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Synthesis and spectroscopic characterization of piperidine/I_2 charge-transfer complex in different chlorinated organic solvents

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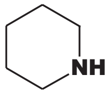
The charge-transfer (CT) complex formed between piperidine (Pip) as donor and iodine (I_2) as σ-acceptor was studied spectrophotometrically. The synthesis and characterization of the piperidine CT-complex of iodine, $[(Pip)_2]I^+.I_3^-$, was described. This complex was readily prepared by the reaction of Pip with I_2 in $CHCl_3$ solvent. Infrared spectra (mid-IR and far-IR), UV-Vis techniques and elemental analyses were used to characterize the piperidine/I_2 charge-transfer complex. Benesi-Hildebrand's method and its modifications were applied to the determination of the association constant (K) and the molar absorption coefficient (ε).

Keywords: Charge-transfer, Iodine, Piperidine, Infrared spectra.

1. INTRODUCTION

Charge-transfer complexes are known to take part in many chemical reactions like addition, substitution and condensation [1,2]. These complexes have attracted great attention as non-linear optical materials and electrical conductors [3-6]. Electron donor-acceptor (EDA) interaction is also important in the field of drug-receptor binding mechanism [7], in solar energy storage [8] and in surface chemistry [9] as well as in many biological fields [10]. On the other hand, the EDA reactions of certain π-acceptors have been successfully utilized in pharmaceutical analysis [11]. For these wide applications extensive studies on CT- complexes of π-acceptors have been performed [12].

Piperidine (Formula I) is an organic compound with the molecular formula $C_5H_{11}N$. It is a cyclic amine with a six-membered ring. It is a clear liquid with pepper-like odor. The piperidine skeleton is present in numerous natural alkaloids such as piperine, the main active chemical agent in black pepper and relatives, pharmaceutical drugs such as raloxifene.



Formula I

The solid charge-transfer complexes formed between iodine and several types of electron donors such as aromatic hydrocarbons, polycyclic amines, mixed oxygen/nitrogen cyclic bases, aromatic/aliphatic amines have been studied and categorized [13-22]. The tri-iodide ion I_3^-, penta-iodide ion I_5^-, and ennea-iodide ion I_9^- were formed through the reaction of iodine with various donors like metal acetylacetonates [23-25], polyazacyclic [26-28], and crown ethers [29-32]. Some of charge-transfer complexes show very interesting applications in the analysis of some drugs in pure form or in pharmaceutical preparations [33, 34]. The charge-transfer in fullerene-based [35, 36] compounds is currently of great interest since these materials can be utilized as superconductors [37] and to produce non-linear optical activity [38].

2. EXPERIMENTAL

2.1. Materials

All chemicals used throughout this work were Analar or extra pure grade. Piperidine ($C_5H_{11}N$), was of analytical reagent grade (Merck reagent). The iodine acceptor was purchased from Aldrich. Stock solutions of piperidine and iodine acceptor were freshly prepared and chloroform of spectroscopic grade (Merck) was used as received.

2.2. Synthesis

[Piperdine]-iodine complex

At room temperature, the solid CT complex of Pip with iodine was prepared by mixing 85.15 mg

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(1.0 mmol) of the donor in chloroform (10 mL), a solution of iodine was added (253.81 mg, 1.0 mmol) in the same solvent (10 mL) upon continuous stirring for about 15 min. A dark brown complex was formed, washed several times with small amounts of chloroform, and dried under vacuum over anhydrous calcium chloride; the empirical formula of the complex $[(Pip)_2]I^+.I_3^-$ is $C_{10}H_{22}N_2I_4$ with molecular weight 677.92 g/mol.

2.3. Methods

The electronic spectra of Pip, iodine acceptor and resulting CT complex were recorded in the region of 200-800 nm by using a Jenway 6405 spectrophotometer with quartz cells of 1.0 cm path length. Photometric titration was performed at 25ºC for the reactions of donor with acceptor in chloroform as follow: the concentration of Pip in the reaction mixtures was kept fixed at 5.0×10^{-4} M, while the concentration of acceptor was changed over a wide range from 1:0.25 to 1:4.00. Infrared measurements (KBr discs) of the solid donors, acceptor and CT complexes were carried out on a Bruker FT-IR spectrophotometer over the range of wave numbers 400_3–4000 cm_3^{-1} with 50 scans at 2 cm^{-1} resolution.

3. RESULTS AND DISCUSSION

The results for the elemental analysis of the piperidine charge-transfer complex are shown in Table 1. It can be seen from the table that the

Table 1: Elemental analysis CHN and physical parameters data of the CT-complex formed in the reaction of the Pip with iodine

Complexes (FW)	Mwt.	C%		H%		N%		Physical data	
		Found	Calc.	Found	Calc.	Found	Calc.	Color	mp (°C)
$[(Pip)_2]I^+.I_3^-$	677.92	17.45	17.70	3.29	3.24	3.99	4.13	Dark brown	< 30

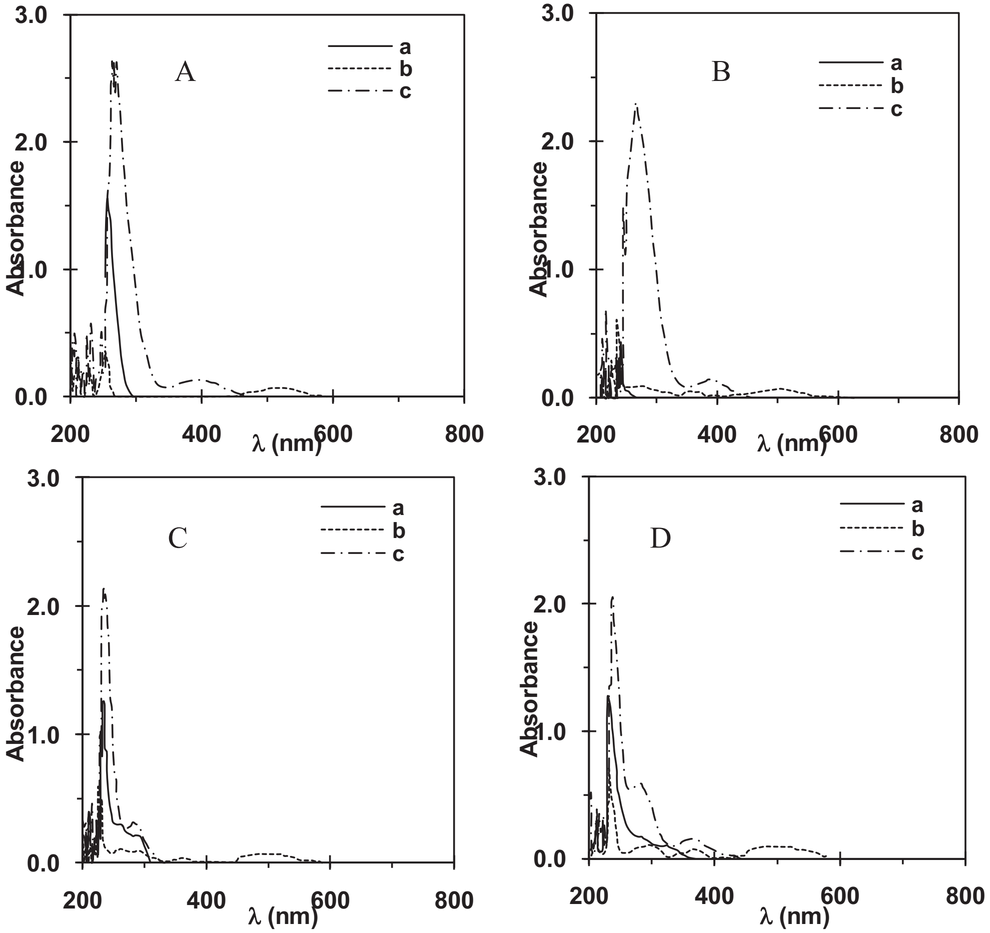


Fig. 1. Electronic absorption spectra of; (A): Pip-iodine reaction in CCl_4, (B): Pip-iodine reaction in $CHCl_3$, (C): Pip-iodine reaction in CH_2Cl_2 and (D): Pip-iodine reaction in 1,2-dichloroethane. (a) = donor (1.0×10^{-4}M), (b) = acceptor (1.0×10^{-4}M) and (c) = CT-complex.

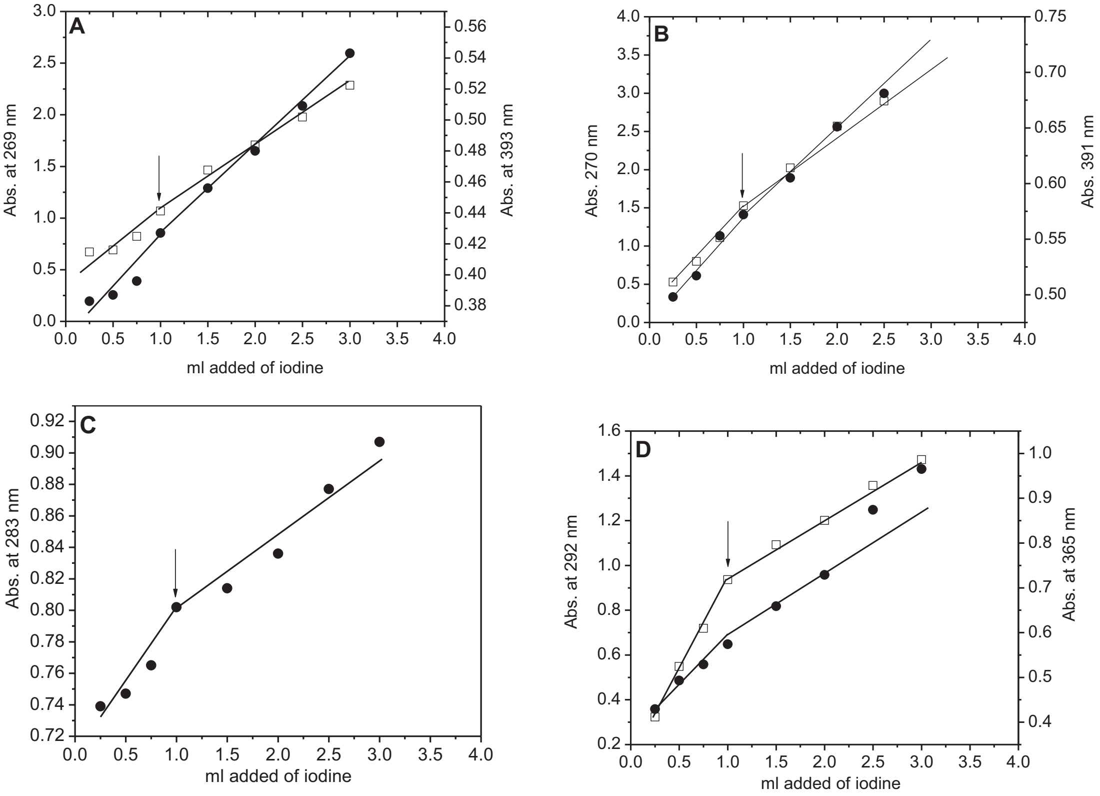


Fig. 2. Photometric titration curves for the Pip-iodine system in various solvents: (A) in CCl_4 at 269 and 393 nm, (B) in $CHCl_3$ at 270 and 391 nm, (C) in CH_2Cl_2 at 283 nm and (D) in 1,2-dichloroethane at 292 and 365 nm.

values found are in agreement with the calculated ones, and the composition of the CT complex is matched with the molar ratios determined from the photometric titration of Pip with the iodine σ-acceptor. All complexes are insoluble in cold and hot water, but easily soluble in DMF and DMSO.

3.1. Electronic absorption spectra of the Pip/iodine system

The electronic (UV-Vis) absorption spectra of the Pip/iodine complex were measured in various solvents such as CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2-dichloroethane. In each solvent the complex was formed by adding X ml of 5.0×10^{-4} M iodine (X = 0.25, 0.50, 0.75, 1.00, 1.50, 2.00, 2.50 and 3.00 ml) to 1.00 ml of 5.0×10^{-4} M Pip. The volume of the mixture in each case was completed to 15 ml for CCl_4, 10 ml for $CHCl_3$, 20 ml for CH_2Cl_2, and 10 ml for 1,2-dichloroethane with the respective solvent. The concentration of Pip in the reaction mixture was kept fixed at 0.33×10^{-4}, 0.50×10^{-4}, 0.25×10^{-4} and 0.50×10^{-4} M in the case of CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2-dichloroethane solvent, respectively, while the concentration of iodine was varied over the range of 0.0833×10^{-4} M to 1.00×10^{-4} M for Pip/I_2 in CCl_4, from 0.125×10^{-4} M to 1.500×10^{-4} M for Pip/I_2 in $CHCl_3$, from 0.0625×10^{-4} M to 0.7500×10^{-4} for Pip/I_2 in CH_2Cl_2 and from 0.125×10^{-4} M to 1.500×10^{-4} M for Pip/I_2 in 1,2-dichloroethane. These concentrations produce Pip:I_2 ratios in the range from 1:0.25 to 1:3.00. The electronic absorption spectra of the 1:1 Pip:I_2 complexes in CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2-dichloroethane together with those of the reactants I_2 and Pip are shown in Figures 1A, B, C and D, respectively.

The spectra show characteristic absorption bands which are not present in the spectra of the reactants free iodine and Pip. These bands at (393 and 269 nm), (391 and 270 nm), 283 nm and (363 and 292 nm) are assigned to the CT-complex formed by the reaction of Pip with I_2 in the solvents CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2-dichloroethane, respectively. Photometric titration curves based on these characteristic absorption bands are given in Figures 2A, B, C and D.

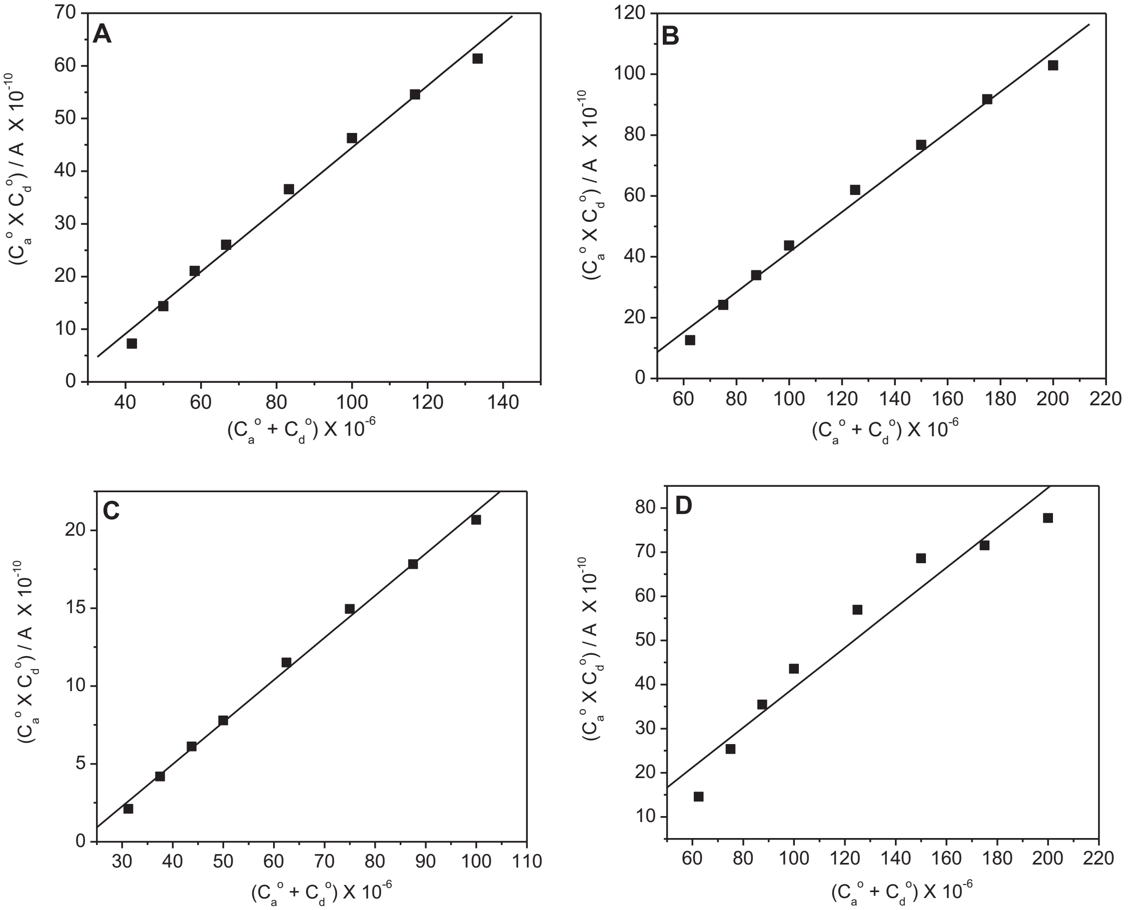


Fig. 3. The plot of (C_d^o+C_a^o) values against (C_d^o.C_a^o/A) values for the Pip-iodine system in various solvents: (A) in CCl_4 at 393 nm, (B) in $CHCl_3$ at 391 nm, (C) in CH_2Cl_2 at 283 nm and (D) in 1,2-dichloroethane at 365 nm.

These photometric titration curves were obtained according to the known methods [39] by plotting absorbance against the X ml added of the iodine σ-acceptor. The equivalence points shown in these curves clearly indicate that the CT complex formed between Pip and iodine is 1:1. The formation of 1:1 complex was also supported by elemental analysis, mid and far infrared spectra.

However, the two absorption bands appearing ≈ 360 and ≈ 290 nm are well known [40-42] to be characteristic for the formation of the tri-iodide ion (I_3^-). Accordingly, the formed complex was formulated as $[(Pip)_2]I^+.I_3^-$.

and A is the absorbance of the definite bands around 290 and 360 nm. From the data obtained C_d^o of Pip, C_a^o of I_2, (C_a^o+C_d^o) and (C_a^o.C_d^o/A) in CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2-dichloroethane were determined. When the C_a^o.C_d^o/A values for each

It was of interest to observe that the solvent has a pronounced effect on the spectral intensities of the formed $[(Pip)_2]I^+.I_3^-$ complex. To study the solvent effect in a quantitative manner, it was necessary to calculate the values of the association constant, *K*, the molar absorption coefficient *ε*, and the oscillator strength, *f*, of the iodine complex in each solvent. The 1:1 modified Benesi-Hildebrand equation [43] was used in the calculations:

$$\frac{C_a^o C_d^o l}{A} = \frac{1}{K\varepsilon} + \frac{C_a^o + C_d^o}{\varepsilon} \tag{1}$$

where C_a^o and C_d^o are the initial concentrations of the acceptor I_2 and the donor Pip, respectively, solvent are plotted against the corresponding (C_a^o+ C_d^o) values, straight lines are obtained with a slope of 1/ε and intercept of 1/kε as shown in Figures 3A, B, C and D for the reactions in CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2–dichloroethane.

Table 2. Spectrophotometric results for the Pip complex with iodine in different solvents at 25°C and the dielectric constants of the solvents

Solvent	λ_{max} (nm)	E_{CT} (eV)	K ($l.mol^{-1}$)	ε_{max} ($l.mol^{-1}.cm^{-1}$)	f	μ	I_p	D
CCl_4	393	3.16	4.09×10^4	1.70×10^4	6.80	23.80	7.71	2.2
$CHCl_3$	391	3.18	2.71×10^4	1.52×10^4	8.75	27.00	7.73	4.7
CH_2Cl_2	361	3.45	6.97×10^4	1.81×10^4	13.44	31.34	7.96	8.9
$(CH_2)_2Cl_2$	365	3.41	7.52×10^4	2.21×10^4	14.30	33.30	8.08	10.4

Table 3. Infrared frequencies[(a)] (cm^{-1}) and tentative assignments for the Pip donor and the $[(Pip)_2]I^+.I_3^-$ complex.

Pip	$[(Pip)_2]I^+.I_3^-$	Assignments[(b)]
3393 s, br	3445 s, br	ν(N-H)
2938 vs	2949 vs	ν_s(C-H)
2854 ms	2833 mw	ν_{as}(C-H)
2809 mw	2803 mw	
2738 mw	2729	
---	2619 mw	Hydrogen bonding
	2502 mw	
	2401 mw	
	2341 mw	
1627 mw	1617 s	δ_{def}(N-H)
		Ring breathing bands
1542 ms	1582 s	C-H deformation
1447 ms	1455 s	
1417 ms		
1321 mw	1313 w	ν(C-C)
1276 s	1255 w	ν(C-N)
1162 mw	1162 w	
1112 mw	1120 w	
1032 mw	1080 w	
	1027 ms	
948 mw	946 mw	(C-H) bend
861 ms	860 mw	δ_{rock}; NH
806 mw	789 vw	CH_2 rock
737 ms	630 vw	Skeletal vibrations
646 vw		
553 ms	549 ms	CNC deformation
438 mw	433 ms	

(a): s = strong, w = weak, m = medium, sh = shoulder, v = very, br = broad. (b): ν, stretching; δ, bending.

The oscillator strength f was obtained from the approximate formula [44]:

$$f = (4.319 \times 10^{-9})\, \varepsilon_{max} \,.\, \nu_{1/2} \,. \qquad (2)$$

Here $\nu_{1/2}$ is the bandwidth for half-intensity in cm^{-1}. The oscillator strength values together with the corresponding dielectric constants, D, of the solvent used are given in Table 2. The trend of the values in this table reveals several facts.

i) The $[(Pip)_2]I^+.I_3^-$ complex shows high values of both the association constant (K) and the molar absorption coefficient (ε). This high value of K reflects the high stability of the iodine complex as a result of the expected high donation power of Pip, while the high value of ε agrees quite well with the existence of the tri-iodide ion, I_3^-, which is known to have a high absorbance value [40-42].

ii) The values of the oscillator strength, f, increase on increasing the dielectric constant (D) of the solvent. This result could be explained on the basis of competitive solvent interactions with the acceptor [21]. Figure 4 shows the obtained linear relationship between f and D.

iii) The formation constant (K) for the $[(Pip)_2]I^+.I_3^-$ complex shows some variations, Table 2, as the solvent is changed, but no clear relation with solvent properties can be derived.

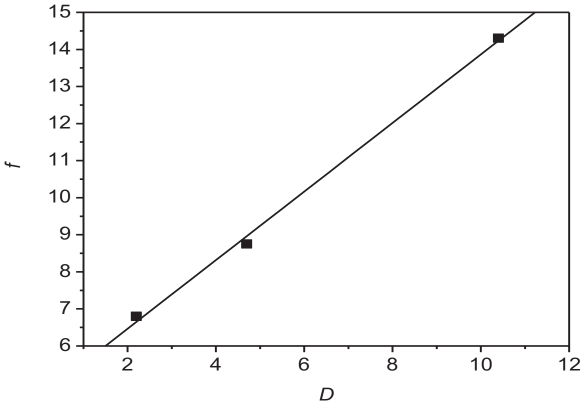


Fig.4. The plot of the oscillator strength *f* of $[(Pip)_2]I^+.I_3^-$ against the dielectric constant of the solvent *D*.

Table 4. Fundamental vibrations for some tri-iodide compounds.

Compounds	Assignments*			References
	ν_1	ν_2	ν_3	
KI_3	111	---	143	[44]
CsI_3	103	69	149	[53]
$[Ni(acac)_2]_2I^+.I_3^-$	101	84	132	[25]
$[(Pip)_2]I^+.I_3^-$	102	79	145	Present work

ν_1^*, ν_s (I-I); ν_2, δ (I_3^-); ν_3, ν_{as} (I-I).

Finally, the general mechanism for the formation of the $[(Pip)_2]I^+.I_3^-$complex is proposed as follows:

$$2Pip+I_2 \rightarrow [Pip]_2I^+.I^-$$
$$[Pip]_2I^+.\ I^-+I_2 \rightarrow [(Pip)_2]I^+.I_3^-$$

The $[Pip]_2I^+.I^-$ reaction intermediate is analogous to the well known species $[(donor)I]^+.I^-$ formed in the reaction of iodine with many donors [19, 26]. It has characteristic [45] absorption, around 250 nm; see Figures 1A, B, C and D.

The transition dipole moment (*μ*) of the iodine complex, Table 2, was calculated from the following equation [46]:

$$\mu = 0.0958[\varepsilon_{max}\, \nu_{1/2}\ /\ \nu_{max}]^{½} \qquad (3)$$

The ionization potential (I_p) of the free donor was determined from the CT energies of the CT band of its complexes with iodine by using the following relationship [47]:

$$E_{CT} = I_p - 5.2 + 1.5\ /\ (I_p - 5.2) \qquad (4)$$

where E_{CT} is the energy of the charge-transfer of the iodine complex; the energy of the π-σ* or *n*-σ* interaction (E_{CT}) is calculated using the following equation [47]:

$$E_{CT} = 1243.667\ /\ \lambda_{CT}\ (nm) \qquad (5)$$

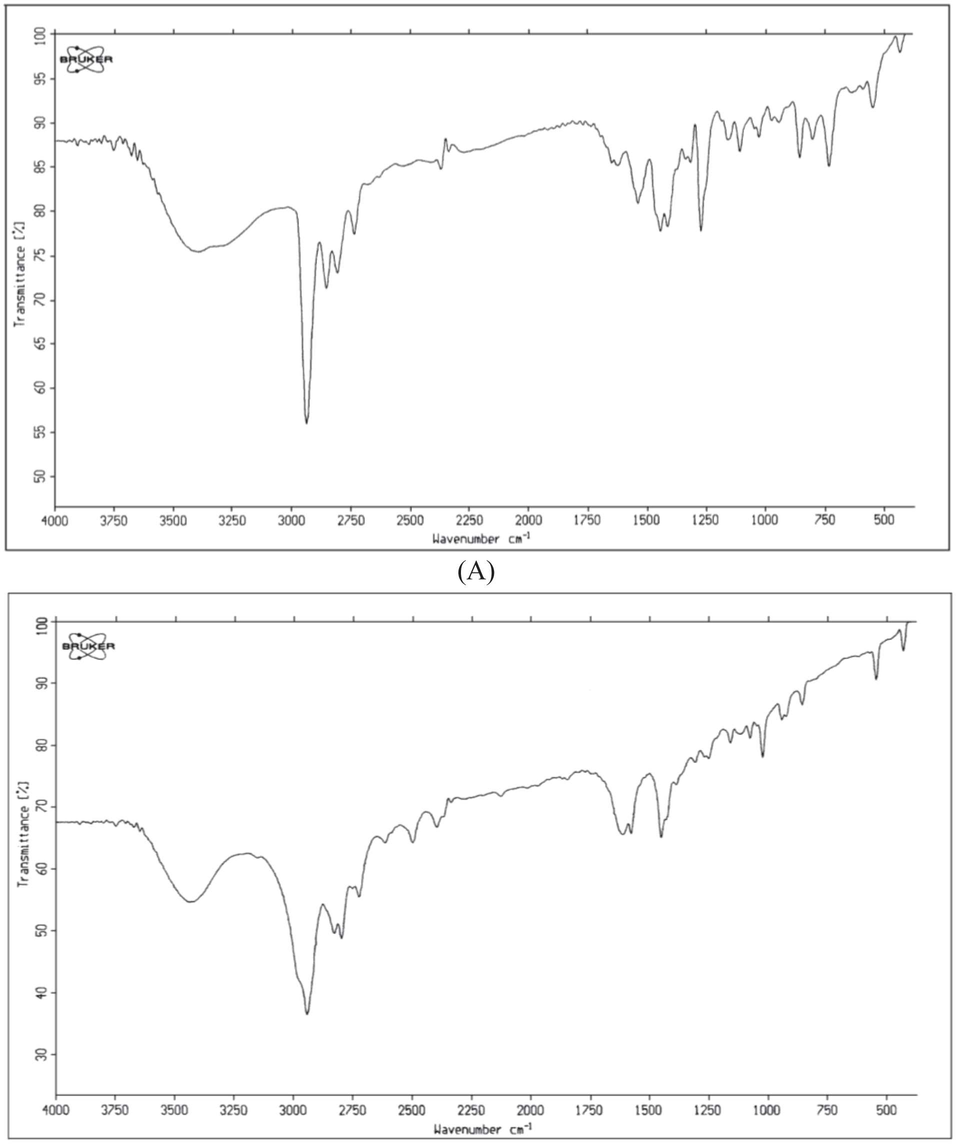
where λ_{CT} is the wavelength of the complexation band.

3.2. Infrared spectra of the Pip-iodine solid complex

The mid infrared spectra of Pip and the formed CT complex, $[(Pip)_2]I^+.I_3^-$, were recorded from KBr discs. These spectra are shown in Figures 5A and B, respectively. The spectral bands are resolved and assigned to their vibrational modes, as given in Table 3. As expected, the bands characteristic for the Pip unit in the $[(Pip)_2]I^+.I_3^-$ CT complex display small changes in band intensities and frequency values. For example, the stretching asymmetry and symmetry of the ν(N-H) vibration occurred at 3393 and 3447 cm^{-1} for free Pip and $[(Pip)_2]I^+.I_3^-$, respectively. There is an increase in the intensity of the δ(N-H) vibration in the case of iodine complex rather than Pip alone as well as a blue shift in the wavenumbers from 1627 cm^{-1}(Pip) to 1617 cm^{-1} (iodine complex). Such changes clearly indicate that the N-H bond in the piperidine donor participates in the complexation process with iodine. On the other hand, the presence of a few bands at 2619, 2502, 2401, and 2341 cm^{-1} could be assigned to the expected hydrogen bonding in the Pip/iodine complex [48]. This fact strongly supports the mode of interaction between Pip and iodine by forming a hydrogen bond between the donor Pip and the iodine acceptor, (N-H---I).

3.3. Far infrared spectra of the Pip-iodine solid complex

The far infrared spectrum of $[(Pip)_2]I^+.I_3^-$ was recorded from Nujol mulls dispersed on polyethylene windows in the region 50-600 cm^{-1} and was focused in the region 50-200 cm^{-1}, as given in Figure 6. The spectrum associated with the $[(Pip)_2]I^+.I_3^-$ complex shows the characteristic bands for the tri-iodide ion, I_3^- at 145, 102 and 79 cm^{-1}. These bands can be attributed to ν_{as}(I-I), ν_s(I-I) and δ(I_3^-), respectively. These three absorption bands do not exist in the spectrum of the donor Pip. However, the I_3^- ion may be linear ($D_{\infty h}$) or non linear (C_{2v}). Group theoretical analysis indicates that the I_3^- ion with C_{2v} symmetry displays three vibrations: ν_s(I-I); A^-_1, ν_{as}(I-I); B_2 and δ(I_3^-); A_1, all infrared active in agreement [40, 49, 50] with the



(A)

(B)

Fig. 5. Infrared spectra of: (A) Pip and (B) the charge-transfer complex, $[(Pip)_2]I^+.I_3^-$.

observed three infrared bands for $[(Pip)_2]I^+.I_3^-$, as shown in Table 4. Accordingly, the formed iodine complex is formulated as $[(Pip)_2]I^+.I_3^-$. The conversion of iodine molecules into polyiodide units is well known in the literature. The formation of I_3^- ions was previously reported [51–53] in the reaction of iodine with various donors such as 4-(dimethylamino) pyridine, 1-aza-15-crown-5,3,6,9,14-tetrathiabicyclo [9.2.1]tetradeca-11,13-diene and nicotine.

3.4. Group theoretical analysis

The geometry of I_3^- in $[(Pip)_2]I^+.I_{33}^-$ may be assigned to one of the two structures: linear structure with ($D_{\infty h}$) symmetry or non-linear structure with (C_{2v}) symmetry. It follows from the above discussion that the shape of the I_{33}^- ion is non linear and displays C_{2v} symmetry. The three modes of vibrations distributed over the symmetry species $2A_1 + B_2$ should be all infrared active. If the complex is linear, i.e., of $D_{\infty h}$ symmetry, it would give only two infrared bands, which is not the case.

CONCLUSION

The characterization techniques like infrared spectra (mid and far), elemental analysis and electronic spectra confirmed that the piperidine-iodine interaction yields a complex of the formula $[(Pip)_2]I^+.I_{33}^-$. The triiodide ion formed belongs to the C_{2v} point group and the

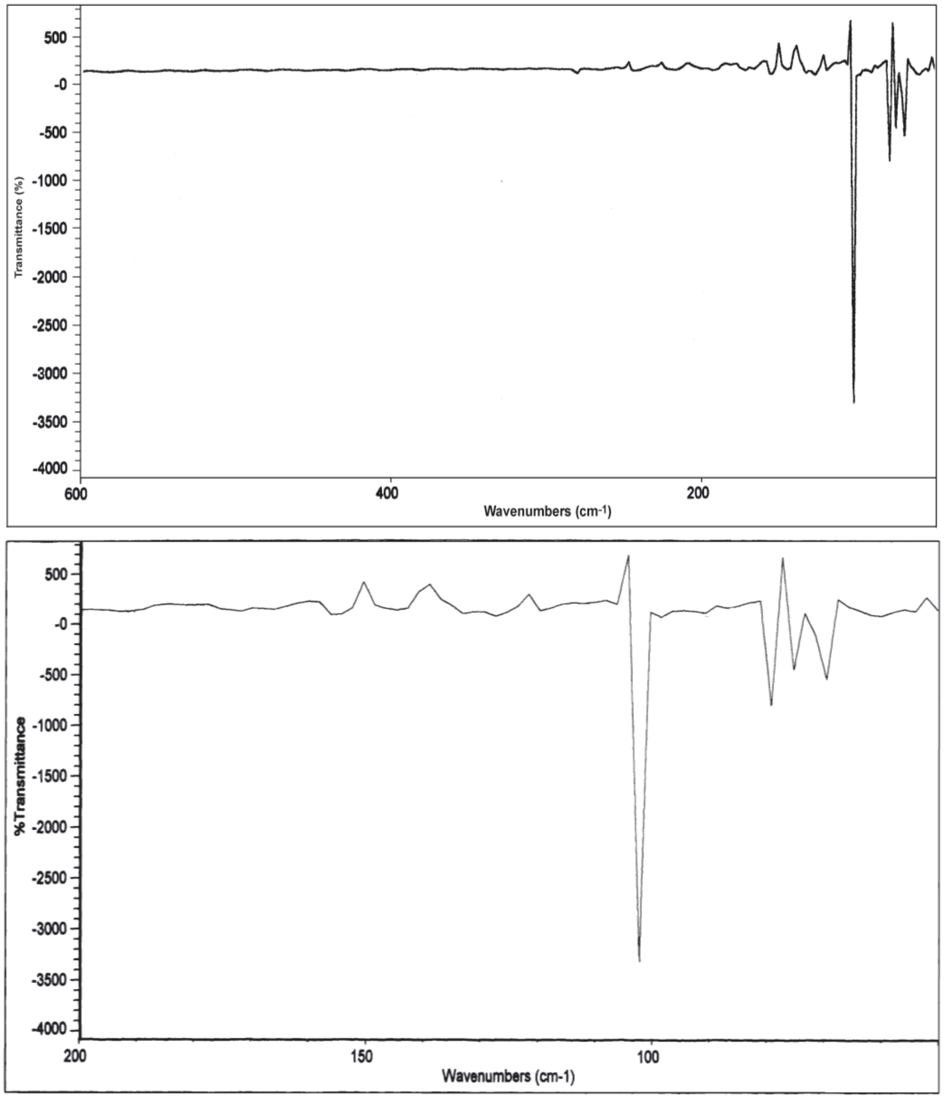


Fig. 6. Far-infrared spectrum of the charge-transfer complex, $[(Pip)_2]I^{+}.I_3^{-}$.

far-infrared spectra play an important role for the assignment of I_{33}^{-}. Some interesting physical data were obtained which throw light on the stability of the piperidine complex. The iodine complex was studied in various solvents like CCl_4, $CHCl_3$, CH_2Cl_2 and 1,2-dichloroethane. It was found that the values of the oscillator strength *f* increase with the increase in the dielectric constant (*D*) of the solvent.

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СИНТЕЗ И СПЕКТРОСКОПСКО ОХАРАКТЕРИЗИРАНЕ НА ПИПЕРИДИН/I_2 КОМПЛЕКС С ПРЕНОС НА ЗАРЯД В РАЗЛИЧНИ ХЛОРИРАНИ ОРГАНИЧНИ РАЗТВОРИТЕЛИ

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(Резюме)

Комплекс с пренос на заряд (ПЗ), образуван между пиперидин (Пип) като донор и йод (I_2) като σ-акцептор, е проучен спектрофотометрично. Описан е синтезът и охарактеризирането на пиперидинови ПТ-комплекс на йод, $[(\text{Пип})_2]I^+.I_3^-$. Този комплекс е получен лесно посредством реакцията на Пип с I_2 в $CHCl_3$ като разтворител. Инфрачервена спектроскопия (mid-IR и far-IR), UV-Vis техники и елементен анализ са използвани, за да се характеризира пиперидин/I_2 комплекс с пренос на заряд. Методът на Банези-Хилдебранд и негови модификации са приложени за определяне на асоциационната константа (K) и моларния абсорбционен коефициент (ε).