



Sudan University of Science and Technology

College of Graduate Studies



**Preparation, Characterization and Complexation of some
Hydroxamic Acids with Iron (III), Vanadium (V), Cobalt (II)
and Copper (II), Cations.**

تحضير وتشخيص وتعقيد بعض الأحماض الهيدروكسيميّة مع كاتيونات

الحديد (III) والفاناديوم (V) والكوبالت (II) والنحاس (II)

A thesis submitted in fulfillment for requirements of the Degree
of Doctor of Philosophy in Chemistry

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Dedication

This effort is dedicated to

The soul of my mother,

Dear father,

Lovely husband, little lovely daughters

Brother and sisters.

R ehab

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Abstract

Five hydroxamic acids were prepared through the esterification route. The prepared esters were reacted with free hydroxylamine under cold ether process. This method was found to be more applicable than the aqueous process. They were benzohydroxamic acid (BHA), cinnamohydroxamic acid (CHA) and salicylohydroxamic acid (SHA). β -phenylbenzohydroxamic acid, (β .PBHA) and β -phenylcinnamohydroxamic acid (β .PCHA), were prepared by the partial reduction of nitrobenzene with zinc dust and ammonium chloride to give β -phenyl hydroxyl amine, which was coupled first with methyl benzoate to give β -phenylbenzohydroxamic and thesecond with methylcinnamate to give β -phenylcinnamohydroxamic. The five synthesized ligands were recrystallized by hot water/acetic acid. The hydroxamic acids were characterized by their standard color test with vanadium (V) and iron (III), melting points for (BHA) (lit 131°C found 127°C), (β .PBHA) (lit 125°C found 121°C), (CHA) (lit. 114°C found 114°C), (β .PCHA) (lit 144-146°C, found 146°C), (SHA) (lit. 168-170°C, found 170°C). I.R. spectra were show the most characteristic bands associated with hydroxamic acids functional group that is due to -OH (BHA 3067, β .PBHA 3082.2,

CHA 3260.2, β .PCHA 3266.4, SHA 3288.09) C=O (BHA 1687.38, β .PBHA 1667, CHA 1663, β .PCHA 1631.67, SHA 1613.64), C-N (BHA 1289, β .PBHA 1289, CHA 1350, β .PCHA 1348.1, SHA 135.7) N-O (BHA 931, β .PBHA 932, CHA 966, β .PCHA 943.1, SHA 907.2) cm^{-1} .

The ^1H NMR spectra of the hydroxamic acids under investigation show δ (ppm) for (BHA 8.0, 7.52, 7.66, 7.97, β .PBHA 7.37, 7.57, CHA 7.37, 7.57, β .PCHA 7.38, 7.56, 7.66, SHA 7.33, 7.71) due to (Ar-H's), for (BHA 3.777, β .PBHA 3.77, CHA 4.93, β .PCHA 3.33, SHA 3.78) attributed to NH, (BHA 6.1, β .PBHA 5.74, CHA 4.945, β .PCHA 5.00, SHA 6.9) assigned to -OH.

The ^{13}C NMR spectra exhibit absorption signal due to carbonyl (C=O) nearly (BHA 167.4, β .PBHA 167.4, CHA 169.7, β .PCHA 163, SHA 166) ppm. The peak at (BHA 134, β .PBHA 132, CHA 134, β .PCHA 134, SHA 133) ppm assigned to two carbon atoms 2 and 6 of the benzene ring, the peak at (BHA 129, β .PBHA 128, CHA 128, β .PCHA 128, SHA 127) ppm assigned to two carbon atoms 3 and 5 of the benzene ring.

The prepared hydroxamic acids were complexed with Fe (III) to obtain colored complex for (BHA violet, β .PBHA red, CHA brown, β .PCHA red brown, SHA dark brown), with V(V) to obtain colored complex for (BHA violet, β .PBHA violet, CHA dark violet, β .PCHA violet, SHA dark violet), with Co(II) to obtain colored complex for (BHA pink, β .PBHA pale violet, CHA pink, β .PCHA pale violet, SHA pink) and with Cu(II) to obtain colored complex for (BHA blue, β .PBHA blue, CHA green, β .PCHA blue, SHA green). They were recrystallized with ethanol and then, characterized by I.R. spectra, ^1H NMR, ^{13}C NMR.

Stoichiometric measurements were carried for (SHA) with the metals Fe (III), V(V), Co (II) and Cu(II) (using continuous variation method (Job's method), the stoichiometric ratio of Fe-salicylhydroxamate, V-salicylhydroxamate, Co-salicylhydroxamate and Cu-salicylhydroxamate, complexes were determined by modified Job's method of continuous variation. The mole ratio of complexes were determined, in different chemical environments, and found to be in ratio of 1:3 for Fe-salicylhydroxamate, V-salicylhydroxamate, Co-salicylhydroxamate, and 1:1 for Cu-salicylhydroxamate (M:L).

المستخلص

تم تحضير خمسة أحماض هيدروكسيميية عن طريق الأسترة. حيث تفاعل الأستر المحضرمع الهيدروكسائل أمين الحر في وسط بارد من الأيثر. ووجد أن هذه الطريقة أجدى تطبيقاً من الوسط المائي. والأحماض هي حمضبنزوهايديروكسيميك، حمض سيناموهايديروكسيميك وحمض ساليسايل هايديروكسيميك. أما حمض فينايل بنزوهايديروكسيميك وحمض فينايل سيناموهايديروكسيميك فتم تحضيرهما بالإختزال الجزئي للنيتروبنزين بواسطة غبار الخارصين مع كلوريدالامونيوم لتعطي فنايل هايديروكسيل أمين ليفاعل أولاً مع ميثيل البنزوات ليعطي حمض فينايل بنزوهايديروكسيميك وثانياً مع ميثيل السينامات ليعطي حمض فينايل سيناموهايديروكسيميك. تمت تنقية الأحماض الهيدروكسيميية الخمس المحضرة بإعادة بلورتها بحمض الخل المخفف /الماء الساخن. تم تشخيص الأحماض الهيدروكسيميية الخمس المحضرة بإظهار الألوان المميزة مع الفانديوم (V) ومع الحديد (III) وأيضاً عن طريق نقاط الانصهار فحمض البنزوهايديروكسيميك اعطي 127°C وحمض بيتا- فنيل- بنزوهايديروكسيميك أعطي 121°C وحمض سيناموهايديروكسيميك اعطي 114°C وحمض بيتا- فنيل سيناموهايديروكسيميك أعطي 146°C وحمض السالسيلوهايديروكسيميك أعطي 170°C وجميع درجات الانصهار كانت قريبة من القيمة النظرية وأيضاً تم التعرف علي الاحماض الهيدروكسيميية المحضرة عن طريق اطياف الاشعة تحت الحمراء التي تشير الي أن الحزم المميزة للاحماض الهيدروكسيميية هي من المجموعات الوظيفة المطلوبة (3067, 3082.2, 3260.2, 3266.4, C=O(1613.64, 1631.67, 1663, 1667, 1687.38)OH (3288.0 N-O(907.2, 943.1, 966, 932, 931)(1348.1 C-N, 1350, 1289, 135.7, 1289) لحمض البنزوهايديروكسيميك وحمض بيتا-فنيل- بنزوهايديروكسيميك وحمض سيناموهايديروكسيميك وحمض بيتا- فنيل سيناموهايديروكسيميك وحمض السالسيلوهايديروكسيميك علي التوالي. طيف الرنين النووي المغناطيسي (هايروجين⁻ ppm) أعطى القيم 7.52, 7.66, 7.97, 8.0 يشير إلي الهيدروجين الأروماتي لحمض البنزوهايديروكسيميك والقيم 7.37, 7.57, لحمض بيتا فنيل بنزوهايديروكسيميك والقيم 7.37, 7.57 لحمض سيناموهايديروكسيميك والقيم 7.66, 7.38, 7.56, لحمض بيتا- فنيل سيناموهايديروكسيميك والقيم 7.33, 7.71 لحمض السالسيلوهايديروكسيميك.

والقيم 3.33,4.93,3.77,3.777 , 3.78 تشير إلي هيدروجين NH والقيم
6.1,5.74,4.945,5.00,6.9 تشير لهيدروجين OH في كل من حمض البنزوهيدروكسيميك

v

وحمض بيتا-فنييل- بنزوهيدروكسيميك وحمض سيناموهيدروكسيميك وحمض بيتا- فنييل
سيناموهيدروكسيميك وحمض الساليلوهيدروكسيميك علي التوالي.
طيف إمتصاص كربون ¹³ يشير الي كربون مجموعة الكربونيل (C=O) 167.4,167.4,
166,163,169.7 والقيم 133,134,134,132,134 يشير الي الكربون رقم 2,6 في حلقة
البنزين والقيم 127,128,128,128,129 يشير الي الكربون رقم 3,5 في حلقة البنزين في كل
من حمض البنزوهيدروكسيميك وحمض بيتا-فنييل- بنزوهيدروكسيميك وحمض
سيناموهيدروكسيميك وحمض بيتا- فنييل سيناموهيدروكسيميك وحمض الساليلوهيدروكسيميك
علي التوالي.

تمت مفاعلة اللواقط المحضرة مع الحديد(III) لإنتاج معقدات ملونة (حمض بنزوهيدروكسيميك-
بنفسجي, حمض بيتا-فنييل- بنزوهيدروكسيميك- أحمر, حمض سيناموهيدروكسيميك- بني , حمض بيتا-
فنييل سيناموهيدروكسيميك - بني محمر, حمض الساليلوهيدروكسيميك-بني غامق) ومع
الفانديوم(V) أعطت معقدات بنفسجية مع كل الأحماض الهيدروكسيمية ومع الكوبالت (II)
أعطت (حمض بنزوهيدروكسيميك-زهري, حمض بيتا-فنييل- بنزوهيدروكسيميك- بنفسجي فاتح
حمض سيناموهيدروكسيميك-زهري , بيتا- فنييل سيناموهيدروكسيميك- بنفسج فاتح , وحمض
الساليلوهيدروكسيميك- زهري) ومع النحاس (II) أعطت لون أرزق مع كل من حمض
بنزوهيدروكسيميك , حمض بيتا-فنييل- بنزوهيدروكسيميك, بيتا- فنييل سيناموهيدروكسيميك , ولون
أخضر مع كل من حمض سيناموهيدروكسيميك , حمض الساليلوهيدروكسيميك. ومن ثم ترسيبها
في ظروف التبريد. وتمت إعادة بلورتها بالايثانول وتشخيصها بطيف الأشعة تحت الحمراء
والرنين النووي المغناطيسي لنظير الهيدروجين-1 والرنين النووي لنظير الكربون-13.

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1:1 للمعقد النحاس .

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