# The Molecular Structure of 6, 8, 6 and Related Systems

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The crystal structures of 1,2;5,6-dibenzoocta-1,5-diene (1) ( $C_{16}H_{16}$ ,  $P_{2_1}/n A = 6.638$ , B = 6.892, C = 12.625 Å,  $\alpha = 90.00^{\circ}$ ,  $\beta = 96.58^{\circ}$ ,  $\gamma = 90.00^{\circ}$ , Z = 2); 6,7,13,14-tetrahydrodipyrido[1,2-a:1,2-e]diazocinium dibromide (11) ( $C_{14}H_{16}N_2Br_2\cdot 2H_2O$ ,  $P_{2_1}/n$ , A = 14.586, B = 6.146, C = 9.159 Å,  $\alpha = 90.00^{\circ}$ ,  $\beta = 104.16^{\circ}$ ,  $\gamma = 90.00^{\circ}$ , Z = 2); 1,2;5,6;9,10-tribenzododeca-1,5,9-triene (12) ( $C_{24}H_{24}$ ,  $P_{2_1}/n$ , A = 19.140, B = 5.122, C = 19.214 Å,  $\alpha = 90.00^{\circ}$ ,  $\beta = 107.12^{\circ}$ ,  $\gamma = 90.00^{\circ}$ , Z = 4) and 1,2;5,6;9,10;13,14tetrabenzohexadeca-1,5,9,13-tetraene (16) ( $C_{32}H_{32}$ ,  $P_{2_1}$ , A = 12.560, B = 33.665, C = 11.312 Å,  $\alpha = 90.00^{\circ}$ ,  $\beta = 90.38^{\circ}$ ,  $\gamma = 90.00^{\circ}$ , Z = 8) have been determined. The first two, which belong to the 6,8,6 ring system, exist in the solid state in the chair conformation, whereas both the chair and the boat forms are present in solution. The central twelve-membered ring of compound 12 exists in the crystal as an almost perfect  $C_2$  propeller, which is also the only observed conformation in solution. Finally, there are four different molecules of compound 16 in the crystal; however, they roughly correspond to the same conformation (and its enantiomer) [(+-+,-+-,++-,---) and (-+-,+-+, --+,+++)]. Partial and complementary to literature NMR (<sup>1</sup>H and <sup>13</sup>C) results agree with previous conformational analyses in solution.

The problem of the molecular structure of the so-called 6,8,6 ring systems, where 6 represents a six-membered aromatic compound, although important, remains practically unexplored. An examination of the literature, both through *Chemical Abstracts* and the CSD,<sup>1</sup> reveals that the simplest representative of this family, 1,2,5,6-dibenzoocta-1,5-diene (1), was studied in 1945 but in an incomplete way (see the following discussion).<sup>2</sup> The X-ray structures of three derivatives of the parent compound have been reported, 2,<sup>3</sup> 3<sup>4</sup> and 4.<sup>5</sup>



Related to 1 are the compounds in which the central cyclooctadiene ring has been modified. The structures of four compounds containing only carbon atoms have been described,  $5,^{6}$  $6,^{7}$  7,<sup>8</sup> and  $8.^{9}$ 

Another, much more common modification, is to introduce heteroatoms in the central ring. All reported structures have the heteroatoms at positions 5, 6, 11 and/or  $12.^{9,10}$  All these compounds have been the subject of careful conformational analyses in solution.<sup>11–14</sup> Considering that related 5,8,5 heterocyclic derivatives  $9^{15}$  and  $10^{16}$  have been described, it is surprising that the structure of the 6,7,13,14-tetrahydrodipyrido[1,2-*a*:1,2-*e*]diazocinium cation (11) has never been published.

It was thus decided to determine the structure of compounds 1 and 11. In the preparation of 1 from  $\alpha, \alpha'$ -dibromo-o-xylene,



a trimer, 1,2;5,6;9,10-tribenzododeca-1,5,9-triene (12), is also formed.<sup>2,12,17</sup>

It is necessary to examine in more detail the seminal work of Baker, Banks, Lyon and Mann.<sup>2</sup> Using molecular models and the scarce crystallographic data available, they discussed the molecular structure of 1 and 12. For 1, Dr. E. M. Davidson determined the unit cell dimensions (a = 6.80, b = 6.84, c = 13.63 Å,  $\beta = 100^{\circ}$ ), the space group (monoclinic,  $P2_1/c$ ) and the experimental density (0.986 g cm<sup>-3</sup>). She concluded that the



molecule has a centre of symmetry, a conclusion which is only consistent with the 'chair' conformation (1c) (for a discussion of the conformational behaviour of 1 in solution, see references  $^{11,18-20}$ ).



In the case of compound 12, Dr. M. Perutz determined that the crystals (needle-shaped with strong negative birefringence) are either orthorhombic or monoclinic. He concluded that of the four possible conformations proposed by Baker *et al.*,<sup>2</sup> only the propeller structure (12c) and the helical structure (12h) were consistent with the crystallographic data (for an NMR study of the conformation of this ring, see ref. 12).

The crystallographic results reported above, although incomplete, were of such relevance that they have been quoted frequently (24 times between 1974 and 1991). The conformations of related compounds will be discussed in detail later on; but for the moment they can be classified as planar, compound 7, chair (some authors call it *anti*), compounds 3, 4, 5, 9 and 10, and boat (also called tub), compounds 2, 6 and 8.

## Results

Synthesis.—Compound 11, a dibromide, was prepared by dimerization of 2-( $\beta$ -bromoethyl)pyridine, according to Boekelheide and Feely.<sup>21</sup> Compound 1 has been described several times. Baker *et al.*<sup>2</sup> obtained it by treating  $\alpha, \alpha'$ -dibromo-*o*xylene with sodium, and obtained 6% of 1 and a much smaller yield of 12. Using the same reaction, Cope and Fenton,<sup>17</sup> after optimizing the procedure, obtained up to 46% of 1 accompanied by small amounts of 12 and 1,2-di(*o*-tolyl)ethane 13. Müller and Röscheisen<sup>22</sup> increased the yield of 12 up to 35%, maintaining the yield of 1 at 40%. Compound 1 can also be obtained (40%) from xylene dianion and 1,2-dibromoethane.<sup>23</sup>

Actually, the Wurtz reaction between  $\alpha, \alpha'$ -dibromo-o-xylene and sodium is rather complex (see the discussion by Ollis and Stoddart).<sup>12</sup> Depending on the experimental conditions, either cyclic compounds 1 and 12 or acyclic hydrocarbons 13,<sup>2,12</sup> 14,<sup>12</sup> and 15<sup>12</sup> were found.

In our hands, the reaction proved even more complex. Working in pure dioxane as solvent (see Experimental section),



a mixture of cyclic compounds, including 1, 12, 16, 17, 18 and 19 was isolated (Table 1).

The  $R_f$  values vary smoothly with the ring size. Thus, using a mixture of hexane-diethyl ether (10:0.2), the  $R_f$  values follow the relationship  $R_f = 1.336-0.441\sqrt{n}$ ,  $r^2 = 0.997$  (*n* is the number of  $C_6H_4CH_2CH_2$  units). For n = 8, the equation predicts  $R_f = 0.09$ , which is compatible with the existence of this macrocycle in the last fraction. Only for n = 2, 3 and 4, were the compounds isolated in sufficient amounts to obtain suitable crystals by repeated crystallisation.



If the reaction is carried out in toluene, then o-xylene, 1,2-dio-tolylethane (13), 1,2-bis(o-methylphenethyl)benzene (14), 1,2bis(o-ethoxymethylphenyl)ethane (17) and  $\alpha, \alpha'$ -diethoxy-o-xylene (18), were isolated.



Compounds 17 and 18 result from the diethyl ether used in the work-up procedure (see Experimental section).

Table 2 summarizes the results obtained with the four compounds, while Figs. 1–4 are different views (drawn with Pluto)<sup>24</sup> of these compounds and their packing modes. Free areas and free volumes were calculated according to Gavezzotti.<sup>25</sup>

1,2;5,6-Dibenzoocta-1,5-diene (1) and Tetrahydrodipyrido-

**Table 1** Cyclic compounds obtained in the Wurtz reaction between  $\alpha, \alpha'$ -dibromo-o-xylene and sodium

							$R_{\rm f}^{\ a}$		
Compound	Formula	М	M.p./°C	Lit. m.p./°C	Yield (%)	MS, <i>M</i> * +	A	В	С
1 12 16 17 18 19	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	208.30 312.45 416.61 520.76 624.91 729.06 833.21	109–110 188.5–189 198–201 204–206	110–112 <sup>22</sup> 184–185 <sup>22</sup>	$\begin{array}{c} 62.0\\ 30.0\\ 5.0\\ 0.8\\ 0.4\\ < 0.4\\ 1.4\end{array}$	208.13 312.22 416.20 520.62 624.79 729.00	0.49 0.43 0.40 0.33 0.29 0.24 0.18	0.71 0.59 0.44 0.35 0.25 0.18 0.10	0.42 0.34 0.25 0.17 0.12 <0.1 <0.1

<sup>a</sup> A, hexane-benzene (10:6.5); B, hexane-diethyl ether (10:0.2); C, hexane-ethyl acetate (10:0.2).

 Table 2
 Crystallographic data for compounds 1, 11, 12 and 16

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	1	11	12	16
Formula	$C_{16}H_{16}$	$C_{14}H_{16}N_{2}Br_{2}\cdot 2H_{2}O$	$C_{24}H_{24}$	$C_{12}H_{32}$
Molecular mass	208.30	406.12	312.45	416.60
Space group	$P2_1/n$	$P2_1/n$	$P2_1/c$	$P2_1$
Â/Å	6.638(1)	14.586(3)	19.140(3)	12.560(2)
<i>B</i> /Å	6.892(1)	6.146(1)	5.122(1)	33.665(4)
Ć/Å	12.625(2)	9.159(2)	19.214(3)	11.312(2)
$\alpha/^{\circ}$	90.00	90.00	90.00	90.00
$\beta'/^{\circ}$	96.58(1)	104.16(1)	107.12(2)	90.38(1)
v/°	90.00	90.00	90.00	90.00
Ż	2	2	4	8
$D_c/\mathrm{g}~\mathrm{cm}^{-3}$	1.206	1.701	1.151	1.159
$V/Å^3$	573.8(2)	796.1(4)	1800.2(6)	4783.0(13)
F(000)	224	408	672	1792
λČu/Å	1.541 78	1.541 78	1.541 78	1.541 78
$\mu/\mathrm{cm}^{-1}$	4.73	65.49	4.52	4.55
Absorption	no	yes	no	yes
Crystallization from	ethanol	H <sub>2</sub> O	acetone	
	methanol	ethanol	ethanol-hexane	benzene
No. of reflections	924	1375	2304	6173
No. of parameters	78	128	232	302-313
$N_{\rm rif}/N_{\rm max}$	11.8	10.7	10.0	20.1
R	0.0574	0.0490	0.0589	0.0679
<i>R</i>	0.0823	0.0592	0.0710	0.0714
Free area/Å <sup>2</sup>	141.48	219.63	352.05	1651.27 "
Free volume/Å <sup>3</sup>	118.87	158.76	537.72	1741.85 <i>°</i>
Molecular volume/Å <sup>3</sup>	113.73	159.34	315.62	1520.56 <sup>a</sup>
Packing coefficient	0.793	0.801	0.701	0.636 ª
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<sup>a</sup> These values refer to calculation performed with the contributions of only 32 carbon atoms of the  $C_{32}H_{32}$  compound, because the dimensions of program OPEC did not permit the introduction of more than 128 atoms, so that the 32 hydrogens of the four independent molecules have been discarded.

[1,2-a:1,2-e]diazocinium Dibromide (11).-Bond distances and angles are in the range usually reported for this class of compound.<sup>1</sup> For instance, bond angles in the bridges are wider than standard sp<sup>3</sup> angles, but this is a general feature of cyclophanes.<sup>5</sup> The six-membered ring forms an angle of 0.86° with the plane through C(1)-C(2)-C(7)-C(8) in 1, and angle of  $0.63^{\circ}$  with the plane N(1)-C(2)-C(7)-C(8) in 11, whereas the angle the same six-membered rings make with the plane constructed with the C(7)-C(8) atoms and the corresponding centrosymmetric C(7')-C(8') are 61.49° and 62.98°, respectively, in the same compounds. The Br atom is 3.446 Å distant from the plane  $N(1) \cdots C(6)$  in 11 as in the bis(pyrazolo)tetrazocinium dibromide (10).<sup>16</sup> Both structures have a chair conformation, their sideviews [Fig. 1(b) and 2(b)] being perfectly superimposable, confirming the hypothesis made in the late work of Baker (note small differences with regard to Davidson's work in the space group, cell dimensions and density).<sup>2</sup>

Concerning the packing of compound 1, there are no carboncarbon intermolecular distances less than 3.5 Å and the closest carbon-hydrogen intermolecular distance is 2.83 Å. That of the salt 11 is more interesting. Owing to the presence of the water molecule and the bromine ion, possible hydrogen bonds are given in Table 3.

1,2;5,6;9,10-Tribenzododeca-1,5,9-triene (12).—Compound  $C_{24}H_{24}$  shows two rings quite parallel [Figs. 3(a) and (b)], the angle between them being 3.71°, while the third one makes quite similar angles with the other two (19.1 and 21.7°, respectively); in this way the molecule assumes the conformation 12c corresponding to the so-called propeller structure. Here again, the expectation based on Perutz's report and the results of molecular models was verified; of the possible conformations the asymmetric one,  $C_2$ , is present in the solid state.

Concerning the packing of this molecule, the minimum intermolecular distances are C(3)-C(12) = 3.500 Å and C(24)-H(23) = 2.774 Å.

1,2;5,6;9,10;13,14-*Tetrabenzohexadeca*-1,5,9,13-*tetraene* 16.—In  $C_{32}H_{32}$  the space group is non-centrosymmetric











Fig. 1 (a) Perspective view of compound 1 ( $C_{16}H_{16}$ ) with the atomic numbering scheme used; (b) sideview of molecule 1; (c) packing diagram of compound 1 with unit cell outlines

whereas for the previous three compounds it was centric. This compound has been solved in space group  $P2_1$  with four independent molecules [Figs. 4(a)-(d)] in the unit cell. The relevant torsional angles for the central sixteen-membered ring are collected in Table 4.



(b)







Fig. 2 (a) Perspective view of compound 11 ( $C_{14}H_{16}N_2Br_2\cdot 2H_2O$ ) with atomic labelling; (b) sideview of molecule 11; (c) projection along the b axis of the atomic arrangement of the molecule 11

Two by two (1 and B, on one hand, and A and C, on the other) these molecules are nearly superimposable by a combination of a translation and a rotation. Furthermore, the averaged



(b)



C(13)



Fig. 3 (a) Molecular structure of compound 12  $(C_{24}H_{24})$  viewed from the direction giving the minimum overlap, with the atomic numbering scheme used; (b) sideview of molecule 12; (c) perspective view of the contents of the unit cell of compound 12 along the b axis

values for 1/B and A/C pairs are very close but for the sign. Since two conformations with the same angles and opposite signs correspond to a pair of enantiomers,<sup>11,12,19</sup> we can conclude that only one conformation (150.2, -172.9, 88.3; -105.1, 171.8, -105.1, 170.

Table 3 Some significant distances and angles pertaining to be hydrogen bonding in salt 11

	Distance/Å	Angle/°
$C(6) \cdots O(9)^a$	3.340(6)	<u> </u>
$O(9) \cdots Br^{b}$	3.351(4)	
$O(9) \cdots Br^{c}$	3.353(4)	
$C(6) \cdots H(6)$	1.08(4)	
$O(9) \cdots H(1)O(9)$	1.07(10)	
$O(9) \cdots H(2)O(9)$	0.94(7)	
$H(6) \cdots O(9)^{a}$	2.278(6)	
$H(1)O(9) \cdots Br^{b}$	2.329(11)	
$H(2)O(9) \cdots Br^{c}$	2.561(8)	
$C(6)-H(6)\cdots O(9)^{a}$		167.1(4)
$O(9) - H(1)O(9) \cdots Br^{b}$		159.5(6.9)
$O(9)-H(2)O(9)\cdots Br^{c}$		169.4(6.9)

<sup>a</sup> Symmetry operator, -x + 1, -y, -z + 1. <sup>b</sup> Symmetry operator  $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$ . <sup>c</sup> Symmetry operator x, y + 1, z.

Table 4 Dihedral angles of the central sixteen-membered ring for the four independent molecules of compound  $C_{32}H_{32}$  (16)



	Molecule						
Dihedral angle	1	A	В	С			
C(1)-C(2)-C(3)-C(4)	153.54	- 151.98	146.31	- 148.99			
C(2)-C(3)-C(4)-C(5)	- 172.93	170.58	- 174.19	173.78			
C(3)-C(4)-C(5)-C(6)	89.64	- 86.38	88.72	- 88.33			
C(5)-C(6)-C(7)-C(8)	- 102.20	101.82	- 106.50	110.07			
C(6)-C(7)-C(8)-C(9)	173.97	174.39	168.92	- 169.89			
C(7)-C(8)-C(9)-C(10)	- 173.82	173.84	- 173.37	168.88			
C(9)-C(10)-C(11)-C(12)	81.52	- 84.22	83.58	- 85.55			
C(10)-C(11)-C(12)-C(13)	67.13	- 62.52	66.06	- 63.74			
C(11)-C(12)-C(13)-C(14)	- 108.85	102.01	- 101.70	103.52			
C(13)-C(14)-C(15)-C(16)	- 92.51	88.43	-93.46	97.06			
C(14)-C(15)-C(16)-C(1)	- 163.89	163.71	-160.71	161.47			
C(15)-C(16)-C(1)-C(2)	- 97.69	100.63	-99.34	95.93			

-172.5; 83.7,64.8, -104.0; -92.8, -162.4, -94.8) and its enantiomer is present in the solid state. Nevertheless, in this very flexible ring system, the aforementioned conformation is not simply related to a conformation of high symmetry.

As regards the packing of this compound, owing to the great number of atoms (128 C and 128 H) the calculation of all intermolecular contacts is beyond the capabilities of the PARST program (maximum number of atoms = 200). Calculations performed with only 128 carbon atoms show that here also there are no intermolecular contacts of less than 3.5 Å, the shortest one being 3.568 Å.

NMR Spectroscopy.—Since <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy have already been used for studying the thermodynamic and kinetic aspects of the conformation of rings 1 and 12, our contribution will be only complementary. A brief summary of the literature results is reported in Table 5.

The main difference between structures 12c and 12d is that the former should present two identical ABCDs and one AA'BB' system, for both phenyl rings and ethanes, whereas the latter should present three identical AA'BB' for phenyls and for



Fig. 4 Best perspective view of the independent molecule 1 of compound  $16(C_{32}H_{32})$  with the atomic numbering scheme used; (b) best perspective view of the independent molecule A of compound 16 with the atomic numbering scheme used; (c) best perspective view of the independent molecule B of compound 16 with the atomic numbering scheme used; (d) best perspective view of the independent molecule C of compound 16 with the atomic numbering scheme used; (e) perspective view of the unit cell of compound 16.



12d

Compound	Thermodynamic	Kinetic	
1 20	In CDCl <sub>3</sub> -CS <sub>2</sub> (2:3) at 183 K a mixture of 60% 1 <b>b</b> -40% 1 <b>c</b>	$1b \Longrightarrow 1c$ $1b \Longrightarrow 1b$	$\Delta G^{\ddagger}$ 10.0 kcal mol <sup>-1</sup> $\Delta G^{\ddagger}$ 7.8 kcal mol <sup>-1</sup>
	In CD <sub>2</sub> Cl <sub>2</sub> C <sub>3</sub> H <sub>6</sub> (2:1) at 147 K 80% <b>1b</b> -20% 1c		
<b>12</b> <sup>19</sup>	Only the propeller structure 12c	$12c \Longrightarrow 12c$	$\Delta G^{\ddagger}$ 10.0 kcal mol <sup>-1</sup>

12c

**Table 6** <sup>1</sup>H NMR data ( $\delta$  in ppm, J in Hz) of compounds 1, 11, 12 and 16

1b

Compound	Solvent	T/K	'H NMR data
1	CDCl <sub>3</sub> <sup><i>a</i></sup>	298	CH <sub>2</sub> : 3.06(s); C <sub>6</sub> H <sub>4</sub> : AA'BB', $\delta_{A} = 6.98$ , $\delta_{B} = 7.00$ $J_{AB} = 7.32$ , $J_{AB'} = 1.22$ , $J_{AA'} = 0.46$ , $J_{BB'} = 7.44$
11	$[^{2}H_{6}]Me_{2}SO^{a}$	353	$CH_2(5)$ : 4.07, $CH_2(6)$ : 5.24, $^{b}J_{AB} = 6.4$ ; 9.14( $H_1$ ), 8.06 ( $H_2$ , $H_3$ ), 8.52( $H_4$ ), $^{b}J_{12} = 5.8$ , $J_{34} = 7.5$
	CDCl <sub>3</sub> "	298	CH <sub>2</sub> : 3.03(s); C <sub>6</sub> H <sub>4</sub> : AA'BB', $\delta_{A} = 7.22$ , $\delta_{B} = 7.32$ $J_{AB} = 7.43$ , $J_{AB'} = 1.29$ , $J_{AA'} = 7.42$ , $J_{BB'} = 0.42$
12	$\mathrm{CD}_{2}\mathrm{Cl}_{2}+\mathrm{CS}_{2}\left(2\!:\!1\right)^{a}$	170	$\begin{array}{l} {\rm CH_2CH_2: AA'BB' + [ABCD]_2} \\ {\rm AA'BB', \delta_A = \delta_{A'} = 2.92, \delta_B = \delta_{B'} = 3.12, J_{gem} \approx -13, J_{gauche} \approx 7, J_{trans} \approx 13} \\ {\rm ABCD, \delta_A = 2.72, \delta_B = 2.74, \delta_C = 3.17, \delta_D = 3.29, J_{gem} \approx -13, J_{gauche} \approx 3, J_{trans} \approx 14} \\ {\rm C_6H_4: [ABCD]_2 + AA'BB', 7.17-7.28 \ (m, 10 \ H), 7.37 \ (d, 2 \ H, J_0 = 7.0)} \end{array}$
16	CDCl <sub>3</sub> <sup><i>a</i></sup>	298	CH <sub>2</sub> : 2.89(s), C <sub>6</sub> H <sub>4</sub> : AA'BB', $\delta_{A} = 7.14$ , $\delta_{B} = 7.24$
o-Xylene	CDCl <sub>3</sub>	298	CH <sub>3</sub> : 2.24; C <sub>6</sub> H <sub>4</sub> : AA'BB', $\delta_A = 7.08$ , $\delta_B = 7.11$ (broadened due to coupling with the CH <sub>3</sub> )

<sup>a</sup> 500 MHz; <sup>b</sup> very broad at 298 K.

Table 7 <sup>13</sup>C NMR data (δ in ppm, <sup>1</sup>H-<sup>13</sup>C coupling constants in Hz) of compounds 1, 11, 12 and 16

Compound	Solvent	T/K	C <sub>1</sub>	C <sub>2</sub>	C <sub>3</sub>	C <sub>4</sub>	C <sub>4a</sub>	C <sub>5</sub>	C <sub>6</sub>
1	CDCl <sub>3</sub>	298	129.6 $^{1}J = 157.9$	126.1 ${}^{1}J = 159.4$ ${}^{3}I = 6.7$			140.5	$^{35.1}_{J} = 128.9$	
1c −35% 1b −65%	[ <sup>2</sup> H <sub>6</sub> ]Acetone	168	130.6 130.6	127.1 127.1			139.5 143.9	37.2 35.4	
11	[ <sup>2</sup> H <sub>6</sub> ]Me <sub>2</sub> SO	313	$^{147.6}_{^{1}J} = 190.5$	$^{128.6}_{^{1}J} = 176.0$	${}^{148.0}_{}^{}J = 171.1$	$^{132.1}_{^{1}J} = 177.3$	153.7 <i>ª</i>	56.4 <i>ª</i>	${}^{34.2}_{}^{}$ ${}^{1}J = 133.9$
12	CDCl <sub>3</sub>	298	$^{130.4}_{^{1}J} = 156.8$	${}^{1}J_{}^{0}=160.0$ ${}^{3}J_{}^{0}=7.7$			140.0	${}^{37.3}_{J} = 129.1$	
16	CDCl <sub>3</sub>	298	${}^{128.7}_{}^{}_{J} = 157.5$	${}^{126.3}_{J} = 159.8$ ${}^{3}J = 7.2$			139.5	${}^{33.3}_{J} = 130.2$	
o-Xylene	CDCl <sub>3</sub>	298	$129.5 {}^{1}J = 157.0 {}^{2}J = 4.6 {}^{3}J = 4.6 {}^{4}J = 1.9 (CH_{3})$	$125.8  {}^{1}J = 159.7  {}^{3}J = 6.5 $			136.4	${}^{19.7}_{}^{}_{J} = 125.6$	

<sup>a</sup> Broad Signals at 298 K.

Table 8 Conformation of the central ring in 6,8,6 systems in the solid state

Compound	Torsion angles/ $^{\circ}$	Conformation	Averaged values/°
1 Chair <sup>25</sup> (C)	$\frac{74.5, -109.3, 74.5}{-74.5, 109.3, -74.5}$	С	74.5, -109.3, 74.5
1 Twist-boat <sup>25</sup> (TB	$\frac{89.1, -26.5, -53.9}{-53.9, -26.5, -89.1}$	ТВ	89.1, -26.5, -53.9
1 Boat <sup>25</sup> (B)	$\frac{74.3, 0, -74.3}{-74.3, 0, -74.3}$	В	74.3, 0, -74.3
1 (This work)	$\frac{74,109, 73}{-73,109,-74}$	С	73.3, -109, 73.5
<b>2</b> <sup>3</sup>	$\frac{74, -8, -67}{93, -37, -44}$	ТВ	84, -22, -56
34	$\frac{73, -100, 70}{-72, 110, -76}$	C	73, -105, 73
<b>4</b> <sup>5</sup>	$\frac{74, -110, 73}{-73, 110, -74}$	C	73.4, -110, 73.4
915	$\frac{69, -101, -74}{-74, -101, -69}$	С	72, -101, 72
1016	$\frac{72, -105, 75}{-75, 105, -72}$	С	73, —105, 73
11 (This work)	$\frac{74, -113, 76}{-74, 113, -76}$	С	75, -113, 75

ethane fragments. Otherwise, the systems are quite similar, for instance, the ethane fragments are always in the staggered conformation (Ar–C–C–Ar dihedral angle near  $\pi/2$ ). Our results are summarized in Tables 6 (<sup>1</sup>H NMR data) and 7 (<sup>13</sup>C NMR data).

The aromatic AA'BB' systems have been analysed and assigned using the small coupling between *ortho* protons [H(1), H(4), H(7), H(10)] and the CH<sub>2</sub> protons. This coupling is also present in *o*-xylene; for this compound the protons of Table 6 and the carbons of Table 7 (assigned according to Kalinowski),<sup>26</sup> have been connected through a 2D ( $^{1}H^{-13}C$ ) correlated spectrum.

The complete analysis of the <sup>1</sup>H NMR spectrum of 12 at 170 K is very complex, even at 500 MHz, owing to couplings between the aromatic ortho protons and the methylene protons and also between the methylene protons of different ethane fragments.<sup>27</sup> Nevertheless, it clearly shows two identical ethane groups in the form of an ABCD system and the third one as a quasi degenerate AA'BB' system, both with staggered-like coupling constants (conformation 12c, Table 5). The <sup>13</sup>C NMR spectrum of compound 1 (Table 7) has not previously been reported. Finally, the dipyridodiazocinium dibromide (11) shows in NMR spectroscopy a dynamic behaviour which considerably enlarges some signals; however, its low solubility in common solvents and its low stability, prevents further studies. Subsequently, for compound 12 a chair-chair interconversion is very unlikely, the broadening probably involving boat or twist-boat forms.

#### Discussion

The 6,8,6 systems crystallize preferably in the chair conformation, compound **2** being the only exception. In Table 8 there is a summary of all available information.

It is noteworthy how accurate Allinger's calculations were,

using his well-known force-field method.<sup>28</sup> The dibenzooctadiene 1 is an almost perfect chair (1c) in agreement with Davidson's inference. The twist-boat conformation of compound 2 in the Allinger sense corresponds to a situation intermediate between the boat (-0+-0+) and the transition state FB1 (0-+0-+) according to Ollis' definition.<sup>19</sup>

The twelve-membered ring of compound 12 in the solid state also has a conformation consistent with one of Perutz's hypothesis and, in this case, coincident with the conformation present in solution (Table 5). It is an almost perfect  $C_2$  propeller, which, according to Ollis' notation (-+-,+-+,+-+) [in fact, it can be better described as  $(-\pi/2-,+\pi/2+,+\pi/2+)$ , since the central Ar-C-C-Ar bond has an almost 180° dihedral angle] can be described as (-103.63,+175.53,-93.28;+88.93,-173.12,+84.55; +82.56,-173.79,+80.49). Simple mechanical calculations performed with the DTMM program,<sup>29</sup> show that 12c is indeed the minimum energy conformation  $(-23.93 \text{ kJ mol}^{-1})$ . Other conformations such as  $D_3$ (12d, see Table 5) and those described by Ollis *et al.*<sup>12</sup> such as  $C_s$  $(+8.86 \text{ kJ mol}^{-1})$ ,  $C_{3v}$   $(+25.70 \text{ kJ mol}^{-1})$  and  $C_{3h}$   $(+59.12 \text{ kJ} mol^{-1})$  are much higher in the energy scale.

Finally, the sixteen-membered central ring of compound **16** is too flexible to be studied by DNMR spectroscopy (nothing is observed at 200 MHz at temperatures as low as 170 K). Views of the conformation present in the solid state are represented in Figs. 4(a)-(d) and the packing in Fig. 4(e).

### Experimental

Chemistry.—Melting points were recorded on a Büchi 530 apparatus and are uncorrected. Mass spectra were carried out on VG-12-250 at 70 eV, Hewlett-Packard GC/MS 5985 and LC/MS 5988A (interface HP1090 LC) spectrometers. The detection mode for the Hewlett-Packard spectrometers was the flow injection particle beam interface. When chemical

 Table 9
 Atomic coordinates for compounds 1, 11, 12 and 16

$ \begin{array}{c} 1 \\ 1 \\ (1) \\ (2) \\ (1) \\ (2$	Atom	x/A	<i>y</i> / <i>B</i>	z/C	Atom	x/A	<i>y</i> / <b>B</b>	z/C
$ \begin{array}{c} C(1) & -0.625  S0(14) & 0.227  S(17) & 0.421  24(10) & C(7) & 0.229  10(24) & 0.086  6(25) & 0.470  32(14) \\ C(2) & -0.226  90(14) & 0.378  60(17) & 0.407  94(10) & C(1A) & -0.065  50(14) & -0.227  64(17) & 0.378  56(17) & 0.337  321(10) & 0.665  56(14) & -0.227  64(17) & 0.378  56(10) \\ C(6) & 0.110  17(14) & 0.356  42(17) & 0.337  27(10) & C(2A) & 0.116  24(27) & -0.150  24(25) & 0.446  90(15) \\ C(6) & 0.110  17(14) & 0.356  42(17) & 0.337  27(10) & C(2A) & 0.116  24(27) & -0.150  24(25) & 0.446  90(15) \\ C(7) & 0.210  57(17) & 0.210  57(17) & 0.217  94(17) & 0.227  94($	1							
$\begin{array}{c} C(2) &0.122\ 27(14) & 0.24\ 98(17) & 0.49\ 78(10) & C(18) &0.175\ 24(27) & 0.102\ 0.223\ 78\ 75(10) \\ C(4) &0.223\ 81(14) & 0.446\ 85(17) & 0.23\ 21(10) & C(1A) &0.05\ 55(14) &0.23\ 81(15) & 0.59\ 77(14) \\ C(5) &0.23\ 74(14) & 0.46\ 65(17) & 0.33\ 74(10) & C(1A) &0.05\ 65(14) &0.246\ 81(25) & 0.59\ 77(14) \\ C(6) & -0.112\ 77(14) & 0.35\ 42(17) & 0.33\ 77(10) & C(1A) &0.125\ 91(26) &0.12\ 91(26) &0.125\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) &0.12\ 91(26) & -0.22\ 91(26) & -0.12\ 91(26) & -0.22\ 91(26) & -0.12\ 91(26) & -0.22\$	C(I)	0.065.50(14)	0 227 65(17)	0.421.24(10)	C(7)	0 223 91(24)	0.086.63(25)	0 470 93(14)
$ \begin{array}{c} C(2) &$	C(2)	-0.122.07(14)	0.24848(17)	0.459.75(10)	C(8)	-0.176.24(27)	0.130.28(25)	0.553.01(13)
$ \begin{array}{c} C(4) &0.22 3 71(4) & -0.48 58(7) & -0.23 2 71(6) & -C(2A) & -0.22 97(4) & -0.28 84(7) & -0.52 97(4) \\ C(5) & -0.037 71(4) & -0.46 58(7) & -0.33 72(0) & C(A) & -0.72 297(2) & -0.190 28(2) & -0.446 997(3) \\ \hline \\ C(5) & -0.037 71(4) & -0.465 62(7) & -0.33 72(0) & C(A) & -0.72 297(2) & -0.192 81(6) & -0.446 997(3) \\ \hline \\ C(7) & -0.038 98(2) & -0.118 97(4) & -0.35 70(5) & C(7) & -9.91 65(3) & -0.152 10(9) & -0.77 90(47) \\ C(2) & -0.56 68(23) & -0.070 13(6) & -0.344 96(41) & O(9) & -0.269 26(24) & -0.688 81(7) & -0.428 77 61(40) \\ C(3) & -57 86(40) & -0.045 508(40) & 0.144 40(41) & O(9) & -0.269 26(24) & -0.688 81(7) & -0.57 81(40) \\ C(4) & 0.57 86(40) & -0.045 508(40) & 0.134 40(7) & C(2A) & 0.483 32(24) & -0.070 13(6) & 0.555 44(4) \\ C(6) & 0.679 18(40) & -0.045 508(40) & 0.134 40(7) & C(2A) & 0.483 32(24) & -0.070 13(6) & 0.555 44(4) \\ C(6) & 0.649 19(26) & -0.257 01(70) & 0.337 74(46) & C(BA) & 0.491 51(23) & -0.227 99(57) & 0.582 34(6) \\ C(6) & 0.649 19(26) & -0.257 01(70) & 0.337 44(46) & C(BA) & 0.491 51(23) & -0.227 99(57) & 0.582 34(6) \\ C(7) & 0.387 58(8) & 0.054 73(24) & 0.079 99(12) & C(15) & 0.998 17(8) & 0.055 11(11) & 0.861 58(8) \\ C(7) & 0.381 51(13) & 0.064 71(3) & 0.611 29(13) & C(16) & 0.944 26(8) & -0.040 72(13) & 0.988 56(8) \\ C(6) & 0.739 58(8) & 0.254 153(8) & 0.057 161(1) & 0.253 28(6) & -0.040 72(13) & 0.988 56(8) \\ C(6) & 0.479 58(8) & 0.226 153(8) & 0.057 16(15) & C(72) & 0.337 53(8) & 0.344 86(8) & -0.645 34(20) & -0.537 97(7) \\ C(1) & 0.568 53(27) & 0.444 65(10) & -0.310 05(22) & C(12) & 0.337 53(8) & 0.344 86(8) & -0.645 34(20) & -0.537 97(7) \\ C(1) & 0.568 53(27) & 0.444 65(10) & -0.310 05(22) & C(12) & 0.337 53(8) & 0.344 8(8) & 0.455 38(7) \\ C(1) & 0.568 53(27) & 0.444 63(10) & -0.310 05(22) & C(12) & 0.337 53(8) & 0.344 8(10) & -0.357 24(7) \\ C(10) & 0.568 53(27) & 0.444 63(10) & -0.310 05(22) & C(12) & 0.337 53(8) & 0.344 8(10) & -0.357 24(7) \\ C(11) & 0.797 20(14) & 0.237 87(16) & 0.737 97(16) & 0.737 97(16) & 0.737 97(16) & 0.737 97(16) & 0.737 97(16) & 0.737 97(16) & 0.7$	C(2)	-0.12207(14) -0.26408(14)	0.24040(17)	0.43975(10)	C(1A)	-0.06550(14)	-0.227.65(17)	0.55571(15) 0.57876(10)
$ \begin{array}{c} C(3) & -0.022 \ 74(14) & 0.046 \ 02(17) & 0.247 \ 02(16) & -0.223 \ 01(24) & -0.085 \ 02(25) & 0.529 \ 07(16) \\ C(6) & 0.110 \ 17(14) & 0.326 \ 02(17) & 0.333 \ 72(10) & C(8A) & 0.175 \ 74(27) & -0.150 \ 28(25) & 0.477 \ 78(047) \\ R(1) & 0.245 \ 77(3) & 0.120 \ 07(7) & 0.333 \ 70(5) & C(7) & 0.315 \ 02(25) & 0.259 \ 07(16) & 0.258 \ 97(23) & 0.227 \ 98(17) & 0.477 \ 78(047) \\ C(2) & 0.561 \ 86(24) & 0.070 \ 18(6) & 0.422 \ 38(40) & C(7) & 0.338 \ 32(24) & 0.0188 \ 88(172) & 0.428 \ 78(44) \\ C(3) & 0.571 \ 85(20) & 0.070 \ 18(6) & 0.244 \ 96(4) & O(9) & 0.259 \ 28(24) & 0.0188 \ 88(172) & 0.248 \ 78(44) \\ C(4) & 0.619 \ 18(24) & 0.070 \ 18(6) & 0.221 \ 14(40,7) & C(2A) & 0.488 \ 32(24) & 0.0178 \ 18(6) & 0.257 \ 88(16) \\ C(6) & 0.687 \ 78(27) & -0.277 \ 95(7) & 0.212 \ 33(49) & C(7A) & 0.488 \ 32(25) & 0.153 \ 18(6) & 0.252 \ 38(4) \\ C(1) & 0.845 \ 95(6) & 0.265 \ 09(31) & 0.733 \ 78(46) & C(1A) & 0.980 \ 87(6) & 0.350 \ 9((13) & 0.852 \ 38(4) \\ C(2) & 0.866 \ 80(10) & 0.025 \ 60(31) & 0.733 \ 33(6) & C(11) & 0.882 \ 56(6) & 0.350 \ 9((13) & 0.852 \ 38(4) \\ C(2) & 0.866 \ 80(10) & 0.025 \ 60(31) & 0.733 \ 70(76) & C(11) & 0.882 \ 56(6) & 0.350 \ 9((31) & 0.852 \ 28(4) \\ C(1) & 0.845 \ 95(6) & 0.265 \ 95(1) & 0.737 \ 97(6) & C(12) & 0.875 \ 18(6) & 0.350 \ 18(1) & 0.881 \ 18(10) \ 80(10) \ $	C(3)	-0.204 98(14)	0.37808(17)	0.32321(10)	C(1A)	-0.00350(14)	-0.22703(17) 0.24848(17)	0.578 70(10)
$ \begin{array}{c} C(6) & 0.110 \ 17(14) & 0.336 \ 42(17) & 0.333 \ 72(10) & C(8A) & 0.176 \ 24(27) & -0.130 \ 28(25) & 0.446 \ 69(15) \\ I \\ H(1) & 0.124 \ 57(2) & 0.120 \ 0777 & 0.353 \ 70(5) & C(7) & 0.591 \ 65(25) & -0.152 \ 81(69) & 0.577 \ 80(47) \\ N(1) & 0.660 \ 86(22) & -0.111 \ 89(45) & 0.422 \ 98(40) & C(8) & 0.388 \ 98(23) & 0.227 \ 98(37) & 0.417 \ 55(44) \\ C(3) & 0.571 \ 88(50) & 0.053 \ 68(50) \ 0.257 \ 88(50$	C(4)	-0.22031(14) -0.03274(14)	0.460.83(17)	0.32321(10) 0.28470(10)	$C(2\mathbf{A})$	-0.223.91(24)	-0.24846(17) -0.08663(25)	0.540 25(10)
$ \begin{array}{c} 11 \\ 11 \\ 11 \\ 11 \\ 11 \\ 11 \\ 11 \\ 11$	C(6)	0.11017(14)	0.33642(17)	0.333 72(10)	C(8A)	0.176 24(27)	-0.13028(25)	0.446 09(13)
	11		· · · ·		, , , , , , , , , , , , , , , , , , ,	· · · ·	· · · · ·	, , , , , , , , , , , , , , , , , , ,
$ \begin{array}{c} N(1) & 0.000 \ \ Se(22) & -0.111 \ \ Se(45) & 0.242 \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ $	$\mathbf{Br}(1)$	0 124 57(3)	0 120 07(7)	0 353 70(5)	C(7)	0 591 65(25)	-0.152.81(69)	0 577 80(47)
$ \begin{array}{c} C(2) \\ C(3) \\ C(3) \\ C(3) \\ C(4) \\ C(4) \\ C(4) \\ C(5) \\ C(5) \\ C(5) \\ C(6) \\ C(7) \\ C(6) \\ C(7) \\ C(6) \\ C(7) \\ C(6) \\ C(7) \\ C$	N(1)	0.124 57(5)	-0.111.89(45)	0.33370(3)	C(8)	0.59105(23)	0.13201(0)) 0.22799(57)	0.37760(47) 0.41762(44)
$ \begin{array}{c} C(3) & 0.571 \ 8530 & 0.03 \ 61655 & 0.202 \ 11409 & N1(A) & 0.399 \ 1422 & 0.117 \ 89445 & 0.577 \ 6160 \ 0.655 \ 04417 & -0.273 \ 81669 & 0.422 \ 23047 \ 0.655 \ 04417 & -0.273 \ 81669 & 0.422 \ 23047 \ 0.655 \ 04417 & -0.273 \ 81669 & 0.422 \ 23047 \ 0.655 \ 04417 \ 0.655 \ 04417 \ 0.655 \ 04417 \ 0.552 \ 81669 & 0.422 \ 23047 \ 0.552 \ 81669 & 0.422 \ 23047 \ 0.552 \ 81669 & 0.452 \ 23047 \ 0.552 \ 81669 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 91611 \ 0.555 \ 9160 \ 0.555 \ 9160 \ 0$	C(2)	0.561.68(24)	0.070 13(61)	0.344.96(41)	O(9)	0.360 + 3(23) 0.269 26(24)	0.227 $33(37)$	0.417 02(44) 0.428 75(44)
$ \begin{array}{c} C(4) & 0.61^9 4430 & -0.045 0 (888) & 0.114 40(47) & C(2A) & 0.435 32(24) & -0.071 316(1) & 0.655 0 (441) \\ C(5) & 0.637 78(27) & -0.227 81(79) & 0.357 44(46) & C(8A) & 0.491 51(23) & -0.227 99(57) & 0.582 38(44) \\ C(1) & 0.645 95(8) & 0.265 09(11) & 0.733 31(8) & C(13) & 0.882 56(8) & 0.360 91(13) & 0.885 24(16) \\ C(2) & 0.876 80(8) & 0.046 27(11) & 0.733 09(8) & C(14) & 0.950 02(8) & 0.255 92(11) & 0.881 24(8) \\ C(2) & 0.876 80(8) & 0.046 27(11) & 0.733 09(8) & C(14) & 0.950 02(8) & 0.255 92(11) & 0.881 24(8) \\ C(3) & 0.935 10(13) & -0.046 65(14) & 0.542 92(8) & C(17) & 0.937 85(6) & -0.058 38(3) & 0.386 66(8) \\ C(6) & 0.799 51(36) & -0.046 65(14) & 0.542 92(8) & C(17) & 0.877 85(6) & -0.058 38(3) & 0.348 66(8) \\ C(7) & 0.679 49(14) & 0.199 28(15) & 0.517 07(6) & C(18) & 0.785 42(6) & -0.048 38(3) & 0.448 55(5) \\ C(7) & 0.679 49(14) & 0.199 28(15) & 0.517 07(16) & C(18) & 0.785 42(6) & -0.048 38(3) & 0.448 55(5) \\ C(9) & 0.600 88(8) & 0.226 15(36) & 0.636 19(7) & C(21) & 0.537 32(8) & 0.539 00(36) & 0.557 95(5) \\ C(10) & 0.634 248(8) & 0.226 15(36) & 0.636 19(7) & C(21) & 0.531 52(8) & 0.539 00(36) & 0.557 58(7) \\ C(11) & 0.716 12(13) & 0.233 8(46) & -0.707 06(7) & C(22) & 0.513 53(8) & 0.599 00(36) & 0.557 58(7) \\ C(11) & 0.716 27(13) & 0.233 8(46) & -0.311 05(32) & C(12A) & 0.937 75(44) & 0.613 94(12) & 0.097 74(45) \\ C(3) & 0.867 24743 & 0.333 28(46) & -0.331 18(32) & C(12A) & 0.937 75(44) & 0.513 86(17) & -0.317 24(7) \\ C(6) & 0.915 53(23) & 0.444 55(10) & -0.331 18(32) & C(12A) & 0.357 12(4) & 0.513 86(17) & -0.377 24(7) \\ C(7) & 0.770 04(8) & 0.223 042(2) & -0.275 81(53) & C(15A) & 0.858 72(43) & 0.513 86(17) & -0.377 24(7) \\ C(7) & 0.770 04(8) & 0.223 042(2) & -0.275 81(53) & C(17A) & 0.663 24(23) & 0.513 18(17) & -0.117 24(34) \\ C(7) & 0.775 28(46) & 0.380 60(2) & -0.351 18(12) & C(12A) & 0.357 12(14) & 0.057 35(12) & 0.077 35(15) \\ C(7) & 0.777 04(8) & 0.224 02(2) & -0.073 81(13) & 0.273 35(13) & 0.077 35(11) & -0.377 35(17) \\ C(7) & 0.777 04(8) & 0.224 02(2) & -0.275 81(53) & C(17A) & 0.868 $	C(2)	0.501 85(24)	0.103.61(65)	0.202 11(49)	N(1A)	$0.209 \ 10(21)$	0.00001(72) 0.111.89(45)	0.577.61(40)
$\begin{array}{c} C(5) & 0.637\ 7827 & -0.27\ 50170 & 0.235\ 73449 & C(7A) & 0.403\ 53255 & 0.125\ 731690 & 0.252\ 531691$	C(4)	0.61944(30)	-0.045.08(88)	0.134 40(47)	C(2A)	0.33911(22) 0.438 32(24)	-0.07013(61)	0.655.04(41)
$ \begin{array}{c} C(6) & 0.649 19(26) & -0.257 01(70) & 0.337 44(46) & C(8A) & 0.491 51(23) & -0.227 99(57) & 0.382 38(44) \\ 12 \\ C(1) & 0.845 95(8) & 0.065 (50(7) & 0.783 33(8) & C(13) & 0.982 56(8) & 0.360 91(31) & 0.852 41(8) \\ C(2) & 0.878 80(8) & 0.064 27(31) & 0.778 03(8) & C(14) & 0.990 02(8) & 0.255 92(31) & 0.891 25(8) \\ C(3) & 0.388 10(13) & -0.057 56(47) & 0.679 95(12) & C(15) & 0.994 22(8) & -0.040 72(31) & 0.591 64(11) \\ C(4) & 0.845 57(13) & 0.064 57(14) & 0.515 29(13) & C(16) & 0.944 22(8) & -0.040 72(31) & 0.791 93(8) \\ C(6) & 0.057 38(10) & -0.065 65(47) & 0.515 29(13) & C(16) & 0.944 22(8) & -0.040 72(31) & 0.791 93(8) \\ C(7) & 0.057 49(14) & -0.065 65(41) & 0.515 29(13) & C(16) & 0.974 25(6) &0.141 13(44) & 0.456 95(8) \\ C(7) & 0.057 49(14) & -0.065 82(25) & 0.977 65(15) & C(20) & 0.677 56(6) &0.141 13(44) & 0.456 95(8) \\ C(9) & 0.600 88(8) & 0.256 13(36) & 0.707 06(7) & C(22) & 0.501 53(8) & 0.559 00(36) & 0.625 78(7) \\ C(11) & 0.054 24(8) & 0.238 12(51) & 0.744 17(13) & C(24) & 0.514 24(8) & 0.537 22(6) & 0.777 29(7) \\ C(12) & 0.771 87(12) & 0.031 82 82(46) & -0.741 17(13) & C(24) & 0.801 24(8) & 0.537 24(26) & 0.777 29(7) \\ 16 \\ C(12) & 0.674 42(8) & 0.444 65(10) & -0.311 05(22) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 744(52) \\ C(13) & 0.874 54(23) & 0.444 65(10) & -0.311 8(22) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 744(53) \\ C(13) & 0.874 54(23) & 0.424 65(10) & -0.235 18(13) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 744(52) \\ C(14) & 0.891 87(53) & 0.336 68(10) & -0.235 18(13) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 744(52) \\ C(13) & 0.874 54(23) & 0.424 65(10) & -0.218 18(22) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 744(52) \\ C(13) & 0.874 54(23) & 0.424 52(10) & -0.235 89(13) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 744(52) \\ C(13) & 0.874 54(23) & 0.424 52(10) & -0.235 89(13) & C(12A) & 0.937 75(14) & 0.613 39(18) & 0.977 75(17) & -0.118 1(77) & -0.125 89(13) & 0.013 81(32) & 0.775 82(11) & -0.215 89(13) & 0.013 81(32) & 0.775 82(11) & 0.013 18(32) &$	C(5)	0.65778(27)	-0.227.83(79)	0.21233(49)	C(7A)	0.408 35(25)	0.15281(69)	0.03301(11) 0.42220(47)
	C(6)	0.649 19(26)	$-0.257\ 01(70)$	0.357 44(46)	C(8A)	0.491 51(23)	-0.227 99(57)	0.582 38(44)
	12							
	C(1)	0.945.05(9)	0.265.00(21)	0 783 33(8)	C(13)	0 882 56(8)	0.260.01(21)	0.852 41(8)
	C(1)	0.843 93(8)	0.20309(31) 0.06427(31)	0.763 33(6) 0.753 00(8)	C(13)	0.062 30(0)	0.30091(31) 0.35502(31)	0.03241(0)
$ \begin{array}{c} (1) & 0.838 \ (101) & -0.038 \ (101) & -0.038 \ (101) & 0.048 \ (101) $	C(2)	$0.870\ 80(8)$	0.004 27(31)	0.73309(8)	C(14)	0.93002(8)	0.23392(31)	0.891 23(8)
$ \begin{array}{c} C(1) \\ C(2) \\ C(3) \\ C(3) \\ C(3) \\ C(4) \\ C(5) \\ C(6) \\ C(6) \\ C(6) \\ C(6) \\ C(7) \\ C(7) \\ C(7) \\ C(7) \\ C(8) \\ C(9) \\ C(12) \\ C(1$	C(3)	0.838 10(13)	-0.03703(47)	0.079.93(12)	C(15)	0.960.67(6)	0.03311(31) 0.04072(21)	0.80101(8)
$ \begin{array}{c} 1(1) & 0.719 \\ (-10) & 0.729 \\ (-10) & 0.719 \\ (-10) & 0.729 \\ (-10) & 0.719 \\ (-10) & 0.719 \\ (-10) & 0.729 \\ (-10) & 0.719 \\ (-10) & 0.719 \\ (-10) & 0.719 \\ (-10) & 0.729 \\ (-10) & 0.719 \\ (-10) &$	C(4)	$0.643 \ 37(13)$ $0.705 \ 13(6)$	0.064 / 1(30)	0.011 29(13) 0.542 02(8)	C(10)	0.344 20(8)	-0.04072(31) 0.22058(34)	0.791.95(8)
	C(5)	0.793 13(0) 0.710 00(6)	-0.04003(34)	0.342.92(8) 0.517.07(8)	C(17)	0.827 83(0)	-0.229  38(34) 0.368 28(34)	0.308 00(8)
$ \begin{array}{c} C(1) & 0.37 \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \ \$	C(0)	0.719.99(0)	-0.00243(34)	0.51707(8)	C(10)	0.763 42(0)	-0.306 26(34) 0.324 06(34)	0.440 33(0) 0.422 60(0)
$ \begin{array}{c} C(2) & 0.305 \ an (1) \\ C(2) & 0.305 \ an (1) \\ C(1) & 0.305 \ an (1) \\ C(1) & 0.505 \ an (1) \\ C(2) \ an (1) \ an (2) \ an $	C(I)	0.07949(14) 0.63581(15)	$0.169\ 26(31)$	0.33170(14) 0.507.63(15)	C(19)	0.71026(0)	-0.32400(34)	0.422 09(8)
$ \begin{array}{c} C(p) & 0.400 \ \text{s}(p) & 0.240 \ \text{s}(p) & 0.242 \ \text{s}(p) & 0.252 \ \text{s}(p)$	$C(\delta)$	0.03381(13)	0.038 22(32) 0.256 15(36)	0.39703(13)	C(20)	0.077 50(0)	-0.141 13(34)	0.430 93(8)
$ \begin{array}{c} C(10) & 0.394 448(1) & 0.392 59(30) & 0.70 08(1) & C(22) & 0.334 89(8) & 0.59 00(50) & 0.022 78(1) \\ C(12) & 0.771 87(12) & 0.383 28(46) & 0.744 17(13) & C(24) & 0.631 48(8) & 0.357 22(36) & 0.373 29(7) \\ \hline 16 \\ \hline C(2) & 0.767 43(25) & 0.424 65(10) & -0.311 05(32) & C(12A) & 0.937 75(44) & 0.613 39(18) & 0.097 34(55) \\ C(2) & 0.767 43(25) & 0.422 55(10) & -0.363 18(32) & C(13A) & 0.838 11(32) & 0.897 34(12) & 0.095 78(45) \\ C(3) & 0.842 79(43) & 0.399 60(0) & -0.286 89(51) & C(14A) & 0.792 48(32) & 0.877 34(12) & 0.095 78(45) \\ C(4) & 0.891 87(53) & 0.326 26(19) & -0.353 27(55) & C(15A) & 0.837 27(43) & 0.581 88(717) & -0.137 14(47) \\ C(5) & 0.995 44(30) & 0.324 68(19) & -0.215 88(35) & C(17A) & 0.688 047(23) & 0.533 11(10) & -0.376 89(33) \\ C(7) & 0.779 00(48) & 0.294 02(22) & -0.213 01(65) & C(18A) & 0.889 047(23) & 0.533 11(10) & -0.396 98(33) \\ C(8) & 0.752 38(46) & 0.308 00(22) & -0.003 94(60) & C(19A) & 0.666 0(23) & 0.552 16(10) & -0.496 93(33) \\ C(10) & 0.585 88(33) & 0.312 62(13) & 0.012 185(34) & C(21A) & 1.238 57(31) & 0.648 80(11) & -0.276 07(34) \\ C(11) & 0.649 92(47) & 0.344 37(0) & 0.118 86(54) & C(22A) & 1.334 60(31) & 0.713 55(11) & -0.157 3(24) \\ C(12) & 0.677 03(45) & 0.384 70(18) & 0.091 72(54) & C(23A) & 1.236 03(1) & 0.713 55(11) & -0.157 3(24) \\ C(13) & 0.581 34(31) & 0.428 26(12) & -0.014 36(25) & C(25A) & 0.288 67(55) & 0.709 17(13) & -0.157 13(24) \\ C(14) & 0.539 99(31) & 0.428 26(12) & -0.014 36(25) & C(25A) & 0.288 (75) & 0.798 9(13) & -0.175 8(32) \\ C(14) & 0.667 71(45) & 0.447 18(17) & -0.177 55(48) & C(27A) & 0.738 38(65) & 0.694 98(13) & -0.025 8(43) \\ C(14) & 0.667 71(45) & 0.447 18(17) & -0.175 54(34) & C(27A) & 0.738 37(35) & 0.709 17(13) & -0.165 19(33) \\ C(14) & 0.667 71(45) & 0.447 18(17) & -0.177 55(48) & C(27A) & 0.738 38(65) & 0.694 98(43) & 0.095 98(13) & 0.105 98(14) \\ C(14) & 0.667 07(14) & 0.448 57(53) & C(18B) & 0.362 34(16) & 0.068 89(11) & -0.055 84(34) \\ C(14) & 0.667 33(3) & 0.288 67(13) & -0.057 34(13) & 0.079 33(23) & 0.678 49(13) & 0.079 33(23) & 0.67$	C(9)	0.00080(8)	0.230 13(30) 0.242 50(36)	0.030 19(7)	C(21)	0.334 32(8)	0.304 30(30)	0.393 33(7)
$ \begin{array}{c} C(1) \\ C(12) \\$	C(10)	0.034 24(8) 0.706 27(12)	0.342 39(30) 0.333 87(51)	0.70700(7) 0.754 50(14)	C(22)	0.501,55(8)	0.33900(30)	0.02378(7)
$ \begin{array}{c} IG(T) & G,T(T) & G,T(T) & G,T(T) & G,T(G) & G,S(G) & G,G,S(G) & G,G,S(G) & G,S(G) & G,G,S(G) & G,S(G) & G,S,S) & G,S(G) & G,S,S) & G,S,S) & G,S,S) & G,S,S) & G,S,S) & G,S,S) & G,S,\mathsf$	C(11)	0.700 27(13) 0.771 87(12)	0.233.87(31) 0.383.28(46)	0.734 39(14) 0.744 17(13)	C(23)	$0.334 \ 69(6)$ 0.601 24(8)	0.04343(30) 0.53722(36)	0.090000(7)
	C(12)	0.771 07(12)	0.505 20(40)	0.744 17(15)	C(24)	0.001 24(0)	0.337 22(30)	0.75729(1)
$ \begin{array}{c} (1) & 0.866 32(2) & 0.444 05(10) & -0.31 10(12) & C(12A) & 0.93 15(44) & 0.013 59(18) & 0.097 34(25) \\ C(2) & 0.677 43(25) & 0.422 55(10) & -0.333 18(32) & C(13A) & 0.838 11(32) & 0.587 34(12) & 0.059 576(26) \\ C(3) & 0.842 79(43) & 0.399 60(0) & -0.286 89(51) & C(14A) & 0.792 48(32) & 0.572 66(12) & -0.008 18(26) \\ C(4) & 0.891 87(53) & 0.362 46(19) & -0.353 27(55) & C(15A) & 0.835 72(43) & 0.551 86(17) & -0.117 224(47) \\ C(5) & 0.959 44(30) & 0.334 68(11) & -0.275 89(35) & C(16A) & 0.922 53(43) & 0.551 86(17) & -0.172 24(47) \\ C(6) & 0.915 53(30) & 0.302 57(11) & -0.215 38(35) & C(17A) & 0.866 24(23) & 0.532 11(10) & -0.349 69(33) \\ C(7) & 0.797 00(48) & 0.294 20(22) & -0.023 01(65) & C(18A) & 0.886 24(23) & 0.552 11(0) & -0.496 98(33) \\ C(8) & 0.752 38(46) & 0.308 0022) & -0.093 94(60) & C(19A) & 0.965 06(23) & 0.552 10(0) & -0.479 63(33) \\ C(10) & 0.885 58(33) & 0.305 77(13) & -0.080 29(34) & C(20A) & 1.037 43(23) & 0.573 25(10) & -0.479 63(33) \\ C(11) & 0.649 92(47) & 0.344 57(20) & 0.118 86(54) & C(22A) & 1.396 37(31) & 0.668 80(11) & -0.215 98(34) \\ C(12) & 0.677 03(45) & 0.384 70(18) & 0.091 72(54) & C(23A) & 1.334 6(31) & 0.713 56(11) & -0.157 51(24) \\ C(13) & 0.581 34(31) & 0.441 65(12) & 0.088 98(25) & C(24A) & 1.245 102(31) & 0.713 56(11) & -0.157 51(24) \\ C(14) & 0.539 93(1) & 0.428 26(12) & -0.014 36(25) & C(25A) & 0.828 67(35) & 0.707 59(13) & -0.171 58(33) \\ C(16) & 0.667 71(45) & 0.447 18(17) & -0.177 55(48) & C(27A) & 0.670 88(35) & 0.694 58(13) & -0.058 84(33) \\ C(19) & 0.678 32(25) & 0.466 67(10) & -0.381 15(12) & C(28A) & 0.733 79(53) & 0.777 59(13) & -0.177 58(12) \\ C(19) & 0.788 72(53) & 0.443 68(1) & -0.059 54(33) & C(13A) & 0.659 80(33) & 0.678 40(13) & 0.031 13(33) \\ C(18) & 0.627 85(25) & 0.446 67(10) & -0.351 15(12) & C(29A) & 0.794 18(32) & 0.577 58(12) & 0.204 92(26) \\ C(20) & 0.778 71(25) & 0.442 25(10) & -0.458 78(32) & C(18B) & 0.496 9(28) & 0.677 57(13) & -0.167 8(34) & 0.668 57(13) & 0.170 579 8(12) & 0.072 19(13) & 0.466 67(10) & 0.267 87(33) & C(18B) & 0.398 23(4) & $	16 C(1)	0 (9( 25(25)	0 444 (5(10)	0.211.05(22)	C(124)	0.027.75(14)	0 (12 20/10)	0.007.24(55)
$ \begin{array}{c} C(2) & 0.767 43(25) & 0.422 53(10) & -0.380 16(22) & C(15A) & 0.383 11(22) & 0.587 54(12) & 0.009 170(26) \\ C(3) & 0.887 17(53) & 0.352 46(19) & -0.353 27(55) & C(15A) & 0.833 72(43) & 0.551 86(17) & -0.137 17(47) \\ C(5) & 0.959 44(30) & 0.334 68(11) & -0.275 89(35) & C(16A) & 0.922 53(43) & 0.551 86(17) & -0.172 24(47) \\ C(6) & 0.915 53(30) & 0.302 57(11) & -0.215 38(35) & C(17A) & 0.886 24(23) & 0.533 16(10) & -0.374 54(33) \\ C(7) & 0.797 00(46) & 0.294 20(22) & -0.213 01(65) & C(18A) & 0.880 47(23) & 0.552 16(10) & -0.496 98(33) \\ C(8) & 0.752 38(46) & 0.308 00(22) & -0.093 94(60) & C(19A) & 0.985 06(23) & 0.552 16(10) & -0.479 63(33) \\ C(10) & 0.654 86(31) & 0.302 77(13) & -0.080 29(34) & C(20A) & 1.037 43(23) & 0.573 25(10) & -0.479 63(33) \\ C(11) & 0.649 92(47) & 0.341 57(20) & 0.118 86(54) & C(12A) & 1.328 637(11) & 0.668 80(11) & -0.215 98(34) \\ C(12) & 0.677 03(45) & 0.384 70(18) & 0.091 72(54) & C(23A) & 1.354 60(13) & 0.713 56(11) & -0.156 71(34) \\ C(13) & 0.581 94(31) & 0.411 65(12) & 0.088 98(25) & C(24A) & 1.245 02(31) & 0.720 32(11) & -0.157 52(34) \\ C(14) & 0.539 99(31) & 0.428 26(12) & -0.014 36(25) & C(25A) & 0.828 67(55) & 0.707 97(13) & -0.171 88(33) \\ C(15) & 0.580 14(44) & 0.4418 22(17) & -0.136 30(47) & C(26A) & 0.718 32(55) & 0.709 17(13) & -0.159 19(33) \\ C(15) & 0.667 71(45) & 0.4471 8(17) & -0.177 55(48) & C(27A) & 0.679 84(52) & 0.553 15(12) & 0.204 92(26) \\ C(19) & 0.778 71(25) & 0.442 52(10) & -0.485 78(32) & C(12A) & 0.702 93(15) & 0.678 40(13) & 0.033 13(33) \\ C(15) & 0.667 71(45) & 0.4471 8(11) & -0.556 25(32) & C(10A) & 0.704 48(32) & 0.553 15(12) & 0.204 92(26) \\ C(20) & 0.778 71(25) & 0.442 52(10) & -0.485 78(32) & C(13A) & 0.704 93(12) & 0.538 47(12) & 0.106 21(26) \\ C(21) & 1.135 06(30) & 0.315 18(11) & -0.205 51(35) & C(13B) & 0.378 23(45) & 0.096 29(18) & 0.479 57(51) \\ C(23) & 0.455 74(31) & 0.423 24(11) & -0.157 92(35) & C(13B) & 0.378 23(45) & 0.096 29(18) & 0.479 57(51) \\ C(24) & 0.478 43(33) & 0.216 07(13) & 0.036 77(34) & C(7B) & 0.324 82(31) & 0.159 6(11) &$	C(1)	0.080 33(25)	0.444 65(10)	-0.31105(32)	C(12A)	0.937 75(44)	0.613.39(18)	0.097 34(55)
$ \begin{array}{c} (13) \\ (13) \\ (14) \\ (14) \\ (15) \\ ($	C(2)	0.76743(25)	0.422(55(10))	-0.303 18(32)	C(13A)	0.83811(32) 0.70248(32)	0.58734(12)	0.095 /6(26)
$ \begin{array}{c} C(7) & 0.39 \ 4(30) & 0.30 \ 40(17) & -0.30 \ 21(25) & C(15A) & 0.30 \ 21(45) & 0.351 \ 86(17) & -0.112 \ 11(47) \\ C(6) & 0.915 \ 53(30) & 0.302 \ 57(11) & -0.215 \ 38(35) & C(17A) & 0.868 \ 24(23) & 0.531 \ 86(10) & -0.374 \ 54(33) \\ C(7) & 0.797 \ 00(48) & 0.294 \ 00(22) & -0.0213 \ 01(65) & C(18A) & 0.868 \ 24(23) & 0.532 \ 11(10) & -0.496 \ 98(33) \\ C(8) & 0.752 \ 38(46) & 0.308 \ 00(22) & -0.093 \ 94(60) & C(19A) & 0.868 \ 24(23) & 0.552 \ 16(10) & -0.496 \ 98(33) \\ C(9) & 0.630 \ 46(33) & 0.305 \ 77(13) & -0.080 \ 29(34) & C(20A) & 1.037 \ 43(23) & 0.573 \ 25(10) & -0.479 \ 63(33) \\ C(10) & 0.858 \ 58(33) & 0.321 \ 62(13) & 0.021 \ 85(34) & C(22A) & 1.386 \ 57(31) & 0.668 \ 80(11) & -0.215 \ 98(4) \\ C(12) & 0.677 \ 03(45) & 0.384 \ 70(18) & 0.091 \ 72(54) & C(22A) & 1.366 \ 60(31) & 0.713 \ 56(11) & -0.156 \ 71(34) \\ C(13) & 0.581 \ 43(31) & 0.411 \ 65(12) & 0.088 \ 98(55) & C(22A) & 1.326 \ 67(33) & 0.070 \ 59(13) & -0.177 \ 58(33) \\ C(14) & 0.581 \ 43(31) & 0.418 \ 22(17) & -0.113 \ 60(47) & C(22A) & 0.788 \ 82(53) & 0.099 \ 58(13) & -0.075 \ 98(33) \\ C(15) & 0.580 \ 14(44) & 0.418 \ 22(17) & -0.113 \ 60(47) & C(22A) & 0.788 \ 82(53) & 0.099 \ 58(13) & -0.059 \ 84(33) \\ C(15) & 0.580 \ 14(44) & 0.418 \ 22(17) & -0.136 \ 30(47) & C(22A) & 0.738 \ 78(53) & 0.694 \ 58(13) & -0.055 \ 84(33) \\ C(17) & 0.616 \ 56(25) & 0.466 \ 71(10) & -0.381 \ 51(32) & C(22A) & 0.738 \ 78(53) & 0.694 \ 58(13) & -0.055 \ 84(33) \\ C(19) & 0.788 \ 71(25) & 0.442 \ 58(10) & -0.356 \ 25(32) & C(30A) & 0.704 \ 62(32) & 0.553 \ 87(12) & 0.106 \ 21(26) \\ C(20) & 0.778 \ 71(25) & 0.422 \ 21(1) & -0.456 \ 71(34) & -0.275 \ 58(35) & C(22A) & 0.702 \ 91(23) & 0.053 \ 87(12) & -0.002 \ 96(26) \\ C(20) & 0.778 \ 71(25) & 0.422 \ 52(10) & -0.485 \ 78(32) & C(2B) & 0.779 \ 53(2) & 0.588 \ 57(12) & 0.106 \ 21(26) \\ C(20) & 0.778 \ 71(25) & 0.442 \ 58(10) & -0.275 \ 55(15) & C(12B) & 0.779 \ 53(2) & 0.588 \ 57(12) & -0.002 \ 96(26) \\ C(20) & 0.758 \ 71(31) & 0.428 \ 58(35) & C(2B) & 0.778 \ 58(31) & 0.159 \ 60(11) \ 0.486 \ 93(33) \\ C($	C(3)	0.042 79(43)	$0.399\ 00(0)$	-0.200 69(51)	C(14A)	0.79240(32) 0.83572(43)	0.37200(12) 0.59197(17)	-0.00010(20)
$ \begin{array}{c} C(3) & 0.524 + C(3) & 0.534 + S(11) & -0.215 + S(35) & C(167) & 0.522 + S(45) & 0.531 + S(11) & -0.172 + S(43) \\ C(7) & 0.797 + 00(48) & 0.294 + 20(22) & -0.213 + 0(65) & C(18A) & 0.880 + 7(23) & 0.532 + 1(10) & -0.374 + S(43) \\ C(7) & 0.797 + 00(48) & 0.294 + 20(22) & -0.213 + 0(65) & C(18A) & 0.880 + 7(23) & 0.532 + 1(10) & -0.496 + 98(33) \\ C(9) & 0.630 + 6(33) & 0.305 + 7(13) & -0.080 + 29(34) & C(20A) & 1.332 + 5(31) & 0.654 + 90(11) & -0.276 + 7(34) \\ C(11) & 0.649 + 92(47) & 0.341 + 57(20) & 0.118 + 86(54) & C(22A) & 1.396 + 37(31) & 0.688 + 80(11) & -0.215 + 98(34) \\ C(12) & 0.677 + 03(45) & 0.384 + 70(18) & 0.091 + 75(44) & C(23A) & 1.354 + 60(31) & 0.713 + 56(11) & -0.156 + 71(34) \\ C(13) & 0.581 + 3(431) & 0.411 + 65(12) & 0.088 + 98(25) & C(22A) & 1.396 + 37(31) & 0.688 + 80(11) & -0.157 + 52(34) \\ C(14) & 0.539 + 99(31) & 0.428 + 26(12) & -0.014 + 36(25) & C(22A) & 1.734 + 60(31) & 0.713 + 56(11) & -0.157 + 52(34) \\ C(14) & 0.539 + 99(31) & 0.428 + 26(12) & -0.014 + 36(25) & C(22A) & 0.718 + 23(25) & 0.709 + 7(13) & -0.159 + 19(33) \\ C(16) & 0.667 + 71(45) & 0.447 + 18(17) & -0.177 + 55(48) & C(27A) & 0.718 + 82(3) & 0.678 + 0(13) & 0.033 + 3(33) \\ C(18) & 0.627 + 85(25) & 0.466 + 67(10) & -0.381 + 5(32) & C(22A) & 0.794 + 18(32) & 0.577 + 58(12) & 0.204 + 92(26) \\ C(20) & 0.778 + 71(25) & 0.422 + 22(10) & -0.487 + 78(32) & C(23A) & 0.709 + 73(32) & 0.678 + 0(13) & 0.033 + 3(33) \\ C(26) & 0.455 + 17(33) & 0.289 + 9(11) & -0.172 + 95(35) & C(12B) & 0.779 + 1(24) & 0.072 + 19(14) & 0.042 + 27(33) \\ C(21) & 1.099 + 10(33) & 0.340 + 98(11) & -0.272 + 95(35) & C(22B) & 0.477 + 1(24) & 0.072 + 19(14) & 0.042 + 27(33) \\ C(22) & 1.555 + 17(33) & 0.289 + 9(11) & -0.172 + 95(35) & C(21B) & 0.384 + 79(12) & 0.065 + 17(33) \\ C(24) & 0.498 + 38(30) & 0.767 + 71(1) & -0.148 + 99(35) & C(2B) & 0.479 + 1(24) & 0.072 + 19(14) & 0.479 + 8(35) \\ C(24) & 0.495 + 13(33) & 0.340 + 98(11) & -0.127 + 95(35) & C(12B) & 0.364 + 23(24) & 0.097 + 75(11) & 0.448 + 96(53) \\ C(25) & 0.455 + 71(33) & 0.288 + 9(13) & -0.15$	C(4)	0.091 07(33)	0.30240(19) 0.33468(11)	-0.33527(33)	C(15A)	0.03372(43)	0.361.67(17)	-0.13171(47)
$ \begin{array}{c} C(0) & 0.915 35(30) & 0.302 3(11) & -0.213 30(65) & C(174) & 0.385 24(25) & 0.335 10(10) & -0.374 34(35) \\ C(7) & 0.797 00(48) & 0.294 20(22) & -0.213 01(65) & C(18A) & 0.886 24(25) & 0.552 11(10) & -0.496 98(33) \\ C(8) & 0.752 38(46) & 0.308 00(22) & -0.093 94(60) & C(19A) & 0.965 06(23) & 0.552 16(10) & -0.479 63(33) \\ C(10) & 0.585 58(33) & 0.321 62(13) & 0.021 85(34) & C(21A) & 1.328 55(31) & 0.654 80(11) & -0.276 07(34) \\ C(11) & 0.649 92(47) & 0.341 57(20) & 0.0118 86(54) & C(22A) & 1.396 57(31) & 0.6680 80(11) & -0.215 98(34) \\ C(12) & 0.677 03(45) & 0.384 70(18) & 0.091 72(54) & C(23A) & 1.238 5(31) & 0.713 56(11) & -0.157 15(34) \\ C(13) & 0.581 34(31) & 0.411 55(12) & 0.088 98(25) & C(22A) & 1.236 40(31) & 0.713 56(11) & -0.157 15(34) \\ C(14) & 0.539 59(31) & 0.428 26(12) & -0.014 36(25) & C(25A) & 0.828 67(35) & 0.707 59(13) & -0.171 58(33) \\ C(15) & 0.580 14(44) & 0.418 22(17) & -0.136 30(47) & C(26A) & 0.718 32(35) & 0.709 17(13) & -0.157 15(34) \\ C(15) & 0.580 14(44) & 0.418 22(17) & -0.136 30(47) & C(26A) & 0.733 79(15) & 0.677 84(0(13) & 0.033 13(33) \\ C(15) & 0.667 71(45) & 0.447 18(17) & -0.177 55(48) & C(27A) & 0.708 8(35) & 0.694 58(13) & -0.056 84(33) \\ C(19) & 0.708 92(25) & 0.466 71(10) & -0.381 51(32) & C(28A) & 0.704 54(32) & 0.553 15(12) & 0.204 492(26) \\ C(20) & 0.778 71(25) & 0.422 52(10) & -0.485 78(32) & C(30A) & 0.702 93(32) & 0.584 77(12) & 0.062 12(66) \\ C(21) & 1.69 20(30) & 0.340 98(11) & -0.279 95(35) & C(32A) & 0.702 93(32) & 0.584 23(12) & -0.002 96(26) \\ C(22) & 1.135 05(30) & 0.315 18(11) & -0.209 51(35) & C(13B) & 0.656 11(24) & 0.052 34(11) & 0.462 77(33) \\ C(24) & 0.981 38(30) & 0.276 77(11) & -0.115 93(35) & C(13B) & 0.365 (33) & 0.079 29(11) & 0.477 38(33) \\ C(24) & 0.981 38(30) & 0.276 77(11) & -0.157 93(35) & C(13B) & 0.468 (36(31) & 0.199 43(11) & 0.589 29(35) \\ C(23) & 0.455 14(33) & 0.328 69(13) & -0.152 92(34) & C(7B) & 0.438 (34(31) & 0.199 43(11) & 0.598 29(35) \\ C(24) & 0.493 13(0, 446 07(12) & 0.039 53(15) & C(11B) & 0.635 29(14) & 0.178 78(120) & 0.$	C(3)	0.93944(30)	0.33406(11) 0.30257(11)	-0.275.69(35)	C(10A)	$0.922 \ 33(43)$	0.331.60(17)	-0.17224(47)
$ \begin{array}{c} C(3) & 0.72 & 30(46) & 0.224 & 20(22) & -0.213 & 01(10) & C(18A) & 0.850 & 7(23) & 0.522 & 11(10) & -0.470 & 96(35) \\ C(8) & 0.630 & 46(33) & 0.305 & 77(13) & -0.080 & 29(34) & C(20A) & 1.037 & 43(23) & 0.573 & 25(10) & -0.479 & 63(33) \\ C(10) & 0.638 & 58(33) & 0.321 & 62(13) & 0.021 & 85(34) & C(21A) & 1.328 & 55(31) & 0.654 & 80(11) & -0.215 & 98(34) \\ C(11) & 0.649 & 92(47) & 0.341 & 57(20) & 0.118 & 86(54) & C(22A) & 1.396 & 37(31) & 0.668 & 80(11) & -0.215 & 98(34) \\ C(12) & 0.677 & 03(43) & 0.411 & 65(12) & 0.088 & 98(25) & C(23A) & 1.354 & 60(31) & 0.713 & 56(11) & -0.157 & 52(34) \\ C(13) & 0.581 & 34(31) & 0.411 & 65(12) & 0.088 & 98(25) & C(22A) & 1.245 & 02(31) & 0.720 & 32(11) & -0.157 & 52(34) \\ C(14) & 0.593 & 99(31) & 0.428 & 26(12) & -0.014 & 36(25) & C(22A) & 0.718 & 32(35) & 0.709 & 17(13) & -0.157 & 52(34) \\ C(15) & 0.667 & 71(45) & 0.447 & 18(17) & -0.177 & 55(48) & C(27A) & 0.738 & 32(35) & 0.678 & 40(13) & 0.033 & 13(33) \\ C(16) & 0.677 & 71(45) & 0.447 & 18(17) & -0.177 & 55(48) & C(27A) & 0.778 & 82(35) & 0.678 & 40(13) & 0.033 & 13(33) \\ C(18) & 0.627 & 85(25) & 0.466 & 671(10) & -0.554 & 25(32) & C(30A) & 0.704 & 18(32) & 0.577 & 58(12) & 0.204 & 92(26) \\ C(20) & 0.778 & 71(25) & 0.442 & 52(10) & -0.485 & 78(32) & C(31A) & 0.659 & 00(32) & 0.538 & 47(12) & 0.106 & 21(26) \\ C(21) & 1.069 & 20(30) & 0.340 & 98(11) & -0.272 & 93(35) & C(23A) & 0.704 & 2(32) & 0.558 & 15(12) & 0.210 & 42(62) \\ C(22) & 1.550 & 0.546 & 57(11) & -0.148 & 99(35) & C(23A) & 0.705 & 91(33) & 0.600 & 09(18) & 0.477 & 91(34) \\ C(25) & 0.565 & 17(33) & 0.288 & 07(11) & -0.151 & 93(35) & C(23B) & 0.338 & 55(58) & 0.133 & 06(20) & 0.414 & 15(58) \\ C(26) & 0.455 & 01(33) & 0.228 & 31(13) & -0.167 & 68(34) & C(4B) & 0.325 & 65(8) & 0.133 & 06(20) & 0.414 & 15(58) \\ C(26) & 0.455 & 17(33) & 0.288 & 69(13) & -0.157 & 59(14) & 0.052 & 91(11) & 0.458 & 82(64) \\ C(25) & 0.565 & 17(33) & 0.288 & 69(13) & -0.157 & 65(14) & 0.778 & 91(11) & 0.578 & 92(15) \\ C(26) & 0.455 & 01(33) & 0.228 & 31(13) & -0.167 & 68(34) & C($	C(0)	0.715 55(50)	0.302 37(11) 0.204 20(22)	-0.213 36(33)	C(17A)	0.80824(23)	0.555 10(10) 0.532 11(10)	-0.37434(33)
$ \begin{array}{c} C(9) & 0.752 \ 36(40) & 0.366 \ 50(22) & -0.080 \ 29(40) & C(19A) & 0.033 \ 50(22) & 0.573 \ 25(10) & -0.479 \ 53(33) \\ C(10) & 0.585 \ 58(33) & 0.321 \ 62(13) & 0.021 \ 85(34) & C(21A) & 1.328 \ 55(31) & 0.654 \ 80(11) & -0.276 \ 07(34) \\ C(12) & 0.677 \ 03(45) & 0.384 \ 70(18) & 0.091 \ 72(54) & C(22A) & 1.396 \ 57(31) & 0.680 \ 80(11) & -0.155 \ 71(34) \\ C(13) & 0.581 \ 34(31) & 0.411 \ 65(12) & 0.088 \ 98(25) & C(22A) & 1.354 \ 60(31) & 0.713 \ 56(11) & -0.155 \ 71(34) \\ C(13) & 0.581 \ 34(31) & 0.411 \ 65(12) & 0.088 \ 98(25) & C(22A) & 0.328 \ 67(35) & 0.707 \ 59(13) & -0.171 \ 58(33) \\ C(14) & 0.595 \ 59(31) & 0.428 \ 26(12) & -0.014 \ 36(25) & C(22A) & 0.328 \ 67(35) & 0.707 \ 59(13) & -0.171 \ 58(33) \\ C(15) & 0.580 \ 14(44) & 0.418 \ 22(17) & -0.136 \ 30(47) & C(26A) & 0.718 \ 32(35) & 0.709 \ 17(13) & -0.159 \ 19(33) \\ C(15) & 0.580 \ 14(44) & 0.418 \ 22(17) & -0.136 \ 30(47) & C(26A) & 0.718 \ 32(35) & 0.709 \ 17(13) & -0.159 \ 19(33) \\ C(17) & 0.616 \ 56(25) & 0.466 \ 71(10) & -0.381 \ 51(32) & C(27A) & 0.670 \ 88(35) & 0.694 \ 84(013) & -0.035 \ 84(33) \\ C(19) & 0.708 \ 92(25) & 0.444 \ 58(10) & -0.556 \ 25(32) & C(30A) & 0.704 \ 62(32) & 0.553 \ 15(12) & 0.210 \ 42(26) \\ C(20) & 0.778 \ 71(25) & 0.422 \ 52(10) & -0.488 \ 78(32) & C(3A) & 0.704 \ 62(32) & 0.553 \ 15(12) & 0.210 \ 42(26) \\ C(21) & 1.069 \ 20(30) & 0.340 \ 98(11) & -0.272 \ 95(35) & C(13A) & 0.702 \ 93(32) & 0.588 \ 84(11) & -0.029 \ 95(25) \\ C(22) & 1.135 \ 05(30) & 0.315 \ 18(11) & -0.279 \ 95(35) & C(13B) & 0.398 \ 23(45) & 0.096 \ 29(18) \ 0.4479 \ 57(51) \\ C(26) & 0.556 \ 17(33) & 0.289 \ 31(13) & -0.152 \ 92(34) & C(7B) & 0.441 \ 73(31) \ 0.159 \ 61(11) & 0.488 \ 69(35) \\ C(29) & 0.555 \ 74(31) & 0.420 \ 56(12) & 0.0376 \ 77(54) & C(6B) & 0.491 \ 93(56) \ 0.158 \ 96(31) & 0.159 \ 96(41) \\ C(30) & 0.448 \ 39(31) & 0.420 \ 56(12) & 0.0376 \ 77(54) & C(7B) \ 0.641 \ 37(31) \ 0.159 \ 30(11) & 0.458 \ 80(53) \\ C(25) & 0.555 \ 17(33) & 0.289 \ 31(13) & -0.050 \ 77(54) & C(7B) \ 0.641 \ 37(31) \ 0.159 \ 30(11) & 0.458 \ 32(25$	C(n)	$0.757 \ 00(46)$	0.234 20(22) 0.308 00(22)	-0.21301(03)	C(10A)	0.06047(23)	0.55211(10)	-0.490 98(33)
$ \begin{array}{c} C(3) & 0.535 \ M(3) & 0.535 \ M(2) & -0.535 \ M(2) & -0.535 \ M(2) & -0.575 \ M(2) & -0.557 \ M(2) & -0.558 \ M(2) \ $	C(0)	0.732 36(40) 0.630 46(33)	$0.308\ 00(22)$ 0.305\ 77(13)	-0.080.29(34)	C(13A)	1.037.43(23)	0.552 10(10) 0.573 25(10)	- 0.349 33(33)
$ \begin{array}{c} C(1) & 0.53 & 0.627 & 0.521 & 0.213 & 0.57 & C(23A) & 1.525 & 0.617 & 0.054 & 0.617 \\ C(12) & 0.647 & 0.348 & 70(18) & 0.091 & 72(54) & C(22A) & 1.354 & 60(31) & 0.713 & 56(11) & -0.156 & 71(34) \\ C(13) & 0.581 & 34(31) & 0.411 & 65(12) & 0.088 & 98(25) & C(24A) & 1.245 & 02(31) & 0.720 & 32(11) & -0.157 & 52(34) \\ C(14) & 0.539 & 99(31) & 0.428 & 26(12) & -0.014 & 36(25) & C(25A) & 0.828 & 67(35) & 0.707 & 59(13) & -0.171 & 58(33) \\ C(15) & 0.580 & 14(44) & 0.418 & 22(17) & -0.136 & 30(47) & C(26A) & 0.718 & 32(35) & 0.694 & 58(13) & -0.056 & 84(33) \\ C(16) & 0.667 & 71(45) & 0.447 & 18(17) & -0.177 & 55(48) & C(27A) & 0.670 & 88(35) & 0.694 & 58(13) & -0.056 & 84(33) \\ C(17) & 0.616 & 56(25) & 0.466 & 67(10) & -0.381 & 15(32) & C(29A) & 0.734 & 78(32) & 0.578 & 40(13) & 0.033 & 13(33) \\ C(18) & 0.627 & 85(25) & 0.466 & 67(10) & -0.504 & 11(32) & C(29A) & 0.794 & 18(32) & 0.577 & 58(12) & 0.204 & 92(26) \\ C(20) & 0.778 & 71(25) & 0.422 & 52(10) & -0.485 & 78(32) & C(31A) & 0.659 & 90(32) & 0.538 & 47(12) & 0.106 & 21(26) \\ C(21) & 1.069 & 20(30) & 0.340 & 98(11) & -0.272 & 95(35) & C(13B) & 0.762 & 93(32) & 0.548 & 23(12) & -0.002 & 96(26) \\ C(22) & 1.135 & 05(30) & 0.315 & 18(11) & -0.209 & 51(35) & C(13B) & 0.356 & 55(8) & 0.133 & 06(20) & 0.414 & 15(8) \\ C(24) & 0.981 & 38(30) & 0.276 & 77(11) & -0.151 & 93(35) & C(28B) & 0.377 & 93(24) & 0.096 & 29(18) & 0.479 & 57(31) \\ C(25) & 0.565 & 17(33) & 0.288 & 9(13) & -0.167 & 68(34) & C(4B) & 0.356 & 55(58) & 0.133 & 06(20) & 0.414 & 15(58) \\ C(27) & 0.410 & 13(33) & 0.304 & 54(13) & -0.050 & 77(34) & C(5B) & 0.228 & 99(31) & 0.159 & 96(11) & 0.488 & 99(35) \\ C(27) & 0.430 & 31(13) & 0.420 & 56(12) & 0.079 & 93(25) & C(18B) & 0.324 & 68(31) & 0.190 & 43(11) & 0.559 & 29(35) \\ C(28) & 0.475 & 51(33) & 0.288 & 69(13) & -0.152 & 92(34) & C(5B) & 0.232 & 93(31) & 0.159 & 6(111) & 0.488 & 99(35) \\ C(24) & 0.433 & 51(12) & -0.088 & 77(25) & C(11B) & 0.666 & 01(31) & 0.173 & 79(12) & 0.784 & 42(84) \\ C(3A) & 1.057 & 94(43) & 0.433 & 76(12) & -0.038 & 77(55) & C$	C(0)	0.585 58(33)	0.30577(13)	0.021.85(34)	C(20A)	1.037 + 5(23)	0.57525(10)	-0.7607(34)
$ \begin{array}{c} C(1) & 0.677 \ 0.347 \ 0.2617 \ 0.347 \ 70(18) & 0.091 \ 72(54) & C(23A) & 1.354 \ 0.36(11) & -0.156 \ 71(34) \\ C(13) & 0.581 \ 34(31) & 0.411 \ 65(12) & 0.088 \ 98(25) & C(24A) & 1.245 \ 02(31) & 0.713 \ 56(11) & -0.157 \ 72(34) \\ C(13) & 0.581 \ 94(31) & 0.411 \ 65(12) & 0.088 \ 98(25) & C(25A) & 0.582 \ 67(35) & 0.707 \ 59(13) & -0.171 \ 58(33) \\ C(15) & 0.580 \ 14(44) & 0.418 \ 22(17) & -0.136 \ 30(47) & C(26A) & 0.718 \ 32(35) & 0.709 \ 17(13) & -0.159 \ 19(33) \\ C(16) & 0.667 \ 71(45) & 0.447 \ 18(17) & -0.177 \ 55(48) & C(27A) & 0.670 \ 88(35) & 0.694 \ 58(13) & -0.056 \ 84(33) \\ C(17) & 0.616 \ 56(25) & 0.466 \ 671(10) & -0.381 \ 51(32) & C(28A) & 0.733 \ 79(35) & 0.678 \ 40(13) \ 0.033 \ 13(33) \\ C(18) & 0.627 \ 85(25) & 0.466 \ 671(10) & -0.504 \ 11(32) & C(29A) & 0.794 \ 18(32) & 0.577 \ 58(12) & 0.204 \ 92(26) \\ C(19) & 0.708 \ 92(25) & 0.444 \ 58(10) & -0.556 \ 25(32) & C(31A) & 0.794 \ 62(32) & 0.553 \ 15(12) & 0.2101 \ 42(26) \\ C(21) & 1.069 \ 20(30) & 0.340 \ 98(11) & -0.272 \ 95(35) & C(12A) & 0.724 \ 93(32) & 0.584 \ 23(12) & -0.002 \ 96(26) \\ C(21) & 1.069 \ 20(30) & 0.340 \ 98(11) & -0.272 \ 95(35) & C(12B) & 0.477 \ 91(24) & 0.072 \ 94(11) & 0.462 \ 57(33) \\ C(24) & 0.981 \ 38(30) & 0.276 \ 77(11) & -0.14 \ 899(35) & C(2B) & 0.477 \ 91(24) & 0.072 \ 19(11) & 0.473 \ 83(33) \\ C(24) & 0.981 \ 38(30) & 0.278 \ 91(13) & -0.152 \ 92(34) & C(7B) & 0.326 \ 58(31) & 0.199 \ 43(11) \ 0.452 \ 57(33) \\ C(27) & 0.410 \ 13(33) & 0.304 \ 54(13) & -0.052 \ 71(34) & C(7B) & 0.326 \ 59(31) & 0.159 \ 61(11) \ 0.488 \ 69(35) \\ C(26) & 0.455 \ 00(33) & 0.288 \ 69(13) & -0.152 \ 92(34) & C(7B) & 0.324 \ 68(31) \ 0.199 \ 43(11) \ 0.452 \ 87(33) \\ C(26) & 0.455 \ 00(33) & 0.288 \ 69(13) & -0.152 \ 92(34) & C(7B) \ 0.324 \ 68(31) \ 0.199 \ 43(11) \ 0.452 \ 87(33) \\ C(25) & 0.455 \ 00(33) & 0.288 \ 69(13) & -0.056 \ 77(34) \ C(18) \ 0.324 \ 68(31) \ 0.199 \ 43(11) \ 0.458 \ 63(32) \\ C(25) & 0.455 \ 00(33) & 0.288 \ 69(13) & -0.056 \ 77(34) \ C(18) \ 0.324 \ 68(31) \ 0.199 \ 43(11) \ 0.559 \ 92(35) \ 0.159 \$	C(10)	0.505 50(55)	0.32102(13) 0.34157(20)	0.02185(54) 0.11886(54)	C(21A)	1.326 35(31) 1 396 37(31)	0.03480(11)	-0.27007(34) -0.21508(34)
$\begin{array}{c} C(13) \\ C(13) \\ C(14) \\ C(13) \\ C(14) \\ C(14) \\ C(15) \\ C(14) \\ C(14) \\ C(15) \\$	C(12)	0.047 92(47) 0.677 03(45)	0.341.57(20) 0.384.70(18)	0.091.72(54)	C(23A)	1.354 60(31)	$0.000\ 00(11)$	-0.15671(34)
$ \begin{array}{c} C(14) & 0.539 \; 9(31) & 0.428 \; 26(12) & -0.014 \; 36(25) & C(25A) & 0.828 \; 6(15) & 0.707 \; 9(13) & -0.171 \; 58(33) \\ C(15) & 0.580 \; 14(44) & 0.418 \; 22(17) & -0.136 \; 50(47) & C(26A) & 0.718 \; 32(35) & 0.709 \; 17(13) & -0.159 \; 19(33) \\ C(15) & 0.667 \; 71(45) & 0.447 \; 18(17) & -0.177 \; 55(48) & C(27A) & 0.670 \; 88(35) & 0.694 \; 58(13) & -0.056 \; 84(33) \\ C(17) & 0.616 \; 56(25) & 0.466 \; 71(10) & -0.381 \; 51(32) & C(28A) & 0.733 \; 79(35) & 0.678 \; 40(13) & 0.033 \; 13(33) \\ C(18) & 0.627 \; 85(25) & 0.446 \; 67(10) & -0.504 \; 11(32) & C(29A) & 0.794 \; 18(32) & 0.577 \; 58(12) & 0.204 \; 92(26) \\ C(20) & 0.778 \; 71(25) & 0.442 \; 52(10) & -0.485 \; 78(32) & C(31A) & 0.659 \; 00(32) & 0.538 \; 47(12) & 0.106 \; 21(26) \\ C(21) & 1.069 \; 20(50) & 0.340 \; 98(11) & -0.272 \; 95(35) & C(32A) & 0.702 \; 93(32) & 0.548 \; 23(12) & -0.002 \; 96(26) \\ C(22) & 1.35 \; 05(30) & 0.315 \; 18(11) & -0.209 \; 51(35) & C(1B) & 0.562 \; 11(24) & 0.052 \; 34(11) & 0.462 \; 57(33) \\ C(23) \; 1.091 \; 14(30) & 0.283 \; 07(11) & -0.154 \; 99(35) & C(3B) & 0.398 \; 23(45) & 0.096 \; 29(18) \; 0.477 \; 97(51) \\ C(25) & 0.565 \; 17(33) & 0.288 \; 9(13) & -0.167 \; 68(34) & C(4B) & 0.356 \; 55(58) & 0.133 \; 06(20) & 0.414 \; 15(58) \\ C(26) & 0.455 \; 00(33) & 0.288 \; 69(13) & -0.152 \; 92(34) & C(6B) & 0.324 \; 68(31) & 0.199 \; 43(11) & 0.458 \; 89(35) \\ C(28) \; 0.475 \; 41(33) & 0.321 \; 00(13) & 0.036 \; 61(34) & C(7B) & 0.441 \; 12(50) & 0.200 \; 22(25) & 0.565 \; 30(65) \\ C(29) \; 0.535 \; 74(31) & 0.420 \; 56(12) & 0.197 \; 93(25) & C(8B) & 0.491 \; 29(45) & 0.187 \; 83(23) & 0.676 \; 90(61) \\ C(31) \; 0.466 \; 64(31) & 0.446 \; 07(12) & 0.033 \; 53(25) & C(1B) & 0.666 \; 01(31) & 0.173 \; 79(12) & 0.784 \; 44(28) \\ C(31) \; 0.466 \; 64(31) & 0.462 \; 67(12) & 0.100 \; 18(25) & C(1B) & 0.666 \; 83(21) & 0.156 \; 80(19) & 0.888 \; 67(51) \\ C(1A) \; 0.940 \; 61(23) & 0.554 \; 25(10) & -0.304 \; 64(33) & C(12B) & 0.569 \; 91(43) & 0.178 \; 83(23) & 0.676 \; 90(61) \\ C(31) \; 0.466 \; 64(31) & 0.462 \; 57(12) & 0.100 \; 18(25) & C(1B) & 0.666 \; 83(21) & 0.155 \; 80(19) & 0.888 \; 87(51) \\ C(4A) \; 1.155$	C(12)	0.581 34(31)	0.301.76(10) 0.411.65(12)	0.088.98(25)	C(24A)	1.33700(31) 1.24502(31)	0.719 30(11) 0.720 32(11)	-0.15752(34)
$\begin{array}{c} C(15) & 0.580 14(44) & 0.418 22(17) & -0.135 30(47) & C(26A) & 0.718 32(35) & 0.709 17(15) & -0.159 19(33) \\ C(16) & 0.667 71(45) & 0.447 18(17) & -0.177 55(48) & C(27A) & 0.670 88(35) & 0.694 58(13) & -0.056 84(33) \\ C(17) & 0.616 56(25) & 0.466 67(10) & -0.504 11(32) & C(28A) & 0.733 79(35) & 0.678 40(13) & 0.033 13(33) \\ C(18) & 0.627 85(25) & 0.466 67(10) & -0.504 11(32) & C(29A) & 0.794 18(32) & 0.577 58(12) & 0.204 92(26) \\ C(19) & 0.708 92(25) & 0.444 58(10) & -0.556 25(32) & C(30A) & 0.704 62(32) & 0.538 47(12) & 0.106 21(26) \\ C(21) & 1.069 20(30) & 0.340 98(11) & -0.272 95(35) & C(32A) & 0.702 93(32) & 0.548 23(12) & -0.002 96(26) \\ C(22) & 1.135 05(30) & 0.315 18(11) & -0.209 51(35) & C(1B) & 0.562 11(24) & 0.052 34(11) & 0.462 57(33) \\ C(24) & 0.981 38(30) & 0.276 77(11) & -0.151 93(35) & C(2B) & 0.477 91(24) & 0.072 19(11) & 0.477 38(33) \\ C(26) & 0.455 00(33) & 0.288 69(13) & -0.152 92(34) & C(5B) & 0.398 23(45) & 0.096 29(18) & 0.479 57(51) \\ C(25) & 0.565 17(33) & 0.288 39(13) & -0.167 68(34) & C(4B) & 0.356 55(58) & 0.133 06(20) & 0.414 15(58) \\ C(27) & 0.410 13(33) & 0.304 54(13) & -0.050 77(34) & C(6B) & 0.324 68(31) & 0.190 43(11) & 0.559 29(35) \\ C(28) & 0.475 41(33) & 0.321 00(13) & 0.036 61(34) & C(7B) & 0.441 12(50) & 0.200 22(25) & 0.565 30(65) \\ C(30) & 0.448 39(31) & 0.446 07(12) & 0.203 57(25) & C(1B) & 0.666 58(31) & 0.189 43(23) & 0.676 90(61) \\ C(30) & 0.448 39(31) & 0.446 07(12) & 0.203 57(25) & C(1B) & 0.666 58(31) & 0.187 83(23) & 0.676 90(61) \\ C(31) & 0.406 64(31) & 0.462 67(12) & 0.100 18(25) & C(10B) & 0.666 58(31) & 0.189 43(23) & 0.676 90(61) \\ C(24) & 1.025 20(23) & 0.574 30(10) & -0.357 19(33) & C(11B) & 0.666 58(31) & 0.080 37(17) & 0.588 87(51) \\ C(1A) & 0.940 61(23) & 0.554 25(10) & -0.308 67(56) & C(11B) & 0.666 58(31) & 0.080 37(17) & 0.588 87(56) \\ C(1A) & 1.045 68(57) & 0.632 87(20) & -0.358 87(56) & C(11B) & 0.661 87(45) & 0.080 37(17) & 0.538 82(46) \\ C(5A) & 1.172 7(13) & 0.661 56(11) & -0.277 60(34) & C(17B) & 0.652 9(24) & 0.031 17(11) & 0.595 90(47) $	C(13)	0.539.59(31)	0.428.26(12)	-0.01436(25)	C(25A)	0.828.67(35)	0.720.52(11) 0.707.59(13)	-0.17158(33)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(15)	0.58014(44)	$0.418\ 22(17)$	$-0.136\ 30(47)$	C(26A)	0.718(32(35))	0.709 17(13)	-0.15919(33)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(16)	0.667 71(45)	$0.447 \ 18(17)$	-0.17755(48)	C(27A)	0.670 88(35)	0.694 58(13)	-0.05684(33)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(17)	0.616 56(25)	0.466 71(10)	-0.38151(32)	C(28A)	0.73379(35)	0.67840(13)	0.033 13(33)
$ \begin{array}{c} C(19) & 0.708\ 92(25) & 0.444\ 58(10) & -0.556\ 25(32) & C(30A) & 0.704\ 62(32) & 0.553\ 15(12) & 0.210\ 14(26) \\ C(20) & 0.778\ 71(25) & 0.422\ 52(10) & -0.485\ 78(32) & C(31A) & 0.659\ 90(32) & 0.538\ 47(12) & 0.106\ 21(26) \\ C(21) & 1.069\ 20(30) & 0.340\ 98(11) & -0.272\ 95(35) & C(32A) & 0.702\ 93(32) & 0.548\ 23(12) & -0.002\ 96(26) \\ C(22) & 1.135\ 05(30) & 0.315\ 18(11) & -0.209\ 51(35) & C(1B) & 0.562\ 11(24) & 0.052\ 34(11) & 0.462\ 57(33) \\ C(23) & 1.091\ 14(30) & 0.283\ 07(11) & -0.14\ 899(35) & C(2B) & 0.477\ 91(24) & 0.072\ 19(11) & 0.407\ 38(33) \\ C(24) & 0.981\ 38(30) & 0.276\ 77(11) & -0.151\ 93(35) & C(3B) & 0.398\ 23(45) & 0.096\ 29(18) & 0.479\ 57(51) \\ C(25) & 0.565\ 17(33) & 0.289\ 31(13) & -0.157\ 98(34) & C(4B) & 0.335\ 55(58) & 0.133\ 06(20) & 0.414\ 15(58) \\ C(26) & 0.455\ 00(33) & 0.288\ 69(13) & -0.152\ 92(34) & C(5B) & 0.285\ 99(31) & 0.159\ 61(11) & 0.488\ 69(35) \\ C(27) & 0.410\ 13(33) & 0.304\ 54(13) & -0.050\ 77(34) & C(6B) & 0.324\ 68(31) & 0.190\ 43(11) & 0.559\ 29(35) \\ C(28) & 0.475\ 41(33) & 0.321\ 00(13) & 0.036\ 61(34) & C(7B) & 0.441\ 12(50) & 0.200\ 22(25) & 0.565\ 30(65) \\ C(30) & 0.448\ 39(31) & 0.446\ 07(12) & 0.203\ 53(25) & C(9B) & 0.614\ 37(31) & 0.189\ 13(12) & 0.684\ 89(28) \\ C(31) & 0.406\ 64(31) & 0.462\ 67(12) & 0.100\ 18(25) & C(10B) & 0.666\ 01(31) & 0.173\ 79(12) & 0.784\ 44(28) \\ C(32) & 0.452\ 24(31) & 0.453\ 76(12) & -0.008\ 77(25) & C(11B) & 0.668\ 83(31) & 0.085\ 02(12) & 0.888\ 67(51) \\ C(3A) & 1.105\ 79(48) & 0.599\ 51(19) & -0.283\ 54(53) & C(12B) & 0.566\ 91(43) & 0.112\ 89(18) & 0.870\ 29(54) \\ C(3A) & 1.105\ 79(48) & 0.599\ 51(19) & -0.283\ 54(53) & C(11B) & 0.666\ 87(31) & 0.085\ 02(12) & 0.866\ 50(24) \\ C(5A) & 1.218\ 97(31) & 0.661\ 56(11) & -0.276\ 88(34) & C(11B) & 0.661\ 87(45) & 0.085\ 03(11) & 0.789\ 90(47) \\ C(AA) & 1.105\ 79(48) & 0.599\ 51(19) & -0.283\ 54(55) & C(11B) & 0.666\ 87(31) & 0.085\ 03(11) & 0.799\ 30(33) \\ C(AA) & 1.105\ 79(48) & 0.599\ 51(19) & -0.283\ 54(55) & C(11B) & 0.661\ 87(45) & 0.085\ 03(11) & 0.595\ 90(47$	C(18)	0.627 85(25)	0.466 67(10)	-0.50411(32)	C(29A)	0.794 18(32)	0.577 58(12)	0.204 92(26)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(19)	0.708 92(25)	0.444 58(10)	-0.55625(32)	C(30A)	0.704 62(32)	0.553 15(12)	0.210 14(26)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(20)	0.778 71(25)	0.422 52(10)	-0.48578(32)	C(31A)	0.659 00(32)	0.538 47(12)	0.106 21(26)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(21)	1.069 20(30)	0.340 98(11)	-0.27295(35)	C(32A)	0.702 93(32)	0.548 23(12)	-0.00296(26)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(22)	1.135 05(30)	0.315 18(11)	-0.209 51(35)	C(1B)	0.562 11(24)	0.052 34(11)	0.462 57(33)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(23)	1.091 14(30)	0.283 07(11)	-0.14 899(35)	C(2B)	0.477 91(24)	0.072 19(11)	0.407 38(33)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(24)	0.981 38(30)	0.276 77(11)	-0.151 93(35)	C(3B)	0.398 23(45)	0.096 29(18)	0.479 57(51)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(25)	0.565 17(33)	0.289 31(13)	-0.167 68(34)	C(4B)	0.356 55(58)	0.133 06(20)	0.414 15(58)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(26)	0.455 00(33)	0.288 69(13)	-0.152 92(34)	C(5B)	0.285 99(31)	0.159 61(11)	0.488 69(35)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(27)	0.410 13(33)	0.304 54(13)	-0.050 77(34)	C(6B)	0.324 68(31)	0.190 43(11)	0.559 29(35)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(28)	0.475 41(33)	0.321 00(13)	0.036 61(34)	C(7B)	0.441 12(50)	0.200 22(25)	0.565 30(65)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(29)	0.535 74(31)	0.420 56(12)	0.197 93(25)	C(8B)	0.491 29(45)	0.187 83(23)	0.676 90(61)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(30)	0.448 39(31)	0.446 07(12)	0.203 53(25)	C(9B)	0.614 37(31)	0.189 13(12)	0.684 89(28)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(31)	0.406 64(31)	0.462 67(12)	0.100 18(25)	C(10B)	0.666 01(31)	0.173 79(12)	0.784 44(28)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(32)	0.452 24(31)	0.453 76(12)	-0.008 77(25)	C(11B)	0.603 82(51)	0.156 80(19)	0.888 67(51)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(1A)	0.940 61(23)	0.554 25(10)	-0.304 64(33)	C(12B)	0.569 91(43)	0.112 89(18)	0.870 29(54)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(2A)	1.025 20(23)	0.574 30(10)	-0.357 19(33)	C(13B)	0.666 58(31)	0.085 02(12)	0.866 50(24)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(3A)	1.105 79(48)	0.599 51(19)	-0.283 54(53)	C(14B)	0.707 25(31)	0.070 00(12)	0.761 10(24)
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	C(4A)	1.154 68(57)	0.632 87(20)	-0.350 87(56)	C(15B)	0.661 87(45)	0.080 37(17)	0.638 82(46)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		1.218 97(31)	0.001 30(11)	-0.27088(34)	C(16B)	0.379.33(43)	0.05051(17)	0.595 90(47)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		1.1//21(31)	0.094 32(11)	-0.217 00(34)	C(1/B)	0.035 29(24)	0.031 1/(11)	0.395 03(33)
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	C(A)	1.030 28(48)	0.701 82(23)	- 0.213 00(09)	C(10B)	0.024 28(24)	0.02980(11)	0.2/2.30(33)
C(10A)         0.844 14(35)         0.676 82(13)         0.020 75(33)         C(21B)         0.176 61(31)         0.152 90(11)         0.479 98(35)           C(11A)         0.910 87(53)         0.658 38(20)         0.115 95(52)         C(22B)         0.105 93(31)         0.177 03(11)         0.541 86(35)	C(0A)	1.011 8/(4/)	0.009 37(23)	-0.090 38(39)	C(19B)	0.340 08(24)	0.049 /0(11)	0.21/11(33)
C(11A) = 0.910 87(53) = 0.658 38(20) = 0.115 95(52) = C(22B) = 0.105 93(31) = 0.177 03(11) = 0.419 98(53)	C(10A)	0.844 14(25)	0.071 + 1(13) 0.676 $82(13)$	-0.00101(33) 0.02075(22)	C(20D)	0.700.09(24)	0.070 87(11)	0.204 03(33)
	C(11A)	$0.910\ 87(53)$	0.658 38(20)	0.115 95(52)	C(22B)	0.105 93(31)	0.177 03(11)	0,541 86(35)

Table 9 (continued)

Atom	x/A	<i>y</i> / <i>B</i>	z/C	Atom	x/A	<i>y</i> / <i>B</i>	z/C
C(23B)	0.144 63(31)	0.207 86(11)	0.612 46(35)	C(12C)	0.169 34(46)	0.385 89(19)	0.141 18(58)
C(24B)	0.254 00(31)	0.214 56(11)	0.621 18(35)	C(13C)	0.074 60(32)	0.413 61(12)	0.148 25(26)
C(25B)	0.673 71(31)	$0.203\ 32(12)$	0.590 01(28)	C(14C)	0.036 54(32)	0.429 21(12)	0.254 12(26)
C(26B)	0.784 68(31)	0.202 16(12)	0.594 67(28)	C(15C)	0.082 30(48)	0.418 15(18)	0.375 66(47)
C(27B)	0.836 32(31)	0.186 82(21)	0.694 22(28)	C(16C)	0.164 38(42)	0.448 64(16)	0.421 44(48)
C(28B)	0.776 98(31)	0.172 63(12)	0.789 11(28)	C(17C)	0.11001(24)	0.466 10(10)	0.625 06(32)
C(29B)	0.713 21(31)	0.074 43(12)	0.974 24(24)	C(18C)	0.12012(24)	0.465 56(10)	0.747 93(32)
C(30B)	0.800 51(31)	0.048 82(12)	0.976 58(24)	C(19C)	0.202 29(24)	0.444 04(10)	0.801 28(32)
C(31B)	0.841 19(31)	0.033 80(12)	0.871 19(24)	C(20C)	0.274 35(24)	0.423 06(10)	0.731 76(32)
C(32B)	0.794 56(31)	0.04 439(12)	0.763 45(24)	C(21C)	0.564 95(31)	0.343 44(12)	0.525 81(36)
C(1C)	0.182 07(24)	0.445 12(10)	0.555 54(32)	C(22C)	0.634 20(31)	0.319 79(12)	0.460 42(36)
C(2C)	0.264 24(24)	0.423 60(10)	0.608 89(32)	C(23C)	0.593 82(31)	0.289 87(12)	0.387 55(36)
C(3C)	0.34 400(50)	0.399 97(20)	0.534 18(58)	C(24C)	0.484 20(31)	0.283 59(12)	0.380 07(36)
C(4C)	0.384 93(52)	0.363 28(19)	0.597 99(59)	C(25C)	0.065 71(33)	0.292 81(12)	0.411 44(30)
C(5C)	0.455 32(31)	0.337 17(12)	0.518 33(36)	C(26C)	-0.04523(33)	0.294 06(12)	0.405 89(30)
C(6C)	0.414 95(31)	0.307 24(12)	0.445 46(36)	C(27C)	-0.09609(33)	0.31 059(12)	0.307 54(30)
C(7C)	0.298 25(53)	0.298 87(27)	0.436 71(68)	C(28C)	-0.03601(33)	0.32 587(12)	0.214 74(30)
C(8C)	0.247 63(49)	0.309 40(24)	0.325 27(59)	C(29C)	0.026 43(32)	0.424 13(12)	0.041 28(26)
C(9C)	0.125 78(33)	0.308 07(12)	0.318 64(30)	C(30C)	-0.05979(32)	0.450 25(12)	0.040 17(26)
C(10Ć)	0.074 92(33)	0.324 60(12)	0.220 29(30)	C(31C)	- 0.097 85(32)	0.465 85(12)	0.146 03(26)
C(11C)	0.137 55(48)	0.342 12(18)	0.121 07(55)	C(32C)	-0.049 68(32)	0.455 33(12)	0.253 01(26)

ionization experiments were carried out, clusters of compounds of Table 1 and ammonia were observed (*e.g.*, compound **18** in positive-Cl NH<sub>3</sub>, present peaks at 748 and 763 Th).\*

Reaction Between  $\alpha, \alpha'$ -Dibromo-o-xylene and Sodium.—To a solution of a,a'-dibromo-o-xylene (12.0 g, 45 mmol) in dry dioxane (450 cm<sup>3</sup>) was added sodium (8.3 g, 361 mmol), finely dispersed in dry dioxane. The resultant suspension was heated at reflux temperature acquiring a deep blue colour. After 52 h the solvent was evaporated off under reduced pressure. The solid obtained was taken up in benzene (4.150 cm<sup>3</sup>), treated with ethanol to destroy the excess of sodium, and the mixture neutralized with 50% aqueous hydrochloric acid. The organic layer was then separated and the solvent eliminated. The residue was treated again with dichloromethane and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> affording 5.6 g of crude product after evaporation of the organic solvent. Column chromatography on silica gel (Merck 60, 70-230 mesh) with hexane and mixtures of hexane-benzene of increasing polarity gave the products shown in Table 1.

When the reaction was performed under similar conditions by refluxing for 24 h in toluene (450 cm<sup>3</sup>) instead of dioxane, a residue of 10.4 g was isolated by extraction with diethyl ether, from which only acyclic hydrocarbons were identified: xylene (60%), 1,2-di(*o*-tolyl)ethane (13),<sup>17</sup>  $R_f(CHCl_3)$  0.71;  $\delta(CDCl_3)$ : 7.3–7.1 (8 H, m, ArH), 2.89 (4 H, s, ArCH<sub>2</sub>CH<sub>2</sub>Ar) and 2.35 (6 H, s, ArMe); 1,2-bis(*o*-ethoxymethylphenyl)ethane (17), oil,  $M^{*+}$ 298 (C<sub>20</sub>H<sub>26</sub>O<sub>2</sub>) (Calc.: C, 80.50; H, 8.8. Found: C, 80.33; H, 8.91)  $R_f(CHCl_3)$ : 0.65;  $\delta(CDCl_3)$ : 7.2–7.1 (8 H, m, ArH), 4.50 (4 H, s, ArCH<sub>2</sub>O), 3.56 (4 H, q, OCH<sub>2</sub>Me), 2.34 (4 H, s, ArCH<sub>2</sub>CH<sub>2</sub>Ar) and 1.25 (6 H, t, OCH<sub>2</sub>Me); and  $\alpha,\alpha'$ -diethoxy*o*-xylene (18), oil,  $M^{*+}$  194 (C<sub>12</sub>H<sub>18</sub>O<sub>2</sub>) (Calc.: C, 74.2; H, 9.3. Found: C, 73.97; H, 9.52)  $R_f(CHCl_3)$ : 0.46;  $\delta(CDCl_3)$ : 7.2–7.4 (8 H, m, ArH), 4.58 (4 H, s, ArCH<sub>2</sub>O), 3.54 (4 H, q, OCH<sub>2</sub>Me) and 1.24 (6 H, t, OCH<sub>2</sub>Me).

6,7,13,14-*Tetrahydrodipyrido*[1,2-a:1,2-e]*diazocinium Dibromide* (11).—This was prepared according to the literature<sup>21</sup> by dimerization of 2-( $\beta$ -bromoethyl)pyridine in 77% yield, m.p. 239–240 °C (from methanol-water, 2:1); lit.<sup>21</sup>: 238–240 °C; *R*<sub>f</sub>(ethyl ether): 0.52. This compound presents a mass spectrum with the most intense peak at 211 Th ( $C^{++}-H^{+}$ ) both using chemical ionization <sup>30</sup> and thermospray positive mode.

Crystallography.—Cell parameters and orientation matrices were obtained by least-squares refinement using 30 reflections in the range  $20 < \theta < 35^{\circ}$ . The data collection, at room temperature, was performed for the four compounds on a Siemens AED diffractometer using  $\theta/2\theta$  scan mode, scan speed  $3-12^{\circ}$  min<sup>-1</sup>, scan width (1.20 + 0.14 tan  $\theta$ ) and  $\theta$  in the range 3-70°. One standard reflection was measured every 100. The reflections were corrected for Lorentz and polarization; absorption correction was applied for compound C14H16N2Br2. 2H<sub>2</sub>O and C<sub>32</sub>H<sub>32</sub> following Uguzzoli.<sup>31</sup> All the calculations were performed on the Gould computer system of the Centro di Strutturistica Diffrattometrica del CNR, Parma, Italy. The structures were solved using the SHELXS program;<sup>32</sup> the refinement (anisotropic for heavy atoms and rigid-body constraint for the phenyl ring, isotropic for the hydrogens) was performed by full-matrix least-squares (block-matrix for the C<sub>32</sub>H<sub>32</sub> compound owing to the high number of parameters to be refined) using the program SHELX76 with the form factors included.<sup>33</sup> Geometric calculations were performed with PARST <sup>34</sup> and CRYSRULER package; <sup>35</sup> drawings were made with the PLUTO<sup>24</sup> program through CRYSRULER package.<sup>†,35</sup> Atomic coordinates and selected bond distances and bond angles are given in Tables 9 and 10.

*NMR Spectroscopy.*—The NMR spectra were recorded on a Bruker AC200, working at 200.13 MHz for <sup>1</sup>H and 50.32 MHz for <sup>13</sup>C, and a Varian unity, operating at 499.84 MHz for <sup>1</sup>H, spectrometers. Both pieces of apparatus were equipped with variable-temperature units. The temperature of the probe was calibrated by the methanol standard method ( $\pm 0.5$  K). <sup>1</sup>H and <sup>13</sup>C chemical shifts ( $\delta$ ) are given from internal TMS with an accuracy of  $\pm 0.01$  and  $\pm 0.1$  ppm, respectively. Coupling constants J were measured with a digital resolution of 0.2 and 0.6 Hz, respectively. The data acquisition parameters for the heteronuclear (<sup>1</sup>H–<sup>13</sup>C) 2D-correlations experiments were F<sub>1</sub>

<sup>\* 1</sup> Th (thomson) = a mass/charge ratio of 1 Da per unit of atomic charge. Rapid. Commun. Mass. Spectrum., 1991, 5, 93.

<sup>†</sup> Lists of thermal components, hydrogen parameters and full lists of bond distances, bond angles and torsion angles have been deposited at the Cambridge Crystallographic Data Centre (CCDC). For details of the deposition scheme, see 'Instructions for Authors', J. Chem. Soc., Perkin Trans. 2, 1992, issue 1.

Table 10 Selected bond distances/Å and bond angles/° for compounds 1, 11, 12 and 16  $\,$ 

1							
C(1)-C(2) C(1)-C(6) C(1)-C(7) C(2)-C(3) C(2)-C(8)	1.395(2) 1.395(2) 1.514(2) 1.395(2) 1.518(2)	C(3)-C(4) C(4)-C(5) C(5)-C(6) C(7)-C(8A)	1.395(2) 1.395(2) 1.395(2) 1.553(2)	C(6)-C(1)-C(7) C(2)-C(1)-C(7) C(2)-C(1)-C(6) C(1)-C(2)-C(8) C(1)-C(2)-C(3) C(3)-C(2)-C(8)	118.2(1) 121.8(1) 120.0(1) 121.2(1) 120.0(1) 118.7(1)	C(2)-C(3)-C(4) C(3)-C(4)-C(5) C(4)-C(5)-C(6) C(1)-C(7)-C(8A) C(2)-C(8)-C(7A)	120.0(1) 120.0(1) 120.0(1) 114.7(1) 114.8(1)
11 N(1)-C(2) N(1)-C(6) N(1)-C(7) C(2)-C(3) C(2)-C(3) C(2)-C(8) C(3)-C(4)	1.372(5) 1.361(6) 1.483(6) 1.368(6) 1.496(5) 1.383(7)	C(4)-C(5) C(5)-C(6) C(7)-C(8A) C(6)-N(1)-C(7) C(2)-N(1)-C(7)	1.374(7) 1.377(6) 1.542(5) 119.1(3) 120.7(3)	C(2)-N(1)-C(6) N(1)-C(2)-C(8) N(1)-C(2)-C(3) C(3)-C(2)-C(8) C(2)-C(3)-C(4) C(3)-C(4)-C(5)	120.2(3) 119.6(3) 119.2(3) 121.1(3) 120.9(4) 119.5(4)	C(5)-C(4)-H(4) C(4)-C(5)-C(6) N(1)-C(6)-C(5) N(1)-C(7)-C(8A) C(2)-C(8)-C(7A)	118.6(5) 119.1(4) 121.1(4) 112.7(3) 114.5(3)
12 C(1)-C(2) C(1)-C(12) C(2)-C(3) C(3)-C(4) C(4)-C(5) C(5)-C(6) C(5)-C(6) C(6)-C(7) C(7)-C(8)	1.395(2) 1.521(2) 1.517(3) 1.538(4) 1.523(3) 1.395(2) 1.520(3) 1.537(4)	C(8)-C(9) C(9)-C(10) C(10)-C(11) C(11)-C(12) C(12)-C(1)-C(13) C(2)-C(1)-C(12) C(1)-C(2)-C(3)	1.522(3) 1.395(2) 1.518(3) 1.533(4) ) 117.7(2) 122.3(2) 122.6(2)	C(3)-C(2)-C(16) C(2)-C(3)-C(4) C(3)-C(4)-C(5) C(4)-C(5)-C(17) C(4)-C(5)-C(6) C(5)-C(6)-C(7) C(7)-C(6)-C(20) C(6)-C(7)-C(8)	117.4(2) 117.5(2) 110.6(2) 117.4(2) 122.5(2) 123.6(2) 116.3(2) 113.7(2)	C(7)-C(8)-C(9) C(8)-C(9)-C(21) C(8)-C(9)-C(10) C(9)-C(10)-C(11) C(11)-C(10)-C(24) C(10)-C(11)-C(12) C(1)-C(12)-C(11)	112.3(2) 116.8(2) 123.1(2) 123.0(2) 117.0(2) 111.8(2) 114.6(2)
$\begin{array}{c} \textbf{16} \\ C(1)-C(2) \\ C(1)-C(16) \\ C(2)-C(3) \\ C(3)-C(4) \\ C(4)-C(5) \\ C(5)-C(6) \\ C(5)-C(6) \\ C(6)-C(7) \\ C(7)-C(8) \\ C(8)-C(9) \\ C(8)-C(9) \\ C(9)-C(10) \\ C(10)-C(11) \\ C(10)-C(11) \\ C(11)-C(12) \\ C(12)-C(13) \\ C(13)-C(14) \\ C(14)-C(15) \\ C(15)-C(16) \\ \end{array}$	$\begin{array}{c} 1.395(5)\\ 1.532(6)\\ 1.492(6)\\ 1.585(7)\\ 1.533(7)\\ 1.395(5)\\ 1.516(7)\\ 1.534(9)\\ 1.542(7)\\ 1.395(6)\\ 1.515(7)\\ 1.523(9)\\ 1.506(7)\\ 1.395(4)\\ 1.512(6)\\ 1.544(8)\end{array}$	$\begin{array}{c} C(1A)-C(2A)\\ C(1A)-C(16A)\\ C(2A)-C(3A)\\ C(3A)-C(4A)\\ C(3A)-C(5A)\\ C(5A)-C(5A)\\ C(5A)-C(6A)\\ C(6A)-C(7A)\\ C(7A)-C(8A)\\ C(8A)-C(9A)\\ C(10A)-C(11A)\\ C(11A)-C(12A)\\ C(12A)-C(13A)\\ C(13A)-C(14A)\\ C(14A)-3(15A)\\ C(15A)-C(16A)\\ \end{array}$	1.395(4) 1.518(6) 1.558(7) 1.491(9) 1.509(8) 1.395(5) 1.541(7) 1.492(10) 1.523(7) 1.395(6) 1.495(7) 1.566(9) 1.528(7) 1.395(4) 1.534(6) 1.558(8)	$\begin{array}{c} C(1B)-C(2B)\\ C(1B)-C(16B)\\ C(2B)-C(3B)\\ C(3B)-C(4B)\\ C(4B)-C(5B)\\ C(5B)-C(6B)\\ C(6B)-C(7B)\\ C(7B)-C(8B)\\ C(7B)-C(8B)\\ C(9B)-C(10B)\\ C(10B)-C(11B)\\ C(10B)-C(11B)\\ C(11B)-C(12B)\\ C(12B)-C(13B)\\ C(13B)-C(14B)\\ C(14B)-C(15B)\\ C(15B)-C(16B)\\ \end{array}$	1.395(5) 1.523(6) 1.529(7) 1.533(9) 1.518(8) 1.395(5) 1.500(7) 1.468(10) 1.548(7) 1.395(5) 1.523(7) 1.552(9) 1.535(7) 1.395(4) 1.533(6) 1.521(8)	C(1C)-C(2C) C(1C)-C(16C) C(2C)-C(3C) C(3C)-C(4C) C(4C)-C(5C) C(5C)-C(6C) C(5C)-C(6C) C(6C)-C(7C) C(7C)-C(8C) C(8C)-C(9C) C(8C)-C(9C) C(9C)-C(10C) C(10C)-C(11C) C(10C)-C(11C) C(11C)-C(12C) C(12C)-C(13C) C(13C)-C(14C) C(14C)-C(15C) C(15C)-C(16C)	$\begin{array}{c} 1.395(5)\\ 1.536(6)\\ 1.537(7)\\ 1.519(9)\\ 1.542(6)\\ 1.395(6)\\ 1.495(8)\\ 1.452(11)\\ 1.532(7)\\ 1.395(5)\\ 1.496(7)\\ 1.543(9)\\ 1.515(7)\\ 1.395(5)\\ 1.533(6)\\ 1.542(8)\end{array}$
$\begin{array}{c} C(16)-C(1)-C(2)-C(1)-C(2)-C(1)-C(2)-C(3)-C(2)-C(3)-C(2)-C(3)-C(2)-C(3)-C(3)-C(4)-C(3)-C(4)-C(5)-C(6)-C(7)-C(6)-C(7)-C(6)-C(7)-C(6)-C(7)-C(6)-C(7)-C(8)-C(9)-C(10)-C(11)-C(10)-C(11)-C(10)-C(11)-C(10)-C(11)-C(12)-C(13)-C(14)-C(13)-C(14)-C(15)-C(15)-C(14)-C(15)$	C(17) $C(16)$ $C(3)$ $C(20)$ $C(4)$ $C(5)$ $C(21)$ $C(6)$ $C(7)$ $C(24)$ $C(8)$ $C(9)$ $C(25)$ $C(10)$ $-C(11)$ $-C(28)$ $-C(12)$ $-C(12)$ $-C(12)$ $-C(13)$ $0-C(14)$ $0-C(15)$ $A)-C(16)$ $-C(15)$ $A)-C(16A)$ $A)-C(16A)$ $A)-C(20A)$ $A)-C(21A)$ $A)-C(5A)$ $A)-C(5A)$	$\begin{array}{c} 115.7(3)\\ 124.3(3)\\ 119.6(4)\\ 120.4(3)\\ 112.4(3)\\ 115.2(4)\\ 117.6(4)\\ 122.2(4)\\ 123.0(4)\\ 117.0(4)\\ 108.9(5)\\ 116.2(5)\\ 121.9(4)\\ 118.1(4)\\ 123.6(4)\\ 116.4(4)\\ 113.3(5)\\ 116.3(4)\\ 123.7(4)\\ 123.7(4)\\ 123.2(4)\\ 116.7(3)\\ 112.3(4)\\ 112.1(4)\\ 115.5(3)\\ 124.4(3)\\ 122.0(4)\\ 118.0(3)\\ 113.9(4)\\ 114.8(5)\\ 115.1(4)\\ 114.8(5)\\ 115.1(4)\\ 124.8(4)\\ \end{array}$	C(5A)-C(6A)-C(7.) C(7A)-C(6A)-C(2.) C(7A)-C(6A)-C(2.) C(6A)-C(7A)-C(8.)-C(9.) C(8A)-C(9A)-C(1.) C(8A)-C(9A)-C(1.) C(9A)-C(10A)-C(1.) C(11A)-C(10A)-C(1.) C(11A)-C(11A)-C(1.) C(12A)-C(13A)-C(1.) C(12A)-C(13A)-C(1.) C(12A)-C(13A)-C(1.) C(12A)-C(13A)-C(1.) C(13A)-C(14A)-C(1.) C(14A)-C(16A)-C(1.) C(16B)-C(1B)-C(1.) C(1B)-C(1B)-C(1.) C(2B)-C(1B)-C(1.) C(2B)-C(1B)-C(1.) C(2B)-C(1B)-C(1.) C(2B)-C(1B)-C(1.) C(2B)-C(2B)-C(2.) C(2B)-C(3B)-C(2.) C(2B)-C(3B)-C(4.) C(3B)-C(4B)-C(5.) C(4B)-C(5B)-C(2.) C(4B)-C(5B)-C(2.) C(4B)-C(5B)-C(2.) C(4B)-C(7.)-C(8.) C(7.)-C(8.)-C(1.) C(7.)-C(8.)-C(1.) C(7.)-C(8.)-C(1.) C(1.)-C(10.)-C(1.) C(1.)-C(1.)-C(1.)-C(1.) C(1.)-C(1.)-C(1.)-C(1.)	A) $121.3(4)$ 4A) $118.6(4)$ A) $111.0(5)$ A) $117.6(5)$ 5A) $119.5(4)$ 0A) $120.5(4)$ $11A$ $120.2(4)$ $(28A)$ $119.7(4)$ $(28A)$ $119.7(4)$ $(21A)$ $115.1(5)$ $(21A)$ $112.3(5)$ $(22A)$ $117.0(4)$ $(21A)$ $123.4(4)$ $(21A)$ $123.4(4)$ $(21A)$ $123.4(4)$ $(21A)$ $123.4(4)$ $(21A)$ $112.9(4)$ $15A)$ $111.4(4)$ $17B)$ $115.6(3)$ 6B) $124.4(3)$ B) $120.8(4)$ OB) $119.2(3)$ B) $113.2(5)$ B) $114.0(5)$ 1B) $116.3(4)$ B) $123.6(4)$ B) $122.9(5)$ B) $117.7(5)$ SB) $120.2(4)$ OB) $119.7(4)$ 11B) $121.6(4)$ $(28B)$ $118.4(4)$	$\begin{array}{c} C(10B) - \\ C(11B) - \\ C(12B) - \\ C(13B) - \\ C(13B) - \\ C(15B) - \\ C(16C) - \\ C(16C) - \\ C(16C) - \\ C(2C) - \\ C(2C) - \\ C(3C) - \\ C(3C) - \\ C(3C) - \\ C(4C) - \\ C(3C) - \\ C(4C) - \\ C(4C) - \\ C(5C) - \\ C(4C) - \\ C(5C) - \\ C(5C) - \\ C(7C) - \\ C(6C) - \\ C(7C) - \\ C(8C) - \\ C(10C) - \\ C(11C) - \\ C(11$	$\begin{array}{c} C(11B)-C(12B) & 11\\ C(12B)-C(13B) & 11\\ C(13B)-C(29B) & 11\\ C(13B)-C(14B) & 12\\ C(14B)-C(15B) & 12\\ C(14B)-C(15B) & 12\\ C(14B)-C(15B) & 11\\ C(15B)-C(16B) & 11\\ C(15B)-C(16B) & 11\\ C(16B)-C(15B) & 11\\ C(1C)-C(17C) & 11\\ C(1C)-C(16C) & 12\\ C(2C)-C(3C) & 12\\ C(2C)-C(3C) & 12\\ C(2C)-C(2C) & 11\\ C(3C)-C(4C) & 11\\ C(4C)-C(5C) & 11\\ C(5C)-C(21C) & 11\\ C(5C)-C(6C) & 12\\ C(5C)-C(2C) & 11\\ C(5C)-C(10C) & 11\\ C(10C)-C(11C) & 12\\ C(10C)-C(13C) & 11\\ -C(11C)-C(13C) & 11\\ -C(13C)-C(14C) & 12\\ -C(14C)-C(15C) & 12\\ -C(14C)-C(15C) & 11\\ C(16C)-C(15C) & 11\\ \end{array}$	3.2(4) 1.7(5) 7.3(4) 2.6(4) 3.5(4) 6.4(3) 2.7(4) 2.4(4) 5.3(3) 4.7(3) 0.9(4) 9.1(3) 2.3(5) 2.3(5) 6.6(4) 3.3(4) 1.8(4) 8.1(5) 5.8(5) 7.8(5) 1.3(4) 8.6(4) 1.0(4) 9.0(4) 3.7(5) 3.2(5) 6.5(4) 3.2(5) 6.5(4) 3.2(5) 6.5(4) 3.4(

domain (SI1: 512W, SW1: 900 Hz, relaxation delay D1: 1),  $F_2$  domain (SI2: 4 K, SW2: 7143 Hz) number of transients per FID, NS: 32, number of preparatory dummy transients per FID, DS: 0.

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