accuracy of the H positions, but distances and angles obtained with calculated H positions are in Table 4. The acceptors are O atoms of planar molecules with sp^2 hybridization and two free electron pairs lying in the plane of the molecule, so angles between the H, O and the atom to which the O is bonded are given, as well as distances from the H to the acceptor plane. Four of the eight H atoms are involved in hydrogen bonding. One bond is between atoms of the same complex, three others link Ca complexes in a three-dimensional net.

There are probably no hydrogen bonds but only van der Waals contacts between N(3)···O(1) and N(2)···O(2) as the H(3)–O(1) and H(1)–O(2) distances of 2·30 Å are too long.

In the structure, only 50% of the possible hydrogen bonds are formed. This can be explained by the H atoms of urea being all in the plane of the molecule and unable to realize simultaneously the geometrical requirements for hydrogen bonding.

The intensity measurement was carried out in the X-ray Laboratory of SLAFiBS, Krakow.

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Biphenyl: Three-Dimensional Data and New Refinement at 293 K*

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Abstract. $C_{12}H_{10}$, monoclinic, $P2_1/a$, $a = 8{\cdot}12$ (2), $b = 5{\cdot}63$ (1), $c = 9{\cdot}51$ (2) Å, $\beta = 95{\cdot}1$ (3)°, $Z = 2$. X-ray diffraction shows the molecule to be planar, but libration around the long axis of the molecule presents an unusually large value 109 (°)^2, thus suggesting a statistically centrosymmetric arrangement.

Introduction. Previous X-ray analyses of biphenyl at room temperature have been published (Trotter, 1961; Robertson, 1961; Hargreaves & Rizvi, 1962). The present study was undertaken to compare structural and thermal parameters, and particularly librational tensors at 293 and 110 K (Charbonneau & Delugeard, 1976). Colourless thin needles, elongated along the crystallographic *b* axis, were grown from a methanol solution by slow evaporation. Unit-cell dimensions used for the refinement were those of Hargreaves & Rizvi (1962). Measurements were made on an Enraf–Nonius three-circle automatic diffractometer with Zr-filtered Mo $K\alpha$ radiation, and the θ–2θ scanning method (scan range 1·30°). A control reflexion, monitored every 50 reflexions, fluctuated no more than 2%. 697 independent reflexions were used in the structure determination (absorption effects were neglected).

The refinement started with the coordinates given by the study at low temperature, with the *ANHAR* program (Baudour, 1972) based on Kay & Behrendt's (1963) formula; this program includes translational and librational tensors of the assumed rigid-body molecule in the structure factor expression, and takes the internal degree of freedom due to the single bond into account. The weight, w, for each reflexion was $w = 1/\sigma^2(|F_o|)$ with $\sigma^2(|F_o|) = (1/N)(F_o^2/4I^2)[\mathrm{CN} + (B_1/4) + (B_2/4) + (I^2/400)]$. CN is the total number of counts collected during the scan, B_1 and B_2 are the background counts, I is the net intensity and N is the number of cycles of measurements for the reflexion. Five cycles of refinement led to the following final values of the agreement indices: $R_1 = \Sigma\, w||F_o| - |F_c||/\Sigma\, w|F_o| = 0{\cdot}063$; $R_2 = |\Sigma\, w(|F_o| - |F_c|)^2/\Sigma\, w(F_o^2)|^{1/2} = 0{\cdot}068$.

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* Structural Transition in Polyphenyls. V.

Atomic positions are given in Table 1.*

Discussion. The interatomic distances and bond angles are shown in Fig. 1. The aromatic ring C—C distances are in the range 1·399–1·387 Å with standard deviations 0·003–0·004 Å, so that none of the differences is significant. The least-squares mean planes relative to the biphenyl molecule and deviations of atoms from the planes are given in Table 2. The observed deviations of the H atoms (maximum 0·06 Å) from these planes are significant and are possibly due to intermolecular packing. Translational and librational tensors at 110 and 293 K are compared in Table 3. One can see that L_{11}, L_{22}, L_{33} are approximately proportional to the absolute temperature T. However, an interesting feature is the unusually large value of L_{22}, 109·17(°)^2 at 293 K, and 45·70(°)^2 at 110 K. This term corresponds to the libration around the long axis of the molecule. In comparison, the corresponding libration motion of the central ring of *p*-terphenyl (Baudour, Delugeard & Cailleau, 1976) was found to be equal to 260·3(°)^2 at 293 K, and to 19·3(°)^2 at 110 K for the ordered phase.

It is generally believed that the molecule is non-planar in the gas phase, in solution and in the melt (Bastiansen & Trætteberg, 1962; Schmid & Brosa, 1972; Barrett & Steele, 1972; Bree, Pang & Rabeneck, 1971). Evidence of a continuous change in molecular and crystal structure, as the temperature is lowered from 75 to 15 K, is provided by Raman spectroscopy (Friedman, Kopelman & Prasad, 1974). These authors interpret the change as a loss of centre of inversion. Preliminary results from X-ray scattering, at room temperature (Cailleau, 1976), reveal a quasi-plane of diffusion centred on ($\frac{1}{2}$,$\frac{1}{2}$,0).

The hypothesis of a structural change below 75 K would be consistent with a statistically centred arrangement found by X-rays at 293 and 110 K. It might explain the large value of the term L_{22}, the two phenyl rings rotating independently around the single bond in a double-minimum potential.

Table 1. *Final atomic parameters* (×10^5; *for* H ×10^4)

Estimated standard deviations are given in parentheses.

	x	*y*	*z*
C(1)	3549 (26)	−52 (41)	37513 (20)
C(2)	−557 (34)	17360 (49)	17016 (25)
C(3)	5825 (36)	17023 (53)	31082 (27)
C(4)	16602 (35)	−854 (56)	35973 (28)
C(5)	20968 (36)	−18238 (55)	26669 (26)
C(6)	14433 (32)	−17915 (47)	12662 (26)
H(2)	−828 (38)	3037 (52)	1351 (28)
H(3)	237 (40)	2888 (56)	3834 (29)
H(4)	2186 (32)	−115 (48)	4595 (29)
H(5)	2952 (39)	−3052 (57)	3030 (27)
H(6)	1722 (35)	−3049 (49)	682 (27)

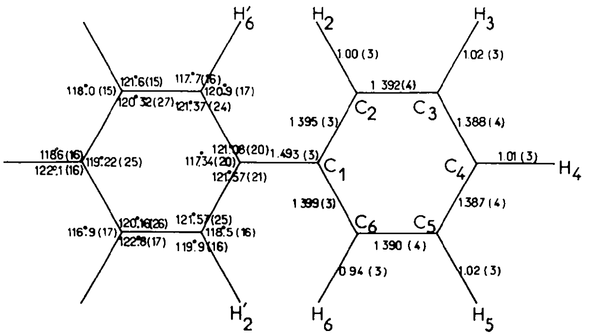

Fig. 1. Bond lengths (Å) and angles (°) of biphenyl. Estimated standard deviations are in parentheses.

Table 2. *Least-squares planes of the molecule with atomic deviations* (Å ×10^4)

The plane constants ($Ax + By + Cz = D$) are referred to crystal axes a, b, c^* and coordinates are in Å.

Plane I: carbon atoms C(1) to C(6)
$0{\cdot}7980X + 0{\cdot}5553Y - 0{\cdot}2341Z = 0{\cdot}0119$

Plane II: 11 atoms of the molecule
$0{\cdot}7918X + 0{\cdot}5612Y - 0{\cdot}2408Z = -0{\cdot}0070$

	I	II		I	II
C(1)	8	119	H(2)	105	359
C(2)	28	180	H(3)	−638	−587
C(3)	−8	27	H(4)	399	209
C(4)	−31	−136	H(5)	699	464
C(5)	51	−79	H(6)	−471	−509
C(6)	−31	−47			

Table 3. *Molecular thermal-motion tensors*

The axes are OX perpendicular to molecular plane, OY in the direction of the long molecular axis, $\mathbf{OZ} = \mathbf{OX} \times \mathbf{OY}$. Standard deviations are given in parentheses.

	Biphenyl 110 K	Biphenyl 293 K
Translation (×10^4 Å^2)		
T_{11}	133 (10)	403 (12)
T_{22}	281 (4)	593 (6)
T_{33}	216 (8)	453 (9)
T_{12}	7 (3)	18 (5)
T_{13}	20 (4)	0 (4)
T_{23}	15 (4)	−12 (5)
Libration, (°)^2		
L_{11}	3·45 (0·40)	11·47 (0·52)
L_{22}	45·70 (2·06)	109·17 (2·82)
L_{33}	2·46 (0·43)	8·39 (0·55)

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32395 (4 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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N-(2-Imidazol-4-ylethyl)phthalimide

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Abstract. Monoclinic, $P2_1/a$, $a = 18{\cdot}434\,(9)$, $b = 7{\cdot}343\,(2)$, $c = 18{\cdot}002\,(7)$ Å, $\beta = 91{\cdot}38\,(4)^\circ$, $C_{13}H_{11}N_3O_2$, $Z = 8$, $D_x = 1{\cdot}315$ g cm^{-3}. The molecules are arranged in infinite NH···N hydrogen-bonded chains with N···N distances of 2·79 and 2·84 Å between imidazolyl groups belonging to symmetry-independent molecules. Parallel stacking of phthalimide groups as well as of alternating phthalimide and imidazolyl groups occurs in the structure.

Introduction. Crystals of $C_{13}H_{11}N_3O_2$ were grown as colorless, fragile plates from an ethyl acetate–petroleum spirit solution. A crystal, approximately 0·2 × 0·2 × 0·05 mm, was used in the X-ray work. Cell parameters and intensity data were measured with a Syntex $P\bar{1}$ diffractometer with graphite-monochromatized Cu $K\alpha$ radiation. 3350 reflections with $\theta < 70^\circ$ were measured. The diffractometer was operated in the θ–2θ variable-speed scan mode, in which the speed of the scan is dependent on a peak-height preview of the reflection intensity. High-intensity reflections were collected at 24° min^{-1} (2θ), low-intensity reflections at 0·5° min^{-1} and medium-intensity reflections at intermediate scan speeds. Scan ranges (2θ) extended from $\alpha_1 - 1^\circ$ to $\alpha_2 + 1^\circ$; stationary background counts were made at the ends of the scans. 1392 reflections with $I > 3\sigma(I)$ were employed in the structure solution and subsequent refinement. Systematic absences of $h0l$ for h odd and of $0k0$ for k odd uniquely identified the space group as $P2_1/a$.

The structure was solved by direct methods with the program *PHASE* (Stewart, 1970), and refined by means of the least-squares program *CLS* (Schilling, 1970). The scattering factors used were those given in *International Tables for X-ray Crystallography* (1968). The weights were taken as $w = [\sigma(F_o) + \alpha|F_o|^2]^{-2}$ where $\sigma(F_o)$ was determined from counting statistics, and α is an empirically adjusted parameter with the optimal value of $6{\cdot}0 \times 10^{-5}$. A secondary extinction parameter, β, was refined and applied to the final structure factor magnitudes by means of the relation (Zachariasen, 1968):

$$|F_c^{\text{corr}}| = |F_c|[1 + \beta|F_c|^2 p_2/(p_1 \sin 2\theta)]^{-0{\cdot}25}$$

where $p_n = (1 + \cos^{2n} 2\theta)/2$. The value of β was determined to be $0{\cdot}377 \times 10^{-5}$. The final R value was 0·046 for the 1392 reflections used in the refinement. The positional parameters of the C, N and O atoms are given in Table 1† and the parameters of the H atoms in Table 2.

Discussion. From kinetic studies of the effect of imidazole on the rate of hydrolysis of *N*-methylphthalimide, Su & Shafer (1969*a*) inferred the existence

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32397 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.