Synthesis of [n]- and [n.n]Cyclophanes Using the Suzuki-Miyaura Coupling

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Supporting Information

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¹ H and ¹³ C NMR spectra for [n.n]Cyclophanes 5-8	S13 to S20
Crystal Structure Data for [n.n]Cyclophanes 5-8	S21 to S49

 AX500.KGP
 SF 400.134
 SY 31.0
 O1 6402.320
 SI 32768
 TD 32768
 SM 4385.965
 HZ/PT 1266
 FM 8.0
 RD 3.13
 AG 3.13
 RG 14
 NS 9
 TE 297
 FM 555
 C2 52.000
 D8 63.10
 LB 500
 SB 0.0
 CX 30.00
 CY 0.0
 FI 10.456
 F2 10.456
 F2/GN 11.856
 PRM/GN 11.804
 SR 103.30

ENDING

SF	100.614
SY	169.0
O	4000.000
SI	65536
TQ	65536
SW	23609.624
HZ/PT	.727

PW	1.9
GO	.730
AG	1.378
RG	327.69
NH	342.7
TH	397

FM	29800
CG	640.000
CG	174.000
LB	.500

SD	0.0
GX	36.00
Gy	0.0
F1	219.044
F2	17.534
HZ/CH	661.535
FM/CH	0.573
SH	5129.25

[8] Metacyclopentane, 3b

¹³C NMR in CDCl₃ at 100 MHz

#	CURSOR	FREQUENCY	PPM	INTENSITY
1	10610	14318.759	142.3140	7.986
2	12654	13116.922	130.3689	10.878
3	12626	12999.042	129.1973	6.746
4	12938	12627.262	125.5022	15.792
5	19610	7779.292	77.3183	7.479
6	19654	7747.325	77.0006	7.625
7	19698	7715.360	76.6829	7.571
8	25335	3619.452	35.9737	18.012
9	25944	3177.042	31.5766	4.714
10	26134	3038.932	30.2039	15.765
11	26601	2699.502	26.8303	16.496
12	27039	2381.282	23.6675	18.616
13	27182	2277.686	22.6379	3.910
14	28366	1417.106	14.0846	5.447

SF 400.134
 CY 30.0
 Q1 16402.920
 SI 32768
 ID 32768
 SM 4385.965
 HZ/PT 369
 AM 5.0
 ED 3.70
 AG 3.736
 AC 64
 NS 397
 PC 3300
 CO 3210.000
 CL 3210.0
 LB 500
 BX 0.0
 CY 36.00
 F1 10.0
 F2 10.4828
 F3 10.4828
 HZ/CM 121.706
 PPM/CM 304
 SR 1402.00

[9]Metacyclophane, 3c

¹H NMR in CDCl₃ at 400 MHz

[10]Metacyclophane, 3d
¹³C NMR in CDCl₃ at 100 MHz

	F1RSOR	FREQUENCY	PPM	INTENSITY
1	.44	14299.721	142.1247	4.396
2	1.280	13111.271	130.3128	4.051
3	1.2523	12934.311	128.5340	4.139
4	1.2877	12677.187	125.9984	8.661
5	1.9609	7785.547	77.3805	5.475
6	1.9653	7753.593	77.0629	5.510
7	1.9697	7721.651	76.7454	5.536
8	2.5432	3554.918	35.3323	8.651
9	26402	2849.837	28.3245	9.633
10	26603	2703.967	26.8747	10.114
11	26691	2639.953	26.2385	10.001
12	26786	2570.768	25.5508	7.999

3-PO SPECTROMETER IN USE, JOB 3
 LOCK LOST DURING ACQUISITION

SF 169.0 100.614
 Q1 4000.000
 SI 65366
 TO 65366
 SM 3309.524
 HZ/PT 727
 PK 1.5
 RD 1.730
 AG 1.376
 RG 32768
 EX 226
 IE 237
 FM 33600
 OF 6400.000
 DP 17H CPD
 LB 1.000
 GB 0.0
 CO 0.0
 CY 0.0
 IL 2.92826
 LS 1.17836
 HN 16.000
 RM 0.000
 SN -0.13.03

48

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= Revision Date = 9-SEP-1999 14:25:18
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= ID COMPLEX
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= 32768
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= 51.7[us]
= 0
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= 10.7[us]
= 18[us]
= 49[us]
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= Spin_off
= Spin_status
= Spin_status
= Temp_get
= Temp_set
= Temp_status
= Temp_status
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= X90_hi
= X90_lo
= X90_duration
= X_domain
= X_freq
= X_offset
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= X_prescans
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= 2.4[us]
= 0.57690944[Hz]
= 18.90359168[Hz]
= X_wstep

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[6.6](2,6)Pyridinophane, 7

^{13}C NMR in CDCl_3 at 50 MHz

77.807
 77.170
 76.532

159.994
 157.197
 156.000
 155.000
 154.000
 153.000

36.8
 34.473
 30.892
 28.070

Solid-State Structure Determination for [6.6]Paracyclophane, 5.

Table 1. Crystallographic data

Crystal Data

Empirical Formula	$C_{24}H_{32}$
Formula weight (g/mol)	320.50
Color; Habit	Colorless, parallelepiped
Size (mm)	0.12 x 0.17 x 0.28
Space Group	Orthorhombic, $P2_1/n$ (#14)
Unit Cell Dimensions	
a (Å)	10.8250(19)
b (Å)	5.9661(10)
c (Å)	16.481(3)
β (°)	100.000(12)
V (Å ³)	1048.2(3)
Formula units/cell	2
D_{calc} (g/cm ³)	1.015
$F(000)$	352

Data Collection*

Unit Cell Reflections	4247 ($I \geq 5 \cdot (I)$)
2θ Range	7.28 to 46.0°
Scan Type	$\bullet/0.5^\circ$ frames
Scan Speed	30 seconds/frame, (two scans)
Reflections Collected	6752
Reflections Unique (R_{int})	1440 (0.062)
Indices	(15 16 ± 9)
Crystal Decomposition	Negligible ($\pm 1\%$)
Absorption Coefficient	0.056 mm ⁻¹
Min./Max. Transmission	N.A.

Refinement*

Final Residuals ($I > 2 \cdot (I)$)	$R_1 = 0.1040$; $wR_2 = 0.2147$
Final Residuals (all data)	$R_1 = 0.1316$; $wR_2 = 0.2357$
Goodness-of-Fit	$S = 1.034$
Extinction coefficient	0.02(3)

Largest $\bullet/\bullet$	0.001
Reflections used (observed)	1440 (1099)
Parameters Refined	118
Data-to-Parameter Ratio	12.2:1
Max./Min. Difference Peaks	0.12/-0.15 $e^-/\text{\AA}^3$

*(Rigaku AFC8/Mercury CCD; graphite monochromated Mo K $\bullet$ radiation ($\bullet = 0.71073 \text{ \AA}$); temperature = $22 \pm 1^\circ\text{C}$; solved by Direct Methods, refined using full-matrix least-squares (on F) on F²(SHELXTL PLUS)).

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}</i>
C(1)	4196(4)	7451(7)	3153(2)	98(1)
C(2)	4204(4)	9278(7)	3663(3)	109(1)
C(3)	3301(4)	9570(7)	4144(3)	106(1)
C(4)	2346(4)	8081(7)	4130(2)	93(1)
C(5)	2335(4)	6260(7)	3622(3)	103(1)
C(6)	3239(4)	5937(7)	3145(2)	105(1)
C(7)	5170(5)	7126(10)	2611(3)	150(2)
C(8)	5912(7)	4985(13)	2810(4)	125(2)
C(8A)	6605(14)	6950(30)	3310(10)	126(5)
C(9)	6853(5)	5160(12)	3558(3)	153(2)
C(10)	7514(7)	3548(11)	4069(4)	170(3)
C(11)	8535(6)	3714(15)	4653(5)	128(3)
C(11A)	7967(13)	1600(20)	4430(8)	113(4)
C(12)	8641(4)	1646(10)	5330(3)	139(2)

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

Table 3. Bond distances (Å) and angles (°)Distances

C(1)-C(6)	1.373(5)	C(1)-C(2)	1.376(6)
C(1)-C(7)	1.507(6)	C(2)-C(3)	1.371(6)
C(3)-C(4)	1.360(5)	C(4)-C(5)	1.371(5)
C(4)-C(12a)	1.513(5)	C(5)-C(6)	1.371(5)
C(7)-C(8)	1.514(8)	C(7)-C(8A)	1.770(16)
C(8)-C(9)	1.460(8)	C(8A)-C(9)	1.157(15)
C(9)-C(10)	1.392(7)	C(10)-C(11)	1.336(8)
C(10)-C(11A)	1.356(13)	C(11)-C(12)	1.655(9)
C(11A)-C(12)	1.536(14)		

Angles

C(6)-C(1)-C(2)	116.8(4)	C(6)-C(1)-C(7)	120.9(5)
C(2)-C(1)-C(7)	122.3(5)	C(3)-C(2)-C(1)	121.7(4)
C(4)-C(3)-C(2)	121.5(4)	C(3)-C(4)-C(5)	117.0(4)
C(3)-C(4)-C(12a)	122.2(4)	C(5)-C(4)-C(12a)	120.8(4)
C(4)-C(5)-C(6)	122.1(4)	C(5)-C(6)-C(1)	120.9(4)
C(1)-C(7)-C(8)	112.3(4)	C(1)-C(7)-C(8A)	104.3(6)
C(9)-C(8)-C(7)	112.9(6)	C(9)-C(8A)-C(7)	114.1(12)
C(10)-C(9)-C(8)	132.2(7)	C(11)-C(10)-C(9)	131.1(8)
C(10)-C(11)-C(12)	112.5(6)	C(10)-C(11A)-C(12)	119.0(10)
C(4a)-C(12)-C(11A)	107.7(6)	C(4a)-C(12)-C(11)	110.1(4)

Atoms labeled with a lower-case character were generated by the following symmetry operation:

- a) $-x+1, -y+1, -z+1$

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

	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	101(3)	99(3)	94(3)	19(2)	16(2)	7(2)
C(2)	102(3)	88(3)	131(3)	11(3)	8(3)	-21(2)
C(3)	119(3)	81(3)	108(3)	-11(2)	-3(3)	1(2)
C(4)	84(2)	100(3)	91(2)	2(2)	1(2)	8(2)
C(5)	96(3)	102(3)	105(3)	0(2)	2(2)	-21(2)
C(6)	130(4)	92(3)	87(3)	-13(2)	4(2)	0(3)
C(7)	146(4)	175(5)	141(4)	64(4)	55(4)	52(4)
C(8)	137(5)	149(6)	94(4)	11(4)	39(4)	26(5)
C(9)	132(4)	189(6)	137(4)	16(4)	19(3)	48(4)
C(10)	187(6)	182(6)	161(5)	69(5)	88(5)	61(5)
C(11)	80(4)	174(7)	132(5)	13(5)	19(4)	-21(5)
C(12)	107(3)	186(5)	125(3)	27(3)	25(3)	49(3)

The anisotropic displacement factor exponent takes the form:
 $-2\pi^2(h^2\mathbf{a}^*U_{11} + \dots + 2h\mathbf{ka}^*\mathbf{b}^*U_{12} + \dots)$

Table 5. H-Atom coordinates ($\times 10^4$) and isotropic displacement coefficients ($\text{\AA}^2 \times 10^3$)

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{iso}</i>
H(2A)	4872	10386	3674	130
H(3A)	3342	10867	4494	127
H(5A)	1659	5167	3615	123
H(6A)	3185	4651	2788	126
H(7A)	5758	8375	2661	180
H(7B)	4714	7060	2046	180
H(7C)	5155	8361	2234	180
H(7D)	5011	5761	2300	180
H(8A)	6374	4645	2369	150
H(8B)	5309	3786	2839	150
H(8C)	6559	7901	3776	152
H(8D)	7313	7445	3055	152
H(9A)	6402	5993	3920	184
H(9B)	7522	6145	3439	184
H(9C)	5989	4823	3647	184
H(9D)	6891	4478	3032	184
H(10A)	7738	2420	3701	204
H(10B)	6879	2875	4349	204
H(10C)	7255	4106	4565	204
H(10D)	8350	4232	4083	204
H(11A)	8499	5129	4925	154
H(11B)	9291	3719	4395	154
H(11C)	8590	1043	4115	135
H(11D)	7271	534	4380	135
H(12A)	9488	1718	5677	167
H(12B)	8555	218	5058	167
H(12C)	9227	2898	5417	167
H(12D)	9135	307	5484	167

Table 6. Torsion angles [°]

C(6)-C(1)-C(2)-C(3)	0.2(6)
C(7)-C(1)-C(2)-C(3)	179.0(4)
C(1)-C(2)-C(3)-C(4)	-0.9(6)
C(2)-C(3)-C(4)-C(5)	0.8(6)
C(2)-C(3)-C(4)-C(12a)	178.7(4)
C(3)-C(4)-C(5)-C(6)	-0.2(6)
C(12a)-C(4)-C(5)-C(6)	-178.1(4)
C(4)-C(5)-C(6)-C(1)	-0.4(6)
C(2)-C(1)-C(6)-C(5)	0.4(6)
C(7)-C(1)-C(6)-C(5)	-178.4(4)
C(6)-C(1)-C(7)-C(8)	-61.7(6)
C(2)-C(1)-C(7)-C(8)	119.6(6)
C(6)-C(1)-C(7)-C(8A)	-120.0(7)
C(2)-C(1)-C(7)-C(8A)	61.3(7)
C(1)-C(7)-C(8)-C(9)	-76.5(7)
C(1)-C(7)-C(8A)-C(9)	86.9(12)
C(7)-C(8A)-C(9)-C(10)	-168.7(16)
C(7)-C(8)-C(9)-C(10)	162.4(6)
C(8A)-C(9)-C(10)-C(11)	-6(3)
C(8)-C(9)-C(10)-C(11)	165.1(7)
C(8A)-C(9)-C(10)-C(11A)	-174(3)
C(8)-C(9)-C(10)-C(11A)	-3(3)
C(9)-C(10)-C(11)-C(12)	156.9(6)
C(9)-C(10)-C(11A)-C(12)	-159(2)
C(10)-C(11A)-C(12)-C(4a)	74.7(11)
C(11)-C(11A)-C(12)-C(4a)	102.1(6)
C(10)-C(11)-C(12)-C(4a)	-71.2(7)

Atoms labeled with a lower-case character were generated by the following symmetry operation:

a) $-x+1, -y+1, -z+1$

Table 7. Least-squares planes data

Least-squares planes (x,y,z in crystal coordinates) and deviations from them
(* indicates atom used to define plane)

$$4.6825 (0.0152) x - 3.0510 (0.0084) y + 10.8142 (0.0187) z = 3.1022 (0.0119)$$

* -0.0014 (0.0027) C1

* -0.0030 (0.0028) C2

* 0.0051 (0.0027) C3

* -0.0028 (0.0026) C4

* -0.0015 (0.0026) C5

* 0.0036 (0.0027) C6

Rms deviation of fitted atoms = 0.0031

Solid-State Structure Determination for [6.6](3,5)Toluenophane, 6.

Table 1. Crystal data for C₂₆H₃₆.

Empirical formula	C ₂₆ H ₃₆
Formula weight	348.55
Crystal size	1.3 x .56 x .8 mm
Crystal habit	cubic
Crystal color	clear
Crystal system	monoclinic
Space group	P2 ₁ /n
Unit cell dimensions	a = 11.9063(11) Å □ = 90° b = 5.5631(5) Å □ = 100.646(7)° c = 16.833(2) Å □ = 90°
Volume	1095.8(2) Å ³
Z	2
Density (calculated)	1.056 Mg*m ⁻³
Absorption coefficient	0.059 mm ⁻¹
F(000)	384
Absorption correction	none

Table 2. Data collection for C₂₆H₃₆.

Diffractometer	Siemens P4
Temperature	294(2) K
Radiation source	fine-focus sealed tube
Wavelength	0.71073 Å (MoKα)
Monochromator	graphite
Theta range, data coll'n	1.94 to 25.00°
Scan type	omega

Index ranges	-14 ≤ h ≤ 1, -1 ≤ k ≤ 6, -19 ≤ l ≤ 20
Reflections collected	2390
Independent reflections	1678 (R(int) = 0.0173)
Standard reflections	-2,-1,4; -6,0,-4;-1,2,8

Table 3. Solution and refinement of C₂₆H₃₆.

System for solution	SHELXS-86 (Sheldrick, 1990)
Structure solution	direct methods
System for refinement	SHELXL-93 (Sheldrick, 1993)
Hydrogen atoms	fixed
Data/ restraints/ parameters	1634 / 0 / 119
Final R indices (1) [I>2σ(I)]	R ₁ = 0.0475, wR ₂ = 0.1424
Reflections observed	1506
R indices (all data)	R ₁ = 0.0529, wR ₂ = 0.1635
Goodness-of-fit (2) on F ²	1.094
Extinction coefficient	0.21(2)
Largest diff. peak and hole	0.186 and -0.123 eÅ ⁻³

$$1) R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|} \quad \text{and} \quad wR_2 = \left[\frac{\sum [w(F_o^2 - F_c^2)^2]}{\sum [w(F_o^2)^2]} \right]^{1/2}$$

$$2) \text{ Goodness-of-Fit} = \left[\frac{\sum [w(F_o^2 - F_c^2)^2]}{(M-N)} \right]^{1/2}$$

where M is the number of reflections and N is the number of parameters refined.

Table 4. Atomic coordinates [× 10⁴] and equivalent isotropic displacement parameters [Å² × 10³] for C₂₆H₃₆. U(eq) is defined as one third of the trace of the orthogonalized U(ij) tensor.

	x	y	z	U(eq)
C(1)	1434(1)	2359(3)	5703(1)	65(1)
C(2)	1465(1)	4199(4)	6257(1)	70(1)
C(3)	2527(2)	4981(4)	6663(1)	72(1)
C(4)	3531(1)	3974(3)	6513(1)	65(1)
C(5)	3460(1)	2141(3)	5956(1)	63(1)
C(6)	2422(1)	1297(3)	5538(1)	60(1)
C(7)	2376(2)	-659(3)	4910(1)	73(1)
C(8)	3078(1)	-98(3)	4260(1)	66(1)
C(9)	2706(1)	2139(3)	3775(1)	64(1)
C(10)	3491(1)	2912(4)	3208(1)	70(1)
C(11)	4646(1)	3867(3)	3629(1)	61(1)
C(12)	5308(2)	5119(4)	3072(1)	83(1)
C(13)	379(2)	5359(5)	6407(2)	105(1)

Table 5. Bond lengths [Å] and angles [°] for C₂₆H₃₆.

C(1)-C(2)	1.381(3)
C(1)-C(6)	1.390(2)
C(2)-C(3)	1.391(3)
C(2)-C(13)	1.507(2)
C(3)-C(4)	1.385(2)
C(4)-C(5)	1.377(2)
C(4)-C(12)#1	1.516(2)
C(5)-C(6)	1.386(2)
C(6)-C(7)	1.511(2)
C(7)-C(8)	1.527(2)
C(8)-C(9)	1.510(2)
C(9)-C(10)	1.517(2)
C(10)-C(11)	1.521(2)
C(11)-C(12)	1.504(2)
C(12)-C(4)#1	1.516(2)
C(2)-C(1)-C(6)	122.1(2)
C(1)-C(2)-C(3)	118.2(2)
C(1)-C(2)-C(13)	120.9(2)
C(3)-C(2)-C(13)	120.9(2)
C(4)-C(3)-C(2)	121.4(2)
C(5)-C(4)-C(3)	118.5(2)
C(5)-C(4)-C(12)#1	119.8(2)
C(3)-C(4)-C(12)#1	121.7(2)
C(4)-C(5)-C(6)	122.2(2)
C(5)-C(6)-C(1)	117.6(2)
C(5)-C(6)-C(7)	120.86(14)
C(1)-C(6)-C(7)	121.5(2)
C(6)-C(7)-C(8)	113.57(14)
C(9)-C(8)-C(7)	114.27(14)
C(8)-C(9)-C(10)	114.86(14)
C(9)-C(10)-C(11)	114.52(13)
C(12)-C(11)-C(10)	113.84(13)
C(11)-C(12)-C(4)#1	113.89(13)

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,-y+1,-z+1

Table 6. Anisotropic displacement parameters [$\text{\AA}^2 \times 10^3$] for $\text{C}_{26}\text{H}_{36}$. The anisotropic displacement factor exponent takes the form: $-2(\square)^2[(ha^*)^2U_{11} + \dots + 2hka^*b^*U_{12}]$

	U11	U22	U33	U23	U13	U12
C(1)	51(1)	78(1)	68(1)	4(1)	16(1)	-4(1)
C(2)	67(1)	83(1)	65(1)	4(1)	24(1)	8(1)
C(3)	84(1)	78(1)	57(1)	-3(1)	24(1)	-4(1)
C(4)	63(1)	81(1)	52(1)	7(1)	13(1)	-11(1)
C(5)	51(1)	70(1)	70(1)	5(1)	16(1)	0(1)
C(6)	54(1)	61(1)	68(1)	5(1)	19(1)	-6(1)
C(7)	66(1)	64(1)	93(1)	-9(1)	23(1)	-11(1)
C(8)	58(1)	60(1)	81(1)	-14(1)	17(1)	2(1)
C(9)	46(1)	75(1)	68(1)	-9(1)	3(1)	4(1)
C(10)	59(1)	85(1)	60(1)	-3(1)	0(1)	-5(1)
C(11)	57(1)	64(1)	59(1)	3(1)	2(1)	1(1)
C(12)	76(1)	113(2)	58(1)	1(1)	6(1)	-23(1)
C(13)	86(1)	130(2)	108(2)	-7(2)	39(1)	27(1)

Table 7. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for $\text{C}_{26}\text{H}_{36}$.

	x	y	z	U(eq)
H(1A)	728(1)	1812(3)	5431(1)	78
H(3A)	2563(2)	6206(4)	7043(1)	86
H(5A)	4131(1)	1446(3)	5857(1)	76
H(7A)	1586(2)	-909(3)	4652(1)	88
H(7B)	2653(2)	-2146(3)	5178(1)	88
H(8A)	3873(1)	83(3)	4517(1)	79
H(8B)	3030(1)	-1455(3)	3894(1)	79
H(9A)	2648(1)	3447(3)	4147(1)	77
H(9B)	1948(1)	1867(3)	3459(1)	77
H(10A)	3110(1)	4150(4)	2851(1)	84
H(10B)	3616(1)	1548(4)	2876(1)	84
H(11A)	4527(1)	4981(3)	4049(1)	73
H(11B)	5098(1)	2537(3)	3888(1)	73
H(12A)	4862(2)	6470(4)	2821(1)	100
H(12B)	5412(2)	4016(4)	2645(1)	100
H(13A)	563(2)	6595(5)	6808(2)	158
H(13B)	-94(2)	4169(5)	6594(2)	158
H(13C)	-23(2)	6054(5)	5913(2)	158

Solid-State Structure Determination for [6.6](2,6)pyridinophane, 7.

chemical formula moiety	?
chemical formula structural	'C22 H30 N2'
chemical formula analytical	'C22 H30 N2'
chemical formula sum	'C22 H30 N2'
chemical formula weight	322.48
chemical melting point	364 K
chemical compound source	?

loop

atom type symbol	
atom type description	
atom type scat dispersion real	
atom type scat dispersion imag	
atom type scat source	
'C' 'C'	0.0033 0.0016
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'	
'H' 'H'	0.0000 0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'	
'N' 'N'	0.0061 0.0033
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'	

symmetry cell setting	Triclinic
symmetry space group name	H-M P-1

loop

symmetry equiv pos as xyz	
'x, y, z'	
'-x, -y, -z'	

cell length a	8.7420(7)
cell length b	12.9751(11)

cell length c 17.3626(14)
 cell angle alpha 90.138(6)
 cell angle beta 90.773(6)
 cell angle gamma 91.771(7)
 cell volume 1968.3(3)
 cell formula units Z 4
 cell measurement temperature 294(2)
 cell measurement reflns used 39
 cell measurement theta min 4.53
 cell measurement theta max 12.49

exptl crystal description cubic
 exptl crystal colour clear
 exptl crystal size max 1.3
 exptl crystal size mid 1.2
 exptl crystal size min 0.7
 exptl crystal density meas ?
 exptl crystal density diffn 1.088
 exptl crystal density method ?
 exptl crystal F 000 704
 exptl absorpt coefficient mu 0.063
 exptl absorpt correction type none
 exptl absorpt correction T min ?
 exptl absorpt correction T max ?

exptl special details

;
 ?
 ;

diffn ambient temperature 294(2)
 diffn radiation wavelength 0.71073
 diffn radiation type MoK α
 diffn radiation source 'fine-focus sealed tube'
 diffn radiation monochromator graphite
 diffn measurement device 'Siemens P4'
 diffn measurement method '\w scans'
 diffn standards number 3
 diffn standards interval count 97
 diffn standards interval time ?
 diffn standards decay % ?
 diffn reflns number 8366
 diffn reflns av R equivalents 0.0145
 diffn reflns av sigmaI/netI 0.0256
 diffn reflns limit h min -10
 diffn reflns limit h max 1
 diffn reflns limit k min -15

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diffn reflns limit k max    15
diffn reflns limit l min   -20
diffn reflns limit l max    20
diffn reflns theta min     1.96
diffn reflns theta max     25.00
reflns number total        6896
reflns number observed      5492
reflns observed criterion   >2sigma(I)

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computing data collection   'Siemens XSCANS'
computing cell refinement   'Siemens XSCANS'
computing data reduction    'Siemens SHELXTL'
computing structure solution 'SHELXS-86 (Sheldrick, 1990)'
computing structure refinement 'SHELXL-93 (Sheldrick, 1993)'
computing molecular graphics 'Siemens SHELXTL'
computing publication material 'Siemens SHELXTL'

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refine special details
;

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Refinement on F^2 for ALL reflections except for 431 with very negative F^2 or flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > 2\sigma(F^2)$ is used only for calculating R factor obs etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

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refine ls structure factor coef Fsqd
refine ls matrix type          full
refine ls weighting scheme
'calc w=1/[s^2*(Fo^2)+(0.1136P)^2+0.1953P] where P=(Fo^2+2Fc^2)/3'
atom sites solution primary    direct
atom sites solution secondary  difmap
atom sites solution hydrogens  geom
refine ls hydrogen treatment   fixed
refine ls extinction method    SHELXL
refine ls extinction coef      0.0482(41)
refine ls extinction expression
'Fc*^=kFc[1+0.001xFc^2/l^3/sin(2\q)]^-1/4'
refine ls number reflns        6465
refine ls number parameters     471
refine ls number restraints     0
refine ls R factor all          0.0690
refine ls R factor obs          0.0562
refine ls wR factor all         0.1982

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refine ls wR factor obs      0.1703
refine ls goodness of fit all 1.029
refine ls goodness of fit obs 1.067
refine ls restrained S all   1.104
refine ls restrained S obs   1.067
refine ls shift/esd max      0.001
refine ls shift/esd mean     0.000

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loop

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atom site label
atom site type symbol
atom site fract x
atom site fract y
atom site fract z
atom site U iso or equiv
atom site thermal displace type
atom site occupancy
atom site calc flag
atom site refinement flags
atom site disorder group
N1 N 0.2241(2) 0.90640(11) 0.88210(8) 0.0620(4) Uani 1 d . .
C1 C 0.1589(2) 0.89582(14) 0.81196(10) 0.0632(4) Uani 1 d . .
N2 N 0.3073(2) 1.05385(12) 1.35290(9) 0.0721(4) Uani 1 d . .
C2 C 0.1602(2) 0.8028(2) 0.77266(11) 0.0761(5) Uani 1 d . .
H2B H 0.1160(2) 0.7967(2) 0.72379(11) 0.091 Uiso 1 calc R .
N3 N 0.1757(2) 0.37434(11) 0.96538(9) 0.0694(4) Uani 1 d . .
C3 C 0.2272(3) 0.7200(2) 0.80638(13) 0.0828(6) Uani 1 d . .
H3B H 0.2292(3) 0.6573(2) 0.78039(13) 0.099 Uiso 1 calc R .
N4 N 0.3307(2) 0.61725(12) 0.46854(9) 0.0728(4) Uani 1 d . .
C4 C 0.2913(2) 0.72976(15) 0.87851(13) 0.0754(5) Uani 1 d . .
H4B H 0.3361(2) 0.67398(15) 0.90246(13) 0.090 Uiso 1 calc R .
C5 C 0.2878(2) 0.82446(13) 0.91486(11) 0.0628(4) Uani 1 d . .
C6 C 0.3463(2) 0.8398(2) 0.99568(11) 0.0724(5) Uani 1 d . .
H6A H 0.4049(2) 0.9044(2) 0.99914(11) 0.087 Uiso 1 calc R .
H6B H 0.4138(2) 0.7846(2) 1.00914(11) 0.087 Uiso 1 calc R .
C7 C 0.2153(2) 0.84134(14) 1.05194(11) 0.0688(5) Uani 1 d . .
H7A H 0.1624(2) 0.77455(14) 1.05067(11) 0.083 Uiso 1 calc R .
H7B H 0.1434(2) 0.89220(14) 1.03485(11) 0.083 Uiso 1 calc R .
C8 C 0.2632(2) 0.8658(2) 1.13497(11) 0.0738(5) Uani 1 d . .
H8A H 0.3197(2) 0.8086(2) 1.15547(11) 0.089 Uiso 1 calc R .
H8B H 0.3313(2) 0.9262(2) 1.13547(11) 0.089 Uiso 1 calc R .
C9 C 0.1293(2) 0.88528(14) 1.18676(10) 0.0690(5) Uani 1 d . .
H9A H 0.0543(2) 0.82904(14) 1.18106(10) 0.083 Uiso 1 calc R .
H9B H 0.1647(2) 0.88654(14) 1.23995(10) 0.083 Uiso 1 calc R .
C10 C 0.0543(2) 0.98531(14) 1.16859(10) 0.0650(4) Uani 1 d . .
H10A H 0.0208(2) 0.98350(14) 1.11511(10) 0.078 Uiso 1 calc R .
H10B H 0.1307(2) 1.04081(14) 1.17395(10) 0.078 Uiso 1 calc R .

```

C11 C 0.0812(2) 0.9893(2) 0.78219(10) 0.0721(5) Uani 1 d . .
 H11A H 0.0467(2) 0.9770(2) 0.72955(10) 0.086 Uiso 1 calc R .
 H11B H 0.1536(2) 1.0475(2) 0.78217(10) 0.086 Uiso 1 calc R .
 C12 C 0.2317(2) 1.14102(15) 1.35970(11) 0.0751(5) Uani 1 d . .
 C13 C 0.2553(3) 1.2230(2) 1.30939(15) 0.0861(6) Uani 1 d . .
 H13A H 0.2024(3) 1.2836(2) 1.31516(15) 0.103 Uiso 1 calc R .
 C14 C 0.3579(3) 1.2130(2) 1.2513(2) 0.0957(7) Uani 1 d . .
 H14A H 0.3743(3) 1.2666(2) 1.2167(2) 0.115 Uiso 1 calc R .
 C15 C 0.4354(2) 1.1249(2) 1.24414(14) 0.0855(6) Uani 1 d . .
 H15A H 0.5050(2) 1.1175(2) 1.20462(14) 0.103 Uiso 1 calc R .
 C16 C 0.4098(2) 1.04565(15) 1.29659(12) 0.0725(5) Uani 1 d . .
 C17 C 0.4990(2) 0.9479(2) 1.29472(12) 0.0785(5) Uani 1 d . .
 C18 C 0.6207(13) 0.9469(9) 1.3552(6) 0.062(2) Uani 0.539(6) d P 1
 H18A H 0.5744(13) 0.9589(9) 1.4047(6) 0.074 Uiso 0.539(6) calc PR 1
 H18B H 0.6928(13) 1.0036(9) 1.3458(6) 0.074 Uiso 0.539(6) calc PR 1
 C19 C 0.7093(11) 0.8465(7) 1.3597(5) 0.068(2) Uani 0.539(6) d P 1
 H19A H 0.6373(11) 0.7886(7) 1.3648(5) 0.082 Uiso 0.539(6) calc PR 1
 H19B H 0.7645(11) 0.8374(7) 1.3121(5) 0.082 Uiso 0.539(6) calc PR 1
 C20 C 0.8223(5) 0.8467(3) 1.4273(2) 0.0698(12) Uani 0.539(6) d P 1
 H20A H 0.8895(5) 0.7890(3) 1.4220(2) 0.084 Uiso 0.539(6) calc PR 1
 H20B H 0.8850(5) 0.9097(3) 1.4263(2) 0.084 Uiso 0.539(6) calc PR 1
 C21 C 0.7425(5) 0.8391(4) 1.5031(3) 0.0697(12) Uani 0.539(6) d P 1
 H21A H 0.6902(5) 0.7724(4) 1.5077(3) 0.084 Uiso 0.539(6) calc PR 1
 H21B H 0.6674(5) 0.8922(4) 1.5070(3) 0.084 Uiso 0.539(6) calc PR 1
 C18' C 0.5898(15) 0.9362(11) 1.3756(7) 0.065(2) Uani 0.461(6) d P 2
 H18C H 0.6655(15) 0.9921(11) 1.3810(7) 0.078 Uiso 0.461(6) calc PR 2
 H18D H 0.5183(15) 0.9414(11) 1.4175(7) 0.078 Uiso 0.461(6) calc PR 2
 C19' C 0.6697(13) 0.8328(9) 1.3808(6) 0.070(2) Uani 0.461(6) d P 2
 C20' C 0.7304(6) 0.8078(3) 1.4595(3) 0.0647(13) Uani 0.461(6) d P 2
 H20C H 0.7746(6) 0.7402(3) 1.4576(3) 0.078 Uiso 0.461(6) calc PR 2
 H20D H 0.6451(6) 0.8036(3) 1.4946(3) 0.078 Uiso 0.461(6) calc PR 2
 C21' C 0.8509(5) 0.8845(3) 1.4925(2) 0.0609(13) Uani 0.461(6) d P 2
 H21C H 0.9449(5) 0.8815(3) 1.4637(2) 0.073 Uiso 0.461(6) calc PR 2
 H21D H 0.8141(5) 0.9542(3) 1.4898(2) 0.073 Uiso 0.461(6) calc PR 2
 C22 C 0.1217(4) 1.1456(2) 1.42555(13) 0.1069(8) Uani 1 d . .
 C23 C 0.2243(2) 0.30458(14) 0.91526(11) 0.0685(5) Uani 1 d . .
 C24 C 0.2402(2) 0.2022(2) 0.93531(14) 0.0788(6) Uani 1 d . .
 H24A H 0.2747(2) 0.1550(2) 0.89962(14) 0.095 Uiso 1 calc R .
 C25 C 0.2043(3) 0.1714(2) 1.0086(2) 0.0856(6) Uani 1 d . .
 H25A H 0.2132(3) 0.1029(2) 1.0231(2) 0.103 Uiso 1 calc R .
 C26 C 0.1554(2) 0.2425(2) 1.06003(13) 0.0789(5) Uani 1 d . .
 H26A H 0.1308(2) 0.2228(2) 1.10993(13) 0.095 Uiso 1 calc R .
 C27 C 0.1428(2) 0.34425(14) 1.03730(11) 0.0674(5) Uani 1 d . .
 C28 C 0.0928(2) 0.4271(2) 1.09116(12) 0.0794(5) Uani 1 d . .
 H28A H 0.0802(2) 0.3978(2) 1.14211(12) 0.095 Uiso 1 calc R .
 H28B H -0.0061(2) 0.4507(2) 1.07396(12) 0.095 Uiso 1 calc R .
 C29 C 0.2039(2) 0.51893(14) 1.09661(11) 0.0715(5) Uani 1 d . .

H29A H 0.2163(2) 0.54807(14) 1.04559(11) 0.086 Uiso 1 calc R .
 H29B H 0.1598(2) 0.57110(14) 1.12888(11) 0.086 Uiso 1 calc R .
 C30 C 0.3596(2) 0.49457(13) 1.12871(11) 0.0687(5) Uani 1 d . .
 H30A H 0.4019(2) 0.44029(13) 1.09779(11) 0.082 Uiso 1 calc R .
 H30B H 0.3478(2) 0.46833(13) 1.18062(11) 0.082 Uiso 1 calc R .
 C31 C 0.4722(2) 0.58555(13) 1.13083(11) 0.0689(5) Uani 1 d . .
 H31A H 0.4842(2) 0.61137(13) 1.07881(11) 0.083 Uiso 1 calc R .
 H31B H 0.4290(2) 0.63998(13) 1.16129(11) 0.083 Uiso 1 calc R .
 C32 C 0.6286(2) 0.56291(15) 1.16343(10) 0.0724(5) Uani 1 d . .
 H32A H 0.6718(2) 0.50809(15) 1.13336(10) 0.087 Uiso 1 calc R .
 H32B H 0.6172(2) 0.53804(15) 1.21575(10) 0.087 Uiso 1 calc R .
 C33 C 0.2594(3) 0.3446(2) 0.83586(12) 0.0849(6) Uani 1 d . .
 H33A H 0.1648(3) 0.3641(2) 0.81085(12) 0.102 Uiso 1 calc R .
 H33B H 0.3021(3) 0.2897(2) 0.80571(12) 0.102 Uiso 1 calc R .
 C34 C 0.2823(2) 0.6836(2) 0.41510(11) 0.0717(5) Uani 1 d . .
 C35 C 0.2599(2) 0.7861(2) 0.43201(15) 0.0853(6) Uani 1 d . .
 H35A H 0.2247(2) 0.8306(2) 0.39426(15) 0.102 Uiso 1 calc R .
 C36 C 0.2906(3) 0.8212(2) 0.5052(2) 0.0936(7) Uani 1 d . .
 H36A H 0.2779(3) 0.8902(2) 0.5175(2) 0.112 Uiso 1 calc R .
 C37 C 0.3402(2) 0.7535(2) 0.56024(14) 0.0854(6) Uani 1 d . .
 H37A H 0.3612(2) 0.7761(2) 0.61026(14) 0.102 Uiso 1 calc R .
 C38 C 0.3585(2) 0.6519(2) 0.54043(11) 0.0714(5) Uani 1 d . .
 C39 C 0.4083(3) 0.5721(2) 0.59747(13) 0.0862(6) Uani 1 d . .
 H39A H 0.4214(3) 0.6041(2) 0.64779(13) 0.103 Uiso 1 calc R .
 H39B H 0.5066(3) 0.5466(2) 0.58225(13) 0.103 Uiso 1 calc R .
 C40 C 0.2942(3) 0.4824(2) 0.60305(12) 0.0809(6) Uani 1 d . .
 H40A H 0.3354(3) 0.4315(2) 0.63781(12) 0.097 Uiso 1 calc R .
 H40B H 0.2825(3) 0.4504(2) 0.55267(12) 0.097 Uiso 1 calc R .
 C41 C 0.1377(3) 0.5111(2) 0.63099(12) 0.0808(6) Uani 1 d . .
 H41A H 0.1490(3) 0.5402(2) 0.68233(12) 0.097 Uiso 1 calc R .
 H41B H 0.0986(3) 0.5643(2) 0.59758(12) 0.097 Uiso 1 calc R .
 C42 C 0.0217(2) 0.4230(2) 0.63334(11) 0.0765(5) Uani 1 d . .
 H42A H 0.0614(2) 0.3694(2) 0.66619(11) 0.092 Uiso 1 calc R .
 H42B H 0.0093(2) 0.3944(2) 0.58187(11) 0.092 Uiso 1 calc R .
 C43 C -0.1341(3) 0.4517(2) 0.66236(11) 0.0826(6) Uani 1 d . .
 H43A H -0.1734(3) 0.5058(2) 0.62986(11) 0.099 Uiso 1 calc R .
 H43B H -0.1220(3) 0.4795(2) 0.71408(11) 0.099 Uiso 1 calc R .
 C44 C 0.2516(3) 0.6378(2) 0.33626(12) 0.0878(6) Uani 1 d . .
 H44A H 0.3467(3) 0.6137(2) 0.31564(12) 0.105 Uiso 1 calc R .
 H44B H 0.2148(3) 0.6911(2) 0.30220(12) 0.105 Uiso 1 calc R .

loop

atom site aniso label
 atom site aniso U 11
 atom site aniso U 22
 atom site aniso U 33
 atom site aniso U 23

atom site aniso U 13

atom site aniso U 12

N1 0.0598(8) 0.0623(8) 0.0637(8) -0.0113(6) -0.0070(6) 0.0060(6)
C1 0.0545(9) 0.0757(11) 0.0592(9) -0.0096(8) -0.0006(7) 0.0019(8)
N2 0.0794(10) 0.0679(9) 0.0685(9) 0.0026(7) -0.0200(8) 0.0007(8)
C2 0.0694(11) 0.0914(14) 0.0669(11) -0.0235(10) -0.0008(9) -0.0036(10)
N3 0.0734(9) 0.0647(9) 0.0694(9) 0.0012(7) -0.0117(7) -0.0033(7)
C3 0.0833(13) 0.0708(12) 0.0941(15) -0.0290(11) 0.0052(11) 0.0025(10)
N4 0.0756(10) 0.0764(10) 0.0666(9) -0.0065(7) 0.0031(7) 0.0064(8)
C4 0.0771(12) 0.0625(10) 0.0869(13) -0.0115(9) 0.0023(10) 0.0086(9)
C5 0.0567(9) 0.0611(9) 0.0710(10) -0.0069(8) -0.0014(8) 0.0091(8)
C6 0.0687(11) 0.0713(11) 0.0776(12) -0.0042(9) -0.0187(9) 0.0153(9)
C7 0.0683(10) 0.0669(10) 0.0711(11) -0.0101(8) -0.0170(9) 0.0090(8)
C8 0.0800(12) 0.0718(11) 0.0698(11) -0.0047(8) -0.0256(9) 0.0203(9)
C9 0.0799(12) 0.0661(10) 0.0608(10) 0.0040(8) -0.0177(9) 0.0051(9)
C10 0.0641(10) 0.0655(10) 0.0654(10) 0.0038(8) -0.0076(8) 0.0035(8)
C11 0.0711(11) 0.0876(13) 0.0574(10) 0.0000(9) -0.0061(8) 0.0033(9)
C12 0.0803(12) 0.0719(11) 0.0725(11) 0.0007(9) -0.0304(10) 0.0049(10)
C13 0.0828(14) 0.0691(12) 0.106(2) 0.0088(11) -0.0332(13) 0.0032(10)
C14 0.0782(14) 0.0874(15) 0.120(2) 0.0374(14) -0.0191(14) -0.0127(12)
C15 0.0635(11) 0.0897(14) 0.102(2) 0.0222(12) -0.0110(10) -0.0109(10)
C16 0.0635(10) 0.0737(11) 0.0791(12) 0.0028(9) -0.0224(9) -0.0089(9)
C17 0.0749(12) 0.0759(12) 0.0841(13) -0.0058(10) -0.0227(10) 0.0047(10)
C18 0.069(4) 0.058(3) 0.058(4) -0.005(3) -0.002(3) 0.003(3)
C19 0.087(5) 0.061(3) 0.056(4) -0.007(3) -0.002(3) 0.001(3)
C20 0.068(2) 0.069(2) 0.073(3) -0.003(2) 0.007(2) 0.015(2)
C21 0.062(2) 0.077(3) 0.069(3) 0.001(2) 0.001(2) 0.006(2)
C18' 0.061(5) 0.070(4) 0.062(6) -0.005(4) -0.009(3) -0.004(3)
C19' 0.079(5) 0.068(4) 0.064(5) -0.007(3) -0.003(3) 0.014(3)
C20' 0.072(3) 0.058(2) 0.065(3) -0.007(2) -0.002(2) 0.008(2)
C21' 0.055(2) 0.068(2) 0.060(2) -0.001(2) 0.004(2) 0.006(2)
C22 0.158(2) 0.101(2) 0.0629(12) -0.0032(11) -0.0063(14) 0.031(2)
C23 0.0619(10) 0.0696(11) 0.0727(11) -0.0062(9) -0.0179(8) -0.0085(8)
C24 0.0672(11) 0.0659(11) 0.102(2) -0.0157(10) -0.0170(10) -0.0056(9)
C25 0.0812(13) 0.0638(11) 0.111(2) 0.0142(11) -0.0116(12) -0.0056(10)
C26 0.0716(12) 0.0751(12) 0.0891(13) 0.0161(11) -0.0068(10) -0.0080(10)
C27 0.0573(9) 0.0709(11) 0.0732(11) 0.0078(9) -0.0091(8) -0.0087(8)
C28 0.0732(12) 0.0866(13) 0.0782(12) 0.0046(10) 0.0067(10) -0.0024(10)
C29 0.0819(12) 0.0655(10) 0.0676(11) -0.0016(8) 0.0062(9) 0.0105(9)
C30 0.0831(12) 0.0597(10) 0.0636(10) 0.0034(8) 0.0025(9) 0.0063(9)
C31 0.0869(12) 0.0574(9) 0.0628(10) 0.0001(7) -0.0015(9) 0.0073(9)
C32 0.0923(13) 0.0711(11) 0.0535(9) 0.0015(8) -0.0080(9) 0.0036(10)
C33 0.0971(15) 0.0893(14) 0.0669(11) -0.0125(10) -0.0192(10) -0.0081(11)
C34 0.0655(10) 0.0815(12) 0.0685(11) 0.0023(9) 0.0122(9) 0.0020(9)
C35 0.0753(12) 0.0774(13) 0.104(2) 0.0102(11) 0.0131(11) 0.0046(10)
C36 0.0879(15) 0.0759(13) 0.117(2) -0.0207(13) 0.0117(14) 0.0017(11)
C37 0.0733(12) 0.0936(15) 0.0888(14) -0.0289(12) 0.0062(11) -0.0055(11)

C38 0.0593(10) 0.0853(13) 0.0693(11) -0.0135(9) 0.0019(8) -0.0018(9)
 C39 0.0763(12) 0.109(2) 0.0734(12) -0.0095(11) -0.0129(10) 0.0037(12)
 C40 0.0937(14) 0.0883(14) 0.0611(11) -0.0034(9) -0.0099(10) 0.0132(11)
 C41 0.0948(14) 0.0819(13) 0.0654(11) -0.0190(9) -0.0078(10) 0.0031(11)
 C42 0.0933(14) 0.0812(12) 0.0552(10) -0.0050(9) -0.0056(9) 0.0077(10)
 C43 0.0998(15) 0.0950(14) 0.0533(10) -0.0146(9) 0.0016(10) 0.0088(12)
 C44 0.0967(15) 0.105(2) 0.0624(11) 0.0061(11) 0.0136(10) 0.0062(13)

geom special details

;

All esds (except the esd in the dihedral angle between two l.s. planes)
 are estimated using the full covariance matrix. The cell esds are taken
 into account individually in the estimation of esds in distances, angles
 and torsion angles; correlations between esds in cell parameters are only
 used when they are defined by crystal symmetry. An approximate (isotropic)
 treatment of cell esds is used for estimating esds involving l.s. planes.

;

loop

geom bond atom site label 1

geom bond atom site label 2

geom bond distance

geom bond site symmetry 2

geom bond publ flag

N1 C5 1.340(2) . ?
 N1 C1 1.342(2) . ?
 C1 C2 1.385(3) . ?
 C1 C11 1.498(3) . ?
 N2 C12 1.334(2) . ?
 N2 C16 1.341(3) . ?
 C2 C3 1.369(3) . ?
 N3 C23 1.337(2) . ?
 N3 C27 1.342(2) . ?
 C3 C4 1.369(3) . ?
 N4 C34 1.341(2) . ?
 N4 C38 1.342(2) . ?
 C4 C5 1.381(3) . ?
 C5 C6 1.498(3) . ?
 C6 C7 1.515(3) . ?
 C7 C8 1.526(3) . ?
 C8 C9 1.513(3) . ?
 C9 C10 1.504(2) . ?
 C10 C11 1.514(3) 2 577 ?
 C11 C10 1.514(3) 2 577 ?
 C12 C13 1.390(3) . ?
 C12 C22 1.506(4) . ?
 C13 C14 1.368(4) . ?

C14 C15 1.352(3) . ?
 C15 C16 1.390(3) . ?
 C16 C17 1.510(3) . ?
 C17 C18 1.485(13) . ?
 C17 C18' 1.613(14) . ?
 C18 C19 1.54(2) . ?
 C19 C20 1.524(11) . ?
 C20 C21 1.500(6) . ?
 C21 C22 1.711(5) 2 678 ?
 C18' C19' 1.53(2) . ?
 C19' C20' 1.499(13) . ?
 C20' C21' 1.532(7) . ?
 C21' C22 1.494(4) 2 678 ?
 C22 C21' 1.494(4) 2 678 ?
 C22 C21 1.711(5) 2 678 ?
 C23 C24 1.384(3) . ?
 C23 C33 1.506(3) . ?
 C24 C25 1.373(3) . ?
 C25 C26 1.364(3) . ?
 C26 C27 1.386(3) . ?
 C27 C28 1.502(3) . ?
 C28 C29 1.516(3) . ?
 C29 C30 1.506(3) . ?
 C30 C31 1.513(3) . ?
 C31 C32 1.510(3) . ?
 C32 C33 1.525(3) 2 667 ?
 C33 C32 1.525(3) 2 667 ?
 C34 C35 1.382(3) . ?
 C34 C44 1.508(3) . ?
 C35 C36 1.368(4) . ?
 C36 C37 1.375(4) . ?
 C37 C38 1.376(3) . ?
 C38 C39 1.505(3) . ?
 C39 C40 1.514(3) . ?
 C40 C41 1.514(3) . ?
 C41 C42 1.506(3) . ?
 C42 C43 1.515(3) . ?
 C43 C44 1.528(3) 2 566 ?
 C44 C43 1.528(3) 2 566 ?

loop

geom angle atom site label 1
 geom angle atom site label 2
 geom angle atom site label 3
 geom angle
 geom angle site symmetry 1
 geom angle site symmetry 3

geom angle publ flag
C5 N1 C1 118.92(15) . . ?
N1 C1 C2 121.1(2) . . ?
N1 C1 C11 115.2(2) . . ?
C2 C1 C11 123.7(2) . . ?
C12 N2 C16 119.1(2) . . ?
C3 C2 C1 119.4(2) . . ?
C23 N3 C27 118.9(2) . . ?
C2 C3 C4 119.8(2) . . ?
C34 N4 C38 118.8(2) . . ?
C3 C4 C5 118.4(2) . . ?
N1 C5 C4 122.4(2) . . ?
N1 C5 C6 115.79(15) . . ?
C4 C5 C6 121.7(2) . . ?
C5 C6 C7 110.93(15) . . ?
C6 C7 C8 114.6(2) . . ?
C9 C8 C7 113.2(2) . . ?
C10 C9 C8 112.3(2) . . ?
C9 C10 C11 115.7(2) . 2 577 ?
C1 C11 C10 110.95(15) . 2 577 ?
N2 C12 C13 121.6(2) . . ?
N2 C12 C22 116.0(2) . . ?
C13 C12 C22 122.3(2) . . ?
C14 C13 C12 118.8(2) . . ?
C15 C14 C13 120.0(2) . . ?
C14 C15 C16 119.2(2) . . ?
N2 C16 C15 121.3(2) . . ?
N2 C16 C17 116.7(2) . . ?
C15 C16 C17 121.9(2) . . ?
C18 C17 C16 112.2(5) . . ?
C16 C17 C18' 108.9(6) . . ?
C17 C18 C19 114.8(8) . . ?
C20 C19 C18 112.1(8) . . ?
C21 C20 C19 111.9(4) . . ?
C20 C21 C22 107.7(3) . 2 678 ?
C19' C18' C17 111.6(9) . . ?
C20' C19' C18' 114.4(8) . . ?
C19' C20' C21' 115.4(5) . . ?
C22 C21' C20' 106.7(4) 2 678 . ?
C21' C22 C12 127.7(3) 2 678 . ?
C12 C22 C21 96.4(2) . 2 678 ?
N3 C23 C24 121.9(2) . . ?
N3 C23 C33 115.7(2) . . ?
C24 C23 C33 122.5(2) . . ?
C25 C24 C23 119.1(2) . . ?
C26 C25 C24 119.2(2) . . ?
C25 C26 C27 119.5(2) . . ?

N3 C27 C26 121.4(2) . . ?
 N3 C27 C28 116.1(2) . . ?
 C26 C27 C28 122.5(2) . . ?
 C27 C28 C29 113.7(2) . . ?
 C30 C29 C28 114.5(2) . . ?
 C29 C30 C31 114.27(15) . . ?
 C32 C31 C30 115.1(2) . . ?
 C31 C32 C33 114.1(2) . 2 667 ?
 C23 C33 C32 113.1(2) . 2 667 ?
 N4 C34 C35 121.9(2) . . ?
 N4 C34 C44 115.2(2) . . ?
 C35 C34 C44 122.8(2) . . ?
 C36 C35 C34 119.0(2) . . ?
 C35 C36 C37 119.4(2) . . ?
 C36 C37 C38 119.2(2) . . ?
 N4 C38 C37 121.8(2) . . ?
 N4 C38 C39 115.5(2) . . ?
 C37 C38 C39 122.7(2) . . ?
 C38 C39 C40 112.3(2) . . ?
 C39 C40 C41 114.5(2) . . ?
 C42 C41 C40 114.7(2) . . ?
 C41 C42 C43 114.5(2) . . ?
 C42 C43 C44 114.2(2) . 2 566 ?
 C34 C44 C43 112.5(2) . 2 566 ?

refine diff density max 0.481
 refine diff density min -0.182
 refine diff density rms 0.037

"#=END"

Solid-State Structure Determination for [6.6](2,7)Fluorenophane, 8.

data 38a (Compound 1)

audit creation method	SHELXL
chemical name systematic	1,28:6,3:13,16:18,21-tetrametheno
dicyclopenta[a,k]2,7,8,9,10,11,12,17,22, 23,24,25,26,27,-tetradacahydro cyclooctacosane	
chemical formula moiety	?
chemical formula structural	'C38 H40'
chemical formula analytical	'C38 H40'
chemical formula sum	'C38 H40'
chemical formula weight	496.70
chemical melting point	513 K
chemical compound source	?

loop

atom type symbol	
atom type description	
atom type scat dispersion real	
atom type scat dispersion imag	
atom type scat source	
'C' 'C'	0.0033 0.0016
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'	
'H' 'H'	0.0000 0.0000
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'	

symmetry cell setting	Monoclinic
symmetry space group name H-M	P2(1)/n

loop

symmetry equiv pos as xyz

'x, y, z'

'-x+1/2, y+1/2, -z+1/2'

'-x, -y, -z'

'x-1/2, -y-1/2, z-1/2'

cell length a 14.537(2)
 cell length b 5.5063(9)
 cell length c 19.003(3)
 cell angle alpha 90.00
 cell angle beta 111.711(13)
 cell angle gamma 90.00
 cell volume 1413.2(4)
 cell formula units Z 2
 cell measurement temperature 294(2)
 cell measurement reflns used 28
 cell measurement theta min 4.55
 cell measurement theta max 12.42

exptl crystal description thin plate
 exptl crystal colour clear
 exptl crystal size max 1.6
 exptl crystal size mid 0.7
 exptl crystal size min 0.2
 exptl crystal density meas ?
 exptl crystal density diffn 1.167
 exptl crystal density method ?
 exptl crystal F 000 536
 exptl absorpt coefficient mu 0.065
 exptl absorpt correction type none
 exptl absorpt correction T min ?
 exptl absorpt correction T max ?

exptl special details

;
 ?
 ;

diffn ambient temperature 294(2)
 diffn radiation wavelength 0.71073
 diffn radiation type MoK α
 diffn radiation source 'fine-focus sealed tube'
 diffn radiation monochromator graphite
 diffn measurement device 'Siemens P4'
 diffn measurement method '\w scans'

```

diffn standards number      3
diffn standards interval count 97
diffn standards interval time ?
diffn standards decay %     ?
diffn reflns number        2953
diffn reflns av R equivalents 0.0187
diffn reflns av sigma/etl   0.0232
diffn reflns limit h min    -1
diffn reflns limit h max    17
diffn reflns limit k min    -1
diffn reflns limit k max    6
diffn reflns limit l min    -22
diffn reflns limit l max    21
diffn reflns theta min      2.21
diffn reflns theta max      25.01
reflns number total         2067
reflns number observed      1702
reflns observed criterion   >2sigma(I)

```

```

computing data collection    'Siemens XSCANS'
computing cell refinement    'Siemens XSCANS'
computing data reduction     'Siemens SHELXTL'
computing structure solution  'SHELXS-86 (Sheldrick, 1990)'
computing structure refinement 'SHELXL-93 (Sheldrick, 1993)'
computing molecular graphics  'Siemens SHELXTL'
computing publication material 'Siemens SHELXTL'

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refine special details

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Refinement on F^2 for ALL reflections except for 102 with very negative F^2 or flagged by the user for potential systematic errors. Weighted R-factors wR and all goodnesses of fit S are based on F^2 , conventional R-factors R are based on F , with F set to zero for negative F^2 . The observed criterion of $F^2 > 2\sigma(F^2)$ is used only for calculating R factor obs etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F , and R-factors based on ALL data will be even larger.

;

```

refine ls structure factor coef Fsqd
refine ls matrix type          full
refine ls weighting scheme
'calc w=1/[s^2(Fo^2)+(0.0683P)^2+0.2092P] where P=(Fo^2+2Fc^2)/3'
atom sites solution primary    direct
atom sites solution secondary  difmap
atom sites solution hydrogens  geom
refine ls hydrogen treatment   fixed

```

```

refine ls extinction method    SHELXL
refine ls extinction coef      0.0320(37)
refine ls extinction expression
'Fc^*=kFc[1+0.001xFc^2/l^3/sin(2\q)]^-1/4'
refine ls number reflns       1965
refine ls number parameters    173
refine ls number restraints    0
refine ls R factor all         0.0524
refine ls R factor obs         0.0428
refine ls wR factor all        0.1296
refine ls wR factor obs        0.1170
refine ls goodness of fit all  1.057
refine ls goodness of fit obs  1.096
refine ls restrained S all     1.092
refine ls restrained S obs     1.096
refine ls shift/esd max        0.000
refine ls shift/esd mean       0.000

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loop

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atom site label
atom site type symbol
atom site fract x
atom site fract y
atom site fract z
atom site U iso or equiv
atom site thermal displace type
atom site occupancy
atom site calc flag
atom site refinement flags
atom site disorder group
C10 C 0.53395(11) 0.9839(3) 0.15090(8) 0.0519(4) Uani 1 d . .
C9 C 0.62879(11) 0.9157(3) 0.14585(8) 0.0487(4) Uani 1 d . .
C5 C 0.74004(12) 1.0724(3) 0.08929(9) 0.0566(5) Uani 1 d . .
H5A H 0.75355(12) 1.1829(3) 0.05737(9) 0.068 Uiso 1 calc R .
C4 C 0.64969(11) 1.0796(3) 0.09776(8) 0.0484(4) Uani 1 d . .
C1 C 0.41384(13) 1.3072(4) 0.10687(9) 0.0631(5) Uani 1 d . .
H1A H 0.39033(13) 1.4435(4) 0.07667(9) 0.076 Uiso 1 calc R .
C8 C 0.69770(13) 0.7398(3) 0.18322(9) 0.0610(5) Uani 1 d . .
H8A H 0.68384(13) 0.6274(3) 0.21446(9) 0.073 Uiso 1 calc R .
C6 C 0.81102(12) 0.9009(3) 0.12818(9) 0.0573(5) Uani 1 d . .
C3 C 0.56551(12) 1.2550(3) 0.06509(9) 0.0543(5) Uani 1 d . .
H3A H 0.53294(12) 1.2331(3) 0.01082(9) 0.065 Uiso 1 calc R .
H3B H 0.58803(12) 1.4217(3) 0.07544(9) 0.065 Uiso 1 calc R .
C2 C 0.49753(12) 1.1877(3) 0.10615(8) 0.0526(5) Uani 1 d . .
C19 C 0.91267(13) 0.9001(4) 0.12379(11) 0.0713(6) Uani 1 d . .
H19A H 0.96174(13) 0.9152(4) 0.17469(11) 0.086 Uiso 1 calc R .
H19B H 0.91860(13) 1.0418(4) 0.09545(11) 0.086 Uiso 1 calc R .

```

C11 C 0.48359(14) 0.8922(4) 0.19488(9) 0.0675(5) Uani 1 d . .
 H11A H 0.50524(14) 0.7517(4) 0.22338(9) 0.081 Uiso 1 calc R .
 C12 C 0.40055(14) 1.0150(5) 0.19520(10) 0.0748(6) Uani 1 d . .
 H12A H 0.36727(14) 0.9558(5) 0.22504(10) 0.090 Uiso 1 calc R .
 C13 C 0.36527(13) 1.2231(4) 0.15263(10) 0.0682(6) Uani 1 d . .
 C7 C 0.78706(14) 0.7336(3) 0.17350(9) 0.0633(5) Uani 1 d . .
 H7A H 0.83275(14) 0.6137(3) 0.19803(9) 0.076 Uiso 1 calc R .
 C16 C 0.85597(14) 0.8373(5) -0.04732(11) 0.0801(6) Uani 1 d . .
 H16A H 0.91998(14) 0.9155(5) -0.03388(11) 0.096 Uiso 1 calc R .
 H16B H 0.81073(14) 0.9553(5) -0.04004(11) 0.096 Uiso 1 calc R .
 C17 C 0.86619(14) 0.6288(4) 0.00643(11) 0.0722(6) Uani 1 d . .
 H17A H 0.88926(14) 0.4873(4) -0.01265(11) 0.087 Uiso 1 calc R .
 H17B H 0.80130(14) 0.5906(4) 0.00696(11) 0.087 Uiso 1 calc R .
 C14 C 0.27878(14) 1.3623(5) 0.15930(12) 0.0927(8) Uani 1 d . .
 H14A H 0.29454(14) 1.4030(5) 0.21214(12) 0.111 Uiso 1 calc R .
 H14B H 0.27134(14) 1.5135(5) 0.13155(12) 0.111 Uiso 1 calc R .
 C18 C 0.93645(14) 0.6761(4) 0.08724(11) 0.0742(6) Uani 1 d . .
 H18A H 0.93580(14) 0.5358(4) 0.11791(11) 0.089 Uiso 1 calc R .
 H18B H 1.00304(14) 0.6924(4) 0.08755(11) 0.089 Uiso 1 calc R .
 C15 C 0.8193(2) 0.7707(6) -0.13038(12) 0.0959(8) Uani 1 d . .
 H15A H 0.8688(2) 0.6697(6) -0.13905(12) 0.115 Uiso 1 calc R .
 H15B H 0.8129(2) 0.9182(6) -0.15974(12) 0.115 Uiso 1 calc R .

loop

atom site aniso label
 atom site aniso U 11
 atom site aniso U 22
 atom site aniso U 33
 atom site aniso U 23
 atom site aniso U 13
 atom site aniso U 12

C10 0.0561(9) 0.0577(11) 0.0392(7) -0.0049(7) 0.0146(6) -0.0079(9)
 C9 0.0580(9) 0.0458(10) 0.0374(7) -0.0015(7) 0.0121(6) -0.0014(8)
 C5 0.0655(10) 0.0544(11) 0.0533(9) 0.0024(8) 0.0261(8) 0.0037(9)
 C4 0.0566(9) 0.0458(10) 0.0411(7) -0.0025(7) 0.0160(6) 0.0011(8)
 C1 0.0615(10) 0.0729(13) 0.0538(9) -0.0078(9) 0.0202(8) 0.0043(10)
 C8 0.0746(11) 0.0591(12) 0.0473(8) 0.0079(8) 0.0202(8) 0.0040(10)
 C6 0.0592(10) 0.0604(12) 0.0492(8) -0.0084(8) 0.0163(7) 0.0072(9)
 C3 0.0599(9) 0.0524(11) 0.0504(8) 0.0037(7) 0.0201(7) 0.0038(9)
 C2 0.0538(9) 0.0588(11) 0.0427(8) -0.0068(8) 0.0150(6) -0.0020(9)
 C19 0.0592(10) 0.0829(15) 0.0690(11) -0.0111(10) 0.0204(9) 0.0061(11)
 C11 0.0712(11) 0.0770(14) 0.0536(9) 0.0047(9) 0.0224(8) -0.0117(11)
 C12 0.0664(11) 0.107(2) 0.0578(10) -0.0094(11) 0.0312(9) -0.0164(12)
 C13 0.0584(10) 0.093(2) 0.0530(9) -0.0202(10) 0.0202(8) -0.0055(11)
 C7 0.0713(11) 0.0619(13) 0.0502(9) 0.0040(8) 0.0148(8) 0.0179(10)
 C16 0.0600(11) 0.109(2) 0.0744(12) 0.0059(12) 0.0284(9) -0.0089(12)
 C17 0.0781(12) 0.0742(14) 0.0768(12) -0.0104(11) 0.0434(10) 0.0031(11)

C14 0.0768(13) 0.135(2) 0.0722(12) -0.0356(14) 0.0350(10) 0.0068(14)
 C18 0.0700(11) 0.0841(15) 0.0731(11) 0.0014(11) 0.0318(9) 0.0200(11)
 C15 0.0704(12) 0.155(2) 0.0748(12) 0.0065(14) 0.0415(10) 0.0119(14)

geom special details

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All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

loop

geom bond atom site label 1

geom bond atom site label 2

geom bond distance

geom bond site symmetry 2

geom bond publ flag

C10 C2 1.389(2) . ?

C10 C11 1.393(2) . ?

C10 C9 1.466(2) . ?

C9 C8 1.386(2) . ?

C9 C4 1.395(2) . ?

C5 C4 1.382(2) . ?

C5 C6 1.392(2) . ?

C4 C3 1.501(2) . ?

C1 C13 1.387(3) . ?

C1 C2 1.388(2) . ?

C8 C7 1.379(2) . ?

C6 C7 1.391(3) . ?

C6 C19 1.511(2) . ?

C3 C2 1.514(2) . ?

C19 C18 1.517(3) . ?

C11 C12 1.386(3) . ?

C12 C13 1.387(3) . ?

C13 C14 1.517(3) . ?

C16 C17 1.507(3) . ?

C16 C15 1.513(3) . ?

C17 C18 1.520(3) . ?

C14 C15 1.514(3) 3 675 ?

C15 C14 1.514(3) 3 675 ?

loop

geom angle atom site label 1

geom angle atom site label 2

geom angle atom site label 3

geom angle

geom angle site symmetry 1
 geom angle site symmetry 3
 geom angle publ flag
 C2 C10 C11 119.6(2) . . ?
 C2 C10 C9 108.75(14) . . ?
 C11 C10 C9 131.5(2) . . ?
 C8 C9 C4 119.8(2) . . ?
 C8 C9 C10 131.65(15) . . ?
 C4 C9 C10 108.38(14) . . ?
 C4 C5 C6 120.5(2) . . ?
 C5 C4 C9 120.28(15) . . ?
 C5 C4 C3 129.42(14) . . ?
 C9 C4 C3 110.20(14) . . ?
 C13 C1 C2 120.0(2) . . ?
 C7 C8 C9 119.1(2) . . ?
 C7 C6 C5 118.1(2) . . ?
 C7 C6 C19 120.4(2) . . ?
 C5 C6 C19 121.4(2) . . ?
 C4 C3 C2 102.53(13) . . ?
 C1 C2 C10 120.9(2) . . ?
 C1 C2 C3 129.1(2) . . ?
 C10 C2 C3 109.85(14) . . ?
 C6 C19 C18 114.9(2) . . ?
 C12 C11 C10 118.5(2) . . ?
 C11 C12 C13 122.4(2) . . ?
 C1 C13 C12 118.5(2) . . ?
 C1 C13 C14 120.9(2) . . ?
 C12 C13 C14 120.5(2) . . ?
 C8 C7 C6 122.1(2) . . ?
 C17 C16 C15 115.5(2) . . ?
 C16 C17 C18 114.5(2) . . ?
 C15 C14 C13 115.1(2) 3 675 . ?
 C19 C18 C17 114.9(2) . . ?
 C16 C15 C14 114.6(2) . 3 675 ?

refine diff density max 0.173
 refine diff density min -0.149
 refine diff density rms 0.032

"#=END"

data pyl1fin (compound 2)

audit creation method	SHELXL
chemical name systematic	23,24-diazotricyclo[17.3.1.1(8,12)]tetracos- 1(230,8,10,12(24)19,21-hexane