)
O(1)#1-Na(1)-O(1)	114.65(6)	C(10)-O(3)-Na(1)#1	170.5(2)
O(3)#2-Na(1)-O(1)	91.38(8)	C(10)-O(3)-Na(1)#4	90.28(15)
O(1)#1-Na(1)-O(3)#3	148.00(8)	Na(1)#1-O(3)-Na(1)#4	87.55(7)
O(3)#2-Na(1)-O(3)#3	113.33(6)	C(10)-O(4)-Na(1)#4	84.14(15)
O(1)-Na(1)-O(3)#3	86.04(7)	C(1)-N(1)-C(3)	109.0(2)
O(1)#1-Na(1)-O(4)#3	101.51(8)	C(1)-N(1)-C(4)	122.43(19)
O(3)#2-Na(1)-O(4)#3	105.08(9)	C(3)-N(1)-C(4)	128.5(2)
O(1)-Na(1)-O(4)#3	139.88(8)	C(1)-N(2)-C(2)	108.5(2)
O(3)#3-Na(1)-O(4)#3	53.88(7)	C(1)-N(2)-C(9)	125.7(2)
O(1)#1-Na(1)-C(10)#3	128.24(8)	C(2)-N(2)-C(9)	125.8(2)
O(3)#2-Na(1)-C(10)#3	105.90(9)	N(1)-C(1)-N(2)	108.4(2)
O(1)-Na(1)-C(10)#3	113.32(8)	C(3)-C(2)-N(2)	107.4(2)
O(3)#3-Na(1)-C(10)#3	27.77(7)	C(2)-C(3)-N(1)	106.6(2)
O(4)#3-Na(1)-C(10)#3	27.23(8)	N(1)-C(4)-C(6)	110.79(19)
O(1)#1-Na(1)-Na(1)#1	47.47(5)	N(1)-C(4)-C(5)	110.27(18)
O(3)#2-Na(1)-Na(1)#1	47.67(5)	C(6)-C(4)-C(5)	111.21(19)
O(1)-Na(1)-Na(1)#1	122.85(6)	O(2)-C(5)-O(1)	127.3(2)
O(3)#3-Na(1)-Na(1)#1	140.69(7)	O(2)-C(5)-C(4)	117.5(2)
O(4)#3-Na(1)-Na(1)#1	94.07(7)	O(1)-C(5)-C(4)	115.2(2)
C(10)#3-Na(1)-Na(1)#1	115.22(7)	N(2)-C(9)-C(11)	111.0(2)
O(1)#1-Na(1)-Na(1)#2	138.36(7)	N(2)-C(9)-C(10)	110.5(2)
O(3)#2-Na(1)-Na(1)#2	119.85(6)	C(11)-C(9)-C(10)	110.2(2)
O(1)-Na(1)-Na(1)#2	44.73(5)	O(4)-C(10)-O(3)	126.1(2)
O(3)#3-Na(1)-Na(1)#2	44.78(5)	O(4)-C(10)-C(9)	117.4(2)
O(4)#3-Na(1)-Na(1)#2	96.35(6)	O(3)-C(10)-C(9)	116.2(2)
C(10)#3-Na(1)-Na(1)#2	72.30(6)	O(4)-C(10)-Na(1)#4	68.63(14)
Na(1)#1-Na(1)-Na(1)#2	165.86(3)	O(3)-C(10)-Na(1)#4	61.96(13)
C(5)-O(1)-Na(1)#2	143.58(17)	C(9)-C(10)-Na(1)#4	151.62(17)
C(5)-O(1)-Na(1)	126.00(16)		

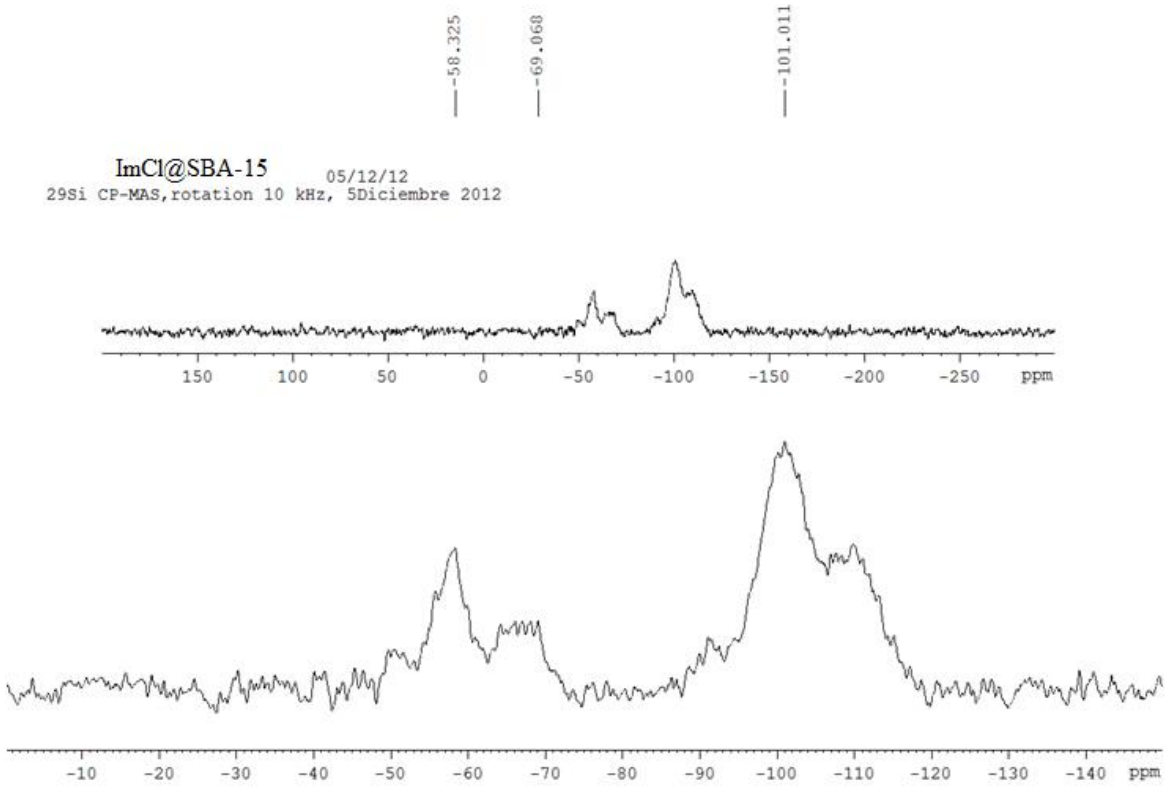
Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1

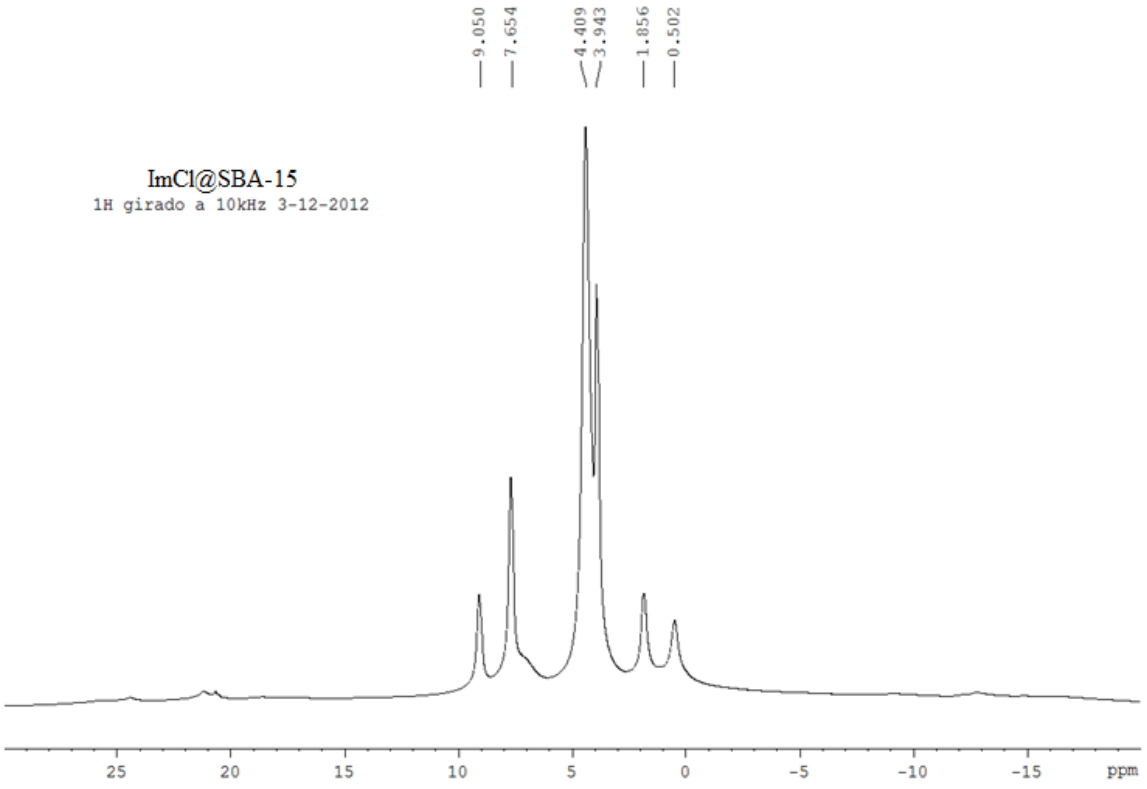
Anexo 14. Espectros de RMN de ^{1}H, ^{13}C y ^{29}Si de los derivados de sílicas mesoporosas SBA-15 y ImCl@SBA-15.

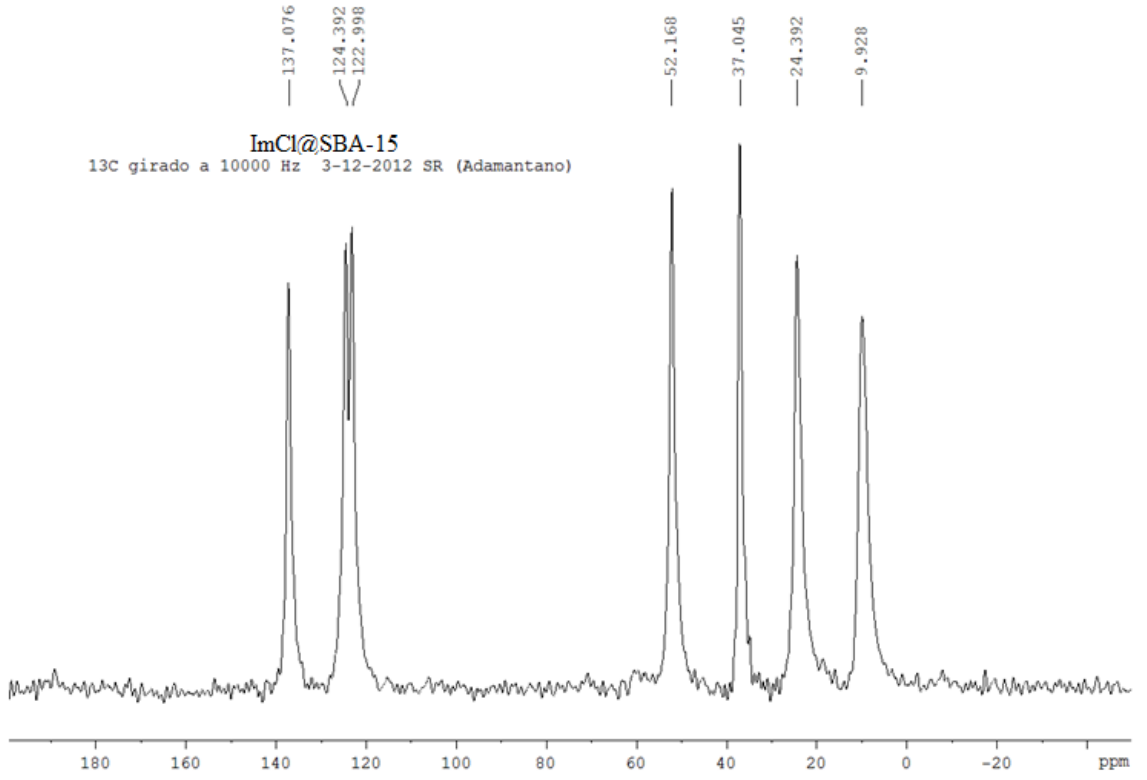





-91.151
-100.370
SBA-15 11-09-12
29Si CP-MAS,rotation 10 kHz, 5Diciembre 2012
50
0
-50
-100
-150
-200
-250
ppm
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm

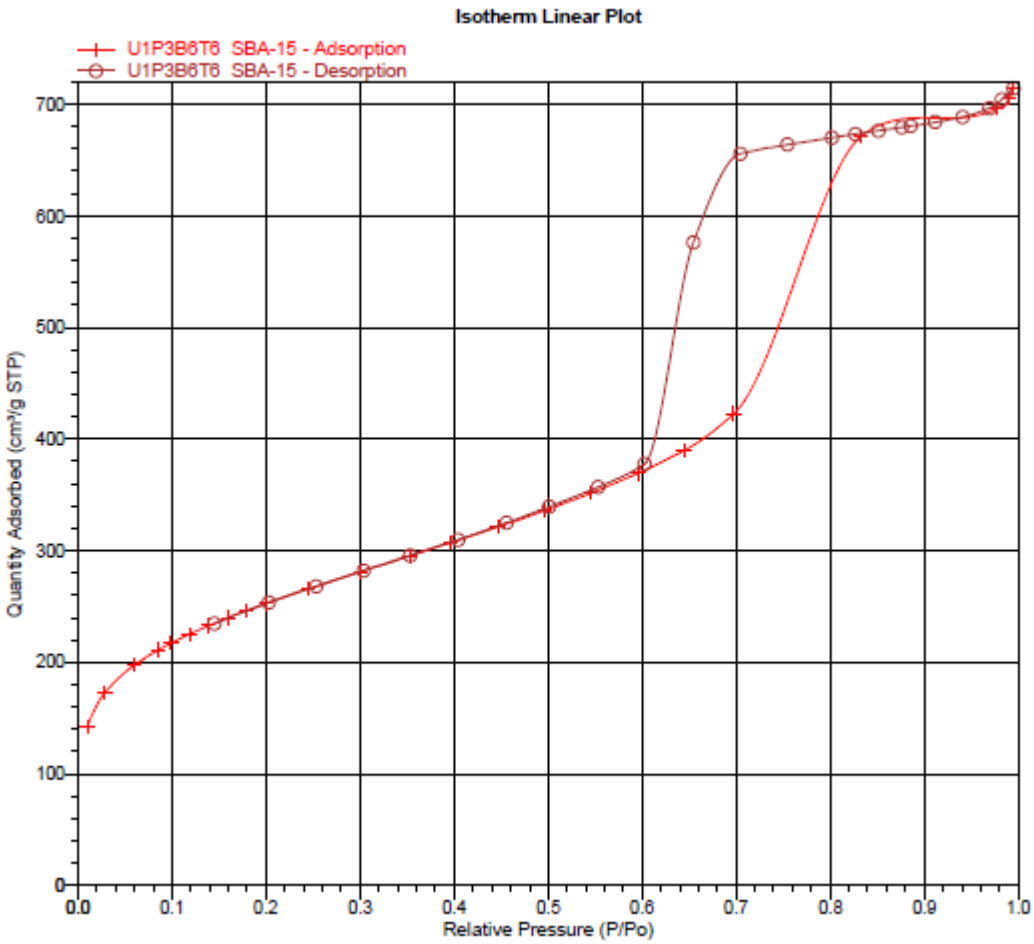

-58.325
-69.068
-101.011
ImCl@SBA-15
05/12/12
29Si CP-MAS,rotation 10 kHz, 5Diciembre 2012
150
100
50
0
-50
-100
-150
-200
-250
ppm
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
ppm


9.050
7.654
4.409
3.943
1.856
0.502
ImCl@SBA-15
1H girado a 10kHz 3-12-2012
25
20
15
10
5
0
-5
-10
-15
ppm

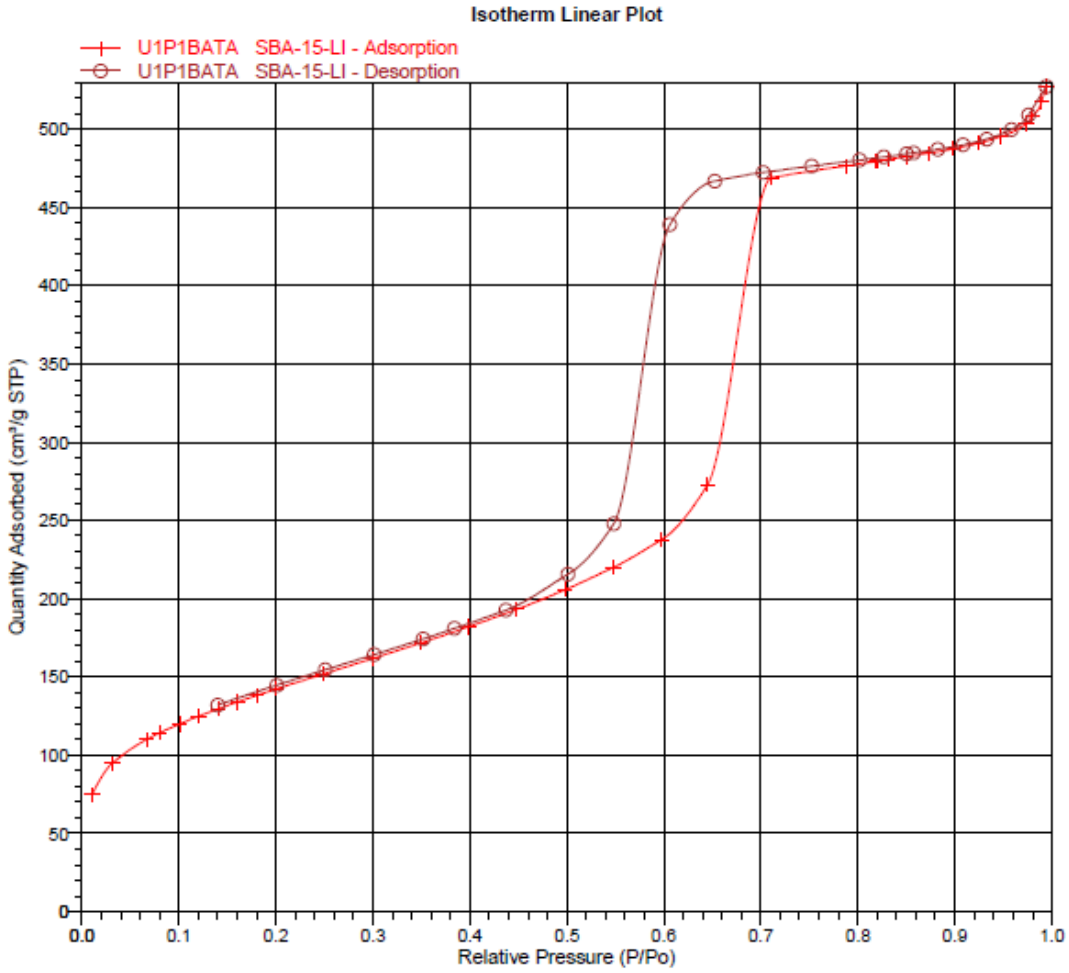

137.076
124.392
122.998
52.168
37.045
24.392
9.928
ImCl@SBA-15
13C girado a 10000 Hz 3-12-2012 SR (Adamantano)
180
160
140
120
100
80
60
40
20
0
-20
ppm

Anexo 15. Isotermas de los materiales porosos (BET).

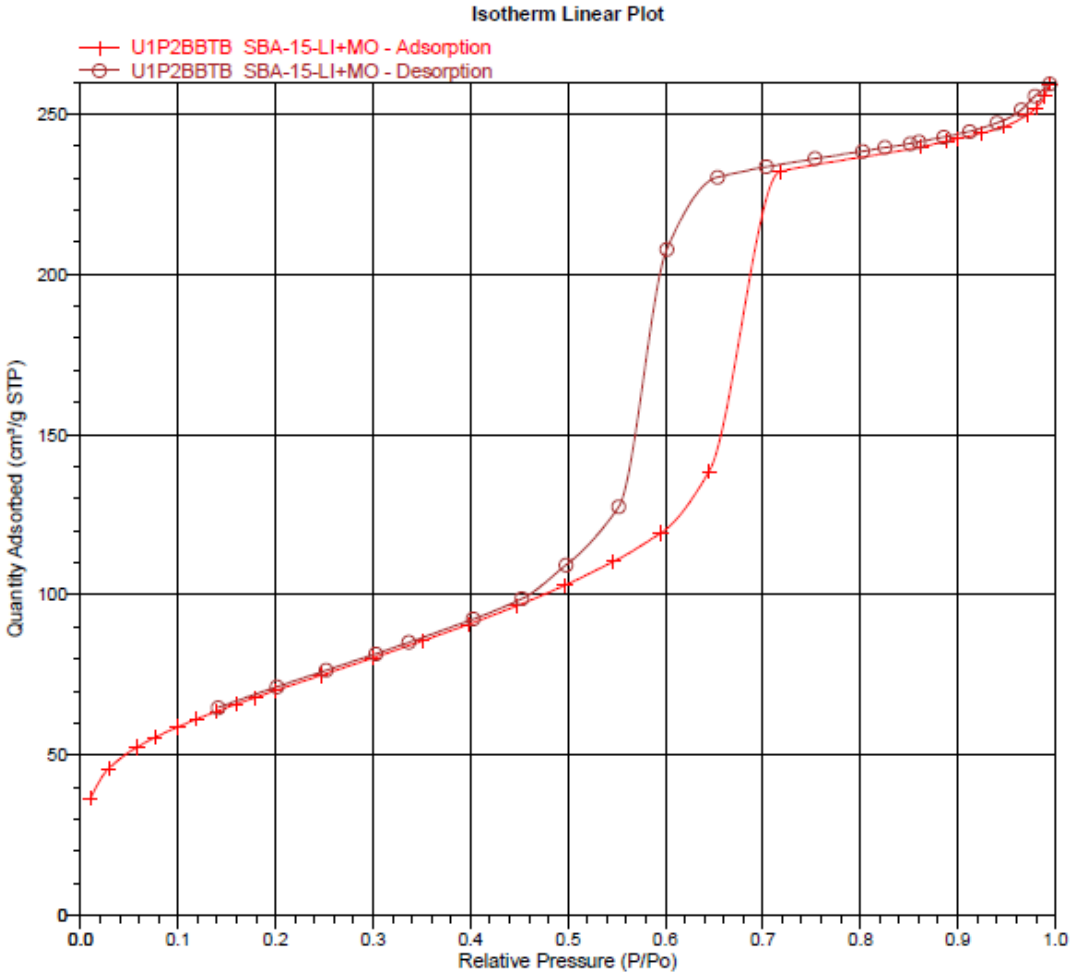
SBA-15



ImCl@SBA-15


Isotherm Linear Plot
U1P1BATA SBA-15-LI - Adsorption
U1P1BATA SBA-15-LI - Desorption
Quantity Adsorbed (cm³/g STP)
Relative Pressure (P/Po)
0
50
100
150
200
250
300
350
400
450
500
0.0
0.1
0.2
0.3
0.4
0.5
0.6
0.7
0.8
0.9
1.0

MoO5@ImCl@SBA-15



Anexo 16. Datos cristalográficos de los compuestos $[MoO(O_2)(Q^{nPe})_2]$, $[MoO(O_2)(Q^{He})_2]$ y $Ph_4P[MoO(O_2)_2(Q^{nPe})_2]$.

Complejo	$[MoO(O_2)(Q^{nPe})_2]$	$[MoO(O_2)(Q^{He})_2]$	$Ph_4P[MoO(O_2)_2(Q^{nPe})_2]$
Fórmula empírica	$C_{32}H_{40}MoN_4O_7$	$C_{34}H_{42}MoN_4O_7$	$C_{40}H_{39}MoN_2O_7P$
Peso Molecular	688.62	714.66	786.64
Sistema cristalográfico	Triclínico	Ortorómbico	Triclínico
Grupo espacial simetría	$P\bar{1}$	*A b a 2*	$P\bar{1}$
a (Å)	9.0124(4)	9.7458(2)	10.5112(6)
b (Å)	13.5773(7)	20.3853(4)	13.1936(7)
c (Å)	13.8352(7)	17.2757(3)	13.8181(8)
α (º)	78.094(2)	90	89.653(2)
β (º)	73.696(2)	90	87.507(2)
γ (º)	82.144(2)	90	71.931(2)
V (Å^3)	1584.30(13)	3432.18(11)	1820.02(18)
Z	2	4	2
ρ (calculado, Mg·m^{-3})	1.444	1.383	1.435
coef abs (mm^{-1})	0.467	0.434	0.457
F(000)	716	1488	812
Tamaño del cristal (mm^3)	0.50 x 0.50 x 0.40	0.40 x 0.30 x 0.20	0.50 x 0.40 x 0.30
Reflexiones recogidas	29597	11966	30917
Reflexiones independientes	5709	2157	6498
Número de parámetros	405	224	464
$R_1(F)$ [$F^2>2\sigma(F^2)$] [a]	0.0254	0.0240	0.0300
$wR_2(F^2)$ [b] (todos los datos incluídos)	0.1039	0.0512	0.0743
S [c] (todos los datos incluídos)	1.055	1.169	1.055

[a] $R_1(F) = \sum(|Fo| - |Fc|)/\sum|Fo|$ para las reflexiones observadas [$F^2>2\sigma(F^2)$]. [b] $wR_2(F^2) = \{\sum [w(F_o^2 - F_c^2)^2]/ \sum w(F_o^2)^2\}^{1/2}$. [c] $S = \{\sum [w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$; (n = número de reflexiones, p = número de parámetros).

Anexo 17. Distancias [Å] y ángulos [°] de enlace del complejo $[MoO(O_2)(Q^{nPe})_2]$.

Mo(1)-O(5)	1.707(2)	C(8)-H(8A)	0.9700
Mo(1)-O(6)	1.830(3)	C(8)-H(8B)	0.9700
Mo(1)-O(7)	1.888(3)	C(8)-H(8C)	0.9700
Mo(1)-O(1)	2.027(2)	C(9)-H(9A)	0.9700
Mo(1)-O(3)	2.028(2)	C(9)-H(9B)	0.9700
Mo(1)-O(2)	2.1207(19)	C(9)-H(9C)	0.9700
Mo(1)-O(4)	2.150(2)	C(10)-H(10A)	0.9700
O(1)-C(3)	1.284(3)	C(10)-H(10B)	0.9700
O(1)-H(1O)	0.9800	C(10)-H(10C)	0.9700
O(2)-C(5)	1.267(3)	C(11)-C(12)	1.370(5)
O(3)-C(19)	1.288(4)	C(11)-C(16)	1.384(4)
O(3)-H(3O)	0.9800	C(12)-C(13)	1.384(5)
O(4)-C(21)	1.274(3)	C(12)-H(12)	0.9400
O(6)-O(7)	1.267(4)	C(13)-C(14)	1.361(5)
N(1)-C(3)	1.343(3)	C(13)-H(13)	0.9400
N(1)-N(2)	1.398(3)	C(14)-C(15)	1.357(6)
N(1)-C(11)	1.429(4)	C(14)-H(14)	0.9400
N(2)-C(1)	1.303(4)	C(15)-C(16)	1.391(5)
N(3)-C(19)	1.335(4)	C(15)-H(15)	0.9400
N(3)-N(4)	1.398(4)	C(16)-H(16)	0.9400
N(3)-C(27)	1.427(4)	C(17)-C(18)	1.441(4)
N(4)-C(17)	1.298(5)	C(17)-C(20)	1.504(5)
C(1)-C(2)	1.437(4)	C(18)-C(21)	1.396(4)
C(1)-C(4)	1.497(4)	C(18)-C(19)	1.413(4)
C(2)-C(5)	1.411(4)	C(20)-H(20A)	0.9700
C(2)-C(3)	1.415(4)	C(20)-H(20B)	0.9700
C(4)-H(4A)	0.9700	C(20)-H(20C)	0.9700
C(4)-H(4B)	0.9700	C(21)-C(22)	1.503(4)
C(4)-H(4C)	0.9700	C(22)-C(23)	1.545(5)
C(5)-C(6)	1.494(4)	C(22)-H(22A)	0.9800
C(6)-C(7)	1.556(4)	C(22)-H(22B)	0.9800
C(6)-H(6A)	0.9800	C(23)-C(24)	1.513(5)
C(6)-H(6B)	0.9800	C(23)-C(25)	1.515(4)
C(7)-C(8)	1.522(4)	C(23)-C(26)	1.533(5)
C(7)-C(10)	1.526(4)	C(24)-H(24A)	0.9700
C(7)-C(9)	1.531(4)	C(24)-H(24B)	0.9700

C(24)-H(24C)	0.9700	O(2)-Mo(1)-O(4)	76.39(8)
C(25)-H(25A)	0.9700	C(3)-O(1)-Mo(1)	128.23(18)
C(25)-H(25B)	0.9700	C(3)-O(1)-H(1O)	104.9
C(25)-H(25C)	0.9700	Mo(1)-O(1)-H(1O)	105.1
C(26)-H(26A)	0.9700	C(5)-O(2)-Mo(1)	135.98(18)
C(26)-H(26B)	0.9700	C(19)-O(3)-Mo(1)	127.67(19)
C(26)-H(26C)	0.9700	C(19)-O(3)-H(3O)	105.0
C(27)-C(32)	1.369(5)	Mo(1)-O(3)-H(3O)	105.2
C(27)-C(28)	1.381(4)	C(21)-O(4)-Mo(1)	134.32(19)
C(28)-C(29)	1.382(5)	O(7)-O(6)-Mo(1)	72.6(2)
C(28)-H(28)	0.9400	O(6)-O(7)-Mo(1)	67.6(2)
C(29)-C(30)	1.364(6)	C(3)-N(1)-N(2)	111.1(2)
C(29)-H(29)	0.9400	C(3)-N(1)-C(11)	130.3(2)
C(30)-C(31)	1.383(6)	N(2)-N(1)-C(11)	118.6(2)
C(30)-H(30)	0.9400	C(1)-N(2)-N(1)	106.3(2)
C(31)-C(32)	1.392(6)	C(19)-N(3)-N(4)	111.5(3)
C(31)-H(31)	0.9400	C(19)-N(3)-C(27)	129.7(3)
C(32)-H(32)	0.9400	N(4)-N(3)-C(27)	118.8(2)
O(5)-Mo(1)-O(6)	104.10(13)	C(17)-N(4)-N(3)	105.6(3)
O(5)-Mo(1)-O(7)	101.20(15)	N(2)-C(1)-C(2)	111.4(3)
O(6)-Mo(1)-O(7)	39.81(13)	N(2)-C(1)-C(4)	118.1(3)
O(5)-Mo(1)-O(1)	98.72(14)	C(2)-C(1)-C(4)	130.5(3)
O(6)-Mo(1)-O(1)	79.27(11)	C(5)-C(2)-C(3)	121.8(2)
O(7)-Mo(1)-O(1)	118.77(14)	C(5)-C(2)-C(1)	134.1(3)
O(5)-Mo(1)-O(3)	90.55(14)	C(3)-C(2)-C(1)	104.0(2)
O(6)-Mo(1)-O(3)	115.33(12)	O(1)-C(3)-N(1)	122.2(3)
O(7)-Mo(1)-O(3)	75.76(14)	O(1)-C(3)-C(2)	130.6(3)
O(1)-Mo(1)-O(3)	160.36(9)	N(1)-C(3)-C(2)	107.2(2)
O(5)-Mo(1)-O(2)	89.18(11)	C(1)-C(4)-H(4A)	109.5
O(6)-Mo(1)-O(2)	159.01(11)	C(1)-C(4)-H(4B)	109.5
O(7)-Mo(1)-O(2)	153.72(13)	H(4A)-C(4)-H(4B)	109.5
O(1)-Mo(1)-O(2)	82.74(8)	C(1)-C(4)-H(4C)	109.5
O(3)-Mo(1)-O(2)	80.11(8)	H(4A)-C(4)-H(4C)	109.5
O(5)-Mo(1)-O(4)	165.07(11)	H(4B)-C(4)-H(4C)	109.5
O(6)-Mo(1)-O(4)	90.82(10)	O(2)-C(5)-C(2)	119.8(2)
O(7)-Mo(1)-O(4)	90.48(13)	O(2)-C(5)-C(6)	115.2(2)
O(1)-Mo(1)-O(4)	83.45(9)	C(2)-C(5)-C(6)	125.0(2)
O(3)-Mo(1)-O(4)	83.28(8)	C(5)-C(6)-C(7)	115.3(2)

C(5)-C(6)-H(6A)	108.5	C(15)-C(14)-C(13)	119.5(3)
C(7)-C(6)-H(6A)	108.5	C(15)-C(14)-H(14)	120.2
C(5)-C(6)-H(6B)	108.5	C(13)-C(14)-H(14)	120.2
C(7)-C(6)-H(6B)	108.5	C(14)-C(15)-C(16)	121.0(3)
H(6A)-C(6)-H(6B)	107.5	C(14)-C(15)-H(15)	119.5
C(8)-C(7)-C(10)	109.5(3)	C(16)-C(15)-H(15)	119.5
C(8)-C(7)-C(9)	109.9(3)	C(11)-C(16)-C(15)	119.0(4)
C(10)-C(7)-C(9)	108.4(3)	C(11)-C(16)-H(16)	120.5
C(8)-C(7)-C(6)	110.8(3)	C(15)-C(16)-H(16)	120.5
C(10)-C(7)-C(6)	111.6(2)	N(4)-C(17)-C(18)	112.1(3)
C(9)-C(7)-C(6)	106.6(3)	N(4)-C(17)-C(20)	118.0(3)
C(7)-C(8)-H(8A)	109.5	C(18)-C(17)-C(20)	129.9(3)
C(7)-C(8)-H(8B)	109.5	C(21)-C(18)-C(19)	122.8(3)
H(8A)-C(8)-H(8B)	109.5	C(21)-C(18)-C(17)	133.7(3)
C(7)-C(8)-H(8C)	109.5	C(19)-C(18)-C(17)	103.2(3)
H(8A)-C(8)-H(8C)	109.5	O(3)-C(19)-N(3)	121.5(3)
H(8B)-C(8)-H(8C)	109.5	O(3)-C(19)-C(18)	131.0(3)
C(7)-C(9)-H(9A)	109.5	N(3)-C(19)-C(18)	107.6(3)
C(7)-C(9)-H(9B)	109.5	C(17)-C(20)-H(20A)	109.5
H(9A)-C(9)-H(9B)	109.5	C(17)-C(20)-H(20B)	109.5
C(7)-C(9)-H(9C)	109.5	H(20A)-C(20)-H(20B)	109.5
H(9A)-C(9)-H(9C)	109.5	C(17)-C(20)-H(20C)	109.5
H(9B)-C(9)-H(9C)	109.5	H(20A)-C(20)-H(20C)	109.5
C(7)-C(10)-H(10A)	109.5	H(20B)-C(20)-H(20C)	109.5
C(7)-C(10)-H(10B)	109.5	O(4)-C(21)-C(18)	120.0(3)
H(10A)-C(10)-H(10B)	109.5	O(4)-C(21)-C(22)	117.0(3)
C(7)-C(10)-H(10C)	109.5	C(18)-C(21)-C(22)	123.0(3)
H(10A)-C(10)-H(10C)	109.5	C(21)-C(22)-C(23)	115.2(3)
H(10B)-C(10)-H(10C)	109.5	C(21)-C(22)-H(22A)	108.5
C(12)-C(11)-C(16)	120.0(3)	C(23)-C(22)-H(22A)	108.5
C(12)-C(11)-N(1)	121.8(3)	C(21)-C(22)-H(22B)	108.5
C(16)-C(11)-N(1)	118.1(3)	C(23)-C(22)-H(22B)	108.5
C(11)-C(12)-C(13)	119.5(3)	H(22A)-C(22)-H(22B)	107.5
C(11)-C(12)-H(12)	120.2	C(24)-C(23)-C(25)	110.4(3)
C(13)-C(12)-H(12)	120.2	C(24)-C(23)-C(26)	108.8(3)
C(14)-C(13)-C(12)	121.0(4)	C(25)-C(23)-C(26)	109.2(3)
C(14)-C(13)-H(13)	119.5		
C(12)-C(13)-H(13)	119.5		

C(24)-C(23)-C(22)	111.0(3)	H(26B)-C(26)-H(26C)	109.5
C(25)-C(23)-C(22)	109.9(3)	C(32)-C(27)-C(28)	121.1(3)
C(26)-C(23)-C(22)	107.5(3)	C(32)-C(27)-N(3)	120.7(3)
C(23)-C(24)-H(24A)	109.5	C(28)-C(27)-N(3)	118.1(3)
C(23)-C(24)-H(24B)	109.5	C(27)-C(28)-C(29)	119.0(4)
H(24A)-C(24)-H(24B)	109.5	C(27)-C(28)-H(28)	120.5
C(23)-C(24)-H(24C)	109.5	C(29)-C(28)-H(28)	120.5
H(24A)-C(24)-H(24C)	109.5	C(30)-C(29)-C(28)	120.9(3)
H(24B)-C(24)-H(24C)	109.5	C(30)-C(29)-H(29)	119.6
C(23)-C(25)-H(25A)	109.5	C(28)-C(29)-H(29)	119.6
C(23)-C(25)-H(25B)	109.5	C(29)-C(30)-C(31)	119.7(4)
H(25A)-C(25)-H(25B)	109.5	C(29)-C(30)-H(30)	120.2
C(23)-C(25)-H(25C)	109.5	C(31)-C(30)-H(30)	120.2
H(25A)-C(25)-H(25C)	109.5	C(30)-C(31)-C(32)	120.2(4)
H(25B)-C(25)-H(25C)	109.5	C(30)-C(31)-H(31)	119.9
C(23)-C(26)-H(26A)	109.5	C(32)-C(31)-H(31)	119.9
C(23)-C(26)-H(26B)	109.5	C(27)-C(32)-C(31)	119.1(4)
H(26A)-C(26)-H(26B)	109.5	C(27)-C(32)-H(32)	120.5
C(23)-C(26)-H(26C)	109.5	C(31)-C(32)-H(32)	120.5
H(26A)-C(26)-H(26C)	109.5		

Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1

Anexo 18. Distancias [Å] y ángulos [°] de enlace del complejo $[MoO(O_2)(Q^{He})_2]$.

Mo(1)-O(3)#1	1.687(8)	C(9)-H(9B)	0.9900
Mo(1)-O(3)	1.687(8)	C(10)-H(10A)	0.9800
Mo(1)-O(5)#1	1.885(8)	C(10)-H(10B)	0.9800
Mo(1)-O(5)	1.885(8)	C(10)-H(10C)	0.9800
Mo(1)-O(4)#1	1.915(5)	C(11)-H(11A)	0.9800
Mo(1)-O(4)	1.915(5)	C(11)-H(11B)	0.9800
Mo(1)-O(1)	2.0380(19)	C(11)-H(11C)	0.9800
Mo(1)-O(1)#1	2.0380(19)	C(12)-C(13)	1.386(5)
Mo(1)-O(2)	2.138(2)	C(12)-C(17)	1.396(4)
Mo(1)-O(2)#1	2.138(2)	C(13)-C(14)	1.393(5)
O(1)-C(1)	1.299(4)	C(13)-H(13)	0.9500
O(2)-C(4)	1.275(4)	C(14)-C(15)	1.370(5)
O(4)-O(5)	1.424(6)	C(14)-H(14)	0.9500
N(1)-C(1)	1.350(4)	C(15)-C(16)	1.365(5)
N(1)-N(2)	1.399(3)	C(15)-H(15)	0.9500
N(1)-C(12)	1.424(4)	C(16)-C(17)	1.382(4)
N(2)-C(3)	1.306(4)	C(16)-H(16)	0.9500
C(1)-C(2)	1.406(4)	C(17)-H(17)	0.9500
C(2)-C(4)	1.402(4)	O(3)#1-Mo(1)-O(3)	98.8(6)
C(2)-C(3)	1.442(4)	O(3)#1-Mo(1)-O(5)#1	15.9(3)
C(3)-C(11)	1.497(4)	O(3)-Mo(1)-O(5)#1	103.2(2)
C(4)-C(5)	1.500(4)	O(3)#1-Mo(1)-O(5)	103.2(2)
C(5)-C(6)	1.524(4)	O(3)-Mo(1)-O(5)	15.9(3)
C(5)-H(5A)	0.9900	O(5)#1-Mo(1)-O(5)	111.4(6)
C(5)-H(5B)	0.9900	O(3)#1-Mo(1)-O(4)#1	29.3(2)
C(6)-C(7)	1.517(4)	O(3)-Mo(1)-O(4)#1	102.4(3)
C(6)-H(6A)	0.9900	O(5)#1-Mo(1)-O(4)#1	44.0(2)
C(6)-H(6B)	0.9900	O(5)-Mo(1)-O(4)#1	98.6(3)
C(7)-C(8)	1.528(4)	O(3)#1-Mo(1)-O(4)	102.4(3)
C(7)-H(7A)	0.9900	O(3)-Mo(1)-O(4)	29.3(2)
C(7)-H(7B)	0.9900	O(5)#1-Mo(1)-O(4)	98.6(3)
C(8)-C(9)	1.511(5)	O(5)-Mo(1)-O(4)	44.0(2)
C(8)-H(8A)	0.9900	O(4)#1-Mo(1)-O(4)	119.3(3)
C(8)-H(8B)	0.9900	O(3)#1-Mo(1)-O(1)	92.2(2)
C(9)-C(10)	1.517(5)	O(3)-Mo(1)-O(1)	103.4(2)
C(9)-H(9A)	0.9900	O(5)#1-Mo(1)-O(1)	76.3(2)

O(5)-Mo(1)-O(1)	118.1(2)	C(4)-C(2)-C(1)	122.5(3)
O(4)#1-Mo(1)-O(1)	118.76(16)	C(4)-C(2)-C(3)	133.0(3)
O(4)-Mo(1)-O(1)	74.28(17)	C(1)-C(2)-C(3)	104.4(3)
O(3)#1-Mo(1)-O(1)#1	103.4(2)	N(2)-C(3)-C(2)	111.0(3)
O(3)-Mo(1)-O(1)#1	92.2(2)	N(2)-C(3)-C(11)	119.3(3)
O(5)#1-Mo(1)-O(1)#1	118.1(2)	C(2)-C(3)-C(11)	129.6(3)
O(5)-Mo(1)-O(1)#1	76.3(2)	O(2)-C(4)-C(2)	120.0(3)
O(4)#1-Mo(1)-O(1)#1	74.28(17)	O(2)-C(4)-C(5)	116.1(3)
O(4)-Mo(1)-O(1)#1	118.76(16)	C(2)-C(4)-C(5)	123.9(3)
O(1)-Mo(1)-O(1)#1	155.99(13)	C(4)-C(5)-C(6)	113.7(3)
O(3)#1-Mo(1)-O(2)	170.5(3)	C(4)-C(5)-H(5A)	108.8
O(3)-Mo(1)-O(2)	90.4(3)	C(6)-C(5)-H(5A)	108.8
O(5)#1-Mo(1)-O(2)	157.6(2)	C(4)-C(5)-H(5B)	108.8
O(5)-Mo(1)-O(2)	86.3(3)	C(6)-C(5)-H(5B)	108.8
O(4)#1-Mo(1)-O(2)	150.19(18)	H(5A)-C(5)-H(5B)	107.7
O(4)-Mo(1)-O(2)	84.45(17)	C(7)-C(6)-C(5)	111.7(3)
O(1)-Mo(1)-O(2)	83.28(8)	C(7)-C(6)-H(6A)	109.3
O(1)#1-Mo(1)-O(2)	78.45(9)	C(5)-C(6)-H(6A)	109.3
O(3)#1-Mo(1)-O(2)#1	90.4(3)	C(7)-C(6)-H(6B)	109.3
O(3)-Mo(1)-O(2)#1	170.5(3)	C(5)-C(6)-H(6B)	109.3
O(5)#1-Mo(1)-O(2)#1	86.3(3)	H(6A)-C(6)-H(6B)	107.9
O(5)-Mo(1)-O(2)#1	157.6(2)	C(6)-C(7)-C(8)	114.6(3)
O(4)#1-Mo(1)-O(2)#1	84.45(17)	C(6)-C(7)-H(7A)	108.6
O(4)-Mo(1)-O(2)#1	150.19(18)	C(8)-C(7)-H(7A)	108.6
O(1)-Mo(1)-O(2)#1	78.45(9)	C(6)-C(7)-H(7B)	108.6
O(1)#1-Mo(1)-O(2)#1	83.28(8)	C(8)-C(7)-H(7B)	108.6
O(2)-Mo(1)-O(2)#1	80.60(14)	H(7A)-C(7)-H(7B)	107.6
C(1)-O(1)-Mo(1)	126.99(18)	C(9)-C(8)-C(7)	112.7(3)
C(4)-O(2)-Mo(1)	134.8(2)	C(9)-C(8)-H(8A)	109.1
O(5)-O(4)-Mo(1)	66.9(4)	C(7)-C(8)-H(8A)	109.1
O(4)-O(5)-Mo(1)	69.1(4)	C(9)-C(8)-H(8B)	109.1
C(1)-N(1)-N(2)	111.0(2)	C(7)-C(8)-H(8B)	109.1
C(1)-N(1)-C(12)	128.5(3)	H(8A)-C(8)-H(8B)	107.8
N(2)-N(1)-C(12)	120.5(2)	C(8)-C(9)-C(10)	113.7(3)
C(3)-N(2)-N(1)	106.3(2)	C(8)-C(9)-H(9A)	108.8
O(1)-C(1)-N(1)	121.4(3)	C(10)-C(9)-H(9A)	108.8
O(1)-C(1)-C(2)	131.4(3)		
N(1)-C(1)-C(2)	107.1(3)		

C(8)-C(9)-H(9B)	108.8	C(17)-C(12)-N(1)	118.4(3)
C(10)-C(9)-H(9B)	108.8	C(12)-C(13)-C(14)	118.2(4)
H(9A)-C(9)-H(9B)	107.7	C(12)-C(13)-H(13)	120.9
C(9)-C(10)-H(10A)	109.5	C(14)-C(13)-H(13)	120.9
C(9)-C(10)-H(10B)	109.5	C(15)-C(14)-C(13)	121.2(4)
H(10A)-C(10)-H(10B)	109.5	C(15)-C(14)-H(14)	119.4
C(9)-C(10)-H(10C)	109.5	C(13)-C(14)-H(14)	119.4
H(10A)-C(10)-H(10C)	109.5	C(16)-C(15)-C(14)	119.6(4)
H(10B)-C(10)-H(10C)	109.5	C(16)-C(15)-H(15)	120.2
C(3)-C(11)-H(11A)	109.5	C(14)-C(15)-H(15)	120.2
C(3)-C(11)-H(11B)	109.5	C(15)-C(16)-C(17)	121.6(4)
H(11A)-C(11)-H(11B)	109.5	C(15)-C(16)-H(16)	119.2
C(3)-C(11)-H(11C)	109.5	C(17)-C(16)-H(16)	119.2
H(11A)-C(11)-H(11C)	109.5	C(16)-C(17)-C(12)	118.2(4)
H(11B)-C(11)-H(11C)	109.5	C(16)-C(17)-H(17)	120.9
C(13)-C(12)-C(17)	121.1(3)	C(12)-C(17)-H(17)	120.9
C(13)-C(12)-N(1)	120.5(3)		

Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1

Anexo 19. Distancias [Å] y ángulos [°] de enlace del complejo $Ph_4P[MoO(O_2)_2(Q^{nPe})]$.

C(10)-C(3)	1.498(2)	C(9)-H(9B)	0.9800
C(10)-H(10A)	0.9800	C(9)-H(9C)	0.9800
C(10)-H(10B)	0.9800	C(11)-C(16)	1.390(3)
C(10)-H(10C)	0.9800	C(11)-C(12)	1.392(3)
Mo(1)-O(3)	1.6856(13)	C(12)-C(13)	1.381(3)
Mo(1)-O(5)	1.9181(15)	C(12)-H(12)	0.9500
Mo(1)-O(6)	1.9214(13)	C(13)-C(14)	1.384(3)
Mo(1)-O(7)	1.9506(14)	C(13)-H(13)	0.9500
Mo(1)-O(4)	1.9517(15)	C(14)-C(15)	1.383(3)
Mo(1)-O(1)	2.0806(12)	C(14)-H(14)	0.9500
Mo(1)-O(2)	2.2783(13)	C(15)-C(16)	1.380(3)
O(1)-C(1)	1.296(2)	C(15)-H(15)	0.9500
O(2)-C(4)	1.251(2)	C(16)-H(16)	0.9500
O(4)-O(5)	1.474(2)	P(1)-C(23)	1.7945(18)
O(6)-O(7)	1.4707(19)	P(1)-C(29)	1.7961(18)
N(1)-C(3)	1.314(2)	P(1)-C(17)	1.7972(18)
N(1)-N(2)	1.398(2)	P(1)-C(35)	1.8002(18)
N(2)-C(1)	1.359(2)	C(17)-C(22)	1.395(3)
N(2)-C(11)	1.421(2)	C(17)-C(18)	1.399(3)
C(1)-C(2)	1.407(2)	C(18)-C(19)	1.390(3)
C(2)-C(4)	1.430(3)	C(18)-H(18)	0.9500
C(2)-C(3)	1.436(2)	C(19)-C(20)	1.384(3)
C(4)-C(5)	1.503(2)	C(19)-H(19)	0.9500
C(5)-C(6)	1.555(3)	C(20)-C(21)	1.383(3)
C(5)-H(5A)	0.9900	C(20)-H(20)	0.9500
C(5)-H(5B)	0.9900	C(21)-C(22)	1.382(3)
C(6)-C(7)	1.524(3)	C(21)-H(21)	0.9500
C(6)-C(9)	1.530(3)	C(22)-H(22)	0.9500
C(6)-C(8)	1.534(3)	C(23)-C(28)	1.391(3)
C(7)-H(7A)	0.9800	C(23)-C(24)	1.396(3)
C(7)-H(7B)	0.9800	C(24)-C(25)	1.381(3)
C(7)-H(7C)	0.9800	C(24)-H(24)	0.9500
C(8)-H(8A)	0.9800	C(25)-C(26)	1.380(3)
C(8)-H(8B)	0.9800	C(25)-H(25)	0.9500
C(8)-H(8C)	0.9800	C(26)-C(27)	1.383(3)
C(9)-H(9A)	0.9800	C(26)-H(26)	0.9500

C(27)-C(28)	1.385(3)	O(5)-Mo(1)-O(4)	44.78(7)
C(27)-H(27)	0.9500	O(6)-Mo(1)-O(4)	130.77(6)
C(28)-H(28)	0.9500	O(7)-Mo(1)-O(4)	158.88(7)
C(29)-C(34)	1.395(3)	O(3)-Mo(1)-O(1)	88.99(6)
C(29)-C(30)	1.395(3)	O(5)-Mo(1)-O(1)	132.98(6)
C(30)-C(31)	1.387(3)	O(6)-Mo(1)-O(1)	134.37(6)
C(30)-H(30)	0.9500	O(7)-Mo(1)-O(1)	90.10(6)
C(31)-C(32)	1.382(3)	O(4)-Mo(1)-O(1)	88.43(6)
C(31)-H(31)	0.9500	O(3)-Mo(1)-O(2)	169.37(6)
C(32)-C(33)	1.379(3)	O(5)-Mo(1)-O(2)	85.70(6)
C(32)-H(32)	0.9500	O(6)-Mo(1)-O(2)	84.49(5)
C(33)-C(34)	1.390(3)	O(7)-Mo(1)-O(2)	79.09(6)
C(33)-H(33)	0.9500	O(4)-Mo(1)-O(2)	79.88(6)
C(34)-H(34)	0.9500	O(1)-Mo(1)-O(2)	80.46(5)
C(35)-C(40)	1.393(3)	C(1)-O(1)-Mo(1)	128.10(11)
C(35)-C(36)	1.396(3)	C(4)-O(2)-Mo(1)	133.12(12)
C(36)-C(37)	1.383(3)	O(5)-O(4)-Mo(1)	66.40(8)
C(36)-H(36)	0.9500	O(4)-O(5)-Mo(1)	68.82(8)
C(37)-C(38)	1.384(3)	O(7)-O(6)-Mo(1)	68.73(8)
C(37)-H(37)	0.9500	O(6)-O(7)-Mo(1)	66.63(8)
C(38)-C(39)	1.382(3)	C(3)-N(1)-N(2)	105.49(14)
C(38)-H(38)	0.9500	C(1)-N(2)-N(1)	111.58(14)
C(39)-C(40)	1.390(3)	C(1)-N(2)-C(11)	129.75(14)
C(39)-H(39)	0.9500	N(1)-N(2)-C(11)	118.53(14)
C(40)-H(40)	0.9500	O(1)-C(1)-N(2)	121.38(16)
C(3)-C(10)-H(10A)	109.5	O(1)-C(1)-C(2)	132.01(16)
C(3)-C(10)-H(10B)	109.5	N(2)-C(1)-C(2)	106.61(15)
H(10A)-C(10)-H(10B)	109.5	C(1)-C(2)-C(4)	121.90(16)
C(3)-C(10)-H(10C)	109.5	C(1)-C(2)-C(3)	104.63(15)
H(10A)-C(10)-H(10C)	109.5	C(4)-C(2)-C(3)	133.46(16)
H(10B)-C(10)-H(10C)	109.5	N(1)-C(3)-C(2)	111.68(15)
O(3)-Mo(1)-O(5)	102.64(7)	N(1)-C(3)-C(10)	116.94(16)
O(3)-Mo(1)-O(6)	102.21(6)	C(2)-C(3)-C(10)	131.37(17)
O(5)-Mo(1)-O(6)	87.93(6)	O(2)-C(4)-C(2)	121.25(16)
O(3)-Mo(1)-O(7)	99.77(7)	O(2)-C(4)-C(5)	117.71(16)
O(5)-Mo(1)-O(7)	130.95(6)	C(2)-C(4)-C(5)	121.04(15)
O(6)-Mo(1)-O(7)	44.64(6)	C(4)-C(5)-C(6)	114.22(15)
O(3)-Mo(1)-O(4)	101.27(7)	C(4)-C(5)-H(5A)	108.7

C(6)-C(5)-H(5A)	108.7	C(15)-C(14)-C(13)	120.00(19)
C(4)-C(5)-H(5B)	108.7	C(15)-C(14)-H(14)	120.0
C(6)-C(5)-H(5B)	108.7	C(13)-C(14)-H(14)	120.0
H(5A)-C(5)-H(5B)	107.6	C(16)-C(15)-C(14)	120.23(19)
C(7)-C(6)-C(9)	108.94(18)	C(16)-C(15)-H(15)	119.9
C(7)-C(6)-C(8)	109.37(18)	C(14)-C(15)-H(15)	119.9
C(9)-C(6)-C(8)	109.89(17)	C(15)-C(16)-C(11)	119.48(18)
C(7)-C(6)-C(5)	110.74(16)	C(15)-C(16)-H(16)	120.3
C(9)-C(6)-C(5)	106.38(17)	C(11)-C(16)-H(16)	120.3
C(8)-C(6)-C(5)	111.45(16)	C(23)-P(1)-C(29)	110.66(8)
C(6)-C(7)-H(7A)	109.5	C(23)-P(1)-C(17)	109.43(8)
C(6)-C(7)-H(7B)	109.5	C(29)-P(1)-C(17)	109.30(8)
H(7A)-C(7)-H(7B)	109.5	C(23)-P(1)-C(35)	108.02(9)
C(6)-C(7)-H(7C)	109.5	C(29)-P(1)-C(35)	108.32(8)
H(7A)-C(7)-H(7C)	109.5	C(17)-P(1)-C(35)	111.12(8)
H(7B)-C(7)-H(7C)	109.5	C(22)-C(17)-C(18)	119.93(17)
C(6)-C(8)-H(8A)	109.5	C(22)-C(17)-P(1)	120.71(15)
C(6)-C(8)-H(8B)	109.5	C(18)-C(17)-P(1)	119.34(13)
H(8A)-C(8)-H(8B)	109.5	C(19)-C(18)-C(17)	119.69(18)
C(6)-C(8)-H(8C)	109.5	C(19)-C(18)-H(18)	120.2
H(8A)-C(8)-H(8C)	109.5	C(17)-C(18)-H(18)	120.2
H(8B)-C(8)-H(8C)	109.5	C(20)-C(19)-C(18)	120.05(19)
C(6)-C(9)-H(9A)	109.5	C(20)-C(19)-H(19)	120.0
C(6)-C(9)-H(9B)	109.5	C(18)-C(19)-H(19)	120.0
H(9A)-C(9)-H(9B)	109.5	C(19)-C(20)-C(21)	120.04(19)
C(6)-C(9)-H(9C)	109.5	C(19)-C(20)-H(20)	120.0
H(9A)-C(9)-H(9C)	109.5	C(21)-C(20)-H(20)	120.0
H(9B)-C(9)-H(9C)	109.5	C(22)-C(21)-C(20)	120.82(19)
C(16)-C(11)-C(12)	120.67(17)	C(22)-C(21)-H(21)	119.6
C(16)-C(11)-N(2)	119.08(16)	C(20)-C(21)-H(21)	119.6
C(12)-C(11)-N(2)	120.25(17)	C(21)-C(22)-C(17)	119.44(19)
C(13)-C(12)-C(11)	118.97(19)	C(21)-C(22)-H(22)	120.3
C(13)-C(12)-H(12)	120.5	C(17)-C(22)-H(22)	120.3
C(11)-C(12)-H(12)	120.5	C(28)-C(23)-C(24)	120.01(17)
C(12)-C(13)-C(14)	120.6(2)	C(28)-C(23)-P(1)	121.57(14)
C(12)-C(13)-H(13)	119.7	C(24)-C(23)-P(1)	118.36(14)
C(14)-C(13)-H(13)	119.7	C(25)-C(24)-C(23)	120.19(19)
C(25)-C(24)-H(24)	119.9	C(23)-C(24)-H(24)	119.9

C(26)-C(25)-C(24)	119.57(19)	C(32)-C(33)-C(34)	120.08(19)
C(26)-C(25)-H(25)	120.2	C(32)-C(33)-H(33)	120.0
C(24)-C(25)-H(25)	120.2	C(34)-C(33)-H(33)	120.0
C(25)-C(26)-C(27)	120.61(19)	C(33)-C(34)-C(29)	119.46(19)
C(25)-C(26)-H(26)	119.7	C(33)-C(34)-H(34)	120.3
C(27)-C(26)-H(26)	119.7	C(29)-C(34)-H(34)	120.3
C(26)-C(27)-C(28)	120.4(2)	C(40)-C(35)-C(36)	120.22(17)
C(26)-C(27)-H(27)	119.8	C(40)-C(35)-P(1)	120.90(14)
C(28)-C(27)-H(27)	119.8	C(36)-C(35)-P(1)	118.68(14)
C(27)-C(28)-C(23)	119.22(19)	C(37)-C(36)-C(35)	119.76(18)
C(27)-C(28)-H(28)	120.4	C(37)-C(36)-H(36)	120.1
C(23)-C(28)-H(28)	120.4	C(35)-C(36)-H(36)	120.1
C(34)-C(29)-C(30)	120.30(17)	C(36)-C(37)-C(38)	119.99(19)
C(34)-C(29)-P(1)	120.45(14)	C(36)-C(37)-H(37)	120.0
C(30)-C(29)-P(1)	119.26(14)	C(38)-C(37)-H(37)	120.0
C(31)-C(30)-C(29)	119.36(18)	C(39)-C(38)-C(37)	120.48(19)
C(31)-C(30)-H(30)	120.3	C(39)-C(38)-H(38)	119.8
C(29)-C(30)-H(30)	120.3	C(37)-C(38)-H(38)	119.8
C(32)-C(31)-C(30)	120.3(2)	C(38)-C(39)-C(40)	120.20(19)
C(32)-C(31)-H(31)	119.9	C(38)-C(39)-H(39)	119.9
C(30)-C(31)-H(31)	119.9	C(40)-C(39)-H(39)	119.9
C(33)-C(32)-C(31)	120.52(19)	C(39)-C(40)-C(35)	119.34(18)
C(33)-C(32)-H(32)	119.7	C(39)-C(40)-H(40)	120.3
C(31)-C(32)-H(32)	119.7	C(35)-C(40)-H(40)	120.3

Transformaciones de simetría usadas para generar átomos equivalentes: #1 -x+1,-y+2,-z #2 -x+2,-y+1,-z+1.