The chelating behavior of urea complexed with copper (II), zinc (II), silver (I), cadmium (II) and mercury (II) ions at room temperature has been studied by elemental analyses, magnetic susceptibility, conductivity measurements, infrared and UV-visible spectroscopy. Some of the formed complexes show high values of molar conductivity, indicating these complexes are electrolytes in nature. The obtained complexes can be formulated as \( \text{CuCl}_2.2\text{U.CH}_3\text{OH}.4\text{H}_2\text{O} \), \( \text{ZnCl}_2.\text{U.CH}_3\text{OH}.4\text{H}_2\text{O} \), \( \text{AgNO}_3.\text{U.CH}_3\text{OH}.6\text{H}_2\text{O} \), \( \text{CdCl}_2.\text{U.CH}_3\text{OH}.4\text{H}_2\text{O} \) and \( \text{HgCl}_2.\text{U.CH}_3\text{OH}.6\text{H}_2\text{O} \).

**Keywords:** Chelating behavior of urea, Conductivity measurements, Magnetic susceptibility, Spectroscopic studies.

*Corresponding author*
INTRODUCTION

Carbamide, carbonyldiamide or the most famous name, urea (Scheme 1), CH$_4$N$_2$O, was first prepared by Wöhler [1] by evaporating a solution containing a mixture of potassium isocyanate and ammonium sulphate.

![Scheme 1: Structure of urea](image)

Industrially [2-4], urea is prepared by allowing liquid carbon dioxide and liquid ammonia to interact, and heating the formed ammonium carbamate at 130-150 °C under about 35 atmospheric pressure. The carbamate is decomposed to form urea and water according to the following reaction;

$$2\text{NH}_3 + \text{CO}_2 \rightarrow \text{NH}_2\text{COONH}_4 \rightarrow \text{CO(NH}_2)_2 + \text{H}_2\text{O}$$

Urea is physiologically very important. It is the chief nitrogenous product of protein metabolism. Adults excrete about 30g of urea per day in the urine, from which it can be extracted by evaporating the urine to small volume and adding nitric acid, to give the slightly soluble urea nitrate, CO(NH$_2$)$_2$.HNO$_3$. Urea has a melting point of 132°C, soluble in water and ethanol, but insoluble in ether. Urea is used for preparing formaldehyde-Urea resin (plastics) [5], barbiturates [6], and fertilizers [7-10]. Urea is also extensively used in the paper industry to soften cellulose and has been used to promote healing in infected wounds and many other applications in the field of medicine [11-13]. Recently, urea is used for the manufacture of hydrazine in which urea is treated with alkaline sodium hypochlorite [6] e.g.,

$$\text{NH}_2\text{CONH}_2 + \text{NaOCl} + 2\text{NaOH} \rightarrow \text{N}_2\text{H}_4 + \text{NaCl} + \text{Na}_2\text{CO}_3 + \text{H}_2\text{O}$$

Urea is used in reactive dyeing [14] which has an effect on the formation and cleavage of covalent bond between the reactive dye and cellulose. The overall effect of urea on reactive dyeing depends on the solvolytic stability of the dye-fiber bond under specific dyeing conditions. Complexes of urea with some metal ions are used as fertilizers [15-18]. Complexes of urea with zinc sulphate and nitrate, [Zn(CON$_2$H$_4$)$_6$]SO$_4$.H$_2$O and [Zn(CON$_2$H$_4$)$_4$](NO$_3$)$_2$.2H$_2$O have very important application in this field [19]. These complexes were found to increase the yield of rice more than a dry mixture of urea-zinc salt does. Calcium nitrate-urea complex, [Ca(urea)$_4$](NO$_3$)$_2$, [20,21] was used also as an adduct fertilizer. Some metal-urea complexes have pharmaceutical application, e.g., the platinum-urea complex which is used as antitumor [22]. Crystal structure studies have shown that in solid urea, both nitrogen atoms are identical. Bond length measurements [6] in urea give the C-N distance as 1.37Å, while, in aliphatic amines the C-N bond length is 1.47Å. This indicates that the C-N bond in urea has some double bond character (about 28%). Urea usually coordinates as a monodentate ligand through the oxygen atom, forming a C=O···M
angle considerably smaller than 180°, in accordance with the sp² hybridization of the O atom (A in Scheme 2). The rare N,O-bidentate coordination mode (B in Scheme 2) has been found in a very limited number of cases [23, 24], while in [Hg₂Cl₄U₂] each U molecule bridges the two Hg²⁺ atoms through the oxygen atom [25] (C in Scheme 2). Of particular chemical/biological interest is the ability of U to undergo metal-promoted deprotonation [26]; the monoanionic ligand H₂NCONH⁻ adopts the μ₂ (D in Scheme 2) and μ₃ (E in Scheme 2) coordination modes. The urea and its derivatives such as the N,N'-dimethylurea and N,N'-diethylurea (Scheme 2) have only been found to coordinate as monodentate ligands through the oxygen atom (F in Scheme 2).

![Scheme 2: The coordination modes of urea towards metal ions](image)

Raman and infrared spectra of urea have been observed by several investigators [27-32]. The normal vibrations of the urea molecule were calculated by Kellner [33] on the assumption of the non-planar model. However, on the basis of the dichronic measurement of the infrared band arising from the N-H stretching vibration by Waldron and Badger [34] and the proton magnetic resonance absorption measurement made by Andrew and Hyndman [35], it is concluded that urea molecule has a planar structure. Yamaguchi et al. [36] calculated the normal vibrations of the C₂ᵥ model of urea molecule as an eight-body problem using a potential function of the Urey-Bradley force field and obtained the force constants which have been refined by the least-squares method. Based on the result of these calculations, Yamaguchi [36], assigned all of the observed frequencies in the spectra of urea and urea-d₄. To the two vibrations of the frequencies 1686 and 1603 cm⁻¹, there are considerable contributions of both CO stretching and NH₂ bending motions, whereas Stewart [37] assigned the 1686 cm⁻¹ band to CO stretching vibration and the 1603 cm⁻¹ band to NH₂ bending motion. The calculations studied by Yamaguchi showed that for the band at 1686 cm⁻¹, the contribution of the NH₂ bending motion is greater than that of CO stretching motion. The band at 1629 cm⁻¹ corresponds to almost pure NH₂ bending vibration. The NH₂ bending motion of A₁ type is equal to that of B₂ type. The A₁ type band should have a frequency of about 1630 cm⁻¹, if there is no coupling between NH₂ bending and CO
stretching motions. On the other hand, the observed frequency of 1610 cm\(^{-1}\) of urea-d\(_4\) is assigned to almost pure skeletal vibration. Therefore, the interaction between the 1630 and 1610 cm\(^{-1}\) vibration gives rise to the two observed bands at 1686 and 1603 cm\(^{-1}\). The infrared bands of urea-d\(_4\) observed at 1245 and 1154 cm\(^{-1}\) are assigned, respectively, to A\(_1\) type and B\(_2\) type, ND\(_2\) bending vibrations. This assignment is consistent with the observed depolarization degrees of the Raman lines. The large frequency difference between the A\(_1\) and B\(_2\) vibrations is due to the fact that in the A\(_1\) vibration, the cross term related to the CN stretching vibration is large. The 1464 cm\(^{-1}\) frequency of urea is assigned to the CN stretching vibration of B\(_2\) type. The corresponding frequency of urea-d\(_4\) is observed at 1490 cm\(^{-1}\). The 1150 cm\(^{-1}\) band is assigned to NH\(_2\) rocking vibrations of both A\(_1\) and B\(_2\) types. The normal vibration calculation yields almost the same values for these frequencies. Urea possesses two types of potential donor atoms, the carbonyl oxygen and amide nitrogens. Penland et al. [38] studied the infrared spectra of urea complexes to determine whether coordination occurs through oxygen or nitrogen atoms. The electronic structure of urea may be represented by a resonance hybrid of structures A-F as shown in scheme 1 with each contributing roughly an equal amount. If coordination occurs through nitrogen, contributions of structure B will decrease. This results in an increase of the CO stretching frequency with a decrease of CN stretching frequency. The N-H stretching frequency in this case may fall in the same range as those of the amido complexes. If coordination occurs through oxygen, the contribution of structure (A) will decreases. This may result in a decrease of the CO stretching frequency but no appreciable change in NH stretching frequency. Since the vibrational spectrum of urea itself has been analyzed completely [36], band shifts caused by coordination can be checked immediately. For example, the effect of the coordination on the spectra of the complexes of urea with Pt(II) and Cr(III) in which the coordination occurs through oxygen and nitrogen atoms, respectively [38]. The mode of coordination of urea with metal ions seems to be dependent upon the type and nature of metal. Pd(II) coordinates to the nitrogen, whereas Fe(III), Zn(II), and Cu(II) coordinate to the oxygen of urea [38]. In urea-metal complexes, if a nitrogen-to-metal bond is present, the vibrational spectrum of this complex differs significantly from that of the free urea molecule. The N-H stretching frequencies would be shifted to lower values, and the C=O bond stretching vibration, (\(v(C=O)\)) would be shifted to higher frequency at about 1700 cm\(^{-1}\) [39]. Recently, urea represents not only an important molecule in biology [40] but also an important raw material in chemical industry [41]. This work aimed to prepare and investigate the resulting compounds formed from the reactions of urea with AgNO\(_3\), CrCl\(_3\).6H\(_2\)O, CdCl\(_2\).H\(_2\)O and ZnCl\(_2\) at room temperature.

MATERIAL AND METHODS

**Synthetic procedure**

Urea, CuCl\(_2\).2H\(_2\)O, ZnCl\(_2\), AgNO\(_3\), CdCl\(_2\).H\(_2\)O, HgCl\(_2\) and methanol were obtained from Aldrich Company. Urea was received from Fluka chemical company. All chemicals used in this study were of analytically reagent grade and used without further purification. The complexes, CuCl\(_2\).2U.CH\(_3\)OH.4H\(_2\)O (1), ZnCl\(_2\).U.CH\(_3\)OH. 4H\(_2\)O (2), AgNO\(_3\).U.CH\(_3\)OH.6H\(_2\)O (3), CdCl\(_2\).U.CH\(_3\)OH.4H\(_2\)O (4), HgCl\(_2\).U.CH\(_3\)OH .6H\(_2\)O (5) were prepared by mixing equal methanolic solutions of CuCl\(_2\).2H\(_2\)O (1.70 g, 0.01 mole), ZnCl\(_2\)(1.36 g, 0.01 mol), AgNO\(_3\) (1.70 g, 0.01 mole), HgCl\(_2\) (2.72 g, 0.01 mole), in 25 mL methanol with a 50 mL volume of urea.
solution (1.2 g, 0.01 mole) in methanol solvent. The mixtures were stirred for about 12 hours under refluxed system at room temperature Ca. 25 °C. The Ag(I), Cd(II) and Zn(II)-urea complexes took a longer time to precipitate. The amount of the formed precipitate increased with time of standing. The complexes formed were filtered off, dried under vacuo over anhydrous calcium chloride.

Characterization

The elemental analyses of carbon, hydrogen and nitrogen contents were performed by the microanalysis unit at Cairo University, Egypt, using a Perkin Elmer CHN 2400 (USA). The molar conductivities of freshly prepared 1.0×10⁻³ mol/cm³ dimethylsulfoxide (DMSO) solutions were measured for the soluble urea complexes using Jenway 4010 conductivity meter. Magnetic measurements were performed on the Magnetic Susceptibility Balance, Sherwood Scientific, and Cambridge Science Park-Cambridge-England. The infrared spectra with KBr discs were recorded on a Bruker FT-IR Spectrophotometer (4000–400 cm⁻¹). The electronic absorption spectra of urea complexes were recorded within 1000–200 nm range using UV2 Unicam UV/Vis Spectrophotometer fitted with a quartz cell of 1.0 cm path length.

RESULTS AND DISCUSSION

Reaction mechanism

Table 1 shows the color, physical characteristic, micro-analytical data and molar conductance measurements of Cu(II), Zn(II), Ag(I), Cd(II), Hg(II)-urea complexes. The elemental analysis data the prepared complexes revealed 1:1 molar ratio (M:U) for all complexes are in a good agreement with the general formulas CuCl₂.2U.CH₃OH.4H₂O (1), ZnCl₂.2U.CH₃OH.4H₂O (2), AgNO₃.2U.CH₃OH.6H₂O (3) CdCl₂.2U.CH₃OH.4H₂O (4) HgCl₂.2U.CH₃OH.6H₂O (5). The reactions can be represented by the stoichiometric equations:

\[
\text{CuCl}_2.2\text{H}_2\text{O} + 2\text{U} + \text{CH}_3\text{OH} \xrightarrow{25^0\text{C}} \text{CuCl}_2.2\text{U}.\text{CH}_3\text{OH}.4\text{H}_2\text{O} (1)
\]
\[
\text{ZnCl}_2 + 2\text{U} + \text{CH}_3\text{OH} \xrightarrow{25^0\text{C}} \text{ZnCl}_2.2\text{U}.\text{CH}_3\text{OH}.4\text{H}_2\text{O} (2)
\]
\[
\text{AgNO}_3 + 2\text{U} + \text{CH}_3\text{OH} \xrightarrow{25^0\text{C}} \text{AgNO}_3.2\text{U}.\text{CH}_3\text{OH}.6\text{H}_2\text{O} (3)
\]
\[
\text{CdCl}_2 + 2\text{U} + \text{CH}_3\text{OH} \xrightarrow{60^0\text{C}} \text{CdCl}_2.2\text{U}.\text{CH}_3\text{OH}.4\text{H}_2\text{O} (4)
\]
\[
\text{HgCl}_2 + 2\text{U} + \text{CH}_3\text{OH} \xrightarrow{25^0\text{C}} \text{HgCl}_2.2\text{U}.\text{CH}_3\text{OH}.6\text{H}_2\text{O} (5)
\]

The complexes are air-stable, hygroscopic, with low melting points, soluble in H₂O and dimethylsulfoxide, DMSO. The molar conductivities of the complex solution prepared in DMSO (10⁻³ g/5mL) (Table 1) indicates that some of the complexes have an electrolytic nature. The molar conductivity values for the urea complexes in DMSO solvent are in the range of (18–335) Ω⁻¹ cm² mol⁻¹, suggesting them to be electrolytes (Table 1). Conductivity measurements have frequently been used in structural of metal complexes (mode of coordination) within the limits of their solubility. They provide a method of testing the degree of ionization of the complexes, the molar ions that a complex liberates in solution (in
case of presence anions outside the coordination sphere), the higher will be its molar conductivity and vice versa. It is clear from the conductivity data that the complexes present seem to be electrolytes. Also the molar conductance values indicate that the anions present outside the coordination sphere. This result was confirmed from the elemental analysis data where Cl\(^{-}\) or NO\(_3\)\(^{-}\) ions are precipitated with colored solution by adding of AgNO\(_3\) or FeSO\(_4\) solutions, respectively, this experimental test is a good matched with CHN data. All these complexes have electrolytic properties. This fact elucidated that the Cl\(^{-}\) or NO\(_3\)\(^{-}\) are present. These results establish the stoichiometries of these complexes, which are in agreement with the general formulas, were suggested. The UV-visible spectra of the urea complexes are given in Table 2, which show the absorption bands of the metal ions toward urea.

**Magnetic measurements**

Magnetic measurements were carried out on a Sherwood Scientific magnetic balance according to the Gauy method. The calculations were evaluated by applying the following equations:

\[
\chi_m = \chi_s \frac{c(R - R_o)}{10^9 M}
\]

\[
\mu_{eff} = 2.828\sqrt{\chi_m T}
\]

where \(\chi\) is mass susceptibility per gm sample

- \(c\) is the calibration constant of the instrument and equal to 0.0816
- \(R\) is the balance reading for the sample and tube
- \(R_o\) is the balance reading for the empty tube
- \(M\) is the weight of the sample in gm
- \(T\) is the absolute temperature

The magnetic moments of the CuCl\(_2\).U.CH\(_3\).4H\(_2\)O (1) complex at \(T = 300\) K and their corresponding hybrid orbitals were calculated. The observed values of the effective magnetic moments \(\mu_{eff}\) measured for the complex was 1.81 B.M., this is in convenient with experimental value of 1.85 B.M. obtained for planar Cu(II) complex with \(dsp^2\) hyperdization [42].

**IR spectra**

The infrared spectra of the Cu(II), Zn(II) Ag(II), Cd(II), Hg(II) and urea complexes at room temperature are shown in Fig. 1. The band locations were measured for the mentioned urea complexes, together with the proposed assignments for the most characteristic vibrations are presented in Tables 3 and 4. In order to facilitate the spectroscopic analysis and to put our hand on proper structure of the prepared complexes, the spectra of the urea complexes were accurately compared with those of the urea and similar complexes in literature survey. The assignments of full vibrational analysis of crystalline urea have been published [38]. Tables 3 and 4 give diagnostic infrared peaks of the free urea ligand, published work and Ag(I), Cr(III), Cd(II) and Zn(II) complexes. Assignments have been given in comparison with the data obtained for the free urea, that is, uncoordinated, U [38] and its [Pt(urea)\(_2\)Cl\(_2\)] and [Cr(urea)\(_6\)]Cl\(_3\) complexes [38]. The effect of the coordination on the spectra of the complexes of urea with [Pt(urea)\(_2\)Cl\(_2\)] and
[Cr(urea)$_2$]Cl$_3$ complexes in which the coordination occurs through nitrogen and oxygen atoms, respectively [38]. The mode of coordination of urea with metal ions seems to be dependent upon the type and nature of metal. Pd(II) ions in [Pt(urea)$_2$Cl$_2$] coordinate to the nitrogen, whereas Fe(III), Zn(II), and Cu(II) coordinate to the oxygen of urea [38]. The distinguished bands of $\nu$(C=N) are exhibited at lower wavenumbers in the spectra of AgNO$_3$.U.CH$_3$OH.6H$_2$O (3) complex than for free urea, whereas the $\nu$(C=O) band shows a frequency at nearly the same wavenumber of free urea. These shifts are consistent with nitrogen coordination, suggesting the presence of $+N=C\cdots O$– resonance features [43], see Scheme 3.

Concerning, CuCl$_2$.U.CH$_3$OH. 4H$_2$O (1), ZnCl$_2$.U. CH$_3$OH .4H$_2$O (2), CdCl$_2$. U. CH$_3$OH.4H$_2$O (4)HgCl$_2$. U. CH$_3$OH .6H$_2$O (5) complexes, the coordination mode take place via oxygen of amide group, the positively charged metal ion stabilizes the negative charge on the oxygen atom; the NCO group now occurs in its polar resonance form and the double bond character of the CN bond increases or still not affected, while the double bond character of the CO bond decreases, resulting in an increase of the CN stretching frequency with a simultaneous decrease in the CO stretching frequency [43, 44]. The one IR-active $\nu$(M–O) vibration of Cr(III), Cd(II) and Zn(II) urea complexes presence at 442, 473, and 443 cm$^{-1}$ [43, 44]. The band related to the stretching vibration $\nu$(O-H) of uncoordinated H$_2$O is observed in the range of (3400-3450) cm$^{-1}$, while the corresponding bending motion of the uncoordinated water, $\delta$(H$_2$O), is observed in the range of (1630-1638) cm$^{-1}$. In silver complex, the coordination mode take place via nitrogens, of amide group the characteristic stretching vibrations of the nitrate group, NO$_3^-$, is observed at around (1385 and 1160 cm$^{-1}$ attributed to $\nu$_s(NO$_2$) and $\nu$_(NO$_2$), respectively [46]. The stretching motion of (\nu(N=O)) is observed at 1475 cm$^{-1}$ as a strong band, while the bending motion of the type $\delta$(NO$_2$) are well resolved and observed at 786 as a medium band.

**Table 1:** Physical characterization, micro-analytical data of urea complexes

<table>
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<tr>
<th>Complexes</th>
<th>Empirical formula (MW.)</th>
<th>Color</th>
<th>Molar ratio</th>
<th>Elemental analysis (%)</th>
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<th>Calcd.</th>
</tr>
</thead>
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<td></td>
<td></td>
<td></td>
<td>C</td>
<td>H</td>
<td>N</td>
</tr>
<tr>
<td>CuCl$_2$.U.Me.4H$_2$O (1)</td>
<td>298.48 g/mol</td>
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<td>08.36 (08.04)</td>
<td>04.83 (05.36)</td>
<td>09.14 (09.36)</td>
</tr>
<tr>
<td>ZnCl$_2$. U.Me.4H$_2$O (2)</td>
<td>300.29 g/mol</td>
<td>White</td>
<td>1:1</td>
<td>08.19 (07.99)</td>
<td>04.66 (05.33)</td>
<td>9.88 (09.32)</td>
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<tr>
<td>AgNO$_3$.U.Me.6H$_2$O (3)</td>
<td>369.87 g/mol</td>
<td>White</td>
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<td>06.51 (06.49)</td>
<td>04.83 (05.41)</td>
<td>08.31 (07.57)</td>
</tr>
<tr>
<td>CdCl$_2$. U.Me.4H$_2$O (4)</td>
<td>347.32 g/mol</td>
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<td>1:1</td>
<td>07.27 (06.91)</td>
<td>04.31 (04.61)</td>
<td>08.19 (08.06)</td>
</tr>
<tr>
<td>HgCl$_2$.U.4Me.6H$_2$O (5)</td>
<td>471.50 g/mol</td>
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<td>05.25 (05.09)</td>
<td>04.09 (04.24)</td>
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Table 2: Molar conductance and magnetic moment data of urea complexes

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<th>Complex</th>
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<th>Electronic transition</th>
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<td></td>
<td>Bands</td>
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<td>233</td>
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<tr>
<td>AgNO₃.U.Me.6H₂O (3)</td>
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<td>-</td>
<td>232</td>
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<td>CdCl₂. U.Me.4H₂O (4)</td>
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<td>235</td>
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<tr>
<td>HgCl₂.U.4Me.6H₂O (5)</td>
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<td>-</td>
<td>238</td>
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</table>

Table 3: Characteristic infrared frequencies (cm⁻¹) and tentative assignments of urea (U), [Pt(urea)₂Cl₂] (A), [Cr(urea)₆]Cl₃ (B), CuCl₂.U.Me.4H₂O (1), ZnCl₂.U.Me.4H₂O (2) and AgNO₃.U.Me.6H₂O(3) complexes.

<table>
<thead>
<tr>
<th>U</th>
<th>A</th>
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<th>3</th>
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<tr>
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<td></td>
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<td>υ(CH₃)CH₂OH</td>
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<tr>
<td>1683</td>
<td>1725</td>
<td>1505</td>
<td>1622</td>
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<td>1505</td>
<td>1471</td>
<td>1489</td>
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Table 4: Characteristic infrared frequencies (cm⁻¹) and tentative assignments of urea (U), [Pt(urea)₂Cl₂] (A), [Cr(urea)₆]Cl₃ (B), CdCl₂.U.Me.4H₂O (4) and HgCl₂.U.4Me.6H₂O (5) complexes.

<table>
<thead>
<tr>
<th>U</th>
<th>A</th>
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<th>5</th>
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<td>1505</td>
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<td>ν(C–N)</td>
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Fig. 1: Infrared spectra of urea complex at room temperature: CuCl₂·U.Me.4H₂O (1), ZnCl₂·U.Me.4H₂O (2), AgNO₃·U.Me.6H₂O (3), CdCl₂·U.Me.4H₂O (4), HgCl₂·U.4Me·6H₂O (5)
REFERENCES