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Synthesis, spectroscopic characterization and structural studies of a new proton transfer (H-bonded) complex of o-phenylenediamine with L-tartaric acid

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HIGHLIGHTS

• A proton transfer complex is synthesized and characterized by spectral techniques.

- Bonding modes and structural information are ascertained from crystallographic studies.
- Single-crystal X-ray studies confirmed the H-bonded network.
- Thermal analysis (TGA-DTA) was also used to confirm the thermal fragmentation and the stability of the CT complex.

• Benesi-Hildebrand equation was used to determine K_{CT}, ε_{CT}, and E_{CT} of the CT complex.

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ABSTRACT

A proton transfer or H-bonded (CT) complex of o-phenylenediamine (OPD) as donor with L-tartaric acid (TART) as acceptor was synthesized and characterized by spectral techniques such as FTIR, ¹H NMR, elemental analysis, TGA–TDA, X-ray crystallography and spectrophotometric studies. The structural investigations exhibit that the cation [OPD⁺] and anion [TART⁻] are linked together through strong N⁺-H···O⁻ type hydrogen bonds due to transfer of proton from acceptor to donor. Formed H-bonded complex exhibits well resolved proton transfer bands in the regions where neither donor nor acceptor has any absorption. The stoichiometry of the H-bonded complex (HBC) was found to be 1:1, determined by straight line methods. Spectrophotometric studies have been performed at room temperature and Benesi–Hildebrand equation was used to determine formation constant (K_{CT}), molar extinction coefficient (ϵ_{CT}) and also transition energy (E_{CT}) of the H-bonded complex. Thermal analysis (TGA–DTA) was also used to confirm the thermal fragmentation and the stability of the synthesized H-bonded complex.

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1. Introduction

L(+)-(2R,3R)-Tartaric acid is a naturally occurring chiral dicarboxylic acid [1]. It has two kinds of different proton-donor centers comprising of two carboxylic and two hydroxyl groups [2]. Studies of molecular packing and hydrogen bonding in the crystals of tartaric acid and its derivatives are relevant for the development of new approaches in crystal engineering [3–6]. Tartaric acid forms a broad range of hydrogen-bonded complexes with amines, betaines and amino acids, and salts of tartaric acid with amines show either 1:1 or 1:2 stoichiometry [7–20]. Some of such complexes can undergo ferroelectric phase transition [21]

and exhibit non-linear optical properties [22-25]. In complexes with organic bases, tartaric acid can interact in three ways: (i) as an acid, without proton transfer to the basic proton-acceptor center, (ii) as a semi-tartrate anion, in which the proton from one carboxyl group is transferred to the basic center, and (iii) as tartrate di-anion. The diastereomeric salts often differ in solubility, making tartaric acid one of the most frequently used resolving agents [1]. The interaction pathways strongly depend on the basicity of the organic bases, the number of the interacting centers, and the molecular structure of the bases used [26,27]. (2R,3R)-Tartaric acid has been used in stereoselective synthesis in the acidic form or with protected hydroxyl or carboxylic groups. Recently, investigations have been performed on the crystal structure and spectroscopic properties of several complexes of betaines with L(+)-(TART) and with meso-TART, whose energy and enthalpy calculations evaluated have been by the DFT calculations [10,15,16,28]. In view of







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the above mentioned potentials of L-tartaric acid, many H-bonded CT complexes of it and other acceptors with organic bases were reported [29–42] and some of them were deposited in the CCDC [43].

Here-in, we report for the first time, to the best of our knowledge, new 1:1 H-bonded complex [(OPD)⁺(TART)⁻] of o-phenylenediamine with tartaric acid. The 1:1 H-bonded complex was synthesized and characterized by absorption spectra, elemental analysis, FTIR, ¹H NMR, and single-crystal X-ray studies. The aim of the work is to determine the reaction stoichiometry, formation constant (K_{CT}), molar extinction coefficient (ε_{CT}), free energy (ΔG°), and also transition energy (E_{CT}) of the H-bonded complex by using Benesi–Hildebrand equation. H-bonded complex was investigated in both solution and solid state and the nature of these interactions was interpreted using above mentioned spectral techniques.

2. Experimental

2.1. Materials

Analytical grade o-phenylenediamine and L-tartaric acid were employed for the synthesis of H-bonded complexes. Methanol, acetonitrile and DMSO were taken as HPLC grade.

2.2. Synthesis of H-bonded complex

The solid $[(OPD)^{+}(TART)^{-}]$ was prepared by mixing the saturated solution of OPD (0.108 g, 1 mmol) in acetonitrile (25 ml) with saturated solution of TART (0.636 g, 1 mmol) also in acetonitrile. A pink color solution was formed upon the mixing and continuous stirring of these two reactants produced pink precipitate. The precipitate was filtered off and washed several times with small amounts (5 ml) of acetonitrile and dried under vacuum over CaCl₂. The melting point and transition energy of H-bonded complex were found to be >300 °C and 4.6061 eV, respectively. The result of the elemental analysis of the H-bonded complex is given as: (theoretical values are shown in brackets): $[(OPD)^{+}(TART)^{-}]$, (C₁₀H₁₄N₂O₆) H-bonded complex (M/w: 258.23 g): C, 46.51% (47.00%); H, 5.42% (5.40%); N, 32.57% (32.77%).

2.3. Single crystal growth

A saturated solution of the synthesized title complex $[(OPD)^+$ (TART)⁻] was prepared in methanol, stirred well for about 1 h and heated slightly to dissolve well. Then the solution was filtered through a Whatmann: 41 grade filter paper to remove the suspended impurities. The clear filtrate was collected in 50 ml conical flask and kept unperturbed in a dust free chamber for the growth of single crystal of the $[(OPD)^+(TART)^-]$. Well defined, bright colored, transparent and needle shaped crystals were harvested at the end of the fifth day.

2.4. Spectrophotometric analyses

When 3 ml solution each of the acceptor and the donor were mixed, a charge transfer complex was formed. The wavelength of maximum absorption of the resulting solution was determined. The H-bonded complex of the 1:1 reaction mixture was kept over night at room temperature to form stable complexes, were analyzed as shown in Fig. 3. The maximum wavelength of the charge transfer complex was determined by spectrophotometer to be 270 nm. The concentration of acceptor (TART) in the reaction mixture was maintained at 1.0×10^{-4} M, whereas the concentration of the donor (OPD) changed over a wide range of concentrations $(1.5 \times 10^{-4}-15 \times 10^{-4}$ M) to maintain the conditions $[D] \gg [A]_0$



Fig. 1. Electronic absorption spectra of (A) L-tartaric acid, Acceptor $(1 \times 10^{-4} \text{ M})$; (B) Donor–Acceptor CT complex and (C) o-phenylenediamine, Donor $(1 \times 10^{-4} \text{ M})$, in methanol at room temperature.



Fig. 2. Benesi–Hildebrand plots of charge transfer complex of o-phenylenediamine with L-tartaric acid, $[D]_o/A$ vs. $1/[A]_o$ in methanol at room temperature.

Table 1

The electronic absorption spectral data for [(OPD)⁺(TART)⁻] CT complex of OPD with TART in methanol at room temperature.

Concentration of donor (×10 ⁻⁴ M)	Concentration of acceptor (M)	Absorbance at λ _{CT} 238 nm	Formation constant (K _{CT}) l mol ⁻¹	Molar extinction coefficient (ε_{CT}) l cm ⁻¹ mol ⁻¹
1.50 2.00 2.77 10.00 15.00	1×10^{-4}	0.481 0.588 0.769 1.609 2.023	1.337×10^3	2.842×10^{5}

or $[D] \ll [A]_o$ for applying Benesi–Hildebrand, which is valid under this condition, to calculate the formation constant, K_{CT} , and the absorptivity, ε_{CT} , values for H-bonded complex in methanol. The stoichiometry of the H-bonded complex was also obtained from the Benesi–Hildebrand plots.

2.5. Spectral and crystal structure Analyses

The electronic absorption spectra of the donor (O-phenylenediamine), acceptor (L-tartaric acid) and the resulting H-bonded

Table 2

Crystallographic and data collection parameters for $[(OPD)^{+}(TART)^{-}]$ CT complex of OPD with TART.

CCDC no.	939527
Empirical formula	$C_{10}H_{14}N_2O_6$
Formula weight (Mr)	258.23
Crystal description	Prism
Temperature (K)	100(2)
Wavelength (Å)	0.71073
Crystal system	Triclinic
Space group	P-1
Unit cell dimensions (Å)	a = 7.114(5)
	b = 9.189(5)
	c = 9.628(5)
	$\alpha = 70.734(5)$
	$\beta = 69.464(5)$
	$\gamma = 81.825(5)$
Unit cell volume	556.1(6) Å ³
Ζ	2
Calculated density (mg/m ³)	1.542
Absorption coefficient (mm ⁻¹)	0.129
F(000)	272
Crystal size (mm ³)	$0.24 \times 0.22 \times 0.20$
H, k, 7 max	8, 11, 12
θ range (°)	2.37-26.49
Reflections collected/unique	3214
Min and max transmission	0.9698, 0.9747
Refinement method	Full matrix least squares on F^2
Goodness of fit on F^2	1.009
$R_1, wR_2 [I > 2\sigma (I)]$	0.0648, 0.1607
R_1, wR_2 (all data)	0.0951, 0.1963

complex in methanol, was recorded in the region of 700–200 nm using a Intra 10 UV–Visible spectrophotometer with a 1 cm quartz cell paths length are shown in Fig. 1. ¹H NMR spectra of the reactants as well as the crystal of $[(OPD)^+(TART)^-]$ were recorded in DMSO on Bruker DRX-300 NMR spectrometer. The elemental analyses were carried out using an Elementar Vario EL III Carlo Erba 1108. The FTIR spectra were recorded employing spectroscopic 2020 FTIR spectrometer using the KBr pellet technique. A single crystal of the H-bonded compound was mounted on a glass capillary and data were collected using graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å) on Bruker SMART APEX CCD diffractometer at 293 K and the thermal analysis (TGA and DTA) were carried out under nitrogen atmosphere with a heating rate of 20 °C/min for TGA and DTA using Shimadzu model DTG-60H Thermal Analyzer.

The data integration and reduction were processed with SAINT [44] software. An empirical absorption correction was applied to the collected reflections with SADABS using XPREP [44]. All the structures were solved by the direct method using SIR-97 [45] and were refined on F^2 by the full-matrix least-squares technique using the SHELXL-97 [45] program package. A summary of crystal-lographic data collection and refinement parameters are given in Table 2.

3. Result and discussion

3.1. X-ray Crystallographic studies of H-bonded complex

The formation of the H-bonded complex has been confirmed by the single crystal X-ray studies. The X-ray data exhibits that the OPD and TART are joined through various H-bonding interactions and the crystal lattice is costabilized via pi-pi interaction which exist between parallel aromatic rings of OPD.

The structure of the complex clearly indicates that the N1 of one of the NH₂ groups of OPD is protonated and strong hydrogen bonding interaction is formed between N1 with O5 of TART (N1–H2–O5 = 2.711 Å). The other N2 of NH₂ group of OPD is also bonded

Table 3

Selected bond lengths (Å), bond angles (°) and torsion angles (°) in crystal structure of CT complex.

Bond	Distance	Bond	Distance
N2 C2	1.416(4)	C5 C4	1.381(4)
O6 C10	1.265(4)	C9 C10	1.531(4)
O4 C9	1.425(4)	C2 C3	1.385(4)
O2 C7	1.216(4)	C2 C1	1.398(4)
O5 C10	1.246(4)	C4 C3	1.382(5)
O3 C8	1.413(4)	C3 H3	0.9300
01 C7	1.302(4)	N1 C1	1.462(4)
C8 C9	1.521(4)	C6 C5	1.386(4)
C8 C7	1.532(4)		
C6 C1	1.387(4)		
Bond	Angle	Bond	Angle
O3 C8 C9	111.8(2)	C4 C5 C6	118.8(3)
O3 C8 C7	109.2(2)	O4 C9 C8	110.2(2)
C9 C8 C7	109.9(2)	O4 C9 C10	110.6(2)
C5 C6 C1	120.4(3)	C8 C9 C10	111.9(2)
C5 C4 C3	120.9(3)	C3 C2 C1	117.7(3)
C4 C3 C2	121.2(3)	C3 C2 N2	120.3(3)
C6 C1 C2	121.0(3)	C1 C2 N2	122.0(3)
C6 C1 N1	118.4(3)	C2 C1 N1	120.6(3)
Bond	Torsion Angle	Bond	Torsion Angle
C1 C6 C5 C4	-0.2(4)	C5 C4 C3 C2 C1	-0.2(5)
O3 C8 C9 O4	-64.3(3)	C2 C3 C4 N2 C2	-0.4(4)
C7 C8 C9 O4	57.1(3)	C3 C4 C5 C6 C1	-177.6(3)
O3 C8 C9 C10	59.2(3)	C2 C5 C6 C1 N1	-0.5(4)
C7 C8 C9 C10	-179.4(3)	C3 C2 C1 C6	-180.0(3)
C6 C5 C4 C3	0.6(4)	N2 C2 C1 C6	0.8(4)
N2 C2 C1 N1	-2.6(4)	C3 C2 C1 N1	177.9(3)
03 C8 C7 02	-4.4(4)	C9 C8 C7 O1	-179.7(3)
C9 C8 C7 O2	-127.4(3)	O4 C9 C10 O5	52.6(3)
03 C8 C7 01	175.5(2)	C8 C9 C10 O5	-3.3(4)
C8 C9 C10 O6	53.7(4)		-126.6(3)
O4 C9 C10 O6	177.0(3)		

Table 4 H-bond geometries	s for the CT compl	ex (Å, °).	
D-H···A	D-H	H···A	D···A

D–H···A	D-H	H···A	D···A	D−H···A
N1-H3···O4	0.934	1.924	2.845	168.47
01-H1106	1.033	1.457	2.489	177.06
N1–H2···05	0.979	1.749	2.711	166.43
03–H8…N2	0.859	1.944	2.801	175.02

the O3 of the alcoholic (OH) group of TART to (N2–H8–O3 = 1.944 Å) The ORTEP view, hydrogen bonding packing diagrams and extended hydrogen bonding network are shown in Figs. 6–8, respectively. The crystal data and refinement parameters are provided in Table 2, and selected bond lengths, bond angles and H-bond geometries for the H-bonded complex are given in Tables 3 and 4. These hydrogen bonding interactions are comparable or even stronger to those reported for proton transfer complexes [46–48] exhibiting the high stability of the H-bonded complex. It is clear from the crystal structure that two rings are parallel to each other and provided ring to ring (pi-pi) interaction which also consolidates the structure of H-bonded complex. Besides the intermolecular H-bonding, there also exists some intramolecular Hbonding forces viz. 05...H9-O4 = 2.118 Å, 01-H11...O2 = 2.420 Å and (N1···H6···N2 = 2.568 Å).

3.2. Observation of CT bands

The spectrophotometric data were used to calculate both formation constant (K_{CT}), and extinction coefficient (ε_{CT}) of the H-bonded complex in the defined solvent based on Benesi–Hildebrand equation [49,50].

$[A]_{o}/A = 1/K_{CT}\varepsilon_{CT} \cdot 1/[D]_{o} + 1/\varepsilon_{CT}$

where $[D]_0$ and $[A]_0$ are the initial concentration of the donor and acceptor, respectively and A is the absorbance of the CT band. The concentration of the donor in the reaction mixture was kept greater than acceptor, $([D]_o) \gg [A]_o$ [49,50], and changed over a wide range of concentration from 1.5×10^{-4} M to 15×10^{-4} M while concentration of the acceptor (TART) in each of the reaction mixture was kept fixed at 1.0×10^{-4} M. These produced solutions with donor: acceptor molar ratios varying from 1.5:1 to 15:1 are reported in Table 1. The electronic absorption spectra of 1×10^{-4} M OPD, 10⁻⁴ M TART and H-bonded complex were recorded against methanol as references are shown in Fig. 1. To obtain the electronic absorption spectra of H-bonded complex, solutions of donor and acceptor were prepared in the same solvents. The straight line was obtained when $[A]_0/A$ was Plotted against $1/[D]_0$ which supports the formation of the charge transfer complex as shown in Fig 2 [49,50]. In this plot, the slope and intercept equals $1/K_{CT} \varepsilon_{CT}$ and $1/\varepsilon_{CT}$ respectively. It was observed that the correlation coefficient (r) value for the straight line was found to be 0.999. The formation constant and molar extinction coefficient of the H-bonded complex were obtained by calculation of intercept on y-axis and slope, and were found to be $1.337 \times 10^3 \,\mathrm{l}\,\mathrm{mol}^{-1}$ and $2.842 \times 10^5 \, l \, cm^{-1} \, mol^{-1}$ respectively. It is observed that new absorption peaks appear in UV region. In some cases multiple peaks were obtained and the longest wavelength peak was considered as H-bonded peak [51-53], which is found at 270 nm. The change of the absorption intensities to higher values for complex on addition of the donor are reported in Table 1. These measurements are based on the H-bonded absorption bands in the spectra of the systems and are mentioned in Figs. 1 and 2.

3.3. FTIR spectra

The FTIR absorption spectra of the o-phenylenediamine, L-tartaric acid and their CT complex employed in the range 4000– 400 cm⁻¹ frequency are shown in Fig. 3. The FTIR spectrum of the H-bonded complex shows the characteristic peaks of the free donor and acceptor, except some peaks being disappeared and some shift to the lower side of frequencies. These FTIR assignments are based on the comparison of the spectrum of the formed H-bonded complex with the spectra of the free acceptor and donor. The formed H-bonded complex shows changes in their band intensities as well as in the values compared to those of the free OPD and TART shifts in the frequencies. The formation of H-bonded complex between



Fig. 3. FTIR of (A) o-phenylenediamine (OPD); (B) ι -tartaric acid (TART); and (C) charge transfer complex of o-phenylenediamine and ι -tartaric acid.



Fig. 4. ¹H NMR spectrum of charge transfer complex of o-phenylenediamine with Ltartaric acid.



Fig. 5. TGA–DTA curves of charge transfer complex of o-phenylenediamine (donor) and L-tartaric acid (acceptor).

donor and acceptor is strongly evidenced by the presence of the main characteristic infra red bands of the OPD and TART in the spectrum of the product. However, the bands of the donor and acceptor are shifted. It is observed that the proton of one of COOH from TART migrated to the nitrogen atom of amine groups of o-phenylenediamine, which contains lone pair of electrons. The intensity of COOH peak was reduced in spectrum of H-bonded complex, which appears at 1600 cm^{-1} (strong) whereas this was observed at 1734 cm⁻¹ (strong) in free TART. The phenolic v (OH) vibrations of the free acceptor (TART) occur with medium and broad band range $3416-3300 \text{ cm}^{-1}$ whereas this was observed at 3422 cm^{-1} (weak) in the spectrum of the H-bonded complex and this new peak is also assigned to the N⁺-H-O⁻ species between the nitrogen atom of one of two NH₂ group of the donor and oxygen of acceptor. This is due to the ring current influence of the magnetic anisotropy of nitrogen atoms and the field effect of the electronic dipoles located on the nitrogen atoms. However, the stretching vibrations of v (N-H) and δ (N–H) appear at (3368 cm⁻¹), medium and strong, (1592 cm⁻¹) strong in free donor, respectively. It can be said that the donation process has been carried out by -NH₂ group in case of TART. This behavior is in accordance with the proton migration from the acceptor to the donor [54,55], which gives an additional



Fig. 6. ORTEP view of crystal of $[(OPD)^{+}(TART)^{-}]$ CT complex showing the numbering and bonding length scheme.



Fig. 7. Packing diagram, in which molecules are bonded through hydrogen bonding between C-H \cdots O and also pi-pi interaction.

evidence for the involvement of the nitrogen atoms of the donor and the COOH group of TART.

These changes strongly support the formation of H-bonded complex and could be attributed to the expected symmetry and electronic structure modifications in both donor and acceptor units in the formed product relative to the free molecules.

3.4. ¹H NMR Spectra

The ¹H NMR spectrum of the proton-transfer complex recorded in DMSO is given as Fig. 4. The peak corresponding to the proton of the solvent (i.e., DMSO) is present at δ = 2.00 ppm in the spectrum of the H-bonded complex. All the protons of OPD and TART indicate their signals at the appropriate positions in the spectrum of the Hbonded complex. The aromatic protons of the protonated OPD moiety appear at δ = 6.82 and 6.72 ppm whereas in free OPD the same were observed at δ = 6.651 ppm while those corresponding to $-NH_3^+$ group of the OPD occur at δ = 3.30 ppm indicating protonation of the donor. The proton transfer occurs from -COOH group of the TART to the -NH₂ group of the OPD part as is clear from the crystal structure of the H-bonded complex. This type of transfer is similar to that reported in earlier work [46,56]. The resonance signal due to the -NH₂ group in OPD is observed at 5.17 ppm. The additional peaks at 7.9 ppm are indicative of the OH group which is undisturbed in the TART part. The peak due to the undissociated -COOH function in TART appears at 2.5 ppm. It was concluded that the above studies are in line with the crystallographic investigation.

3.5. Study of thermogram of the H-bonded complex of ophenylenediamine and L-tartaric acid

Thermogravimetric (TG) and differential thermogravimetric (DTG) analysis were carried out for H-bonded complex of OPD and TART under on N_2 flow in order to confirm its formula, structure and thermal stability. The thermogram for H-bonded complex is depicted in Fig. 5. The degradation data clearly support the proposed molecular structure of the H-bonded complex. However, it is difficult to propose a definite decomposition reaction.

It was observed that the thermal decomposition of the Hbonded complex of OPD and TART exhibits various degradation steps differing in weight loss. The thermogram for the H-bonded complex (Fig. 5) shows two stages of decomposition. The first step occurs within the range 150–250 °C which corresponds to 31.78% weight loss of the acceptor (TART). The second step is the decomposition of the donor (OPD) which occurs within the range 280– 500 °C with weight loss 67.37% as decomposition of OPD in its CT complex, with picric acid [56]. It was also observed that H-bonded complex has been completed with endothermic process, as indicated by absorption of 45.99 and 109.07 J/g in the 1st and 2nd steps of its degradation, respectively. This has been attributed that the H-bonded complex is more stable than its constituents.



Fig. 8. H-bonding scheme in extended structure of [(OPD)⁺(TART)⁻] CT complex.

4. Conclusions

The formation of 1:1 hydrogen bonded proton or H-bonded complex of OPD and TART was studied spectrophotometrically in methanol as well as in solid phase at room temperature. The formation constant and molar extinction coefficient of the H-bonded Complex, were estimated employing Benesi-Hildebrand equation, and also, the stoichiometry of the H-bonded complex was found to be 1:1 consistent with the formula [(OPD)⁺(TART)⁻]. FTIR, ¹H NMR and electronic absorption spectra provide evidence for the existence of new bands of the H-bonded complex, which indicate a H-bonded interaction associated with proton migration from the acceptor to the donor followed by intermolecular hydrogen. X-ray data confirm the formation of 1:1 H-bonded complex and presence of extensive hydrogen bonding interactions. TGA-DTA studies show thermal stability of the H-bonded complex as well as show good agreement with the proposed stoichiometry by elemental analysis.

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