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Crystallographic Comparison of 4-cyano-4′-n-Decycloxybiphenyl and 4-Cyano-4′-n-Dodecycloxybiphenyl

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CRYSTALLOGRAPHIC COMPARISON OF 4-CYANO-4′-n-DECYCLOXYBIPHENYL AND 4-CYANO-4′-n-DODECYCLOXYBIPHENYL

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*The crystal structures of 4-cyano-4′-n-decycloxybiphenyl ($C_{23}H_{29}NO$) (**I**) and 4-cyano-4′-n-dodecycloxybiphenyl ($C_{25}H_{33}NO$) (**II**) have been determined by X-ray diffraction methods.* **I** *and* **II** *crystallize in the monoclinic system with space group C2/c. The final R-factor for I is 0.066 ($F_o > 4\sigma(F_o)$) and 0.085 for all data, $wR(F^2) = 0.189$ for 2444) and it is 0.068 for* **II** *($F_o > 4\sigma(F_o)$) and 0.111 for all data, $wR(F^2) = 0.164$ for 1723). Both phenyl rings are almost planar, and dihedral angle between both rings is 31.2(7)° and 31.0(2)° for* **I** *and* **II**, *respectively.*

INTRODUCTION

Biphenyl and its derivatives have been studied extensively in the past because of the difference found in the inter-ring torsion angles in the solid state [1–3] and in the gas phase [4,5]. This has entailed extended studies of the molecular geometry, crystal packing, and thermal motion effects [6–9]. The present study is part of our ongoing work on the preparation of X-ray diffraction quality single crystals of biphenyls by employing solvent-loss and vapor diffusion techniques [10–17] and investigating their three-dimensional molecular and crystal structures. This study is also investigates the phenomenon of polymorphism in biphenyls [6–9]. X-ray diffraction quality single crystals of both the compounds were obtained

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TABLE 1 Atomic Coordinates and Equivalent Isotropic Thermal Parameters (Å^2) for Nonhydrogen Atoms

Atom	x	y	z	U_{eq}*
		Molecule **I**		
O1	0.1226(1)	0.7251(3)	0.5039(3)	0.063(1)
N1	−0.0058(1)	0.7416(4)	−0.0237(4)	0.077(1)
C1	0.0578(1)	0.7547(3)	0.2017(3)	0.049(1)
C2	0.0514(1)	0.6077(4)	0.0891(4)	0.061(1)
C3	0.0355(1)	0.6036(4)	0.0265(4)	0.063(1)
C4	0.0247(1)	0.7475(3)	0.0748(3)	0.054(1)
C5	0.0310(1)	0.8967(3)	0.1839(3)	0.060(1)
C6	0.0470(1)	0.9002(3)	0.2459(3)	0.053(1)
C7	0.0748(1)	0.7543(3)	0.2783(3)	0.050(1)
C8	0.0878(1)	0.6700(3)	0.1792(3)	0.050(1)
C9	0.1034(1)	0.6648(3)	0.2561(4)	0.056(1)
C10	0.1071(1)	0.7449(3)	0.4371(3)	0.050(1)
C11	0.0946(1)	0.8356(4)	0.5329(4)	0.060(1)
C12	0.0790(1)	0.8384(3)	0.4545(4)	0.054(1)
C13	0.0078(1)	0.7415(4)	0.0173(3)	0.059(1)
C14	0.1265(1)	0.7892(4)	0.6974(4)	0.062(1)
C15	0.1431(1)	0.7190(4)	0.7509(4)	0.062(1)
C16	0.1488(1)	0.7865(4)	0.9521(4)	0.059(1)
C17	0.1656(1)	0.7172(4)	1.0080(4)	0.060(1)
C18	0.1717(1)	0.7821(4)	1.2082(4)	0.062(1)
C19	0.1887(1)	0.7163(4)	1.2626(4)	0.065(1)
C20	0.1950(1)	0.7839(5)	1.4604(5)	0.074(1)
C21	0.2118(10)	0.7228(5)	1.5162(6)	0.082(1)
C22	0.2183(10)	0.7977(9)	1.7081(9)	0.124(3)
C23	0.2350(10)	0.7374(10)	1.7663(9)	0.171(4)
		Molecule **II**		
O1	0.1126(1)	0.2738(3)	0.1092(3)	0.073(1)
N1	−0.0054(1)	0.2584(3)	0.5200(4)	0.087(1)
C1	0.0534(1)	0.2471(3)	0.3497(4)	0.057(1)
C2	0.0473(1)	0.3914(4)	0.4541(4)	0.070(1)
C3	0.0326(1)	0.3977(4)	0.5048(4)	0.072(1)
C4	0.0226(1)	0.2528(3)	0.4465(3)	0.066(1)
C5	0.0284(1)	0.1039(3)	0.3451(4)	0.068(1)
C6	0.0429(1)	0.0988(3)	0.2951(3)	0.060(1)
C7	0.0686(1)	0.2455(3)	0.2924(3)	0.057(1)
C8	0.0805(1)	0.3307(3)	0.3997(4)	0.058(1)
C9	0.0948(1)	0.3353(3)	0.3407(3)	0.066(1)
C10	0.0982(1)	0.2567(3)	0.1613(4)	0.059(1)
C11	0.0867(1)	0.1638(4)	0.0596(4)	0.070(1)
C12	0.0724(1)	0.1623(3)	0.1172(4)	0.062(1)
C13	0.1160(1)	0.2125(4)	−0.0774(4)	0.068(1)

(*Continued*)

TABLE 1 (Continued)

Atom	x	y	z	U_{eq}*
C14	0.1313(1)	0.2790(4)	−0.1179(4)	0.068(1)
C15	0.1363(1)	0.2158(4)	−0.3115(4)	0.068(1)
C16	0.1519(1)	0.2809(3)	−0.3571(4)	0.065(1)
C17	0.1572(1)	0.2159(4)	−0.5487(4)	0.071(1)
C18	0.1728(1)	0.2870(4)	−0.5917(4)	0.069(1)
C19	0.1785(1)	0.2153(4)	−0.7818(5)	0.071(1)
C20	0.1938(1)	0.2851(4)	−0.8265(4)	0.076(1)
C21	0.1995(1)	0.2160(4)	−1.0079(5)	0.080(1)
C22	0.2154(1)	0.2790(5)	−1.0514(6)	0.096(2)
C23	0.2206(1)	0.2048(6)	−1.2346(7)	0.120(2)
C24	0.2364(1)	0.2613(7)	−1.2765(8)	0.160(3)
C25	0.0074(1)	0.2554(3)	0.4862(3)	0.063(1)

*$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* (a_i \cdot a_j)$.
E.s.d's are given in parentheses.

using vapor precipitation technique with dimethylformamide acting as a good solvent system for both the compounds, while cyclohexane and petrol ether were found to be poor solvents for achieving crystallization of **I** and **II** in platey crystals.

EXPERIMENTAL

Three-dimensional intensity data of **I** and **II** were collected on an Enraf-Nonius CAD-4 diffractometer using CuKα radiation ($\lambda = 1.5418$ Å). $\omega/2\theta$ scan mode was employed for data collection. The cell parameters were obtained from a least-squares fit of the setting angle of 25 reflections. The θ range for the data collection is $2.17 < \theta < 67.94°$ in **I** and $6.17 < \theta < 67.97°$ in **II**. Total of 3597 reflections were collected in the case of **I**, from which 3513 were found to be unique ($-95 \le h \le 97$, $0 \le k \le 8$, $-4 \le l \le 8$) and 2444 were considered to be observed ($F_o > 4\sigma(F_o)$). In the case of **II**, the total number of reflections as collected is 3487, of which 3214 were found to be unique ($-105 \le h \le 106$, $-8 \le k \le 8$, $-8 \le l \le 8$) and 1723 were considered to be observed ($F_o > 4\sigma(F_o)$). Reflection data for both the compounds were corrected for Lorentz and polarization effects. Absorption and extinction corrections were not applied.

Both the structures have been solved by using SHELXS97 program [18], and all nonhydrogen atoms were located from the E-map. Full-matrix least-squares refinement of the structures has been carried out by using SHELXL97 program [19]. The positional and thermal parameters of non-hydrogen atoms were refined isotropically. All hydrogen atoms were located on the difference Fourier map, and their positional and isotropic

TABLE 2 Bond Distances (Å) and Bond Angles (°) for Nonhydrogen Atoms

Molecule **I**			
O1–C10	1.342(1)	C8–C9	1.367(1)
O1–C14	1.441(3)	C9–C10	1.401(3)
N1–C13	1.137(1)	C10–C11	1.387(2)
C1–C2	1.405(3)	C11–C12	1.370(1)
C1–C6	1.404(2)	C14–C15	1.482(2)
C1–C7	1.471(1)	C15–C16	1.533(4)
C2–C3	1.356(1)	C16–C17	1.498(2)
C3–C4	1.402(3)	C17–C18	1.530(4)
C4–C5	1.401(3)	C18–C19	1.503(2)
C4–C13	1.425(1)	C19–C20	1.526(4)
C5–C6	1.363(1)	C20–C21	1.480(8)
C7–C8	1.409(2)	C21–C22	1.514(8)
C7–C12	1.393(3)	C22–C23	1.475(11)
C10–O1–C14	118.1(1)	O1–C10–C9	116.4(1)
C6–C1–C7	120.8(1)	C9–C10–C11	118.1(1)
C2–C1–C7	122.2(1)	O1–C10–C11	125.5(1)
C2–C1–C6	116.9(1)	C10–C11–C12	120.1(1)
C1–C2–C3	122.2(1)	C7–C12–C11	123.1(1)
C2–C3–C4	120.5(1)	N1–C13–C4	177.5(1)
C3–C4–C13	121.2(1)	O1–C14–C15	107.7(1)
C3–C4–C5	117.9(1)	C14–C15–C16	112.0(1)
C5–C4–C13	120.9(1)	C15–C16–C17	112.7(1)
C4–C5–C6	121.3(1)	C16–C17–C18	114.1(1)
C1–C6–C5	121.1(1)	C17–C18–C19	114.2(1)
C1–C7–C12	121.6(1)	C18–C19–C20	114.4(1)
C1–C7–C8	122.5(1)	C19–C20–C21	115.4(2)
C8–C7–C12	115.9(1)	C20–C21–C22	115.2(4)
C7–C8–C9	121.7(1)	C21–C22–C23	115.9(6)
C8–C9–C10	121.0(1)		
Molecule **II**			
O1–C10	1.352(2)	C9–C10	1.414(10)
O1–C13	1.415(11)	C10–C11	1.375(4)
N1–C25	1.172(1)	C11–C12	1.351(2)
C1–C2	1.393(6)	C13–C14	1.480(2)
C1–C6	1.450(4)	C14–C15	1.507(11)
C1–C7	1.427(2)	C15–C16	1.510(2)
C2–C3	1.370(2)	C16–C17	1.508(11)
C3–C4	1.411(4)	C17–C18	1.520(2)
C4–C5	1.396(6)	C18–C19	1.528(11)
C4–C25	1.390(1)	C19–C20	1.495(2)
C5–C6	1.351(2)	C20–C21	1.468(10)
C7–C8	1.393(4)	C21–C22	1.527(2)
C7–C12	1.412(10)	C22–C23	1.476(12)
C8–C9	1.355(2)	C23–C24	1.502(12)

(*Continued*)

TABLE 2 (Continued)

Molecule **II**			
C10–O1–C13	118.2(2)	C9–C10–C11	116.9(3)
C6–C1–C7	121.5(1)	O1–C10–C11	126.1(1)
C2–C1–C7	123.6(2)	C10–C11–C12	122.1(1)
C2–C1–C6	114.9(2)	C7–C12–C11	121.8(2)
C1–C2–C3	124.2(2)	O1–C13–C14	108.7(2)
C2–C3–C4	119.5(1)	C13–C14–C15	113.2(2)
C3–C4–C25	122.1(1)	C14–C15–C16	114.5(2)
C3–C4–C5	117.9(2)	C15–C16–C17	115.2(2)
C5–C4–C25	120.0(2)	C16–C17–C18	113.8(2)
C4–C5–C6	122.3(2)	C17–C18–C19	114.1(2)
C1–C6–C5	121.2(1)	C18–C19–C20	114.6(2)
C1–C7–C12	121.4(2)	C19–C20–C21	115.3(2)
C1–C7–C8	122.9(1)	C20–C21–C22	116.0(2)
C8–C7–C12	115.6(3)	C21–C22–C23	113.7(4)
C7–C8–C9	122.7(1)	C22–C23–C24	114.3(7)
C8–C9–C10	120.6(2)	N1–C25–C4	179.7(2)
O1–C10–C9	117.0(2)		

E.s.d.'s are given in parentheses.

temperature factors were refined. Few cycles of refinement with anisotropic thermal parameters for nonhydrogen atoms yielded a final value of R factor = 0.066 for **I** and 0.068 for **II**, respectively. Atomic scattering factors were taken from International Tables for Crystallography (1992, Vol. C Tables 4.2.6.8 and 6.1.1.4).

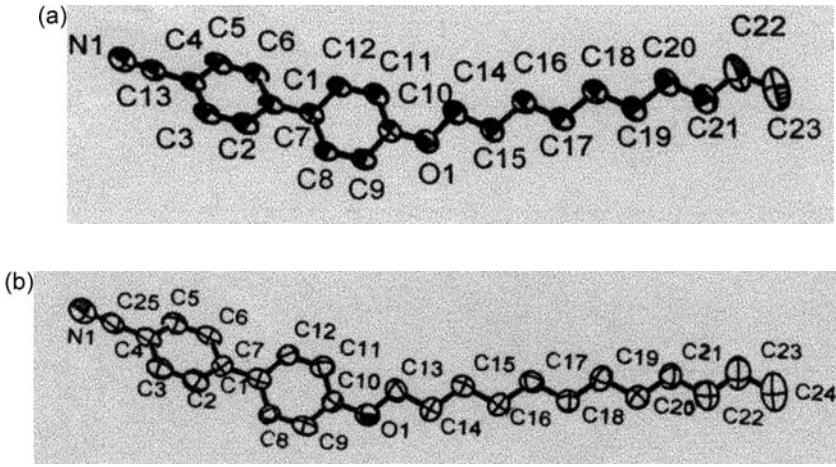

FIGURE 1 (a) A general view of the molecule **I** indicating numbering scheme. (b) A general view of the molecule **II** indicating numbering scheme.

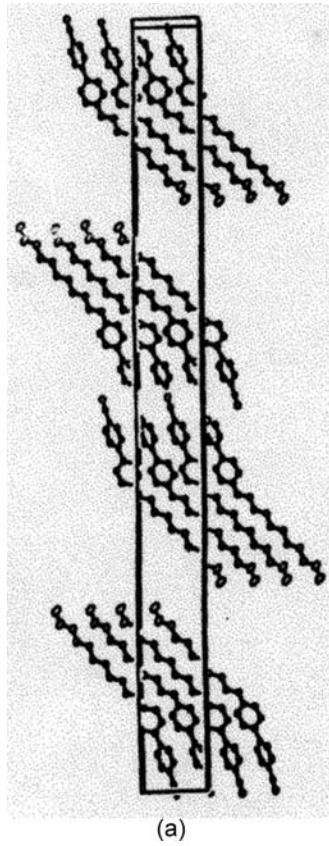

FIGURE 2 (a) Packing view for molecule **I**. (b) Packing view for molecule **II**.

RESULTS AND DISCUSSION

The final atomic positions and equivalent isotropic displacement parameters for all the nonhydrogen atoms are listed in (Table 1). Bond lengths and angles are presented in (Table 2). A general view of the molecules indicating atom numbering scheme (thermal ellipsoid drawn at 50% probability) is shown in Figures 1a and 1b [20].

The geometrical calculations were performed using PARST program [21]. The bond distances and angles agree well with the values reported in the literature for some analogous structures [10–17, 22–27]. The bond length C13 ≡ N1 (1.137(1) Å in **I** and 1.172(1) in **II** is slightly less than the standard value [28]. The four internal ring bond angles at C1 (116.9(1)°), C4 (117.9(1)°), C7 (115.9(1)°), and C10 (118.1(1)°) for **I** are significantly

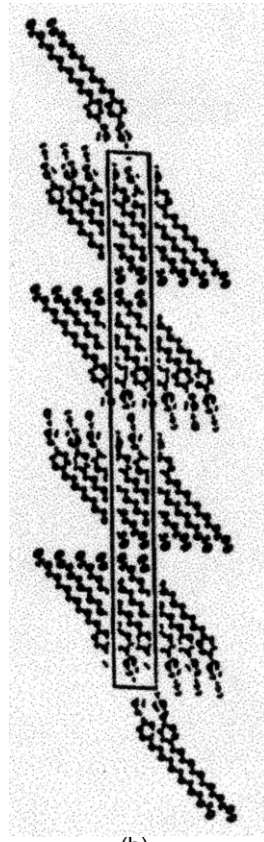

FIGURE 2 (Continued)

smaller than the ideal angles of 120° but slightly greater than the corresponding bond angles obtained in **II**. The length of the bond joining two phenyl rings (C1—C7 = 1.471(1)Å and 1.427(2) Å in **I** and **II**, respectively) is quite close to the standard value for a single bond length between trigonally linked carbon atoms [28]. The bond angles of phenyl rings have normal values. The C13 atom is found slightly out of the plane of the phenyl ring atoms (a deviation of −0.061(2) Å and −0.065(2) for **I** and **II**, respectively). The dihedral angle between the two phenyl rings is 31.2(2)° and 31.0(2)° for molecule **I** and **II**, respectively.

A packing view of the molecule in unit cell for both the structures are shown in Figures 2a and 2b [20].

Since the a-axis is exceptionally large in both the cases as compared to the other two axes, a molecular view down the c-axis has been chosen to show the alignment of the molecules. Molecular layers which form a head-to-head configuration appear to be extending diagonally across the ab-plane, giving rise to a ladder type of molecular packing.

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APPENDIX

Supplementary Data of 4-cyano-4′-n-decyloxybiphenyl [Compound-I] and 4-cyano-4′-n-dodecyloxybiphenyl [Compound-II]

TABLE 1 Fractional Coordinates Equivalent and Isotropic Thermal Parameters ($Å^2$) for Hydrogen Atoms

Atom	x	y	z	U_{iso}
		Compound **I**		
H2	0.062(4)	0.521(5)	0.059(5)	0.099(11)
H3	0.031(4)	0.515(5)	−0.051(5)	0.088(10)
H5	0.023(3)	0.998(4)	0.220(4)	0.067(8)
H6	0.054(3)	1.012(4)	0.321(4)	0.068(8)
H8	0.085(4)	0.619(4)	0.049(5)	0.080(9)
H9	0.113(3)	0.619(4)	0.185(4)	0.058(7)
H11	0.099(4)	0.896(4)	0.644(4)	0.070(8)
H12	0.069(3)	0.881(3)	0.512(3)	0.041(6)
H141	0.126(4)	0.932(5)	0.694(5)	0.100(11)
H142	0.117(4)	0.743(4)	0.786(4)	0.055(7)
H151	0.153(4)	0.745(4)	0.685(4)	0.055(8)
H152	0.143(4)	0.578(5)	0.752(4)	0.079(9)
H161	0.150(4)	0.939(5)	0.959(5)	0.102(11)
H162	0.141(5)	0.743(5)	1.045(6)	0.106(13)
H171	0.174(3)	0.746(3)	0.945(3)	0.033(6)
H172	0.166(4)	0.570(5)	1.009(4)	0.073(9)
H181	0.164(4)	0.742(4)	1.312(5)	0.072(9)
H182	0.171(4)	0.921(5)	1.216(5)	0.088(10)
H191	0.197(4)	0.757(4)	1.172(5)	0.084(11)
H192	0.190(4)	0.579(5)	1.265(5)	0.093(11)
H201	0.186(4)	0.734(4)	1.559(5)	0.074(10)
H202	0.196(4)	0.929(5)	1.459(5)	0.085(10)
H211	0.221(5)	0.759(5)	1.433(6)	0.092(12)
H212	0.212(4)	0.587(6)	1.509(5)	0.094(11)
H221	0.218(6)	0.929(8)	1.695(8)	0.152(21)
H222	0.209(8)	0.760(8)	1.798(9)	0.165(26)
*H23A	0.238(1)	0.792(10)	1.889(9)	
*H23B	0.243(1)	0.778(10)	1.670(9)	
*H23C	0.235(1)	0.604(10)	1.777(9)	
		Compound **II**		
H2	0.054(1)	0.490(4)	0.492(4)	
H3	0.029(1)	0.497(4)	0.577(4)	
H5	0.022(1)	0.005(3)	0.311(4)	
H6	0.046(1)	−0.002(3)	0.224(3)	

(*Continued*)

TABLE 1 (Continued)

Atom	x	y	z	U_{iso}
		Compound **II**		
H8	0.079(1)	0.387(3)	0.517(4)	
H9	0.103(1)	0.391(3)	0.419(3)	
H11	0.089(1)	0.010(4)	−0.052(4)	
H12	0.065(1)	0.105(3)	0.039(4)	
H13A	0.109(1)	0.261(4)	−0.174(4)	
H13B	0.116(1)	0.078(4)	−0.083(4)	
H14A	0.138(1)	0.234(4)	−0.017(4)	
H14B	0.131(1)	0.414(4)	−0.114(4)	
H15A	0.129(1)	0.261(4)	−0.412(4)	
H15B	0.136(1)	0.081(4)	−0.315(4)	
H16A	0.152(1)	0.416(3)	−0.355(4)	
H16B	0.159(1)	0.238(3)	−0.255(4)	
H17A	0.150(1)	0.256(4)	−0.651(4)	
H17B	0.157(1)	0.081(4)	−0.550(4)	
H18A	0.180(1)	0.251(4)	−0.487(4)	
H18B	0.173(1)	0.422(4)	−0.596(4)	
H19A	0.179(1)	0.081(4)	−0.777(5)	
H19B	0.171(1)	0.251(4)	−0.887(5)	
H20A	0.193(1)	0.420(4)	−0.832(4)	
H20B	0.201(1)	0.251(4)	−0.721(4)	
H21A	0.193(1)	0.255(4)	−1.114(5)	
H21B	0.199(1)	0.081(4)	−1.005(5)	
H22A	0.216(1)	0.414(5)	−1.057(6)	
H22B	0.223(1)	0.240(5)	−0.946(6)	
H23A	0.214(10)	0.247(6)	−1.340(7)	
H23B	0.220(10)	0.070(6)	−1.231(7)	
H24A	0.239(10)	0.208(7)	−1.397(8)	
H24B	0.243(10)	0.218(7)	−1.175(8)	
H24C	0.237(10)	0.394(7)	−1.284(8)	

*Atoms fixed stereochemically.
e.s.d.'s are given in parentheses.

TABLE 2 Anisotropic Thermal Parameters ($Å^2$) for Nonhydrogen Atoms

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
			Compound **I**			
O1	0.073(1)	0.060(1)	0.055(1)	−0.007(1)	−0.004(1)	0.003(1)
N1	0.095(2)	0.076(2)	0.059(1)	−0.003(1)	−0.001(1)	0.005(1)
C1	0.078(2)	0.035(1)	0.033(1)	0.001(1)	0.009(1)	−0.001(1)
C2	0.087(2)	0.045(1)	0.051(1)	−0.009(1)	0.002(1)	0.004(1)
C3	0.096(2)	0.049(1)	0.044(1)	−0.012(1)	0.003(1)	−0.001(1)
C4	0.081(2)	0.050(1)	0.029(1)	0.003(1)	0.004(1)	0.001(1)
C5	0.098(2)	0.041(1)	0.040(1)	−0.002(1)	0.005(1)	0.006(1)
C6	0.085(2)	0.034(1)	0.041(1)	−0.001(1)	0.002(1)	0.001(1)
C7	0.078(2)	0.029(1)	0.042(1)	−0.001(1)	0.003(1)	0.000(1)
C8	0.068(2)	0.040(1)	0.043(1)	−0.007(1)	0.005(1)	0.005(1)
C9	0.075(2)	0.042(1)	0.051(1)	−0.005(1)	0.009(1)	0.002(1)
C10	0.067(2)	0.034(1)	0.049(1)	0.003(1)	0.001(1)	−0.004(1)
C11	0.088(2)	0.044(1)	0.049(1)	−0.010(1)	−0.003(1)	0.001(1)
C12	0.077(2)	0.041(1)	0.046(1)	−0.011(1)	0.004(1)	0.007(1)
C13	0.074(2)	0.062(2)	0.041(1)	−0.001(1)	0.005(1)	0.004(1)
C14	0.081(2)	0.050(2)	0.056(1)	−0.004(1)	−0.005(1)	−0.001(1)
C15	0.079(2)	0.049(2)	0.058(2)	−0.006(1)	−0.006(1)	0.003(1)
C16	0.078(2)	0.043(1)	0.056(1)	−0.003(1)	0.001(1)	0.002(1)
C17	0.078(2)	0.051(2)	0.052(1)	−0.006(1)	−0.001(1)	−0.004(1)
C18	0.084(2)	0.047(1)	0.056(1)	−0.002(1)	−0.002(1)	0.001(1)
C19	0.087(2)	0.051(2)	0.056(1)	−0.002(1)	−0.007(1)	−0.001(1)
C20	0.100(3)	0.058(2)	0.063(2)	−0.002(1)	−0.014(2)	0.001(2)
C21	0.100(3)	0.067(2)	0.077(2)	−0.001(2)	−0.017(2)	−0.001(2)
C22	0.154(6)	0.116(4)	0.100(3)	−0.007(3)	−0.055(4)	0.005(4)
C23	0.160(7)	0.213(8)	0.104(5)	0.012(5)	−0.071(4)	−0.003(5)
			Compound **II**			
O1	0.075(2)	0.077(1)	0.0668(1)	−0.003(1)	0.001(1)	−0.006(1)
N1	0.103(2)	0.086(2)	0.0737(2)	−0.002(1)	0.007(2)	−0.005(1)
C1	0.049(2)	0.053(1)	0.0689(1)	−0.001(1)	−0.001(1)	−0.001(1)
C2	0.090(2)	0.056(1)	0.0646(1)	−0.009(1)	0.001(1)	−0.006(1)
C3	0.087(2)	0.063(2)	0.0662(1)	−0.015(1)	−0.001(1)	−0.002(1)
C4	0.093(2)	0.063(2)	0.0410(1)	−0.001(1)	0.001(1)	−0.006(1)
C5	0.085(2)	0.057(1)	0.0596(1)	−0.004(1)	−0.002(1)	−0.005(1)
C6	0.090(2)	0.049(1)	0.0401(1)	−0.001(1)	−0.005(1)	−0.000(1)
C7	0.075(2)	0.040(1)	0.0554(1)	−0.001(1)	−0.003(1)	0.002(1)
C8	0.053(2)	0.055(1)	0.0657(1)	−0.008(1)	0.001(1)	−0.007(1)
C9	0.088(2)	0.053(1)	0.0549(1)	−0.010(1)	−0.001(1)	0.004(1)
C10	0.051(2)	0.061(1)	0.0642(1)	0.001(1)	0.002(1)	0.004(1)
C11	0.070(2)	0.064(2)	0.0734(2)	−0.008(1)	−0.004(1)	−0.004(1)
C12	0.053(2)	0.061(1)	0.0693(1)	−0.100(1)	−0.002(1)	0.001(1)
C13	0.071(2)	0.068(2)	0.0667(1)	−0.009(1)	0.011(1)	−0.001(1)
C14	0.056(2)	0.066(2)	0.0812(2)	0.004(1)	0.008(1)	−0.002(1)

(*Continued*)

TABLE 2 (Continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
			Compound **II**			
C15	0.075(2)	0.066(2)	0.0622(1)	−0.005(1)	0.001(1)	−0.001(1)
C16	0.055(2)	0.063(2)	0.0763(2)	0.002(1)	0.004(1)	−0.001(1)
C17	0.078(2)	0.066(2)	0.0665(1)	−0.015(1)	−0.001(1)	−0.003(1)
C18	0.066(2)	0.071(2)	0.0708(2)	−0.005(1)	0.007(1)	−0.002(1)
C19	0.063(2)	0.063(2)	0.0875(2)	0.001(1)	0.014(2)	−0.005(1)
C20	0.069(2)	0.079(2)	0.0789(2)	−0.003(1)	0.008(2)	−0.009(2)
C21	0.069(3)	0.081(2)	0.0917(2)	−0.012(2)	0.019(2)	−0.001(2)
C22	0.090(3)	0.101(2)	0.1008(2)	−0.001(2)	0.026(2)	0.007(2)
C23	0.118(4)	0.113(3)	0.1341(4)	−0.012(3)	0.046(3)	−0.010(3)
C24	0.151(6)	0.179(6)	0.1574(5)	0.015(4)	0.069(4)	−0.007(4)
C25	0.077(2)	0.068(2)	0.0435(1)	−0.003(1)	0.005(1)	−0.008(1)

e.s.d.'s are given in parentheses.

TABLE 3 Bond Distances (Å) and Bond Angles (°) for Nonhydrogen Atoms

		Compound **I**	
O1–C10	1.342(1)	C8–C9	1.367(1)
O1–C14	1.441(3)	C9–C10	1.401(3)
N1–C13	1.137(1)	C10–C11	1.387(2)
C1–C2	1.405(3)	C11–C12	1.370(1)
C1–C6	1.404(2)	C14–C15	1.482(2)
C1–C7	1.471(1)	C15–C16	1.533(4)
C2–C3	1.356(1)	C16–C17	1.498(2)
C3–C4	1.402(3)	C17–C18	1.530(4)
C4–C5	1.401(3)	C18–C19	1.503(2)
C4–C13	1.425(1)	C19–C20	1.526(4)
C5–C6	1.363(1)	C20–C21	1.480(8)
C7–C8	1.409(2)	C21–C22	1.514(8)
C7–C12	1.393(3)	C22–C23	1.475(11)
C10–O1–C14	118.1(1)	O1–C10–C9	116.4(1)
C6–C1–C7	120.8(1)	C9–C10–C11	118.1(1)
C2–C1–C7	122.2(1)	O1–C10–C11	125.5(1)
C2–C1–C6	116.9(1)	C10–C11–C12	120.1(1)
C1–C2–C3	122.2(1)	C7–C12–C11	123.1(1)
C2–C3–C4	120.5(1)	N1–C13–C4	177.5(1)
C3–C4–C13	121.2(1)	O1–C14–C15	107.7(1)
C3–C4–C5	117.9(1)	C14–C15–C16	112.0(1)
C5–C4–C13	120.9(1)	C15–C16–C17	112.7(1)
C4–C5–C6	121.3(1)	C16–C17–C18	114.1(1)
C1–C6–C5	121.1(1)	C17–C18–C19	114.2(1)
C1–C7–C12	121.6(1)	C18–C19–C20	114.4(1)

(*Continued*)

TABLE 3 (Continued)

Compound **I**			
C1–C7–C8	122.5(1)	C19–C20–C21	115.4(2)
C8–C7–C12	115.9(1)	C20–C21–C22	115.2(4)
C7–C8–C9	121.7(1)	C21–C22–C23	115.9(6)
C8–C9–C10	121.0(1)		
Compound **II**			
O1–C10	1.352(2)	C9–C10	1.414(10)
O1–C13	1.415(11)	C10–C11	1.375(4)
N1–C25	1.172(1)	C11–C12	1.351(2)
C1–C2	1.393(6)	C13–C14	1.480(2)
C1–C6	1.450(4)	C14–C15	1.507(11)
C1–C7	1.427(2)	C15–C16	1.510(2)
C2–C3	1.370(2)	C16–C17	1.508(11)
C3–C4	1.411(4)	C17–C18	1.520(2)
C4–C5	1.396(6)	C18–C19	1.528(11)
C4–C25	1.390(1)	C19–C20	1.495(2)
C5–C6	1.351(2)	C20–C21	1.468(10)
C7–C8	1.393(4)	C21–C22	1.527(2)
C7–C12	1.412(10)	C22–C23	1.476(12)
C8–C9	1.355(2)	C23–C24	1.502(12)
C10–O1–C13	118.2(2)	C9–C10–C11	116.9(3)
C6–C1–C7	121.5(1)	O1–C10–C11	126.1(1)
C2–C1–C7	123.6(2)	C10–C11–C12	122.1(1)
C2–C1–C6	114.9(2)	C7–C12–C11	121.8(2)
C1–C2–C3	124.2(2)	O1–C13–C14	108.7(2)
C2–C3–C4	119.5(1)	C13–C14–C15	113.2(2)
C3–C4–C25	122.1(1)	C14–C15–C16	114.5(2)
C3–C4–C5	117.9(2)	C15–C16–C17	115.2(2)
C5–C4–C25	120.0(2)	C16–C17–C18	113.8(2)
C4–C5–C6	122.3(2)	C17–C18–C19	114.1(2)
C1–C6–C5	121.2(1)	C18–C19–C20	114.6(2)
C1–C7–C12	121.4(2)	C19–C20–C21	115.3(2)
C1–C7–C8	122.9(1)	C20–C21–C22	116.0(2)
C8–C7–C12	115.6(3)	C21–C22–C23	113.7(4)
C7–C8–C9	122.7(1)	C22–C23–C24	114.3(7)
C8–C9–C10	120.6(2)	N1–C25–C4	179.7(2)
O1–C10–C9	117.0(2)		

e.s.d.'s are given in parentheses.

TABLE 4 Torsion Angles (°) for Nonhydrogen Atoms

Compound **I**			
C10–O1–C13–C14	168.5(5)	C1–C7–C8–C9	−177.2(6)
C13–O1–C10–C9	−174.0(5)	C8–C7–C12–C11	0.8(8)
C13–O1–C10–C11	8.7(9)	C12–C7–C8–C9	0.4(8)
C6–C1–C7–C8	−151.7(6)	C7–C8–C9–C10	1.8(9)
C2–C1–C7–C8	30.8(9)	C8–C9–C10–O1	177.3(5)
C6–C1–C7–C12	30.7(8)	C8–C9–C10–C11	−5.1(8)
C2–C1–C7–C12	−146.7(6)	C9–C10–C11–C12	6.4(9)
C7–C1–C6–C5	−178.1(5)	O1–C10–C11–C12	−176.3(6)
C2–C1–C6–C5	−0.4(8)	C10–C11–C12–C7	−4.4(9)
C6–C1–C2–C3	0.2(9)	O1–C13–C14–C15	178.1(5)
C7–C1–C2–C3	177.8(6)	C13–C14–C15–C16	−179.8(5)
C1–C2–C3–C4	−1.4(9)	C14–C15–C16–C17	178.9(5)
C2–C3–C4–C5	2.6(8)	C15–C16–C17–C18	178.7(5)
C2–C3–C4–C25	−177.1(5)	C16–C17–C18–C19	177.7(5)
C3–C4–C25–N1	5.3(11)	C17–C18–C19–C20	179.6(5)
C5–C4–C25–N1	−174.4(11)	C18–C19–C20–C21	179.4(5)
C3–C4–C5–C6	−2.8(9)	C19–C20–C21–C22	−177.4(5)
C25–C4–C5–C6	176.9(5)	C20–C21–C22–C23	179.7(6)
C4–C5–C6–C1	1.8(9)	C21–C22–C23–C24	−178.4(6)
C1–C7–C12–C11	178.5(5)		
Compound **II**			
C10–O1–C14–C15	168.9(2)	C1–C7–C12–C11	177.9(2)
C14–O1–C10–C9	−174.0(2)	C1–C7–C8–C9	−177.4(2)
C14–O1–C10–C11	5.3(3)	C8–C7–C12–C11	−2.2(3)
C6–C1–C7–C8	−150.6(2)	C12–C7–C8–C9	2.7(3)
C2–C1–C7–C8	32.2(3)	C7–C8–C9–C10	−0.5(3)
C6–C1–C7–C12	29.3(3)	C8–C9–C10–O1	177.1(2)
C2–C1–C7–C12	−147.9(2)	C8–C9–C10–C11	−2.3(3)
C7–C1–C6–C5	−176.5(2)	C9–C10–C11–C12	2.7(3)
C2–C1–C6–C5	0.8(3)	O1–C10–C11–C12	−176.5(2)
C6–C1–C2–C3	−0.6(3)	C10–C11–C12–C7	−0.5(3)
C7–C1–C2–C3	176.7(2)	O1–C14–C15–C16	177.5(2)
C1–C2–C3–C4	−0.7(3)	C14–C15–C16–C17	−179.8(2)
C2–C3–C4–C5	1.9(3)	C15–C16–C17–C18	179.9(2)
C2–C3–C4–C13	−177.3(2)	C16–C17–C18–C19	−178.7(2)
C3–C4–C13–N1	163.2(5)	C17–C18–C19–C20	178.8(2)
C5–C4–C13–N1	−15.9(5)	C18–C19–C20–C21	−179.3(3)
C3–C4–C5–C6	−1.7(3)	C19–C20–C21–C22	177.2(4)
C13–C4–C5–C6	177.4(2)	C20–C21–C22–C23	179.7(5)
C4–C5–C6–C1	0.4(3)		

e.s.d.'s are given in parentheses.

TABLE 5 Least-squares Planes and Dihedral Angles (°) Between Various Planes

Compound **I**			
Plane 1. Equation: 0.2656(8)X+0.4781(9)Y−0.8372(6)Z−2.677(6) = 0			
Atom	Deviation (Å)	Atom	Deviation (Å)
C1	−0.007(2)	C5	0.005(2)
C2	0.004(3)	C6	0.003(2)
C3	0.010(3)	C7*	−0.086(2)
C4	−0.010(2)	C13*	−0.061(2)
Plane 2. Equation: 0.2171(11)X+0.8597(5)Y−0.4623(9)−5.103(7) = 0			
Atom	Deviation (Å)	Atom	Deviation (Å)
C7	−0.017(2)	C11	0.015(3)
C8	0.011(2)	C12	0.009(2)
C9	0.007(2)	C1*	−0.069(2)
C10	−0.016(2)	O1*	−0.079(2)
Plane	Plane	Angle (°)	
1	2	31.2(7)	
Compound **II**			
Plane 1. Equation: −0.203(3)X+0.476(3)Y−0.855(2)+2.15(1) = 0			
Atom	Deviation(Å)	Atom	Deviation (Å)
C1	0.003(8)	C5	−0.011(8)
C2	0.001(8)	C6	0.002(8)
C3	−0.009(8)	C7*	0.058(8)
C4	0.014(8)	C25*	0.065(8)
Plane 2. Equation: −0.181(2)X+0.857(1)Y−0.482(2)+0.55(2) = 0			
Atom	Deviation (Å)	Atom	Deviation (Å)
C7	0.010(5)	C11	−0.028(5)
C9	−0.014(5)	C1*	0.077(5)
C10	0.030(5)	O1*	0.073(5)
Plane	Plane	Angle (°)	
1	2	31.0(2)	

e.s.d.'s are given in parentheses.
*atoms not included in the plane calculation.