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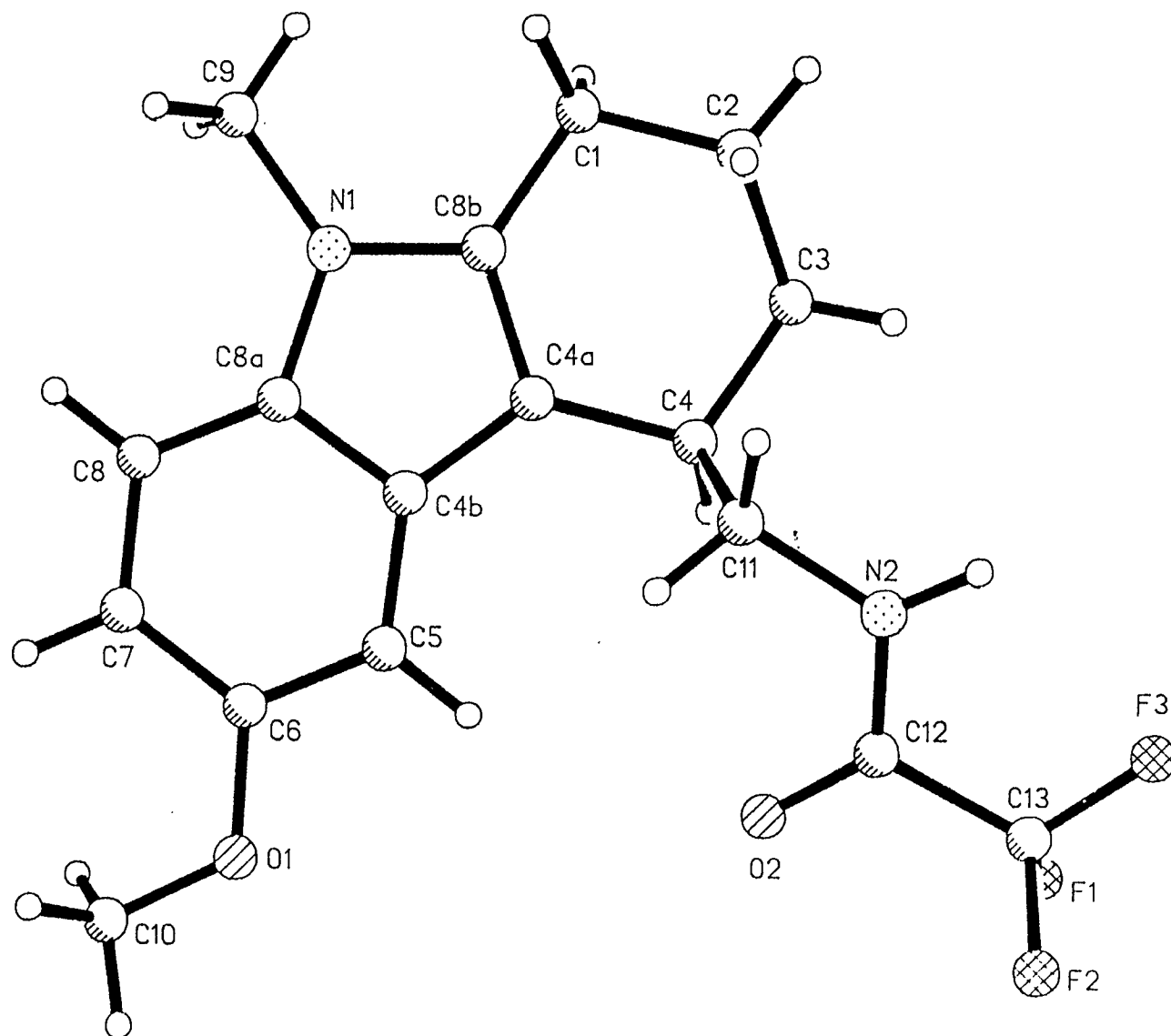
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Experimental

A single crystal of approximate size 0.80x0.32x0.31 mm was mounted on a glass fibre. All geometric and intensity data were taken from this sample at 293 K using an automated four-circle diffractometer (Nicolet R3mV) equipped with Mo-K α radiation ($\lambda = 0.71073$ Å).

The lattice vectors were identified by the application of the automatic indexing routine of the diffractometer to the positions of 30 reflections taken from a rotation photograph and centred by the diffractometer. The ω -2 θ technique was used to measure 3169 reflections (2787 unique) in the range $5^\circ < 2\theta < 50^\circ$. Three standard reflections (remeasured every 97 scans) showed no significant loss in intensity during data collection. The data were corrected for Lorentz and polarisation effects, and empirically for absorption. The 1469 unique data with $I < 3.0\sigma(I)$ were used to solve and refine the structure in the orthorhombic space group Pnaa.

Crystal Data: C₁₇H₁₉N₂O₂F₃, M = 340.38, orthorhombic, space group Pnaa, a = 10.021(1), b = 14.221(3), c = 22.992(4) Å, U = 3276 Å³, Z = 8, D_c = 1.38 g cm⁻³, F(000) = 1424, μ (Mo-K α) = 1.08 cm⁻¹.

The structure was solved by direct methods and developed by using alternating cycles of least-squares refinement and difference-fourier synthesis. The non-hydrogen atoms were refined anisotropically while the hydrogens were placed in idealised positions (C-H = 0.96 Å) and assigned a common isotropic thermal parameter (U = 0.08 Å²). The final cycle of least-squares refinement included 217 parameters for 1469 variables and did not shift any parameter by more than 0.001 times its standard deviation. The final R values were 0.0589 and 0.0586 and the final difference fourier was featureless with no peaks greater than 0.20 e. Å³.

Calculations were performed on a MicroVax II computer equipped with SHELXTL PLUS software.

POOR QUALITY ORIGINAL

	X	Y	Z	U(eq)
F(1)	915(4)	2714(3)	8167(2)	117(2)
F(2)	1448(4)	3859(3)	8686(1)	106(2)
F(3)	2918(4)	3196(3)	8180(1)	110(2)
O(1)	-3224(3)	6376(3)	6835(2)	77(1)
O(2)	149(3)	4360(2)	7572(1)	63(1)
N(1)	679(4)	6283(3)	5165(2)	46(1)
N(2)	2318(3)	4423(2)	7164(2)	43(1)
C(1)	2773(5)	5281(3)	5160(2)	56(2)
C(2)	3587(5)	4783(4)	5632(2)	65(2)
C(3)	2758(5)	4126(3)	6007(2)	58(2)
C(4)	1631(4)	4636(3)	6332(2)	43(2)
C(4A)	996(4)	5357(3)	5941(2)	39(1)
C(4B)	-228(4)	5850(3)	6022(2)	39(1)
C(5)	-1203(5)	5860(3)	6452(2)	47(2)
C(6)	-2291(5)	6428(3)	6084(2)	52(2)
C(7)	-2433(5)	7019(3)	5910(2)	56(2)
C(8)	-1485(5)	7023(3)	5478(2)	52(2)
C(8A)	-403(5)	6430(3)	5528(2)	42(1)
C(8B)	1524(4)	5628(3)	5118(2)	42(2)
C(9)	859(5)	6707(4)	4597(2)	64(2)
C(10)	-4395(5)	6861(4)	6788(3)	85(3)
C(11)	2106(5)	5107(3)	6897(2)	47(2)
C(12)	1307(5)	4131(3)	7605(2)	43(2)
C(13)	1673(6)	3467(4)	8180(2)	63(2)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 2. Bond lengths (Å)

F(1)-C(13)	1.314 (7)	F(2)-C(13)	1.309 (6)
F(3)-C(13)	1.306 (7)	O(1)-C(6)	1.379 (6)
O(1)-C(10)	1.364 (7)	O(2)-C(12)	1.211 (6)
N(1)-C(8A)	1.384 (6)	N(1)-C(8B)	1.387 (6)
N(1)-C(9)	1.449 (6)	N(2)-C(11)	1.464 (5)
N(2)-C(12)	1.320 (6)	C(1)-C(2)	1.532 (7)
C(1)-C(8B)	1.471 (6)	C(2)-C(3)	1.519 (7)
C(3)-C(4)	1.536 (6)	C(4)-C(4A)	1.505 (6)
C(4)-C(11)	1.539 (6)	C(4A)-C(4B)	1.424 (6)
C(4A)-C(8B)	1.369 (6)	C(4B)-C(5)	1.391 (6)
C(4B)-C(8A)	1.413 (6)	C(5)-C(6)	1.363 (7)
C(6)-C(7)	1.405 (7)	C(7)-C(8)	1.375 (7)
C(8)-C(8A)	1.378 (7)	C(12)-C(13)	1.525 (7)

C(6)-O(1)-C(10)	119.9(4)	C(8A)-N(1)-C(8B)	109.1(3)
C(8A)-N(1)-C(9)	125.3(4)	C(8B)-N(1)-C(9)	125.5(4)
C(11)-N(2)-C(12)	120.5(4)	C(2)-C(1)-C(8B)	108.8(4)
C(1)-C(2)-C(3)	113.3(4)	C(2)-C(3)-C(4)	112.8(4)
C(3)-C(4)-C(4A)	110.1(3)	C(3)-C(4)-C(11)	112.9(4)
C(4A)-C(4)-C(11)	109.8(3)	C(4)-C(4A)-C(4B)	128.5(4)
C(4)-C(4A)-C(8B)	123.5(4)	C(4B)-C(4A)-C(8B)	108.0(4)
C(4A)-C(4B)-C(5)	134.6(4)	C(4A)-C(4B)-C(8A)	106.8(4)
C(5)-C(4B)-C(8A)	118.6(4)	C(4B)-C(5)-C(6)	120.0(4)
O(1)-C(6)-C(5)	116.2(4)	O(1)-C(6)-C(7)	123.1(4)
C(5)-C(6)-C(7)	120.8(4)	C(6)-C(7)-C(8)	120.4(5)
C(7)-C(8)-C(8A)	118.7(4)	N(1)-C(8A)-C(4B)	107.4(4)
N(1)-C(8A)-C(8)	131.1(4)	C(4B)-C(8A)-C(8)	121.5(4)
N(1)-C(8B)-C(1)	125.1(4)	N(1)-C(8B)-C(4A)	108.8(4)
C(1)-C(8B)-C(4A)	126.1(4)	N(2)-C(11)-C(4)	112.0(3)
O(2)-C(12)-N(2)	126.5(4)	O(2)-C(12)-C(13)	118.1(4)
N(2)-C(12)-C(13)	115.3(4)	F(1)-C(13)-F(2)	105.6(5)
F(1)-C(13)-F(3)	108.2(5)	F(2)-C(13)-F(3)	107.0(4)
F(1)-C(13)-C(12)	110.4(4)	F(2)-C(13)-C(12)	111.0(5)
F(3)-C(13)-C(12)	114.3(4)		

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$)

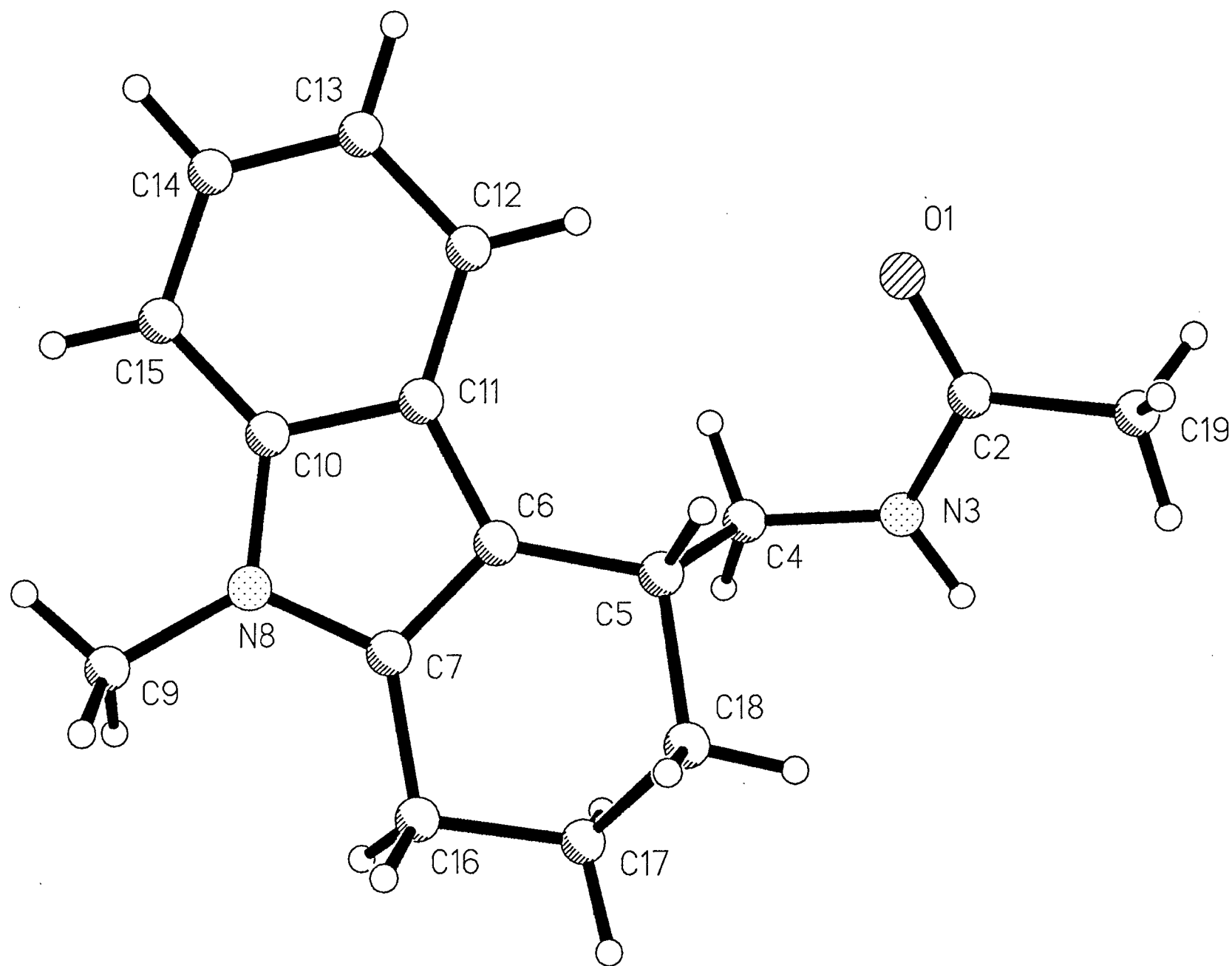
	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
F(1)	154(4)	87(3)	109(3)	41(2)	-18(3)	-40(3)
F(2)	135(3)	138(3)	45(2)	0(2)	1(2)	21(3)
F(3)	82(2)	155(3)	93(3)	65(2)	11(2)	52(2)
O(1)	59(2)	93(3)	78(3)	26(2)	21(2)	32(2)
O(2)	34(2)	85(2)	70(2)	3(2)	1(2)	5(2)
N(1)	61(3)	42(2)	35(2)	7(2)	-5(2)	6(2)
N(2)	34(2)	50(2)	44(2)	3(2)	-7(2)	6(2)
C(1)	64(3)	56(3)	48(3)	-4(3)	11(3)	-3(3)
C(2)	55(3)	77(4)	64(3)	-8(3)	13(3)	12(3)
C(3)	62(3)	56(3)	55(3)	-6(3)	8(3)	13(3)
C(4)	48(3)	40(3)	42(3)	-3(2)	-5(2)	3(2)
C(4A)	42(3)	40(2)	34(2)	2(2)	-1(2)	1(2)
C(4B)	40(2)	38(3)	38(2)	4(2)	-4(2)	-4(2)
C(5)	50(3)	46(3)	44(3)	10(2)	-4(2)	1(3)
C(6)	47(3)	53(3)	55(3)	4(3)	5(2)	9(3)
C(7)	59(3)	50(3)	58(3)	14(3)	-7(3)	12(3)
C(8)	63(3)	45(3)	47(3)	13(2)	-6(3)	0(3)
C(8A)	50(3)	39(3)	36(2)	1(2)	-5(2)	10(2)
C(8B)	50(3)	40(3)	37(2)	-2(2)	-1(2)	-8(2)
C(9)	80(4)	70(3)	42(3)	9(3)	-5(3)	15(3)
C(10)	68(4)	97(5)	90(4)	8(4)	18(3)	23(4)
C(11)	49(3)	45(3)	46(3)	1(2)	-3(2)	0(2)
C(12)	35(3)	55(3)	38(2)	-4(2)	0(2)	2(2)
C(13)	62(4)	77(4)	52(3)	9(3)	9(3)	9(3)

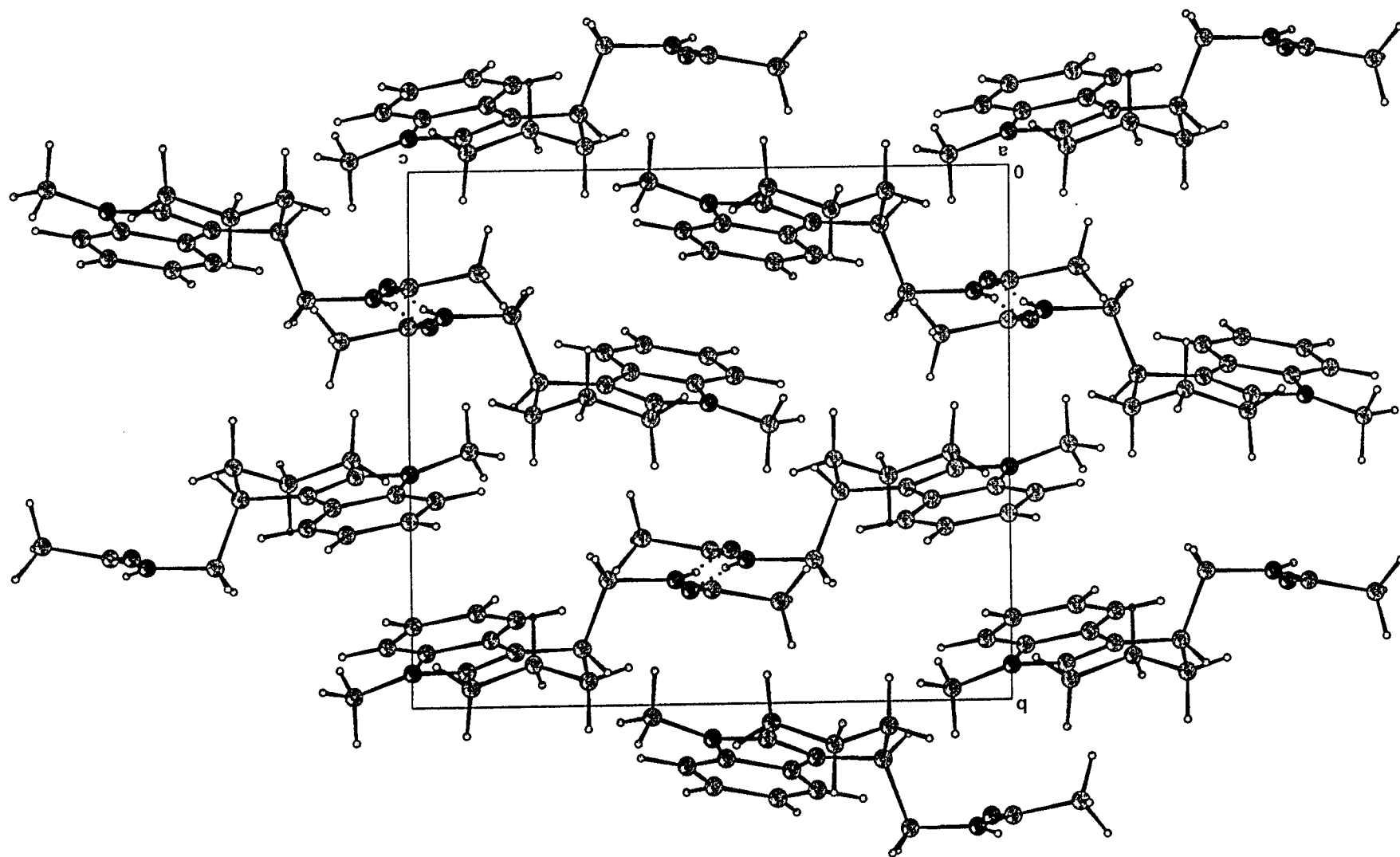
The anisotropic displacement exponent takes the form:

$$-2\pi^2(h^2a^{*2}U_{11} + \dots + 2hka^*b^*U_{12})$$

displacement parameters ($\text{\AA}^2 \times 10^3$)

	x	y	z	U
H(2C)	3141	4197	7434	80
H(1A)	2572	4842	4855	80
H(1B)	3268	5715	4996	80
H(2A)	3970	5255	5879	80
H(2B)	4305	4418	5456	80
H(3A)	3325	3823	6287	80
H(3B)	2387	3547	5761	80
H(4A)	966	4181	6437	80
H(5A)	-1104	5471	6790	80
H(7A)	-3198	7423	5882	80
H(8A)	-1559	7411	5146	80
H(9A)	1692	6502	4435	80
H(9B)	141	5514	4048	80
H(9C)	861	7380	4632	80
H(10A)	-4932	5046	7126	80
H(10B)	-4206	7521	6759	80
H(10C)	-4868	6658	6447	80
H(11A)	2926	5432	6818	80
H(11B)	1457	5562	7021	80





0.84 H₂O

O(1)	-0.2275(1)	-0.2129(1)	-0.46541(9)	0.0587	1.0000
C(2)	-0.1102(1)	-0.2127(1)	-0.5012(1)	0.0474	1.0000
N(3)	0.0017(1)	-0.2319(1)	-0.44108(9)	0.0471	1.0000
C(4)	-0.0083(1)	-0.2335(1)	-0.3281(1)	0.0471	1.0000
C(5)	-0.0073(1)	-0.1061(1)	-0.2838(1)	0.0460	1.0000
C(6)	-0.0554(1)	-0.1007(1)	-0.1727(1)	0.0441	1.0000
C(7)	0.0266(1)	-0.0655(1)	-0.0917(1)	0.0468	1.0000
N(8)	-0.0504(1)	-0.0639(1)	-0.00067(9)	0.0509	1.0000
C(9)	-0.0027(2)	-0.0224(2)	0.0995(1)	0.0665	1.0000
C(10)	-0.1826(2)	-0.1006(1)	-0.0234(1)	0.0499	1.0000
C(11)	-0.1897(2)	-0.1239(1)	-0.1312(1)	0.0468	1.0000
C(12)	-0.3151(2)	-0.1613(1)	-0.1746(1)	0.0562	1.0000
C(13)	-0.4290(2)	-0.1744(2)	-0.1086(2)	0.0668	1.0000
C(14)	-0.4183(2)	-0.1515(2)	-0.0021(2)	0.0681	1.0000
C(15)	-0.2967(2)	-0.1140(1)	0.0423(1)	0.0613	1.0000
C(16)	0.1760(2)	-0.0355(1)	-0.0983(1)	0.0540	1.0000
C(17)	0.2311(2)	-0.0777(2)	-0.2037(1)	0.0590	1.0000
C(18)	0.1337(2)	-0.0455(1)	-0.2939(1)	0.0566	1.0000
C(19)	-0.0818(2)	-0.1882(2)	-0.6144(1)	0.0641	1.0000

Atom	x/a	y/b	z/c	U(iso)	Occ
H(41)	-0.0967	-0.2737	-0.3074	0.0479	1.0000
H(42)	0.0714	-0.2787	-0.2985	0.0479	1.0000
H(51)	-0.0766	-0.0607	-0.3261	0.0458	1.0000
H(91)	-0.0791	-0.0319	0.1521	0.0677	1.0000
H(92)	0.0785	-0.0714	0.1226	0.0677	1.0000
H(93)	0.0244	0.0626	0.0950	0.0677	1.0000
H(121)	-0.3230	-0.1778	-0.2507	0.0570	1.0000
H(131)	-0.5197	-0.2009	-0.1395	0.0680	1.0000
H(141)	-0.5023	-0.1613	0.0429	0.0724	1.0000
H(151)	-0.2907	-0.0975	0.1183	0.0640	1.0000
H(161)	0.2278	-0.0761	-0.0408	0.0549	1.0000
H(162)	0.1886	0.0527	-0.0919	0.0549	1.0000
H(171)	0.3232	-0.0403	-0.2170	0.0603	1.0000
H(172)	0.2416	-0.1665	-0.2022	0.0603	1.0000
H(181)	0.1772	-0.0698	-0.3613	0.0570	1.0000
H(182)	0.1199	0.0433	-0.2941	0.0570	1.0000
H(191)	0.0193	-0.1918	-0.6281	0.0647	1.0000
H(192)	-0.1305	-0.2489	-0.6584	0.0647	1.0000
H(193)	-0.1175	-0.1069	-0.6327	0.0647	1.0000
H(1)	0.0934	-0.2460	-0.4752	0.0500	1.0000

O(1)	- C(2)	1.226(2)
C(2)	- N(3)	1.347(2)
C(2)	- C(19)	1.503(2)
N(3)	- C(4)	1.453(2)
C(4)	- C(5)	1.539(2)
C(5)	- C(6)	1.501(2)
C(5)	- C(18)	1.531(2)
C(6)	- C(7)	1.366(2)
C(6)	- C(11)	1.429(2)
C(7)	- N(8)	1.385(2)
C(7)	- C(16)	1.487(2)
N(8)	- C(9)	1.443(2)
N(8)	- C(10)	1.376(2)
C(10)	- C(11)	1.409(2)
C(10)	- C(15)	1.398(2)
C(11)	- C(12)	1.399(2)
C(12)	- C(13)	1.398(3)
C(13)	- C(14)	1.395(3)
C(14)	- C(15)	1.374(3)
C(16)	- C(17)	1.528(2)
C(17)	- C(18)	1.536(2)

_om	U(11)	U(22)	U(33)	U(23)	U(13)	U(12)
O(1)	0.0377(5)	0.0862(7)	0.0629(6)	-0.0018(5)	-0.0041(4)	-0.0031(5)
C(2)	0.0418(7)	0.0552(6)	0.0479(6)	-0.0077(5)	-0.0039(5)	-0.0021(5)
N(3)	0.0358(5)	0.0679(6)	0.0443(5)	-0.0086(5)	-0.0017(4)	0.0007(5)
C(4)	0.0432(7)	0.0547(6)	0.0445(6)	-0.0009(5)	-0.0035(5)	0.0011(5)
C(5)	0.0429(7)	0.0545(6)	0.0420(5)	0.0006(5)	-0.0035(5)	0.0001(5)
C(6)	0.0392(6)	0.0524(6)	0.0422(6)	-0.0025(5)	-0.0025(5)	0.0030(5)
C(7)	0.0449(7)	0.0535(6)	0.0434(6)	-0.0050(5)	-0.0030(5)	0.0019(5)
N(8)	0.0526(7)	0.0609(6)	0.0425(5)	-0.0080(4)	0.0004(5)	0.0036(5)
C(9)	0.089(1)	0.0762(9)	0.0451(7)	-0.0110(7)	-0.0029(8)	-0.0062(9)
C(10)	0.0526(8)	0.0508(6)	0.0491(7)	-0.0030(5)	0.0068(6)	0.0087(6)
C(11)	0.0421(7)	0.0510(6)	0.0487(6)	-0.0008(5)	-0.0015(5)	0.0056(5)
C(12)	0.0410(7)	0.0635(8)	0.0692(9)	-0.0009(7)	-0.0055(7)	0.0024(6)
C(13)	0.0427(8)	0.0722(9)	0.098(1)	0.0029(9)	0.0054(9)	-0.0000(7)
C(14)	0.056(1)	0.0681(9)	0.095(1)	0.0033(9)	0.0262(9)	0.0047(7)
C(15)	0.067(1)	0.0629(8)	0.0620(9)	0.0005(7)	0.0198(7)	0.0090(7)
C(16)	0.0457(7)	0.0662(7)	0.0553(7)	-0.0057(6)	-0.0096(6)	-0.0049(6)
C(17)	0.0420(8)	0.082(1)	0.0607(8)	-0.0050(7)	-0.0006(6)	-0.0070(7)
C(18)	0.0541(9)	0.0683(8)	0.0508(7)	-0.0022(6)	0.0033(6)	-0.0108(7)
C(19)	0.071(1)	0.0803(9)	0.0469(7)	-0.0049(7)	-0.0044(7)	-0.0026(8)

N(3) - C(2) - C(19)	115.8(1)
C(2) - N(3) - C(4)	121.4(1)
N(3) - C(4) - C(5)	110.9(1)
C(4) - C(5) - C(6)	112.7(1)
C(4) - C(5) - C(18)	112.8(1)
C(6) - C(5) - C(18)	109.8(1)
C(5) - C(6) - C(7)	123.7(1)
C(5) - C(6) - C(11)	128.9(1)
C(7) - C(6) - C(11)	107.3(1)
C(6) - C(7) - N(8)	109.4(1)
C(6) - C(7) - C(16)	125.9(1)
N(8) - C(7) - C(16)	124.6(1)
C(7) - N(8) - C(9)	125.6(1)
C(7) - N(8) - C(10)	108.5(1)
C(9) - N(8) - C(10)	125.7(1)
N(8) - C(10) - C(11)	108.0(1)
N(8) - C(10) - C(15)	129.7(1)
C(11) - C(10) - C(15)	122.3(2)
C(6) - C(11) - C(10)	106.7(1)
C(6) - C(11) - C(12)	134.1(1)
C(10) - C(11) - C(12)	119.2(1)
C(11) - C(12) - C(13)	118.4(2)
C(12) - C(13) - C(14)	121.0(2)
C(13) - C(14) - C(15)	121.8(2)
C(10) - C(15) - C(14)	117.4(2)
C(7) - C(16) - C(17)	108.7(1)
C(16) - C(17) - C(18)	112.3(1)
C(5) - C(18) - C(17)	112.3(1)
_audit_creation_method	CRYSTALS
_audit_creation_date	05/02/96
_cell_length_a	9.68(1)
_cell_angle_alpha	90.0000(3)
_cell_length_b	11.23(1)
_cell_angle_beta	90.0000(3)
_cell_length_c	12.83(1)
_cell_angle_gamma	90.0000(3)
_cell_volume	1394.73
_symmetry_space_group_name_H-M	'P 21 21 21'
loop_	
_symmetry_equiv_pos_as_xyz	
'X,Y,Z'	
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'-X,Y+1/2,-Z+1/2'	
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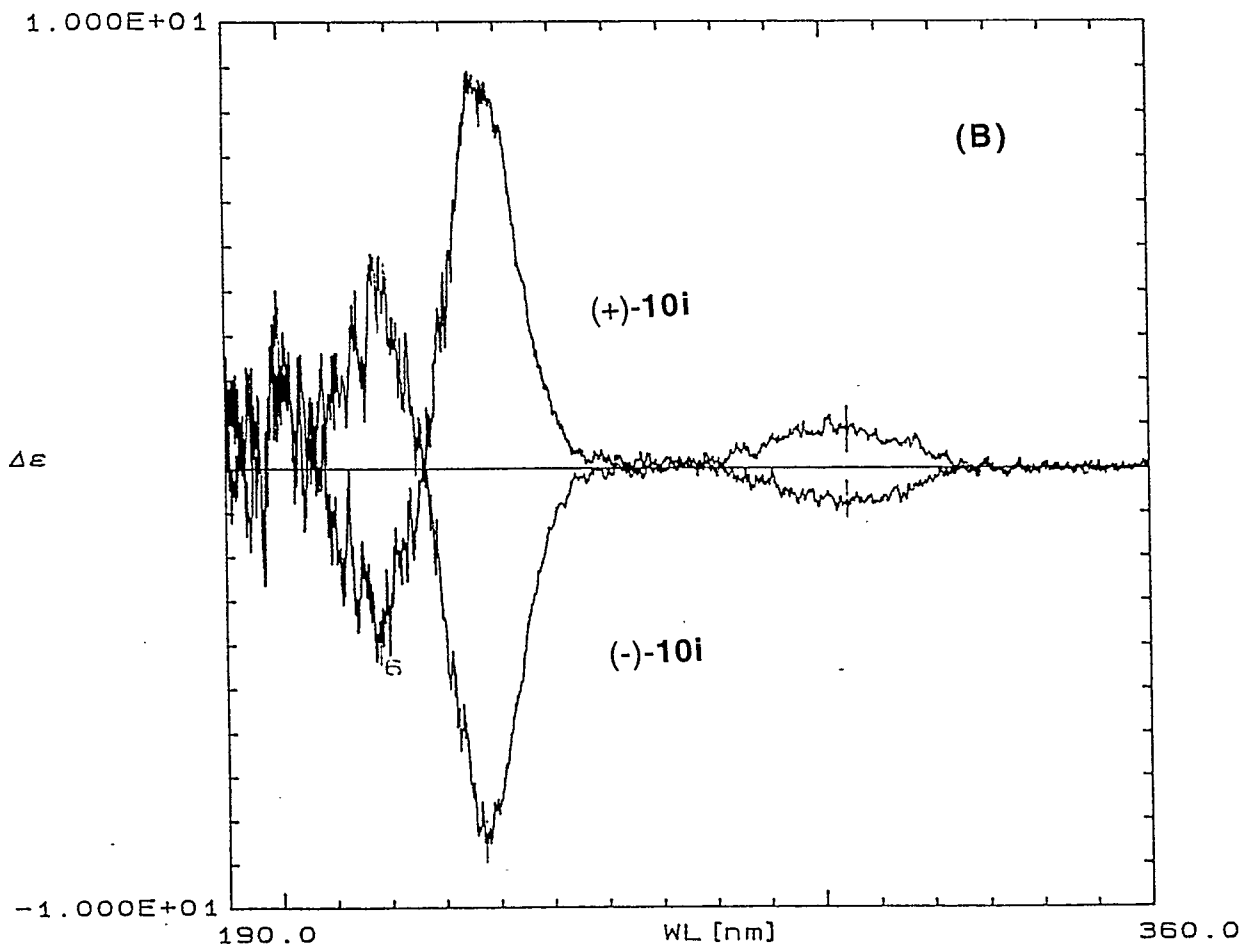
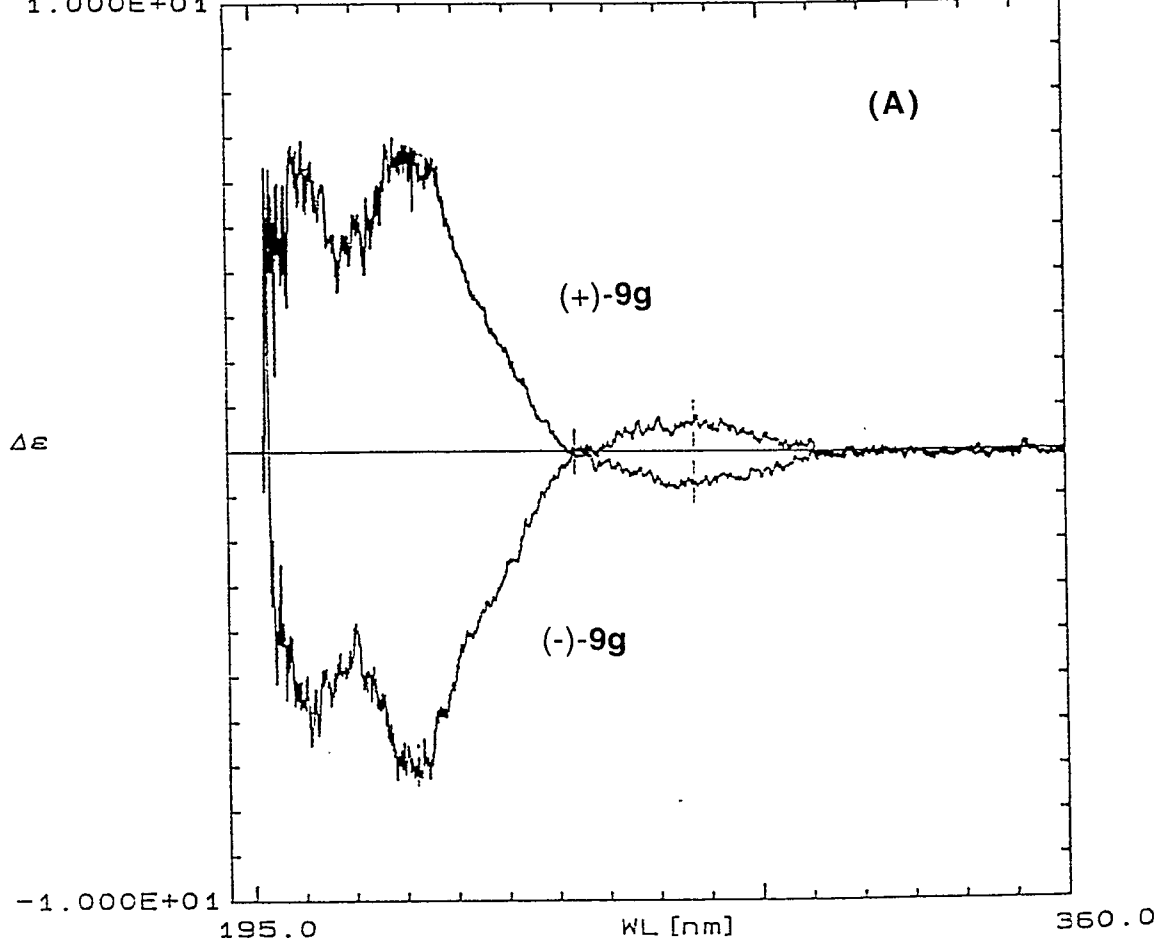
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NOW ALL THE GOODIES TO BE ENTERED BY HAND

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H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S	H	K	L	10FO	10FC	10S
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