ADDITIONAL MATERIAL

Unexpected formation of bis(hydrazinecarboximidamide) via ultrasound promoted rearrangement of epoxy ketones

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IR spectra of compounds 2a-e

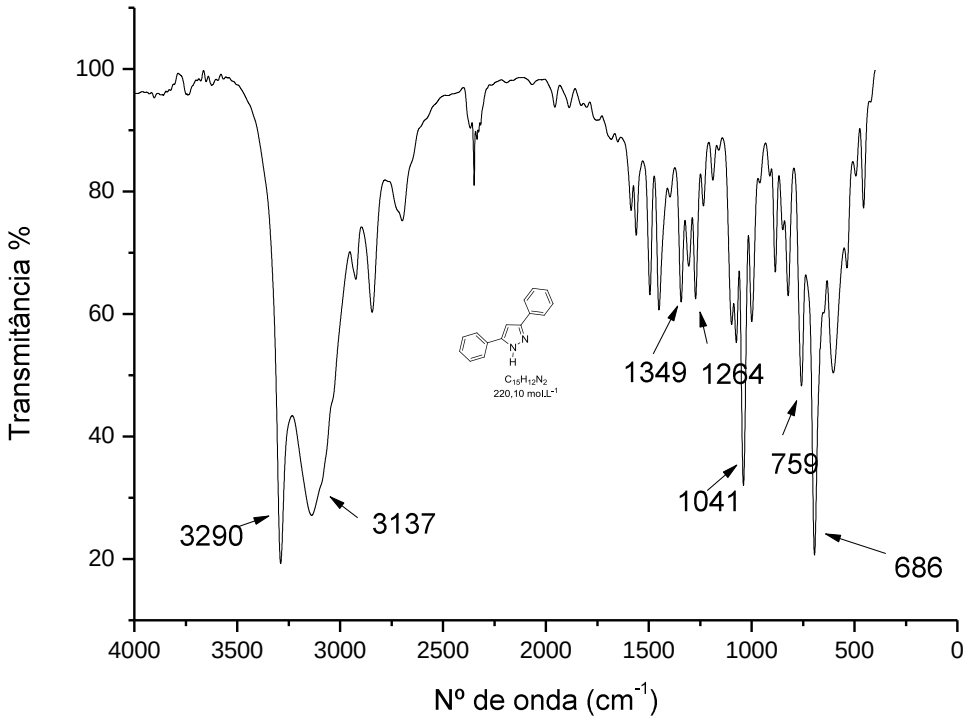

Figure S1: IR spectrum 3,5-diphenyl-*1H*-pyrazole (**2a**).

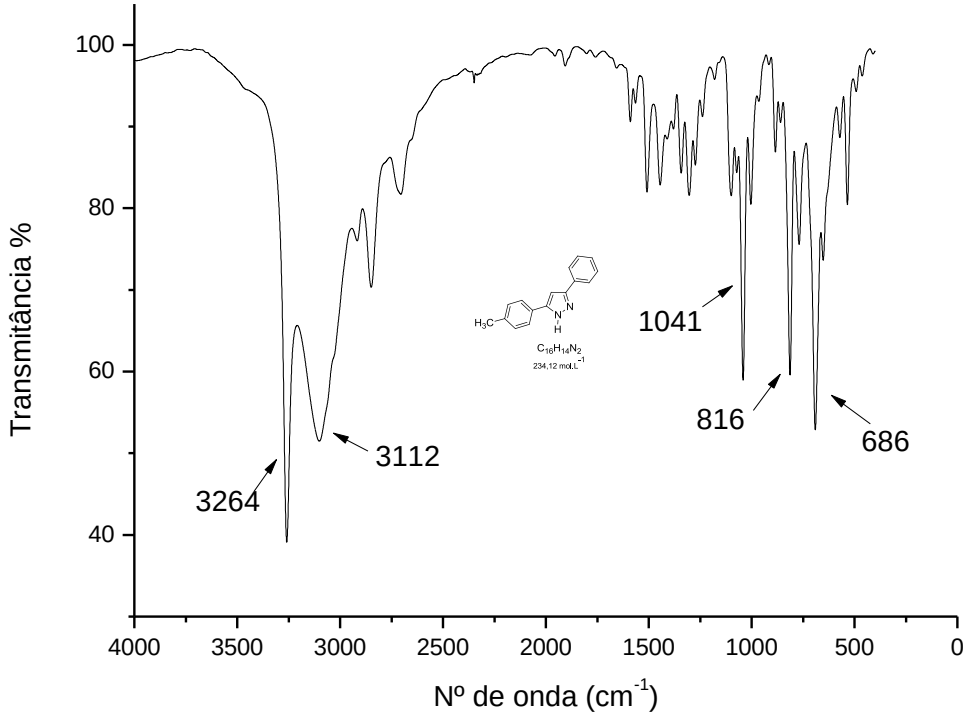

Figure S2: IR spectrum of 3-phenyl-5-(4-tolyl)-*1H*-pyrazole (**2b**).

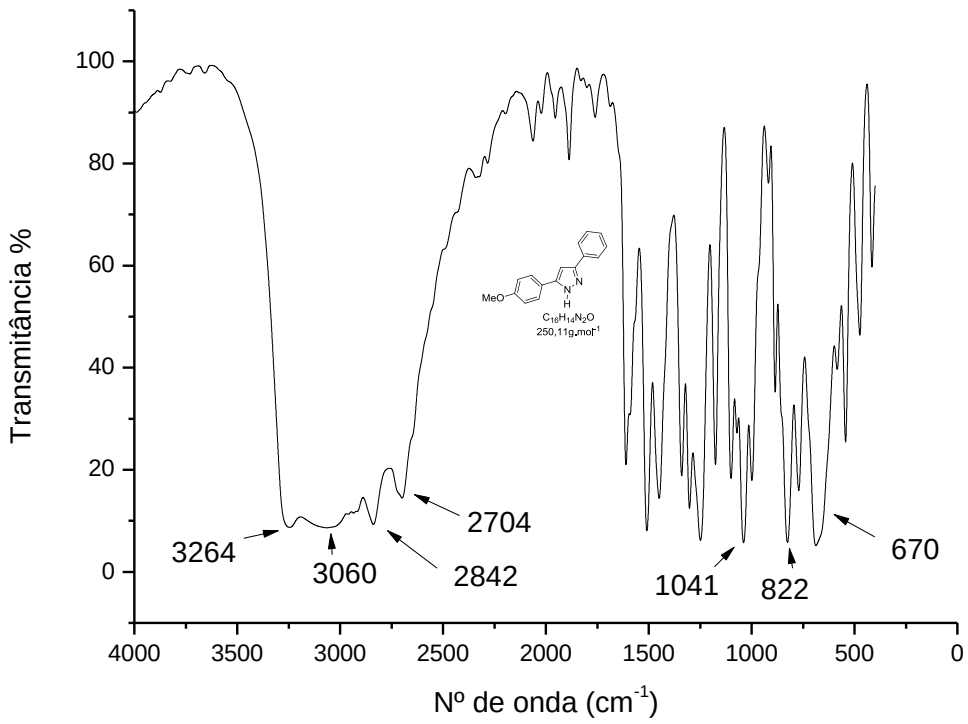

Figure S3: IR spectrum of 3-phenyl-5-(4-methoxyphenyl)-*1H*-pyrazole (**2c**).

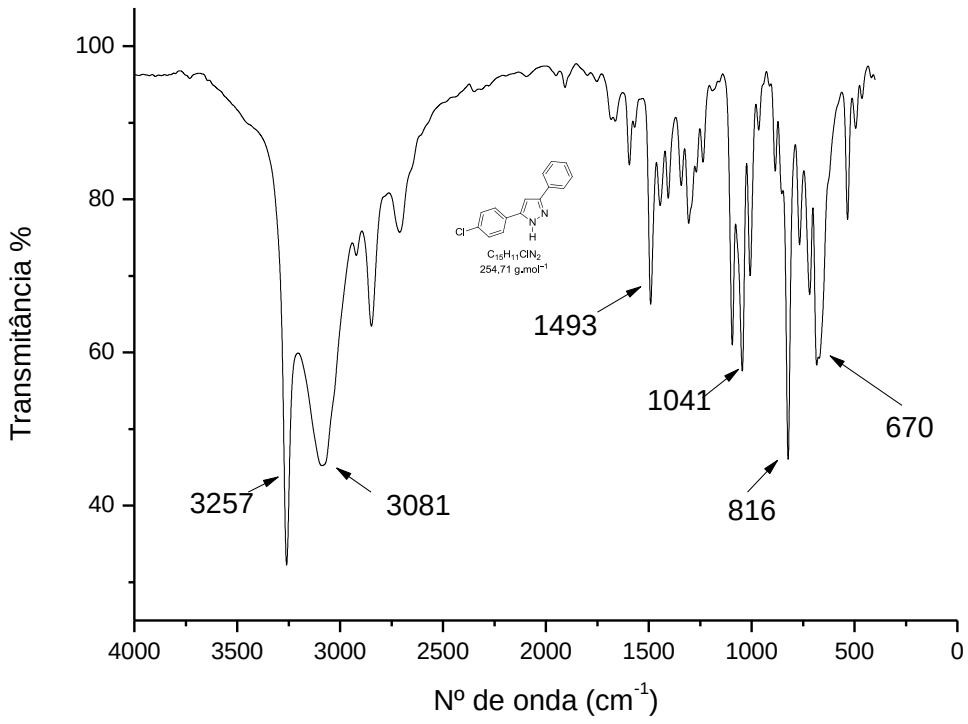

Figure S4: IR spectra of 3-phenyl-5-(4-chlorophenyl)-*1H*-pyrazole (**2d**).

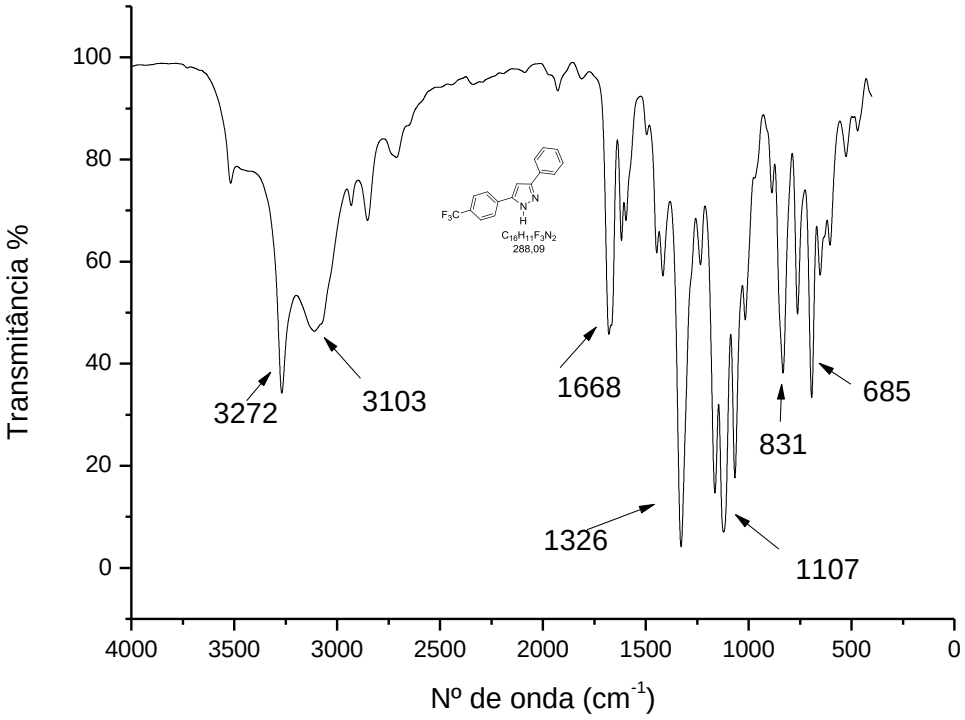

Figure S5: IR spectrum of 3-phenyl-5-(4-trifluorophenyl)-*1H*-pyrazole (**2e**).

Low-resolution mass spectra of compounds 2a-f

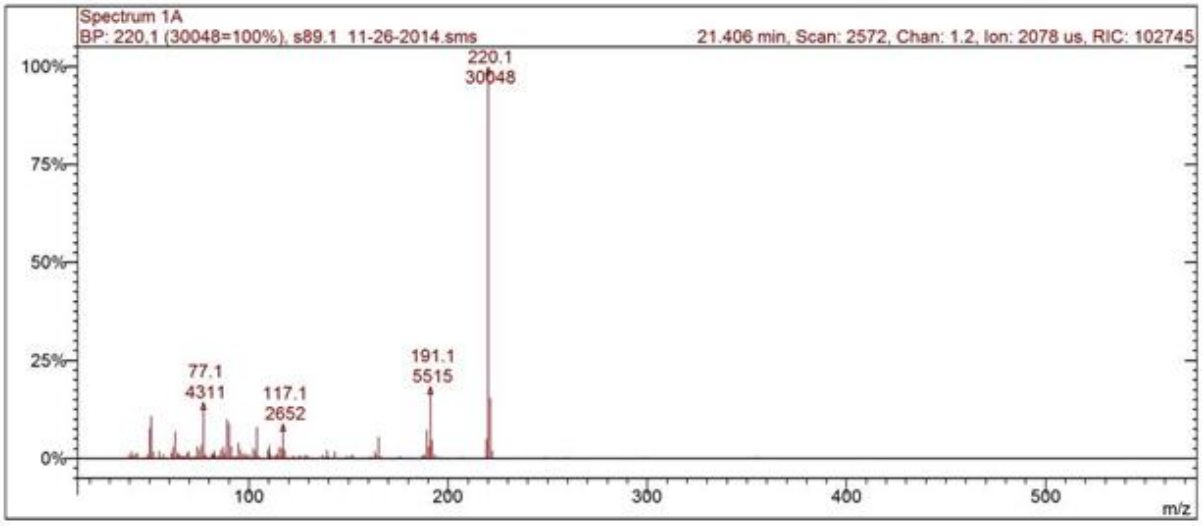

Figure S6: LRMS of 3,5-diphenyl-*1H*-pyrazole (**2a**).

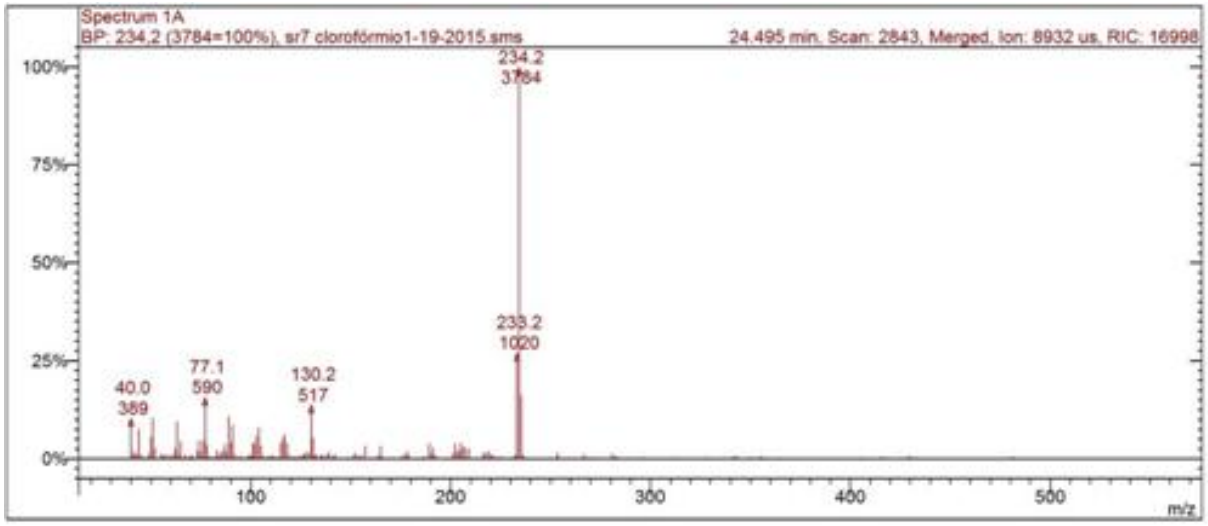

Figure S7: LRMS of 3-phenyl-5-(4-tolyl)-*1H*-pyrazole (**2b**).

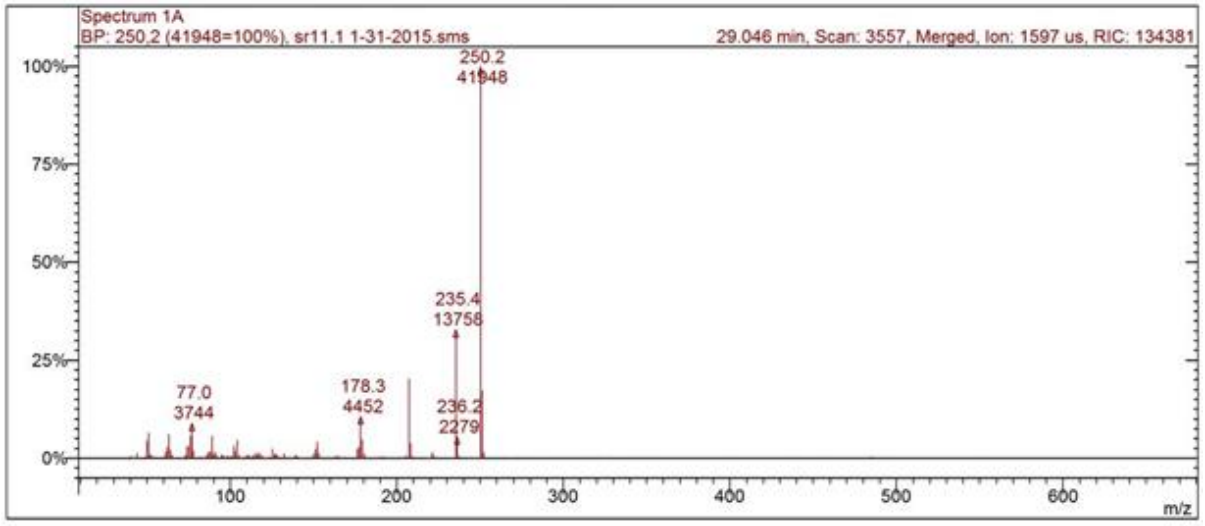

Figure S8: LRMS of 3-phenyl-5-(4-methoxyphenyl)-*1H*-pyrazole (**2c**).

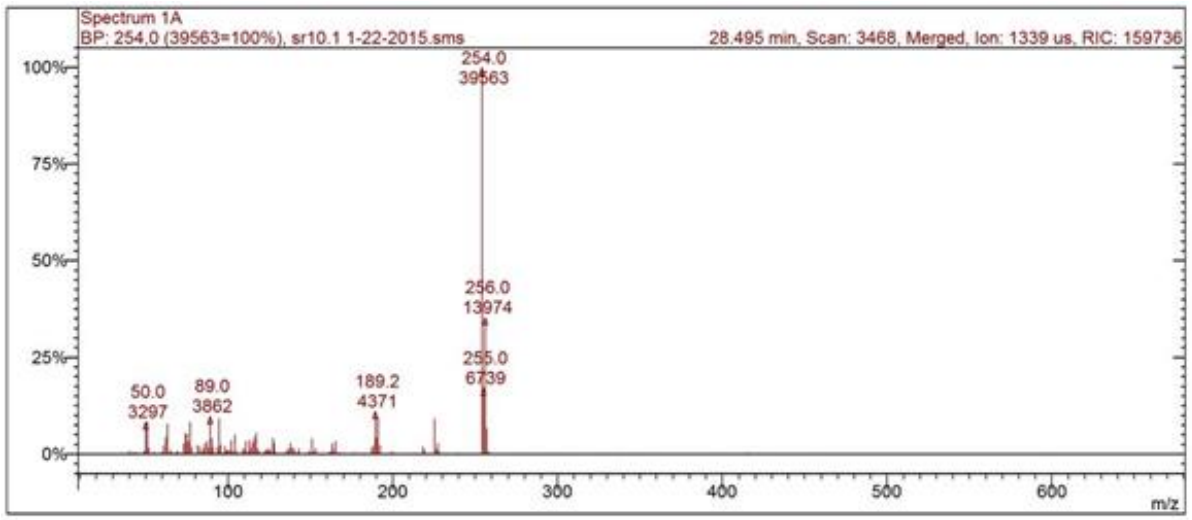

Figure S9: LRMS of 3-phenyl-5-(4-chlorophenyl)-*1H*-pyrazole (**2d**).

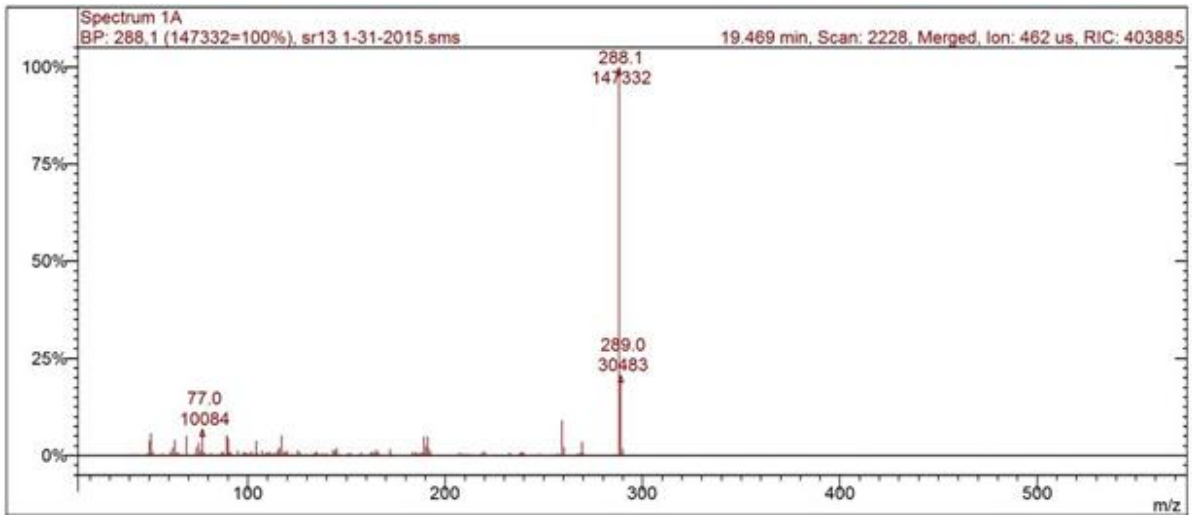

Figure S10: LRMS of 3-phenyl-5-(4-trifluoromethylphenyl)-*1H*-pyrazole (**2e**).

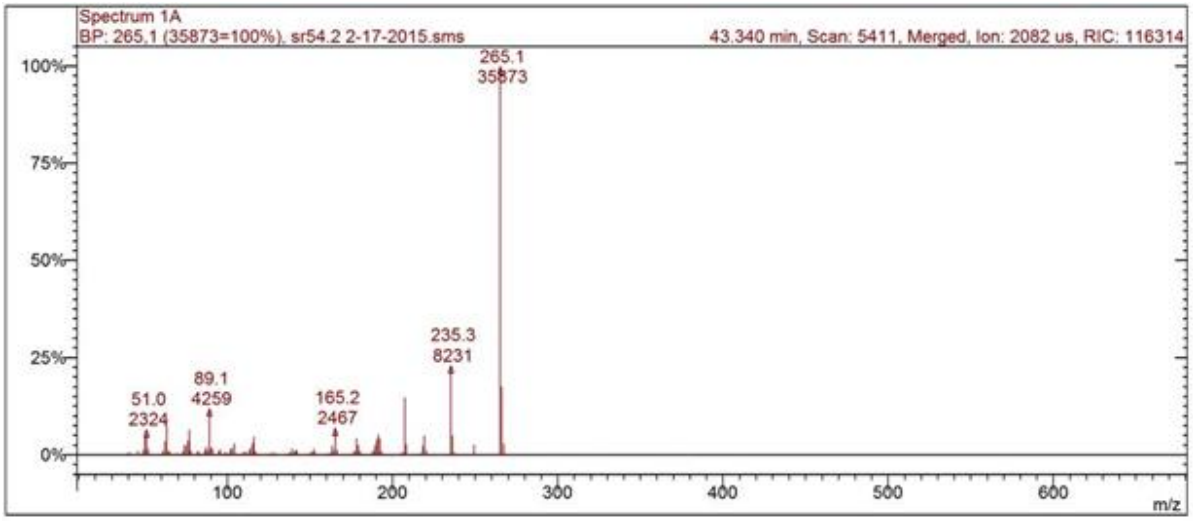

Figure S11: LRMS of 3-phenyl-5-(4-nitrophenyl)-*1H*-pyrazole (**2f**).

Chromatogram and low-resolution mass spectrum of amidino pyrazole 3a

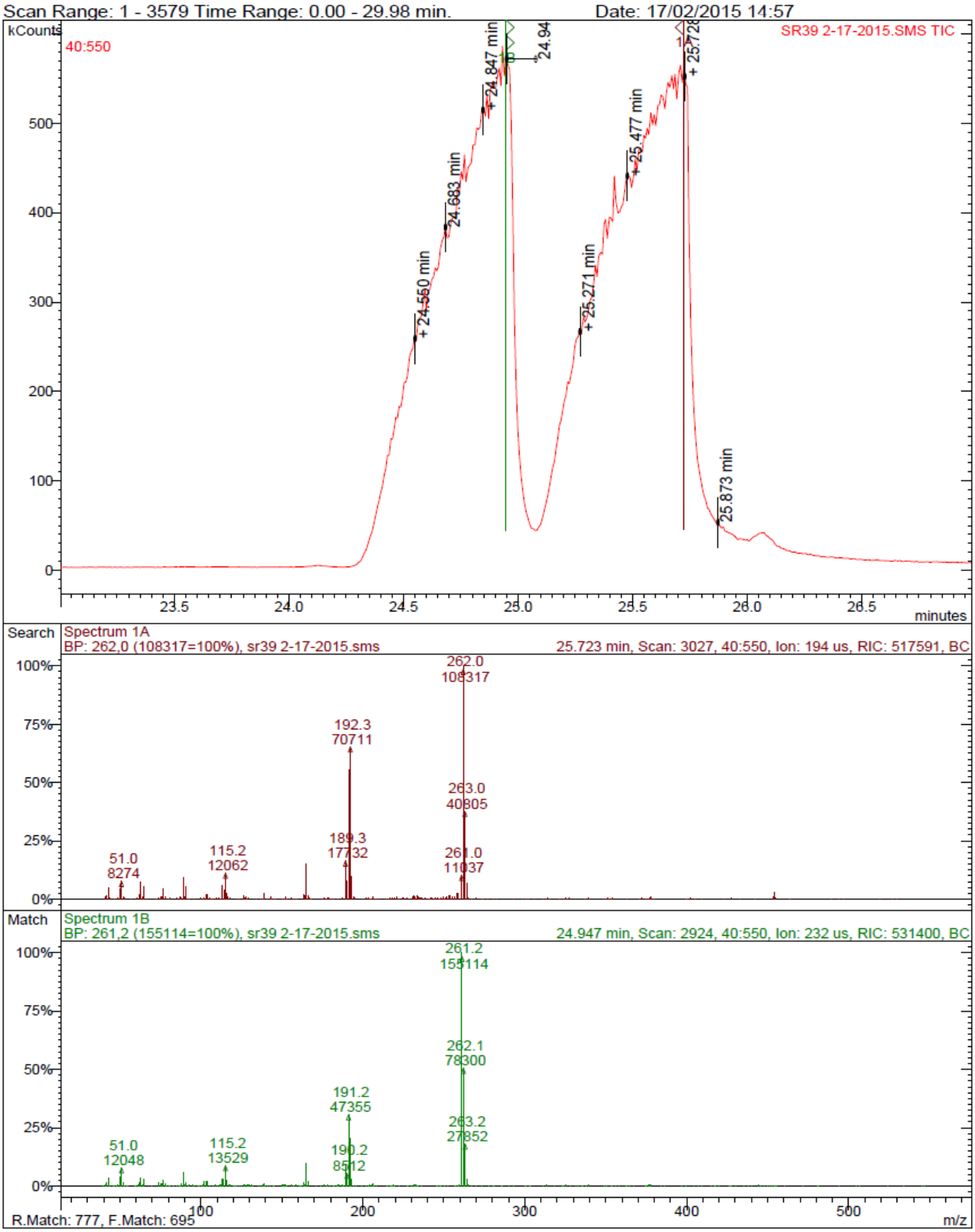

Figure S12: Chromatogram and LRMS of 3,5-diphenyl-1*H*-pyrazole-1-carboximidamide (**3a**).

NMR spectra of bis(hydrazinecarboximidamide) 4a

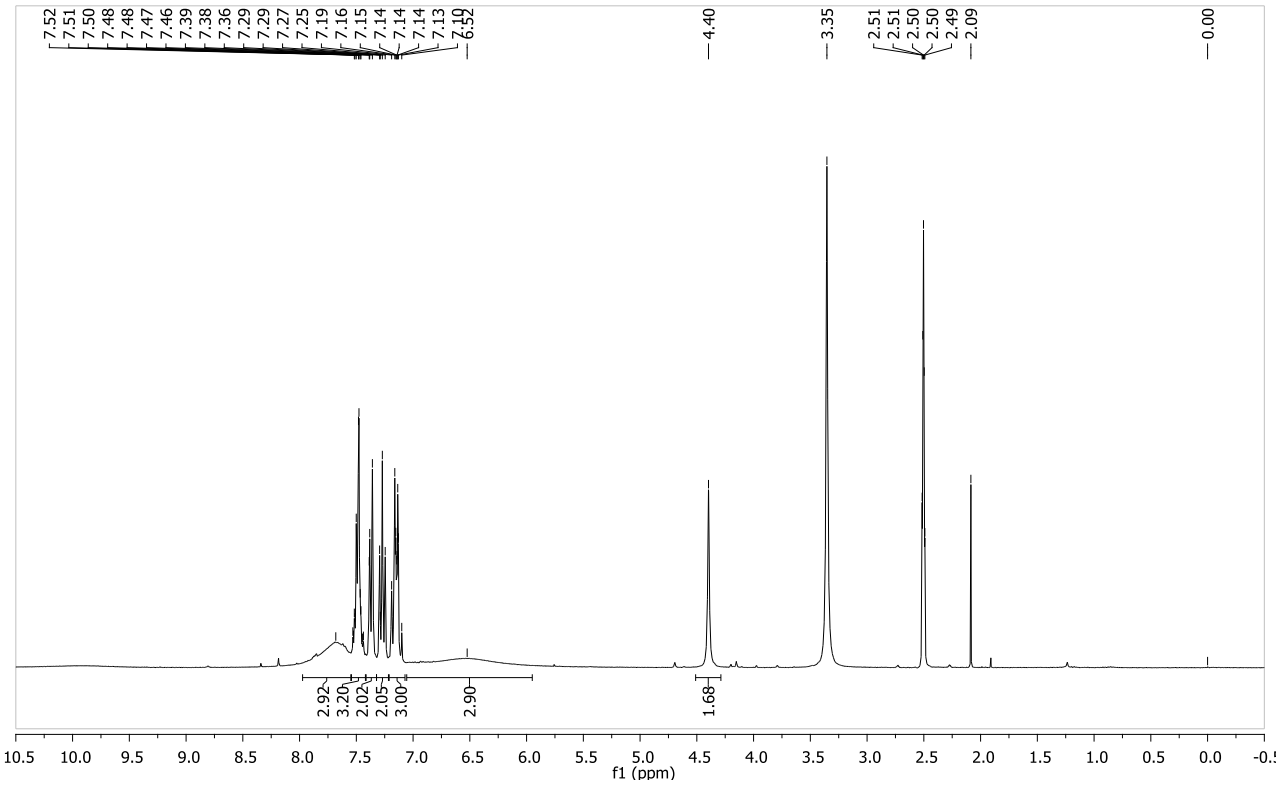

Figure S13: ^{1}H NMR spectrum of **4a** (300 MHz, DMSO-d_6).

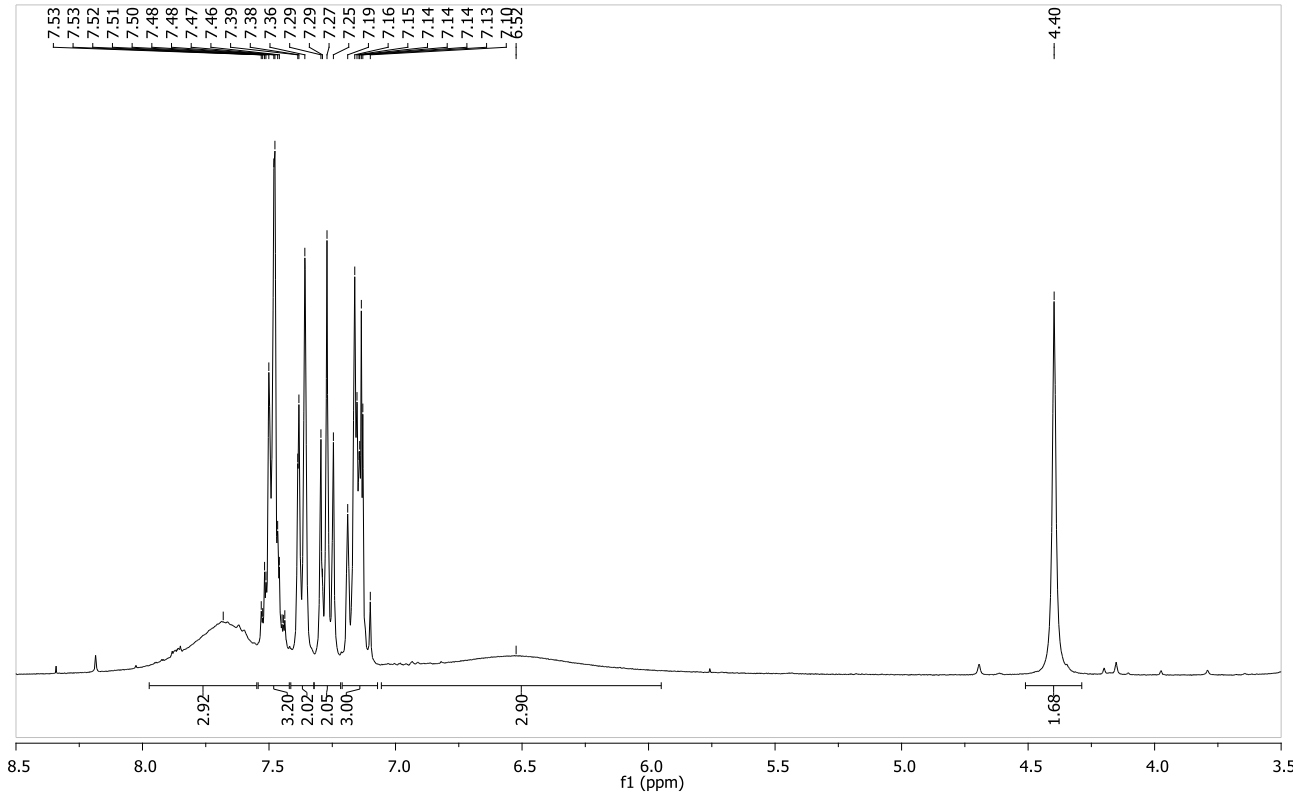

Figure S14: Expansion of the ^{1}H NMR spectrum of **4a** (300 MHz, DMSO-d_6).

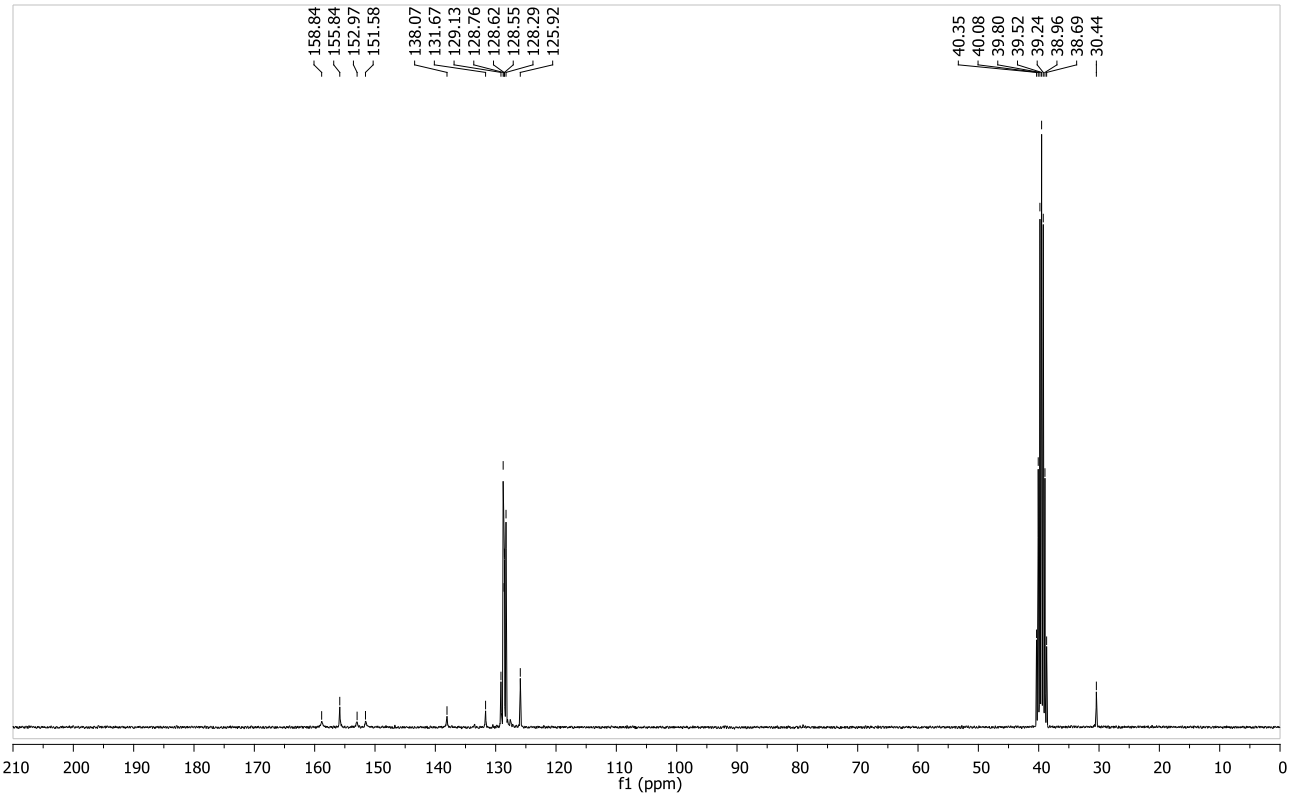

Figure S15: ^{13}C NMR spectrum of **4a** (75 MHz, DMSO-d_6).

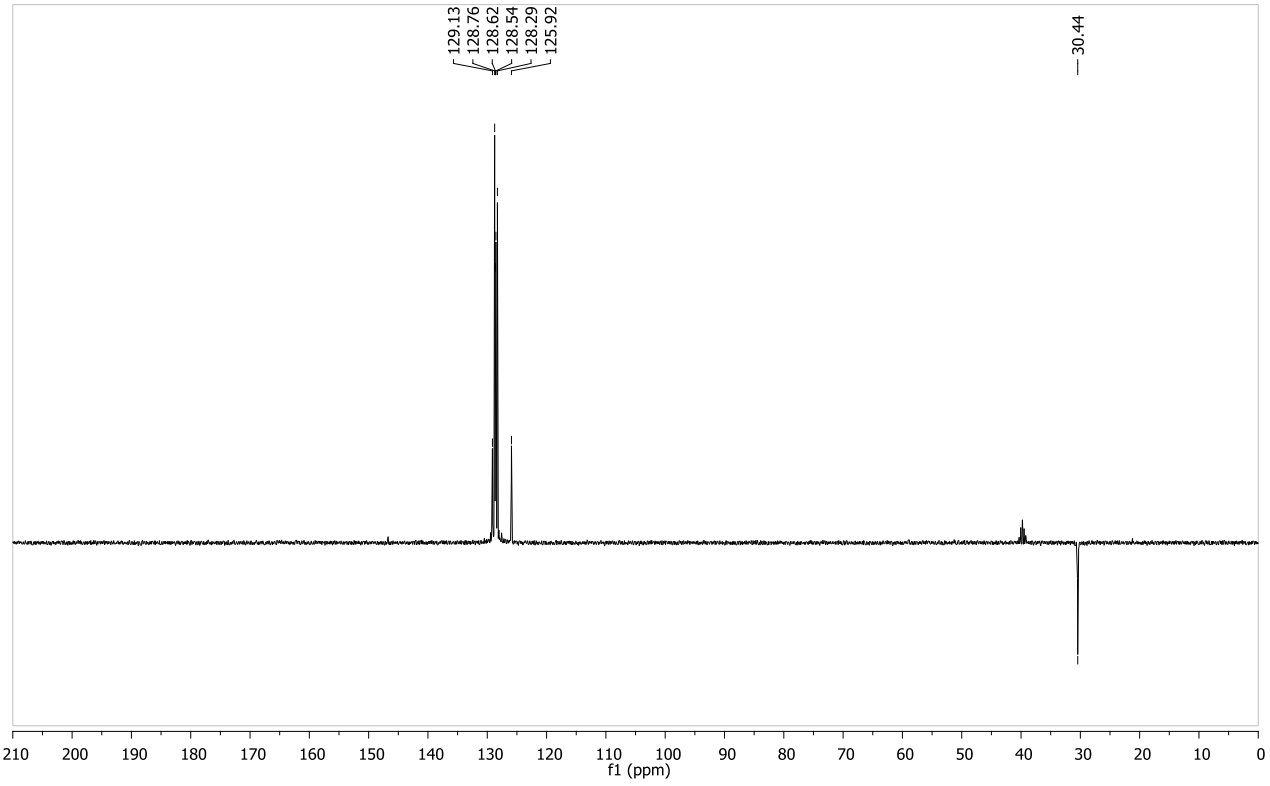

Figure S16: DEPT spectrum of **4a** (75 MHz, DMSO-d_6).

Selected crystallographic data for 4a

CCDC 1536540 contains the supplementary crystallographic data for **4a**. These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

Table S1. Bond lengths and angles for **4a**.

Bond lengths (Å)		**Angles (°)**	
N(6)-C(17)	1.346(2)	C(17)-N(6)-N(5)	116.71(13)
N(6)-N(5)	1.280(2)	C(13)-N(5)-N(6)	118.19(14)
N(8)-C(17)	1.314(2)	C(14)-N(1)-N(2)	120.09(15)
N(1)-C(14)	1.280(2)	C(18)-N(2)-N(1)	115.30(15)
N(1)-N(2)	1.373(2)	N(1)-C(14)-C(13)	112.46(15)
N(2)-C(18)	1.344(2)	N(1)-C(14)-C(15)	129.13(16)
C(14)-C(13)	1.492(2)	C(13)-C(14)-C(15)	118.36(14)
C(14)-C(15)	1.511(2)	N(8)-C(17)-N(7)	121.75(16)
N(7)-C(17)	1.318(2)	N(8)-C(17)-N(6)	118.54(15)
N(3)-C(18)	1.315(2)	N(7)-C(17)-N(6)	119.67(15)
N(4)-C(18)	1.309(3)	C(7)-C(12)-C(11)	119.69(18)
C(12)-C(7)	1.383(3)	C(7)-C(12)-C(13)	117.95(16)
C(12)-C(11)	1.384(3)	C(11)-C(12)-C(13)	122.24(17)
C(12)-C(13)	1.490(2)	N(5)-C(13)-C(12)	126.17(15)
C(1)-C(2)	1.370(3)	N(5)-C(13)-C(14)	116.03(14)
C(1)-C(6)	1.379(3)	C(12)-C(13)-C(14)	117.75(14)
C(1)-C(15)	1.510(3)	N(4)-C(18)-N(3)	120.27(18)
C(7)-C(8)	1.392(3)	N(4)-C(18)-N(2)	120.26(17)
C(11)-C(10)	1.390(3)	N(3)-C(18)-N(2)	119.46(18)
C(9)-C(10)	1.362(4)	C(2)-C(1)-C(6)	117.9(2)
C(9)-C(8)	1.364(4)	C(2)-C(1)-C(15)	121.0(2)
C(6)-C(5)	1.375(4)	C(6)-C(1)-C(15)	121.1(2)
C(5)-C(4)	1.361(5)	C(1)-C(15)-C(14)	109.57(15)
C(2)-C(3)	1.382(5)	C(12)-C(7)-C(8)	119.8(2)
C(3)-C(4)	1.356(6)	C(12)-C(11)-C(10)	119.4(2)
		C(10)-C(9)-C(8)	120.6(2)
		C(9)-C(8)-C(7)	120.0(2)
		C(5)-C(6)-C(1)	120.8(3)
		C(9)-C(10)-C(11)	120.5(2)
		C(4)-C(5)-C(6)	120.4(3)
		C(1)-C(2)-C(3)	120.9(3)
		C(4)-C(3)-C(2)	120.4(3)
		C(3)-C(4)-C(5)	119.5(3)

Table S2. Atomic coordinates (10^4) and equivalent isotropic displacement parameters U(eq)[a] ($Å^2$ x 10^3) for **4a**.

Atom	x	y	z	U(eq)
N(6)	9072(1)	3343(1)	3334(1)	33(1)
N(5)	8711(1)	2772(1)	2878(1)	34(1)
N(8)	9304(1)	4352(1)	4872(1)	42(1)
N(1)	8224(1)	2031(1)	122(1)	39(1)
N(2)	7851(1)	1546(1)	−467(1)	42(1)
C(14)	8332(1)	1888(1)	1283(2)	36(1)
N(7)	8643(1)	3401(1)	5173(2)	47(1)
O	7228(1)	137(2)	198(2)	94(1)
C(17)	9001(1)	3710(1)	4473(2)	33(1)
N(3)	7405(1)	1343(1)	−2312(2)	57(1)
N(4)	7963(1)	2498(1)	−2149(2)	60(1)
C(12)	9173(1)	2621(1)	914(2)	37(1)
C(13)	8748(1)	2471(1)	1757(2)	33(1)
C(18)	7743(1)	1796(1)	−1656(2)	43(1)
C(1)	8477(1)	451(1)	2426(2)	50(1)
C(15)	8112(1)	1208(1)	2173(2)	44(1)
C(7)	9579(1)	2054(2)	1049(2)	53(1)
C(11)	9158(1)	3263(1)	−34(2)	49(1)
C(9)	9948(1)	2757(2)	−716(2)	67(1)
C(8)	9968(1)	2129(2)	225(2)	68(1)
C(6)	8494(1)	−299(2)	1653(3)	74(1)
C(10)	9551(1)	3322(2)	−849(2)	65(1)
C(5)	8834(2)	−978(2)	1874(3)	97(1)
C(2)	8806(1)	490(2)	3427(3)	84(1)
C(3)	9149(2)	−191(3)	3637(4)	117(2)
C(4)	9164(2)	−919(3)	2860(4)	110(1)
Cl(1)	10000	4602(1)	2500	51(1)
Cl(2)	7500	2500	−5000	75(1)
Cl(3)	8412(1)	4683(1)	7416(1)	66(1)

[a] U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Table S3. Anisotropic displacement parameters ($A^2 \times 10^3$) for **4a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 (a^*)^2 U11 + ... + 2 h k (a^*)(b^*) U12]$.

Atom	U11	U22	U33	U23	U13	U12
N(6)	33(1)	39(1)	29(1)	−2(1)	1(1)	−5(1)
N(5)	34(1)	37(1)	31(1)	0(1)	−2(1)	−3(1)
N(8)	48(1)	42(1)	35(1)	−8(1)	1(1)	−5(1)
N(1)	40(1)	43(1)	33(1)	−2(1)	−5(1)	−8(1)
N(2)	44(1)	48(1)	34(1)	2(1)	−4(1)	−15(1)
C(14)	38(1)	37(1)	31(1)	−2(1)	0(1)	−2(1)
N(7)	52(1)	55(1)	34(1)	−7(1)	11(1)	−11(1)
O	107(2)	127(2)	48(1)	2(1)	0(1)	−74(1)
C(17)	35(1)	33(1)	30(1)	1(1)	−1(1)	4(1)
N(3)	53(1)	76(1)	41(1)	−4(1)	−12(1)	−14(1)
N(4)	70(1)	70(1)	37(1)	12(1)	−12(1)	−21(1)
C(12)	40(1)	40(1)	30(1)	−6(1)	−1(1)	−8(1)
C(13)	36(1)	35(1)	28(1)	2(1)	−2(1)	−1(1)
C(18)	40(1)	55(1)	34(1)	−4(1)	−4(1)	−3(1)
C(1)	62(1)	46(1)	41(1)	11(1)	0(1)	−12(1)
C(15)	45(1)	52(1)	34(1)	0(1)	2(1)	−13(1)
C(7)	44(1)	69(1)	44(1)	−1(1)	−3(1)	3(10
C(11)	63(1)	41(1)	44(1)	1(1)	5(1)	−11(1)
C(9)	61(1)	90(2)	52(1)	−26(1)	20(1)	−31(1)
C(8)	43(1)	97(2)	65(2)	−21(1)	5(1)	2(1)
C(6)	97(2)	62(2)	63(2)	−5(1)	−6(1)	1(1)
C(10)	90(2)	61(1)	43(1)	−4(1)	16(1)	−33(1)
C(5)	135(3)	67(2)	90(2)	−2(2)	3(2)	21(2)
C(2)	120(2)	59(2)	71(2)	10(1)	−41(2)	1(2)
C(3)	148(3)	81(2)	117(3)	25(2)	−67(3)	11(2)
C(4)	134(3)	74(2)	122(3)	31(2)	−23(3)	27(2)
Cl(1)	52(1)	49(1)	55(1)	0	21(1)	0
Cl(2)	56(1)	139(1)	28(1)	10(1)	−5(1)	−38(1)
Cl(3)	75(1)	64(1)	59(1)	−21(1)	1(1)	6(1)

Table S4. Hydrogen coordinates (10^4) and isotropic displacement parameters (A^2 x 10^3) for **4a**.

Hydrogen	x	y	z	U(eq)
H(6)	9332	3463	2914	40
H(8A)	9271	4582	5607	50
H(8B)	9535	4543	4397	50
H(2)	7698	1113	−101	51
H(7A)	8602	3620	5912	56
H(7B)	8450	2979	4891	56
H(3A)	7329	1507	−3068	68
H(3B)	7261	882	−1985	68
H(4A)	7887	2664	−2904	71
H(4B)	8183	2793	−1718	71
H(15A)	8033	1506	2955	53
H(15B)	7808	962	1806	53
H(7)	9591	1624	1688	63
H(11)	8889	3651	−126	59
H(9)	10207	2799	−1271	81
H(8)	10242	1751	316	82
H(6A)	8271	−345	974	89
H(10)	9543	3752	−1489	77
H(5)	8839	−1480	1346	117
H(2A)	8797	982	3973	101
H(3)	9372	−150	4315	140
H(4)	9397	−1375	2999	132
H(1B)	6982	148	874	50
H(1A)	7009	−83	−416	50