

Metal Complexes as Ligands: Binuclear Alkali Metal and Trinuclear Alkaline Earth Metal Complexes with Nickel (II) and Copper (II) Metal Complexes of isonitrosoacetylacetone

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Abstract: *In the present study we have synthesized new binuclear complexes of alkali metal salts and trinuclear complexes of alkaline earth metal salts with interaction of stable metal chelates of isonitrosoacetylacetone of Ni(II) & Cu(II) having general formula $[Ma(INAA)_2.M_bL]$ and $[Ma(INAA)_2.M_bX]$ for alkali metal adducts $[Ma(INAA)_2.M_bL_2]$ and $[Ma(INAA)_2.M_bX_2]$ for alkaline earth metal adducts, where $M_a=Ni(II)$ or $Cu(II)$ and $M_b=Li^+, Na^+, K^+$ or Mg^{++} , $INAA=$ deprotonated isonitrosoacetylacetone, $L=$ deprotonated 1-nitroso-2-naphthol or 8-hydroxyquinoline and, $X= ClO_4$ or SCN^- . The IR spectral studies suggests that the Ni(II) & Cu(II) metal chelates act as ligand and coordination towards alkali metal salts and alkaline earth metal salts takes place through oxygen atom of bonded ketonic oxygen. The diffuse reflectance electronic spectra and magnetic moment values of the metal chelates and binuclear & trinuclear complexes with alkali metal and alkaline earth metal salts indicates the change in geometry during oxygen bridged complex formation.*

Keywords: isonitrosoacetylacetone, 1-nitroso-2-naphthol, 8-hydroxyquinoline, Metal complexes as ligands

1. Introduction

The isonitrosoacetylacetone has found to be good extractant and spectrophotometric reagent.¹ It has also been used as ambidentate ligand in complexes of Fe(II) and Fe(III) by Robar et al.² Co(III) and Pd(II) complexes of isonitrosoacetylacetone have been reported by Haldar et al.^{3,4} Octahedral and square planar structure for Co(III) and Pd(II) complexes respectively have been proposed on the basis of magnetic and spectral measurements. Taylor and Ewbank⁵ have reported that the reaction between alcoholic isonitrosoacetylacetone (HINAA) and aqueous ammoniacal nickel acetate produced a green compound which changed rapidly into a red-brown compound. Djordjevic et al.⁶ have shown that the action of nitrite ions on bis(acetylacetonato) nickel(II) in the presence of ammonium acetate results in the formation of a bright red complex $Ni(C_5H_7N_2O_2)_2$, which had the same composition as that of the red-brown nickel complex cited above. A systematic study of the reaction of nickel salts with HINAA yields six compounds. $Ni(C_5H_6NO_3)_2$ (olive green), $Ni(C_5H_6NO_3)_2$ (Violet), $Ni(C_5H_6NO_3)_2 \cdot 2Py$ (Violet), $Ni(C_5H_{10}N_2O_4)$ (Pale Green), $Ni(C_5H_7N_2O_2)_2$ (Orange Red) and $Ni(C_5H_7N_2O_2)_2$ (Red), The first three have been prepared by Haldar et al.⁷ Complexes of Cu(II) with isonitrosoacetylacetone have also been reported⁸ and their structures have been investigated by magnetic and spectral measurements. It has been observed that olive green coloured Cu(II) metal chelates has 29 % monomeric and 71 % dimeric form. Banerjee et al.⁹ have synthesized and reported number of stable hetero-binuclear alkali metal complexes with magnesium(II) and Zinc(II) acetylacetonates. Later on they also reported¹⁰ hetero-trinuclear alkali metal complexes with Cu(II) and Ni(II) acetylacetonates. They concluded that the

oxygen atoms of metal chelates provide coordination sites for attachment of the alkali metals in the formation of hetero poly-nuclear alkali metal complexes. Recently Gupta et al.¹¹⁻¹³ have reported the hetero binuclear and trinuclear complexes of alkali & alkaline earth metal salts with stable Ni(II) & Cu(II) metal chelates of para-methyl isonitrosoacetophenone, isonitrosopropiophenone and isonitrosobarbituric acid. In view of the above information, we are reporting here synthesis of a number of stable bi- and tri-nuclear complexes of alkali/alkaline earth metal salts with "complex ligand" i.e., Ni(II) and Cu(II) metal chelates of isonitrosoacetylacetone denoted by $Ni(INAA)_2$ and $Cu(INAA)_2$ respectively.

2. Experimental

Material

Ni (II) & Cu(II) acetate were used of E. Merck quality. The organic acid were used; 8-hydroxyquinoline and 1-nitroso-2-naphthol of BDH A.R. Quality, isonitrosoacetylacetone was prepared by the published method.¹⁴

Preparation of Ni (II) & Cu (II) metal chelates of isonitrosoacetylacetone

Bis (isonitrosoacetylacetonate) Ni(II) and Cu(II) chelates were prepared according to reported method.^{7,8}

Preparation of alkali metal salts of organic acids.

95 % ethanolic solution of 0.01 mole of organic acid and 0.01 mole of alkali metal hydroxide were mixed. The mixture was refluxed on magnetic hot plate for 1 hours with continuous stirring at 80⁰ C & on cooling the resulting solution, a characteristic color precipitate was obtained. It was filtered

washed with pure solvent and dried in an electric oven at 100° C.

filtered, washed several times with absolute ethanol and dried in oven at 80° C.

Preparation of Binuclear and trinuclear complexes of Ni(II) and Cu(II) metal chelates with alkali & alkaline earth metal salts

Our usual method of synthesis was to take metal chelates Ni(INAA)₂ or Cu(INAA)₂ in an absolute ethanol and to add alkali metal salts to it in 1:1 mole ratio, usually slight excess of alkali metal salts were taken. In the case of alkaline earth metal salts 2:1 mole ratio were taken. The reaction mixture was refluxed with constant stirring in hot magnetic plate for about 3 to 4 hours at 60° C. The whole substance went in to the solution and subsequently the adducts were precipitated in hot condition during the process of refluxing. They were

3. Results and Discussion

The adducts are stable under dry condition, but decompose on exposure to moisture, as such they were kept in a desiccators over anhydrous CaCl₂. Some physical properties and analytical data of the metal chelates and their adducts are listed in Table-1. From the results, It is evident that the adducts have characteristic color and are different from the metal chelates. All the adducts show high melting/decomposition temperatures with few exception showing there greater stability.

Table 1: Color, Decomposition temperature, magnetic moments & Elemental analysis of the complexes

Compound	Color	Transition (t ⁰ C) or decomposition (d ⁰ C) temperature	Magnetic moment values in B.M.	%Analysis found/calc.				
				C	H	N	M _a	M _b
Ni(INAA) ₂	Olive Green	218d	3.09	38.62 (38.13)	4.02 (3.81)	8.79 (8.89)	18.54 (18.65)	
Ni(INAA) ₂ .LiIN2N	Brown	290d	----	47.92 (48.61)	3.89 (3.64)	8.37 (8.50)	11.02 (11.89)	
Ni(INAA) ₂ .NaIN2N	Dark Orange Brown	285d	----	46.34 (47.08)	3.85 (3.53)	7.98 (8.23)	11.13 (11.51)	4.95 (5.51)
Ni(INAA) ₂ .KIN2N	Brown	290d	3.39	44.86 (45.64)	3.69 (3.42)	7.38 (7.98)	10.83 (11.16)	7.21 (7.43)
Ni(INAA) ₂ .Na8HQ	Light Yellow	>320	----	46.81 (47.33)	3.94 (3.73)	8.54 (8.71)	11.75 (12.18)	4.48 (4.77)
Ni(INAA) ₂ .K8HQ	Cream	>320	3.42	44.92 (45.80)	3.82 (3.61)	8.16 (8.43)	11.38 (11.79)	7.39 (7.85)
Ni(INAA) ₂ .NaClO ₄	Bottle Green	225d	3.41	26.74 (27.44)	2.95 (2.74)	5.93 (6.40)	13.26 (13.42)	4.98 (5.26)
Ni(INAA) ₂ .KSCN	Yellowish green	158d	3.38	31.26 (32.06)	3.13 (2.91)	9.79 (10.20)	13.93 (14.26)	9.21 (9.47)
Ni(INAA) ₂ .Mg(8HQ) ₂	Greenish yellow	310d	3.37	47.53 (48.43)	4.11 (3.82)	8.63 (8.92)	11.97 (12.47)	2.38 (2.64)
Cu (INAA) ₂	Olive Green	135d	1.22	37.32 (37.56)	3.8 (3.7)	8.48 (8.70)	19.69 (19.84)	
Cu (INAA) ₂ .NaIN2N	Greenish Black	>320	1.88	45.79 (46.64)	3.68 (3.49)	7.84 (8.16)	11.94 (12.34)	4.28 (4.47)
Cu (INAA) ₂ .KIN2N	Greenish Black	>320	44.65 (45.22)	3.64 (3.39)	7.56 (7.91)	11.49 (11.97)	7.16 (7.35)
Cu (INAA) ₂ .Na8HQ	Greenish Brown	>320	-----	45.98 (46.86)	3.86 (3.69)	8.30 (8.61)	12.78 (13.05)	4.68 (4.72)
Cu (INAA) ₂ .K8HQ	Brown	>320	1.92	44.49 (45.36)	3.76 (3.58)	7.96 (8.35)	12.13 (12.64)	7.48 (7.76)
Cu (INAA) ₂ .NaClO ₄	Green	230d	1.83	26.81 (27.14)	2.92 (2.71)	5.69 (6.33)	14.06 (14.37)	4.96 (5.20)
Cu (INAA) ₂ .KSCN	Greenish Brown	210d	1.73	30.86 (31.68)	3.02 (2.88)	9.73 (10.08)	14.89 (15.25)	9.14 (9.36)
Cu (INAA) ₂ .Mg(8HQ) ₂	Brownish Green	235d	-----	46.79 (47.94)	3.86 (3.78)	8.68 (8.83)	13.07 (13.36)	2.38 (2.52)
Cu (INAA) ₂ .Mg(ClO ₄) ₂	Greenish Brown	>320	1.76	27.32 (27.83)	3.02 (2.78)	6.21 (6.49)	14.27 (14.74)	2.46 (2.78)

Infrared spectra

Infrared measurements of all the complexes were recorded in the region between 4000-200cm⁻¹ and 4000-650cm⁻¹. In KBr

disc with the help of spectrophotometer. Pertinent IR data for these compounds were recorded in Table-2. The IR spectra of the olive green complex of Ni(INAA)₂⁷ shows absorption band at 3500 cm⁻¹. Which has been assigned to hydrogen

bonded OH stretching vibration. The strong bond at 1660 cm^{-1} in the $\text{Ni}(\text{INAA})_2$

Table 2: Pertinent IR data (cm^{-1}) for metal chelates as well as their adducts with alkali & alkaline earth metal salts

Adducts	$\nu\text{C}=\text{O}/\text{SCN}^-$	Non-Bonded $\nu\text{C}=\text{O}$	Bonded $\text{C}=\text{O}$	$\nu\text{C}=\text{N}$	$\nu\text{M}-\text{N}$	$\nu\text{M}-\text{O}$
$\text{Ni}(\text{INAA})_2$	---	1660s	1600br	1582sh	572s	470br,440br,380br,350br
$\text{Ni}(\text{INAA})_2.\text{NaIN}_2\text{N}$	---	1660sh	1575m	1590m		--
$\text{Ni}(\text{INAA})_2.\text{Na}_8\text{HQ}$	---	1660s	1575br	1590m		--
$(\text{Ni}(\text{INAA})_2).\text{Mg}(\text{8HQ})_2$	---	1660s	1580s	1590m		--
$\text{Ni}(\text{INAA})_2.\text{NaClO}_4$	1100m, 1110m, 932s	1660s	1575m	1592sh	570vs	470m,440br,380br,260br,330br
$\text{Ni}(\text{INAA})_2.\text{KSCN}$	2040vs	1665vs	1575s	1585sh	570s	460m,445s,380m,365br
$\text{Cu}(\text{INAA})_2$	---	1680-1650br	1610w	1600w	598s	495w,450m
$\text{Cu}(\text{INAA})_2.\text{Na}_8\text{HQ}$	---	1680sh	1590sh	1585s		
$\text{Cu}(\text{INAA})_2.\text{K}_8\text{HQ}$	---	1680sh	1600m	1585s		
$\text{Cu}(\text{INAA})_2.\text{KIN}_2\text{N}$	---	1685w	1590m	1580sh		
$\text{Cu}(\text{INAA})_2.\text{NaClO}_4$	1136s, 1105s, 1076s, 930m	1680sh	1600br	1595sh	615s	470br,450br
$\text{Cu}(\text{INAA})_2.\text{KSCN}$	2070vs	1680sh	1590br	1595br	603s	470br,450br,370br
$[\text{Cu}(\text{INAA})_2]_2.\text{Mg}(\text{8HQ})_2$		1675m	1600m	1580s		
$[\text{Cu}(\text{INAA})_2]_2.\text{Mg}(\text{ClO}_4)_2$	1080s, 1120br, 1110w, 935s	1680sh	1600s	1595sh	595m	518s,485s,455m,420vs,375m,330vs

S=strong, m=medium, w=weak, sh=shoulder and br=broad

complexes may be attributed to the free $\text{C}=\text{O}$ stretching frequency which is reported to occur at 1684 cm^{-1} in $\text{Co}(\text{INAA})_2^3$ and at 1640 cm^{-1} in $\text{Pd}(\text{INAA})_2^4$. The peaks at 1605 cm^{-1} and 1582 cm^{-1} in the olive green compound may arise from hydrogen bonded $\text{C}=\text{O}$, $\text{C}=\text{O}$ bonded to metal and/or perturbed $\text{C}=\text{N}$ stretching modes. The broadness of these bands is possibly due to the overlapping of the $\text{C}=\text{N}$ frequency. The complex does not show any band in the range $1300-1200\text{ cm}^{-1}$, since the N-oxide stretching frequency usually appears in this region⁴. It is reasonable to conclude that the olive-green compound does not contain an N-oxide linkage.⁷ In the lower region, band at 572 cm^{-1} might be attributed to a Ni-N vibration^{3,4}. The appearance of more than one band in the region at $470,440,380$ and 350 cm^{-1} clearly indicates the cis-structure of the metal chelates of $\text{Ni}(\text{INAA})_2^{15-17}$. In the alkali/alkaline earth metal adducts of $\text{Ni}(\text{INAA})_2$ the appreciable shift of bonded $\nu\text{C}=\text{O}$ is assigned in between $1575-1580\text{ cm}^{-1}$. Slight change in $\nu\text{C}=\text{N}$ stretching modes are also observed in adducts. It is also evident that in lower region M-O bands appear with slight shift. So it is quite apparent that bonded ketonic oxygen atoms are now no longer remained in that form but they are most likely, attached to the alkali/alkaline earth metal forming hetero bi-or trinuclear complexes. Banerjee et al.⁹ while synthesizing the $\text{Mg}(\text{acac})_2$ with alkali metal salt of organic acids isolated 1:1 binuclear complexes of alkali metal organic acid salts and made same observations.

Similarly the infrared spectra of $\text{Cu}(\text{INAA})_2^8$ exhibits a broad maximum at 3400 cm^{-1} which, from its position and shape, has

been assigned to the hydrogen bonded OH. It also reveals a broad band at $1680-1650\text{ cm}^{-1}$, which may include stretching frequency of free $\text{C}=\text{O}$ and stretching mode of $\text{C}=\text{C}$. The perturbed $\text{C}=\text{O}$ and/or perturbed $\text{C}=\text{N}$ stretching vibrations appear as a broad band near $1600-1610\text{ cm}^{-1}$. The peak near 598 cm^{-1} may be assigned to the Cu-N vibration, where as the peak near 495 and 450 cm^{-1} may be assigned due to Cu-O vibration and more than one band in this region indicate the cis-configuration of the metal chelates.¹⁵⁻¹⁷ In alkali/alkaline earth metal adducts of $\text{Cu}(\text{INAA})_2$ the bonded $\text{C}=\text{O}$ frequency appear in the region $1600-1590\text{ cm}^{-1}$. The shift of bonded $\text{C}=\text{O}$ frequency in higher region indicate the coordination of alkali/alkaline earth metal through bonded ketonic oxygen atoms. In the adducts of alkali metal thiocyanate the thiocyanate peak appears at $2040-2070\text{ cm}^{-1}$. The shifting of the $\nu\text{C}=\text{N}$ to higher frequency (for KSCN it is 2020 cm^{-1})¹⁸. Suggest coordination of alkali metal through ketonic oxygen atoms. The medium intensity peaks in the region at $1080-1136\text{ cm}^{-1}$ and extra peak at $930-935\text{ cm}^{-1}$ suggest the coordinated perchlorate¹⁹ in the adducts of alkali/alkaline earth metal perchlorate.

Magnetic Measurement

Magnetic susceptibility measurements were made using the faraday technique at 34°C . The results are recorded in Table-01.

Halder et al.⁷ reported the magnetic moment of $\text{Ni}(\text{INAA})_2$, 3.11 B.M. at 296.5°K . He concluded that $\text{Ni}(\text{INAA})_2$ complex had most probably octahedral or pseudo-octahedral structure.

We observed magnetic moment value of Ni(INAA)₂ at R.T., almost equal to the magnetic value observed by Halder et al.⁷. The magnetic moment values of isolated alkali/alkaline earth metal adducts are found in between 3.38 to 3.80 B.M., which is higher than the magnetic moment value of Ni(INAA)₂. The higher magnetic moment value of alkali/alkaline earth metal adducts of Ni(INAA)₂ may be due to change in the geometry Ni(II) complexes of isonitrosoacetylacetone in the adducts from octahedral structure to tetrahedral structure.

Magnetic susceptibility of Cu(INAA)₂ complex was also studied by Halder et al.⁸. He observed that magnetic moment of Cu(INAA)₂ complex was substantially below the spin only value for one unpaired electron. The magnetic moment of complex changed from 1.08 B.M to 1.26 B.M. over the temperature range 87^o-295^o K, such sub-normal values of magnetic moments have been reported for many copper(II) complexes.²⁰⁻²¹ The anomalous magnetic behavior of Cu(INAA)₂ complex could be explained on the basis of the antiferromagnetic interaction. The least fit for Cu(INAA)₂ was observed.⁸ by assuming it to be a mixture of 29% of the monomeric form obeying the Curie-Weiss law with a magnetic moment of 1.9 B.M., Corresponding to one unpaired electron, 71% of the binuclear variety showing antiferromagnetic exchange interaction with exchange coupling constant, $J=300\text{ cm}^{-1}$ and $g=2$. The χ_m values for the dimeric form at various temperature were calculated by using the equation derived by Bleamy and Bowers.²² We observed magnetic moment values 1.22 B.M for Cu(INAA)₂ complex which is almost equal to the reported value. The magnetic moment values of isolated alkali/alkaline earth metal adducts are in between 1.85 to 1.95 B.M (Table-1)

Therefore, the stereochemistry of Cu(INAA)₂ unit in the alkali/alkaline earth metal complexes may be due to change in geometry from dimeric form to monomeric form.

4. Electronic Spectra

Diffuse reflectance electronic spectra were recorded on SHIMADZU UV-VIS-160A spectrophotometer in Nujol Mull/Paraffin liquid. The bands observed in diffuse reflectance spectra of Ni(INAA)₂ and Cu(INAA)₂ and of their some adducts are given in Table 3.

Table 3: Major diffuse reflectance bands (nm) of metal chelates and their adducts

Compound	Diffuse reflectance (nm)
Ni(INAA) ₂	980w,622h,921br,246s,
Ni(INAA) ₂ .KIN2N	665w,320br,301s,
Ni(INAA) ₂ .NaClO ₄	1052h,660w,392br,305s,237br
Ni(INAA) ₂ .KSCN ₂	1061h,690sh,463s,238br

Cu(INAA) ₂	705br,400br,325s,300s,215s
Cu(INAA) ₂ .KIN2N	1100w,975w,655br,366s,300s,250br
Cu(INAA) ₂ .NaClO ₄	720sh,366br,350s,300s,234br
Cu(INAA) ₂ .Mg(ClO ₄) ₂	724sh,390br,348s,280s,230br

Br=broad, s=strong,m=Medium,w=weak, sh=shoulder and h=hump

The diffuse electronic spectra of Ni(INAA)₂⁷ complex in paraffin liquid display bands in region 246-321nm due to charge transfer, The weak hump at 622nm is appeared possibly due to d-d transition. The absorption band of alkali metal adducts derived from Ni(INAA)₂ is found in the range of 660-690nm and a band of medium intensity observed at 1052-1061nm. Had it been six coordinated complex of Ni(II), the absorption band must have been obtained like Ni(INAA)₂ i.e. the "complex ligand" But the nature of absorption band of alkali metal adducts of Ni(INAA)₂ is suggesting tetrahedral geometry of the Ni(II) and the magnetic moment values observed higher than 3.38B.M., further confirm the tetrahedral structure of Ni(II) in the alkali metal adducts. The diffuse reflectance spectrum of Cu(INAA)₂²⁰ reveals a broad maximum at 705nm, which is attributed to d-d transition. Peaks at 215, 300,325nm are likely to be due to $\pi \rightarrow \pi$ transitions in the ligand.

The shifts in the charge transfer region as well as d-d transition region have been observed in the alkali/alkaline earth metal adducts of Cu (INAA)₂. These shifts have been interpreted in terms of slight distortion from planer or tetragonal towards tetrahedral configuration²³⁻²⁴, as well as adducts formation of Cu(INAA)₂ with alkali/alkaline earth metal salts.

Structure & Bonding

On the basis of elemental analysis, the molecular formula of binuclear or trinuclear alkali/alkaline earth metal adducts has been suggested as [M_a (INAA)₂.M_bL] or [M_a (INAA)₂.M_bX] for alkali metal adducts and [M_a (INAA)₂.M_bL₂] or [M_a (INAA)₂.M_bX₂] for alkaline earth metal adducts, where M_a=Ni (II) or Cu (II) and M_b=Li⁺, Na⁺, K⁺ or Mg⁺⁺ L=deprotonated 1-nitroso-2-naphthol and 8-hydroxyquinoline, X= ClO₄⁻, SCN⁻ or Cl⁻.

The infrared spectral studies of these adducts, suggest that complex ligand, i.e. Ni(II) and Cu(II) chelates of isonitrosoacetylacetone acts as a Lewis base to the Lewis acid (alkali/alkaline earth metal salts); Coordination is taking through the oxygen atoms of bonded ketonic oxygen. The probable structure of the complex compound is shown in Figure -01 & 02 may be produced schematically as such.

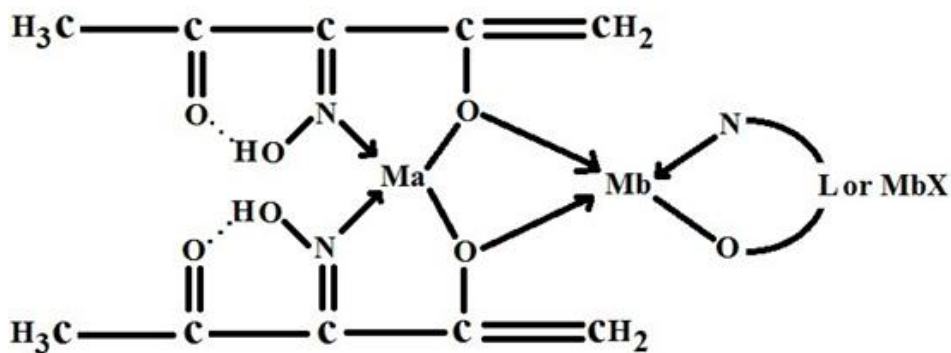


Figure 1

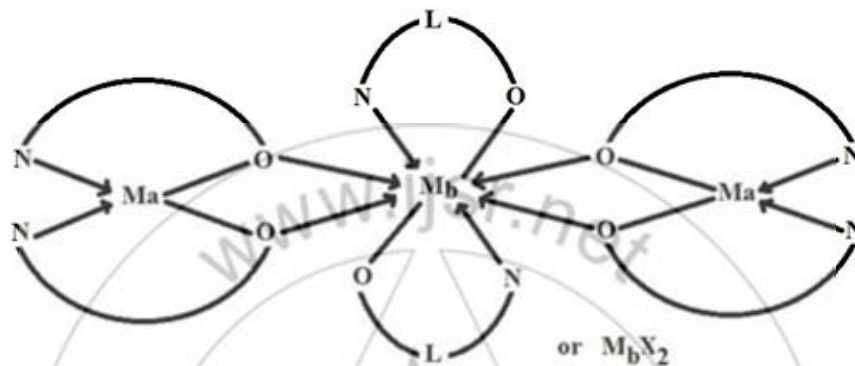


Figure 2

5. Acknowledgement

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