

***SUPPLEMENTARY MATERIAL FOR  
DEPOSITION AND REFEREES***

## MATERIAL FOR DEPOSIT

Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^{-3}$ ) for the 100K neutron structure of DCNP.

Atom	U11	U22	U33	U23	U13	U12
N(1)	34(1)	21(1)	27(1)	3(1)	-12(1)	4(1)
N(2)	32(1)	19(1)	33(1)	-9(1)	-4(1)	-3(1)
N(3)	24(1)	12(1)	14(1)	-2(1)	-2(1)	0(1)
N(4)	27(1)	16(1)	16(1)	-1(1)	0(1)	0(1)
C(1)	18(1)	17(1)	18(2)	0(1)	-3(1)	1(1)
C(2)	17(2)	15(2)	25(2)	-4(1)	-1(1)	-6(1)
C(3)	13(1)	12(1)	15(1)	-2(1)	-1(1)	-1(1)
C(4)	17(1)	11(1)	16(1)	-2(1)	1(1)	3(1)
C(5)	15(1)	9(1)	17(1)	-1(1)	0(1)	-1(1)
C(6)	23(2)	15(1)	19(2)	1(1)	3(1)	-1(1)
C(7)	23(2)	15(1)	16(1)	1(1)	2(1)	-1(1)
C(8)	14(2)	23(2)	19(2)	-2(1)	1(1)	0(1)
C(9)	20(2)	28(2)	16(1)	-1(1)	2(1)	-9(1)
C(10)	25(2)	47(2)	19(2)	-9(2)	4(1)	-19(2)
C(11)	15(2)	71(3)	18(2)	1(2)	-3(1)	-10(2)
C(12)	14(2)	64(3)	21(2)	9(2)	2(1)	2(2)
C(13)	15(2)	38(2)	18(2)	10(2)	1(1)	5(1)
H(4)	51(4)	17(3)	34(3)	-10(3)	-8(3)	6(3)
H(6A)	44(4)	44(4)	42(4)	13(3)	7(3)	-20(3)

H(6B)	37(3)	30(3)	43(4)	-9(3)	0(3)	13(3)
H(7A)	41(4)	33(3)	52(4)	6(3)	-15(3)	-17(3)
H(7B)	55(4)	43(4)	27(3)	-1(3)	21(3)	18(3)
H(9)	47(4)	27(3)	36(4)	3(3)	4(3)	-6(3)
H(10)	67(5)	67(5)	37(4)	-19(4)	9(4)	-29(4)
H(11)	37(5)	123(9)	38(4)	-7(5)	-15(4)	-22(4)
H(12)	44(5)	89(6)	38(5)	21(5)	1(4)	15(4)
H(13)	55(5)	37(4)	48(5)	6(4)	-4(4)	20(4)

*Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for the 90K X-ray structure of DCNP.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	35(1)	26(1)	32(1)	0(1)	-9(1)	-1(1)
N(2)	35(1)	27(1)	32(1)	-3(1)	-8(1)	-2(1)
N(3)	27(1)	23(1)	24(1)	0(1)	-1(1)	1(1)
N(4)	33(1)	22(1)	21(1)	-1(1)	-3(1)	0(1)
C(1)	25(1)	22(1)	28(1)	-2(1)	-2(1)	-2(1)
C(2)	21(1)	22(1)	24(1)	1(1)	0(1)	0(1)
C(3)	21(1)	20(1)	23(1)	-2(1)	0(1)	-3(1)
C(4)	24(1)	18(1)	27(1)	-1(1)	0(1)	0(1)
C(5)	22(1)	21(1)	21(1)	0(1)	1(1)	-1(1)
C(6)	30(1)	19(1)	25(1)	3(1)	1(1)	1(1)
C(7)	32(1)	19(1)	23(1)	1(1)	1(1)	-1(1)

C(8)	19(1)	32(1)	21(1)	1(1)	0(1)	-1(1)
C(9)	28(1)	36(1)	24(1)	-1(1)	1(1)	-7(1)
C(10)	29(1)	50(1)	24(1)	-8(1)	1(1)	-15(1)
C(11)	24(1)	69(2)	24(1)	-6(1)	-2(1)	-12(1)
C(12)	21(1)	61(2)	27(1)	8(1)	-2(1)	6(1)
C(13)	23(1)	39(1)	25(1)	8(1)	0(1)	6(1)

*Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for the 100K X-ray structure of DCNP.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	36(1)	26(1)	32(1)	0(1)	-10(1)	0(1)
N(2)	36(1)	27(1)	31(1)	-4(1)	-6(1)	-3(1)
N(3)	29(1)	20(1)	22(1)	1(1)	0(1)	0(1)
N(4)	33(1)	20(1)	21(1)	-1(1)	-2(1)	0(1)
C(1)	25(1)	22(1)	26(1)	-3(1)	-2(1)	-2(1)
C(2)	21(1)	21(1)	22(1)	1(1)	-1(1)	-1(1)
C(3)	19(1)	19(1)	22(1)	0(1)	0(1)	-1(1)
C(4)	24(1)	15(1)	24(1)	-1(1)	1(1)	-1(1)
C(5)	22(1)	16(1)	21(1)	-1(1)	3(1)	-1(1)
C(6)	31(1)	16(1)	24(1)	3(1)	2(1)	1(1)
C(7)	32(1)	16(1)	22(1)	1(1)	2(1)	0(1)
C(8)	19(1)	29(1)	18(1)	0(1)	1(1)	0(1)
C(9)	27(1)	33(1)	23(1)	-2(1)	3(1)	-6(1)

C(10)	28(1)	49(1)	24(1)	-10(1)	3(1)	-16(1)
C(11)	23(1)	73(1)	22(1)	-6(1)	-1(1)	-12(1)
C(12)	23(1)	61(1)	27(1)	9(1)	0(1)	5(1)
C(13)	24(1)	38(1)	24(1)	8(1)	1(1)	6(1)

*Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for the 200K X-ray structure of DCNP.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	75(1)	50(1)	63(1)	3(1)	-20(1)	2(1)
N(2)	72(1)	49(1)	61(1)	-10(1)	-12(1)	-4(1)
N(3)	55(1)	34(1)	41(1)	1(1)	-1(1)	0(1)
N(4)	62(1)	36(1)	41(1)	0(1)	-2(1)	0(1)
C(1)	48(1)	40(1)	52(1)	-6(1)	-5(1)	-1(1)
C(2)	42(1)	40(1)	43(1)	1(1)	-1(1)	-2(1)
C(3)	41(1)	37(1)	40(1)	0(1)	3(1)	-1(1)
C(4)	45(1)	32(1)	47(1)	-1(1)	2(1)	-1(1)
C(5)	44(1)	32(1)	39(1)	-2(1)	4(1)	0(1)
C(6)	57(1)	36(1)	48(1)	3(1)	2(1)	1(1)
C(7)	57(1)	34(1)	46(1)	-1(1)	4(1)	-4(1)
C(8)	38(1)	53(1)	36(1)	-1(1)	3(1)	1(1)
C(9)	52(1)	59(2)	44(1)	-5(1)	6(1)	-9(1)
C(10)	59(2)	83(2)	50(1)	-19(1)	5(1)	-28(1)
C(11)	45(2)	126(3)	41(2)	-5(2)	-2(1)	-19(1)

C(12)	45(2)	106(2)	46(2)	12(2)	-3(1)	2(1)
C(13)	49(1)	68(2)	46(1)	11(1)	4(1)	8(1)

*Anisotropic displacement parameters ( $\text{\AA}^2 \times 10^3$ ) for the 290K X-ray structure of DCNP.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	108(2)	72(2)	91(3)	4(2)	-27(2)	4(2)
N(2)	105(3)	69(2)	92(3)	-12(2)	-16(2)	-3(2)
N(3)	76(2)	51(1)	59(2)	1(1)	-4(2)	1(1)
N(4)	90(2)	53(2)	61(2)	1(2)	-1(2)	-2(2)
C(1)	73(2)	61(2)	71(3)	0(2)	-10(2)	-1(2)
C(2)	64(2)	58(2)	66(2)	-1(2)	0(2)	-4(2)
C(3)	57(2)	51(2)	65(2)	-1(2)	3(2)	-5(1)
C(4)	67(2)	43(2)	66(2)	-2(2)	2(2)	-3(2)
C(5)	63(2)	46(2)	56(2)	0(2)	6(2)	-3(2)
C(6)	84(3)	50(2)	69(3)	4(2)	5(2)	-3(2)
C(7)	81(3)	46(2)	65(2)	1(2)	6(2)	0(2)
C(8)	58(2)	71(2)	54(2)	2(2)	4(2)	2(2)
C(9)	75(3)	82(3)	64(2)	-4(2)	5(2)	-13(2)
C(10)	87(3)	120(4)	69(3)	-23(3)	6(2)	-39(3)
C(11)	71(3)	179(6)	61(3)	-7(4)	-3(2)	-27(3)
C(12)	69(3)	142(4)	67(3)	15(3)	-8(3)	6(3)
C(13)	70(3)	93(3)	71(3)	13(3)	3(2)	8(2)

*The overall libration in the structural refinements.*

The Type of Analysis	Libration (degrees <sup>2</sup> )	Root mean square of libration
100K neutron	3.735 <sup>&amp;</sup>	1.933 <sup>&amp;</sup>
90K X-ray	1.806	1.344
100K X-ray	2.257	1.502
200K X-ray	3.781	1.944
290K X-ray	5.534	2.352

*The librational coefficients calculated in the given analyses.*

Analysis	L11 (deg <sup>2</sup> )	L12 (deg <sup>2</sup> )	L13 (deg <sup>2</sup> )	L22 (deg <sup>2</sup> )	L23 (deg <sup>2</sup> )	L33 (deg <sup>2</sup> )
100K neutron	5(7) <sup>&amp;</sup>	0.6(4) <sup>&amp;</sup>	5(1) <sup>&amp;</sup>	-0.2(4) <sup>&amp;</sup>	0.0(4) <sup>&amp;</sup>	16(2) <sup>&amp;</sup>
90K X-ray	3.0(6)	0.1(3)	3.2(8)	-0.1(3)	0.2(3)	9(2)
100K X-ray	3.3(6)	0.2(3)	3.0(8)	-0.1(3)	0.3(3)	10(2)
200K X-ray	7(1)	0.4(5)	7(2)	0.3(5)	0.5(5)	19(3)
290K X-ray	10(1)	0.3(7)	10(2)	0.6(7)	0.8(7)	27(4)

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<sup>&</sup> The values obtained from the 100K neutron analysis differ from those obtained from the 100K X-ray analysis. This is expected since the two different techniques determined anisotropic displacement parameters in a different manner (Blessing, 1995). Suitable scaling methods exist in order to minimize this difference (Blessing, 1995). However, scaling was unsuitable in this case since the original neutron derived parameters were necessary in order to determine a riding correction (see later) for the X-H distances.

*Translational coefficients for the given analysis*

Analysis	T11 (Å <sup>2</sup> )	T12 (Å <sup>2</sup> )	T13 (Å <sup>2</sup> )	T22 (Å <sup>2</sup> )	T23 (Å <sup>2</sup> )	T33 (Å <sup>2</sup> )
100K neutron	0.015(2)&	0.000(1)&	0.000(1)&	0.013(1)&	- 0.001(1)&	0.018(1)&
90K X-ray	0.022(1)	-0.001(1)	-0.001(1)	0.021(1)	0.000(1)	0.023(1)
100K X-ray	0.022(1)	-0.001(1)	0.001(1)	0.018(1)	0.000(1)	0.022(1)
200K X-ray	0.042(2)	-0.001(1)	0.002(1)	0.034(2)	-0.001(1)	0.042(1)
290K X-ray	0.062(2)	-0.002(2)	0.002(1)	0.048(2)	0.000(1)	0.061(2)

*Corrections to bond distances involving libration effected non-hydrogen atoms.*

Bond	Libration corrected distance (Å)				
	100K neutron	90K X-ray	100K X-ray	200K X-ray <sup>#</sup>	290K X-ray <sup>#</sup>
C(8)-C(9)	1.393	1.397	1.396	1.400	1.401
C(8)-C(13)	1.406	1.412	1.409	1.411	1.407
C(9)-C(10)	1.389	1.392	1.388	1.392	1.390
C(10)-C(11)	1.397	1.400	1.406	1.393	1.395
C(11)-C(12)	1.397	1.387	1.384	1.377	1.368
C(12)-C(13)	1.388	1.409	1.394	1.401	1.396

<sup>#</sup> A rigid-body correction was also made on all non-hydrogen atoms for these two data sets (see following page) and the values given include this correction.



Average correction to libration	+0.0011	+0.0015	+0.0017	+0.0029	+0.0036
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*THMA11 corrected anisotropic displacement parameters for the 100K neutron refinement.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	0.0346	0.0245	0.0256	0.0022	-0.0093	0.0042
N(2)	0.0326	0.0167	0.0258	-0.0085	-0.0025	-0.0048
N(3)	0.0169	0.0135	0.0174	-0.0017	0.0001	-0.0014
N(4)	0.0163	0.0186	0.0179	0.0008	0.0011	0.0002
C(1)	0.0223	0.0180	0.0211	-0.0005	-0.0042	0.0009
C(2)	0.0178	0.0146	0.0203	-0.0052	0.0004	-0.0033
C(3)	0.0147	0.0128	0.0184	-0.0026	-0.0004	-0.0012
C(4)	0.0178	0.0097	0.0185	-0.0023	-0.0013	-0.0006
C(5)	0.0151	0.0101	0.0178	-0.0018	0.0007	-0.0004
C(6)	0.0303	0.0154	0.0243	0.0030	-0.0023	0.0022
C(7)	0.0265	0.0104	0.0230	-0.0010	-0.0014	0.0013
C(8)	0.0156	0.0289	0.0168	0.0014	0.0016	-0.0017
C(9)	0.0264	0.0320	0.0177	-0.0031	0.0011	-0.0073
C(10)	0.0312	0.0502	0.0195	-0.0070	0.0009	-0.0144
C(11)	0.0176	0.0703	0.0156	-0.0009	0.0025	-0.0102
C(12)	0.0175	0.0629	0.0180	0.0095	0.0025	0.0009

C(13)	0.0204	0.0400	0.0211	0.0080	0.0014	0.0022
H(4)	0.0310	0.0096	0.0221	-0.0025	-0.0060	0.0004
H(6A)	0.0378	0.0207	0.0271	0.0080	-0.0042	-0.0073
H(6B)	0.0427	0.0222	0.0292	0.0023	-0.0056	0.0133
H(7A)	0.0344	0.0121	0.0259	0.0007	-0.0031	-0.0070
H(7B)	0.0322	0.0159	0.0254	-0.0049	-0.0030	0.0099
H(9)	0.0365	0.0252	0.0211	-0.0060	-0.0053	-0.0070
H(10)	0.0509	0.0511	0.0232	-0.0105	-0.0077	-0.0167
H(11)	0.0189	0.0788	0.0148	0.0006	0.0025	-0.0133
H(12)	0.0240	0.0686	0.0212	0.0138	0.0001	0.0033
H(13)	0.0347	0.0369	0.0260	0.0101	-0.0034	0.0070

*THMA11 Corrected Anisotropic Displacement Parameters for the 90K X-ray Refinement.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	0.0340	0.0260	0.0290	0.0017	-0.0081	0.0005
N(2)	0.0343	0.0228	0.0271	-0.0039	-0.0030	-0.0016
N(3)	0.0222	0.0216	0.0238	-0.0001	-0.0014	-0.0016
N(4)	0.0225	0.0241	0.0234	0.0008	-0.0003	-0.0001
C(1)	0.0265	0.0232	0.0261	0.0006	-0.0045	-0.0010
C(2)	0.0248	0.0218	0.0244	-0.0020	-0.0010	-0.0017
C(3)	0.0220	0.0208	0.0241	-0.0004	-0.0016	-0.0016
C(4)	0.0232	0.0195	0.0244	-0.0002	-0.0023	-0.0016

C(5)	0.0219	0.0198	0.0235	-0.0001	-0.0008	-0.0011
C(6)	0.0323	0.0225	0.0260	0.0018	-0.0022	0.0012
C(7)	0.0300	0.0200	0.0255	0.0000	-0.0018	0.0006
C(8)	0.0216	0.0292	0.0231	0.0009	-0.0003	-0.0005
C(9)	0.0293	0.0323	0.0241	-0.0038	0.0007	-0.0057
C(10)	0.0328	0.0500	0.0251	-0.0075	0.0017	-0.0133
C(11)	0.0224	0.0695	0.0225	-0.0021	0.0008	-0.0089
C(12)	0.0230	0.0628	0.0239	0.0074	0.0006	0.0034
C(13)	0.0259	0.0402	0.0257	0.0066	0.0006	0.0043

*THMA11 corrected Anisotropic Displacement Parameters of the 100K X-ray refinement.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	0.0344	0.0263	0.0287	0.0025	-0.0079	0.0015
N(2)	0.0347	0.0217	0.0262	-0.0051	-0.0017	-0.0022
N(3)	0.0221	0.0183	0.0220	-0.0002	-0.0001	-0.0014
N(4)	0.0229	0.0210	0.0215	0.0008	0.0011	0.0003
C(1)	0.0265	0.0223	0.0253	0.0009	-0.0040	-0.0005
C(2)	0.0247	0.0203	0.0233	-0.0027	0.0000	-0.0019
C(3)	0.0218	0.0189	0.0229	-0.0006	-0.0009	-0.0015
C(4)	0.0229	0.0167	0.0231	-0.0002	-0.0014	-0.0014
C(5)	0.0219	0.0166	0.0220	-0.0003	0.0004	-0.0008
C(6)	0.0336	0.0192	0.0246	0.0017	-0.0007	0.0018

C(7)	0.0307	0.0168	0.0241	-0.0003	-0.0004	0.0009
C(8)	0.0219	0.0268	0.0210	0.0009	0.0013	-0.0003
C(9)	0.0288	0.0300	0.0234	-0.0041	0.0026	-0.0059
C(10)	0.0316	0.0497	0.0255	-0.0098	0.0034	-0.0130
C(11)	0.0226	0.0720	0.0206	-0.0035	0.0027	-0.0086
C(12)	0.0237	0.0637	0.0220	0.0087	0.0021	0.0027
C(13)	0.0260	0.0385	0.0247	0.0076	0.0016	0.0040

*THMA11 Corrected Anisotropic Displacement Parameters for the 200K X-ray refinement.*

Atom	U11	U22	U33	U23	U13	U12
N(1)	0.0710	0.0501	0.0566	0.0044	-0.0165	0.0029
N(2)	0.0679	0.0422	0.0514	-0.0096	-0.0055	-0.0031
N(3)	0.0435	0.0351	0.0425	-0.0011	0.0006	-0.0020
N(4)	0.0440	0.0396	0.0416	0.0009	0.0024	-0.0002
C(1)	0.0543	0.0432	0.0494	0.0013	-0.0079	-0.0003
C(2)	0.0487	0.0396	0.0452	-0.0051	-0.0005	-0.0029
C(3)	0.0437	0.0371	0.0442	-0.0014	-0.0013	-0.0020
C(4)	0.0456	0.0330	0.0446	-0.0010	-0.0020	-0.0017
C(5)	0.0425	0.0325	0.0423	-0.0011	0.0014	-0.0013
C(6)	0.0636	0.0368	0.0481	0.0029	-0.0026	0.0012
C(7)	0.0583	0.0327	0.0470	-0.0010	-0.0016	0.0007
C(8)	0.0431	0.0493	0.0408	0.0010	0.0028	-0.0011

C(9)	0.0572	0.0545	0.0464	-0.0080	0.0040	-0.0085
C(10)	0.0651	0.0865	0.0489	-0.0169	0.0056	-0.0216
C(11)	0.0474	0.1226	0.0404	-0.0060	0.0046	-0.0172
C(12)	0.0475	0.1093	0.0426	0.0134	0.0030	0.0022
C(13)	0.0519	0.0681	0.0460	0.0116	0.0028	0.0053

*THMA11 Corrected Anisotropic Displacement Parameters for the 290K X-ray Refinement.*

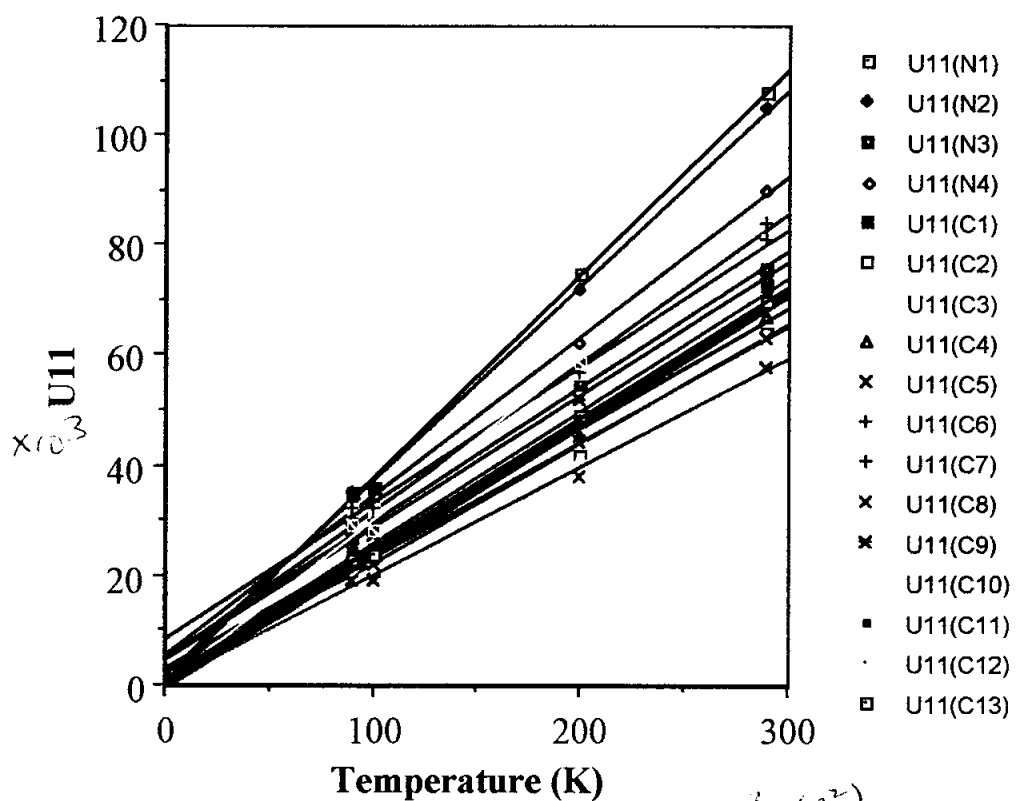
Atom	U11	U22	U33	U23	U13	U12
N(1)	0.1023	0.0731	0.0822	0.0075	-0.0239	0.0043
N(2)	0.0970	0.0615	0.0748	-0.0137	-0.0074	-0.0041
N(3)	0.0637	0.0494	0.0622	-0.0003	0.0003	-0.0035
N(4)	0.0651	0.0556	0.0607	0.0024	0.0032	-0.0006
C(1)	0.0789	0.0627	0.0720	0.0030	-0.0119	-0.0005
C(2)	0.0710	0.0575	0.0660	-0.0069	-0.0009	-0.0042
C(3)	0.0640	0.0536	0.0647	-0.0010	-0.0022	-0.0032
C(4)	0.0663	0.0471	0.0651	-0.0001	-0.0032	-0.0032
C(5)	0.0623	0.0461	0.0618	-0.0004	0.0017	-0.0026
C(6)	0.0923	0.0517	0.0695	0.0051	-0.0029	0.0016
C(7)	0.0844	0.0463	0.0681	-0.0005	-0.0017	0.0003
C(8)	0.0644	0.0693	0.0598	0.0024	0.0033	-0.0012
C(9)	0.0843	0.0767	0.0660	-0.0097	0.0047	-0.0117
C(10)	0.0955	0.1199	0.0700	-0.0211	0.0058	-0.0292

C(11)	0.0722	0.1675	0.0592	-0.0060	0.0046	-0.0226
C(12)	0.0716	0.1495	0.0626	0.0198	0.0028	0.0024
C(13)	0.0759	0.0947	0.0676	0.0172	0.0026	0.0067

*R-factors for all  $U_{ij}$  values and just the diagonal  $U_{ii}$  values for each analysis.*

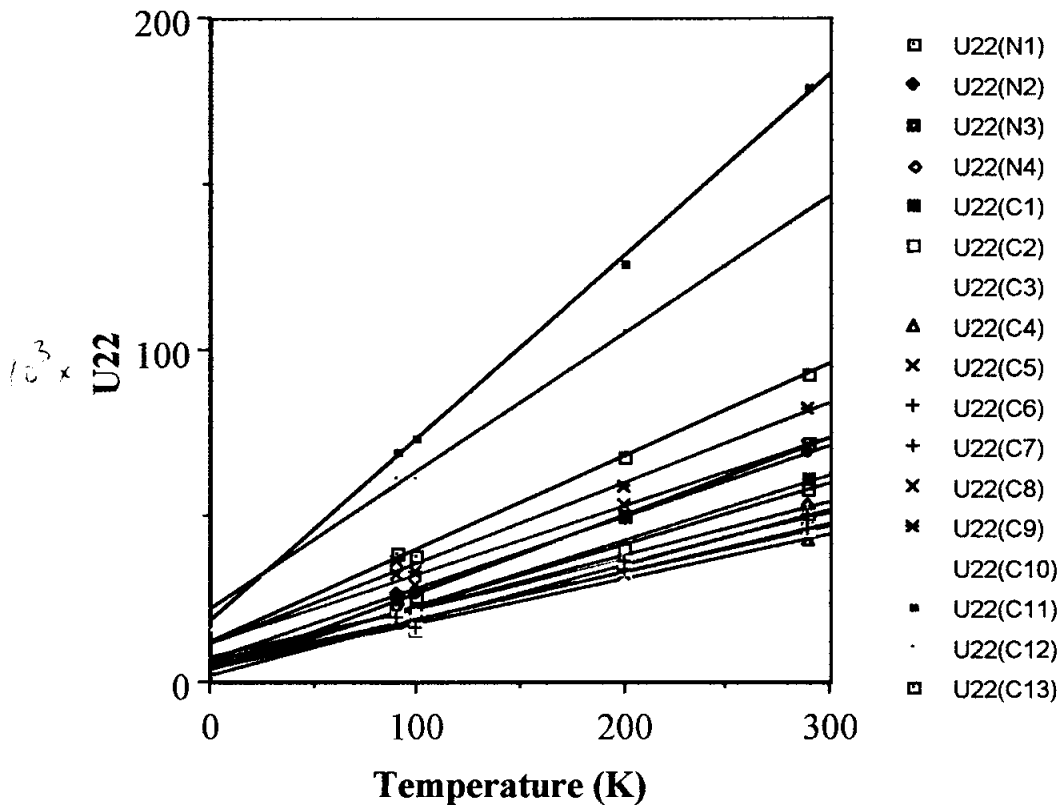
Type of Analysis	R-factor (of all $U_{ij}$ values)	R-factor (of just $U_{ii}$ values)
100K neutron	0.276(5)	0.237(5)
90K X-ray	0.126(2)	0.097(2)
100K X-ray	0.131(3)	0.103(3)
200K X-ray	0.124(4)	0.100(4)
290K X-ray	0.110(6)	0.091(6)

### U11 versus Temperature (DCNP)



Plots of the anisotropic displacement parameter,  $U_{11}$  versus temperature for each non-hydrogen atom in DCNP.

### U22 versus Temperature (DCNP)

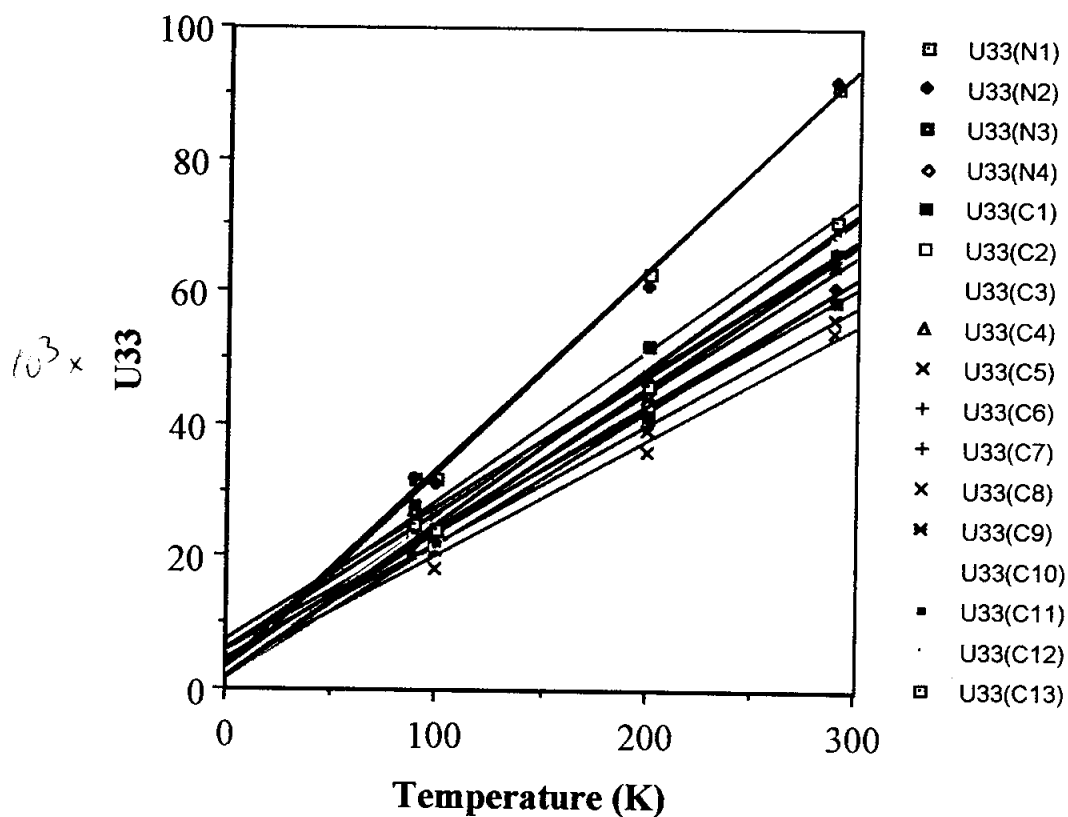


× 10<sup>3</sup> (Å<sup>2</sup>)

Plots of the anisotropic displacement parameter,  $U_{22}$ , versus temperature for each non-hydrogen atom in DCNP.

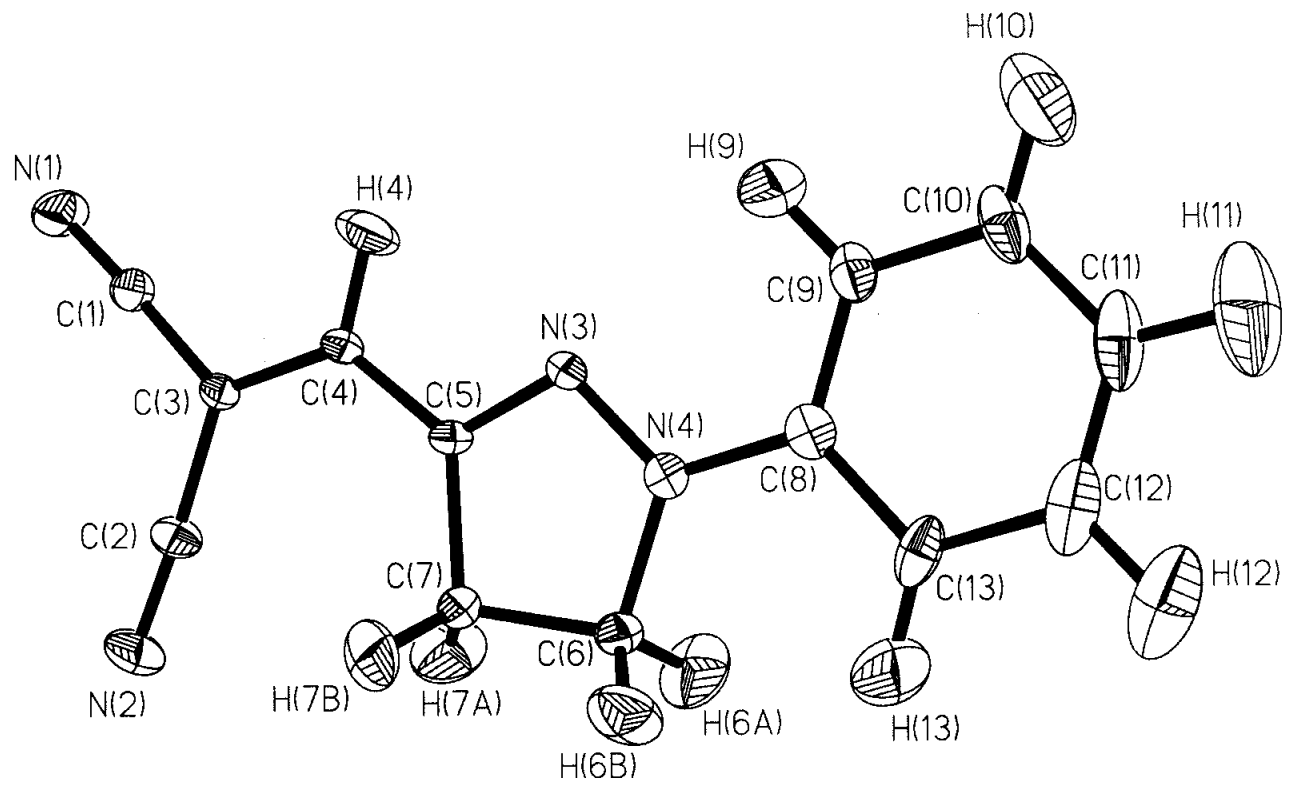


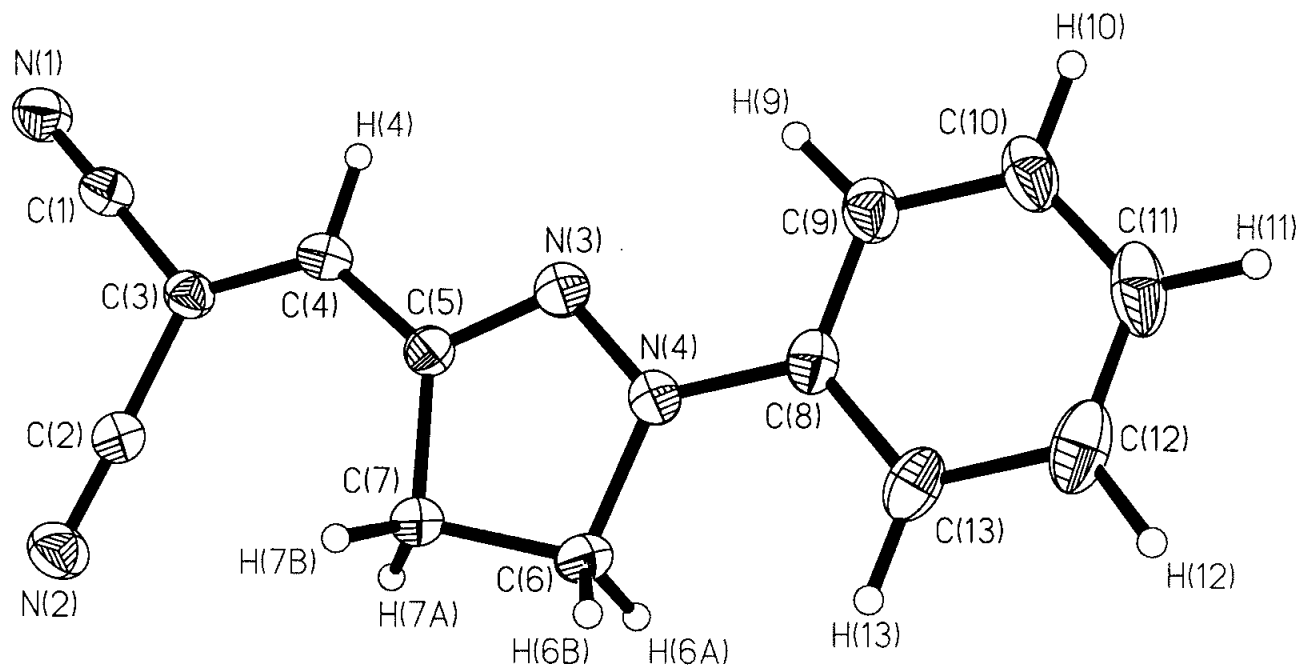
### U33 versus Temperature (DCNP)



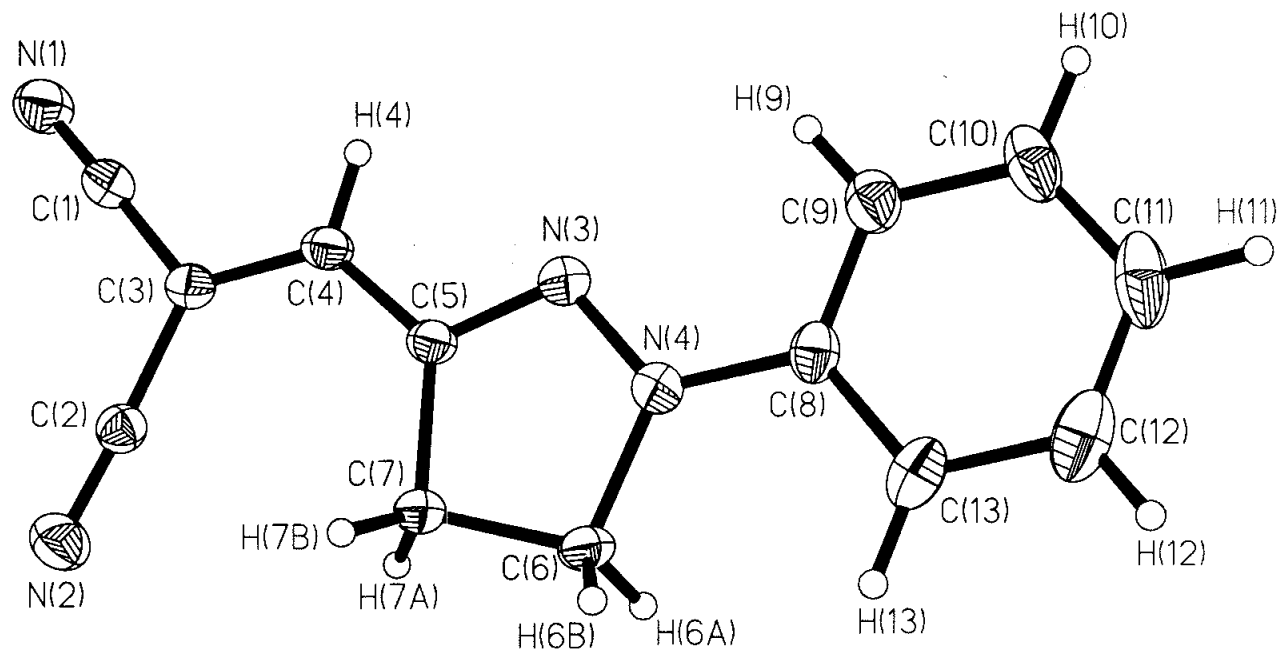
Plots of the anisotropic displacement parameter,  $U_{33} \times 10^3$  (Å<sup>2</sup>) versus temperature for each non-hydrogen atom in DCNP.

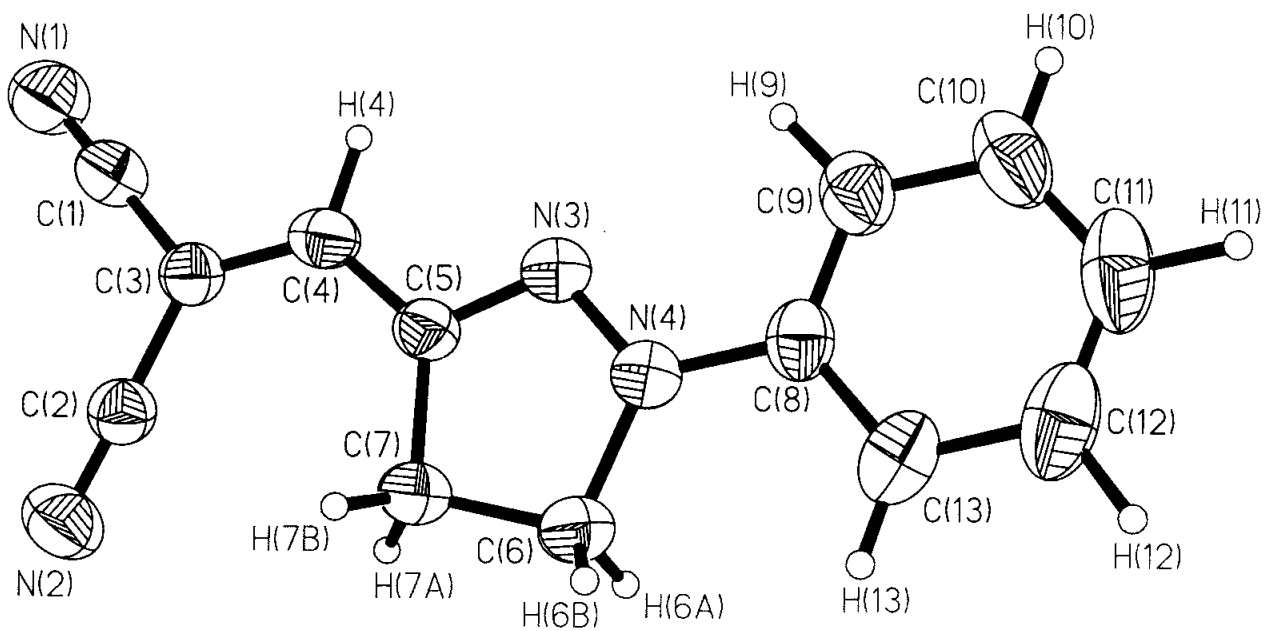
100K NEUTRON STRUCTURE





1051c X-RAY STRUCTURE





290K X-RAY STRUCTURE

