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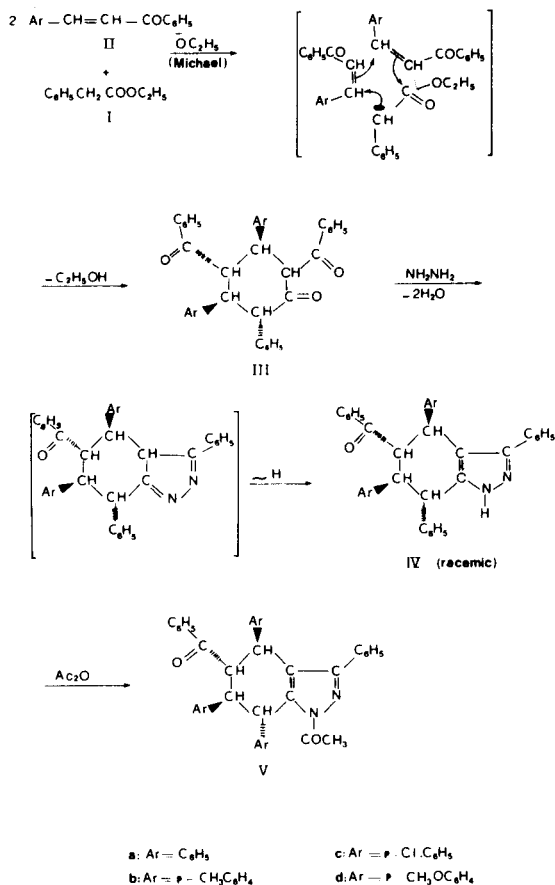
Reactions of 3-aryl-1-phenyl-2-propen-1-ones IIa-d with ethyl phenylacetate (I) in the presence of sodium ethoxide under reflux for one hour gave only the corresponding 3,5-diaryl-2,4-dibenzoyl-6-phenyl-cyclohexanones IIIa-d. The reaction of these compounds with hydrazine hydrate afforded the corresponding 5-benzoyl-4,6-diaryl-3,7-diphenyl-1,4,5,6,7-pentahydro-1*H*-indazole IVa-d. The structures of the products were established by spectroscopic (ir, uv, nmr and mass spectra), single-crystal X-ray analysis and chemical evidence.

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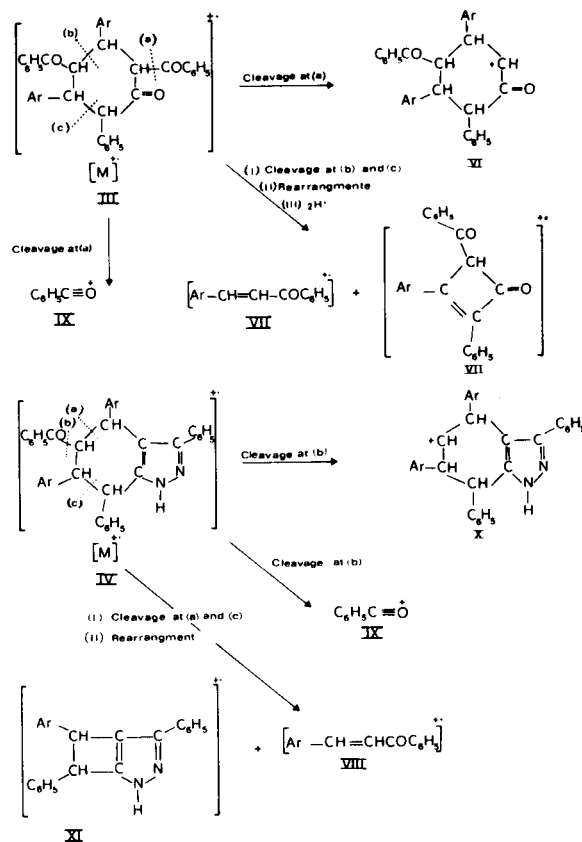
The reaction of 3-aryl-1-phenyl-2-propen-1-ones IIa-d with ethyl phenylacetate (I) under reflux for one hour have been reported previously to give the corresponding 4-aryl-3,6-diphenyl-3,4-dihydro-2*H*-pyran-2-ones (2). The present investigation was intended to study the reaction between two moles of 3-aryl-1-phenyl-2-propen-1-ones IIa-d and one mole of ethyl phenylacetate (I) in the presence of two moles of sodium ethoxide and to throw further light on the

reaction mechanism as well as to establish the structure and configuration of the products.

When two moles of 3-phenyl- (IIa), 3-*p*-tolyl- (IIb), 3-*p*-chlorophenyl- (IIc) and 3-*p*-methoxyphenyl-1-phenyl-2-propen-1-ones (II d) were refluxed for one hour with the sodium salt of ethyl phenylacetate (prepared from one mole of the ester and two moles of sodium ethoxide) in dry



Scheme 1



Scheme 2

Table 1

Infrared, Ultraviolet and Nuclear Magnetic Resonance Spectral Data for 3,5-Diaryl-2,4-dibenzoyl-6-phenylcyclohexanones (IIIa-d), 5-Benzoyl-4,6-diaryl-3,7-diphenyl-1,4,5,6,7-pentahydro-1*H*-indazoles (IVa-d) and the *N*-Acetyl Derivative (Va)

Compound No.	Infrared Spectra (Nujol)		Ultraviolet Spectra (Ethanol)		Nmr Spectra (Deuteriochloroform)	
	cm ⁻¹	ν	λ max (nm)	ϵ	δ	Assignment (No. of Protons)
IIIa	1725 (s)	C=O	284	12,920	8.02-6.58 (m)	(25) ArH
	1690 (s)		248	28,990	5.43-4.27 (m)	(5) >CH
	1660 (s)					
IIIb	1725 (s)	C=O	248	24,270	7.92-6.48 (m)	(23) ArH
	1690 (s)				5.45-3.98 (m)	(5) >CH
	1660 (s)				2.03 (s)	(3) Ar-CH ₃
					2.00 (s)	(3) Ar-CH ₃
IIIc	1725 (s)	C=O	247	26,630	8.0-6.63 (m)	(23) ArH
	1690 (s)		220	28,680	5.43-3.93 (m)	(5) >CH
	1665 (s)					
III d	1715 (s)	C=O	246	23,700	8.16-6.22 (m)	(23) ArH
	1690 (s)				5.48-4.15 (m)	(5) >CH
	1670 (s)				3.58 (s)	(3) Ar-OCH ₃
IVa	3280 (br)	NH	248	21,860	7.55-6.55 (m)	(26) ArH + NH
	1670 (s)	C=O			5.22-3.38 (m)	(4) >CH
	1660 (m)	C=C and/or C=N				
IVb	3240 (br)	NH	248	23,340	7.43-6.43 (m)	(24) ArH + NH
	1670 (s)	C=O			5.03-3.33 (m)	(4) >CH
	1665 (sh)	C=C and/or C=N			2.00 (s)	(6) 2 ArCH ₃
IVc	3400 (s)	NH	249	25,160	7.85 (br)	(1) NH
	3200 (br)				7.55-6.38 (m)	(23) ArH
	1670 (s)	C=O			5.22-3.88 (m)	(4) >CH
	1665 (sh)	C=C and/or C=N				
IVd	3260 (b)	NH	249	25,110	7.60 (br)	(1) NH
	1670 (s)	C=O			7.42-6.08 (m)	(23) ArH
	1660 (sh)	C=C and/or C=N			5.12-3.95 (m)	(4) >CH
					3.52 (s)	(6) 2ArOCH ₃
Va	1730 (s)	NCOCH ₃	249	21,860	7.60-6.36 (m)	(25) ArH
	1670 (s)	C=O			5.01-3.23 (m)	(4) >CH
		(benzoyl group)			2.40 (s)	(3) COCH ₃

benzene, they gave the corresponding 5-benzoyl-4,6-diaryl-3,7-diphenyl-1,4,5,6,7-pentahydro-1*H*-cyclohexanones IIIa-d. The reaction seems to proceed by Michael addition between the acetate I and the α,β -unsaturated ketones II with subsequent cyclization to give the cyclohexanone derivative III (*cf.*, Scheme 1).

The structures of these compounds were established by both spectroscopic and chemical evidence. Thus, the infrared spectra of the cyclohexanones IIIa-d (Table I) show

three strong bands in the 1725-1715 cm⁻¹, 1690 cm⁻¹, and 1680-1660 cm⁻¹ regions which are attributable to the three carbonyl stretching frequencies of the cyclohexanone and the two benzoyl groups, respectively (3). The nmr spectra of these cyclohexanones (*cf.*, Table I) show signals correlated to the methine protons in addition to the aromatic protons. Compounds IIIb and III d also show two signals attributable to two methyl and to two methoxy protons, respectively, present in different environments (Table I).

Table II

Relative Intensity of Major Ions in the Mass Spectra for 3,5-Diaryl-2,4-dibenzoyl-6-phenylcyclohexanones (IIIa-d) and the corresponding 1*H*-Indazole Derivatives (IVa-d)

Compound No.	m/e	Relative Intensity (%)	Compound No.	m/e	Relative Intensity (%)	Compound No.	m/e (Cl ³⁵ , Cl ³⁷)	Relative Intensity (%)	Compound No.	m/e	Relative Intensity (%)	Assignments	Number (cf., Scheme 2)
IIIa	534	25.8	IIIb	562	6.7	IIIc	602, 604, 606,	3, 2, 0.33,	IIId	594	4.7	[M] ⁺	III
	429	93.6		457	53.3		497, 499, 501,	18.2, 12.1, 3,		489	65.6	[M-COC ₆ H ₅] ⁺	VI
	324	5.1		338	13.3		358, 360,	6.1, 2.0,		354	34.4	[M-VIII-2H] ⁺	VII
	208	48.4		222	31.1		242,244,	9.1, 3.1,		238	56.3	[Ar-CH=CH-CO-C ₆ H ₅] ⁺	VIII
	105	100		105	100		105	100		105	100	[COC ₆ H ₅] ⁺	IX
IVa	530	23	IVb	558	15	IVc	598, 600, 602,	15.3, 10, 1.5,	IVd	590	14.5	[M] ⁺	IV
	425	20		453	17		493, 495, 497,	18, 11.7, 2.2,		485	14	[M-COC ₆ H ₅] ⁺	X
	322	50		336	45		356, 358,	60, 18.5,		352	40	[M-VIII] ⁺	XI
	208	5		222	11		242, 244,	20, 6.1,		238	6.1	[Ar-CH=CH-CO-C ₆ H ₅] ⁺	VIII
	105	100		105	100		105	100		105	100	[C ₆ H ₅ CO] ⁺	IX

The ultraviolet spectra of the cyclohexanone derivatives IIIa-d give further evidence for the proposed structure. They show an absorption in the region 248-246 nm which is attributed to the π - π^* transition band of the acetophenone moiety (4). Further support for the structures of these compounds was gained from their mass spectra, which showed peaks corresponding to $[M]^+$, $[M-C_6H_5CO]^+$ (VI); $[M-2H-Ar-CH=CHCOC_6H_5]^+$ (VII), $[Ar-CH=CH-COC_6H_5]^+$ (VIII) and $[C_6H_5C=O]^+$ (cf. Table II, Scheme 2).

The chemical reactions of the cyclohexanones IIIa-d gave further evidence for the assigned structure. Thus, the boiling of these compounds with hydrazine hydrate in ethanol gave the corresponding 5-benzoyl-4,6-diaryl-3,7-diphenyl-1,4,5,6,7-pentahydro-1*H*-indazoles (IVa-d). The reaction seems to be similar to the reactions of hydrazine hydrate with β -diketones and β -ketoesters (5,6). The structures of these compounds were identified from their spectral data (ir, uv, nmr and ms), single-crystal x-ray analysis and chemical reaction. Thus, their ir spectra show a broad band in the range 3400-3200 cm^{-1} attributable to ν NH in addition to two strong bands in the region 1670-1650 cm^{-1} that can be ascribed to the ν C=O and (benzoyl group) and ν C=C or/and ν C=N of the indazole system (3). The uv spectra of these compounds are identical and similar to those spectra reported for the cyclohexanone derivatives III. They show absorption maxima in the range 249-248 nm (cf., Table I). This is an π - π^* transition band which can be attributed to the K-band of the acetophenone moiety (2). The nmr spectra of these compounds also gave another evidence for their structure, showing signals correlated to the methine and aromatic protons (Table I). The mass spectra of these compounds also support the assigned structures, showing peaks corresponding to $[M]^+$, $[M-C_6H_5CO]^+$ (X), $[M-Ar-CH=CH-COC_6H_5]^+$ (XI), $[Ar-CH=CH-COC_6H_5]^+$ (VIII) and $[C_6H_5CO]^+$ (IX) molecular ions (Scheme 2, Table II).

Structure Determination of IVc by X-Ray Analysis.

The structure and configuration of IVc have been

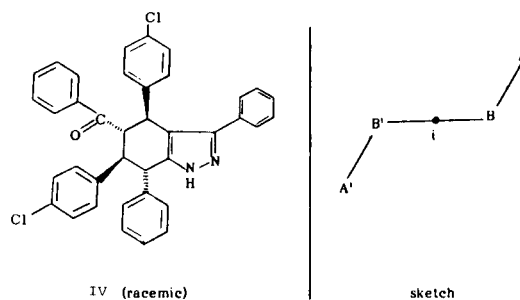


Figure 1

established by a single-crystal X-ray analysis as 5-benzoyl-4,6-bis-*p*-chlorophenyl-3,7-diphenyl-1,4,5,6,7-pentahydro-1*H*-indazole (racemic) and is shown in Figure 1.

The structure was solved by a multiple solution procedure (7). Full-matrix least squares was used for all refinements. Early in the analysis it became apparent that there were solvent molecules present in the crystals. These molecules are located close to the inversion center at $\frac{1}{2}$, 0, 0. Two atoms, A and B, were found (see sketch, Figure 1). Atom B was closest to the inversion center *i* and was only about 1.6 Å from atom B', the atom related to B by the inversion center. These four atoms (A, B, B', A') were taken to be due to methylene chloride disordered about the inversion center. The presence of methylene chloride was confirmed by 100 MHz nmr. Thus, the atoms Cl-C-Cl sometimes take the positions A-B-B' and sometimes the positions B-B'-A'. In the refinement, atom A was assigned the chlorine scattering curve while the scattering curve used for atom B was the sum of those for chlorine and carbon. Atoms A and B were given atom multipliers of 0.5 (because the one half of the methylene chloride molecules are in the position A-B-B' and one half of the molecules are in the position B-B'-A'). Towards the conclusion of the refinement; the atom multiplier of atom B was permitted to vary (and the multiplier for atom A was reset to the new value for atom B after each least squares cycle). The final

Table III

Final Positional and Anisotropic Thermal Parameters for IVc with Estimated Standard Deviations in Parentheses

Anisotropic Thermal Parameters are in the Form $\exp - (h^2\beta_{11} + K^2\beta_{22} + I^2\beta_{33} + 2hK\beta_{12} + 2hI\beta_{13} + 2KI\beta_{23})$

Atom	X	Y	Z	$\beta_{11} \times 10^4$	$\beta_{22} \times 10^4$	$\beta_{33} \times 10^4$	$\beta_{12} \times 10^4$	$\beta_{13} \times 10^4$	$\beta_{23} \times 10^4$
Cl(1)	0.8303(2)	0.4910(1)	0.5817(1)	312(3)	103(1)	39(1)	39(1)	-35(1)	-24(1)
Cl(2)	0.4695(2)	-0.0870(1)	0.2272(1)	341(3)	34(1)	80(1)	-13(1)	26(1)	2(1)
O(1)	0.3424(3)	0.2562(2)	0.3140(2)	80(3)	64(2)	47(1)	7(2)	25(2)	12(1)
N(1)	0.5091(3)	0.4327(2)	0.0697(2)	126(4)	35(2)	26(1)	-3(2)	15(2)	-1(1)
N(2)	0.5128(3)	0.5070(2)	0.0962(2)	128(4)	38(2)	29(2)	0(2)	13(2)	1(1)
C(3)	0.5037(3)	0.5023(2)	0.1712(2)	103(5)	35(2)	28(2)	6(2)	12(2)	1(2)
C(4)	0.4824(3)	0.3866(2)	0.2674(2)	88(4)	36(2)	26(2)	10(2)	16(2)	4(1)
C(5)	0.5041(3)	0.2974(2)	0.2648(2)	79(4)	35(2)	28(3)	6(2)	15(2)	4(1)
C(6)	0.4385(3)	0.2609(2)	0.1823(2)	69(4)	33(2)	30(2)	1(2)	9(2)	0(1)
C(7)	0.4886(3)	0.2950(2)	0.1156(2)	77(4)	35(2)	27(2)	-1(2)	10(2)	-2(1)
C(8)	0.4974(3)	0.3809(2)	0.1258(2)	85(4)	33(2)	27(2)	-2(2)	8(2)	3(2)
C(9)	0.4943(3)	0.4239(2)	0.1925(2)	88(4)	33(4)	26(2)	6(2)	8(2)	1(1)
C(10)	0.4536(4)	0.2581(2)	0.3278(2)	82(4)	34(2)	35(2)	4(2)	21(2)	1(1)
C(11)	0.5053(4)	0.5739(2)	0.2182(2)	129(5)	31(2)	29(2)	14(3)	5(3)	4(2)
C(12)	0.4345(4)	0.5804(3)	0.2699(3)	177(6)	38(2)	40(2)	24(3)	30(3)	5(2)
C(13)	0.4365(6)	0.6481(3)	0.3136(3)	237(8)	52(3)	37(2)	46(4)	35(3)	13(2)
C(14)	0.5101(7)	0.7087(3)	0.3059(3)	263(10)	53(3)	37(2)	31(4)	1(4)	-9(2)
C(15)	0.5795(5)	0.7046(3)	0.2538(4)	196(7)	44(2)	63(3)	-5(3)	8(4)	-9(2)
C(16)	0.5775(4)	0.6371(3)	0.2102(3)	156(6)	44(2)	45(2)	0(3)	16(3)	-7(2)
C(21)	0.5676(4)	0.4187(2)	0.3447(2)	120(5)	32(2)	26(2)	16(2)	17(3)	3(1)
C(22)	0.5295(4)	0.4206(2)	0.4129(3)	171(6)	41(2)	34(2)	24(3)	30(3)	1(2)
C(23)	0.6112(7)	0.4426(3)	0.4864(3)	236(9)	55(2)	31(2)	42(4)	35(4)	2(2)
C(24)	0.7282(7)	0.4626(3)	0.4893(3)	225(9)	52(2)	30(2)	36(4)	-9(4)	-9(2)
C(25)	0.7684(4)	0.4624(3)	0.4234(3)	151(6)	49(2)	37(2)	5(3)	-5(3)	-7(2)
C(26)	0.6870(4)	0.4393(2)	0.3507(2)	130(6)	41(2)	30(2)	7(3)	12(3)	0(1)
C(31)	0.5335(3)	0.2240(2)	0.4024(2)	89(5)	32(2)	32(2)	1(2)	19(2)	3(1)
C(32)	0.4800(4)	0.1879(2)	0.4544(3)	104(5)	44(2)	45(2)	5(2)	29(3)	10(2)
C(33)	0.5495(5)	0.1584(3)	0.5267(3)	153(7)	53(2)	46(2)	0(3)	34(3)	17(2)
C(34)	0.6754(5)	0.1650(3)	0.5482(3)	162(7)	55(2)	39(2)	-1(3)	7(3)	15(2)
C(35)	0.7312(4)	0.1995(3)	0.4975(3)	103(5)	74(3)	46(2)	-5(3)	1(3)	21(2)
C(36)	0.6606(4)	0.2290(3)	0.4247(2)	97(5)	56(2)	38(2)	-4(3)	15(3)	15(2)
C(41)	0.4469(3)	0.1731(2)	0.1870(2)	83(4)	38(2)	27(2)	-5(2)	12(2)	-1(1)
C(42)	0.3434(4)	0.1272(3)	0.1692(3)	92(5)	47(2)	55(2)	-9(3)	19(3)	-5(2)
C(43)	0.3488(5)	0.0475(3)	0.1802(3)	153(7)	41(2)	62(3)	-28(3)	36(3)	-5(2)
C(44)	0.4612(6)	0.0127(3)	0.2095(3)	187(7)	32(2)	45(2)	-7(3)	24(3)	0(2)
C(45)	0.5659(5)	0.0557(3)	0.2259(3)	144(6)	39(2)	44(2)	15(3)	-1(3)	2(2)
C(46)	0.5659(5)	0.1350(3)	0.2145(2)	91(5)	37(2)	43(2)	0(2)	6(2)	0(2)
C(51)	0.4077(4)	0.2697(2)	0.0340(2)	100(5)	39(2)	29(2)	-5(2)	12(2)	0(2)
C(52)	0.2942(5)	0.3009(4)	0.0015(3)	119(6)	108(4)	46(2)	34(4)	-12(3)	-21(2)
C(53)	0.2175(5)	0.2737(5)	-0.0715(4)	120(6)	150(5)	53(3)	4(5)	-13(4)	-9(3)
C(54)	0.2538(7)	0.2185(5)	-0.1112(3)	203(9)	101(4)	42(3)	-38(5)	7(4)	-18(3)
C(55)	0.3661(8)	0.1865(5)	-0.0798(4)	270(11)	122(5)	63(3)	31(6)	5(5)	-51(3)
C(56)	0.4422(5)	0.2125(4)	-0.0074(3)	160(7)	106(4)	46(3)	31(4)	2(3)	-31(3)
Cl + C	0.5676(4)	0.0140(3)	-0.0028(3)	282(8)	114(3)	114(3)	-12(3)	51(4)	7(2)
Cl	0.6862(6)	0.0159(3)	0.0675(5)	244(8)	119(3)	185(5)	-36(4)	52(6)	-7(3)

value of the multiplier for atom B was 0.412(4).

In the final refinement, anisotropic thermal parameters were used for the non-hydrogen atom (including solvent atoms A and B) and isotropic temperature factors were used for the hydrogen atoms. The hydrogen atoms were included in the structure factor calculations but their parameters were not refined. The final discrepancy

indices are $R = 0.054$ and $WR = 0.070$ for the remaining 2746 observed reflections. The final difference map has no peaks greater than $\pm 0.4 \text{ eA}^{-3}$.

The final atomic parameters with their estimated standard deviations are listed in Tables III and IV. The molecular stereoscopic drawing of a molecule of IVc is shown in Figure 2. The bond lengths and angles are given

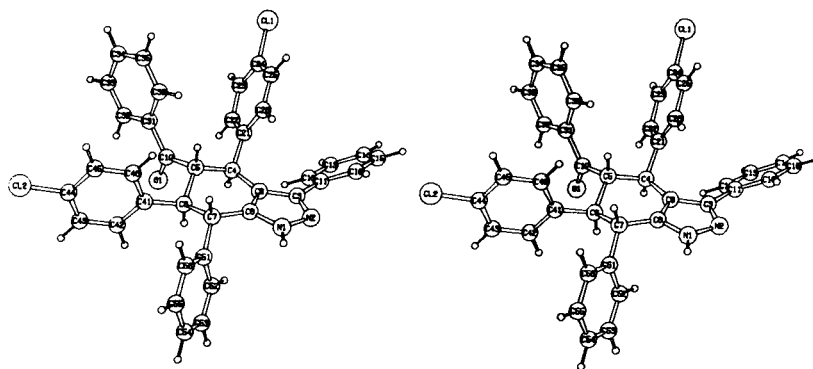


Figure 2: Molecular Stereoscopic drawing of a molecule of IVc.

Table IV

Final Hydrogen-Atom Positional Parameters and Thermal Parameters (a)

Atom	X	Y	Z	B
HN(1)	0.514	0.418	0.018	5.0
H(4)	0.396	0.395	0.269	4.0
H(5)	0.594	0.287	0.278	4.0
H(6)	0.350	0.275	0.169	4.0
H(7)	0.573	0.274	0.123	4.0
H(12)	0.381	0.536	0.276	6.0
H(13)	0.384	0.653	0.350	8.0
H(14)	0.514	0.757	0.339	8.0
H(15)	0.631	0.750	0.247	8.0
H(16)	0.629	0.633	0.172	6.0
H(22)	0.443	0.406	0.409	6.0
H(23)	0.584	0.444	0.536	7.0
H(25)	0.854	0.479	0.427	7.0
H(26)	0.716	0.437	0.302	6.0
H(32)	0.388	0.183	0.439	6.0
H(33)	0.509	0.132	0.564	7.0
H(34)	0.726	0.144	0.601	7.0
H(35)	0.823	0.204	0.513	7.0
H(36)	0.701	0.254	0.388	6.0
H(42)	0.261	0.152	0.147	6.0
H(43)	0.272	0.016	0.167	7.0
H(45)	0.648	0.029	0.246	6.0
H(46)	0.637	0.166	0.226	5.0
H(52)	0.265	0.344	0.030	9.0
H(53)	0.134	0.296	-0.094	11.0
H(54)	0.199	0.200	-0.164	11.0
H(55)	0.395	0.144	-0.109	11.0
H(56)	0.525	0.188	0.015	9.0
HCL + CA	0.586	-0.021	-0.044	14.0
HCL + CB	0.556	0.069	-0.024	14.0

(a) The hydrogen atoms are numbered according to the atoms to which they are attached.

in Tables V and VI, respectively.

Further evidence for the structure of the 1*H*-indazole derivatives was gained from the treatment of IVa with acetic anhydride which gave the corresponding *N*-acetyl derivative Va; the structure of this was established by its

Table V

Bond Lengths (Å°) in IVc with Standard Deviations in Parentheses

Cl(1)-C(24)	1.758(5)	C(21)-C(22)	1.383(7)
Cl(2)-C(44)	1.736(6)	C(21)-C(26)	1.380(7)
O(1)-C(10)	1.219(5)	C(22)-C(23)	1.397(6)
N(1)-N (2)	1.353(5)	C(23)-C(24)	1.364(11)
N(1)-C (8)	1.358(5)	C(24)-C(25)	1.360(9)
N(2)-C (3)	1.346(6)	C(25)-C(26)	1.390(6)
C(3)-C (9)	1.408(5)	C(31)-C(32)	1.381(6)
C(3)-C(11)	1.476(6)	C(31)-C(36)	1.387(6)
C(4)-C (5)	1.553(5)	C(32)-C(33)	1.373(6)
C(4)-C (9)	1.499(6)	C(33)-C(34)	1.377(8)
C(4)-C(21)	1.515(5)	C(34)-C(35)	1.369(8)
C(5)-C (6)	1.546(5)	C(35)-C(36)	1.385(6)
C(5)-C(10)	1.539(6)	C(41)-C(42)	1.376(6)
C(6)-C (7)	1.554(5)	C(41)-C(46)	1.393(5)
C(6)-C(41)	1.511(5)	C(42)-C(43)	1.379(7)
C(7)-C (8)	1.485(5)	C(43)-C(44)	1.368(8)
C(7)-C(51)	1.515(5)	C(44)-C(45)	1.360(8)
C(8)-C (9)	1.388(6)	C(45)-C(46)	1.374(7)
C(10)-C(31)	1.474(5)	C(51)-C(52)	1.360(6)
C(11)-C(12)	1.382(7)	C(51)-C(56)	1.345(8)
C(11)-C(16)	1.393(7)	C(52)-C(53)	1.396(8)
C(12)-C(13)	1.387(7)	C(53)-C(54)	1.310(11)
C(13)-C(14)	1.367(9)	C(54)-C(55)	1.352(11)
C(14)-C(15)	1.373(10)	C(55)-C(56)	1.379(8)
C(15)-C(16)	1.384(7)		

spectral data (ir, uv, and nmr) (Table 1). The mass spectrum of the *N*-acetyl derivative Va also support the assigned structure, showing peaks corresponding to the following *m/e* values: (a) 572 (20%) [M]⁺, (b) 529 (10%) [M-COCH₃]⁺, 467 (30%) [M-COC₆H₅]⁺, 364 (10%) [M-C₆H₅CH=CH-COC₆H₅]⁺, 322 (35%) [XI]⁺ and 105 (100%) [C₆H₅CO]⁺ (Scheme 2).

EXPERIMENTAL

Microanalyses were performed by Dr. A. Bernhardt, Microanalytisches Laboratorium, West Germany. The nmr spectra were recorded on a JEOL JNM-PM Spectrometer using TMS as internal standard. The infrared and uv spectra were measured on a Perkin Elmer 577 Grating Infrared (Nujol) and Beckman ACTA MVI (ethanol) Spectrophotometers, respectively. Mass spectra were measured with Varian MAT 311A mass

Table VI

Bond Angles (A°) in IVc with Standard Deviations in Parentheses

N(2)-N (1)-C (8)	111.7(3)	C(22)-C(21)-C(26)	118.8(3)
N(1)-N (2)-C (3)	105.8(3)	C(21)-C(22)-C(23)	120.3(5)
N(2)-C (3)-C (9)	110.4(3)	C(22)-C(23)-C(24)	118.7(5)
N(2)-C (3)-C(11)	120.0(3)	Cl(1)-C(24)-C(23)	118.7(5)
C(9)-C (3)-C(11)	129.6(4)	Cl(1)-C(24)-C(25)	118.7(5)
C(5)-C (4)-C (9)	109.8(3)	C(23)-C(24)-C(25)	122.6(5)
C(5)-C (4)-C(21)	108.6(3)	C(24)-C(25)-C(26)	118.2(5)
C(9)-C (4)-C(21)	115.0(3)	C(21)-C(26)-C(25)	121.4(4)
C(4)-C (5)-C (6)	112.9(3)	C(10)-C(31)-C(32)	118.9(4)
C(4)-C (5)-C(10)	108.3(3)	C(10)-C(31)-C(36)	123.0(4)
C(6)-C (5)-C(10)	108.0(3)	C(32)-C(31)-C(36)	118.1(3)
C(5)-C (6)-C (7)	111.3(3)	C(31)-C(32)-C(33)	121.5(4)
C(5)-C (6)-C(41)	110.3(3)	C(32)-C(33)-C(34)	119.5(5)
C(7)-C (6)-C(41)	112.5(3)	C(33)-C(34)-C(35)	120.3(4)
C(6)-C (7)-C (8)	108.1(3)	C(34)-C(35)-C(36)	119.8(4)
C(6)-C (7)-C(51)	110.0(3)	C(31)-C(36)-C(35)	120.7(4)
C(8)-C (7)-C(51)	113.4(3)	C(6)-C(41)-C(42)	121.5(3)
N(1)-C (8)-C (7)	125.5(3)	C(6)-C(41)-C(46)	121.6(3)
N(1)-C (8)-C (9)	106.8(3)	C(42)-C(41)-C(46)	116.7(4)
C(7)-C (8)-C (9)	127.7(4)	C(41)-C(42)-C(43)	122.5(4)
C(3)-C (9)-C (4)	132.3(4)	C(42)-C(43)-C(44)	118.9(5)
C(3)-C (9)-C (8)	105.3(3)	Cl (2)-C(44)-C(43)	119.5(4)
C(4)-C (9)-C (8)	122.4(3)	Cl (2)-C(44)-C(45)	119.9(4)
O(1)-C(10)-C (5)	117.2(3)	C(43)-C(44)-C(45)	120.6(4)
O(1)-C(10)-C(31)	120.0(4)	C(44)-C(45)-C(46)	120.1(4)
C(5)-C(10)-C(31)	122.9(4)	C(41)-C(46)-C(45)	121.3(4)
C(3)-C(11)-C(12)	121.2(4)	C(7)-C(51)-C(52)	121.4(4)
C(3)-C(11)-C(16)	120.2(4)	C(7)-C(51)-C(56)	121.4(4)
C(12)-C(11)-C(16)	118.6(4)	C(52)-C(51)-C(56)	117.1(4)
C(11)-C(12)-C(13)	120.6(5)	C(51)-C(52)-C(53)	120.8(6)
C(12)-C(13)-C(14)	119.6(6)	C(52)-C(53)-C(54)	120.9(6)
C(13)-C(14)-C(15)	121.1(5)	C(53)-C(54)-C(55)	119.3(6)
C(14)-C(15)-C(16)	119.3(5)	C(54)-C(55)-C(56)	120.3(7)
C(11)-C(16)-C(15)	120.7(5)	C(51)-C(56)-C(55)	121.6(6)
C(4)-C(21)-C(22)	119.6(4)		
C(4)-C(21)-C(26)	121.3(4)		

spectrometer. Evaporation was performed *in vacuo* on a rotary evaporator.

3,5-Diaryl-2,4-dibenzoyl-6-phenyl-cyclohexanones (IIIa-d).

General Procedure.

3-Aryl-1-phenyl-2-propen-1-one II (0.03 mole) was added to a suspension of sodium ethoxide (0.03 mole) and ethyl phenylacetate (I) (0.015 mole) in dry benzene (200 ml.). The reaction mixture, which gradually acquired a deep orange colour, was heated on a boiling water-bath for one hour with occasional stirring, then poured into cold water (250 ml.). The benzene layer was washed, dried (sodium sulfate) and evaporated. The colourless residue was crystallized from a suitable solvent to give the corresponding 3,5-diaryl-2,4-dibenzoyl-6-phenylcyclohexanones III as colourless crystals. The results are reported in Table VII.

Acidification of the alkaline aqueous layer with dilute hydrochloric acid gave phenylacetic acid, m.p. and mixed m.p. 76-77°.

5-Benzoyl-4,6-diaryl-3,7-diphenyl-1,4,5,6,7-pentahydro-1H-indazoles (IVa-d).

General Procedure.

A solution of the pyrone III (2.0 g.) in ethanol (25 ml.) was refluxed with hydrazine hydrate (99% w/w; 3 ml.) on a boiling water-bath for 3 hours. The precipitated solid was filtered and crystallized from a suitable solvent to give the corresponding 1H-indazole derivative IV as colourless crystals. The results are reported in Table VII.

Heating of IVa (1.0 g.) with acetic anhydride (3 ml.) on a boiling water-bath for 30 minutes afforded the corresponding *N*-acetyl derivative Va as colourless leaflets m.p. 267-268°, yield 88%.

Anal. Calcd. for C₄₀H₃₂N₂O₂: C, 83.89; H, 5.63; N, 4.89; M. W. 572. Found: C, 83.43; H, 5.75; N, 4.69; M. W. (ms) 572.

X-Ray Crystallographic Analysis of IVc.

Good single crystals of IVc were obtained upon recrystallization from methylene chloride/petroleum ether. The crystals were monoclinic, space group P2₁/a, with a = 11.399(4), b = 17.161(3), c = 17.466(4) Å, β = 107.16(2)°, and d calcd. = 1.288 g cm⁻³ for Z = 4 (C₃₈H₂₈Cl₂N₂O · 0.4 CH₂Cl₂) M.W. = 633.53). The intensity data were measured on a Hilger-watts diffractometer (nickel filtered copper Kα radiation, θ-2θ scans, plus height discrimination). A crystal measuring approximately 0.35 × 0.40 × 0.45 mm was used for data collection; the data were corrected for

Table VII

3,5-Diaryl-2,4-dibenzoyl-6-phenylcyclohexanones IIIa-d and 1H-indazole Derivatives (IVa-d)

Compound No.	Yield (%)	M.p., °C	Formula	Calcd. %					Found %				
				C	H	N	Cl	M.W	C	H	N	Cl	M.W (MS)
IIIa	92	274-275 (a)	C ₃₈ H ₃₀ O ₃	85.37	5.66	—	—	534	85.36	5.64	—	—	534
IIIb	95	269-270 (b)	C ₄₀ H ₃₄ O ₃	85.38	6.09	—	—	562	85.36	5.92	—	—	562
IIIc	89	228-229 (b)	C ₃₈ H ₂₈ Cl ₂ O ₃	75.62	4.68	—	11.75	602,	75.66	4.77	—	11.64	602,
								604, 606					604, 606
IIIId	92	226-227 (c)	C ₄₀ H ₃₀ O ₃	80.79	5.76	—	—	594	80.49	5.87	—	—	594
IVa	95	280-281 (c)	C ₃₈ H ₃₀ N ₂ O	86.01	5.70	5.28	—	530	86.27	5.90	5.23	—	530
IVa	97	255-256 (c)	C ₄₀ H ₃₄ N ₂ O	85.99	6.13	5.01	—	558	85.65	6.20	5.06	—	558
IVc	95	257-258 (a)	C ₃₈ H ₂₈ Cl ₂ N ₂ O	76.13	4.71	4.67	11.83	598,	76.34	4.86	4.59	—	598,
								600, 602					600, 602
IVd	94	272-273 (a)	C ₄₀ H ₃₄ N ₂ O ₃	81.33	5.80	4.74	—	590	81.48	5.93	4.64	—	590

(a) Crystallized from benzene. (b) Crystallized from methanol. (c) Crystallized from methanol-benzene.

absorption ($\mu = 26.5 \text{ cm}^{-1}$). A total of 3059 reflections were measured for $\theta < 48^\circ$, of which 2748 were considered to be observed [$1 > 2.50 \delta(1)$]. Two reflections, which were strongly affected by extinction, were excluded from the final refinement and difference map.

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