THE STRUCTURE OF 1,2,4,4,6-PENTAPHENYL-1,4-DIHYDROPYRIDINE

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Title compound is triclinic, $M_r = 461.60$; $P\bar{1}$, $a = 9.158(1)$, $b = 16.062(3)$, $c = 19.472(3)$ Å, $\alpha = 110.69(1)°$, $\beta = 89.70(1)°$, $\gamma = 103.17(1)°$, $V = 2\,600(1)$ Å^3, $Z = 4$, $D_o = 1.15(3)$, $D_c = 1.179(1)$ Mg m^{-3}, $\lambda(CuK_\alpha) = 1.5418$ Å, $\mu = 0.509$ mm^{-1}, $F(000) = 976$, $T = 297$ K, $R = 0.040$ for 8 059 unique observed reflections. Both symmetrically independent molecules show a different geometry of the 1,4-dihydropyridine ring: either the boat conformation with apexes $C(sp^3)$, N and boat angles $14.7(3)°$ and $10.3(2)°$ respectively, or the planar conformation. The conformation has been compared with similar dihydropyridines obtained from Cambridge Structural Database.

The crystal structure of 1,2,4,4,6-pentaphenyl-1,4-dihydropyridine (*I*) has been solved by the X-ray diffraction method in connection with a study of materials which show the photochromic behaviour. The colourless crystals turn to violet during the light and X irradiation. Similar properties were observed with previously solved structures of differently substituted 1,4-dihydropyridines and 4*H*-pyrans[1-3]. The molecular structure is supposed to be significant for the explanation of photochromic properties.

EXPERIMENTAL

The crystals were grown from an oxygen free benzene solution[4]. The density was measured by flotation in a KBr aqueous solution. The quality of the crystal (a prism, size 0.2 × 0.3 × 0.3 mm), space group and approximate cell dimensions were established by Weissenberg photographs. The $\Theta-2\Theta$ scan on the SYNTEX P21 diffractometer was used for data collection and a precise cell-parameters determination from 15 centered reflections ($12° < 2\Theta < 30°$). Total 8 828 unique reflections were measured, of which 8 059 with $I > 1.96\sigma(I)$ were treated as observed. Three standard reflections ($\bar{4}00, 050, 004$) showed no significant fluctuations during the measurement. No changes in the diffracted intensities of 15 selected reflections were observed during the colouration of the crystal.

RESULTS AND DISCUSSION

The phase problem was solved by direct methods using the MULTAN 87 program[5] and refined by the SHELX 76 program[6] in the full matrix mode, 854 parameters

being freely refined in four blocks, anisotropically for all non-hydrogen atoms and isotropically for hydrogen atoms, minimizing $\sum w(|F_o| - |F_c|)^2$ with $w = = 1/[\sigma^2(F_o) + 0.0009F_o^2]$. The empirical isotropic extinction factor was refined to $0.796 . 10^{-6}$. No significant correlations among the refined parameters were observed. The refinement was finished when all shifts fell below 0.12 of their e.s.d.'s. The final $R = \sum(|F_o| - |F_c|)/\sum|F_o| = 0.040$, $R_g = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{-1/2}$

TABLE I

Final coordinates (. 10^4) and equivalent isotropic thermal parameters (. $10^4 \mathring{A}^2$) with e.s.d.'s in parentheses for the non-H atoms. $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\ a_i^*\ a_j^*\ a_i a_j$

Atom	x	y	z	U_{eq}	Atom	x	y	z	U_{eq}
C1A	1798(1)	2627(1)	6005(1)	427(4)	C1B	7542(1)	2332(1)	1863(1)	447(4)
C2A	791(1)	1889(1)	6014(1)	465(4)	C2B	7545(2)	1471(1)	1771(1)	480(5)
C3A	828(1)	1470(1)	6599(1)	441(4)	C3B	8297(1)	848(1)	1175(1)	470(4)
C4A	1891(1)	2163(1)	7242(1)	437(5)	C4B	8997(1)	1387(1)	709(1)	472(5)
C5A	2956(1)	2859(1)	7192(1)	415(4)	C5B	9022(1)	2263(1)	832(1)	442(5)
C6A	4063(1)	3480(1)	7819(1)	455(4)	C6B	9805(1)	2721(1)	342(1)	461(4)
C7A	4599(1)	3145(1)	8314(1)	527(5)	C7B	11253(2)	2638(1)	163(1)	581(5)
C8A	5589(2)	3722(1)	8913(1)	683(7)	C8B	11973(2)	3009(1)	-325(1)	706(7)
C9A	6070(2)	4644(2)	9031(1)	797(8)	C9B	11279(2)	3480(1)	-637(1)	714(7)
C10A	5558(2)	4985(2)	8546(1)	791(8)	C10B	9845(2)	3577(1)	-462(1)	728(8)
C11A	4560(2)	4409(1)	7942(1)	603(6)	C11B	9115(2)	3202(1)	24(1)	597(6)
C12A	1601(1)	3054(1)	5457(1)	467(5)	C12B	6635(2)	2832(1)	2433(1)	502(5)
C13A	1624(2)	3985(1)	5687(1)	556(6)	C13B	5557(2)	3214(1)	2252(1)	637(6)
C14A	1332(2)	4364(1)	5177(1)	681(7)	C14B	4629(2)	3604(1)	2778(1)	855(10)
C15A	1013(2)	3832(1)	4447(1)	752(8)	C15B	4771(3)	3613(1)	3476(1)	957(10)
C16A	979(1)	2911(1)	4212(1)	757(8)	C16B	5833(3)	3243(2)	3664(1)	979(11)
C17A	1277(2)	2521(1)	4711(1)	615(7)	C17B	6784(2)	2856(1)	3149(1)	741(7)
C18A	-805(1)	1260(1)	6807(1)	483(5)	C18B	7095(2)	-24(1)	730(1)	501(5)
C19A	-1315(2)	1887(1)	7391(1)	630(6)	C19B	6880(2)	-334(1)	-29(1)	603(5)
C20A	-2820(2)	1733(2)	7528(1)	811(9)	C20B	5798(2)	-1129(1)	-420(1)	716(6)
C21A	-3840(2)	958(2)	7077(1)	829(9)	C21B	4918(2)	-1621(1)	-60(1)	721(7)
C22A	-3346(2)	324(1)	6502(1)	768(9)	C22B	5111(2)	-1319(1)	701(1)	751(7)
C23A	-1833(2)	473(1)	6367(1)	613(6)	C23B	6198(2)	-532(1)	1090(1)	660(7)
C24A	1429(1)	596(1)	6336(1)	435(4)	C24B	9610(2)	602(1)	1499(1)	494(5)
C25A	1271(2)	43(1)	6759(1)	536(5)	C25B	9836(2)	-283(1)	1256(1)	594(6)
C26A	1874(2)	-715(1)	6554(1)	610(7)	C26B	11105(2)	-463(1)	1493(1)	718(7)
C27A	2644(2)	-944(1)	5924(1)	632(6)	C27B	12190(2)	241(1)	1983(1)	779(9)
C28A	2816(2)	-398(1)	5510(1)	632(6)	C28B	11979(2)	1115(2)	2240(1)	820(8)
C29A	2217(2)	371(1)	5714(1)	539(5)	C29B	10696(2)	1293(1)	2000(1)	671(7)
N30A	3042(1)	3087(1)	6547(1)	435(4)	N30B	8270(1)	2792(1)	1407(1)	446(4)
C31A	4505(1)	3165(1)	6249(1)	448(4)	C31B	9084(1)	3743(1)	1791(1)	454(4)
C32A	5087(2)	3867(1)	5998(1)	570(6)	C32B	10423(2)	3942(1)	2196(1)	704(7)
C33A	6471(2)	3907(1)	5700(1)	720(8)	C33B	11203(2)	4844(1)	2558(1)	869(9)
C34A	7286(2)	3270(1)	5668(1)	781(8)	C34B	10630(2)	5551(1)	2518(1)	730(6)
C35A	6717(2)	2587(1)	5932(1)	712(7)	C35B	9294(2)	5352(1)	2115(1)	721(7)
C36A	5322(2)	2523(1)	6219(1)	558(6)	C36B	8514(2)	4450(1)	1749(1)	618(6)

$= 0.072$. Atomic scattering factors were taken from International Tables for X-ray Crystallography[7]. The mlecular geometry was calculated by the PARST program[8].

The final positional parameters and equivalent isotropic thermal parameters for non-hydrogen atoms are listed in Table I, those for hydrogen atoms in Table II. The molecular structure of the title compound with atom numbering is shown in Fig. 1. Symmetrically independent molecules are marked A and B. Bond distances and angles are listed in Table III. Selected torsion angles are listed in Table IV.

The geometry of 1,4-dihydropyridine ring was studied using a set of 36 structures found in Cambridge Structural Database (CSD)[9], release 1989. Only the structures with no disorder and no cyclic routes (i.e. no atom in the dihydropyridine fragment may be connected by a cyclic bond to any atom outside the fragment) were considered. A comparison of mean values of selected parameters of these 36 structures and those determined for I is given in Table V. Both independent molecules are

TABLE II

Final coordinates ($. 10^4$) and isotropic thermal parameters ($. 10^3 \text{Å}^2$) with e.s.d.'s in parentheses for the H atoms

Atom	x	y	z	U_{iso}	Atom	x	y	z	U_{iso}
H2A	-57(17)	1620(9)	5628(8)	49(4)	H2B	6930(17)	1210(10)	2113(8)	55(4)
H4A	1808(15)	2061(9)	7708(8)	45(3)	H4B	9464(18)	1077(10)	273(9)	58(4)
H7A	4171(17)	2443(11)	8226(11)	57(4)	H7B	11838(20)	2307(12)	393(9)	71(5)
H8A	6019(21)	3456(13)	9242(11)	85(6)	H8B	13034(23)	2891(13)	-499(11)	87(6)
H9A	6749(25)	5033(15)	9432(13)	98(6)	H9B	11834(22)	3759(13)	-994(11)	87(6)
H10A	5796(22)	5688(14)	8651(11)	90(6)	H10B	9349(21)	3893(13)	-716(11)	84(6)
H11A	4275(19)	4657(11)	7585(10)	68(5)	H11B	8130(20)	3255(11)	136(9)	62(4)
H13A	1803(17)	4355(10)	6227(9)	55(4)	H13B	5475(21)	3208(12)	1745(11)	77(5)
H14A	1415(21)	5068(14)	5374(10)	83(5)	H14B	3776(24)	3823(14)	2590(11)	94(6)
H15A	882(23)	4093(14)	4090(12)	93(6)	H15B	4197(29)	3896(17)	3913(15)	119(8)
H16A	825(23)	2598(14)	3759(12)	81(6)	H16B	6092(29)	3249(17)	4159(16)	125(9)
H17A	1279(18)	1856(11)	4545(11)	62(4)	H17B	7666(24)	2600(14)	3294(11)	93(6)
H19A	-447(23)	2475(14)	7740(11)	84(5)	H19B	7478(21)	14(12)	-298(10)	76(5)
H20A	-3021(30)	2248(18)	7938(15)	121(8)	H20B	5521(21)	-1339(12)	-966(11)	80(5)
H21A	-5017(23)	835(12)	7162(10)	82(5)	H21B	4146(21)	-2158(13)	-326(10)	79(5)
H22A	-4089(24)	-266(14)	6151(11)	86(6)	H22B	4523(24)	-1673(14)	968(12)	96(6)
H23A	-1380(21)	-29(13)	5922(11)	79(5)	H23B	6372(20)	-287(12)	1660(11)	75(5)
H25A	693(19)	160(11)	7199(10)	66(4)	H25B	9012(20)	-786(12)	915(10)	73(5)
H26A	1788(20)	-1104(13)	6851(10)	78(5)	H26B	11231(24)	-1068(15)	1326(11)	92(6)
H27A	2982(19)	-1502(12)	5755(9)	69(5)	H27B	13222(24)	128(14)	2147(11)	92(6)
H28A	3397(20)	-529(12)	5065(10)	71(5)	H28B	12748(26)	1635(15)	2562(12)	97(7)
H29A	2389(18)	775(11)	5415(9)	65(4)	H29B	10514(22)	1941(14)	2159(11)	89(6)
H32A	4560(22)	4302(13)	6017(10)	80(5)	H32B	10817(23)	3404(14)	2226(11)	91(6)
H33A	6789(23)	4343(14)	5479(11)	87(6)	H33B	12230(25)	4974(14)	2885(12)	94(6)
H34A	8270(28)	3343(15)	5469(13)	111(7)	H34B	11180(21)	6210(14)	2783(11)	84(5)
H35A	7338(25)	2153(15)	5927(12)	98(6)	H35B	8836(22)	5903(14)	2137(11)	89(6)
H36A	4950(20)	2005(12)	6410(10)	72(5)	H36B	7369(22)	4277(13)	1453(10)	85(6)

described separately, due to a significant difference in their conformation. The molecule A shows the boat conformation with the apexes N and C(sp^3), while the molecule B shows an almost planar conformation. It could be explained by the minimal energy difference between the planar and boat conformation in the case of 1,4-di-

TABLE III

Bond distances ($\mathring{A}$) and angles (°) with e.s.d.'s in parentheses

Bond	Bond length		Bond	Bond length	
	A	B		A	B
C1-C2	1.331(2)	1.331(3)	C1-C12	1.489(3)	1.494(2)
C1-N30	1.426(2)	1.418(3)	C2-C3	1.517(3)	1.519(2)
C3-C4	1.512(2)	1.510(3)	C3-C18	1.541(2)	1.541(2)
C3-C24	1.542(2)	1.549(3)	C4-C5	1.338(2)	1.336(2)
C5-C6	1.484(2)	1.488(3)	C5-N30	1.424(3)	1.427(2)
C6-C7	1.396(3)	1.393(2)	C6-C11	1.390(2)	1.394(3)
C7-C8	1.380(2)	1.381(3)	C8-C9	1.380(2)	1.376(2)
C9-C10	1.377(3)	1.384(3)	C10-C11	1.389(2)	1.385(3)
C12-C13	1.397(2)	1.381(3)	C12-C17	1.393(2)	1.387(3)
C13-C14	1.390(3)	1.394(3)	C14-C15	1.366(3)	1.360(3)
C15-C16	1.379(2)	1.361(4)	C16-C17	1.386(3)	1.396(3)
C18-C19	1.387(2)	1.381(3)	C18-C23	1.385(2)	1.387(3)
C19-C20	1.385(3)	1.392(2)	C20-C21	1.378(3)	1.364(3)
C21-C22	1.376(3)	1.384(3)	C22-C23	1.391(3)	1.386(2)
C24-C25	1.395(3)	1.395(2)	C24-C29	1.385(3)	1.383(2)
C25-C26	1.381(3)	1.378(3)	C26-C27	1.388(3)	1.382(2)
C27-C28	1.371(3)	1.373(4)	C28-C29	1.395(3)	1.390(3)
N30-C31	1.454(2)	1.459(2)	C31-C32	1.381(3)	1.371(2)
C31-C36	1.391(3)	1.380(3)	C32-C33	1.389(3)	1.385(2)
C33-C34	1.382(3)	1.381(3)	C34-C35	1.369(3)	1.367(3)
C35-C36	1.388(3)	1.386(2)			

Atoms	Bond angle		Atoms	Bond angle	
C2-C1-N30	122.2(3)	123.4(3)	C1-C2-C3	124.0(3)	125.7(3)
C2-C3-C4	107.9(3)	106.8(3)	C3-C4-C5	123.8(2)	126.0(2)
C4-C5-N30	122.0(3)	122.9(3)	C1-N30-C5	115.9(3)	155.0(3)
C12-C1-N30	116.7(3)	116.9(3)	C6-C11-C10	120.4(3)	120.8(3)
C2-C1-C12	120.9(3)	119.6(3)	C1-C12-C13	120.6(2)	121.4(2)
C2-C3-C24	113.2(2)	112.1(2)	C13-C12-C17	118.6(3)	118.6(3)
C2-C3-C18	105.7(2)	108.5(2)	C4-C5-C6	122.4(2)	120.3(3)
C18-C3-C24	110.9(3)	110.8(3)	C12-C17-C16	120.3(3)	119.9(3)
C4-C3-C24	106.8(2)	106.0(2)	C3-C18-C19	121.2(3)	122.1(3)
C4-C3-C18	112.4(2)	112.6(2)	C19-C18-C23	118.5(3)	117.8(3)
C6-C5-N30	115.5(2)	116.8(3)	C18-C23-C22	120.6(2)	121.1(2)
C5-C6-C7	120.4(3)	119.6(3)	C3-C24-C25	119.6(2)	122.8(2)
C7-C6-C11	118.3(2)	118.1(3)	C25-C24-C29	118.3(3)	117.5(3)
C5-N30-C31	115.4(2)	115.3(3)	C1-N30-C31	114.4(2)	115.4(2)
N30-C31-C36	119.3(3)	120.3(3)	C32-C31-C36	120.0(3)	119.4(3)
C31-C32-C33	119.2(3)	120.4(3)	C31-C36-C35	119.8(3)	120.0(3)

hydropyridines, confirmed by EHT and CNDO/2 calculations[10]. However, the bond distances and angles of both symmetrically independent molecules do not show any significant difference.

TABLE IV

Selected torsion angles (°) with e.s.d.'s in parentheses

Sequence	Angles		Sequence	Angles	
	A	B		A	B
C2-C1-N30-C5	-14.7(4)	-0.0(4)	N30-C1-C2-C3	1.3(5)	-0.5(5)
C1-C2-C3-C4	15.2(4)	-1.1(4)	C2-C3-C4-C5	-20.4(4)	3.8(4)
C3-C4-C5-N30	9.3(5)	-4.9(5)	C4-C5-N30-C1	9.5(4)	2.6(4)
C12-C1-N30-C5	160.5(3)	-175.1(3)	C2-C1-C12-C13	125.8(3)	-123.8(3)
N30-C1-C12-C17	135.4(3)	-133.7(3)	C4-C3-C24-C25	73.2(3)	-107.1(3)
C1-C2-C3-C18	135.6(3)	-122.7(3)	C4-C3-C18-C19	24.6(4)	9.5(4)
C1-C2-C3-C24	-102.8(3)	114.6(3)	C18-C3-C4-C5	-136.6(3)	122.7(3)
C2-C3-C24-C25	-168.2(3)	136.7(3)	C24-C3-C4-C5	101.6(3)	-115.9(3)
C12-C1-C2-C3	-173.7(3)	174.5(3)	C24-C3-C18-C19	144.0(3)	-109.1(3)
C18-C3-C24-C25	-49.5(4)	15.3(4)	C3-C4-C5-C6	-174.7(3)	177.7(3)
N30-C5-C6-C11	32.5(4)	-48.2(4)	C6-C5-N30-C1	-166.8(3)	-179.8(3)
C2-C3-C18-C19	-92.9(3)	127.5(3)	C4-C5-C6-C7	34.4(4)	-47.6(4)
C5-C6-C11-C10	177.8(3)	-176.4(3)	C1-C12-C17-C16	174.9(3)	-173.6(3)
C3-C18-C23-C22	-173.0(3)	-179.9(3)	C3-C24-C29-C28	176.6(3)	-172.7(3)
C1-N30-C31-C32	83.8(4)	73.5(4)	C5-N30-C31-C36	42.1(4)	115.5(3)

TABLE V

Comparison of the geometry of 1,4-dihydropyridines with e.s.d.'s in parentheses

Parameter	A	B	CSD
Average bond length			
$<C(sp^2)-C(sp^2)>$	1.335(5)	1.334(4)	1.35(1)
$<C(sp^2)-C(sp^3)>$	1.515(4)	1.514(6)	1.52(1)
$<C(sp^2)-N>$	1.425(3)	1.423(6)	1.38(1)
Average angles			
$C(sp^2)-C(sp^3)-C(sp^2)$	107.9(2)	106.8(2)	110(1)
$<C(sp^2)-C(sp^2)-C(sp^3)>$	123.9(2)	125.8(2)	121(2)
$<C(sp^2)-C(sp^2)-N>$	122.1(2)	122.9(3)	120(2)
$C(sp^2)-N-C(sp^2)$	115.9(3)	115.0(3)	122(3)
Dihedral angles			
boat angles P_1,P_2 [a]	14.7(3)	2.1(3)	16(7)
boat angles P_1,P_3 [a]	10.3(2)	1.3(2)	9(4)
fold angles	16.7(1)	0.8(1)	16(7)
$\sum_{n=1}^{6}|\tau_n|$	70(2)	13(3)	71(30)

[a] P_1 mean plane C1,C2,C4,C5; P_2 plane C2,C3,C4; P_3 plane C1,O30,C5.

The average bond distance $\langle C(sp^3)\text{-}C(sp^2)\rangle$ of 1,4-dihydropyridine ring for I corresponds to the other structures from CSD, but $\langle C(sp^2)\text{-}C(sp^2)\rangle$ is smaller and $\langle C(sp^2)\text{-}N\rangle$ greater than the corresponding mean values from CSD. This effect was observed in the case of the similarly substituted 1,4-dihydropyridines[2] and attributed to methoxycarbonyl groups at the positions 3,5 and to the 1-phenylsubstituents.

TABLE VI

Dihedral angles between phenyl planes and the plane P_1 ($°$) and bond-plane angles between the phenyl substituents bonds and plane P_1 ($°$) with e.s.d.'s in parentheses

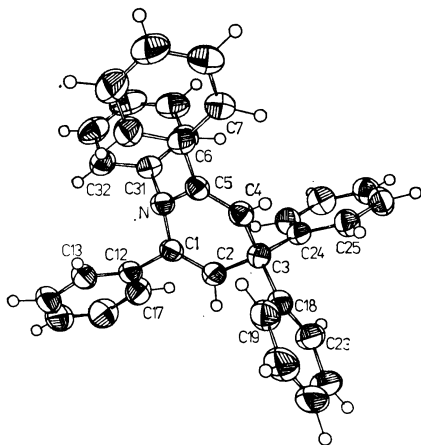| Dihedral angle	A	B	Bond-plane angle	A	B
C31-C32-C33-C34-C35-C36	74.1(2)	86.8(2)	N-C31	47.6(2)	35.9(2)
C6-C7-C8-C9-C10-C11	30.1(2)	47.9(2)	C1-C12	13.1(2)	5.4(2)
C18-C19-C20-C21-C22-C23	71.1(1)	55.9(2)	C3-C18	41.4(2)	51.8(2)
C24-C25-C26-C27-C28-C29	81.7(1)	86.5(2)	C3-C24	68.6(2)	58.5(2)
C12-C13-C14-C15-C16-C17	49.1(1)	52.6(2)	C5-C6	5.4(2)	0.1(2)

FIG. 1
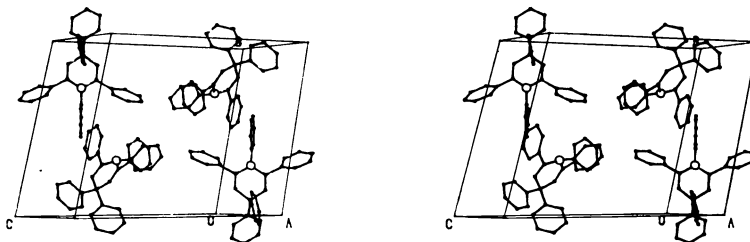
Molecular structure with the atom numbering

FIG. 2

Stereoscopic view of the unit cell (the hydrogen atoms were omitted for clarity)

The average angle on the N atom and the $C(sp^3)$ atom of I are smaller and the other angles are wider than the corresponding values from CSD. Different conformations of 1,4-dihydropyridines are described by dihedral angles between the planes P_1, P_2 and P_1, P_3 (boat angles) and dihedral angles between both $N-C(sp^2)-C(sp^2)-C(sp^3)$ planes (fold angles), or by the sum of absolute values of the six 1,4-dihydropyridine torsion angles $\sum_{n=1}^{6} |\tau_n|$ in Table V.

The orientation of phenyl substituents of the title compound is summarized in Table VI. The χ^2 values for all ten phenyl planes range from 2 to 26. The average lengths $\langle C(sp^2)-C(phenyl)\rangle = 1\cdot489(4)$ Å, $\langle C(sp^3)-C(phenyl)\rangle = 1\cdot543(4)$ Å and $\langle N-C(phenyl)\rangle = 1\cdot456(4)$ Å correspond to typical values within 4 e.s.d.'s. Similar effects were observed at the inner phenyl angles at the site of connection with the rest of the molecule, similarly to the case of previously studied 1,4-dihydropyridines and 4H-pyrans[3].

Fig. 2 is a stereoscopic view of the crystal structure. No intermolecular non--hydrogen atomic distances shorter than 3·4 Å have been found. It seems impossible that photochromic properties could originate in the charge transfer between phenyl substituents, because no tendency to a parallel stacking of phenyl rings of neighbour molecules has been observed.

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