THERMAL STABILITY AND KINETIC STUDIES OF COBALT (II), NICKEL (II), COPPER (II), CADMIUM (II) AND MERCURY (II) COMPLEXES DERIVED FROM N-SALICYLIDENE SCHIFF BASES

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ABSTRACT

The thermal properties of a number of Schiff base metal complexes in nitrogen atmosphere have been investigated by thermogravimetry (TG) and differential thermogravimetry (DTG). The kinetic analyses of the thermal decomposition were studied using the Coats-Redfern and Horowitz-Metzger equations. The obtained kinetic parameters show the kinetic compensation effect.

Keywords: Metal Schiff base complexes, thermogravimetry, thermal stability.

INTRODUCTION

Schiff bases have played and continue to play an important role in the development of coordination chemistry. Schiff bases and their metal complexes are becoming increasingly important in recent years due to their biological activity and their uses as catalysts and as compounds of interesting photoluminescent and electroluminescent properties.

In spite of the relatively large number of thermal analysis reports on Schiff base metal complexes, less work was published on the calculation of the kinetic and thermodynamic parameters of their decomposition.

In this paper we report on the kinetics of the thermal decomposition and the accompanying compensation effect for a number of Schiff base complexes of Co(II), Ni(II), Cu(II), Cd(II) and Hg(II).

EXPERIMENTAL

All chemicals were of analytical grade.

Aqua (N-salicylideneanthranilinato)cobalt(II)dihydrate, aqua (N-salicylideneanthranilinato)nickel(II), aqua(N-salicylideneanthranilinato) copper(II), aqua(N-salicylidene-o-iminophenolato)cobalt(II)dihydrate, aqua(N-salicylidene-o-iminophenolato)nickel(II), aqua(N-salicylidene-o-iminophenolato) copper(II), dichloro-(N-salicylidene-o-toluidine)mercury(II) were prepared according to literature procedures. Thermogravimetric analyses were performed on a Shimadzu TGA-50 H thermal analyser in a dynamic nitrogen atmosphere (20 mL min⁻¹) at a heating rate of 10°C min⁻¹.

RESULTS AND DISCUSSION

The thermal decomposition data of the compounds are collected in Tables 1 and 2. The complexes are thermally relatively stable and their thermal decompositions are multistage processes. The complexes are subjected to a TG analysis from 50-700°C in nitrogen atmosphere.

Aqua(N-salicylideneanthranilinato)cobalt(II)dihydrate, [Co(SAA)(H₂O)] · 2H₂O

The complex undergoes decomposition in three steps in the temperature ranges 50-140, 145-270 and 275-460°C (Fig. 1). The first step is in accord with the elimination of two water molecules (calc. 10.2%, found 10.5%) and the corresponding activation energy amounts to 45.3 kJ/mol. The DTG peak of this hydration step is symmetrical, indicating that the two water molecules are isoenergetically bonded and are lost in one step. The second step (calc. 5.1%, found 5.5%) and the third step (26.5%) correspond to the loss of the third water molecule together with unidentified decomposition products, indicating that the third water molecule is more tightly bonded to the cobalt(II). The second step has a narrow temperature range which prevents its kinetic analysis. For the third step the computed activation energy amounts to 133.5 kJ/mol. Cobalt carbonate is supposed to be formed at the end of this step (calc. 33.7%, found 34.0%).

\[ \text{[Co(SAA)(H₂O)] · 2H₂O} \xrightarrow{50-140°C} \text{[Co(SAA)(H₂O)]} + 2H₂O \quad (1) \]

Aqua(N-salicylideneanthranilinato)nickel(II), [Ni(SAA)(H₂O)]

Degradation of this complex takes place in three successive steps at 69-125, 125-275 and 340-500°C. The first decrease in mass corresponds closely to the detachment of one water molecule (calc. 5.7%, found 6.5%). This step has an activation energy value of 62.1 kJ/mol. The second step has an activation energy of 105.6 kJ/mol.

\[ \text{[Ni(SAA)(H₂O)]} \xrightarrow{69-125°C} \text{[Ni(SAA)] + H₂O} \quad (2) \]

The third mass loss of 29.5% is characterized by an activation energy of 169.5 kJ/mol. The second step of mass loss 4.0% is a slow small step. The third step of 29.0% is characterized by an activation energy of 169.5 kJ/mol. The fourth step of mass loss 31.5% is composed of three overlapping steps as indicated from its DTG peak. The mass loss consideration indicated that the end product should be CuO (calc. 24.8%, found 25.0%).

\[ \text{[Cu(SAA)(H₂O)]} \xrightarrow{65-150°C} \text{[Cu(SAA)] + H₂O} \quad (3) \]

Aqua(N-salicylidene-o-iminophenolato)cobalt(II)dihydrate, [Co(SOP)(H₂O)] · 2H₂O

The thermal decomposition of the complex involves three decomposition stages well discernible in its TG curve in the temperature ranges 50-152, 215-375 and 375-477 °C. The first decomposition step corresponds to the release of two water molecules (calc. 11.1%, found 11.3%) with activation energy of 57.3 kJ/mol. The second step of mass loss 13.0% is a slow small step. The third step of mass loss 29.0% is characterized by an activation energy of 169.5 kJ/mol. The fourth step of mass loss 31.5% is composed of three overlapping steps as indicated from its DTG peak. The mass loss consideration indicated that the end product should be CuO (calc. 24.8%, found 25.0%).

\[ \text{[Co(SOP)(H₂O)]} \xrightarrow{50-152°C} \text{[Co(SOP)(H₂O)]} + 2H₂O \quad (4) \]

Aqua(N-salicylidene-o-iminophenolato)nickel(II), [Ni(SOP)(H₂O)]

The complex undergoes decomposition in three steps in the temperature ranges 98-203, 205-305 and 355-450°C. The mass loss during the first step is associated with the loss of one water molecule (calc. 6.2%, found 7.1%). The second step corresponds to a mass loss of 13.0% and its activation energy amounts to 62.1 kJ/mol. The third step with a mass loss of 38.0% has an activation energy of 105.6 kJ/mol.

\[ \text{[Ni(SOP)(H₂O)]} \xrightarrow{98-203°C} \text{[Ni(SOP)] + H₂O} \quad (5) \]
**Dehydration of a hydrate compound includes more than one constituent water in hydrates is usually regarded as complex.**

The Toluidine radical may abstract hydrogen to afford toluene or dimerize to radical (II) corresponding to the expulsion of two Toluidine radicals leaving the complex (III). The Toluidine radical may abstract hydrogen to afford toluene or dimerize to give the diphenyl derivative (Scheme A).

**Decomposition of the complex proceeds stepwise in a two-stage decomposition pattern in the temperature ranges 50-210 and 300-420°C. The mass loss associated with the first step involves the loss of one water molecule (calc. 6.1 %, found 5.5 %). The second step mass loss of 67 % is a major step with an activation energy of 59.9 kJ/mol. The final residue is suggested to be CuO (calc. 27.1 %, found 27.5 %).**

$$[\text{Cu(SOP)}(\text{H}_2\text{O})]^{50-210^\circ\text{C}} \rightarrow [\text{Cu(SOP)}] + \text{H}_2\text{O} \quad (6)$$

**Dichlorobis(N-salicylidene-o-toluidine)cadmium(II), [CdCl_2(SOT)]**

The TG trace of this complex reveals four decomposition steps in the temperature ranges 105-150, 170-245, 250-357 and 520-700°C. The first step displays a 4.0 % mass loss and the kinetic parameters for this step could not be calculated because the temperature range of this step is too narrow for enough data to be obtained. The second step of mass loss 8.5 % is connected with an activation energy of 89.5 kJ/mol. It is to be noted that the first, second and third steps correspond to the expulsion of two toluidine radicals (calc. 30.0 %, found 30.8 %) leaving a cadmium complex which seems to be very stable as indicated from the long plateau extending from 374.5°C. The fourth mass loss of 63.0 % is accompanied with an activation energy of 174.5 kJ/mol. The first, second and third steps in the TG curve represent mass losses corresponding to the expulsion of two toluidine radicals leaving the complex radical (II). The latter loses hydrogen to give a cadmium nitrile complex (III). The Toluidine radical may abstract hydrogen to afford toluene or dimerize to give the diphenyl derivative (Scheme A).

**Decomposition products**

Scheme A

**Dichlorobis(N-salicylidene-o-toluidine)mercury(II), [HgCl_2(SOT)]**

The TG profile of this complex exhibits three decomposition steps in the temperature ranges 100-334, 334-415 and 415-520°C. The first step is a major step (89.5 %) and the decomposition at this step is accompanied by an activation energy of 70.3 kJ/mol. The second step is a small step associated with a mass loss amounts to 3.0 %. The TG trace exhibits further a mass loss of 10.5 % corresponding to the third step with an activation energy of 149.5 kJ/mol. The thermal decomposition finishes at 507 °C and the complex decomposes completely.

**Kinetic analyses**

Non-isothermal kinetic analyses for the thermal decomposition of the complexes were carried out with the application of two different procedures, namely the Coats-Redfern and Horowitz-Metzger methods.

The kinetic parameters were evaluated only for clear-cut and non-overlapping stages. Kinetic studies were not attempted for decomposition stages, which occur within a very narrow temperature range resulting in a TG curve too steep for enough data to be collected. Furthermore, some decomposition steps are so small that within the accuracy of the measurements a sufficient number of points from the TG curve could not be obtained to derive meaningful results for kinetic parameters. The kinetic parameters $n$, $E$ and $Z$ were calculated according to the above two methods. Comparison of the two sets of kinetic parameters shows that deviation between them is not so great, considering the approximations involved in the Horowitz-Metzger method.

**Coats-Redfern equation**

$$\ln \left[ \frac{1-(1-\alpha)^{1-n}}{(1-n)T^2} \right] = \frac{M}{T} + B$$

for $n \neq 1 \quad (7)$

$$\ln \left[ \frac{-\ln (1-\alpha)}{T^2} \right] = \frac{M}{T} + B$$

for $n = 1 \quad (8)$

where $\alpha$ is the fraction of material reacted, $n$ the order of the decomposition reaction and $M = E/R$ and $B = Z/R \cdot E$; $E$, $R$, $Z$ and $\phi$ are the activation energy, gas constant, pre-exponential factor and heating rate, respectively.

**Horowitz-Metzger equation**

$$\ln \left[ \frac{1-(1-\alpha)^{1-n}}{1-n} \right] = \ln \left[ \frac{ZRT_e^2}{\phi E} \right] + \frac{E}{RT_e} - \frac{RT_e}{R}$$

for $n \neq 1 \quad (9)$

$$\ln \left[ -\ln (1-\alpha) \right] = \frac{E}{RT_e}$$

for $n = 1 \quad (10)$

where $\phi = T - T_e$, $T_e$ is the temperature at the DTG peak.

The correlation coefficient $r$ is computed using the least squares method for equations (7), (8), (9) and (10). Linear curves were drawn for different values of $n$ ranging from 0 to 2. The value of $n$, which gave the best fit, was chosen as the order parameter for the decomposition stage of interest. The kinetic parameters were calculated from the data of the left hand side of equations (7) and (8) against $1/T$ and against $\theta$ for equations (9) and (10).

The fractionary values of the decomposition reaction order $n$ between 0.5 and unity show a mixed regime of control (kinetic and diffusion) for a reaction with the purely kinetic regime corresponding to $n = 1$.

In order to account for the mechanisms involved in the dehydration of the compounds in this investigation, the following arguments should be taken first into consideration:

- **Dehydration of a hydrate compound includes more than one type of a chemical change upon heating:**
  a) release of coordinated water can be regarded as a thermal decomposition of the complex compound, b) dehydration accompanied by structural reorganization of a lattice destabilized by the removal of constituent hydrogen bonded water, c) hydroxyl groups that combine to give oxide and water.

- **Discussion of dehydration frequently portrays the chemical changes as occurring within an active advancing interface and many investigations of dehydration** lead to the recognition of the importance of the reaction interface in this respect. This is the zone, in the immediate vicinity of the contact between reactant and product phases, within which chemical changes occur preferentially.

- **The constituent water in hydrates is usually regarded as participating in extensive structural hydrogen bonding and some or all may also be coordinated.** These local linkages must be important in characterizing the first step towards $\text{H}_2\text{O}$ release.

For the two cobalt complexes $[\text{Co(SAA)}(\text{H}_2\text{O})]_2\text{H}_2\text{O}$ and $[\text{Co(SOP)}(\text{H}_2\text{O})]_2\text{H}_2\text{O}$, the DTG peaks corresponding to the release of water of crystallization are broad or unsymmetrical, indicating that the water molecules are not isoenergetically bound and that they are involved in a structural hydrogen bonding. Furthermore, we assume that dehydration in the compounds under investigation leads to chemical changes in the complexes and occurs within an active reaction interface. The values of activation energies corresponding to the dehydrations in the two cobalt complexes (the first decomposition stage for each compound) are somewhat larger than the range reported for water of crystallization, emphasizing involvement of water in hydrogen bonding.

From the above data it is to be concluded that the thermal decomposition of the complexes generally involves overlapping steps which together with the great diversity of possible intermediate products preclude exhaustive...
interpretations. In most cases attempts to correlate the results with the theoretical mass losses were not successful, except for the anhydrous complexes and the final solid products.

It was noted that the thermal behaviour of Co(II), Ni(II), Cu(II), Cd(II) and Hg(II) complexes of the same ligand depends on the nature of the metal ion and it resembles the thermal decomposition reaction:

$$A_{n0} + B_{n0} + C_{n0}$$

which can be studied kinetically.

It is of interest to note that the TG-DTG curves of [CdCl₂(SOT)] permit verification of the formation of a nitrile intermediate (and probably also for the other complexes under study) during thermal decomposition. Formation of such nitrile intermediates was also reported for the thermal decomposition of some aromatic Schiff bases.

The two complexes of Cd(II) and Hg(II) exhibit striking similar thermal decomposition patterns, indicating their isostructural relationships. Both degradations occur in three well defined steps.

It is notable that the kinetic parameters based on the Horowitz-Metzger equation are higher than that based on the Coats-Redfern equation. This is due to the inherent error involved in the approximation method employed in the derivation of the Horowitz-Metzger equation.

### Table 1. Thermal decomposition data of the complexes.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Step</th>
<th>TG/DTG</th>
<th>Mass Loss (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>T&lt;sub&gt;j&lt;/sub&gt;°C</td>
<td>T&lt;sub&gt;max&lt;/sub&gt;°C</td>
</tr>
<tr>
<td>[Co(SAA)(H₂O)].2H₂O</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>69</td>
<td>99</td>
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<tr>
<td></td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>125</td>
<td>169-200</td>
</tr>
<tr>
<td></td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>340</td>
<td>415</td>
</tr>
<tr>
<td>[Ni(SAA)(H₂O)]</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>65</td>
<td>91</td>
</tr>
<tr>
<td></td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>160</td>
<td>220</td>
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<td></td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>290</td>
<td>321</td>
</tr>
<tr>
<td></td>
<td>4&lt;sup&gt;th&lt;/sup&gt;</td>
<td>403</td>
<td>450</td>
</tr>
<tr>
<td>[Cu(SOP)(H₂O)].2H₂O</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>50</td>
<td>84</td>
</tr>
<tr>
<td></td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>215</td>
<td>280-325</td>
</tr>
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<td></td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>375</td>
<td>400</td>
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<tr>
<td>[Ni(SOP)(H₂O)]</td>
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<td>98</td>
<td>190</td>
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<td>205</td>
<td>279</td>
</tr>
<tr>
<td></td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>355</td>
<td>400</td>
</tr>
<tr>
<td>[Cu(SOP)(H₂O)]</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>50</td>
<td>110</td>
</tr>
<tr>
<td></td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>300</td>
<td>300</td>
</tr>
<tr>
<td>[CdCl₂(SOT)]</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>105</td>
<td>125</td>
</tr>
<tr>
<td></td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>170</td>
<td>205</td>
</tr>
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<td></td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>250</td>
<td>300</td>
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<td></td>
<td>4&lt;sup&gt;th&lt;/sup&gt;</td>
<td>520</td>
<td>600</td>
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<tr>
<td>[HgCl₂(SOT)]</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>100</td>
<td>226</td>
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<tr>
<td></td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>334</td>
<td>367</td>
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<tr>
<td></td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>415</td>
<td>477</td>
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</tbody>
</table>

### Table 2. Kinetic parameters of the thermal decomposition of the complexes

<table>
<thead>
<tr>
<th>Compound</th>
<th>Step</th>
<th>Coats-Redfern</th>
<th>Horowitz-Metzger</th>
</tr>
</thead>
<tbody>
<tr>
<td>[Co(SAA)(H₂O)].2H₂O</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>n = 0.993, r = 45.3, E = 1.3x10&lt;sup&gt;11&lt;/sup&gt;</td>
<td>n = 0.993, E = 56.3</td>
</tr>
<tr>
<td>[Ni(SAA)(H₂O)].H₂O</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>n = 0.993, r = 62.1, E = 7.9x10&lt;sup&gt;10&lt;/sup&gt;</td>
<td>n = 0.997, E = 47.2</td>
</tr>
<tr>
<td>[Cu(SAA)(H₂O)]</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>n = 0.995, r = 58.6, E = 5.6x10&lt;sup&gt;8&lt;/sup&gt;</td>
<td>n = 0.994, E = 67.6</td>
</tr>
<tr>
<td>[Co(SOP)(H₂O)].2H₂O</td>
<td>1&lt;sup&gt;st&lt;/sup&gt;</td>
<td>n = 0.993, r = 57.3, E = 1.3x10&lt;sup&gt;10&lt;/sup&gt;</td>
<td>n = 0.991, E = 56.3</td>
</tr>
<tr>
<td>[Ni(SOP)(H₂O)]</td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>n = 0.997, r = 62.2, E = 8.9x10&lt;sup&gt;9&lt;/sup&gt;</td>
<td>n = 0.998, E = 73.8</td>
</tr>
<tr>
<td>[Cu(SOP)(H₂O)]</td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>n = 0.995, r = 105.6, E = 7.1x10&lt;sup&gt;8&lt;/sup&gt;</td>
<td>n = 0.996, E = 117.3</td>
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<tr>
<td>[CdCl₂(SOT)]</td>
<td>2&lt;sup&gt;nd&lt;/sup&gt;</td>
<td>n = 0.998, r = 45.6, E = 6.3x10&lt;sup&gt;9&lt;/sup&gt;</td>
<td>n = 0.997, E = 50.3</td>
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<tr>
<td>[HgCl₂(SOT)]</td>
<td>3&lt;sup&gt;rd&lt;/sup&gt;</td>
<td>n = 0.996, r = 89.3, E = 4.1x10&lt;sup&gt;9&lt;/sup&gt;</td>
<td>n = 0.999, E = 89.9</td>
</tr>
</tbody>
</table>

E in kJ/mol and Z in s<sup>-1</sup>
Kinetic compensation effect

The effect of sample weights on the TG curves of \([\text{Co(SAA)}(\text{H}_2\text{O})]_2\text{H}_2\text{O},\) \([\text{Ni(SAA)}(\text{H}_2\text{O})]\) and \([\text{Cu(SAA)}(\text{H}_2\text{O})]\) (Fig. 2) was studied. The TG curves exhibit a shift towards higher temperatures by increasing sample weights. The concept of the so-called kinetic compensation effect, which was observed in many non-isothermal processes\(^{17,18}\) is seen to be valid here.

It was reported that for a particular process, the value of \(E\) bears a definite linear relationship to \(\ln Z\), viz.

\[
\ln Z = a E + b
\]  

(11)

Where \(a\) and \(b\) are constants.

The equations to this straight line, as obtained by linear squares method for the above three complexes (Fig. 3) are written as follows:

For \([\text{Co(SAA)}(\text{H}_2\text{O})]_2\text{H}_2\text{O}\)
\[
\ln Z = 0.237 E - 3.831
\]  

(12)

For \([\text{Ni(SAA)}(\text{H}_2\text{O})]\)
\[
\ln Z = 0.270 E - 9.909
\]  

(13)

For \([\text{Cu(SAA)}(\text{H}_2\text{O})]\)
\[
\ln Z = 0.260 E + 1.379
\]  

(14)

Garn\(^{19,20}\) defined a characteristic temperature \(T_c\) which is related to the slope \(a\) of the straight line plot according to

\[
T_c = \frac{1}{Ra}
\]  

(15)

The physical significance of \(T_c\) is, however, not very clear. For each of the above three complexes, the value of \(T_c\) was calculated and it was seen that the value of \(T_c\) falls within the experimental temperature ranges 507-773 K for \([\text{Co(SAA)}(\text{H}_2\text{O})]_2\text{H}_2\text{O}\), 445-773 K for \([\text{Ni(SAA)}(\text{H}_2\text{O})]\), and 462-773 K for \([\text{Cu(SAA)}(\text{H}_2\text{O})]\) (Table 3). So the kinetic compensation effect holds good for the non-isothermal decomposition of the three complexes. Table 3 reveals that there is a direct correlation between activation energy and the value of \(a\). According to Zsako et al.\(^{21}\) who assumed that the compensation parameter \(a\) can also be correlated with the strength of the bond to be broken, we can connect the value of this parameter with the strength of water bond to the metal ion.

<table>
<thead>
<tr>
<th>Compound</th>
<th>Weight (mg)</th>
<th>(E)</th>
<th>(\ln Z)</th>
<th>Slope (a)</th>
<th>Intercept (b)</th>
<th>Correlation Coefficient (r)</th>
<th>Mean Temperature (T_c)(K)</th>
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<tr>
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<td>31.8</td>
<td>7.8</td>
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<td>-3.831</td>
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<td>507</td>
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<td>4.3</td>
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<td>2.7</td>
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<td>1.8</td>
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<td>13.1</td>
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<td>11.5</td>
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<td>3.1</td>
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<td>8.2</td>
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<td>0.986</td>
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Table 3.- Kinetic compensation effect for the dehydration of \([\text{Co(SAA)}(\text{H}_2\text{O})]_2\text{H}_2\text{O}, [\text{Ni(SAA)}(\text{H}_2\text{O})]\) and \([\text{Cu(SAA)}(\text{H}_2\text{O})]\).
Fig. 3. - Kinetic compensation effect of [Co(SAA)(H$_2$O)$_2$].2H$_2$O

REFERENCES