

PREPARATION AND CHARACTERIZATION OF N-SUBSTITUTED OCTANOHYDROXAMIC ACIDS METAL COMPLEXES

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ABSTRACT

Organic ligands of octano, N-methyl, N-phenyl, N-(p-chloro phenyl) octano-hydroxamic acids were prepared. The interaction of such ligands with La(III), Co(IV), Th(IV), ZrO(II) and UO₂(II) ions under pH control in (1:1), (1:2) and (1:3) Molar ratios (M:L) led to the formation of solid complexes which gave analytical data suggested the (1:1), (1:2) and (1:3) based on elemental analysis, IR and ¹HNMR techniques.

INTRODUCTION

Hydroxamic acids are weak organic acids and have a wide range variety of applications. They are used as inhibitors for copper corrosion, antifungal agents, food additives and in nuclear fuel processing. Hydroxamic acids are also important as gravimetric colourimetric reagents and strong chelating (Smith, 1960; Jenks, 1958; Fishchbein, 1967; Benhard *et al.* 1964; Shome, 1950; Galiardi *et al.* 1962; Brandt, 1960; Dutta, 1960; Cassidy and Ryan, 1968; Coutts, 1967; Feigi, 1975; Munson and Kehl, 1982 and Chang *et al.* 1987).

In monohydroxamic acid metal complexes, the metal atom is coordinated through the oxygen atoms (Brown *et al.*, 1979). Many different types of hydroxamic acids have been synthesized by adopting Blatt's method (Blatt, 1963). Anindya *et al.*, 2002 reported that, in their reaction with transition metal ions, the N-phenylbenzohydroxamic acids may play two different roles depending on the oxidation state of the metal ion. This is well manifested in their reaction with rhodium(I) and rhodium(III). For rhodium(I) the N-phenylbenzohydroxamic acids serve as oxidizing agents, they oxidize rhodium(I) to rhodium(III), and at the same time themselves are reduced to the corresponding amides. With rhodium(III), however, the same hydroxamic acids only undergo complexation in the usual fashion.

MATERIALS AND METHODS

All materials used for synthesis of N-substituted hydroxamic acids and complexes are of analytical reagent. Adigital Philips PW9404 pH-meter was calibrated by means of standard buffer solutions (BDH). The IR spectra of studied complexes were recorded by Perkin-Elmer 580 spectrophotometer using KBr discs.

Micro analysis of C, H and N were recorded by Perkin-Elmer Model 2400 elemental analyzer. ¹HNMR spectra were run on Jool JNL-GX-90 FTNMR spectrometer using CDCl₃, CD₃OD as solvent and TMS as the reference standard.

Preparation of organic ligands:

Octanohydroxamic acid was prepared by adding octanoylchloride (15.9gm, 0.1mol) with stirring cooled mixture of KOH (11.2gm, 0.2mol) and hydroxylamine hydrochloride dissolved in methanol (250ml), the resulting mixture was stirred for 5hours. The reaction mixture was extracted with ether and the ethereal layer was evaporated under reduced pressure. The product obtained in the form of shiny white crystals, which recrystallized from chloroform, the melting point is (61-62) °C. N-methyl octanohydroxamic acid was prepared by adding (15.9gm, 0.1mol) octanoylchloride to an ice mixture of Na₂CO₃ (21.2gm, 0.2mol) and N-methylhydrochloride (8.35gm, 0.1mol) in methanol (300ml), according to the same procedure described above, the product was in the form of a pale yellow oil. Phenyl hydroxyl amine was prepared by adding (2:1) molar ratio of Zn dust to nitrobenzene according to Brink and Crumbliss method (Brink and Crumbliss, 1982) the product was in the form of yellow needle crystals, (m.p.61-62)°C N-phenyl octanohydroxamic acid was prepared by the same procedure as discussed before, the product was in the form of green oil, (It decompose by long storage). N-(P-chlorophenyl) octanohydroxamic acid was prepared by adding (15.9gm, 0.1mol) octanoylchloride to ice cooled mixture of Na₂CO₃ (21.0gm, 0.2mol) and N-(P-chlorophenyl)-hydroxylamine (14.35gm, 0.1mol) in methanol (250ml). The resulting mixture was stirred for 7hours, filtered, evaporated and dried. The product in the form of crude, (m.p. 156-158)°C

Preparation of metal complexes:

Preparation of Lanthanum (III) complexes:

(a) La(C₈H₁₆NO₂)₃.6H₂O

(3.18gm, 0.02mol) of octanohydroxamic acid in (50ml) ethanol was added with stirring to (4.33gm, 0.01mol) of La(NO₃)₃.6H₂O in (50ml)ethanol, the pH was adjusted between 2.27 to 5.68 using 10% sodium hydroxide solution, white precipitate was allowed to cool then filtered off under nitrogen and dried over calcium chloride.

(b) La [(C₉H₁₈NO₂).2OH].3H₂O and La [(C₁₄H₂₀NO₂).2OH].3H₂O

complexes were prepared as discussed before, the pH had adjusted between 3.71 to 6.67 and .48 to 9.38 respectively.

(c) La [(C₁₄H₁₉NO₂Cl).2OH].6 H₂O

gm, 0.02mol of N-(p-chlorophenyl) octanohydroxamic acid was (5.39) added to (4.3302 gm, 0.01mol) of La (NO₃)₃.6H₂O in (50ml)ethanol, then the mixture was stirred for one hour. The pH had been adjusted between 1.55 to 9.33, a dark yellow precipitate was obtained.

Preparation of Cerium (IV) complex:

Ce[(C₁₄H₁₉NO₂Cl)₂.2OH].8H₂O was prepared by adding (4.042gm, 0.01mol) of Ce(SO₄)₂.4H₂O in (75ml) double distilled water to (5.39gm, 0.02mol) of N-(p-chlorophenyl) octanohydroxamic acid in (75ml) warm ethanol, the pH raised to 2.19, the brown precipitate obtained was allowed to refrigerate overnight.

Preparation of Thorium (IV) complexes:

(a) Th[(C₈H₁₆NO₂)₂.2OH].6H₂O Was prepared by adding (3.18gm, 0.02mol) of octanohydroxamic acid in (50ml) ethanol to (4.33gm, 0.01mol) of

$\text{Th}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$ in (50ml) ethanol, the pH was adjusted between 1.88 to 6.30, a white precipitate was obtained.

- (b) $\text{Th}[(\text{C}_9\text{H}_{18}\text{NO}_2) \cdot 3\text{OH}] \cdot \text{H}_2\text{O}$ Was prepared as discussed before, where the pH had been adjusted between 2.29 to 5.21, a white precipitate was obtained.
- (c) $\text{Th}[(\text{C}_{14}\text{H}_{20}\text{NO}_2) \cdot 3\text{OH}] \cdot 6\text{H}_2\text{O}$ The same procedure has been used, where the pH had been adjusted between 1.07 to 6.36, a white precipitate was obtained.

Preparation of $\text{UO}_2(\text{II})$ complexes:

- (a) $\text{UO}_2[(\text{C}_8\text{H}_{16}\text{NO}_2)_2] \cdot 10\text{H}_2\text{O}$ Was prepared by dissolving of (5.02gm, 0.01mol) Uranyl nitrate in (50ml) ethanol and add to (3.18gm, 0.02mol) of octanohydroxamic acid. This resulted in a color change to red-brown. The mixture was stirred for 30minutes and 10% solution of sodium hydroxide was add drop wise to the mixture until a change of color occurred then the pH raised to 6.55, the solution became cloudy and a change in color from red-brown to orange. The reaction mixture was left to cool over night, the obtained solid product was filtered and dried.
- (b) $\text{UO}_2[(\text{C}_{14}\text{H}_{19}\text{NO}_2\text{Cl})_2] \cdot 6 \text{H}_2\text{O}$ Was prepared by adding (5.02gm, 0.01mol) of Uranyl nitrate to (5.39gm, 0.02mol) of N-(p-chloro phenyl) octanohydroxamic acid, according to the same procedure described above (pH = 8.57).

Preparation of $\text{ZrO}(\text{II})$ complex:

$\text{ZrO}[(\text{C}_8\text{H}_{16}\text{NO}_2)_2] \cdot 6\text{H}_2\text{O}$ Was prepared by adding (3.2235gm, 0.01mol) of Zirconyl-chloride-8-hydrate to (3.18gm, 0.02mol) of octanohydroxamic acid, the mixture was stirred for 30minutes, the pH was raised to 9.45. Awhite precipitation was obtained.

RESULTS AND DISCUSSION

The IR spectra of octanohydroxamic ligand show sharp band in the $1665\text{-}1615 \text{ Cm}^{-1}$ region which attributed to the amide carbonyl frequency, the shift of this band is due to intermolecular hydrogen-bonding on complexation, this band undergoes a shift of about 40-60 wave numbers, which is consistent with chelation by carbonyl oxygen atom. This frequency shift in the carbonyl stretching is due to a reduction of C=O bond order caused by the interaction with metal, abroad band at $3566\text{-}2422 \text{ Cm}^{-1}$ in the spectra of the free ligands was assigned to free (OH)group, which disappears on complexation in all cases. The free hydroxamic acids are bidentate and the bonding occurs through the two oxygen atom (Brown *et al.*, 1979). In the region bellow 950 Cm^{-1} most bands are sensitive to the nature of the metal ion and the substituent.

The IR spectra of octanohydroxamic acid, N-methyl octanohydroxamic acid and p-chloro-N-phenyl octanohydroxamic acid are shown in Table(1). A strong band at $1423\text{-}1467 \text{ Cm}^{-1}$ is assigned to C-N stretching frequency and N-H deformation band (Chamberlain and Gonzlez, 1952). A strong band at $1182\text{-}1116 \text{ Cm}^{-1}$ is assigned to N-O stretching frequency. The O-H bonded appears at 2957 Cm^{-1} while OH free at 3054 Cm^{-1}

¹ (Rundle and Parasol, 1952). The C-H of N-CH₃ stretching appears at 2868-2412 Cm⁻¹ (James, 1980).

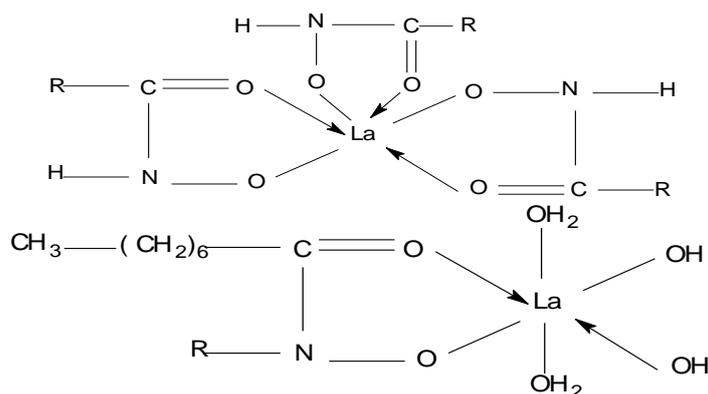
Table (1): The infrared spectra of unsubstituted and N-substitute octano-hydroxamic acids.

Compound	ν C=O	ν C-N	ν (OH) ^b	ν (OH) ^f	ν C-H
	1655	1423	3256	3566	2956
	1615	1432	3054	3372	2912
	1629	1463	3195	3422	2951

Lanthanium (III) form 1:3 and 1:1 complexes with ligand octano, N-methyl, N-phenyl and N-(p-chlorophenyl) octanohydroxamic acids. The IR spectra of Lanthanium complexes are listed in Table (2).

The IR spectra of the amide carbonyl (C=O) shifted in the solid complexes at lower frequency 1601, 1599, 1560 and 1564 Cm⁻¹ in octano, N-methyl, N-phenyl and N-(p-chlorophenyl) octanohydroxamic acids respectively.

The expected structures of complexes are as follows:



Where R=CH₃, C₆H₅ and C₆H₅Cl

Cerium (IV) form complexes of a molar ratio 1:2M:L with P-chloro- N-phenyl octanohydroxamic acid. The IR spectra are listed in Table (2).

The above results give clear sign that the two oxygen atoms of hydroxamic acids are involved in the chelation to metal atom which caused electron withdrawal from the carbonyl group, this increases electron density in the C-N bond resulting in lowering of 65Cm⁻¹ in the carbonyl. The above results give clear sign that the two oxygen atoms of hydroxamic acids frequency compared to the free ligand and an increase by about 15Cm⁻¹ in

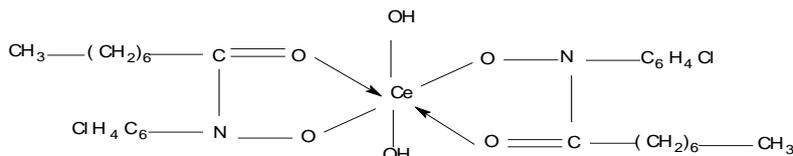
the C-N frequency, the band observed in all complexes in the range 1457 to 1489 Cm^{-1} are attributed to C-N stretching frequency. A broad band which is observed in all complexes in the range 3300-3500 Cm^{-1} is assigned to $\nu(\text{OH})$ of coordinated water molecules. A strong band which is common to all complexes spectra in the range 460-485 Cm^{-1} is assigned to $\nu(\text{La-O})$ vibration (Elmer *et al.*, 1982).

There is band at 428 Cm^{-1} observed at lower frequency which is absent in the free ligand is attributed to the metal oxygen interactions M-O (Baraniak *et al.*, 1976).

Table (2): Infrared spectra of complexes.

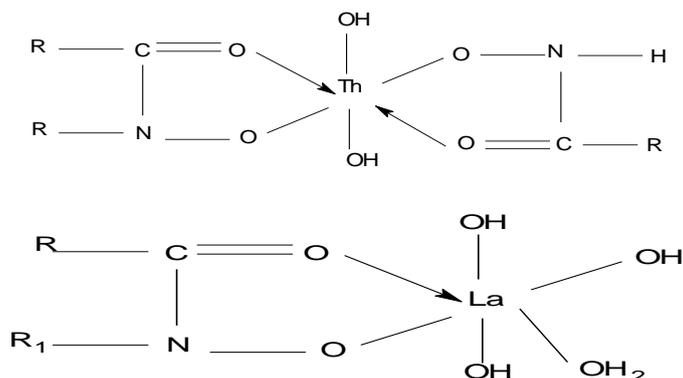
Compound	$\nu \text{ C=O}$	$\nu \text{ C-N}$	$\nu \text{ (M-O)}$	$\nu \text{ (O-H)}$
$\text{La}[\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-NH-O}]_3 \cdot 6\text{H}_2\text{O}$	1601	1465	472	3300
$\text{La}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_2 \cdot 2\text{OH}] \cdot 3\text{H}_2\text{O}$ CH_3	1599	1483	482	3498
$\text{La}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_2 \cdot 2\text{OH}] \cdot 3\text{H}_2\text{O}$ C_6H_5	1560	1457	477	3508
$\text{La}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_2 \cdot 2\text{OH}] \cdot 6\text{H}_2\text{O}$ $\text{C}_6\text{H}_4\text{Cl}$	1564	1489	462	3423
$\text{Ce}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_2 \cdot 2\text{OH}] \cdot 8\text{H}_2\text{O}$ $\text{C}_6\text{H}_4\text{Cl}$	1662	1483	428	3304
$\text{Th}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-NH-O})_2 \cdot 2\text{OH}] \cdot 6\text{H}_2\text{O}$	1599	1483	413	3304
$\text{Th}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_3 \cdot 3\text{OH}] \cdot \text{H}_2\text{O}$ CH_3	1599	1482	414	3422
$\text{Th}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_3 \cdot 3\text{OH}] \cdot 6\text{H}_2\text{O}$ C_6H_5	1600	1464	403	3277
$\text{ZrO}[(\text{CH}_3\text{-(CH}_2\text{)}_6\overset{\text{O}}{\parallel}\text{C-N-O})_2] \cdot 6\text{H}_2\text{O}$ $\text{C}_6\text{H}_4\text{Cl}$	1603	1483	409	3675

The expected structure of the complex is as follow:



The IR spectra of thorium complexes of the general formula MA_2 and MA are listed in Table (2). On comparison with IR spectra of the free ligand, there are clear differences, the main difference is the disappearance of the O-H peak on complex formation. The carbonyl absorption which occurs at 1665cm^{-1} and 1615cm^{-1} in the free ligand is shifted to 1599cm^{-1} and 1600cm^{-1} in the complexes. There is an intense absorption bands at 1483cm^{-1} and 1464cm^{-1} attributed to C-N stretching frequency. Strong bands at 1181cm^{-1} and 1175cm^{-1} is assigned to $\nu(\text{N-O})$ stretching frequency in complexes. Broad bands at 3422cm^{-1} and 3277cm^{-1} respectively, are assigned to $\nu(\text{O-H})$ of coordinated water to metal ion. A strong band at 988cm^{-1} is assigned to bending mode of coordinated OH group to metal ion $\nu(\text{M-OH})$ (Scargill, 1961). A strong band at $(438-403)\text{cm}^{-1}$ is assigned to $\nu(\text{Th-O})$ vibration (Nakomato, 1970). Finally the bands at 2854cm^{-1} and 2956cm^{-1} are assigned to C-H stretching of N- CH_3 .

The expected structures of the complexes are as follows:

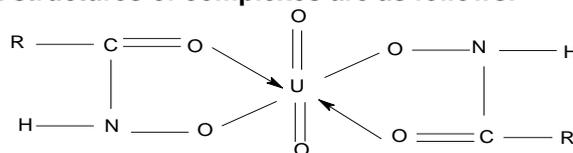


Where $R = \text{Heptyl}$ and $R_1 = \text{CH}_3$ or C_6H_5

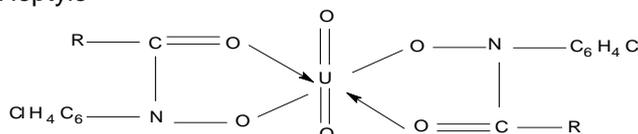
On comparing the IR spectra of the complexes $\text{UO}_2[\text{C}_8\text{H}_{16}\text{NO}_2]_2 \cdot 10\text{H}_2\text{O}$, $\text{UO}_2[\text{C}_{14}\text{H}_{19}\text{NO}_2\text{Cl}]_2 \cdot 6\text{H}_2\text{O}$ with the IR spectra of free ligand, we find that there are clear differences, the main difference is the disappearance of O-H peaks on the complex formation. The presence of a broad band at 3285cm^{-1} and 3269cm^{-1} in the IR spectra of uranyl complexes show that these complexes contain coordinated water molecules. The major peaks are represented in Table (2). The uranyl ion has intense absorption band at 915cm^{-1} in $\text{UO}_2[\text{C}_8\text{H}_{16}\text{NO}_2]_2 \cdot 10\text{H}_2\text{O}$, 830cm^{-1} in

$\text{UO}_2[\text{C}_{14}\text{H}_{19}\text{NO}_2\text{C}]_2 \cdot 6\text{H}_2\text{O}$ and 1000Cm^{-1} in both complexes, there are similar uranyl stretching.

The expected structures of complexes are as follows:

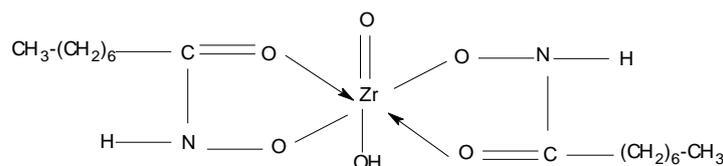


Where R=Heptyle



Zirconyl form complexes with octanohydroxamic acid only in molar ratio 1:2 (M:L), the IR spectra are listed in Table (2). An intense absorption band at 997Cm^{-1} in complex spectra which is absent in the spectra of the free corresponding hydroxamic acid is attributed to $\nu(\text{Zr}=\text{O})$ (Dehnicke and Weidlein, 1966). The Zr-O stretching frequency resulting from the interaction of oxygen of N-substituted hydroxamato-ligands occurs in the range 409Cm^{-1} (Nakomato and Martell, 1960 and Nakomato *et al.*, 1961,1962). Broad bands is observed in the range $3653\text{-}3182\text{Cm}^{-1}$ are assigned to $\nu(\text{N-H})$ and $\nu(\text{O-H})$ of coordinated water to metal ion.

The expected structure of the complex as follows:



$^1\text{HNMR}$ spectra of octanohydroxamic acid in (CDCl_3) in Table (3) shows the following signals, triplet at $\delta=0.9$ ppm ($J=8.0$ Hz) corresponding to 3proton of CH_3 , multiplet at $\delta=1.4$ ppm equivalent to 8protons of four methylene groups (4 CH_2 groups), multiplet at $\delta=1.7$ ppm corresponding to CH_2 group and triplet signal at $\delta=2.2$ ppm ($J=5.6\text{Hz}$) due to CH_2 group.

Table (3): $^1\text{HNMR}$ spectra of unsubstituted and N-substituted octabohydroxamic acids of the general formula $\text{CH}_3\text{-(CH}_2\text{)}_6\text{-CO-N(R)OH}$ measured in (CDCl_3) at 25°C .

Ligand	CH3	(CH ₂) ^v	(CH ₂) ^b	(CH ₂) ^a	N-(CH ₃)
$\begin{array}{c} \text{O} \\ \parallel \\ (\text{CH}_3\text{-(CH}_2\text{)}_4\text{-CH}_2\text{-CH}_2\text{-C-NH-OH)} \end{array}$	0.9(t)	1.4(m)	1.7(m)	2.2(t)	-
$\begin{array}{c} \text{O} \\ \parallel \\ (\text{CH}_3\text{-(CH}_2\text{)}_4\text{-CH}_2\text{-CH}_2\text{-C-N-OH)} \\ \\ \text{CH}_3 \end{array}$	0.87(t)	1.24(m)	1.6(q)	2.4(t)	3.44(s)

Table (4): The elemental analysis of unsubstituted, N-substituted octanohydroxamic acid and complexes are shown in the following the carbon, hydrogen and nitrogen are in good agreement with theoretical percentage.

Compound	C %		H %		N %	
	Found	Calc.	Found	Calc.	Found	Calc.
$\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-NH-OH}$	61.05	60.73	10.77	10.69	7.52	8.80
$\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(CH}_3\text{)-OH}$	59.6	60.00	3.40	10.98	5.50	5.80
$\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(C}_6\text{H}_4\text{Cl)-OH}$	60.1	62.40	4.60	7.34	5.90	5.20
$\text{La}[\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-NH-O}]_3 \cdot 6\text{H}_2\text{O}$	39.40	39.9	5.00	8.32	12.10	5.82
$\text{La}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(CH}_3\text{)-O})_2 \cdot 2\text{OH}] \cdot 3\text{H}_2\text{O}$	26.30	27.07	4.40	6.51	5.80	3.50
$\text{La}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(C}_6\text{H}_5\text{)-O})_2 \cdot 2\text{OH}] \cdot 3\text{H}_2\text{O}$	36.80	36.45	4.50	6.07	4.90	3.03
$\text{La}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(C}_6\text{H}_4\text{Cl)-O})_2 \cdot 2\text{OH}] \cdot 6\text{H}_2\text{O}$	30.20	30.57	4.80	6.00	4.70	2.55
$\text{Ce}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(C}_6\text{H}_4\text{Cl)-O})_2 \cdot 2\text{OH}] \cdot 8\text{H}_2\text{O}$	38.00	39.20	3.70	5.16	6.54	3.27
$\text{Th}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-NH-O})_2 \cdot 2\text{OH}]_6 \cdot 6\text{H}_2\text{O}$	27.20	27.88	4.80	6.86	3.30	4.06
$\text{Th}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(CH}_3\text{)-O})_3 \cdot 3\text{OH}] \cdot 2\text{H}_2\text{O}$	22.20	22.83	4.20	4.86	3.10	2.96
$\text{Th}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(C}_6\text{H}_5\text{)-O})_3 \cdot 3\text{OH}] \cdot 6\text{H}_2\text{O}$	25.70	25.47	4.70	5.30	1.30	2.12
$\text{UO}_2[\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-NH-O}]_2 \cdot 10\text{H}_2\text{O}$	22.50	25.06	4.50	6.78	4.20	3.65
$\text{UO}_2[\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-N(C}_6\text{H}_4\text{Cl)-O}]_2 \cdot 6\text{H}_2\text{O}$	36.00	36.70	4.10	5.46	3.90	3.06
$\text{ZrO}[(\text{CH}_3\text{-(CH}_2)_6\text{-C(=O)-NH-O})_2]_6 \cdot 6\text{H}_2\text{O}$	36.10	36.14	5.10	8.28	4.40	5.27

The disappearance of OH proton may be attributed to complex formation. ¹HNMR spectrum of N-(p-chlorophenyl) octabohydroxamic acid shows triplet signal at $\delta=0.9$ ppm ($J=7.2$ Hz) corresponding to CH₃ group, multiple signal at $\delta=1.2$ ppm equivalent to 8proton due to four CH₂ groups, triplet signal at $\delta=2.55$ ppm ($J=0.9$ Hz) corresponding to CH₂ group and two duplets at $\delta=7.2$ ppm, $\delta=7.45$ ppm ($J=7.9$ Hz) corresponding to four protons of p-substituted phenyl group. The disappearance of OH proton may be attributed to complex formation.

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تحضير بعض المعقدات الناتجة من تفاعل مشتقات الأحماض الهيدروكسيميية الثمانية

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تم فى هذا البحث دراسة معقدات ناتجة من تفاعل مشتقات الأحماض الهيدروكسيميية الثمانية مع ايونات $UO_2(VI)$, $La(III)$, $ZrO(VI)$, $Ce(IV)$ هيدروكسيل أمين وبارا كلوروفينيل هيدروكسيل أمين , والليجندات العضوية اوكتانو , ن – فينيل , ن – (4 – كلورفينيل) اوكتانو حمض الهيدروكسيمييك . وقد تم تحضير المعقدات نتيجة لتفاعل تلك الليجندات مع ايونات الفلزات السابقة , تحت التحكم فى قيم الأس الهيدروجيني . وتم إجراء التحليل الطيفى لوضع صيغ جزيئية لهذه المعقدات . وقد تم اعتماد تراكيب المعقدات الصلبة بنسبة 1:1 و 1:2 و 1:3 (فلز : ليجاند) عن طريق تقنيات أطياف الأشعة تحت الحمراء والتحليل العنصري والرنين النووي المغناطيسي لنواة الهيدروجين .

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