REACTIONS OF PHENYLISOINDOLES WITH SOME SELECTED ORAGANIC ACCEPTORS

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Abstract: 1-Phenylisoindole **1** and 2-phenyl-1-phenylimino-2,3-dihydro-1H-isoindole **11** (electron donors) reacted with tetracyanoethylene (TCNE) and gave 3-dicyanomethyleneisoindole **2** and 1-phenylisoindol-3-one 3. Using 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) and 2,3,5,6-tetrachloro-1,4-benzoquinone (CHL-*p*) as π-acceptors afforded mainly 3,3'-bisphenylisoindole **4** as well as condensation products. 2,3-Dichloro-1,4-naphthoquinone (DCHNQ) gave miscellaneous reaction products, in addition to the isoindole dimer **4**. The two donor systems exhibit, in general, the same behaviour towards the π-acceptors investigated.

Introduction:

Among the fused five membered heterocylic systems, indole and isoindole are of interest in connection with our previous investigation on isoindolines (1-5). Whereas indole chemistry has long enjoyed considerable attention, its isomer isoindole, has not received the same interest due to its unstability under normal reaction conditions (6). Consequently, isoindoles are considered as highly reactive intermediates, whose activity, however, may be reduced by substitution in the position three of the five membered ring.

Results and Discussions:

As a part of our continuing study in the synthesis of new heterocyclic ring systems via charge-transfer (CT) complexation (7-12), we became interested in employing 1-phenylisoindoles **1** (13) and 2-phenyl-1-phenylimino-2,3-dihydro-1H-isoindole **11** (14) as electron donors in donor-acceptor reactions. Our overall strategy is outlined in Schemes 1 and 2.

1-Phenyl-3-dicyanomethyleneisoindole **2** and 1-phenylisoindol-3-one **3** (15) were obtained by addition of equimolar amounts of tetracyanoethyiene (TCNE) to 1-phenylisoindole **1** in ethyl acetate, after CT complexes have been formed initially (Scheme 1).

On the other hand, upon adding ethyl acetate solutions of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) to the solution of donor **1** a pink colour was observed (CT-complex), which rapidly changed to brown

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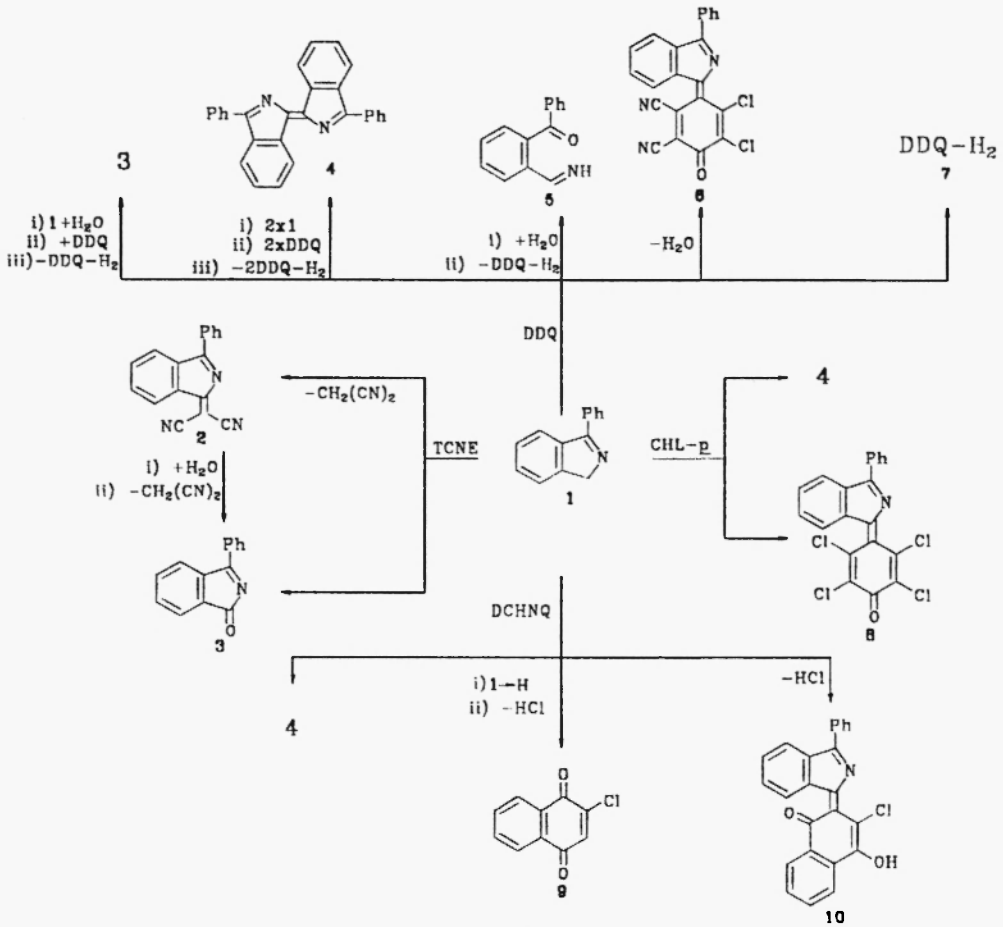
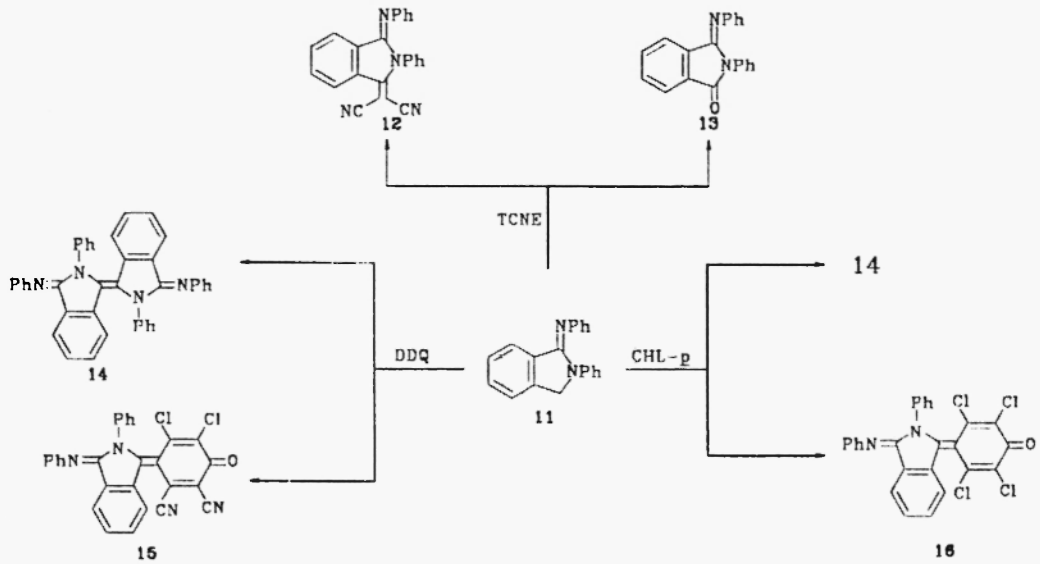


Scheme 1



Scheme 2

PLT A:\ASHRAF1D 01-13-1997 Scale: .40 Ht: 3.60

and afforded the dimer **4**, the condensation product **6**, and hydroquinone derivative **7** as well as compounds **3** and **5**.

In the same solvent compound **1** immediately formed CT-complex with 2,3,5,6-tetrachloro-1,4-benzoquinone (CHL-*p*), transformed to the dimer **4** as well as the condensation product **8** within two days (Scheme 1).

In a different manner, 2,3-dichloro-1,4-naphthoquinone (DCHNQ) reacted with the isoindole derivative **1** via CT-complex formation to yield the dimer **4**, and 2-chloronaphthoquinone **9** as well as the product 1-phenyl-3(1'-hydroxy-2'-chloro-4'-oxo-naphtha-3'-ylidene)isoindole **10** (Scheme 1).

Addition of **1** to another electron acceptor namely 7,7,8,8- tetracyanoquinodimethane (TCNQ) in ethyl acetate results in formation of a crystalline CT-complex (see exp.).

In view of these promising results, it is interesting to extend our studies on another recently prepared isoindole donor system (14), 2-phenyl-1-phenylimino-2,3-dihydro-1H-isoindole **11**, aiming to shed more light on the effect of azomethine group on the electron-donative nature of this system.

Scheme 2 summarizes the reaction products obtained on subjecting the isoindole derivative **11** to the action of TCNE, DDQ and CHL-*p*, it can be conculded that the donor character of **11** towards TCNE and CHL-*p* is similar to that of **1**. In case of DDQ, however, only two products were formed.

Conclusion:

From the above findings that, isoindoles **1** and **11** are interesting donor systems which produce promising reaction products on treatment by a variety of organic acceptors. The methylene group in the donor molecules studied is the active center responsible for all subsequent chemical reactions.

Acknowledgement:

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Experimental:

Melting points are uncorrected. IR spectra (KBr) were obtained on Nicolet 320 FT-IR. ^{1}H-NMR (400.134 MHz.) and ^{13}C-NMR (100.164 MHz.) spectra were measured on Bruker AM 400 and the chemical shifts were expressed as δ (ppm) using Me_4 Si an internal reference. J values were expressed in Hz. Mass spectra were recorded on a Finnigan MAT 8430 instrument at 70 e.V. Microanalyses were performed by Microanalytical Laboratory Kronach, Braunschweig, Germany .

Materials.- The organic acceptors, TCNE, DDQ, CHL-*p*, TCNQ and DCHNQ were purified as reported in previous works. Ethyl acetate (B.D.H.) was purified following Vogel (16), dried and distilled. 1-Phenyl-

isoindole **1** was synthesized accerding references 13 and 17. 2-Phenyl-1-phenylimino-2,3-dihydro-1H-isoindole **11** was prepared according to reference 14.

Reaction of 1-phenylisoindole **1** *with TCNE.*- To a cold solution of **1** (386 mg, 0.002 mol) in dry ethyl acetate (5 ml) at 0^0C, an ice cooled solution of TCNE (256 mg, 0.002 mol) in dry ethyl acetate (10 ml) was added, and the reaction mixture was stirred at this temperature for 3 h and at room temperature for 48 h. Concentration and chromatography on thin layer plates using toluene : ethyl acetate (1:1) as eluent yielded compounds **2** and **3**.

1-Phenyl-3-dicyanomethyleneisoindole **2**.- (250 mg, 49 %) had mp 250^0C, yellow crystals from ethanol; ν_{max}/cm^{-1} 3120-3003 (Ar-CH), 2214 (CN), 1630 (C=C), 1580 (C=N); δ_H ([$^2H_6$]DMSO), 7.30-7.91(m, 7H, Ar-H), 8.22-8.39 (m, 2H, Ar-H); δ_c ([$^2H_6$]DMSO),116.31, 116.66(CN); 125.56, 125.66, 125.84, 128.17, 128.99, 129.46, 129.45, 129.81, 129.49, 129.86, 133.64, 134.40, 139.70, 149.71 and 163.70; *m/z* 255 (M^+, 100), 254(92), 229(90), 228(68), 202(16), 196(20), 190(34), 181(10), 152(18), 114(14) and 98(16) (Found: C, 79.82; H, 3.51; N, 16.47. $C_{17}H_9N_3$ requires: C, 79.99; H, 3.53; N, 16.46 %).

1-Phenylisoindol-3-one **3**.- (91 mg, 22%) had mp 143^0C (lit. (15) , 144^0C).

Reactions of 1-phenylisoindole **1** *with DDQ.*- To a cold solution of **1** (386 mg, 0.002 mol) in dry ethyl acetate (10 ml) at 0^0C, an ice cooled solution of DDQ (454 mg, 0.002 mol) in dry ethyl acetate (10 ml) was added with stirring at this temperature for 3 h. and at room temperature for 48 h. Evaporation and chromatographic separation of the residue using toluene as eluent afforded compounds **3**-**6**.

3.- (150 mg, 36%). Its spectral data is identical in every way to that of compound obtained in case of the reaction of **1** with TCNE.

3,3'-Bisphenylisoindole 4.- (310 mg, 41%) had mp $261\text{-}262^0$C, orange crystals from ethanol; ν_{max}/cm^{-1} 3010-3000 (Ar-CH), 1665 (C=C), 1580 (C=N); δ_H ($CDCl_3$), 7.26-7.46 (m, 10H, Ar-H), 7.97-8.00 (dd, 2H, J=9.67, Ar-H), 8.32-8.34 (m, 4H, Ar-H), 9.01-9.11(dd, 2H, J=8.43, Ar-H); δ_c ($CDCl_3$), 126.18, 128.98, 129.28, 130.18, 135.91, 137.32, 138.66, 143.61, 144.82, 145.30, 147.09, 149.73, 155.42 and 164.80; *m/z* 382 (M^+. 100), 381(24), 379(8), 280(6), 279(8), 278(10), 191(12), 190(6),111(12), 98(10), 97(12), 81(36), 69(94) and 57(24) (Found: C, 87.80; H, 4.80; N, 7.30. $C_{28}H_{18}N_2$ requires: C, 87.93; H, 4.74; N, 7.32 %).

o-Iminomethylenebenzophenone **5**.- (30 mg, 7%) had mp 130^0C yellow crystals from acetonitrile; ν_{max}/cm^{-1} 3380-3200 (NH), 3010-3000(Ar-CH), 1700(CO); δ_H ($CDCl_3$), 7.28-7.40 (m, 5H, Ar-H),), 7.48-7.59 (m, 4H Ar-H), 7.62 (s, 1H, CH=NH), 9.40 (s, 1H, NH); δ_c ($CDCl_3$), 120.75, 122.09, 123.46, 125.18, 126.27, 128.87, 128.95, 129.18, 130.00, 130.90, 133.88, 135.66, 138.15 (CH=N) and 190.00 (CO); *m/z* 209 (M^+, 16), 208(100), 165(8), 152(10), 148(12), 131(16), 130(30) and 102(12) (Found: C, 80.40; H, 5.29; N, 6.69. $C_{14}H_{11}NO$ requires: C, 80.36; H, 5.30; N, 6.69 %).

1-Phenyl-3(2',3'-dichloro-5',6'-dicyano-4'-oxo-cyclohexylidene-2',5'-diene)isoindole **6**.- (100 mg, 12%) had mp $310\text{-}312^0$C yellow crystals from ethanol; ν_{max}/cm^{-1} 3010-3000 (Ar-CH), 2210(CN), 1685(CO); δ_H ($CDCl_3$), 7.88-8.15(m, 5H, Ar-H), 8.20-8.23 (M, 3H Ar-H), 8.30-8.32 (dd, 1H, Ar-H); δ_c ($CDCl_3$), 117.19,

118.22(CN); 125.08, 126.95, 129.50, 129.63, 130.46, 133.60, 135.62, 136.30, 140.00, 146.21, 146.33, 148.21, 150.33, 151.91, 161.72, 163.48, 167.81, 168.34, 170.50 and 186.50(CO); *m/z* 404(M^{+2}, 38), 403(M^{+1}, 72), 402(M^{+}, 100), 401(38), 400(30), 375(60), 339(12), 303(68), 278(15), 230(16), 218(20), 195(44), and 167(18). (Found: C, 65.70; H, 2.24; Cl, 17.58; N, 10.46. $C_{22}H_9Cl_2N_3O$ requires: C, 65.69; H, 2.26; Cl, 17.63; N, 10.45 %).

Reaction of 1-phenylisoindole 1 *with CHL-p.*- The reaction was carried out as in **2** using (386 mg, 0.002 mol) of 1 and (488 mg, 0.002 mol) of CHL-*p* and a mixture of toluene: ethyl acetate(10:1) as eluent.

4.- (200 mg, 26%). This compound was identified as mentioned before.

1-Phenyl-3(2',3',5',6'-tetrachloro-4'-oxo-cyclohexylidene-2',5'-diene)isoindole **8**.- (300 mg, 36%) had mp 300^{0}C yellow crystals from ethanol; ν_{max}/cm^{-1} 3010-3000 (Ar-CH), 1680(CO); δ_H ($CDCl_3$), 7.50-7.59 (m, 2H, Ar-H), 7.66-7.72 (m, 5H, Ar-H), 7.99-8.10 (m, 2H, Ar-H); δ_c ($CDCl_3$), 123.88, 125.97, 130.32, 130.42, 133.16, 135.43, 140.22, 145.19, 148.16, 148.48, 150.19, 151.77, 152.39, 154.11, 157.20, 158.88, 159.09, 160.98, 165.27, 189.18(CO); *m/z* 425(M^{+4},18), 424(M^{+3}, 32), 423(M^{+2}, 58), 422(M^{+}, 72), 421(M^{+}, 100), 420(14), 418(32), 384(80), 351(40), 348(42), 310(18), 275(22), 242(18), 241(22), 240(16), 205(32), 204(34), 203(30), 191(14), 190(16), 189(12), 167(48), 166(54), 165(52), 127(32), 126(38) and 125(28) (Found: C, 57.18; H, 2.16; Cl, 33.60; N, 3.30. requires: $C_{20}H_9Cl_4NO$ C, 57.04; H, 2.15; Cl, 33.68; N, 3.33 %).

Reaction of 1-phenylisoindole **1** *with DCHNQ*.- To a stirred cold solution (0^{0}C) of 1 (386 mg, 0.002 mol) in ethyl acetate (5 ml) a cold solution (0^{0}C) of DCHNQ (455 mg, 0.002 mol) was added, and stirring was continued for 3 h. at this temperature and 72 h. at room temperature. Evaporation of the solvent and chromatograohic separation using toluene as eluent yielded products **4**, **9** and **10**.

4.- (310 mg, 34%).

2-Chloronaphthoquinone **9**.- (100 mg, 26%) had mp 150^{0}C yellow crystals from hexane; ν_{max}/cm^{-1} 3090-3000(Ar-CH) 1700(CO); δ_H ($CDCl_3$), 7.26(s, 1H), 7.60-7.75 (m, 2H, Ar-H), 8.00-8.40 (m, 2H, Ar-H); δ_C ($CDCl_3$), 128.63, 129.52, 129.66, 130.39, 131.83, 133.99, 134.80, 135.63, 180.62 (-CO-CH), and 186.62(-CO-C-Cl); *m/z* 193(M^{+1}, 30), 192(M^{+}, 100), 190(42), 157(63) and 131(12) (Found: C, 62.20; H, 2.60; Cl, 18.33. requires: $C_{10}H_5ClO_2$ C, 62.36; H, 2.62; Cl, 18.41 %).

1-Phenyl-3(1'-hydroxy-2'-chloro-4'-oxo-naphtha-3'-ylidene)isoindole **10**.- (250 mg, 33%) had mp 260-262^{0}C decomp. green crystals from toluene; ν_{max}/ cm^{-1} 3600-3504(OH), 3010-3000(Ar-H) 1620(C=C), 1582(C=N), 1682(CO); δ_H ($CDCl_3$), 7.30-7.83 (m, 11H), 8.00-8.22(m, 2H), 8.12 (s, 1H, OH); δ_c ($CDCl_3$), 125.21, 126. 83, 127.24, 127.38, 128.78, 129.07, 131.11, 135.93, 138.18, 143.23, 150.22, 151.44, 151.66, 151.83, 152.98, 153.16, 153.28, 154.16, 154.33, 155.44, 155.83, 156.20, 164.18 and 185.12(CO); *m/z* 384(M^{+1}, 30), 383(M^{+}, 100), 365(12), 349(18), 348(20), 290(18), 248(22), 189(14), 165(12) and 145(16). (Found: C, 74.88; H, 3.60; Cl, 9.10; N, 3.60. requires $C_{24}H_{14}ClNO_2$ C, 75.10; H, 3.68; Cl, 9.24; N, 3.65 %).

Charge-transfer complex of 1-*TCNQ*.- Addition of equimolar amounts of 1 and TCNQ (0.002 mol) in dry ethyl acetate (10 ml) at 0^{0}C to each other gave blue crystals. They were filtered, dreid and analyzed, mp

330°C decomp. UV ($CHCl_3$, λ_{max}, nm): 780. (Found: C, 78.38; H, 3.80; N, 17.60. requires: $C_{26}H_{15}N_2$ C, 78.57; H, 3.80; N, 17.62 %).

Reaction of isoindole 11 *with TCNE:* To a solution of 11 (568 mg, 0.002 mol) dry ethyl acetate (10 ml), a solution of TCNE (256 mg, 0.002 mol) dry ethyl acetate (10 ml) was added, and the reaction mixture was stirred for 48 h. Evaporation of the solvent and chromatographic separation of the residue using toluene as eluent afforded compounds **12** and **13.**

2-Phenyl-1-phenylimino-3-dicyanomethyleneisoindole **12.**- (250 mg, 35%) had mp 180°C, yellow crystals from toluene; ν_{max} /cm^{-1} 3090-3000 (Ar-CH), 1610(C=C), 1566(C=N), 2220 (CN); δ_H ($CDCl_3$), 7.30-7.72(m, 9H, Ar-H), 7.80-8.10 (m, 5H Ar-H); δ_C ($CDCl_3$), 114.32, 115.28(CN); 129.60, 131.34, 133.68, 135.24, 139.35, 142.46, 143.65, 144.08, 144.20, 145.33, 149.28, 151.17, 151.23, 151.63, 151.99, 152.29, 152.43, 153.66, 154.38, 160.18 and 163.82; *m/z* 346(M^+, 100), 320(22), 295(36), 282(80), 274(42), 272(20) and 202(48) (Found: C, 79.60; H, 4.03; N, 16.15. requires: $C_{23}H_{14}N_4$ C, 79.75; H, 4.07; N, 16.17 %).

2-Phenyl-1-phenylimino-isoindol-3-one 13.- (180 mg, 22%) had mp 163°C, yellow crystals from toluene; ν_{max} /cm^{-1} 3080-3010 (Ar-CH), 1695 (CO); δ_H ($CDCl_3$), 7.28-7.63(m, 9H, Ar-H), 7.73-7.92 (m, 5H Ar-H); δ_C ($CDCl_3$), 128.43, 129.52, 130.90, 133.09, 133.28, 134.44, 135.71, 138.24, 140.62, 143.08, 143.28, 145.00, 145.19, 149.33, 150.64, 161.16, 162.85, 164.73, 168.16 and 190.28(CO); *m/z* 298(M^+, 100), 269(16), 226(38), 154(60), 126(42) and 112(28) (Found: C, 80.40; H, 4.71; N, 9.30. requires $C_{20}H_{14}N_2O$: C, 80.52; H, 4.73; N, 9.39 %).

Reaction of isoindole 11 *with DDQ:* The reaction was carreid out as in **6** using 11 (568 mg, 0.002 mol) and DDQ (454 mg, 0.002 mol), and a mixture of toluene : ethyl acetate (10:1) as eluent to produce compounds **14** and **15**.

3,3'-Bis(2-phenyl-1-phenylimino-isoindole) **14.**- (300 mg, 27%) had mp 260°C blue crystals from ethanol; ν_{max} / cm^{-1} 3080-3000 (Ar-CH), 1660(C=C), 1575(C=N); δ_H ($CDCl_3$), 7.52-7.90 (m, 18H, Ar-H), 8.20-8.35(m, 10H, Ar-H); δ_C ($CDCl_3$), 120.91, 121.43, 128.91, 129.13, 129.27, 135.93, 137.08, 138.38, 140.18, 142.66, 143.77, 144.28, 145.24, 146.37, 148.46, 149.98, 155.20, 158.29, 160.18 and 164.90; *m/z* 564(M^+, 100), 492(20), 419(48), 392(60), 320(22) and 277(36) (Found: C, 84.90; H, 4.96; N, 9.90. requires: $C_{40}H_{28}N_4$ C, 85.08; H, 5.00; N, 9.92 %).

2-Phenyl-1-phenylimino-3(2',3'-dichloro-5',6'-dicyano-4'-oxo-cyclohexylidene-2',5'-diene)isoindole **15.**- (210 mg, 21%) had mp 298-300°C green crystals ($CHCl_3$/ EtOEt); ν_{max}/ cm^{-1} 3050- 3000 (Ar-CH), 2218(CN), 1683(CO); δ_H ([$^2H_6$] DMSO), 7.83-8.12 (m, 10H, Ar-H), 8.15-8.25 (m, 4H, Ar-H); δ_C ([$^2H_6$]DMSO), 120.28, 122.16, 128.00, 128.20, 129.14, 131.35, 134.92,136.98, 138.24, 140.19, 143.31, 145.55, 148.29, 150.18, 151.79, 152.22, 155.39, 157.20, 158.09, 158.18, 160.32, 161.71, 163.22, 164.81, 165.20, 168.34, 169.63 and 188.18(CO); *m/z* 495(M^{+2}, 28), 494(M^{+1}, 62), 493(M^+,100), 491(30), 458(60), 430(18), 429(16), 404(22), 403(18), 282(20) and 281(24) (Found: C, 68.20; H, 2.85; Cl, 14.30; N, 11.30. requires: $C_{28}H_{14}Cl_2N_4O$ C, 68.17; H, 2.86; Cl, 14.37; N, 11.36 %).

Reaction of isoindole **11** *with CHL-p:* The reaction was carreid out as in **7** using **11** (568 mg, 0.002 mol) and CHL-*p* (488 mg, 0.002 mol).

14.- (450 mg, 40%). It has same the spectral and analytical data as given before.

2-Phenyl-phenylimino-3(2',3',5',6'-tetrachloro-4'-oxo-cyclohexylidene-2',5'-diene)isoindole **16**.- (200 mg, 20%) had mp 276-278^{0}C decomp. yellow crystals from EtOEt/Cyclohexane; ν/cm^{-1} 3030-3000 (Ar-CH), 1682(CO); δ_H ([$^2H_6$] DMSO), 7.72-8.00 (m, 10H, Ar-H), 8.15-8.20 (m, 4H, Ar-H); δ_C ([$^2H_6$] DMSO), 118.71, 119.39, 130.53, 130.94, 133.81, 136.72, 140.02, 141.24, 141.94, 142.49, 143.18, 143.88, 144.20, 146.09, 148.18, 150.29, 153.63, 155.22, 158.63, 160.29, 164.28, 165.36, 166.22, 168.26, 169.18 and 185.92(CO); *m/z* 516(M^{+4}, 18), 515(M^{+3}, 32), 514(M^{+2}, 58), 513(M^{+1},72), 512(M^+,100), 511(60), 509(28), 474(20), 473(28), 472(10), 436(18), 435(20), 434(22), 403(32), 402(32), 401(10) and 118(69) (Found: C, 60.81; H, 2.70; Cl, 27.52; N, 5.49. requires: $C_{26}H_{14}Cl_4N_2O$ C, 60.97; H, 2.75; Cl, 27.69; N, 5.47 %).

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