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Heats of dissociation and hydrolysis of Doxycycline hydrochloride and Salbutamol sulphate: A conductometric and pH-metric study

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Abstract

Conductometric and pH-metric studies on aqueous solutions of Doxycycline hydrochloride and Salbutamol sulphate reveal two modes of variation with temperature. In each mode both dissociation and hydrolysis occur simultaneously but the overall process is exothermic in one mode and endothermic in the other. The endothermic dissociation process dominates over the exothermic hydrolysis process in one mode and the reverse trend is observed in the other. For both the drugs transition from one mode to the other occurs at about 295K. Dissociation Constants of these drugs have been determined with various concentration and temperature.

Keywords: Doxycycline hydrochloride, Salbutamol sulphate, conductance, degree of dissociation, Heat of reaction, Hydrolysis constant.

Introduction

Doxycycline hydrochloride (**D**) is especially suitable for the treatment of a typical pneumonia caused by Legionella, Chlamydia or Mycoplasma and diseases caused by Borrelia and Rickettsiae (Elston, D. M., 2010). An ion-complexed doxycycline as an antibiotic prepared by simple mixing of positively charged doxycycline hyclate and negatively charged multivalent Na_2HPO_4 ($2Na^+ + HPO_4^{2-}$) in aqueous solutions (Oh, et al., 2016). Overall, its main application is in the treatment of respiratory and urinary tract infections. Salbutamol Sulphate (**S**) is typically used to treat bronchospasm (due to any cause, allergen asthma or exercise-induced), as well as chronic obstructive pulmonary disease (Ahee, P. 2000). Physicochemical properties of a drug molecule in solution determine the mechanism of action of the drug (Angelakis, et al., 2015). Investigation of thermodynamic properties (Alberty et al., 2011) of drug molecules in solution is of importance in pharmacokinetics. Thus, the effect of temperature on the structure and hydration of sodium cloxacillin and sodium dicloxacillin penicillins in aqueous solution have been examined by measurements of molal volumes and adiabatic compressibilities (Taboada et al., 2003). Recently it is found that mesoporous silica (SiO_2) materials behave as adsorbent of the antibiotic doxycycline at several values of pH, temperature, ionic strength (Brigante and Avena, 2016). Drug **D** has been shown to form charge transfer complexes with a number of electron acceptors in aqueous ethanol medium and the complexation process has been shown to occur after salt hydrolysis of the drug molecule (Saha and Mukherjee, 2004). The surface physicochemical properties of two anionic penicillinscloxacillin and dicloxacillins in mixed ethanol-water solvent were investigated by surface tension and dynamic light scattering (Barbosa et al., 2006). Recently, Absolute Rate Constants have been determined for Hydroxyl-Radical-Induced Degradative Oxidation of *β*-Lactam Antibiotics in Water (Dail and Mezyk, 2010) and Excitation Energy Transfer in Europium Chelate with

Doxycycline in the presence of a Second Ligand has been studied in Micellar Solutions of Nonionic Surfactants (Smirnova et al., 2011). Using modulated temperature differential scanning calorimetry (MTDSC) and thermogravimetric analysis (TGA), the effect of water uptake on the glass transition of spray dried and milled salbutamol sulphate has been performed (Grisedale et al., 2012). The Physicochemical and in vitro deposition properties of spray dried Drug **S** (Muddle et al., 2015) and the spray dried drug/excipient mixtures Drug **S** /lactose and Drug **S** /PEG have been investigated (Corrigan et al., 2006). Recently experimental solubilities of salbutamol and salbutamol sulphate in ethanol-water mixtures at 25 °C have been studied (Ali et al., 2012). Depending on temperature and concentration, the electrical conductance of the solutions can make conclusion about the role of solvent in the dissociation of the solvated substance, ion salvation, thermodynamics and kinetics of the ion reactions in the solutions, etc (Vlaev et al., 2007). Nasal delivery of salbutamol sulphate is used for therapeutic improvement (Salunke et al., 2016). These works indicate the importance of physicochemical studies on pharmaceutical molecules in vitro. Such studies are also important in removal of residual antibiotic from water (Avisar et al., 2010; Koyuncu et al., 2008; Hylton et al., 2009).

In the present work the variation of degree of dissociation (K_d) and degree of hydrolysis (K_h) of the two antibiotics e.g., Drug **D** and Drug **S** with temperature has been studied conductometrically and pH-metrically and related heats of the reaction have been analyzed.

Materials and Methods

Doxycycline hydrochloride and salbutamol sulphate (having molecular structures shown in Figure 1) were collected from Sigma and were used without further purification. All experiments were done on a microprocessor based conductivity meter from Systronics (model 306) and a microprocessor based pH meter from Elico (Model LI-614). Sample solutions were made in triple distilled water.

Results and Discussion

Conductance values of Doxycycline Hydrochloride and Salbutamol Sulphate solutions at different concentration and temperature are given in Tables 1 and 2. These two drug molecules (Fig. 1) behave as weak electrolytes. Using these data in the following rearranged form of the Ostwald dilution law, an apparent dissociation constant (K_{app}) and equivalent conductance at infinite dilution (Λ_0) could be determined.

$$\frac{1}{\Lambda} = \frac{1}{\Lambda_0} + \left(\frac{1}{K_{app}\Lambda_0^2}\right)\Lambda C \qquad \ldots(1)$$

where Λ is the equivalent conductance at concentration C. One typical plot is shown in Fig. 2

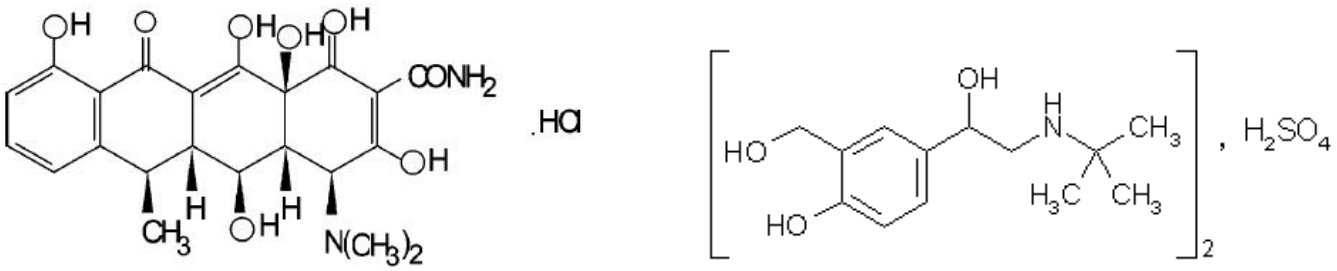
.

Table 1: Conductance of Doxycycline Hydrochloride solution at different concentration and temperature keeping cell constant of the electrode one.

Temp. (K)	Conductance of Doxycycline Hydrochloride (mS)						
	0.1(M)	**5 ò 10^{-2} (M)**	**2.5 ò 10^{-2} (M)**	**1.25 ò 10^{-2} (M)**	**6.25 ò 10^{-3} (M)**	**3.13 ò 10^{-3} (M)**	**1.56 ò 10^{-3} (M)**
288	7.415	4.665	2.936	1.718	1.059	0.6446	0.3706
289	7.470	4.701	3.006	1.748	1.084	0.6509	0.3799
290	7.550	4.756	3.077	1.795	1.105	0.6583	0.3894
291	7.605	4.813	3.138	1.820	1.127	0.6671	0.397
292	7.704	4.865	3.200	1.854	1.148	0.6774	0.4044
293	7.786	4.945	3.25	1.889	1.169	0.6888	0.4107
294	7.880	5.024	3.313	1.929	1.191	0.7010	0.4178
295	7.980	5.108	3.373	1.968	1.212	0.7129	0.4256
296	8.150	5.208	3.435	2.007	1.236	0.7261	0.4328
297	8.329	5.287	3.497	2.046	1.260	0.7401	0.4408
298	8.494	5.392	3.567	2.086	1.285	0.7536	0.4500
299	8.706	5.497	3.637	2.129	1.312	0.7692	0.4580
300	8.874	5.61	3.702	2.171	1.332	0.7827	0.4657
301	9.112	5.695	3.775	2.213	1.360	0.7969	0.4733
302	9.259	5.826	3.860	2.260	1.383	0.8121	0.4811
303	9.502	5.914	3.910	2.302	1.408	0.8266	0.4894

Table 2: Conductance of Salbutamol Sulphate solution at different concentration and temperature

Temp. (K)	Conductance of Salbutamol Sulphate (ms)						
	0.1(M)	5 ò 10^{-2} (M)	2.5 ò 10^{-2} (M)	1.25 ò 10^{-2} (M)	6.25 ò 10^{-3} (M)	3.13 ò 10^{-3} (M)	1.56 ò 10^{-3} (M)
288	7.675	5.500	3.325	1.885	1.016	0.5400	0.2503
289	8.164	5.763	3.362	1.962	1.033	0.5540	0.2528
290	8.470	5.944	3.418	1.999	1.056	0.5641	0.2568
291	8.941	6.210	3.480	2.038	1.082	0.5741	0.2628
292	9.279	6.417	3.555	2.072	1.102	0.5934	0.2670
293	9.462	6.550	3.640	2.105	1.119	0.6100	0.2738
294	9.761	6.744	3.727	2.154	1.146	0.6246	0.2800
295	9.985	6.901	3.817	2.197	1.169	0.6348	0.2870
296	10.098	7.002	3.906	2.240	1.189	0.6500	0.2933
297	10.283	7.138	3.996	2.286	1.213	0.6640	0.3005
298	10.376	7.232	4.088	2.327	1.239	0.6750	0.3069
299	10.591	7.387	4.183	2.370	1.270	0.6910	0.3145
300	11.044	7.663	4.286	2.423	1.304	0.7019	0.3220
301	11.061	7.726	4.391	2.469	1.331	0.7146	0.3295
302	11.266	7.872	4.478	2.515	1.355	0.7306	0.3355
303	11.493	8.031	4.569	2.577	1.380	0.7460	0.3426



(a) (b)

Fig. 1: Structure of (a) Doxycycline hydrochloride and (b) Salbutamol sulphate

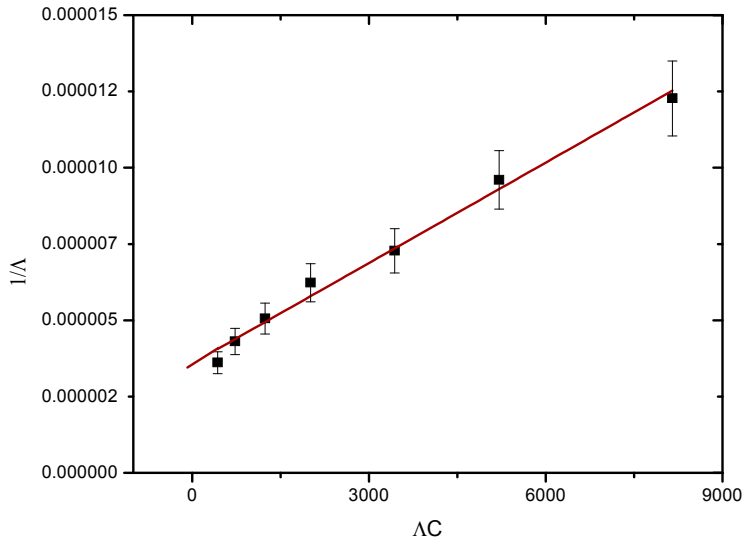


Fig. 2: Plot showing validity of Ostwald dilution law by Doxycycline hydrochloride at 296 K

To improve the precision of the calculation of Λ_0 and K_{app}, Eq.(1) was rewritten by Fuoss and Kraus as follows:

$$\frac{F(z)}{\Lambda} = \frac{1}{\Lambda_0} + \frac{\Lambda C \gamma_{\pm}^2}{F(z)\Lambda_0^2 K_{app}} \quad \text{.....(2)}$$

where $F(z)$ is the Fuoss function and $\gamma_{\pm}^2$ is the mean molar activity coefficient of the ions. This equation is recommended by some authors (Ergin et al., 1972; Izmailov et al., 1976; Barabanov et al., 1974; Sagulenko et al., 1997) for the determination of Λ_0 and K_{app} for weak electrolytes. Based on the values of Λ_0 calculated with Eq. (1), the values of the Fuoss function $F(z)$ were calculated using the following relation:

$$F(z) = 1 - \frac{S\sqrt{\Lambda C}}{\Lambda_0^{3/2}} \quad \text{.....(3)}$$

where $S = A\Lambda_0 + B$. The coefficients A and B depend on the nature of the solvent

$$A = \frac{8.204 \times 10^5}{(\varepsilon T)^{3/2}} \text{ and } B = \frac{82.501}{\eta (\varepsilon T)^{1/2}} \quad \text{....(4)}$$

where ε is the permitivity and η is the viscosity of the solvent at a given temperature T.

Using the resulting values of $F(z)$, the values of the degree of hydrolysis α and the mean molar activity coefficient of ions $\gamma_{\pm}^2$ were calculated for each concentration according to the equations:

$$\sim = \frac{\Lambda}{\Lambda_0 F(z)} \quad \text{.....(5)}$$

and

$$-\log_{10} \gamma_{\pm}^2 = 2 \quad (\sim C)^{1/2} \quad \text{...(6)}$$

where $2 = \frac{727.07}{\varepsilon^{3/2}}$ (Sagulenko et al., 1997). For the temperature dependence dielectric constant ε and the viscosity η of water, the following empirical equations were used (Vidulich et al., 1967; Robert et al., 1969; Korson 1969; Vlaev et al., 2007):

$$\log_{10}\varepsilon_{H2O} = 1.94409 - 1.991 \times 10^{-3} t \quad \text{.... (7)}$$

$$\log_{10}\left(\frac{\eta_t}{\eta_{20}}\right) = \frac{1.1709(20-t) - 0.00067(t-20)^2}{t + 96.71} \quad \text{....(8)}$$

where t is the temperature in degree Celsius.

Equations (5) and (6) give the values of α and $\gamma_{\pm}^2$ at different temperatures and concentrations of these drugs. The values of α decrease with an increase of both solution temperature and concentration due to the decrease of the values of ε and enhancement of the intermolecular interaction.

When the K_{app} values were plotted against the reciprocal of temperature, two linear branches were obtained for each of the drugs studied as shown in Fig. 3. Occurrence of the two branches can be explained by assuming that dissociation of the salt (drug molecules) and cation hydrolysis take place simultaneously:

$Z.H_mX \leftrightarrow Z.mH^+ + X^{m-}$ Dissociation const = K_d, Heat of reaction ΔH_d^0 ....(9)

$Z.mH^+ + mH_2O \leftrightarrow Z + mH_3O^+$ Hydrolysis const = K_h, Heat of reaction ΔH_h^0 .... (10)

where $Z.mH^+$ is the cationic part of doxycycline hydrochloride or salbutamol sulphate. Here the apparent dissociation constant is given by

$$K_{app} = K_h K_d \quad \text{.....(11)}$$

and the temperature variation is given by the well known van't Hoff equation:

$$\frac{d\ln K_d K_h}{dT} = \frac{d\ln K_{app}}{dT} = \frac{\Delta H_d^0 + \Delta H_h^0}{RT^2} = \frac{\Delta H_{app}^0}{RT^2} \quad \text{....(12)}$$

where ΔH_d^0 and ΔH_h^0 are the heats of dissociation and hydrolysis respectively. The former process is endothermic and the latter is exothermic. Hence depending on the relative magnitudes of the ΔH^0 quantities, the slope of the plot ln K_{app} vs. 1/T may be positive or negative as found in Fig. 3. The negative slopes of these figures indicate that K_d of the endothermic dissociation process changes more rapidly with temperature than the K_h of the exothermic hydrolysis constant. This trend persists up to a temperature of 295K above which the opposite trend follows for both the drugs. The individual values of K_d and K_h were obtained as follows. The degree of hydrolysis α, is obtained using eqn (5) at any concentration C. This is related to hydrolysis constant and pH by the following equation:

$$pH = pK_h + \log\frac{\sim}{1-\sim} \quad \text{.....(13)}$$

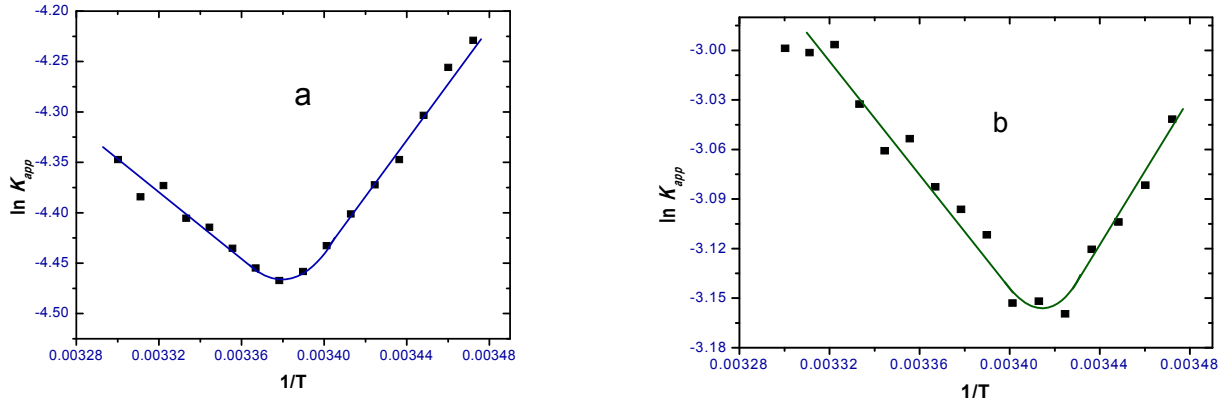


Fig. 3: Plots showing variation of K_{app} with reciprocal of temperature for (a) Doxycyclic Hydrochloride (b) Salbutamol Sulphate

Table 3: Dissociation constant, hydrolysis constant, apparent dissociation constants, Equivalent conductance, heats of dissociation and hydrolysis of the drug molecules in pure water

Drug	Temp	K_{App}	i_0 Mho cm^{-1} mol^{-1}	α 0.025 (M) soln	pH 0.025 (M) soln	K_d	K_h	ΔH_{app}^0 (kJ mol^{-1})	ΔH_d^0 (kJ mol^{-1})	ΔH_h^0 (kJ mol^{-1})
D	288	1.50×10^{-2}	228	0.516	2.57	5.23	2.87×10^{-3}	-32.0 ± 0.1	-34.0 ± 0.2	2.0 ± 0.2
	289	1.42×10^{-2}	235	0.512	2.56	4.90	2.89×10^{-3}			
	290	1.35×10^{-2}	242	0.508	2.55	4.64	2.91×10^{-3}			
	291	1.29×10^{-2}	249	0.504	2.54	4.41	2.93×10^{-3}			
	292	1.26×10^{-2}	255	0.502	2.53	4.23	2.98×10^{-3}			
	293	1.23×10^{-2}	261	0.498	2.52	4.09	3.00×10^{-3}			
	294	1.19×10^{-2}	268	0.490	2.50	3.84	3.10×10^{-3}			
	295	1.16×10^{-2}	275	0.493	2.49	3.70	3.13×10^{-3}			
	296	1.17×10^{-2}	279	0.492	2.48	3.64	3.21×10^{-3}	12.0 ± 0.3	-13.0 ± 0.2	25.0 ± 0.5
	297	1.14×10^{-2}	286	0.489	2.46	3.43	3.32×10^{-3}			
	298	1.19×10^{-2}	289	0.494	2.46	3.50	3.39×10^{-3}			
	299	1.21×10^{-2}	293	0.496	2.44	3.38	3.58×10^{-3}			
	300	1.22×10^{-2}	298	0.497	2.43	3.32	3.68×10^{-3}			
	301	1.26×10^{-2}	301	0.502	2.42	3.29	3.84×10^{-3}			
	302	1.25×10^{-2}	308	0.501	2.41	3.18	3.92×10^{-3}			
	303	1.29×10^{-2}	310	0.505	2.40	3.19	4.06×10^{-3}			
S	288	4.92×10^{-2}	182	0.722	5.97	1.81×10^{4}	2.72×10^{-6}	-19.0 ± 0.2	-28.0 ± 0.2	9.0 ± 0.4
	289	4.59×10^{-2}	188	0.714	5.96	1.67×10^{4}	2.74×10^{-6}			
	290	4.49×10^{-2}	192	0.711	5.95	1.63×10^{4}	2.75×10^{-6}			
	291	4.41×10^{-2}	197	0.708	5.94	1.59×10^{4}	2.78×10^{-6}			
	292	4.25×10^{-2}	202	0.703	5.93	1.52×10^{4}	2.79×10^{-6}			
	293	4.28×10^{-2}	206	0.706	5.92	1.48×10^{4}	2.89×10^{-6}	13.0 ± 0.3	-20.0 ± 0.1	33.0 ± 0.4
	294	4.27×10^{-2}	211	0.706	5.91	1.44×10^{4}	2.96×10^{-6}			
	295	4.45×10^{-2}	214	0.713	5.9	1.42×10^{4}	3.13×10^{-6}			
	296	4.52×10^{-2}	218	0.716	5.88	1.36×10^{4}	3.33×10^{-6}			
	297	4.58×10^{-2}	222	0.719	5.87	1.33×10^{4}	3.45×10^{-6}			
	298	4.72×10^{-2}	226	0.724	5.86	1.31×10^{4}	3.62×10^{-6}			
	299	4.69×10^{-2}	231	0.723	5.85	1.27×10^{4}	3.70×10^{-6}			
	300	4.82×10^{-2}	236	0.728	5.84	1.25×10^{4}	3.86×10^{-6}			
	301	5.00×10^{-2}	239	0.734	5.82	1.20×10^{4}	4.17×10^{-6}			
	302	4.97×10^{-2}	244	0.734	5.81	1.17×10^{4}	4.26×10^{-6}			
	303	4.99×10^{-2}	249	0.733	5.79	1.12×10^{4}	4.46×10^{-6}			

Thus K_h at any temperature can be obtained from the measured pH and conductance. Hence K_d is obtained from eqn. (11). Individual heats of reaction ΔH_d^0 and ΔH_h^0 were then determined using van't Hoff equation. Results are summarized in Table 3. It is to be noted that the sum of ΔH_d^0 and ΔH_h^0 equals ΔH_{app}^0 as expected from eqn (12).

Conclusion

Two steps of interaction of each drug molecules under consideration with water could be studied individually and the heats of dissociation and hydrolysis could be determined separately by conductometric and pH metric method.

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