

## Spectroscopic Studies of Charge-Transfer Complexation of Iodine with a New Benzo-Substituted Macrocyclic Diamide in Chloroform, Dichloromethane and Their 1:1 Mixture

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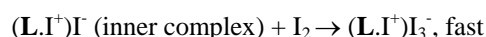
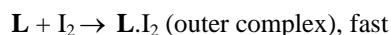
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Charge-transfer complexation of iodine with a new benzo-substituted macrocyclic diamide 5,6,7,8,9,10-hexahydro-2H-1,13,4,7,10-benzodioxatriazacyclopentadecine-3,11(4H,12H)-dione (**L**) with iodine was studied spectrophotometrically in chloroform, dichloromethane and their 1:1 (v/v) mixture. The observed time dependence of the charge-transfer band and subsequent formation of I<sub>3</sub><sup>-</sup> ion are related to the slow formation of the initially formed 1:1 **L**.I<sub>2</sub> outer complex to an inner electron donor-acceptor (EDA) complex, followed by fast reaction of the inner complex with iodine to form a triiodide ion, as follows:



The pseudo-first-order rate constants for the transformation process were evaluated in different solvent systems. The stability constants of the resulting EDAr complexes were also evaluated and the solvent effect on their stability is discussed. The resulting complexes were isolated and characterized by FTIR and <sup>1</sup>H NMR spectroscopy.

**Keywords:** Charge-transfer complex, Iodine, Macrocyclic diamide, Kinetics, Formation constant, Spectroscopy

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### INTRODUCTION

Crown ethers are macrocyclic polyethers [1] well known for their ability to form stable and selective inclusion complexes with many metal ions, including alkali and alkaline earth ions [2,3]. The stability of the resulting complexes

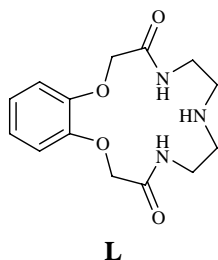
appears to be related to the macrocycles' cavity sizes; it is at maximum when the cavity diameter matches the ionic size. The substitution of some oxygen atoms of the crown ethers by -NH-, and -S- groups has found to alter their complexing ability towards different metal ions [4]. During the past two decades, a growing interest has been focused on the complexing ability of these macrocyclic ligands as electron-pair molecules towards neutral and electron accepting

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molecules [5-16] and, especially, molecular iodine [17-28]. Interest in such molecular complexes is largely due to their possible applications in areas like organic semiconduction, separation processes, catalysis of chemical reactions, biomimetic receptors and conversion of chemical reactions into electronic or optical signals [5,29-31].

In continuation of our previous studies made on the charge transfer complexes of iodine with macrocyclic crown ethers and their aza- and thia-derivatives in various solvents [20-26], in this paper we wish to report on the results of kinetic and spectral studies concerning the complexation of iodine with a recently synthesized benzo-substituted macrocyclic diamide 5,6,7,8,9,10-hexahydro-2H-,1,13,4,7,10-benzodioxatriaza-cyclopentadecine-3,11(4H,12H)-dione (**L**) in chloroform, dichloromethane and their 1:1 (v/v) mixture.



## EXPERIMENTAL

### Reagents and Apparatus

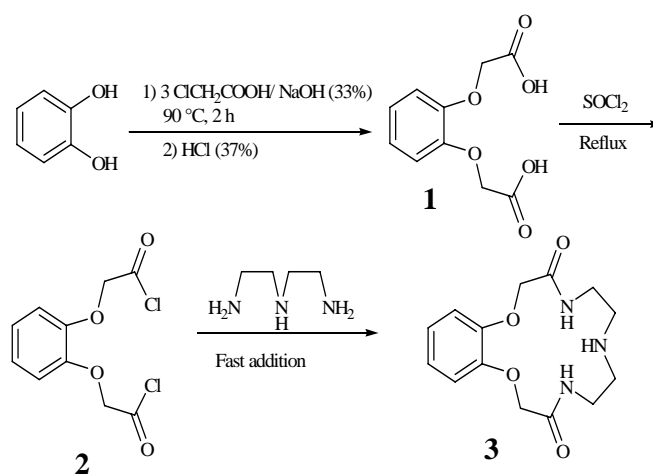
Resublimed iodine of analytical grade (Merck) was used as received. Reagent grade chloroform and dichloromethane (from Fluka) were used without any further purification. The electronic absorption spectra of the **L**, iodine and their charge transfer complex in chloroform, dichloromethane and 1:1 (v/v) mixture of the two solvents were recorded in the region 600-200 nm using a Shimadzu double beam spectrometer model 2100 using a quartz cell of 1-cm path length. The proton NMR spectra were recorded on a Bruker 500 MHz FT NMR spectrometer operating at a field of 11.74T using CDCl<sub>3</sub> (Merck) as a pure deuterated solvent with tetramethylsilan (TMS) as an internal standard. The mid infrared spectra (4000-400 cm<sup>-1</sup>) were recorded from KBr discs using a Nicolet FT-IR model 560 spectrometer. The charge transfer complexes in the crystalline form were prepared by dissolving appropriate amounts of the crown ether and iodine in chloroform, as

follows. To a solution of **L** (0.0293 g, 0.10 mmol) in CHCl<sub>3</sub> (20 ml) was added a solution of excess iodine (0.152 g, 0.60 mmol) in CHCl<sub>3</sub> (50 ml) and stirred for 18 h. The dark brown precipitates formed were filtered off, washed two times with minimum amounts of CHCl<sub>3</sub> and dried under vacuum over P<sub>2</sub>O<sub>5</sub>.

### Synthesis Macroyclic Diamide **L**

The macrocyclic diamide **L** was synthesized according to Scheme 1:

**Phenylenedioxydiacetic acid 1.** To a mixture of catechol (1 g, 0.1 mol), and chloroacetic acid (28.4 g, 0.3 mol) at 90 °C on a water-bath was added a solution of NaOH 33%(w/v) (48 g, in 97.5 ml water) drop wise. The mixture was stirred at 90 °C for 2 h, and after cooling to room temperature (by keeping the solution in an ice bath) to the solution was added concentrated HCl drop wise, under stirring, to get an acidic solution. The mixture was allowed to warm to room temperature and the solution was filtered and the resulting white precipitate washed with cool water. Crystallization of the residue from hot water afforded the solid compound **1** (20.5 g, 91% yield); m.p.: 178-179 °C (Lit: 177-178 °C [31]); IR (KBr): 662 (w), 753 (m), 781 (w), 822 (w), 858 (w), 1062 (m), 1193 (s), 1209 (s), 1232 (s), 1402 (w), 1433 (m), 1508 (s), 1598 (w), 740 (s), 2335 (w), 2631 (w), 2850 (m) cm<sup>-1</sup>; <sup>1</sup>H NMR (DMSO-d<sub>6</sub>): δ = 4.69 (s, 4H), 6.88-6.90 (m, 4H) ppm; <sup>13</sup>C NMR (DMSO-d<sub>6</sub>): δ = 65.5, 114.4, 121.8, 147.7, 170.8



Scheme 1

ppm; Mass  $m/z$  (%): 228 ( $M^+ + 2$ , 107), 227 ( $M^+ + 1$ , 10.1), 226 ( $M^+$ , 87.0), 181 (8.9), 168 (14.3), 151 (27.9), 135 (15.2), 123 (100), 107 (38.7), 81 (52.7), 64 (43.4), 43 (25.7); Anal. Calcd. for  $C_{10}H_{10}O_6$  (226.18): C = 53.10%; H = 4.46%. Found: C = 53.01%; H = 4.40%.

**1,2-Phenylenedioxydiacetyl chloride 2.** 1,2-Phenylenedioxy diacetic acid **1** (5.65 g, 0.025 mol) was heated in 50 ml thionyl chloride for 4 h at 50-60 °C. The solvent was slowly evaporated at low temperature to get compound **2** as a white cream solid (5.4 g, 85% yield); m.p.: 48-49 °C; IR (KBr): 747 (s), 770 (m), 812 (m), 945 (s), 1138 (s), 1195 (s), 1525 (s), 1421 (m), 1501 (m), 1595 (m), 1805 (s), 2927 (w), 2977 (w), 3075 (w)  $cm^{-1}$ ;  $^1H$  NMR ( $CDCl_3$ )  $\delta$  = 4.85 (s, 4H), 6.8 (s, 4H) ppm;  $^{13}C$  NMR ( $CDCl_3$ ):  $\delta$  = 73.6, 116.3, 123.5, 146.8, 170.6 ppm; Anal. Calcd. for  $C_{10}H_8Cl_2O_4$  (263.07): C = 45.66%, H = 3.07%; Found: C = 45.75%; H = 2.95%.

**5,6,7,8,9,10-Hexahydro-2H-1,13,4,7,10-benzodioxatriazacyclopentadecine-3,11 (4H, 12H)-dione 3 (L).** A solution of diethylenetriamine (0.2 mmol) in dry ethyl acetate (50 ml) was added quickly to a vigorously stirring solution of 2-phenylenedioxydiacetyl chloride **2** (2 mmol) in dry ethyl acetate (50 ml) at room temperature. The mixture was stirred for a further 20 min and then was washed with bicarbonate solution (2  $\times$  50 ml) and water (2  $\times$  50 ml). The organic layer was dried over  $MgSO_4$  and the solvent was evaporated to give a solid product. The crude product was purified by column chromatography using petroleum ether/ethyl acetate as eluent (45% yield); decomposed at 233 °C [32]; IR (KBr): 750 (s), 1040 (s), 1120 (s), 1210 (s), 1250 (vs.), 1320 (s), 1420 (s), 1450 (s), 1500 (s), 1540 (s), 1590 (w), 1640 (vs.), 2850 (s), 2950 (s), 3030 (w), 3380 (s), 3430 (br)  $cm^{-1}$ ;  $^1H$  NMR ( $CDCl_3$ ):  $\delta$  = 1.75 (s, 1H), 2.94 (t, 4H), 3.50 (t, 4H), 4.48 (s, 4H), 6.84-7.00 (m, 4H), 7.81 (s, 2H) ppm;  $^{13}C$  NMR ( $CDCl_3$ ):  $\delta$  = 38.5, 47.8, 67.2, 112.6, 122.4, 146.4, 167.6 ppm; Mass  $m/z$  (%): 295 ( $M^+ + 2$ , 0.9), 294 ( $M^+ + 1$ , 2.9), 293 ( $M^+$ , 5.4), 225 (50.6), 180 (10.5), 167 (17.3), 150 (14.9), 121 (17.7), 85 (33.6), 69 (78.7), 56 (78.4), 43 (100). Anal. Calcd. for  $C_{14}H_{19}N_3O_4$  (293.32): C = 57.33%; H = 6.53%; Found: C = 57.39%; H = 6.45%.

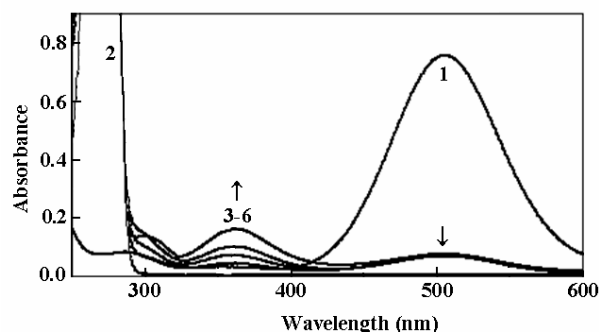
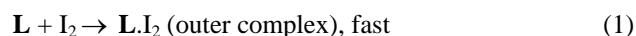
## RESULTS AND DISCUSSION

The electronic absorption spectra of **L** in the presence of

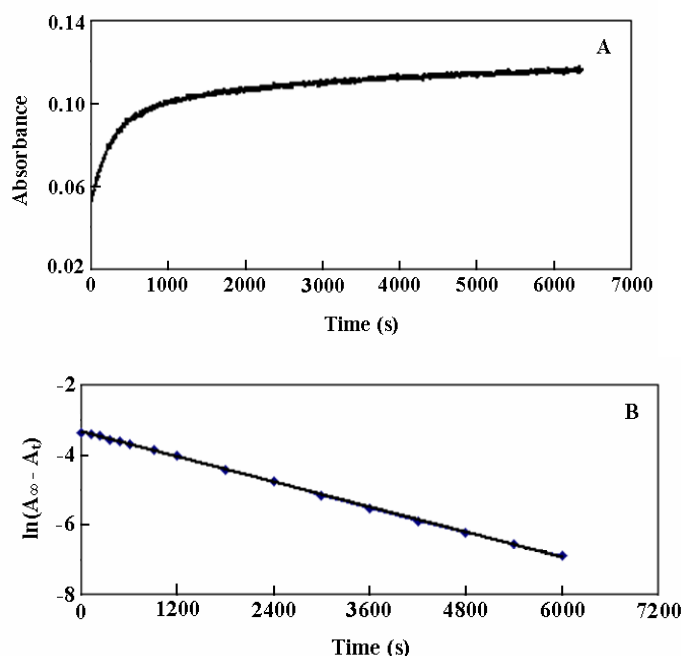
an excess amount of iodine (*i.e.*,  $[L]/[I_2] = 10$ ) in chloroform, dichloromethane and their 1:1 mixture were obtained as a function of time at 25 °C and sample spectra are shown in Fig. 1. As it is seen from Fig. 1, while none of the reactants show any considerable absorption in the 290-450 nm range, the spectrum obtained immediately after mixing iodine with the macrocyclic diamide resulted in two strong absorptions in the 290-450 nm region. The first absorption, located at about 296 nm, corresponds to a charge-transfer band and the second at about 364 nm is the blue-shifted iodine band [32].

From Fig. 1, it is obvious that the spectra recorded for the complexes between **L** and iodine are time dependent. As seen, the intensity of the iodine band at 510 nm decreased significantly with the elapse of time, while an intensification of new bands at 296 and 364 nm was observed. It should be noted that the absorptions at 296 and 364 nm are well-known characteristic bands for the formation of triiodide ion,  $I_3^-$ , in the process of complex formation between iodine and different electron-pair donor ligands [17-28,33,34].

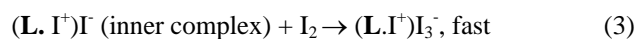
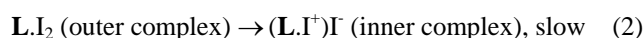
The observed time dependence of the charge-transfer band and the subsequent formation of the  $I_3^-$  ion in solution are most probably due to a transformation of the initially formed outer complex into an inner CT complex followed by a fast reaction of the resulting inner complex with iodine to form a triiodide ion, as follows [26-28,35,36]:



**Fig. 1.** Absorption spectra of  $7.4 \times 10^{-4}$  M iodine (1),  $1.36 \times 10^{-3}$  M ligand (2) and their mixture  $I_2$  ( $6.72 \times 10^{-5}$  M) + **L** ( $1.21 \times 10^{-3}$  M) in chloroform just after mixing (3) and after 30 min (4), 60 min (5), 14 h (6).



**Fig. 2.** Absorbance-time plot at 25 °C for a  $7.4 \times 10^{-4}$  M iodine solution in chloroform in the presence of excess ligand at  $[\text{ligand}]/[\text{I}_2] = 20$  (A) and the corresponding  $\ln(A_\infty - A_t)$  vs. time plot (B).



The kinetics of transformation of the outer CT complex into the inner CT complex was followed by the time dependence of the triiodide absorption band at 364 nm, in order to obtain more information about the kinetics and mechanism of the interaction of **L** with iodine. The absorbance of solutions containing reactants at a  $[\text{L}]/[\text{I}_2]$  molar ratio of 20

in all three solvent systems was monitored as a function of time at 364 nm, and a sample absorbance-time plot in chloroform is shown in Fig. 2A. As is obvious, the transformation of the outer complex into the corresponding inner complex (Eq. 2) found to follow pseudo-first-order kinetics.

The rate constant  $k$  was then evaluated based on a simple first-order kinetic model using the following equation:

$$A_t = A_\infty + (A_0 - A_t) \exp(-kt) \quad (4)$$

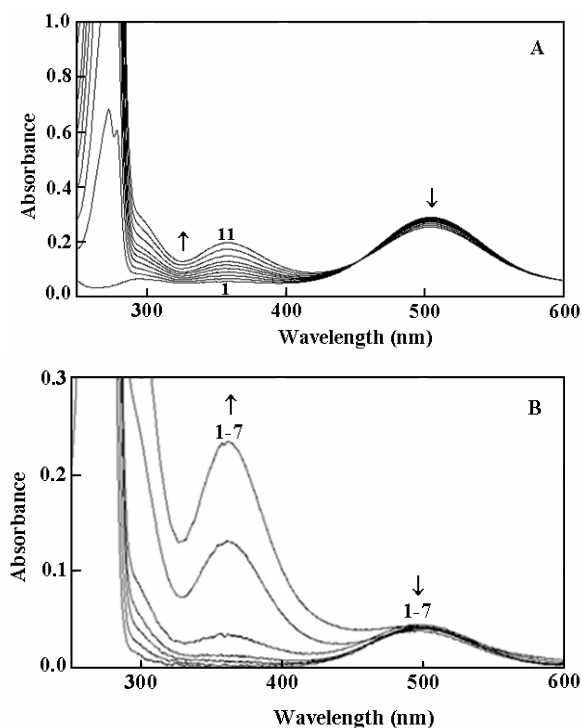
where  $A_0$ ,  $A_\infty$  and  $A_t$  are the measured at initial and infinite times and time  $t$ , respectively. According to Eq. 4, the  $k$  values can be evaluated from the slope of the resulting linear  $\ln(A_\infty - A_t)$ -time plots (see Fig. 2B). The rate constants thus calculated for all three solvent systems used are given in Table 1. Interestingly, in support of the proposed three-step mechanism, the rate constant increases with increasing dielectric constant of the solvent system. In fact, for the slow rate determining step (Eq. 2), the resulting charged transition state is expected to be more stabilized in more polar solvents of higher relative permittivity, which in turn results in decreased activation energy and increased rate of transformation of the outer complex to the inner one [37].

In the next step, the electronic absorption spectra of iodine ( $3.15 \times 10^{-4}$  M, Fig. 3A and  $3.15 \times 10^{-5}$  M, Fig. 3B) in the presence of increasing amount of **L** in chloroform, dichloromethane and their 1:1 mixture were recorded and typical results in dichloromethane are shown in Fig. 3. As seen, the resulting **L**-iodine complex has a rather sharp absorption at 292 nm and a weaker and broader absorption at 360 nm, which are associated with the formation of donor-acceptor molecular complexes between the macrocyclic diamide **L** and iodine in solution [17-28].

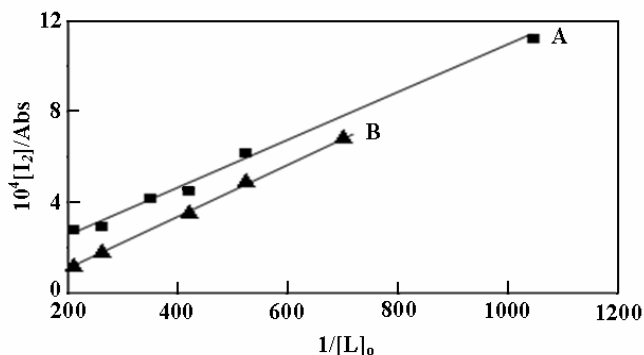
**Table 1.** Rate constants,  $k$ , Formation Constants,  $K_f$ , and Molar Absorptivities,  $\epsilon_{\text{CT}}$ , for the Formation of **L**- $\text{I}_2$  Complex in Different Solvents at 25 °C

Solvent	D	$\lambda_{\text{CT}}$ (nm)	$10^2 k$ ( $\text{min}^{-1}$ )	LogKf	$\epsilon_{\text{CT}}$
$\text{CHCl}_3$	4.8	364	$3.6 \pm 0.2$	$1.62 \pm 0.02$	$2327 \pm 20$
$\text{CH}_2\text{Cl}_2$	9.1	360	$3.9 \pm 0.2$	$2.02 \pm 0.03$	$829 \pm 67$
$\text{CHCl}_3 + \text{CH}_2\text{Cl}_2$	7.3	362	$3.7 \pm 0.1$	$1.79 \pm 0.04$	$1578 \pm 43$

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**Fig. 3.** (A) Absorption spectra of  $3.15 \times 10^{-4}$  M iodine in dichloromethane at 25 °C in the absence (1) and presence of **L** at  $[L]/[I_2]$  mole ratios of 0.1 (2), 0.2 (3), 0.3 (4), 0.4 (5), 0.5 (6), 0.6 (7), 0.7 (8), 0.8 (9), 0.9 (10) and 1.0 (11). (B) Absorbance spectra of  $3.15 \times 10^{-5}$  M iodine in chloroform at 25 °C in the absence (1) and presence of varying concentration of **L**: (2)  $4.76 \times 10^{-4}$  M, (3)  $9.52 \times 10^{-4}$  M, (4)  $1.43 \times 10^{-3}$  M, (5)  $1.90 \times 10^{-3}$  M, (6)  $2.38 \times 10^{-3}$  M and (7)  $3.81 \times 10^{-3}$  M.



**Fig. 4.** Benesi-Hildebrand plot for **L**- $I_2$  in chloroform (A) and in dichloromethane (B) at 25 °C.

Formation constants of the resulting 1:1 donor-acceptor complexes were determined by measuring the absorbance at 360 nm for a series of solutions with varying excess amounts ligand and constant iodine concentration in all three solvent systems used. The formation constants,  $K_f$ , and molar absorptivity,  $\epsilon_{CT}$ , of the complex were evaluated using Benesi-Hildebrand equation [38]:

$$[I_2]_0 / \text{Abs} = (1/K_f \epsilon_{CT})(1/[L]_0) + 1/\epsilon_{CT} \quad (6)$$

where  $[I_2]_0$  and  $[L]_0$  are the initial concentrations of reactants. Sample linear plots of  $[I_2]_0 / \text{Abs}$  vs.  $1/[L]_0$  are shown in Fig. 4, from the intercept and slope of which the  $\epsilon_{CT}$  and  $K_f$  values can be evaluated. Such a fair linear relationship in the resulting Benesi-Hildebrand plots is a good evidence for the 1:1 stoichiometry of the charge transfer complexes studied. The calculated  $\epsilon_{CT}$  and  $K_f$  values are also included in Table 1. It is interesting to note that, as expected by the Mulliken's charge transfer theory [39], the formation constant of the **L**- $I_2$  complex increases with increasing polarity of medium as  $\text{CH}_2\text{Cl}_2 > \text{CH}_2\text{Cl}_2 + \text{CHCl}_3 > \text{CHCl}_3$ . The observed trend could be due to the high stabilization of the excited states in more polar solvents, where the charges are more separated than those in the ground state [39,40].

The crystalline  $(\text{L} \cdot \text{I}^+) \text{I}_3^-$  adduct was prepared, as described in the experimental section, and its IR spectrum together with that of **L** were recorded. The corresponding spectral data of the ligand and the complex together with the tentative assignment of the most important IR-frequencies that provide structural evidence of the complex are listed in Table 2.

From the data given in Table 2 it is clear that most of the fundamental frequencies of the electron-pair donor compound **L** show significant shifts, strongly supporting the formation of the **L**- $I_2$  complex. Generally,  $\nu(\text{C}=\text{O})$ ,  $\nu(-\text{CH}_2-)$ ,  $\nu(\text{C}-\text{H})$  and  $\nu(\text{C}-\text{O})$  bands of the free donors are shifted to lower frequencies on complex formation. This behavior is in accordance with the charge migration from donors to acceptors. Meanwhile, such small changes clearly indicate that these bonds in **L** are not involved in the complexation process with iodine. On the other hand, the  $\nu(\text{C}-\text{N})$  and  $\nu(\text{N}-\text{H})$  of the macrocycle **L** show the largest shifts upon complexation with iodine. These changes clearly show that, as expected [20-23,26], the nitrogen atoms as n-donors in **L** molecule are

**Table 2.** Infrared Frequencies ( $\text{cm}^{-1}$ ) and Tentative Assignments for Macrocycle Diamide **L** and its Charge Transfer Complex with Iodine

<b>L</b>	$(\mathbf{L}^+)\mathbf{I}_3^-$	Assignment
3382 (vs), 3278 (s)	3411 (s, br)	$\nu$ (N-H)
3047 (w)	3056 (w, sh)	$\nu$ (C-H)aromatic
2907 (s)	2923 (w)	$\nu$ (C-H)aliphatic
1648 (vs)	1640 (s)	$\nu$ (C=O)
1550 (s), 1508 (s)	1547 (m), 1494 (m)	$\nu$ (-CH <sub>2</sub> -)
1435 (s)	1424 (m)	$\nu$ (CH)
1333 (s)	1260 (m)	$\nu$ (C-N)
1218 (s)	1133 (m)	$\nu$ (C-N)
1133 (s)	1112 (m)	$\nu$ (C-O)

(vs) very strong; (s) strong; (m) medium; (sh) shoulder; (br) broad; (w) weak.

**Table 3.**  $^1\text{H}$  NMR Spectral Data of **L** and its Charge Transfer Complex with Iodine in Chloroform

<b>L</b>	1.85 (s, 1H) -NH (Amine)	2.89 (t, 4H) -CH <sub>2</sub> -NH- (Amine)	3.43 (t, 4H) -CH <sub>2</sub> -NH- (Amide)	4.43 (s, 4H) -O-CH <sub>2</sub> -CO-	6.82-6.95 (complex, 4H) (Benzo)	7.76 (s, 2H) -NH (Amide)
<b>L-I<sub>2</sub></b>	1.55	2.86-2.93	3.69	4.20	7.24	7.51

mainly involved in the complexation with iodine as an electron-accepting molecule.

Interesting results were also obtained from the  $^1\text{H}$  NMR spectra of **L** and its iodine complex in chloroform solution (Table 4). As is obvious from Table 3, the  $^1\text{H}$  NMR spectrum of **L** showed 6 series of signals for different protons involved; these consisted of a singlet at 1.85 ppm for the -NH proton, a triplet at 2.89 ppm due to amine -OCH<sub>2</sub>-NH protons, another triplet at 3.43 ppm for amide -OCH<sub>2</sub>-NH protons, a singlet at 4.43 due to -OCH<sub>2</sub>-CO- protons, a complex signal at 6-82-6-95 ppm for phenyl-H protons and finally a sharp singlet at 7.76 ppm for the two amide -NH protons. Meanwhile, all protons of the macrocycle **L** show some pronounced shifts upon complexation with iodine, the shifts in both amine and amide -NH protons being larger than that of other macrocycles' protons. In support of the IR results, the observed chemical shift behavior may reveal the transfer of charge from nitrogen atoms of the macrocyclic ligand to the

acceptor molecule iodine, during the course of complexation reaction [11].

## ACKNOWLEDGMENTS

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