SUPPLEMENTARY FILE

Using of Benzotraizolium Iodide Ionic Liquids for Extractive Desulfurization of Model Diesel Oil

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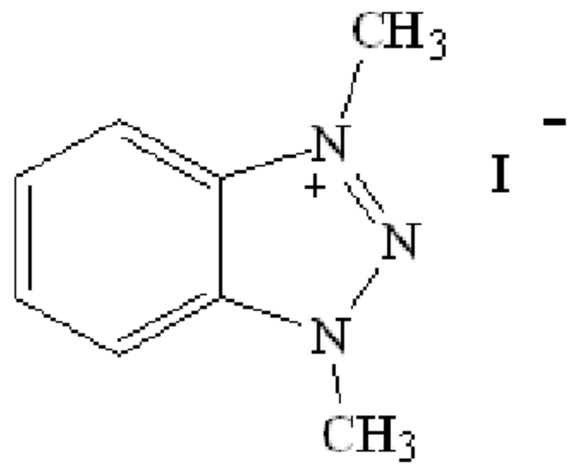
[rel]

8.3732
8.3644
8.3569
8.01176
8.0108
8.0013
7.9938
7.8950
7.4168

4.6344
4.4953

3.3737

2.5129



15

10

5

0

4.2022

0.1458

0.0787

6.1107

8

7

6

5

4

3

[ppm]

Table 1. Crystal data and structure refinement for . Assim A. Sabah

Empirical formula	$(C_8 H_{10} N_3)^+ . I^-$	
Formula weight	275.09	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pnma	
Unit cell dimensions	a = 8.8972(4) Å	α= 90°
	b = 6.4646(3) Å	β= 90°
	c = 17.0708(7) Å	γ = 90°
Volume	981.86(8) $Å^3$	
Z	4	
Density (calculated)	1.861 Mg/m^3	
Absorption coefficient	3.213 mm^{-1}	
F(000)	528	
Crystal size	0.52 x 0.51 x 0.40 mm^3	
Theta range for data collection	2.39 to 33.04°.	
Index ranges	-13<=h<=13, -9<=k<=9, -26<=l<=26	
Reflections collected	16404	
Independent reflections	1922 [R(int) = 0.0606]	
Completeness to theta = 32.00°	99.7 %	
Absorption correction	multi-scan	
Max. and min. transmission	0.3597 and 0.2858	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	1922 / 0 / 76	
Goodness-of-fit on F^2	1.005	
Final R indices [I>2sigma(I)]	R1 = 0.0322, wR2 = 0.0773	
R indices (all data)	R1 = 0.0418, wR2 = 0.0816	
Extinction coefficient	0.0398(18)	
Largest diff. peak and hole	1.310 and -1.612 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for TAQA1. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
I1	766(1)	2500	3267(1)	31(1)
N1	5179(3)	2500	6481(2)	24(1)
N2	3703(3)	2500	6511(2)	26(1)
N3	3240(3)	2500	5776(1)	23(1)
C1	5697(3)	2500	5727(2)	22(1)
C2	7154(3)	2500	5417(2)	27(1)
C3	7240(4)	2500	4610(2)	30(1)
C4	5930(3)	2500	4137(2)	29(1)
C5	4502(4)	2500	4438(2)	25(1)
C6	4412(3)	2500	5256(2)	21(1)
C7	6070(4)	2500	7205(2)	35(1)
C8	1615(3)	2500	5616(2)	29(1)

Table 3. Bond lengths [Å] and angles [°] for TAQA1.

N1-N2	1.314(4)
N1-C1	1.368(4)
N1-C7	1.468(4)
N2-N3	1.321(3)
N3-C6	1.369(4)
N3-C8	1.471(4)
C1-C6	1.397(4)
C1-C2	1.400(4)
C2-C3	1.380(5)
C2-H2	0.9500
C3-C4	1.419(5)
C3-H3	0.9500
C4-C5	1.371(4)
C4-H4	0.9500
C5-C6	1.400(4)
C5-H5	0.9500
C7-H7A	0.9800
C7-H7B	0.9800
C7-H7C	0.9800
C8-H8A	0.9800
C8-H8B	0.9800
C8-H8C	0.9800
N2-N1-C1	111.9(2)
N2-N1-C7	120.5(3)
C1-N1-C7	127.6(3)
N1-N2-N3	105.9(2)
N2-N3-C6	112.2(2)
N2-N3-C8	118.8(2)
C6-N3-C8	129.0(2)
N1-C1-C6	105.4(2)
N1-C1-C2	131.9(3)
C6-C1-C2	122.7(3)
C3-C2-C1	115.4(3)

C3-C2-H2	122.3
C1-C2-H2	122.3
C2-C3-C4	121.5(3)
C2-C3-H3	119.2
C4-C3-H3	119.2
C5-C4-C3	123.3(3)
C5-C4-H4	118.4
C3-C4-H4	118.4
C4-C5-C6	115.3(3)
C4-C5-H5	122.4
C6-C5-H5	122.4
N3-C6-C1	104.6(2)
N3-C6-C5	133.6(3)
C1-C6-C5	121.8(3)
N1-C7-H7A	109.5
N1-C7-H7B	109.5
H7A-C7-H7B	109.5
N1-C7-H7C	109.5
H7A-C7-H7C	109.5
H7B-C7-H7C	109.5
N3-C8-H8A	109.5
N3-C8-H8B	109.5
H8A-C8-H8B	109.5
N3-C8-H8C	109.5
H8A-C8-H8C	109.5
H8B-C8-H8C	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for TAQA1. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
I1	27(1)	45(1)	23(1)	0	1(1)	0
N1	26(1)	28(1)	18(1)	0	-1(1)	0
N2	28(1)	30(1)	20(1)	0	3(1)	0
N3	23(1)	25(1)	20(1)	0	2(1)	0
C1	23(1)	21(1)	22(1)	0	2(1)	0
C2	22(1)	25(1)	35(2)	0	4(1)	0
C3	32(1)	24(1)	34(2)	0	14(1)	0
C4	40(2)	22(1)	24(1)	0	9(1)	0
C5	34(1)	21(1)	20(1)	0	2(1)	0
C6	26(1)	18(1)	19(1)	0	1(1)	0
C7	41(2)	42(2)	22(1)	0	-11(1)	0
C8	21(1)	33(2)	34(2)	0	3(1)	0

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for TAQA1.

	x	y	z	U(eq)
H2	8023	2500	5740	33
H3	8200	2500	4366	36
H4	6049	2500	3584	34
H5	3634	2500	4113	29
H7A	6929	3435	7145	53
H7B	6432	1096	7311	53
H7C	5443	2969	7642	53
H8A	1141	1343	5893	44
H8B	1445	2352	5052	44
H8C	1176	3805	5798	44

Table 6. Torsion angles [°] for TAQA1.

C1-N1-N2-N3	0.0
C7-N1-N2-N3	180.0
N1-N2-N3-C6	0.0
N1-N2-N3-C8	180.0
N2-N1-C1-C6	0.0
C7-N1-C1-C6	180.0
N2-N1-C1-C2	180.0
C7-N1-C1-C2	0.000(1)
N1-C1-C2-C3	180.0
C6-C1-C2-C3	0.000(1)
C1-C2-C3-C4	0.0
C2-C3-C4-C5	0.0
C3-C4-C5-C6	0.0
N2-N3-C6-C1	0.0
C8-N3-C6-C1	180.0
N2-N3-C6-C5	180.0
C8-N3-C6-C5	0.000(1)
N1-C1-C6-N3	0.0
C2-C1-C6-N3	180.0
N1-C1-C6-C5	180.0
C2-C1-C6-C5	0.0
C4-C5-C6-N3	180.0
C4-C5-C6-C1	0.0

Table 7. Hydrogen bonds for TAQA1 [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
C5-H5...I1	0.95	2.93	3.877(3)	174
C3-H3...I1[i]	0.95	2.95	3.885(3)	167

Symmetry transformations used to generate equivalent atoms:

(i): x+1,y,z

And some nice pictures:

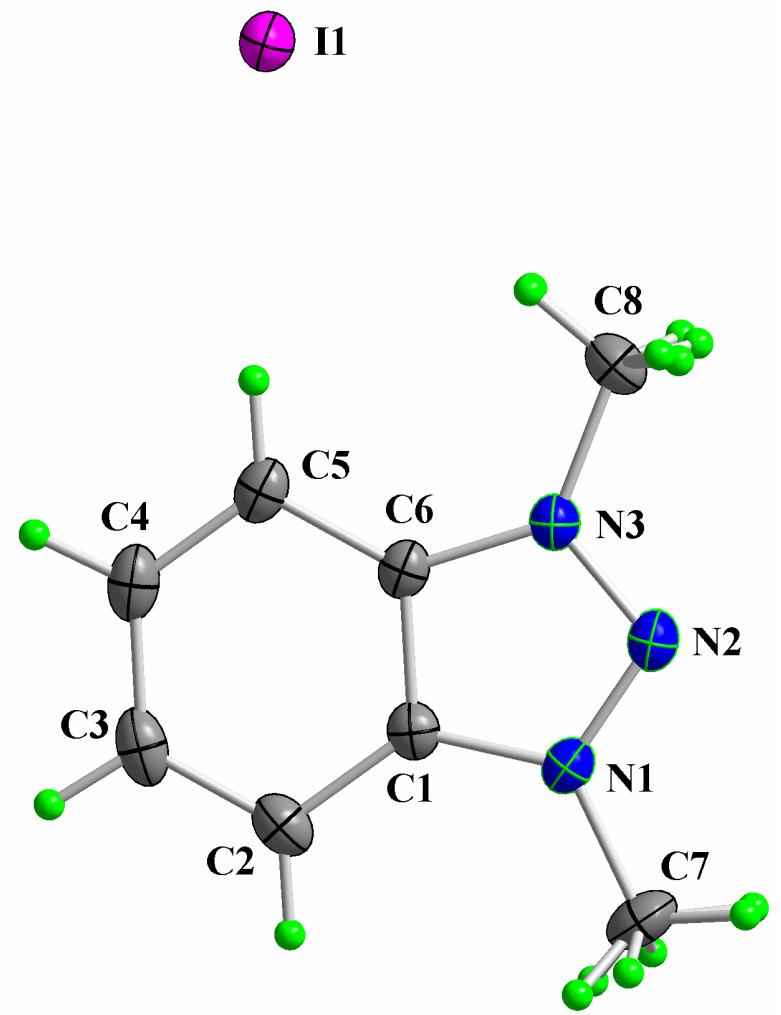


Fig.1. A perspective drawing of the title compound, showing the atom-numbering scheme. Displacement ellipsoids are drawn at 50% probability level and H atoms are shown as small spheres of arbitrary radii. Organic ions are located on the crystallographic mirror plane are hence planar. Methyl hydrogens are disordered with 50% occupancy.

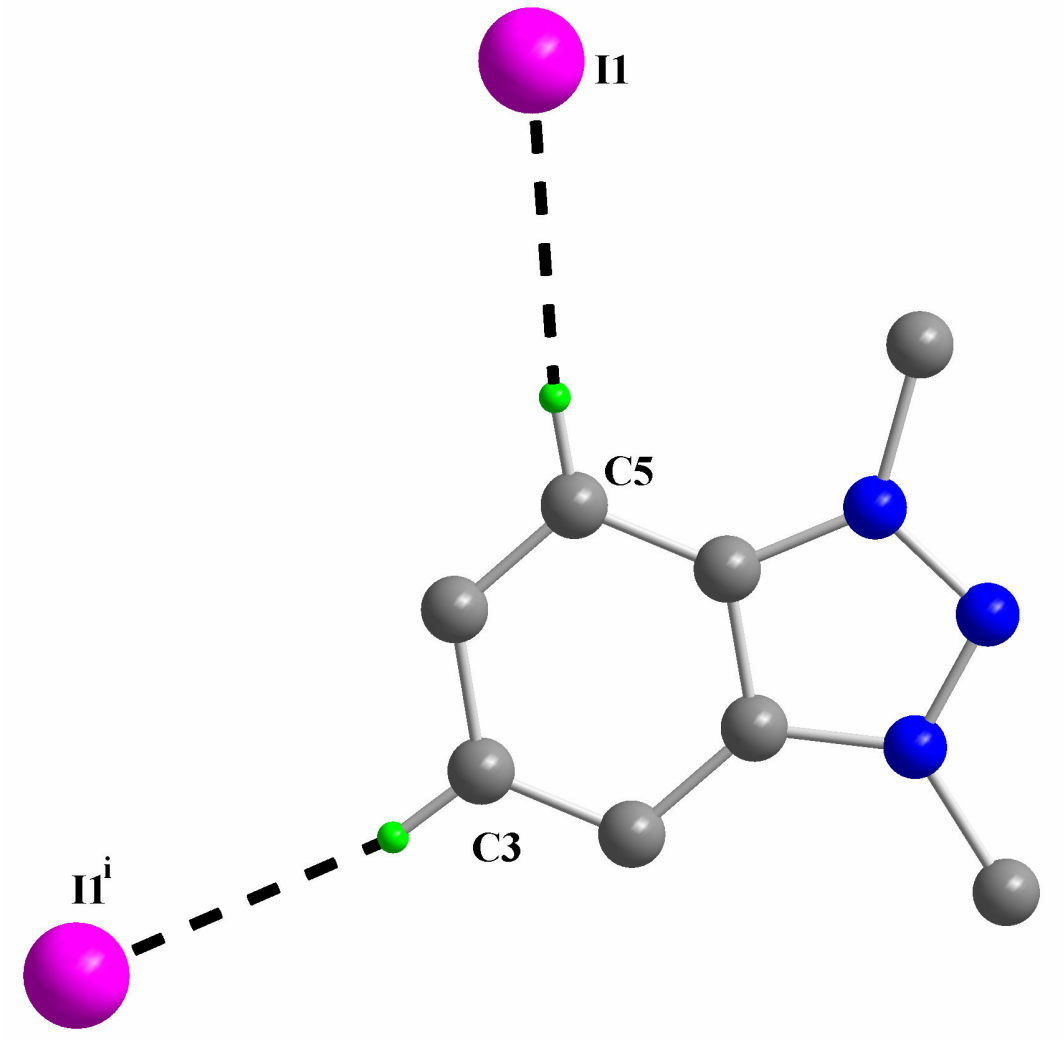


Fig.2. The hydrogen-pattern (dashed lines). For full details, see Table 7. Hydrogen atoms not taking part in the hydrogen bonds were omitted for clarity.

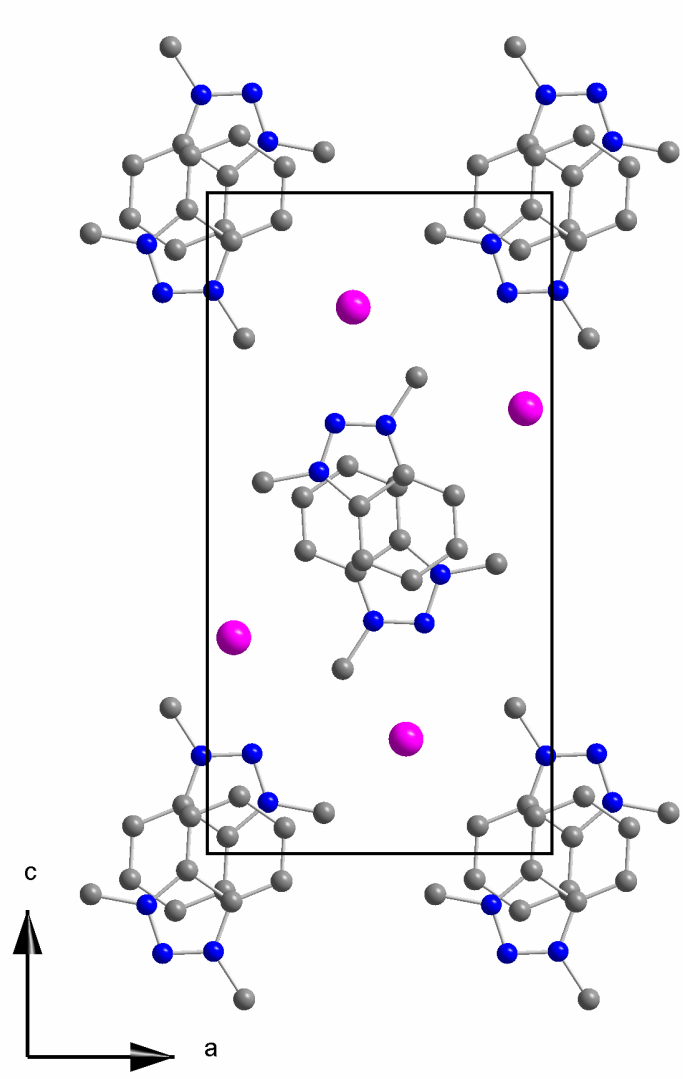
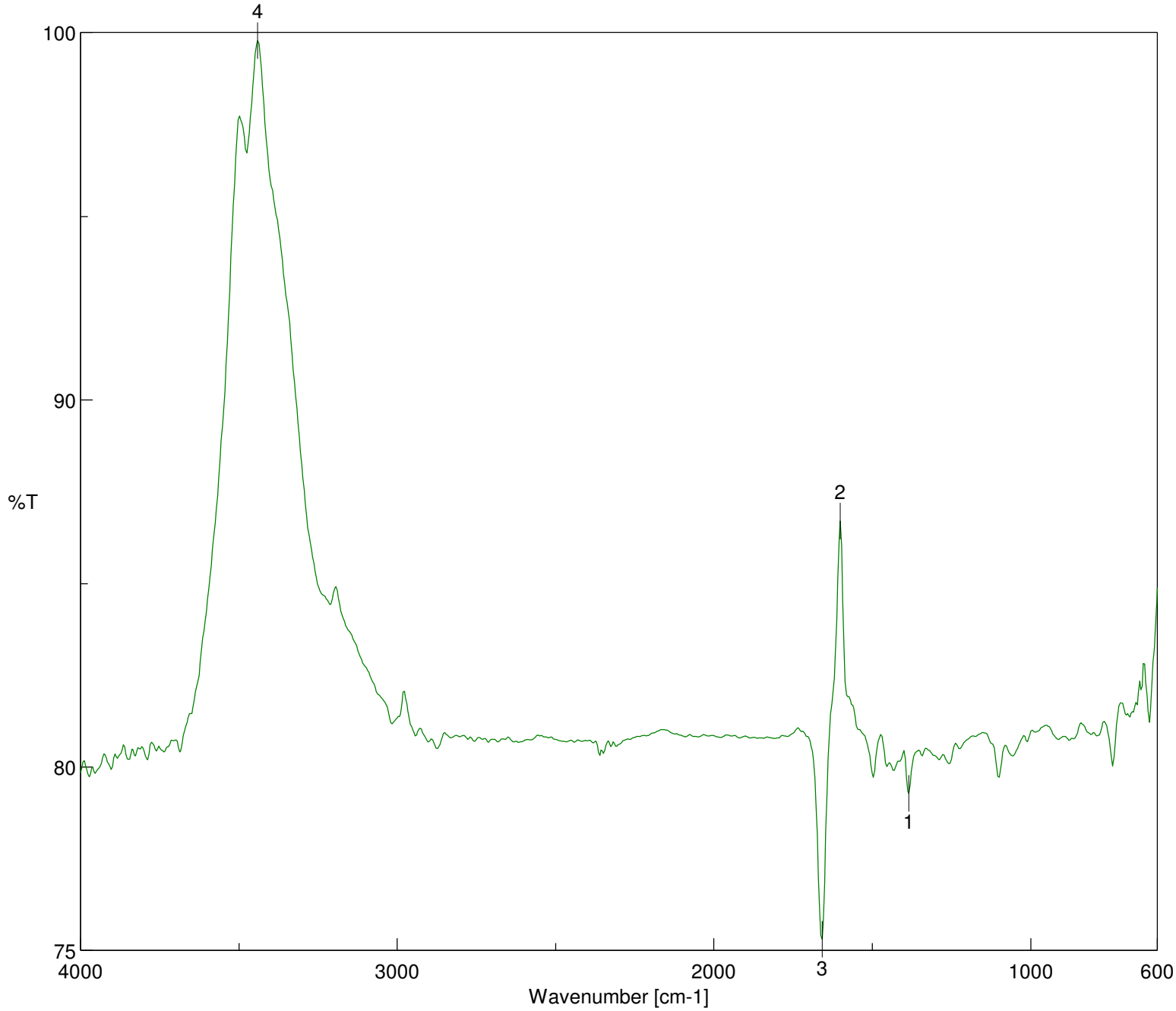


Fig.3. Projection of the structure along the b-axis. Hydrogen atoms were omitted for clarity.



Results of Peak Find

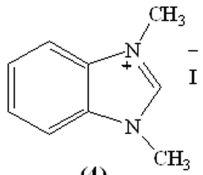
No.	Position	Intensity
1	1384.64	79.2774
2	1600.63	86.6885
3	1658.48	75.2986
4	3440.39	99.77

[FileName] Memory-64

[Date] 21/02/2011 17:07

Sample ID: 4...A.SABAH.. CI+(NH3) SOLID PROBE TRIO-1000 Acquired on 09-Mar-2011 at 16:56:32

V223 32 (2.692) Scan CI+ 1.72e6



(4)

"sample 6" 1 1 M: Assim

8.3957 8.3697 8.3402 8.0234 8.0002

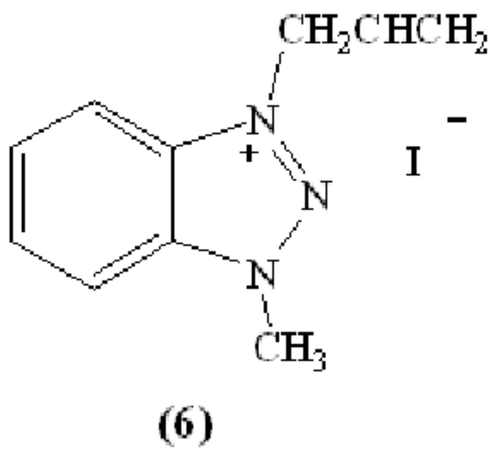
6.2201 6.1701 6.1301 5.7270 5.7101 5.5501 5.5285 5.5101 5.4823 5.4601

4.6401

3.3601

2.5101

2.1000



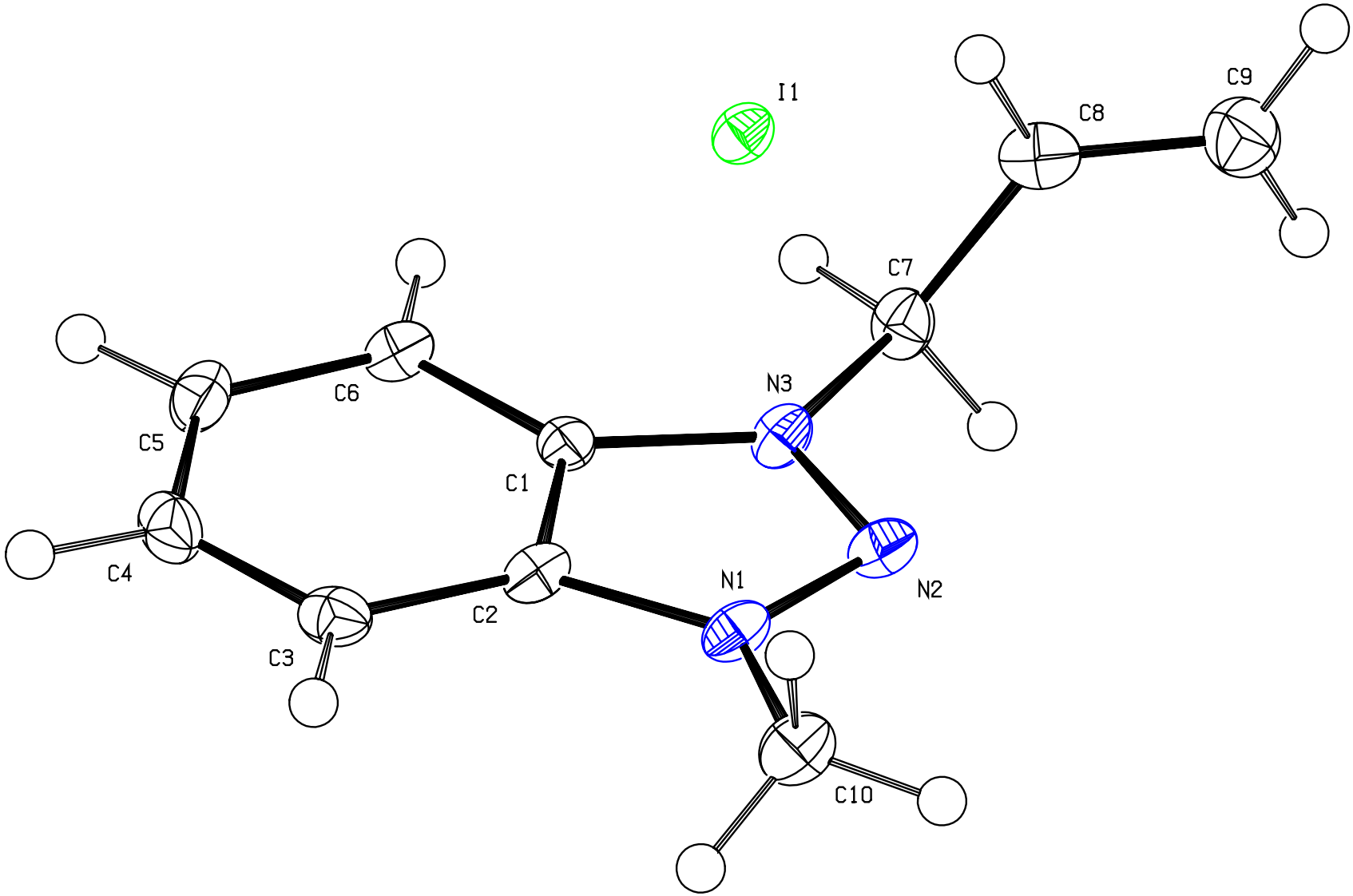
(6)

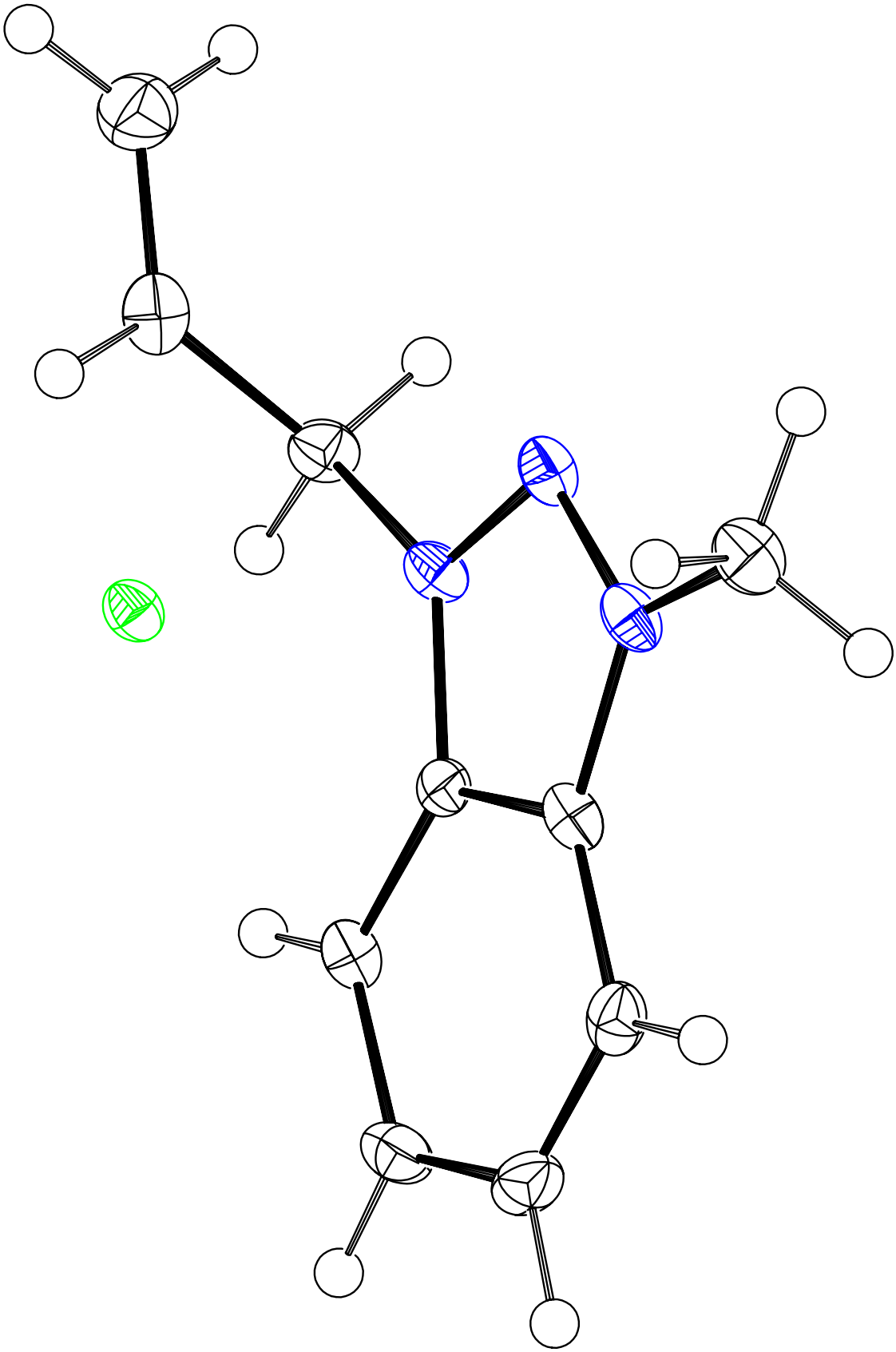
1.8595 1.8070 0.2338 1.7877 1.0427 1.8492 2.9596

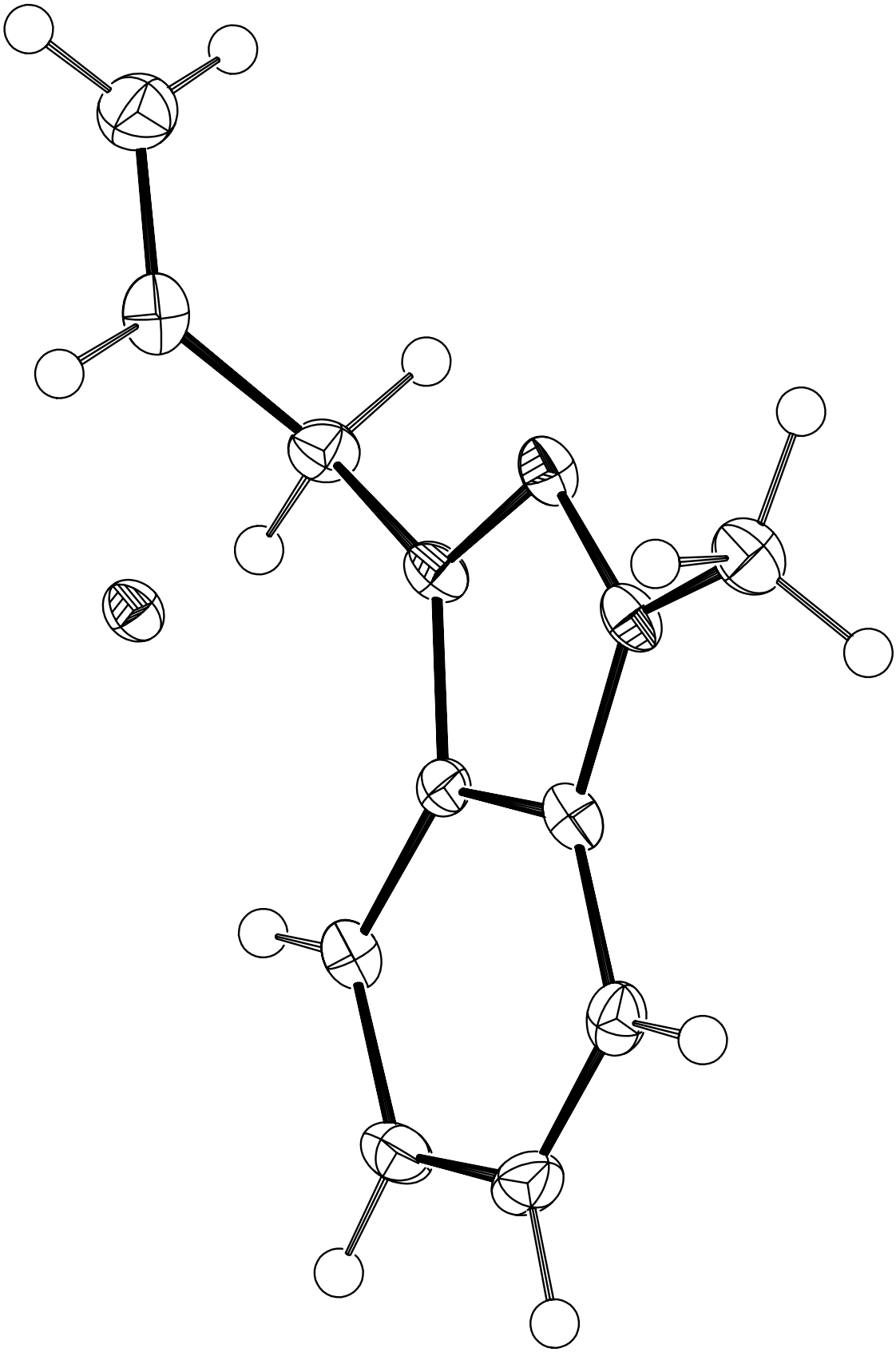
[rel]

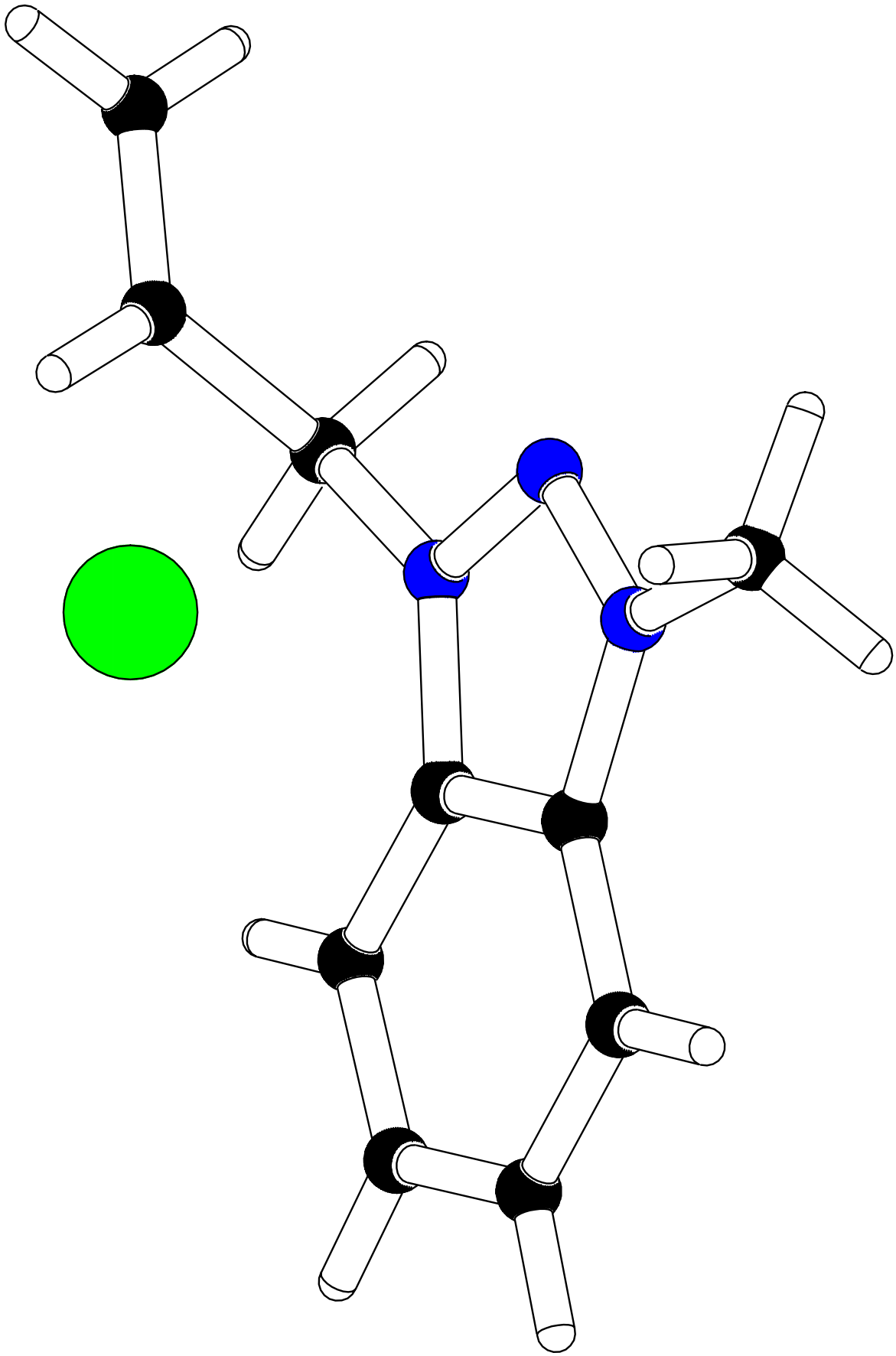
15 10 5 0

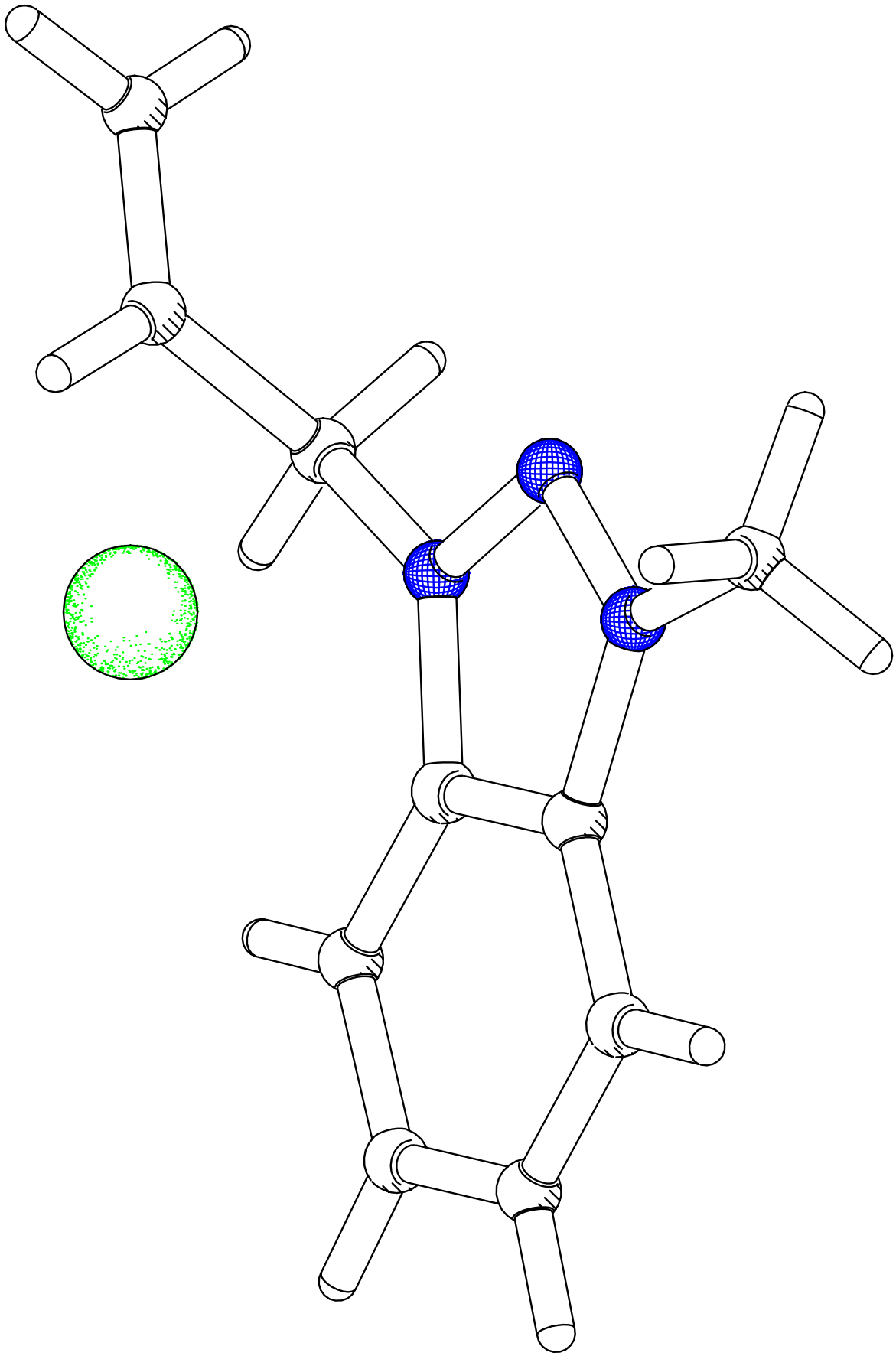
8 7 6 5 4 3 [ppm]

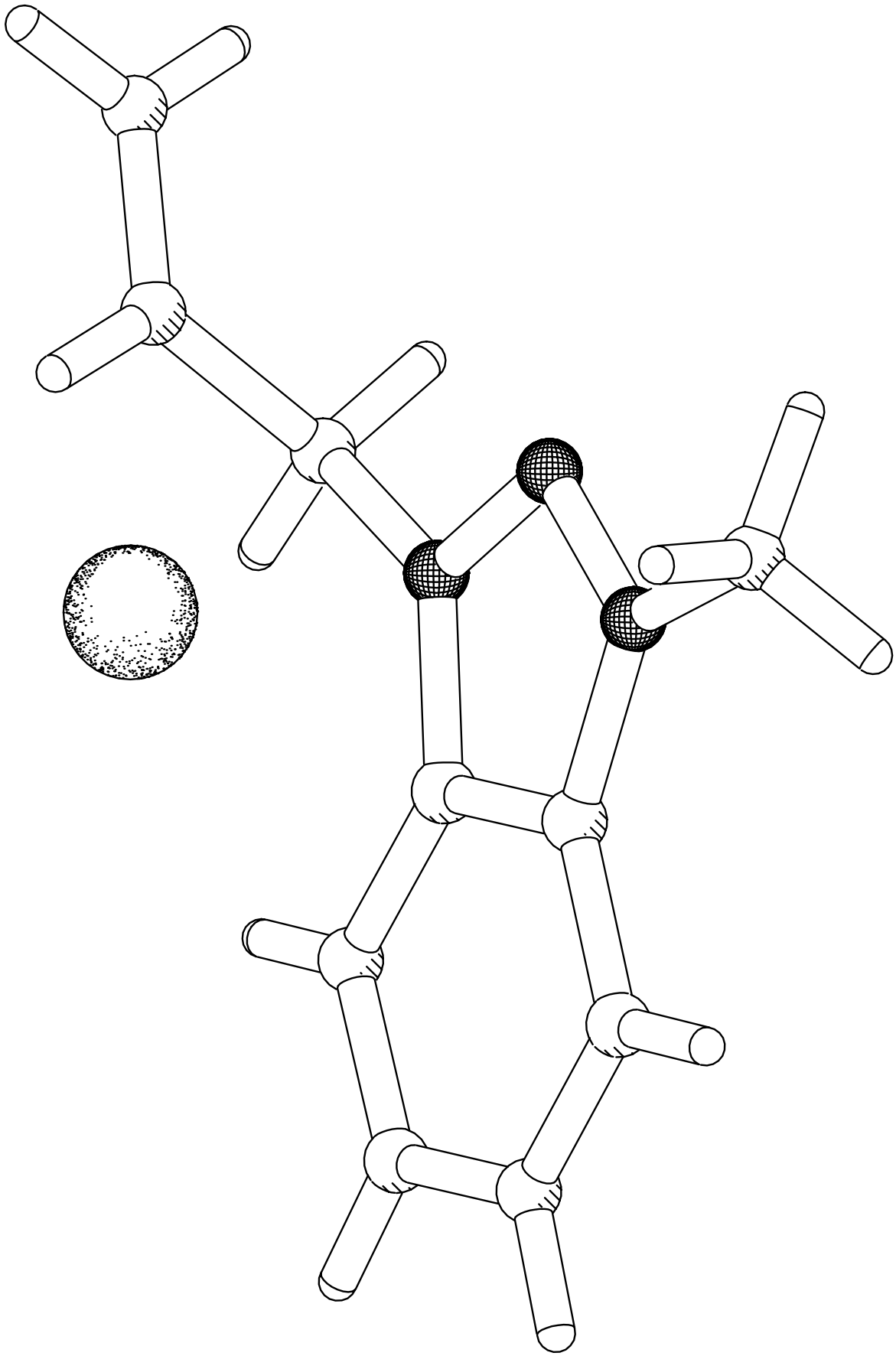

I1
C8
C9
C7
N3
C6
C5
C1
C4
N1
N2
C2
C3
C10

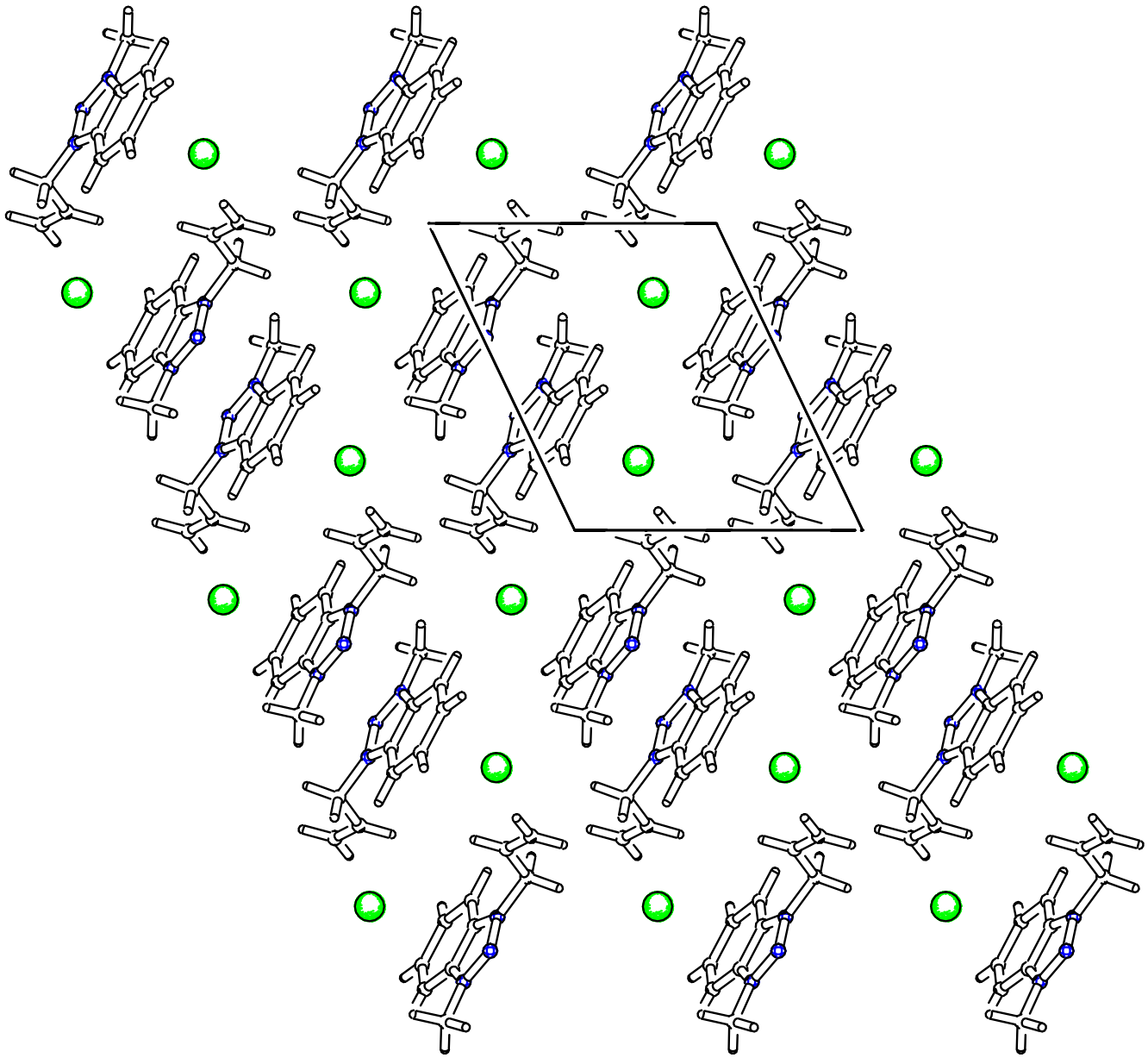


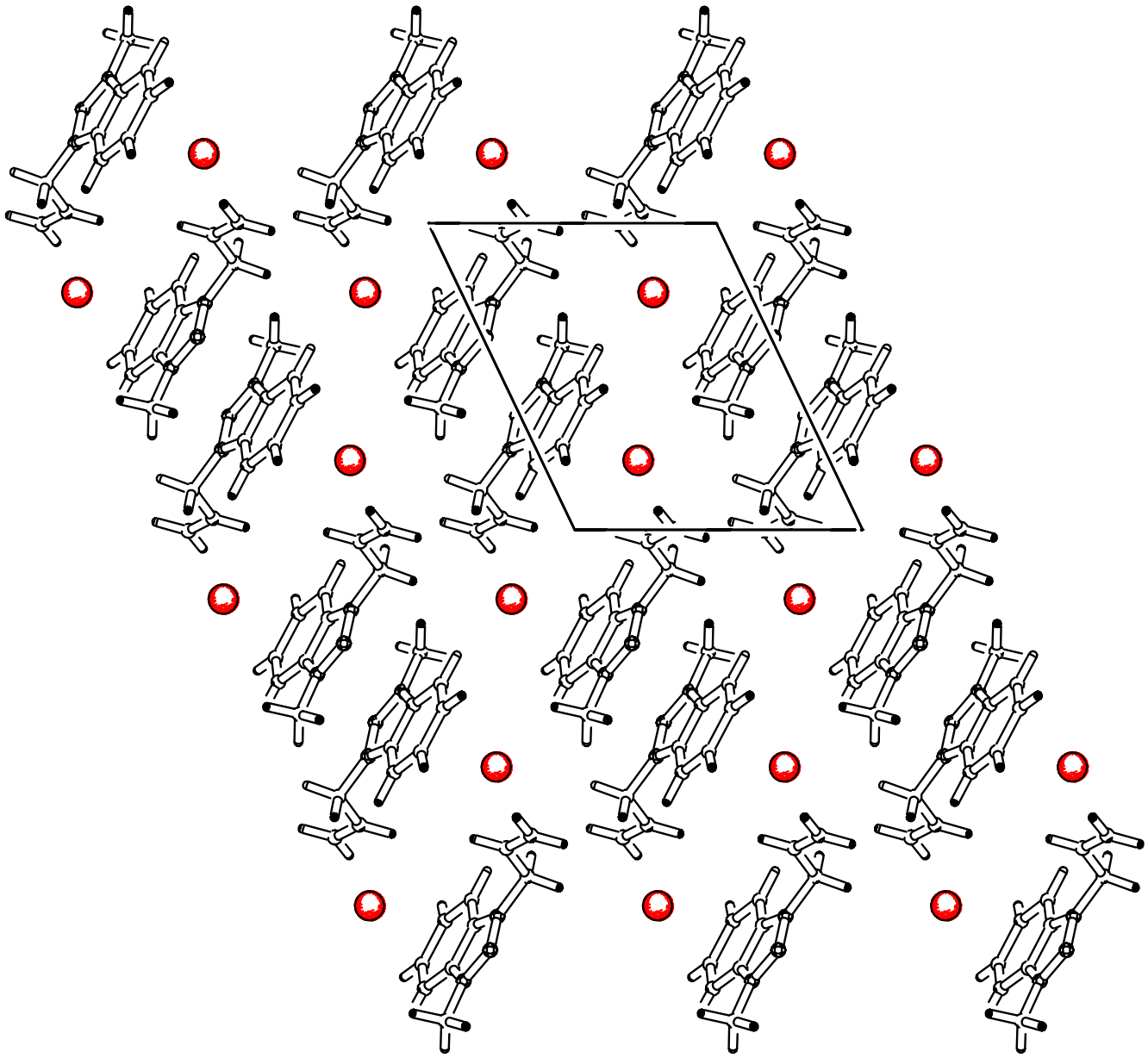












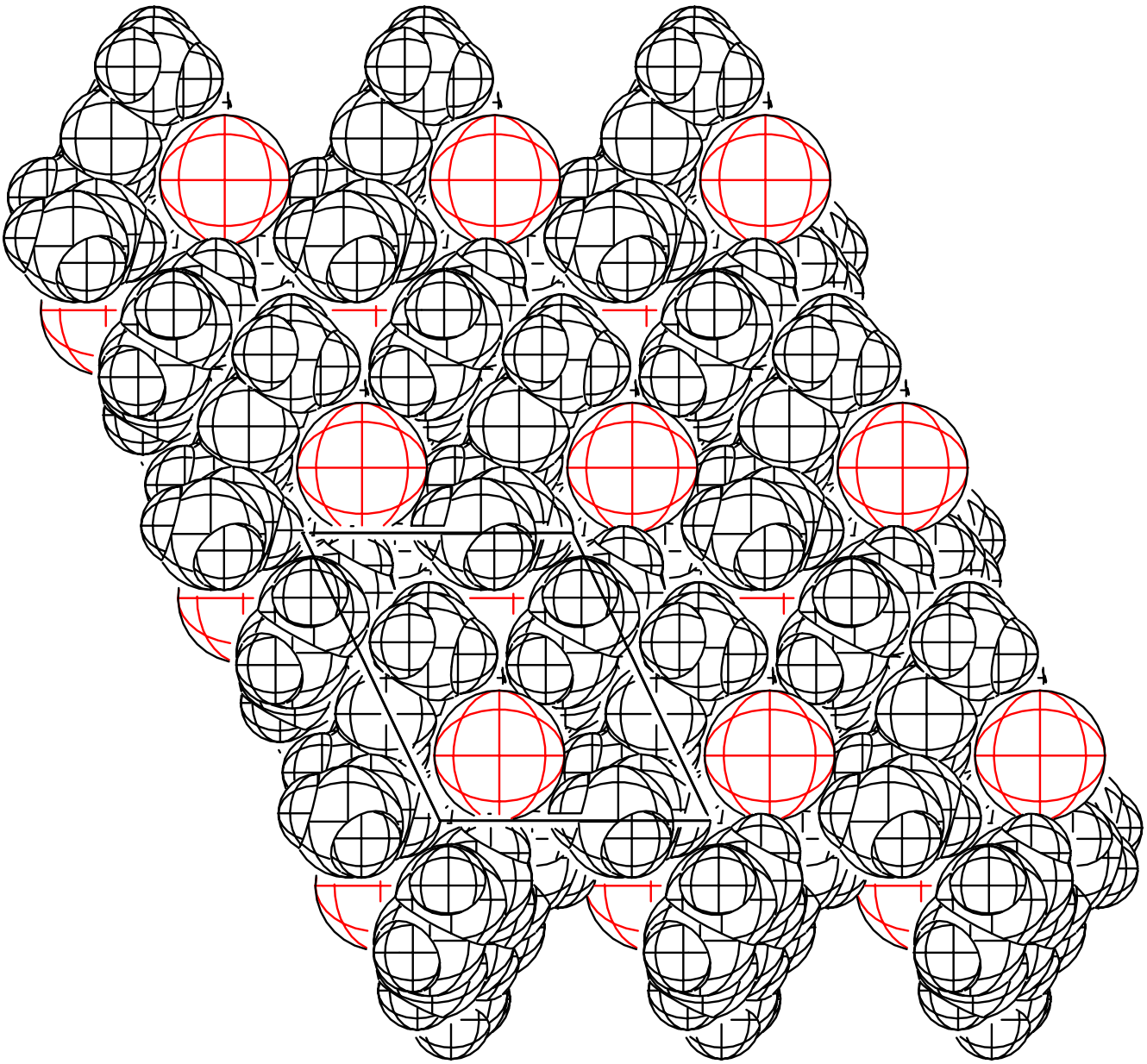


Table 1. Crystal data and structure refinement for c10_a02.

Identification code	taqa
Empirical formula	C10 H12 I N3
Formula weight	301.13
Temperature	100(2) K
Wavelength	.71073 A
Crystal system	Triclinic
Space group	P -1
Unit cell dimensions	a = 7.8839(12) A alpha = 114.093(2) deg. b = 8.2265(14) A beta = 104.033(15) deg. c = 9.9957(17) A gamma = 92.201(13) deg.
Volume	567.20(16) A^3
Z	2
Density (calculated)	1.763 Mg/m^3
Absorption coefficient	2.790 mm^-1

F(000)	292
Crystal size	.39 x .04 x .01 mm
Theta range for data collection	2.33 to 28.31 deg.
Index ranges	-10<=h<=10, -10<=k<=10, -13<=l<=13
Reflections collected	7816
Independent reflections	2798 [R(int) = 0.0894]
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.972 and 0.409
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2798 / 0 / 128
Goodness-of-fit on F^2	0.955
Final R indices [I>2sigma(I)]	R1 = 0.0349, wR2 = 0.0641
R indices (all data)	R1 = 0.0399, wR2 = 0.0661
Largest diff. peak and hole	1.581 and -1.354 e.A^-3

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (^2 x 10^3) for c10_a02. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
N(1)	7209(4)	1330(4)	4718(3)	16(1)
N(2)	7597(4)	-107(4)	3708(3)	18(1)
N(3)	6104(4)	-914(4)	2612(3)	15(1)
C(1)	4725(5)	22(5)	2899(4)	14(1)
C(2)	5465(5)	1512(5)	4296(4)	15(1)
C(3)	4457(5)	2804(5)	4962(4)	19(1)
C(4)	2706(5)	2495(5)	4171(4)	21(1)
C(5)	1956(5)	978(5)	2754(4)	20(1)
C(6)	2947(5)	-288(5)	2084(4)	17(1)
C(7)	6077(5)	-2619(5)	1295(4)	18(1)
C(8)	7214(5)	-2358(5)	381(4)	21(1)
C(9)	8387(5)	-3421(6)	-34(5)	27(1)
C(10)	8626(5)	2617(5)	6012(4)	22(1)
I(1)	7737(1)	3349(1)	2263(1)	15(1)

Table 3. Bond lengths [A] and angles [deg] for c10_a02.

N(1)-N(2)	1.309(4)
N(1)-C(2)	1.370(5)
N(1)-C(10)	1.460(4)
N(2)-N(3)	1.322(4)
N(3)-C(1)	1.376(4)
N(3)-C(7)	1.476(4)
C(1)-C(2)	1.394(5)
C(1)-C(6)	1.394(5)
C(2)-C(3)	1.396(5)
C(3)-C(4)	1.370(5)
C(3)-H(3)	.9500
C(4)-C(5)	1.417(5)
C(4)-H(4)	.9500
C(5)-C(6)	1.375(5)
C(5)-H(5)	.9500
C(6)-H(6)	.9500
C(7)-C(8)	1.492(5)
C(7)-H(7A)	.9900
C(7)-H(7B)	.9900
C(8)-C(9)	1.321(5)
C(8)-H(8)	.9500
C(9)-H(9A)	.9500
C(9)-H(9B)	.9500
C(10)-H(10A)	.9800
C(10)-H(10B)	.9800

C(10)-H(10C)	.9800
N(2)-N(1)-C(2)	112.4(3)
N(2)-N(1)-C(10)	119.4(3)
C(2)-N(1)-C(10)	127.7(3)
N(1)-N(2)-N(3)	105.9(3)
N(2)-N(3)-C(1)	111.9(3)
N(2)-N(3)-C(7)	119.8(3)
C(1)-N(3)-C(7)	128.3(3)
N(3)-C(1)-C(2)	104.8(3)
N(3)-C(1)-C(6)	132.4(3)
C(2)-C(1)-C(6)	122.8(3)
N(1)-C(2)-C(1)	105.0(3)
N(1)-C(2)-C(3)	133.4(4)
C(1)-C(2)-C(3)	121.6(4)
C(4)-C(3)-C(2)	115.8(4)
C(4)-C(3)-H(3)	122.1
C(2)-C(3)-H(3)	122.1
C(3)-C(4)-C(5)	122.5(4)
C(3)-C(4)-H(4)	118.7
C(5)-C(4)-H(4)	118.7
C(6)-C(5)-C(4)	121.9(4)
C(6)-C(5)-H(5)	119.1
C(4)-C(5)-H(5)	119.1
C(5)-C(6)-C(1)	115.4(3)
C(5)-C(6)-H(6)	122.3
C(1)-C(6)-H(6)	122.3
N(3)-C(7)-C(8)	111.4(3)
N(3)-C(7)-H(7A)	109.3

C(8)-C(7)-H(7A)	109.3
N(3)-C(7)-H(7B)	109.3
C(8)-C(7)-H(7B)	109.3
H(7A)-C(7)-H(7B)	108.0
C(9)-C(8)-C(7)	122.1(4)
C(9)-C(8)-H(8)	118.9
C(7)-C(8)-H(8)	118.9
C(8)-C(9)-H(9A)	120.0
C(8)-C(9)-H(9B)	120.0
H(9A)-C(9)-H(9B)	120.0
N(1)-C(10)-H(10A)	109.5
N(1)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
N(1)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (^2 x 10^3) for c10_a02.

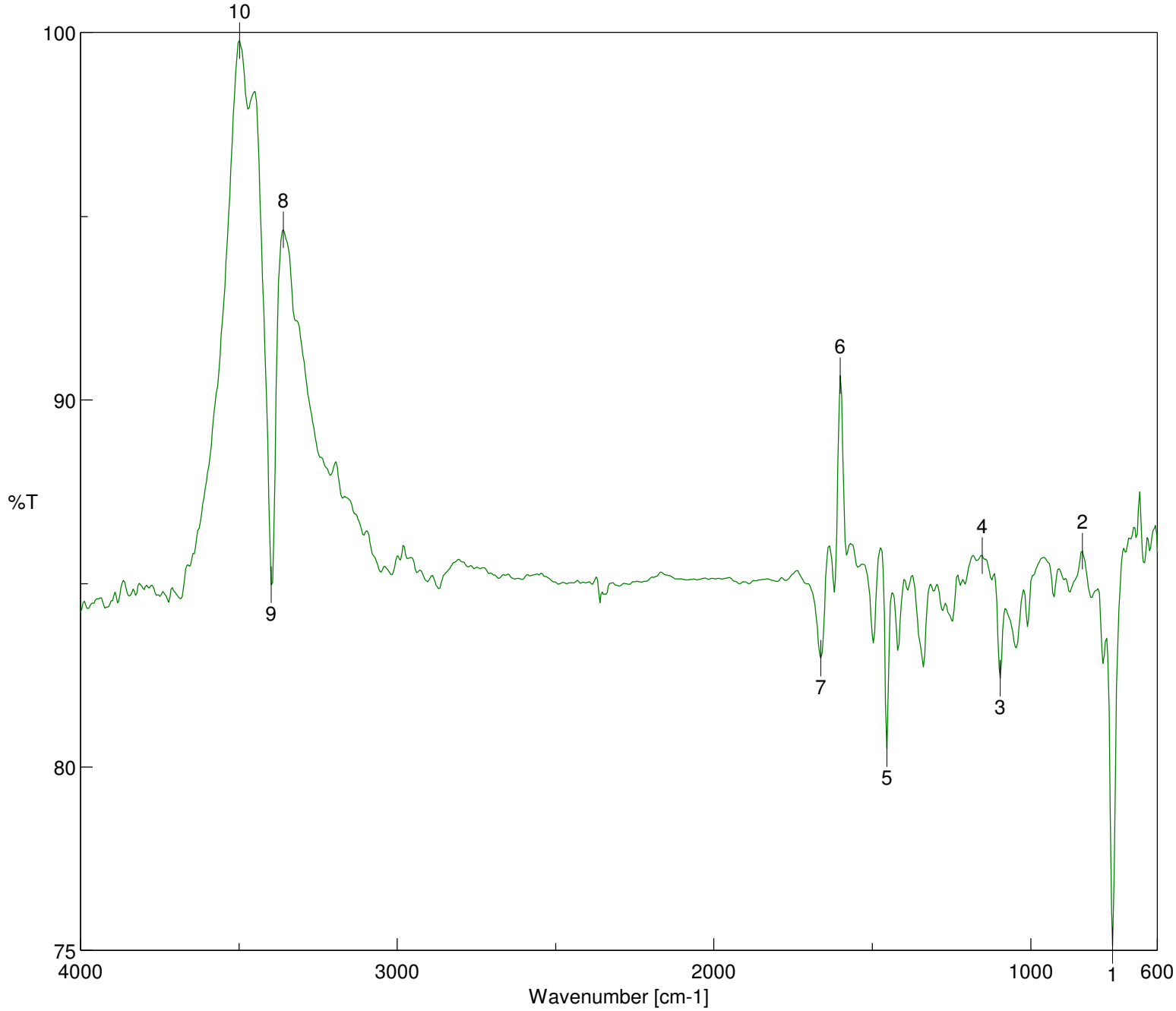
	x	y	z	U(eq)
H(3)	4955	3832	5906	22
H(4)	1965	3331	4589	25
H(5)	733	831	2252	24
H(6)	2454	-1304	1131	20
H(7A)	4846	-3094	634	21
H(7B)	6502	-3519	1665	21
H(8)	7091	-1396	89	25
H(9A)	8527	-4390	250	33
H(9B)	9091	-3218	-615	33
H(10A)	9640	2009	6201	33
H(10B)	8201	3103	6922	33
H(10C)	8987	3604	5781	33

Table 5. Anisotropic displacement parameters (^2 x 10^3) for c10_a02.

The anisotropic displacement factor exponent takes the form:

-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
N(1)	17(2)	15(2)	18(2)	10(1)	1(1)	1(1)
N(2)	20(2)	17(2)	16(2)	9(2)	2(1)	3(1)
N(3)	15(2)	10(2)	17(2)	6(1)	2(1)	2(1)
C(1)	19(2)	13(2)	16(2)	10(2)	8(2)	6(2)
C(2)	14(2)	12(2)	17(2)	8(2)	0(2)	-2(2)
C(3)	27(2)	15(2)	17(2)	8(2)	8(2)	4(2)
C(4)	27(2)	18(2)	24(2)	10(2)	14(2)	10(2)
C(5)	17(2)	22(2)	25(2)	14(2)	5(2)	6(2)
C(6)	19(2)	15(2)	15(2)	7(2)	1(2)	2(2)
C(7)	19(2)	13(2)	19(2)	5(2)	4(2)	4(2)
C(8)	26(2)	14(2)	17(2)	5(2)	3(2)	-1(2)
C(9)	23(2)	31(3)	25(2)	10(2)	6(2)	4(2)
C(10)	18(2)	22(2)	19(2)	6(2)	-1(2)	-2(2)
I(1)	16(1)	13(1)	16(1)	7(1)	3(1)	3(1)



Results of Peak Find

No.	Position	Intensity
1	740.531	75.1263
2	836.955	85.8707
3	1095.37	82.4138
4	1153.22	85.754
5	1454.06	80.4986
6	1600.63	90.645
7	1662.34	82.9609
8	3359.39	94.6179
9	3397.96	84.9524
10	3498.24	99.77

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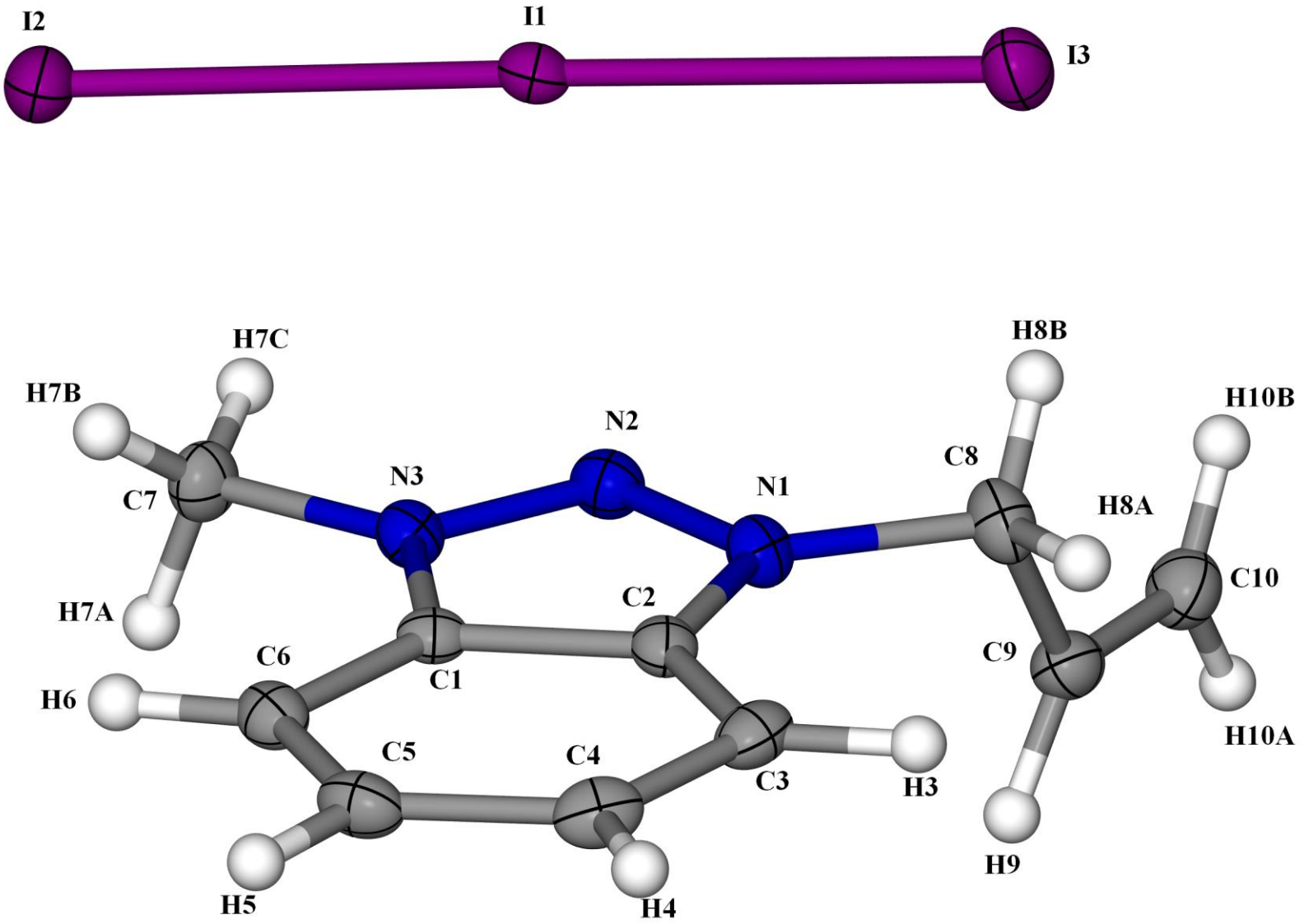
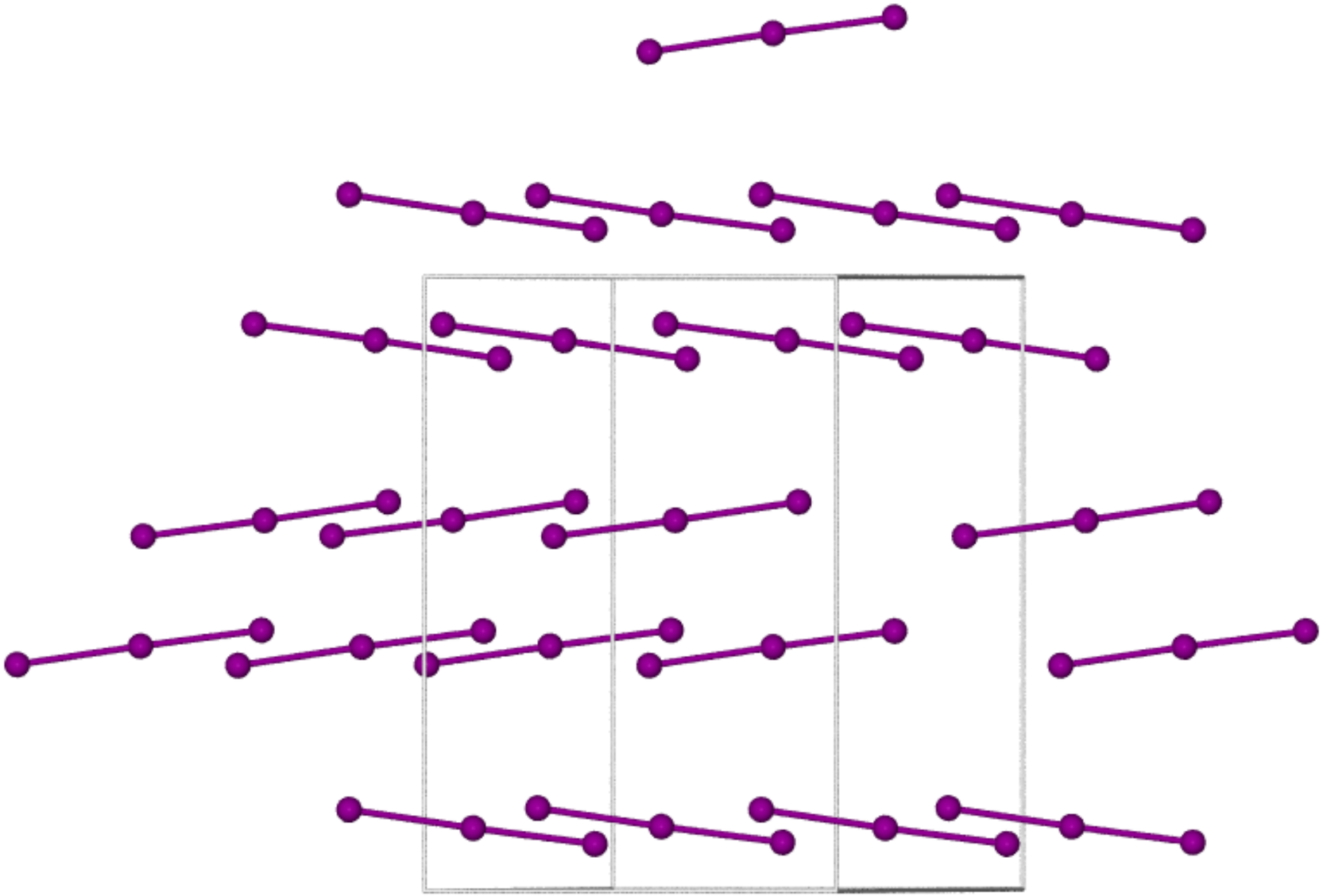
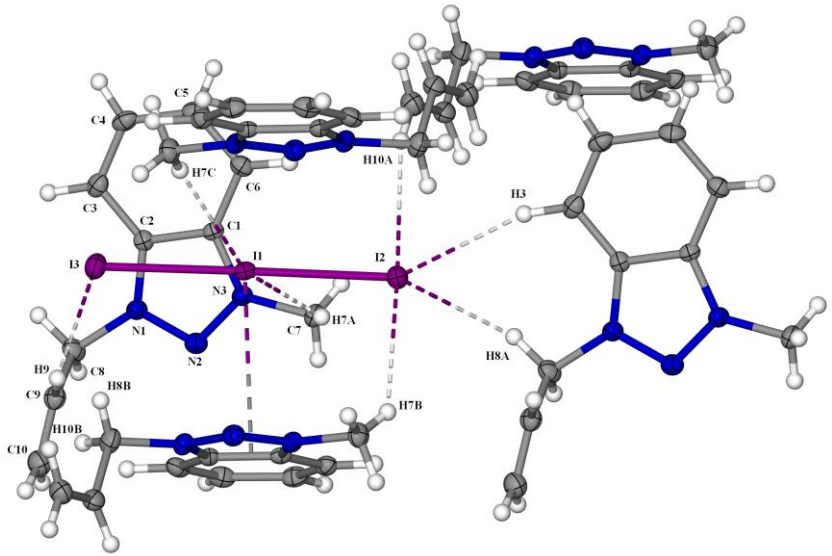
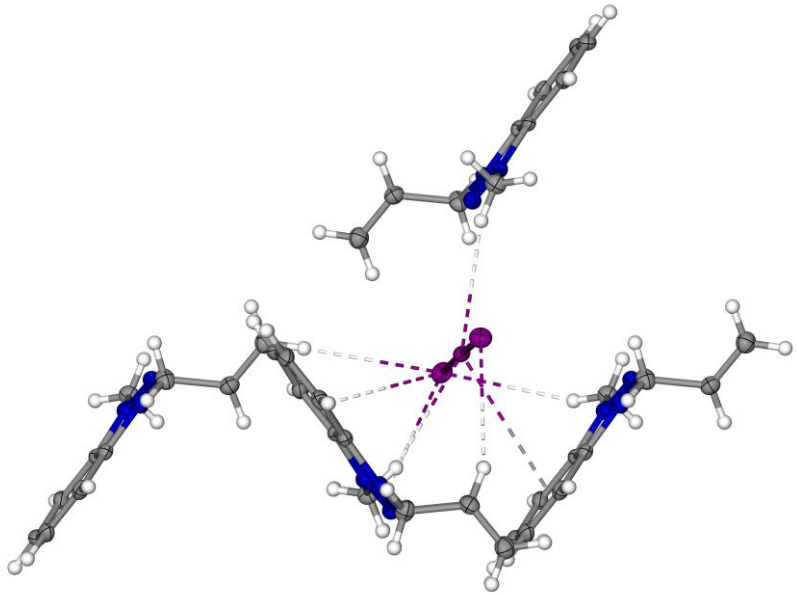


Fig. 1. A perspective drawing of the title compound showing the atom numbering scheme. Displacement ellipsoids are drawn at a 50% probability level and H atoms are shown as spheres of arbitrary radii.

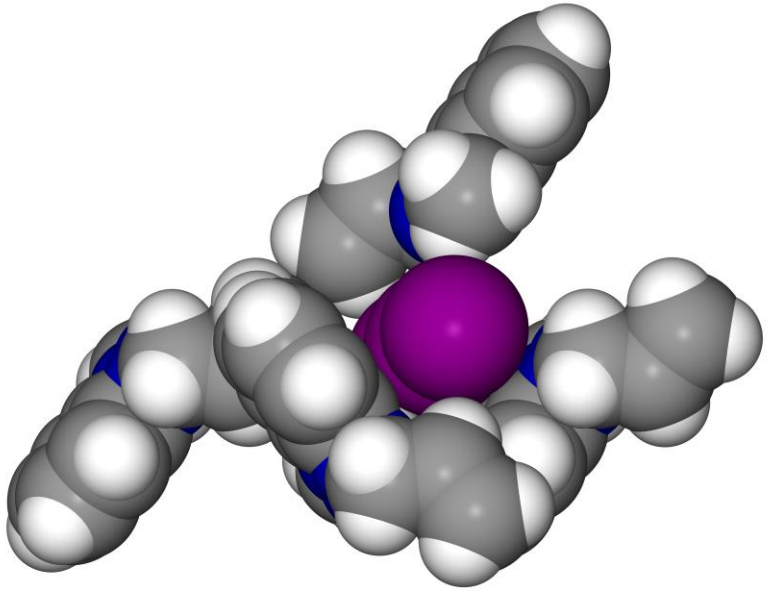




a)

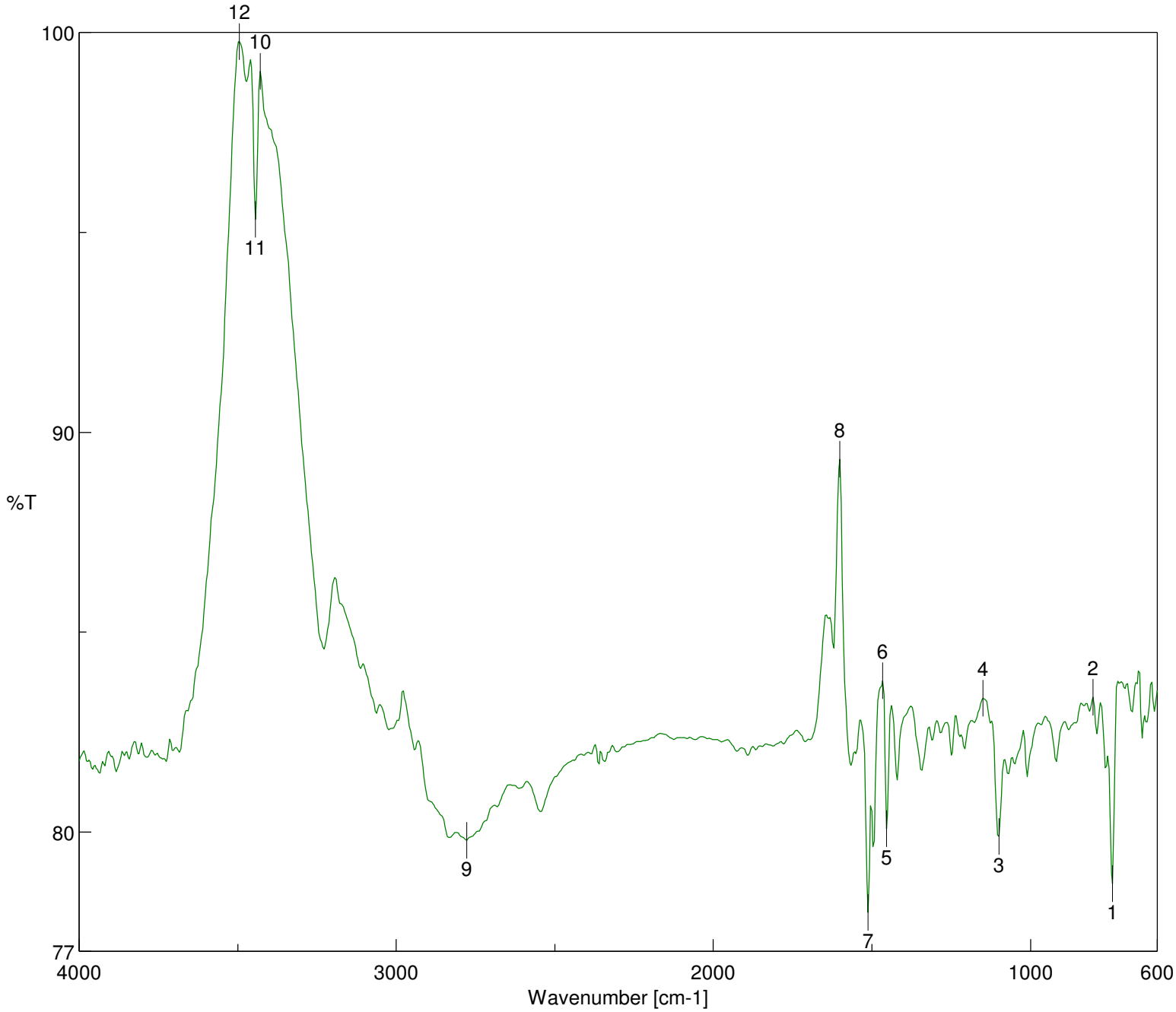


b)



c)

Fig. 2. Different views of a fragment of the crystal structure of the title compound. The linear triodide anion, due to its large size, is ideal for forming numerous weak C-H hydrogen bonds and for interacting with the electropositive phenyl rings of the cations. The overall positive charge of the cation is spread out over the hydrogen atoms. Each triiodide anion is surrounded by five cations. The organic molecules wrap around the I_3^- ions forming a cage-like structure. The distances between the I atoms in the triiodide anion are different (I1-I2, 2.9451(5) Å; I1-I3, 2.8822(5) Å) with the terminal I atom of the longer bond (I2) accepting more hydrogen bonds from the cations. Displacement ellipsoids (2a and 2b) are drawn at a 50% probability level and H atoms are shown as spheres of arbitrary radii while 2c is a space filling plot with atoms shown as spheres with van der Waals radii.



Results of Peak Find

No.	Position	Intensity
1	740.531	78.6877
2	802.242	83.3579
3	1099.23	79.8786
4	1149.37	83.3375
5	1454.06	80.0737
6	1465.63	83.7676
7	1511.92	77.9766
8	1600.63	89.3167
9	2776.99	79.7822
10	3428.81	99.0248
11	3444.24	95.3224
12	3494.38	99.77

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