(*Z*)- and (*E*)-Arylidene-1,3-dihydroindol-2-ones: Configuration, Conformation, and Infrared Carbonyl Stretching Frequencies

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The configuration and conformation of both (*Z*)- and (*E*)-arylidene-1,3-dihydroindol-2-ones were investigated. In the solid state the *Z*-isomer is planar whereas the *E*-isomer has the aryl group significantly twisted, as shown by two *X*-ray structures. A linear correlation was found between $\tilde{\nu}_{(C=O)}$ and the σ_p^+ of the substituents and this allows the conformation in solution and the degree of conjugation of the Ar–C=C–C=O system to be inferred.

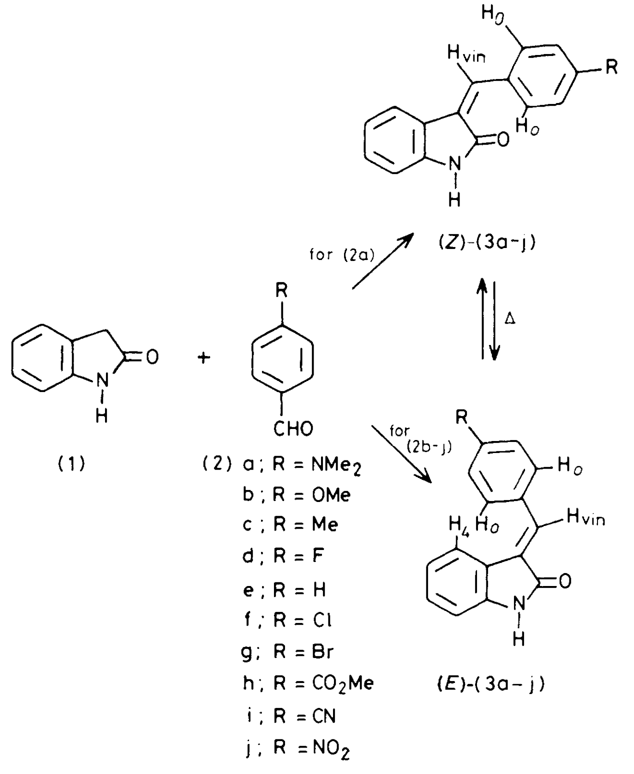
The configuration and conformation of (*Z*)- and (*E*)-1,3-dihydro-2-oxoindol-3-ylidene derivatives are topics that attracted interest some 30 years ago when Abramovitch and Hey [1] suggested the existence of two configurational isomers of benzylidene-1,3-dihydroindol-2-one.

The question of configuration was solved first by Autrey and Tahk [2] and then by other groups [3,4] using n.m.r., as the chemical shifts of both the vinylic and *ortho*-arylidene protons are significantly influenced by configuration.

Although the configuration is now readily determined, the conformation is still unresolved, as it has been suggested that either the *Z*- or the *E*-isomers [5,6] could adopt a twisted conformation.

As a result of our interest in 1,3-dihydro-2-oxoindol-3-ylidene derivatives as substrates in heterodiene reactions,[7] in a previous paper [8] we reported the effect of configuration on the transmission of the electronic effects to the carbonyl group of (*Z*)- and (*E*)-3-alkylidene-1,3-dihydroindol-2-ones.

In this paper we complete the previous investigations by taking into account 3-benzylidene derivatives.



Scheme 1.

Results

3-Arylidene-1,3-dihydroindol-2-ones were obtained by condensation of 1,3-dihydroindol-2-one (1) and aromatic aldehydes (2) in accordance with the literature method,[9] but this route gives *E*-isomers (3b–j) except for the *p*-dimethylamino-derivative which gives (*Z*)-(3a) only. A thermal equilibration of these products was then carried out giving both isomers, which were easily separated in good yields by column chromatography (Scheme 1, see Experimental section for details).

The configuration was assigned to pure (*Z*)- and (*E*)-(3) by n.m.r., taking into account previously recorded chemical-shift values (see Table 2 in the experimental part). Obviously the vinylic proton is more deshielded in *E*-isomers due to the carbonyl influence, whereas *ortho*-benzylidene protons are more deshielded in *Z*-isomers for the same reason.

Having defined the configuration for all the isomers, their conformation was investigated to clarify previously mentioned literature discrepancies.[5,6]

It is known that arylidene derivatives of pyrazolone [10] and isoxazolone [11] are planar in the *Z*-configuration and in the solid state. This was determined by an *X*-ray analysis of their crystal structures. Thus to obtain unequivocal information about the conformation of (*Z*)- and (*E*)-(3), the crystal structures of two isomers were determined and the results are reported in Figure 1.[12]

The *Z*-isomer [Figure 1(*a*)] is planar with strongly divergent bond angles of the O=C–C=C–Ar fragment which allows the aryl group to be planar. The distance between the *ortho*-arylidene hydrogen and the oxygen of the carbonyl group is 2.1 Å, the same value that was found for (*Z*)-benzylidene-pyrazolone [10] and -isoxazolone.[11]

The *E*-isomer [Figure 1(*b*)] has the aryl group significantly twisted (the dihedral angle between C=C–C=O and the aryl system is 49°). The reason for this lies in a strong steric interaction between the *ortho*-benzylidene protons and the 4-H of the 1,3-dihydroindol-2-one, the distance of which was found to be 2.48 Å in the twisted conformation. Some calculations were performed on simulated planar structures and, if the experimental bond lengths and angles found in (*E*)-(3e) were retained, the distance 4-H–*o*-H in a planar structure would be 0.7 Å. Furthermore, if a distance of 2 Å were imposed on these hydrogens in a hypothetically planar structure, the deformation of the angles involved would become highly improbable. Thus if one or two angles are kept in the range 120—130° the third reaches 150—190° and

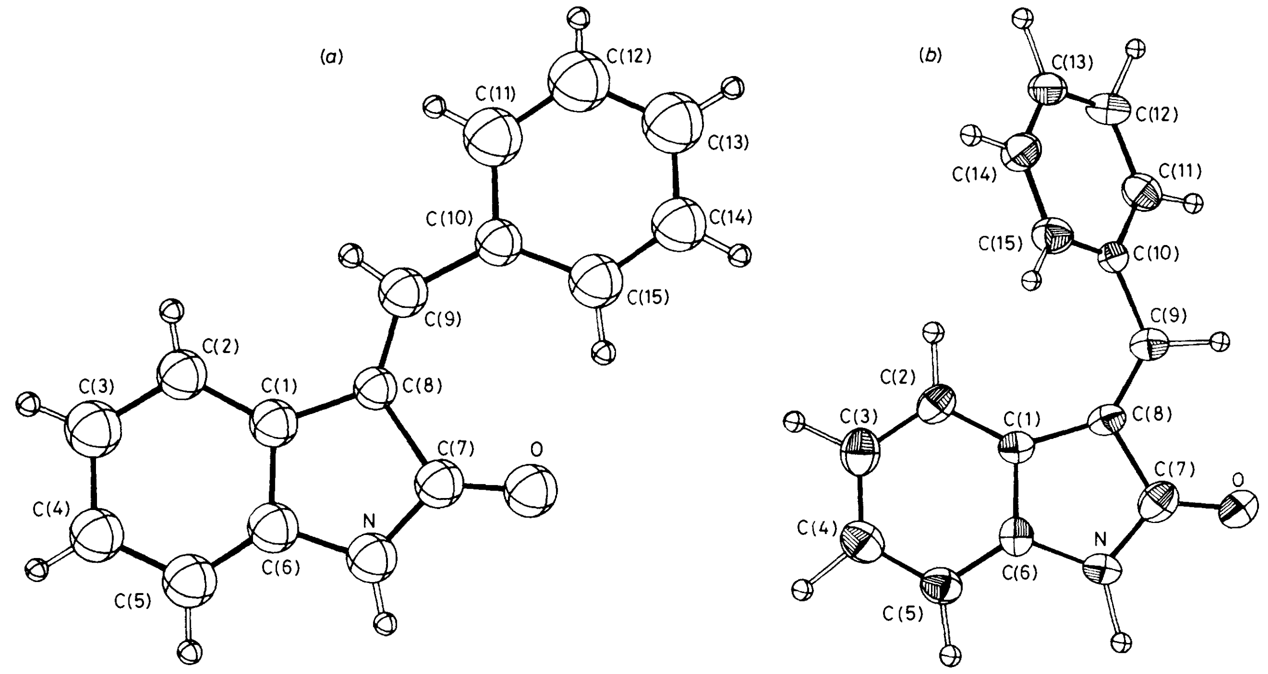


Figure 1. Perspective view of (*Z*)-(3e) and (*E*)-(3e) [(*a*) and (*b*), respectively] in crystals as drawn by ORTEP

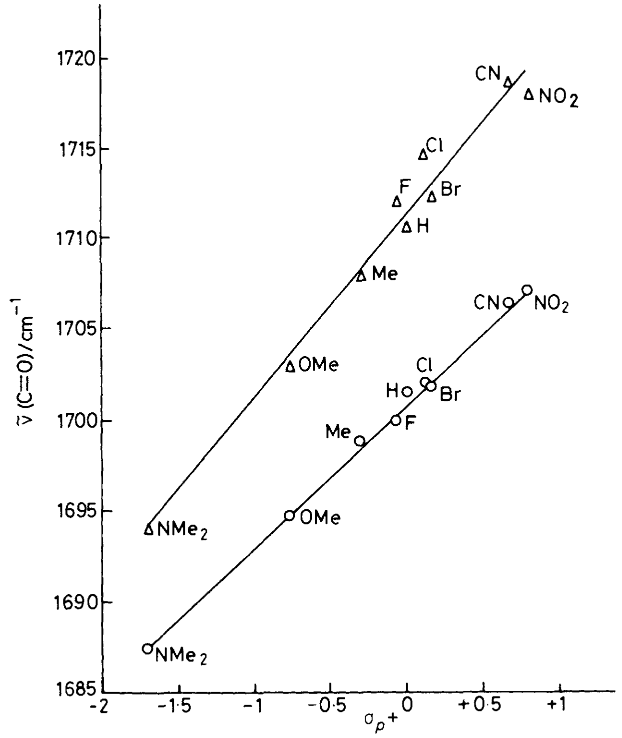


Figure 2. Plot of $\tilde{\nu}_{(C=O)}$ *versus* σ_p^+ for (*Z*)-(3) (○) and (*E*)-(3) (Δ)

if the best compromise is reached C(1)–C(8)–C(9), C(8)–C(9)–C(10), and C(9)–C(10)–C(15) are diverging to 138°. This allows the prediction that not only in the solid state but also in solution a planar conformation as a moment of free rotation of the aryl group is highly unlikely, and the molecule, in an effort to balance a better conjugation and a severe steric interaction, will adopt a twisted conformation as the best compromise.

Table 1. Carbonyl stretching frequencies (cm^{-1}) of (*Z*)- and (*E*)-3-arylidene-1,3-dihydroindol-2-ones (3) in $CHCl_3$ solutions

Compound	(*Z*)-(3)	(*E*)-(3)
a	1 687.4	1 694.3
b	1 694.6	1 703.0
c	1 699.0	1 707.9
d	1 700.1	1 712.0
e	1 701.6	1 710.6
f	1 702.2	1 714.7
g	1 701.9	1 712.4
h[a]	—	—
i	1 706.5	1 718.8
j	1 707.2	1 717.9

[a] Not determined for overlapping of ester band.

Bond lengths, angles, and the most significant dihedral angle of (*Z*)- and (*E*)-(3e) are listed in the Experimental section. Supplementary material on crystal structures and conformational calculations have been deposited as Supplementary Publication No. SUP 23813 (22 pp.).*

In general the more extended the conjugation of an Ar–C=C–C=O system, the lower the stretching frequency of the carbonyl group. Thus, the results reported here were checked in solution using i.r. spectroscopy to see if the transmission of the substituents effect is also possible in the twisted *E*-configuration.

The carbonyl stretching frequencies of all (*Z*)- and (*E*)-3-arylidene-1,3-dihydroindol-2-ones (3) were measured in $CHCl_3$ solutions (for details see the Experimental section) and their values are listed in Table 1.

All isomers (3a—j) readily give intramolecular hydrogen bonds,[13] due to the presence of both C=O and NH groups. Hence both the position of the bands and their shape are

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* For details of the Supplementary Publication Scheme see Instructions for Authors, *J. Chem. Soc., Perkin Trans. 2*, 1984, Issue 1.

Table 2. Analytical and physical data for compounds (*Z*)- and (*E*)-(3a—j)

Compound		Physical aspect and yield (%)[a]	M.p. (°C)	Elemental analysis (%) and lit. m.p. (°C)	N.m.r.[b] Chemical shifts (δ from Me_4Si)	
					$H_{vin.}$	H_o
(3a)	*Z*	Bright red prisms (30)	233—234[c]	194—195;[f,g] 241—242;[f,h] 197[i,j]	7.45	8.39
	E	Deep orange crystals (51)	204—205[d]		7.77	7.70
(3b)	*Z*	Bright orange needles (29)	175—176[c]	157;[f,g] 163;[i,j] 159—161;[i,k] 183—185[i,k]	7.50	8.35
	E	Deep yellow needles (59)	156—157[e]		7.79	7.66
(3c)	*Z*	Soft yellow needles (30)	205—206[e]	Found: C, 81.45; H, 5.49; N, 5.85	7.52	8.22
	E	Yellow crystals (64)	195—196[e]	Found: C, 81.57; H, 5.60; N, 5.88 Calc. for $C_{16}H_{13}NO$: C, 81.68; H, 5.57; N, 5.95	7.82	7.59
(3d)	*Z*	Bronze coloured platelets (36)	205—206[e]	194—196;[f,m] 196—197[i,j]	7.49	8.34
	E	Soft yellow needles (63)	189—190[e]		7.75	7.60
(3e)	*Z*	Deep orange needles (32)	180—181[e]	175—176;[f,g] 176;[f,o] 177—178;[f,p] 181—183;[f,m] 176;[f,n]	7.55	8.28
	E	Bright yellow needles (66)	175—176[e]	165—168;[f,q] 181—183;[i,j] 175—176;[i,k] 171—174;[k,l] 181—183;[i,r] 167—168;[i,r] 176—177;[i,s] 168—169[i,s]	7.84	7.62
(3f)	*Z*	Light orange needles (35)	210—211[c]	184;[f,n] 185—188;[f,t] 188—190;[f,t] 190—191;[i,k]	7.45	8.25
	E	Soft yellow needles (64)	188—189[e]	210—213;[i,k] 183—184[i,j]	7.74	7.53
(3g)	*Z*	Orange needles (33)	232—233[d]	Found: C, 59.90; H, 3.30; N, 4.58; Br, 26.35	7.45	8.14
	E	Soft yellow needles (62)	195—196[c]	Found: C, 60.12; H, 3.42; N, 4.75; Br, 26.45 Calc. for $C_{15}H_{10}BrNO$: C, 60.01; H, 3.35; N, 4.66; Br, 26.62	7.71	7.57
(3h)	*Z*	Soft orange needles (30)	212—213[d]	Found: C, 73.05; H, 4.61; N, 5.10	7.53	8.10
	E	Bright yellow crystals (59)	238—239[d]	Found: C, 72.98; H, 4.70; N, 5.15 Calc. for $C_{17}H_{13}NO_3$: C, 73.11; H, 4.69; N, 5.02	7.81	7.75
(3i)	*Z*	Deep orange crystals (30)	205—206[d]	Found: C, 78.15; H, 4.20; N, 11.25	7.47	8.27
	E	Orange crystals (48)	242—243[d]	Found: C, 78.19; H, 4.15; N, 11.42 Calc. for $C_{16}H_{10}N_2O$: C, 78.03; H, 4.09; N, 11.38	7.74	7.74
(3j)	*Z*	Deep red crystals (25)	252—253[c]	229;[f,n] 224—225;[f,u] 254—255;[i,j] 240—243;[i,k] 212—219[i,k]	7.52	8.29
	E	Brick red crystals (30)	255—256[c]		7.77	7.80

[a] Yields of the crude isomers after chromatographic separation. [b] For $H_{vin.}$ and H_o see Scheme 1, formulae (3a—j). [c] From dioxane. [d] From dioxane–EtOH, 1 : 1. [e] From EtOH. [f] Described with undetermined configuration. [g] Ref. 9. [h] Ref. 25. [i] Described with *E*-configuration. [j] Ref. 26. [k] Ref. 3. [l] Described with *Z*-configuration. [m] Ref. 27. [n] Ref. 28. [o] Ref. 29. [p] Ref. 30. [q] Ref. 31. [r] Ref. 4. [s] Ref. 32. [t] Ref. 33. [u] Ref. 34.

concentration dependent and all the reported values were at high dilution (5×10^{-4}M)[8] when no further modification of the spectrum occured.

All (*Z*)-3-arylidene-1,3-dihydroindol-2-ones absorb at a frequency lower than that of the corresponding *E*-isomers.

Furthermore, all frequencies depend significantly on the *para* substituent in the arylidene group and if $\tilde{\nu}_{(C=O)}$ is plotted *versus* σ_p^+ values of R,[13] good linear relationships are obtained with comparable ρ values (8.03 for the *Z*-series and 10.07 for the *E*) (Figure 2).

Discussion and Conclusions

Some conclusions can be drawn from the above reported results.

The carbonyl stretching frequency, lower in the *Z*-series than in the *E*, could be interpreted as the result of the better coplanarity of the *Z*-isomers, which allows a more extended conjugation. This effect certainly operates, but results for *all* five-membered ring derivatives found in the literature give the *trans* (CO) isomer * at a higher frequency than the *cis*: *e.g.*, 1-[15,16] and 2-indanones,[17] 1,2-dihydroindol-3-ones,[18] aurones,[19] and pyrazolones.[20]

In general this is certainly the result of a more extended conjugation. However, this is not the case for aurone (2-arylidene-2*H*-benzo[*b*]furan-3-ones) whose *trans* (CO) isomer (the *Z*-isomer) was found to have a fully planar crystal structure.[21]

Therefore, even if it is significant, the carbonyl stretching frequency cannot be recommended as a basis for defining the degree of planarity of an α,β-unsaturated carbonyl system.

Since $\tilde{\nu}_{(C=O)}$ in both the *Z*- and the *E*-series correlates with σ_p^+, the twisted conformation of the *E*-series does not prevent

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* The presence of heteroatoms in the five-membered ring makes the *trans* (CO) isomer [the *E*-isomer of (3)] sometimes the *E*, and sometimes the *Z*.

Table 3. Bond lengths (Å) and angles (°) of (*Z*)-(3e); e.s.d. in parentheses

(*a*) Bond length

C(1)–C(2)	1.393(11)	C(7)–C(8)	1.547(10)
C(1)–C(6)	1.399(10)	C(8)–C(9)	1.350(9)
C(1)–C(8)	1.439(10)	C(9)–C(10)	1.440(10)
C(2)–C(3)	1.386(11)	C(10)–C(11)	1.423(12)
C(3)–C(4)	1.430(11)	C(10)–C(15)	1.402(11)
C(4)–C(5)	1.378(11)	C(11)–C(12)	1.385(12)
C(5)–C(6)	1.332(10)	C(12)–C(13)	1.378(13)
C(6)–N	1.376(10)	C(13)–C(14)	1.378(12)
N–C(7)	1.351(9)	C(14)–C(15)	1.349(10)
C(7)–O	1.224(9)		

(*b*) Angle

C(2)–C(1)–C(6)	117.5(8)	C(7)–C(8)–C(1)	103.9(7)
C(2)–C(1)–C(8)	132.7(8)	C(7)–C(8)–C(9)	130.8(8)
C(6)–C(1)–C(8)	109.8(8)	C(1)–C(8)–C(9)	125.2(8)
C(1)–C(2)–C(3)	120.4(9)	C(8)–C(9)–C(10)	133.9(8)
C(1)–C(2)–H(2)	126.2(9)	C(8)–C(9)–H(9)	109.2(8)
C(3)–C(2)–H(2)	113.4(1.0)	C(10)–C(9)–H(9)	116.8(9)
C(2)–C(3)–C(4)	119.9(9)	C(9)–C(10)–C(11)	115.1(9)
C(2)–C(3)–H(3)	126.5(1.0)	C(9)–C(10)–C(15)	129.5(9)
C(4)–C(3)–H(3)	113.5(9)	C(11)–C(10)–C(15)	115.3(8)
C(3)–C(4)–C(5)	118.1(9)	C(10)–C(11)–C(12)	120.1(1.0)
C(3)–C(4)–H(4)	119.0(1.0)	C(10)–C(11)–H(11)	121.4(1.0)
C(5)–C(4)–H(4)	122.1(1.0)	C(12)–C(11)–H(11)	118.5(1.2)
C(4)–C(5)–C(6)	120.7(1.0)	C(11)–C(12)–C(13)	122.4(1.1)
C(4)–C(5)–H(5)	116.2(9)	C(11)–C(12)–H(12)	123.5(1.3)
C(6)–C(5)–H(5)	122.4(1.0)	C(13)–C(12)–H(12)	113.7(1.1)
C(5)–C(6)–C(1)	123.1(1.0)	C(12)–C(13)–C(14)	117.2(1.0)
C(1)–C(6)–N	107.1(8)	C(12)–C(13)–H(13)	125.1(1.2)
C(5)–C(6)–N	129.6(1.0)	C(14)–C(13)–H(13)	117.7(1.2)
C(6)–N–C(7)	114.6(8)	C(13)–C(14)–C(15)	121.8(1.0)
C(6)–N–H	119.4(8)	C(13)–C(14)–H(14)	120.4(1.0)
C(7)–N–H	126.0(8)	C(15)–C(14)–H(14)	117.8(1.1)
N–C(7)–C(8)	104.3(8)	C(14)–C(15)–C(10)	123.0(1.0)
N–C(7)–O	128.5(9)	C(14)–C(15)–H(15)	119.1(1.0)
C(8)–C(7)–O	127.0(8)	C(10)–C(15)–H(15)	117.9(8)

Dihedral angle between phenyl and 1,3-dihydro-2-oxoindol-3-ylidene planes is 8.0(9)°.

Table 4. Bond lengths (Å) and angles (°) of (*E*)-(3e); e.s.d. in parentheses

(*a*) Bond length

C(1)–C(2)	1.397(5)	C(7)–O	1.244(5)
C(1)–C(6)	1.404(6)	C(8)–C(9)	1.337(6)
C(1)–C(8)	1.465(5)	C(9)–C(10)	1.488(5)
C(2)–C(3)	1.396(6)	C(10)–C(11)	1.386(5)
C(3)–C(4)	1.400(6)	C(10)–C(15)	1.387(6)
C(4)–C(5)	1.395(6)	C(11)–C(12)	1.394(6)
C(5)–C(6)	1.382(6)	C(12)–C(13)	1.396(6)
C(6)–N	1.413(5)	C(13)–C(14)	1.381(6)
N–C(7)	1.358(5)	C(14)–C(15)	1.385(6)
C(7)–C(8)	1.499(6)		

(*b*) Angle

C(2)–C(1)–C(6)	118.2(4)	C(8)–C(9)–C(10)	128.0(4)
C(2)–C(1)–C(8)	134.6(4)	C(8)–C(9)–H(9)	121.9(4)
C(6)–C(1)–C(8)	107.2(4)	C(10)–C(9)–H(9)	110.1(3)
C(1)–C(2)–C(3)	118.9(4)	C(9)–C(10)–C(11)	119.7(4)
C(1)–C(2)–H(2)	120.9(4)	C(9)–C(10)–C(15)	121.2(4)
C(3)–C(2)–H(2)	119.5(4)	C(11)–C(10)–C(15)	119.0(4)
C(2)–C(3)–C(4)	121.5(4)	C(10)–C(11)–C(12)	121.2(4)
C(2)–C(3)–H(3)	122.5(4)	C(10)–C(11)–H(11)	123.3(4)
C(4)–C(3)–H(3)	116.0(4)	C(12)–C(11)–H(11)	115.5(4)
C(3)–C(4)–C(5)	120.3(4)	C(11)–C(12)–C(13)	118.7(4)
C(3)–C(4)–H(4)	128.1(4)	C(11)–C(12)–H(12)	128.0(4)
C(5)–C(4)–H(4)	111.4(4)	C(13)–C(12)–H(12)	112.3(4)
C(4)–C(5)–C(6)	117.2(4)	C(12)–C(13)–C(14)	120.5(4)
C(4)–C(5)–H(5)	126.1(4)	C(12)–C(13)–H(13)	114.7(4)
C(6)–C(5)–H(5)	116.6(4)	C(14)–C(13)–H(13)	124.8(4)
C(1)–C(6)–C(5)	123.8(4)	C(13)–C(14)–C(15)	119.9(4)
C(1)–C(6)–N	110.0(4)	C(13)–C(14)–H(14)	117.2(4)
C(5)–C(6)–N	126.2(4)	C(15)–C(14)–H(14)	122.6(4)
C(8)–C(7)–N	108.6(3)	C(10)–C(15)–C(14)	120.7(4)
C(8)–C(7)–O	126.5(4)	C(10)–C(15)–H(15)	121.0(4)
N–C(7)–O	124.9(4)	C(6)–N–C(7)	109.5(3)
C(1)–C(8)–C(7)	104.7(3)	C(6)–N–H	124.2(3)
C(1)–C(8)–C(9)	135.8(4)	C(7)–N–H	125.9(3)
C(7)–C(8)–C(9)	119.4(4)	C(14)–C(15)–H(15)	118.2(4)

Dihedral angle between phenyl and 1,3-dihydro-2-oxoindol-3-ylidene planes is 49.7(4)°.

the transmission of the electronic effects from R to the carbonyl group by resonance. This means that the residual degree of planarity of (*E*)-(3) is enough to allow conjugation. The result is not unexpected, taking into account that conjugation is still possible even if the system deviates considerably from planarity.[16,22–24]

The $\tilde{\nu}_{(C=O)}$ frequencies of the *Z*-series are lower than those of the *E*, but the ρ value is greater in the latter (10.07) than in the former (8.03). Since the ρ value, even in i.r. spectroscopy, is considered to be a function of the degree of transmission of the electronic effects, this fact would contrast with all previous considerations.

Several factors certainly determine the ρ value. Among them, the field effect between C=C and C=O in *cisoid* and *transoid* open-chain C=C–C=O systems was found to be significant.[23] The different field effects that operate between the aryl and the carbonyl groups in *Z*- and *E*-series could give rise to two ρ values which do not differ significantly.

In conclusion, this investigation on 3-arylidene, and the previous one on (*Z*)- and (*E*)-3-alkylidene,[3] illustrate the ground state of the 1,3-dihydro-2-oxoindol-3-ylidene derivatives, the steric and electronic effects that determine both configuration and conformation, and finally clarify the discrepancies in the literature.

Experimental

All m.p.s are uncorrected. ^1H N.m.r. spectra were obtained with a Bruker WP80SY spectrometer equipped with an ASPECT 2000 computer: solutions were prepared using $CDCl_3$ as solvent (10^{-3}M), Me_4Si was used as internal standard. Microanalyses were performed by Dr. Lucia Maggi Dacrema.

3-Arylidene-1,3-dihydroindol-2-ones (3).—Prepared according to the literature method.[9] Equimolecular amounts (0.01 mol) of 1,3-dihydroindol-2-one (1) and of the appropriate aldehyde (2a—j) in MeOH [20 ml; 40 ml for (2h)] and piperidine (0.2 ml) were heated under reflux for 5 h. Then, for (2c—f) and (2 h) the solvent was partially evaporated. After cooling, the coloured solid residue was filtered off and washed with a large amount of water (yields 84—92%). The crude adducts (3b—j) have the *E*-configuration, only (3a) has the *Z*-configuration.

Thermal Equilibration and Chromatographic Separation of (Z)- *and* (E)-(3).—The above reported crude products (3.5 mmol) were heated at 300—310 °C for 5 min, with occasional stirring, in a bath of fusible metal alloy (Wood's alloy). The coloured solids were dissolved in the minimum amount of AcOEt at room temperature and chromatographed (column: 120 cm length and 5.0 cm diameter; Kieselgel, Merck, 0.060—0.200; cyclohexane–ethyl acetate 1 : 1 as eluant). Pure (*Z*)- and (*E*)-(3) were obtained as the first and second fractions, respectively. Yields and physical characteristics of the isomers are reported in Table 2.

I.r. Frequencies.—The i.r. frequencies were determined using a Perkin-Elmer 983 spectrophotometer in the expanded scales (scan conditions: scan rates 12.5 cm^{-1} min^{-1}; noise filter 4; slit 0.092 mm; extra smooth 1). Calibration was performed using bands at 1 801.6 and 1 601.4 cm^{-1} of a polystyrene film. The spectra were taken on freshly prepared solutions (0.0005M) of (3) in spectral grade chloroform at room temperature in an NaCl cell of path length 5.00 mm. The values reported in Table 1 are the average of five different scans and the maximum deviation observed was in the range ± 0.5 cm^{-1}. Different scans on different samples always gave reproducible results.

X-Ray Analysis.—*Crystal data.* [(*Z*)-(3e)]: $C_{15}H_{11}NO$, orange plates, monoclinic, space group $I2/c$, $Z = 8$, $a = 22.973(4)$, $b = 5.915(1)$, $c = 17.517(3)$ Å, $\beta = 105.53(2)°$. The sample dimensions were 0.30 × 0.30 × 0.05 mm.

[(*E*)-3e]: $C_{15}H_{11}NO$, yellow needles, monoclinic, space group $P2_1/n$, $Z = 4$, $a = 12.294(2)$, $b = 22.232(4)$, $c = 4.100(1)$ Å, $\beta = 95.41(1)°$. The sample dimensions were 0.12 × 0.18 × 0.60 mm.

The intensities were recorded with a Philips PW1100 four-circle diffractometer using monochromatized Cu-K_α radiation, $\lambda = 1.5418$ Å, [(*Z*)-(3e)] 1 172, [(*E*)-(3e)] 1 403 independent reflections were collected in the range [(*Z*)-(3e)] $3 \leqslant 2\theta \leqslant 50°$, [(*E*)-(3e)] $3 \leqslant 2\theta \leqslant 55°$, but only [(*Z*)-(3e)] 426, [(*E*)-(3e)] 761 with $|F_0| \geqslant 3\sigma(F_0)$ were processed. The structures were solved by direct methods (MULTAN 78).[35] The co-ordinates of the hydrogen atoms were calculated on the basis of geometrical considerations and checked on a difference-Fourier map. All their calculated co-ordinates were inserted but not allowed to vary in the least-squares refinement carried out [(*Z*)-(3e)] isotropically, [(*E*)-(3e)] anisotropically on all non-hydrogen atoms. The final *R* for all reflections was [(*Z*)-(3e)] 8.3%, [(*E*)-(3e)] 5.2%. The scattering factors were those listed in the International Tables for *X*-ray crystallography (1974).[36]

The bond lengths and angles are given in Tables 3 and 4.

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