Monatshefte für Chemie 118, 369—379 (1987)

**Monatshefte für Chemie**
*Chemical Monthly*

# Interactions of Indole Derivatives with Organic Acceptors. III**

**Giorgio Tosi**[a,] *, **Liberato Cardellini**[a], **Barbara Cardillo**[a],
and **Gabriele Bocelli**[b]

[a] Dipartimento di Scienze dei Materiali e della Terra, Facoltà di Ingegneria,
Università degli Studi di Ancona, I-60130 Ancona, Italy

[b] Centro di Studio C.N.R. per la Strutturistica Diffrattometrica,
Università di Parma, I-43100 Parma, Italy

*(Received 18 December 1985. Accepted 4 March 1986)*

Interactions of 1-hydroxy-2-phenylindole and 2,2′-diphenyl-$\Delta^{3,3'}$-bi-3*H*-indole with common organic acceptors are analyzed. Depending on the nature of donor-acceptor partners, the reaction may afford molecular complexes, redox products or some kind of adduct. The reaction of 1-hydroxy-2-phenylindole with tetracyanoethylene leads to the formation of 1-oxy-2-phenyl-3-(dicyanomethylidene) indole whose crystal structure is reported.

*(Keywords: Charge-transfer complexes; 1,1,2,2-Ethenetetracarbonitrile; Molecular associations)*

*Wechselwirkungen von Indol-Derivaten mit organischen Akzeptoren, 3. Mitt.*

Es werden die Wechselwirkungen von 1-Hydroxy-2-phenylindol und 2,2′-Diphenyl-$\Delta^{3,3'}$-bi-3*H*-indol mit organischen Akzeptoren geprüft. Je nach den Eigenschaften des Akzeptors und des Donors kann die Reaktion molekulare Komplexe, Redox-Produkte oder andere Addukte ergeben. Die Reaktion von 1-Hydroxy-2-phenylindol mit Tetracyanoethylen bildet 1-Oxy-2-phenyl-3-(dicyanomethylidene)indol, dessen Kristallstruktur beschrieben wird.

## Introduction

Molecular complexes of indole derivatives have encountered interest mainly because many biologically active compounds contain indole moieties.

---

** Reference [9] is considered part I while part II is in press in Gazz. Chim. Ital.

Indole itself does not form stable charge transfer complexes (CTC) even in apolar solvents [1]; therefore in polar solvents (such as dichloromethane) with 1,1,2,2-ethenetetracarbonitrile (*TCNE*), the initial complex decomposes affording 3-tricyanovinylindole [2]. Methyl- and methoxy-substituted indoles are able to form stable CTC in solution with a large range of organic acceptors as evidenced with spectroscopic and thermodynamic studies [3, 7].

1-Hydroxy-2-phenylindole, I, and 2,2′-diphenyl-$\Delta^{3,3'}$-bi-3*H*-indole, II, are two particular indole derivatives of large interest in our studies. Both compounds are potential donors like simpler indoles: as a matter of fact, 1-hydroxy-2-phenylindole forms isolable complexes with arylazopyridines [8, 9] through a strong hydrogen bond as evidenced by X-ray determinations. In this paper we report a study on interactions of I and II with some available organic acceptors: tetracyanoethylene (*TCNE*), 2,3-dicyano-5,6-dichloro-1,4-benzoquinone (*DDQ*), chloranil (*CA*), tetracyanoquinodimethane (*TCNQ*), and piromellitic anhydride (*PA*).

## Results and Discussion

The reaction of compound I with acceptors like *DDQ* and *CA*, after initial molecular interactions ($\lambda$ = 387 nm, for *DDQ* and $\lambda$ = 460, 486 nm for *CA*), led to redox compounds and unmelting compounds (in the case of *DDQ*) as reported in the experimental section. The compound I with *TCNQ* formed only very weak interactions as evidenced by the light blue colour and by spectroscopic measurements. No reaction was evidenced between the hydroxyindole I and *PA*. With *TCNE*, the hydroxyindole *I*, after an initial unstable CT interaction (multiple bands between 468–405 nm), affords (in quantitative yield) 1-oxy-2-phenyl-3-(dicyano methylidene)indole, III (whose crystal structure is reported).

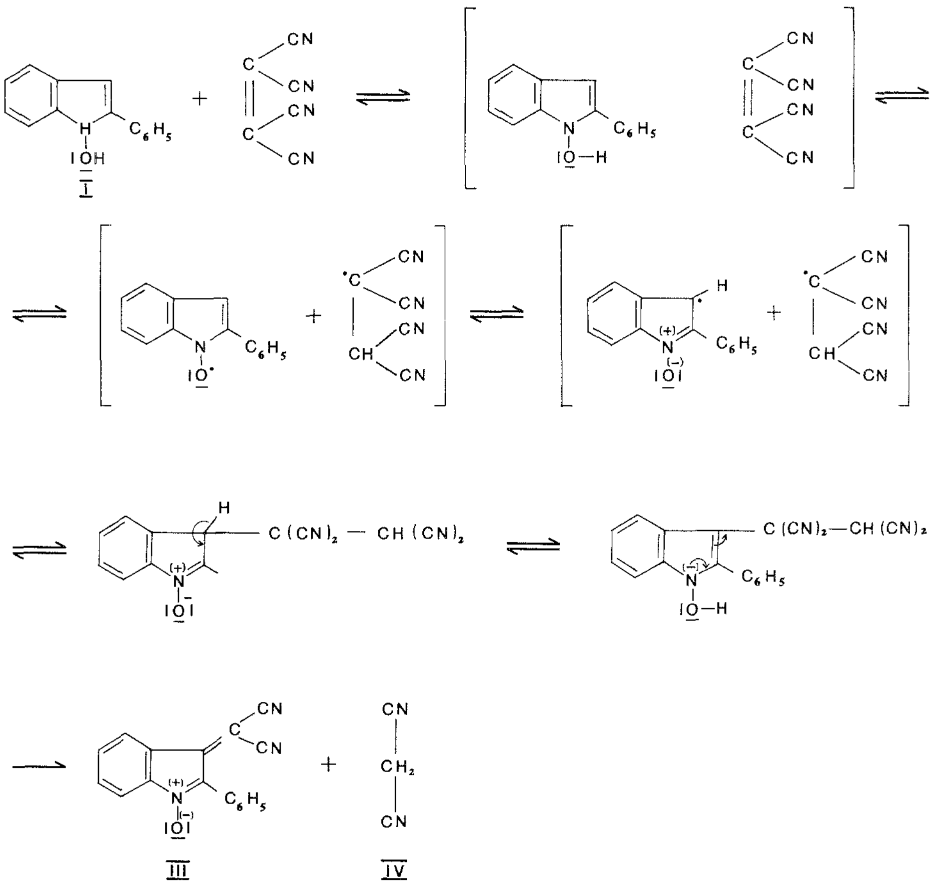
The malononitrile IV was detected and characterized with TLC and HPLC measurements.

Compound III may be considered the first example of synthesis of malononitrile derivatives "via" *TCNE*, the synthesis of yledene derivatives being related only to the reactions of malononitrile with carbonyl compounds [10, 11].

It can be pointed out that a similar mechanism is proposed by *Colonna* in the reaction of 1-hydroxy-2-phenylindole for a wide range of compounds [12].

The molecule as a whole is formed by three practically planar fragments (Fig. 1): the indole group, the aromatic ring and the dicyano group. The phenyl ring forms a dihedral angle of 78.1 (1)° with respect to the indole plane, which, in turn, is bent by 4.0 (1)° with respect to the

*Scheme 1*



planar dicyano group. Moreover the fused rings of the indole form a dihedral angle of 2.7 (1)° to each other.

All bond lengths and angles have values consistent with those of similar structures. The sum of the bond angles at nitrogen is 360° and the N—O bond makes an angle of only 0.7 (1)° with the plane of the indole ring and of 0.3 (1)° with that of the five-membered ring.

The N—O, N—C8 and C8—C9 lengths 1.256 (3), 1.336 (3), 1.467 (3) Å indicate than a certain degree of $\pi$ delocalisation is typically for these bonds.

No distances shorter than the normal *Van der Waals* interactions were found. The mass spectrum of III (Fig. 2) is in agreement with the X-ray

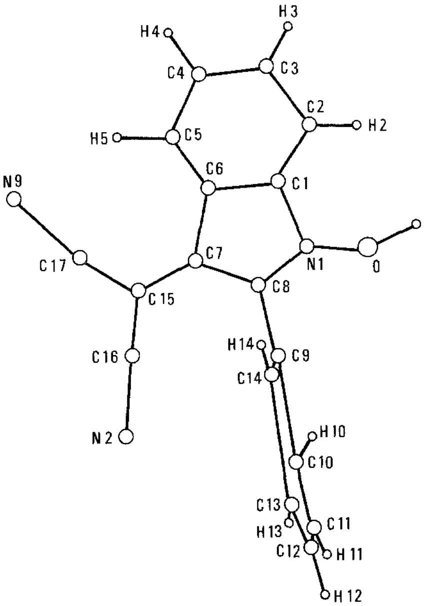


Fig. 1

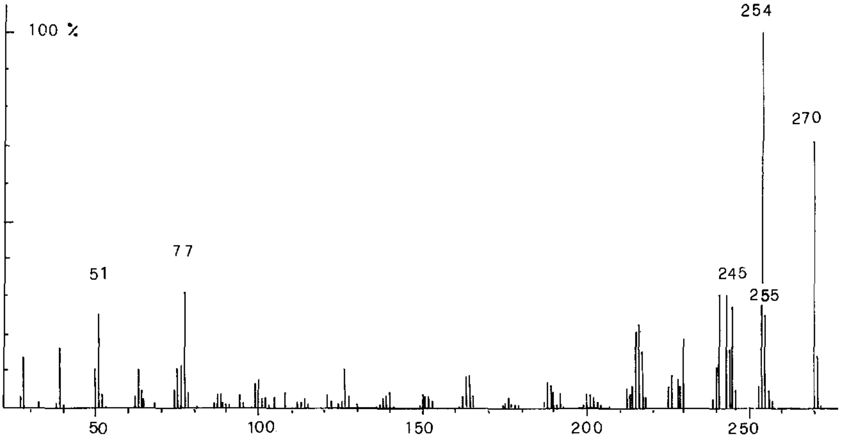


Fig. 2

structure (Fig. 1) (parent peak at 270.4 *m*/*z* with an intense peak at 253.6 *m*/*z* due to the loss of the oxygen atom in the N—O moiety).

The biindole II shows a better behaviour with organic acceptors (Table 1): as a matter of fact, compound II forms stable molecular complexes with *TCNE* and *DDQ* in presence of different solvents (benzene, acetonitrile, 1,2-dichloroethane); with *TCNE*, the CT bonding is quite

Table 1. *Characteristic infrared ($cm^{-1}$) and visible (nm) absorptions for 2,2′-diphenyl-$\Delta^{3,3'}$-bi-3 H-indole, II, complexes with TCNE and DDQ in different solvents*

| Solvent | *TCNE* | Biindole · *TCNE* | | | Biindole | Biindole · *DDQ* | | | | $\nu_{DDQ}$ | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $\nu_{CN}$ | $\nu_{NH}$ | $\nu_{CN}$ | $\lambda_{CT}$ | $\nu_{NH}$ | $\nu_{NH}$ | $\nu_{CN}$ | $\nu_{CO}$ | $\lambda_{CT}$ | $\nu_{CN}$ | $\nu_{CO}$ |
| Benzene | 2 200 | 3 435 | 2 210 | | 3 425 | | | | | | |
| | 2 252 | | 2 248 | | | | | | | | |
| Acetonitrile | 2 130 | 3 370 | 2 130 | 557 | 3 360 | 3 350 | 2 210 | 1 685 | 586 | 2 218 | 1 680 |
| | 2 180 | | 2 185 | 415 | | | 2 235 | 1 690 | 543 | 2 230 | 1 685 |
| Dichloroethane | 2 210 | 3 450 | 2 218 | 565 | 3 440 | 3 450 | 2 205 | 1 684 | 594 | 2 217 | 1 680 |
| | 2 240 | | 2 243 | 535 | | | 2 235 | 1 695 | | 2 230 | 1 690 |

stable to allow the determination of complexation thermodynamic parameters (Tables 2, 3). Any attempt to isolate the CTC in the solid state failed, recovering only unchanged starting materials.

The green coloured solution of *DDQ* and II [*Me*CN ($\lambda = 586$, 543 nm), dichloroethane ($\lambda = 594$ nm)] is not very stable: the band of the oxidation product, 2,2′-dimethyl-$\Delta^{3,3'}$-bi-3*H*-indole-1,1′-dioxide, overcomes in short time the disappearing CT absorption.

The interaction between the biindole II and *TCNE* or *DDQ* are characterized by an intense colour that may be due either to $\pi$ and to n orbital contributions.

In the case of the complex between the biindole II and *TCNE*, the shifts of the donor NH band and of the CN bands of the acceptor denote the occurrence of an interaction characterized by a n-$\pi^*$ transition with transfer of a n-electron from the heterocyclic nitrogen atom to the lowest vacant $\pi$-level of the CN group of *TCNE*.

On the other hand the small amounts of the shifts indicate that the n-$\pi^*$ energy is low enough to overcome the $\pi$-$\pi^*$ polarizing influence that, anyway, must be also present due to the small values of the shifts (as supported also by literature reports) [3, 5, 13–15].

A mixed participation $\pi$-$\pi^*$ may be hypothesized also for the complexes between the biindole II and *DDQ*. The nature of the shifts for the NH band of the donor and for the CN bands of the acceptor reveals a controversial behaviour: (i) the NH stretching for the complex in *Me*CN is shifted to higher wavenumbers while in dichloroethane falls to lower wavenumbers; (ii) the two bands of CN stretching vibrations, in both solvents, are shifted toward opposite directions: in both cases the ratio between the intensities of the two bands in the complex is reversed with respect to starting *DDQ*. A linear increase of wavenumbers of CO

Table 2. *Data for determination of K for 2,2'-diphenyl-$\Delta^{3,3'}$-bi-3H-indole-tetracyanoethylene in dichloroethane*

| $[D] \times 100$ | $d$ at 15 °C | Standard deviation × $10^3$ | $d$ at 25 °C | Standard deviation × $10^3$ | $d$ at 35 °C | Standard deviation × $10^3$ |
|---|---|---|---|---|---|---|
| 2.307 | 0.1675 | 5.15 | 0.1389 | 4.75 | 0.1146 | 3.59 |
| 2.873 | 0.2052 | 2.82 | 0.1693 | 2.59 | 0.1403 | 2.77 |
| 3.501 | 0.2557 | 4.59 | 0.2127 | 4.21 | 0.1749 | 3.47 |
| 4.052 | 0.2842 | 2.80 | 0.2378 | 2.46 | 0.2001 | 2.41 |
| 4.489 | 0.2985 | 5.02 | 0.2485 | 4.30 | 0.2054 | 3.80 |

Table 3. *Values of equilibrium constant (K), extinction coefficient (ε), enthalpy (ΔH), free energy (ΔG), and entropy (ΔS) of formation of the complex between biindole II and tetracyanoethylene in dichloroethane*

| $T$(°K) | Intercept | Slope | Correlation coefficient | $K$ | $\varepsilon$ | $-\Delta S$ kcal/mol | $-\Delta H$ kcal/mol | $\Delta S$ cal/mol |
|---|---|---|---|---|---|---|---|---|
| 288 | $1.0373 \pm 5.2809 \cdot 10^{-1}$ | $3.4757 \cdot 10^{-1} \pm 1.6680 \cdot 10^{-2}$ | 0.9966 | $2.984 \pm 1.663$ | $964 \pm 490$ | 1.608 | 2.687 | 3.683 |
| 298 | $1.0809 \pm 6.8054 \cdot 10^{-1}$ | $4.2399 \cdot 10^{-1} \pm 2.1496 \cdot 10^{-2}$ | 0.9962 | $2.549 \pm 1.734$ | $925 \pm 582$ | 1.403 | 3.378 | 6.518 |
| 308 | $1.0985 \pm 3.5135 \cdot 10^{-1}$ | $5.1865 \cdot 10^{-1} \pm 2.6891 \cdot 10^{-2}$ | 0.9960 | $2.118 \pm 1.751$ | $910 \pm 705$ | | | |

stretching vibration modes can be noted going from *DDQ* to the complex with retention of the appearance.

The latter fact denotes a negligible participation of the CO group in the complex formation: as a matter of fact, consistent higher wavenumbers values are typical for a CT bonding participation of the CO group as a consequence of an increased polarization of this group in the molecular complex [13].

Finally it appears, from the cited infrared reports, that the influence of the solvent on the formation of a CT bonding and on the stability of the complex is negligible.

The absence of CT interactions between the biindole II and *DDQ*, using benzene as a solvent, is due to the low solubility of starting materials in this solvent.

CT bonding between II and *TCNQ* is characterized by weak multiple bands; some authors [16] exclude the presence of CTC interactions if the electronic spectra show multiple bands, but we agree with *Foster* [17], supporting in this case the presence of contact CT bondings.

A 2 : 1 complex between *TCNE* and II may be supposed because the biindole II has the same steric and electronic situation like a simple indole with the possibility that two molecules of *TCNE* can be located over the indole moietes of compound II (Fig. 3).

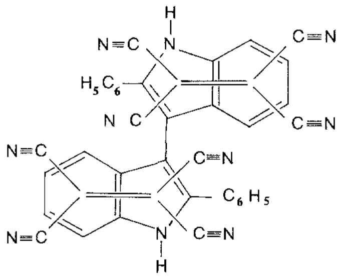


Fig. 3

## Experimental

1-Hydroxy-2-phenylindole I and 2,2′-diphenyl-$\Delta^{3,3'}$-bi-3*H*-indole II were prepared according the literature [18]. Tetracyanoethylene, 2,3-dicyano-5,6-dichloro-1,4-benzoquinone, tetracyanoquinodimethane, chloranil and piromellitic anhydride were Fluka pure reagents and were purified by appropriate solvents before using. All solvents were Fluka RP-ACS grade and were dried and purified according to *Vogel* [19].

UV and IR spectra were registred on Perkin-Elmer 554 and Perkin-Elmer 298 instruments. The mass spectra were performed on a Varian MAT 311 A. Melting points are uncorrected and were determined with an electrothermal melting point apparatus.

### *Reaction of 1-Hydroxy-2-phenylindole I with TCNE*

Equimolar amounts of I and *TCNE* were dissolved in anhydrous dichloroethane and mixed at room temperature under nitrogen. The blue color of the solution rapidly turned red. After 24 h the red precipitate was collected and purified from benzene-hexane. The compound was 1-oxy-2-phenyl-3-(dicyanomethylidene)indole III, m.p. 204 °C. The yield was almost quantitative. Analysis % for $C_{16}H_9N_8O$, calcd. (found): C, 75.26 (75.07); H, 3.34 (3.33); N, 15.49 (15.23).

### *Reaction of the Compound I with DDQ*

Equimolar amounts of I and *DDQ* were dissolved in anhydrous dichloroethane and mixed at room temperature under nitrogen. The colour of the solution was black. After standing 24 h the precipitate was collected and washed with benzene: m.p. over 300 °C. The solvent was evaporated and after purification with benzene 1,1′-dioxide-2,2′-diphenyl-$\Delta^{3,3'}$-bi-3*H*-indole and the corresponding hydroquinone were recovered as evidenced by mixed m.p. with reference compounds.

### *Reaction of the Compound I with TCNQ*

Equimolar amounts of I and *TCNQ* were dissolved in anhydrous dichloroethane and mixed at room temperature under nitrogen. The green colour of the solution was unchanged after 24 h. Working up the solution, starting materials were recovered.

### *Reaction of the Compound I with Chloranil*

Equimolar amounts of I and *CA* were dissolved in anhydrous dichloroethane and mixed at room temperature under nitrogen. After standing 24 h, the brown-red precipitate was collected, washed with cold dichloroethane, obtaining 2,3,5,6-tetrachlorohydroquinone (70%) as evidenced by mixed melting point (reference compound) and by infrared determinations. The mother liquors were taken into dryness and the residue purified with benzene. Firstly precipitated the starting chloranil (15%) and then 2,2′-diphenyl-$\Delta^{3,3'}$-bi-3*H*-indole-1,1-dioxide (40%) with traces of 1,1′-dihydroxide-2,2′-diphenyl-$\Delta^{3,3'}$-bi-3*H*-indole.

### *Reaction of Compound I with Piromellitic Anhydride*

Equimolar amounts of I and *PA*, dissolved in anhydrous dichloroethane, were mixed at room temperature under nitrogen. After standing 24 h the solution retained the initial colour. The white precipitate was starting piromellitic anhydride. From the solution the remaining amounts of starting materials were recovered.

### *X-Ray Determination of Compound III*

Cell dimension and symmetry information were obtained by least-squares by use of 29 $(\vartheta, \chi, \varphi)_{hkl}$ measurements taken on a Siemens AED single crystal diffractometer, employing a prismatic specimen of about $0.6 \times 0.7 \times 0.11$ mm. Crystal data—$C_{17}H_9N_3O$, $M = 271.28$, monoclinic, space group $Pz_1/c$, $a = 10.913(2)$, $b = 17.712(2)$, $c = 7.004(3)$ Å, $\beta = 99.85(2)°$, $Z = 4$, $V = 1\,341.5$ Å$^3$, $D_c = 1.34$ g cm$^{-3}$, CuK$\alpha$ radiation, $\lambda = 1.54178$ Å, $\mu = 6.64$ cm$^{-1}$, $F(000) = 560$.

Intensity data were collected in the range $3 < \vartheta < 70°$ with Ni-filtered CuK$\alpha$ radiation employing a modified version [20] of the *Lehmann* and *Larsen* procedure [21]. Throughout the data collection a standard reflection was checked every fifty measurements and indicated no decomposition or misalignement of the specimen.

A total of 2 874 reflections was measured (2 574 unique, $R_{\text{int}} = 0.021$) and 1 894 were considered observed having $I > 2\sigma(I)$. The data were corrected for *Lorentz* and polarisation effects, not for absorption.

The structure was solved by direct methods with SHELX [22]. The isotropic refinement of all heavy atoms gave $R = 0.16$. Several cycles of full-matrix anisotropic least-squares reduced $R$ to 0.09. A difference synthesis computed at this stage revealed peaks near to the positions were the hydrogens were expected to occur. Any attempt to localize hydrogen connected to the oxygen atom failed. Further refinement of all atoms (the hydrogens isotropically) gave the final values of $R = 0.053$, $R_w = 0.066$ with $w = 1.0/(\sigma^2 F + 0.13044 F^2)$ for 1 876 reflections (18 were omitted in the final cycles because probably affected by extinction). The $(\Delta/\sigma)_{\max}$ was 0.19 and the higher peak in the final difference synthesis was 0.17 e Å$^{-3}$. The atomic scattering factors used for the analysis were those of SHELX.

Table 4. *Atomic fractional coordinates* ($\times 10^4$) *and U-EQ* ($\times 10^4 - A^2$)

| | X/A | Y/B | Z/C | U/EQ |
|---|---|---|---|---|
| O1 | 8 212(2) | 2 919(1) | − 4 052(3) | 752(7) |
| N1 | 8 312(2) | 3 271(1) | − 2 470(3) | 514(6) |
| N2 | 4 913(2) | 4 206(1) | 1 260(3) | 644(7) |
| N3 | 8 349(2) | 4 776(2) | 4 693(4) | 875(10) |
| C 1 | 9 512(2) | 3 478(1) | − 1 356(3) | 521(8) |
| C 2 | 10 656(2) | 3 347(2) | − 1 834(4) | 705(10) |
| C 3 | 11 670(2) | 3 642(2) | − 545(5) | 732(10) |
| C 4 | 11 494(2) | 4 029(2) | 1 073(4) | 671(9) |
| C 5 | 10 308(2) | 4 143(1) | 1 540(4) | 592(9) |
| C 6 | 9 294(2) | 3 862(1) | 258(3) | 473(7) |
| C 7 | 7 931(2) | 3 884(1) | 168(3) | 432(6) |
| C 8 | 7 387(2) | 3 521(1) | − 1 615(3) | 440(7) |
| C 9 | 6 075(2) | 3 416(1) | − 2 472(3) | 433(6) |
| C 10 | 5 505(2) | 2 727(1) | − 2 370(3) | 526(7) |
| C 11 | 4 245(2) | 2 646(2) | − 3 181(4) | 601(8) |
| C 12 | 3 603(2) | 3 237(2) | − 4 104(4) | 626(9) |
| C 13 | 4 181(3) | 3 928(2) | − 4 242(4) | 656(9) |
| C 14 | 5 414(2) | 4 018(1) | − 3 411(4) | 584(8) |
| C 15 | 7 297(2) | 4 179(1) | 1 486(3) | 478(6) |
| C 16 | 5 957(2) | 4 183(1) | 1 299(3) | 504(7) |
| C 17 | 7 912(2) | 4 510(1) | 3 269(3) | 590(7) |

Table 5. *Bond distances (Å) and angles (°) with estimated deviations in parentheses*

| | | | |
|---|---|---|---|
| O1—N1 | 1.265(3) | C7—C8 | 1.445(3) |
| N1—C1 | 1.454(3) | C7—C15 | 1.354(3) |
| N1—C8 | 1.336(3) | C8—C9 | 1.467(3) |
| N2—C16 | 1.136(3) | C9—C10 | 1.377(3) |
| N3—C17 | 1.137(3) | C9—C14 | 1.390(3) |
| C1—C2 | 1.366(3) | C10—C11 | 1.403(3) |
| C1—C6 | 1.380(3) | C11—C12 | 1.361(4) |
| C2—C3 | 1.406(4) | C12—C13 | 1.388(5) |
| C3—C4 | 1.371(5) | C13—C14 | 1.382(4) |
| C4—C5 | 1.403(3) | C15—C16 | 1.466(3) |
| C5—C6 | 1.394(3) | C15—C17 | 1.443(3) |
| C6—C7 | 1.478(3) | | |

| | | | |
|---|---|---|---|
| O1—N1—C8 | 127.0(2) | N1—C8—C7 | 108.0(2) |
| O1—N1—C1 | 122.2(2) | C7—C8—C9 | 129.9(1) |
| C1—N1—C8 | 110.7(1) | N1—C8—C9 | 122.1(1) |
| N1—C1—C6 | 107.6(2) | C8—C9—C14 | 119.3(1) |
| N1—C1—C2 | 126.9(2) | C8—C9—C10 | 120.6(1) |
| C2—C1—C6 | 125.4(2) | C10—C9—C14 | 120.2(2) |
| C1—C2—C3 | 115.4(2) | C9—C10—C11 | 119.3(2) |
| C2—C3—C4 | 121.0(2) | C10—C11—C12 | 120.3(2) |
| C3—C4—C5 | 122.2(2) | C11—C12—C13 | 120.6(2) |
| C4—C5—C6 | 117.2(2) | C12—C13—C14 | 119.6(2) |
| C1—C6—C5 | 118.7(2) | C9—C14—C13 | 120.1(2) |
| C5—C6—C7 | 134.3(2) | C7—C15—C17 | 122.5(2) |
| C1—C6—C7 | 107.0(1) | C7—C15—C16 | 124.5(2) |
| C6—C7—C15 | 127.5(2) | C16—C15—C17 | 112.9(1) |
| C6—C7—C8 | 106.6(1) | N2—C16—C15 | 175.8(2) |
| C8—C7—C15 | 125.8(2) | N3—C17—C15 | 177.1(2) |

The final positional parameters are compiled in Table 4 while Table 5 reports a list of bond distances and angles of the molecule which is illustrated in Fig. 1. All calculations were performed on a GOULD SEL 32/77 computer*.

### *UV-Visible Spectrophotometric Measurements*

Measurements of the interactions between compound I and acceptors were performed in anhydrous dichloroethane using a ratio [*D*]/[*A*] = 1/1.

Measurements of the interactions between compound II and acceptors were performed in anhydrous benzene, acetonytrile and dichloroethane. The ratio [*D*]/[*A*] was 10/1. The values for CT bonding are reported in Table 1. Only very small interactions between II and *TCNQ* were detected while no interactions were evidenced between compound II and *PA*.

---

* List of structure factors, of anisotropic thermal parameters, and of H coordinates with bond distances involving hydrogens may be obtained upon request from the authors.

For the UV-visible spectrophotometric measurements on the complex of biindole II with *TCNE* in dichloroethane stock dichloroethane solutions of compound II and *TCNE*, in a ratio ranging from $[D]/[A] = 1$ to the maximum possible value $[D]/[A] = 16.56$ (owing to the low solubility of the donor) were used; $A_0 = 2.71 \cdot 10^{-3}\,M$. Five samples were made, mixing 1.0, 1.5, 2.0, 2.5, 3.0 $cm^3$ of biindole solution with 1 $cm^3$ of *TCNE* solution. Each mixture was prepared in five separate samples leading to a total of 25 samples, on which the determination of the absorbance ($d$) at the $\lambda$ max was performed at 15.0, 25.0 and 35.0 °C. Reproducibility tests were performed on 22 samples ($[D]/[A] = 8.082$) with $d = 0.3065 \pm 5.4 \cdot 10^{-3}$. A consistent part of the measurements (86.4%) was in the range of the ± 2%. A linear correlation was obtained by plotting $1/[D_0]$ versus $[A_0] \cdot 10^3/d$ (Table 2); the calculated thermodynamic parameters are listed in Table 3.

## References

[1] *Szent Györgyi A, Isenberg I, Metaglin J* (1961) Proc Natl Acad Sci USA 47: 1089

[2] *Foster R, Hanson P* (1965) Tetrahedron 21: 255

[3] *Sung MT, Parker JA* (1972) Proc Natl Acad Sci USA 69: 1196

[4] *Foster R, Fyfe CA* (1966) J Chem Soc B: 926

[5] *Cipiciani A, Sartini S, Savelli G* (1979) J Chem Soc Faraday I: 497

[6] *Foster R, Hanson P* (1964) J Chem Soc Faraday II 60: 2189

[7] *Sabourault B, Bourdais J* (1972) CR Acad Sci Paris 274: 813

[8] *Colonna M, Bruni P* (1969) Gazz Chim Ital 99: 885

[9] *Tosi G, Battistoni P, Bruni P, Cardellini L, Bocelli G* (1983) Gazz Chim Ital 113: 153

[10] *Freuman F* (1980) Chem Rev 80: 329

[11] *Fatiadi AJ* (1983) Preparation and systhetic applications of cyano compounds. In: *Patai S* (ed) The chemistry of triple bonded groups. Suppl C. Wiley, p 1057

[12] *Colonna M, Bruni P* (1967) Gazz Chim Ital 97: 1569

[13] *Hindawey AM, Nassar AMG, Issa RM* (1977) Acta Acad Sci Hungaricae 92(3): 263

[14] *Issa RM, Abel-Ghani NT, El-Ansary AL, Shoukry AF* (1981) Rev Roumanie Chim 26: 667

[15] *Issa RM, El-Essawey MM* (1973) J Phys Chem (Leipzig) 253: 96; *Knoss RD, Fassel VA* (1960) J Am Chem Soc 79: 316

[16] *Spatz AM, Shackle DR* (1976) Tappi 59: 128

[17] *Foster R* (1979) Molecular association, vol 2. Academic Press, London

[18] *Colonna M, Monti A* (1962) Gazz Chim Ital 92: 1401

[19] *Vogel AI* (1957) A textbook of practical organic chemistry, 3rd edn. Longmans, London

[20] *Lehmann HS, Larsen FK* (1974) Acta Cryst A 30: 580

[21] *Belletti D, Ugozzoli F, Cantoni A, Pasquinelli G* (1979) Gestione on line di diffrattometro a cristallo singolo Siemens AED con sistema general automation Jumbo 220. Centro di Studio per la Strutturistica Diffrattometrica del CNR, Parma. Internal Reports 1–3/79

[22] *Sheldrick GM*, SHELX (1976) Program for crystal structure determination. Univ of Cambridge, England