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Synthesis and X-Ray Crystal Structure of 1',7',7'-Trimethylbicyclo[2.2.1] heptane[2',3'-*b*]-2,3-dicyanopyrazine

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Синтез и структура 1′,7′,7′–триметилбицикло[2.2.1]–гептано[2′,3′–*b*]–2,3–дицианопиразина по данным РСА

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Figure S1. EI MS spectrum for **1**

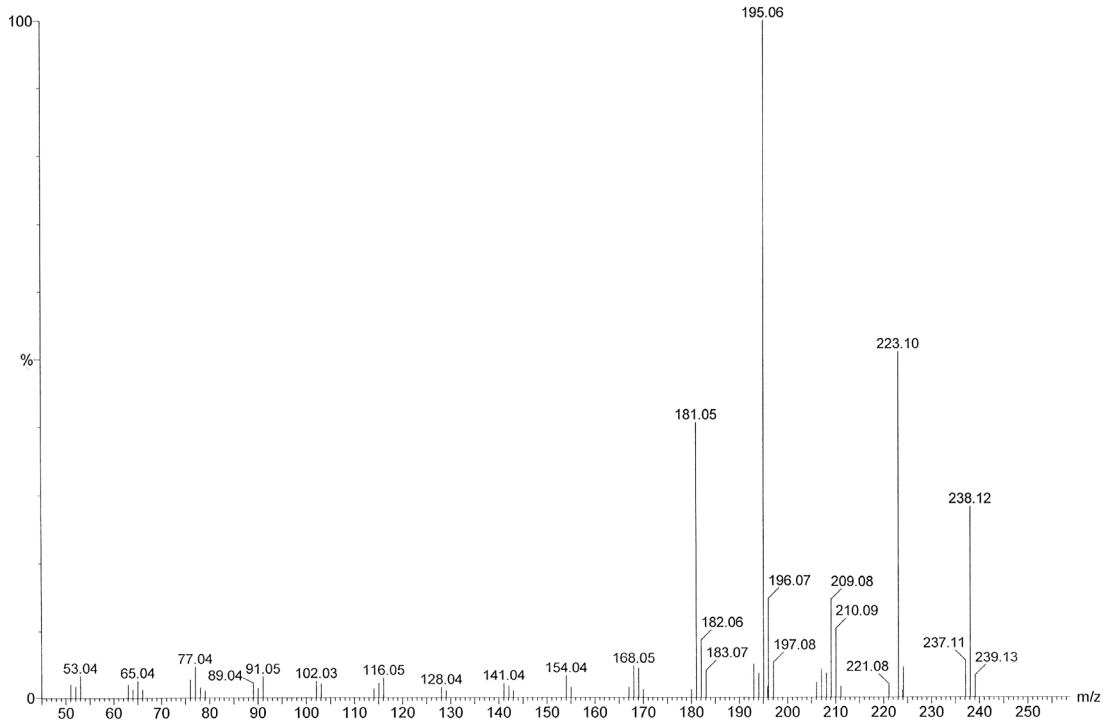

Figure S2. ^{1}H NMR spectrum for **1** in $CDCl_3$

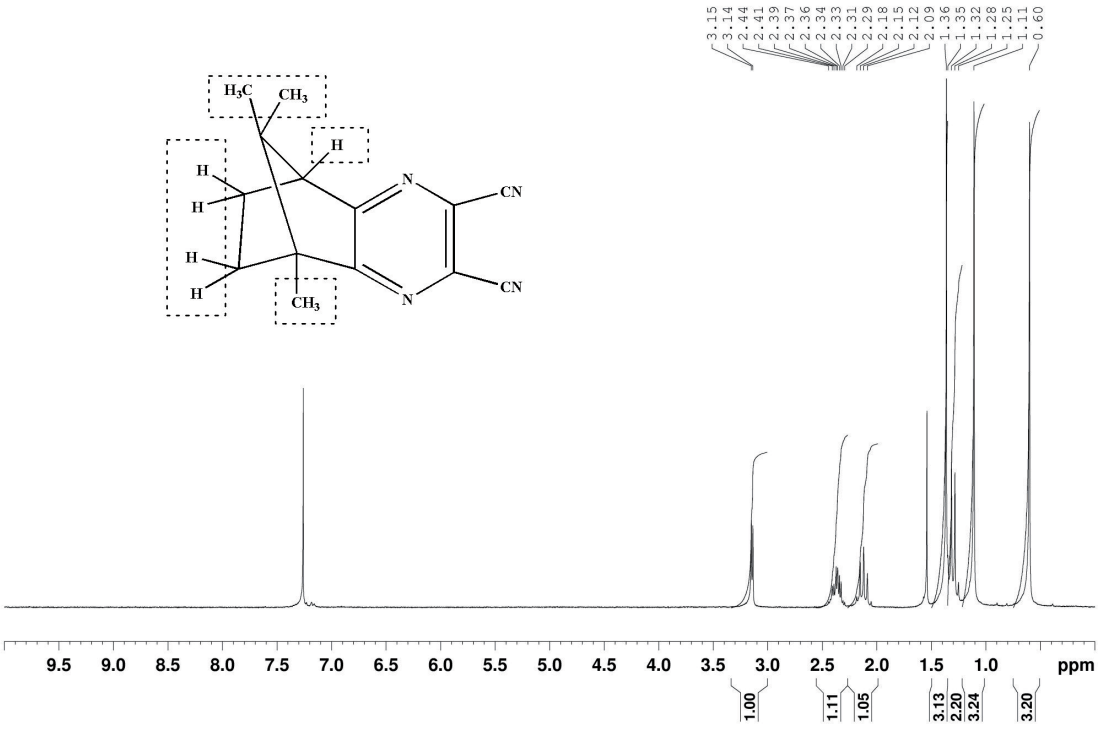

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Figure S3. ^{13}C NMR spectrum for **1** in $CDCl_3$

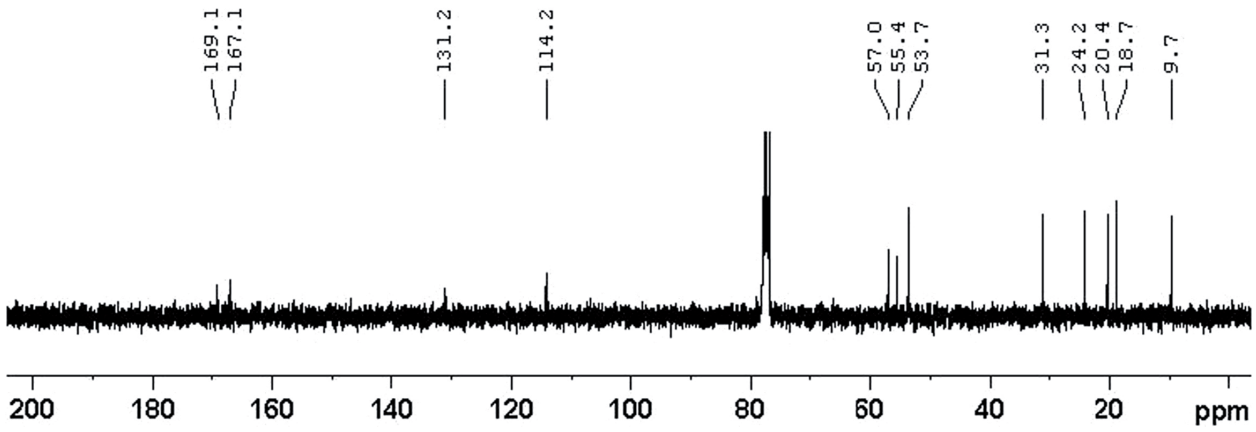

Figure S4. View of **R(-)1**optimized by B3LYP/ cc-pVTZ

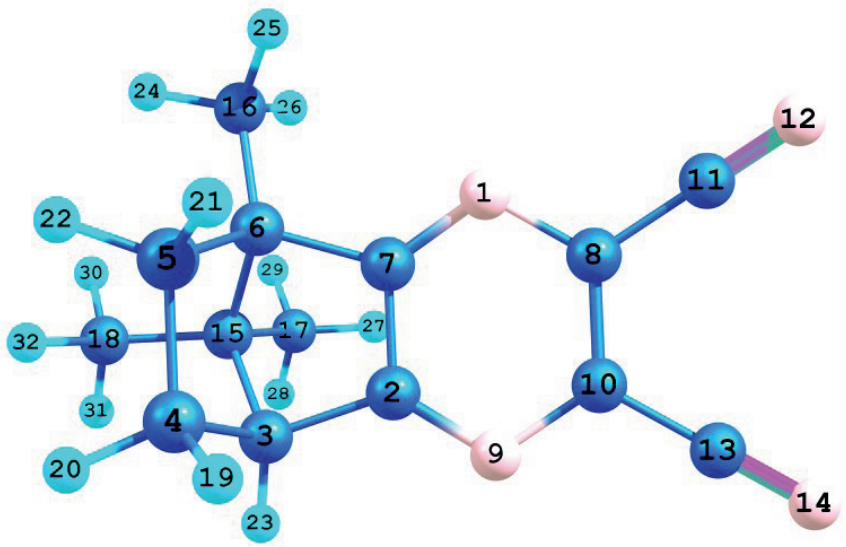

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Table 1. Geometric parameters of **R(-)1** calculated at the B3LYP/ cc-pVTZ level

E_{tot} = -760.666481 a.u.

7	0.802627670	-1.861780116	-0.079081701
6	-0.270700512	0.175548899	0.578878847
6	0.202352811	1.582613869	0.788673720
6	1.093396105	1.503220287	2.068009143
6	2.323656919	0.679893863	1.594789623
6	2.029938896	0.369188968	0.085500848
6	0.853019465	-0.572714103	0.152250685
6	-0.413272478	-2.427926685	0.099764952
7	-1.451921214	-0.358925373	0.772795184
6	-1.525832392	-1.685892855	0.517476063
6	-0.514128949	-3.834470188	-0.156098410
7	-0.595127263	-4.964949317	-0.359796506
6	-2.805497334	-2.305842065	0.696668113
7	-3.833889639	-2.803470344	0.840865448
6	1.271607322	1.693344284	-0.354648814
6	3.222341357	-0.100803665	-0.720693650
6	0.664092199	1.620000566	-1.762080505
6	2.125233957	2.962671423	-0.282905081
1	0.562761115	1.034249312	2.894871841
1	1.375108787	2.502957576	2.393141418
1	2.462648595	-0.238097482	2.164258565
1	3.248424090	1.249447996	1.677148479
1	-0.577938032	2.338578842	0.814745577
1	4.022496623	0.639839219	-0.699666898
1	3.616636938	-1.033496563	-0.315815051
1	2.955113623	-0.287967103	-1.761203492
1	0.034357224	0.744859223	-1.920028046
1	0.051080889	2.502430044	-1.953494581
1	1.450103755	1.599712147	-2.518441291
1	2.893231925	2.946970510	-1.057774762
1	1.500940168	3.839377270	-0.466212294
1	2.623122955	3.108240388	0.672238722

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Figure S5. View of **S(+)1**optimized by B3LYP/ cc-pVTZ

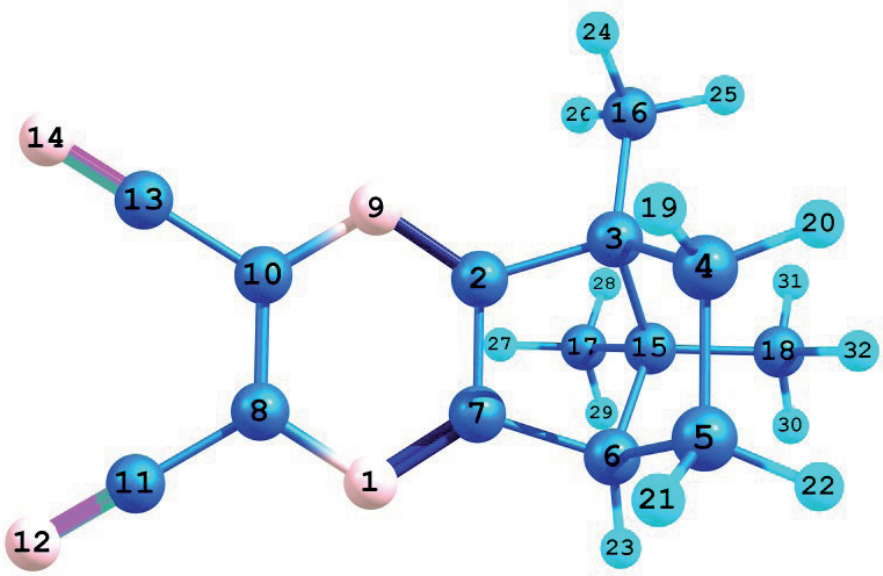

Table 2. Geometric parameters of **S(+)1** calculated at the B3LYP/ cc-pVTZ level

E_{tot} = -760.666484 a.u.

7	0.139060959	-1.187814139	-0.469129756
6	-0.931667348	0.835320393	0.240072162
6	-0.482130044	2.259639426	0.453496456
6	0.460975536	2.152352268	1.702934297
6	1.690063904	1.349006931	1.193496242
6	1.334146413	1.059362822	-0.298145732
6	0.184760643	0.099300439	-0.224604630
6	-1.068162567	-1.764215859	-0.264372327
7	-2.102465082	0.288615664	0.459532322
6	-2.174144419	-1.035931099	0.192977226
6	-1.166496323	-3.168086009	-0.535544275
7	-1.245417843	-4.296355387	-0.751704509
6	-3.443266138	-1.667988011	0.402676966
7	-4.463573176	-2.174285409	0.571884735
6	0.579844966	2.370507498	-0.720153839
6	-1.578851127	3.298882349	0.561847989
6	-0.034835471	2.289086814	-2.123928137
6	1.450037726	3.629411675	-0.666828596
1	-0.045983414	1.667788207	2.536355350
1	0.739762436	3.151038335	2.036384946
1	1.853617823	0.427875348	1.750257117

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1	2.608676267	1.929296844	1.256192577
1	2.167676262	0.742496233	-0.919435093
1	-2.237005433	3.074401286	1.401851051
1	-1.159890013	4.293404184	0.719238633
1	-2.198237783	3.325067589	-0.335331958
1	-0.682556632	1.424588825	-2.266980721
1	-0.625794864	3.181141348	-2.336171172
1	0.753663195	2.234726925	-2.876619186
1	2.243384201	3.562730191	-1.413807425
1	0.854566236	4.511324970	-0.907709093
1	1.920023099	3.802871839	0.297743550

Table 3. Crystal data and structure refinement for **1**

Identification code	datos_m	
Empirical formula	C14 H14 N4	
Formula weight	238.29	
Temperature	100(2) K	
Wavelength	1.54178 E	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 7.1410(5) E	α= 90°.
	b = 33.579(2) E	β= 95.116(4)°.
	c = 10.5417(8) E	γ = 90°.
Volume	2517.7(3) E^3	
Z	8	
Density (calculated)	1.257 Mg/m^3	
Absorption coefficient	0.623 mm^{-1}	
F(000)	1008	
Crystal size	0.20 x 0.08 x 0.04 mm^3	
Theta range for data collection	2.63 to 67.77°.	
Index ranges	-8<=h<=7, -39<=k<=40, -11<=l<=12	
Reflections collected	12189	
Independent reflections	4326 [R(int) = 0.0385]	
Completeness to theta = 67.77°	94.7 %	
Absorption correction	Semi-empirical from equivalents	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4326 / 0 / 331	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0998, wR2 = 0.2606	
R indices (all data)	R1 = 0.1157, wR2 = 0.2743	
Largest diff. peak and hole	1.482 and -0.684 $e.E^{-3}$	

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Table 4. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($Å^2 \times 10^3$) for **1**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C1	10868(7)	115871(14)	18349(5)	36(4)
C2	10423(6)	116174(13)	16988(4)	28(3)
C3	11106(6)	109049(14)	16774(4)	27(9)
C4	10468(6)	112797(13)	16234(4)	26(4)
C5	9572(6)	119776(16)	15229(5)	42(9)
C6	9066(8)	123167(17)	14302(7)	62(9)
C7	6886(7)	122502(15)	13995(6)	44(4)
C8	6824(7)	118719(17)	13186(5)	44(1)
C9	8927(8)	11749(2)	13136(5)	57(5)
C10	9532(6)	116292(16)	14494(5)	37(5)
C11	9881(8)	12142(2)	13070(6)	67(1)
C12	12036(9)	12103(3)	13177(8)	93(9)
C13	9318(10)	12369(2)	11838(7)	75(7)
C14	9679(10)	127128(19)	14659(7)	67(6)
N1	11223(7)	115536(15)	19424(5)	54(1)
N2	11659(6)	106071(12)	17183(4)	40(1)
N3	9960(5)	119825(11)	16487(4)	42(1)
N4	9971(5)	112796(13)	14958(4)	36(9)
C15	5783(6)	112184(13)	16676(4)	31(2)
C16	6372(6)	108317(12)	17181(4)	26(9)
C17	6550(7)	110944(14)	19371(5)	35(3)
C18	6712(6)	107728(12)	18484(4)	25(8)
C19	6918(6)	101885(12)	16807(4)	24(3)
C20	7156(6)	197910(13)	16185(4)	27(8)
C21	9300(6)	197030(13)	16410(4)	27(5)
C22	9611(6)	196300(13)	17855(4)	27(4)
C23	7626(6)	196876(12)	18347(4)	25(6)
C24	7239(5)	101219(12)	18149(4)	23(7)
C25	6319(6)	195163(13)	17186(4)	27(3)
C26	6600(7)	190743(13)	16945(4)	33(2)
C27	4233(6)	195869(14)	17310(5)	32(7)
C28	7428(7)	195298(14)	19664(4)	31(8)
N5	5291(6)	115225(13)	6287(5)	46(2)
N6	6401(7)	113508(14)	10077(5)	53(8)
N7	6488(5)	105367(11)	6299(3)	27(2)
N8	7174(5)	104085(10)	8994(3)	25(2)

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Table 5. Bond lengths [Å] and angles [°] for **1**

Atom1	Atom2	Length
C1	N1	1.145(7)
C2	C4	1.387(6)
C2	N3	1.364(6)
C3	C4	1.439(6)
C3	N2	1.145(6)
C4	N4	1.360(6)
C5	C6	1.523(8)
C5	C10	1.402(8)
C5	N3	1.330(7)
C6	C7	1.577(8)
C6	C11	1.58(1)
C6	C14	1.440(9)
C7	H7A	0.991(6)
C7	H7B	0.989(5)
C7	C8	1.528(8)
C8	H8A	0.990(5)
C8	H8B	0.990(6)
C8	C9	1.563(8)
C9	H9	0.999(6)
C9	C10	1.512(7)
C9	C11	1.490(9)
C10	N4	1.299(7)
C11	C12	1.539(9)
C11	C13	1.528(9)
C12	H12A	0.979(9)
C12	H12B	0.982(9)
C12	H12C	0.980(8)
C13	H13A	0.981(7)
C13	H13B	0.978(7)
C13	H13C	0.980(7)
C14	H14A	0.980(7)
C14	H14B	0.980(7)
C14	H14C	0.980(7)
C15	C16	1.451(6)
C15	N5	1.144(6)
C16	C18	1.388(6)
C16	N7	1.366(5)
C17	C18	1.440(6)
C17	N6	1.149(7)
C18	N8	1.365(5)
C19	C20	1.503(6)
C19	C24	1.430(6)
C19	N7	1.311(5)
C20	H20	1.000(4)
C20	C21	1.557(6)
C20	C25	1.560(6)

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C21	H21A	0.989(4)
C21	H21B	0.991(4)
C21	C22	1.539(6)
C22	H22A	0.991(4)
C22	H22B	0.990(4)
C22	C23	1.564(6)
C23	C24	1.495(6)
C23	C25	1.580(6)
C23	C28	1.504(6)
C24	N8	1.315(5)
C25	C26	1.522(6)
C25	C27	1.525(6)
C26	H26A	0.980(5)
C26	H26B	0.979(5)
C26	H26C	0.980(4)
C27	H27A	0.980(5)
C27	H27B	0.979(5)
C27	H27C	0.979(5)
C28	H28A	0.980(4)
C28	H28B	0.981(5)
C28	H28C	0.980(5)

Atom1	Atom2	Atom3	Angle
C2	C1	N1	178.4(5)
C1	C2	C4	119.8(4)
C1	C2	N3	118.2(4)
C4	C2	N3	122.0(4)
C4	C3	N2	178.0(5)
C2	C4	C3	120.7(4)
C2	C4	N4	123.3(4)
C3	C4	N4	115.9(4)
C6	C5	C10	106.1(4)
C6	C5	N3	130.3(5)
C10	C5	N3	123.6(5)
C5	C6	C7	101.5(5)
C5	C6	C11	99.5(5)
C5	C6	C14	118.3(5)
C7	C6	C11	102.0(5)
C7	C6	C14	117.1(5)
C11	C6	C14	115.6(5)
C6	C7	H7A	111.3(5)
C6	C7	H7B	111.4(5)
C6	C7	C8	102.2(4)
H7A	C7	H7B	109.3(5)
H7A	C7	C8	111.3(5)
H7B	C7	C8	111.3(5)

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C7	C8	H8A	110.7(5)
C7	C8	H8B	110.8(5)
C7	C8	C9	105.0(4)
H8A	C8	H8B	108.8(5)
H8A	C8	C9	110.8(5)
H8B	C8	C9	110.7(5)
C8	C9	H9	116.1(5)
C8	C9	C10	103.4(4)
C8	C9	C11	102.3(5)
H9	C9	C10	116.1(5)
H9	C9	C11	116.0(5)
C10	C9	C11	100.8(5)
C5	C10	C9	106.9(4)
C5	C10	N4	123.6(5)
C9	C10	N4	129.5(5)
C6	C11	C9	95.0(5)
C6	C11	C12	114.2(5)
C6	C11	C13	115.1(5)
C9	C11	C12	112.3(5)
C9	C11	C13	113.3(5)
C12	C11	C13	106.9(6)
C11	C12	H12A	109.6(7)
C11	C12	H12B	109.4(7)
C11	C12	H12C	109.5(7)
H12A	C12	H12B	109.4(8)
H12A	C12	H12C	109.6(8)
H12B	C12	H12C	109.4(8)
C11	C13	H13A	109.4(6)
C11	C13	H13B	109.5(6)
C11	C13	H13C	109.5(6)
H13A	C13	H13B	109.6(7)
H13A	C13	H13C	109.4(7)
H13B	C13	H13C	109.5(7)
C6	C14	H14A	109.5(6)
C6	C14	H14B	109.4(6)
C6	C14	H14C	109.5(6)
H14A	C14	H14B	109.5(7)
H14A	C14	H14C	109.5(7)
H14B	C14	H14C	109.4(7)
C2	N3	C5	113.4(4)
C4	N4	C10	113.9(4)
C16	C15	N5	178.9(5)
C15	C16	C18	120.7(4)

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C15	C16	N7	115.6(4)
C18	C16	N7	123.7(4)
C18	C17	N6	179.3(5)
C16	C18	C17	121.3(4)
C16	C18	N8	122.2(4)
C17	C18	N8	116.6(4)
C20	C19	C24	106.2(3)
C20	C19	N7	130.2(4)
C24	C19	N7	123.7(4)
C19	C20	H20	116.2(4)
C19	C20	C21	104.5(3)
C19	C20	C25	99.5(3)
H20	C20	C21	116.0(4)
H20	C20	C25	116.0(4)
C21	C20	C25	102.5(3)
C20	C21	H21A	111.0(4)
C20	C21	H21B	111.0(4)
C20	C21	C22	103.5(3)
H21A	C21	H21B	109.1(4)
H21A	C21	C22	111.1(4)
H21B	C21	C22	111.0(4)
C21	C22	H22A	110.8(4)
C21	C22	H22B	110.8(4)
C21	C22	C23	104.7(3)
H22A	C22	H22B	108.9(4)
H22A	C22	C23	110.8(4)
H22B	C22	C23	110.8(4)
C22	C23	C24	103.6(3)
C22	C23	C25	100.9(3)
C22	C23	C28	115.1(4)
C24	C23	C25	99.1(3)
C24	C23	C28	116.2(4)
C25	C23	C28	119.3(4)
C19	C24	C23	107.6(3)
C19	C24	N8	122.9(4)
C23	C24	N8	129.5(4)
C20	C25	C23	94.2(3)
C20	C25	C26	113.5(4)
C20	C25	C27	113.7(4)
C23	C25	C26	114.0(4)
C23	C25	C27	113.0(4)
C26	C25	C27	108.1(4)
C25	C26	H26A	109.4(4)

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C25	C26	H26B	109.4(4)
C25	C26	H26C	109.5(4)
H26A	C26	H26B	109.5(4)
H26A	C26	H26C	109.5(4)
H26B	C26	H26C	109.5(4)
C25	C27	H27A	109.5(4)
C25	C27	H27B	109.5(4)
C25	C27	H27C	109.5(4)
H27A	C27	H27B	109.5(5)
H27A	C27	H27C	109.5(5)
H27B	C27	H27C	109.4(5)
C23	C28	H28A	109.5(4)
C23	C28	H28B	109.5(4)
C23	C28	H28C	109.4(4)
H28A	C28	H28B	109.4(4)
H28A	C28	H28C	109.6(4)
H28B	C28	H28C	109.5(4)
C16	N7	C19	113.3(4)
C18	N8	C24	114.3(3)