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SALITSILAMIDNING Ni(II)NITRAT TUZI BILAN KOMPLEKS BIRIKMASI SINTEZI VA KRISTALL TUZILISHI

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Annotatsiya. Ushbu maqolada $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2(\text{H}_2\text{O})_2]$ (Sal-salitsilamid) tarkibli kompleks birikmaning sintezi va uning kristall tuzilishi muvaffaqiyatli qilingan. Sintez qilingan kompleks birikma tarkibi element analizi va tuzilishi rentgen strukturaviy analiz usuli yordamida o'rganildi. Nikel tanlangan ligand salitsilamid bilan karbonil guruhidagi kislorod atomi orqali koordinatsiyaga uchrashi, shuningdek koordinatsiyalanishda gidroksil guruhi qatnashmayotganligi rentgen tuzilish tahlili (RTT) usuli yordamida ko'rsatib berildi. Sintezlangan koordinatsion birikma monokristali o'stirilib, unga tegishli parametrlar ya'ni bog' uzunligi, burchaklar kattaligi va kompleks birikma molekulasidagi atomlar orasida kuzatilayotgan vodorod bog'lar aniqlandi hamda struktura tahlil qilindi. Sintezlangan kompleks birikmada nikelning koordinatsion soni 6 ga teng bo'lib, uning gibridlanishi sp^3d^2 ekanligi aniqlandi. Gidroksibenzamid izomerlari ichidan (2-gidroksibenzamid(L_1), 3-gidroksibenzamid(L_2), 4-gidroksibenzamid(L_3)) ichidan ayrim fizik-kimyoviy xossalari o'rganish orqali salitsilamidni tanlab, uni bir nechta oraliq metallar bilan kompleks birikmalari sintezi amalga oshirildi. Natijada, salitsilamidning 3d metallari bilan koordinatsion birikmalarini sintez qilishning yangi usuli yaratildi va keyingi tajribalarda qo'llash bo'yicha tavsiyalar berildi.

Kalit so‘zlar: Nikel(II)nitrat, salitsilamid, ligand, kompleks birikma, sintez, analiz, rentgen tuzilish tahlili (RTT), element analizi, koordinatsion bog‘, eruvchanlik, suyuqlanish harorati, reaksiya unumi.

СИНТЕЗ И КРИСТАЛЛИЧЕСКАЯ СТРУКТУРА КОМПЛЕКСНОГО СОЕДИНЕНИЯ САЛИЦИЛАМИДА С СОЛЮ НИТРАТ Ni(II)

Аннотация. В данной статье рассматривается синтез и кристаллическая структура комплексного соединения $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2(\text{H}_2\text{O})_2]$ (Sal-салициламид). Синтезированное комплексное соединение исследовано методами элементного анализа и рентгеноструктурного анализа. Методом рентгеноструктурного анализа (РСА) было показано, что никель координируется с выбранным лигандом салициламидом через атом кислорода карбонильной группы, а гидроксильная группа не участвует в координации. Был выращен монокристалл синтезированного координационного соединения, определены его важные параметры, а именно длина связи, величина угла и водородные связи, наблюдаемые между атомами в молекуле комплексного соединения, а также проанализирована структура. Координационное число никеля в синтезированном комплексном соединении равно 6, а его гибридизация имеет вид sp^3d^2 . Изучая некоторые физико-химические свойства изомеров гидроксибензамида (2-гидроксибензамид (L_1), 3-гидроксибензамид (L_2), 4-гидроксибензамид (L_3)) был выбран салициламид и синтезированы его комплексные соединения с несколькими промежуточными металлами. В результате разработан новый метод синтеза координационных соединений салициламида с 3d-металлами и даны рекомендации по его использованию в дальнейших экспериментах.

Ключевые слова: Нитрат никеля(II), салициламид, лиганд, комплексное соединение, синтез, анализ, рентгеноструктурный анализ (РСА), элементный анализ, координационная связь, растворимость, температура ликвидуса, выход реакции.

SYNTHESIS AND CRYSTAL STRUCTURE OF THE COMPLEX COMPOUND OF SALICYLAMIDE WITH Ni(II) NITRATE SALT

Annotation. This article discusses the synthesis and crystal structure of the complex compound $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2(\text{H}_2\text{O})_2]$ (Sal-salicylamide). The synthesized complex compound was studied using elemental analysis of the composition and structure of the complex. It was shown by X-ray structural analysis (XSA) that nickel is coordinated with the selected ligand salicylamide through the oxygen atom of the carbonyl group, and that the hydroxyl group does not participate in the coordination. A single crystal of the synthesized coordination compound was grown, its relevant parameters, namely the bond length, angle size, and hydrogen bonds observed between the atoms in the complex molecule, were determined, and the structure was analyzed. The coordination number of nickel in the synthesized complex compound is 6, and its hybridization was found to be sp^3d^2 . By studying some of the physicochemical properties of

hydroxybenzamide isomers (2-hydroxybenzamide (L_1), 3-hydroxybenzamide (L_2), 4-hydroxybenzamide (L_3)), salicylamide was selected and its complex compounds with several intermediate metals were synthesized. As a result, a new method for the synthesis of coordination compounds of salicylamide with 3d metals was developed and recommendations for its use in further experiments were given.

Key words: Nickel(II) nitrate, salicylamide, ligand, complex compound, synthesis, analysis, X-ray structural analysis (XSA), elemental analysis, coordination bond, solubility, liquidus temperature, reaction yield.

KIRISH. Bugungi kunda dunyo aholisi sonining jadal suratlar bilan o‘sayotganligini e‘tirof qilish mumkin. Bu jarayonni, 2019-2024 yillar oralig‘ida dunyo aholisining sonining quyidagi o‘zgarishlaridan ko‘rish mumkin.

1-jadval

Dunyo aholisining 2019-2024-yillar davomida demografiya statistikasi holati

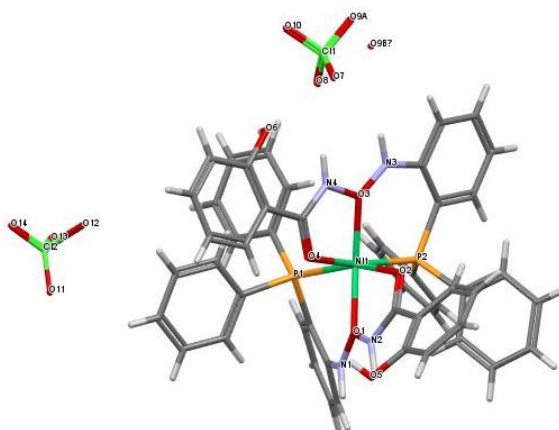
Yillar	2019	2020	2021	2022	2023	2024
Dunyo aholisining soni (mlrd hisobida)	7,674	7,809	7,888	8,016	8,074	8,114

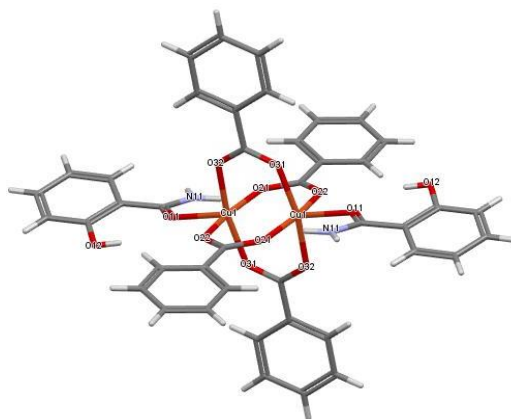
Aholi sonining ortishi bilan, tabiiyki ular o‘zi uchun qulay yashash sharoiti yaratishga intiladi. Barcha tirik jonzoqlar kabi, inson yashash sharoitiga ham albatta, abiotik va biotik omillar ta’sir qiladi. Buning natijasida hozirgi kunda ekologiyada bo‘layotgan jiddiy o‘zgarishlar tufayli inson salomatligiga ta’sir qiluvchi bakteriyalar, viruslar va zamburug‘lar soni ham keskin ortib bormoqda. Bugungi kunda, zararli bakteriyalarning -3 mlndan ortig‘i, viruslarning 2 mingdan ortiq turlari o‘rganilgan [1]. Bu bakteriya va viruslar insonlar salomatligiga, yovvoyi va uy hayvonlari sog‘ligiga va o‘simliklarning o‘sishi hamda rivojlanishiga ta’sir qilib, ularni normal funksional holatini izdan chiqaradi. Shu kabi salbiy ta’sirlarni o‘rganish orqali, o‘simliklarning o‘sishiga va hosildorligiga ijobiy ta’sir qiluvchi salitsil kislota hosilalardan salitsilamid (2-gidroksibenzamid)ning kompleks birikmalarini sintez qilish, ularni tarkibi va tuzilishini o‘rganish ishlari amalga oshirildi [2]. 3d metallardan hisoblangan nikelning salitsilamid bilan eritma muhitida koordinatsion birikmasini sintez qilindi va monokristallari o‘stirilib, uni rentgen struktura analizi amalga oshirildi hamda tegishli parametrlari aniqlandi.

ADABIYOTLAR TAHLILI VA METODLAR. Jahonning yetakchi ilmiy markazlari va kimyogar olimlari tomonidan salitsil kislota hamda uning hosilalarining koordinatsion birikmalari sintezlanib, tarkibi, tuzilishi va biologik faolligi o‘rganilganligini ko‘plab ilmiy baza ma’lumotlari, shuningdek adabiyot ma’lumotlarini tahlil qilish orqali ko‘rish mumkin [3]. Ingliz olimi E. Furiya tomonidan “Suvli eritmada temir (III) ion va 2-gidroksibenzamid o‘rtasidagi komplekslanish muvozanatini o‘rganish” nomli maqolasida suvli eritmada salitsilamid va temir

(III) tuzlarining o‘zaro tasirlashib, koordinatsion birikma hosil qilishi va reaksiya muvozanati haqida ma’lumotlar berilgan [4]. Raffaella Porto va Emilia Furialar tomonidan “Mis (II) ionining 2-gidroksibenzamid bilan kompleks birikmasi” nomli maqolasida Cu^{2+} ionining 2-gidroksibenzamid (salitsilamid, HL) bilan kompleks birikmasi 0,5 dan 3 mol/dm³ gacha bo‘lgan ion kuchi uchun NaClO_4 muhitida shisha elektrod bilan potentsiometrik o‘lchovlar bilan 25°C da o‘rganilganligi aytib o‘tilgan [5]. [T. Göktürk](#), [C. G. Topkaya](#), [T. Hökelek](#), [R. Güplar](#) tomonidan yozilgan “Oktaedral Ni(II) kompleksining serendipit sintezi: yagona kristall strukturasi, Xirshfeld sirti va bo‘shliqlar tahlili” nomi maqolasida

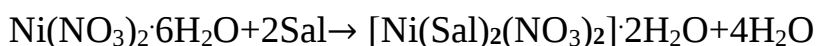
$\text{C}_{50}\text{H}_{42}\text{N}_4\text{NiO}_6\text{P}_2 \cdot 2(\text{ClO}_4)$ yangi sintezlangan Schiff asosi ligand va PPh_3 bilan reaksiyasi natijasida serendipit tarzda sintez qilindi va uning molekulyar hamda kristall tuzilmalari monokristalli rentgen tahlili bilan aniqlandi. Triklirik tizimga tegishli $a = 10,7936(4) \text{ \AA}$, $b = 12,2926(4) \text{ \AA}$, $c = 19,6272(6) \text{ \AA}$, $\alpha = 92,574(3)^\circ$, $\beta = 95,527(3)^\circ$, $\gamma = 102,898(4)^\circ$ bo‘lgan fazo guruhi, $Z = 2$ va $V = 2520.73(15) \text{ \AA}^3$.





2-rasm. [Cux[RCOO]yLz] tarkibli kompleks birikma molekulasining tuzilishi

Nikelning salitsilamid bilan kompleks birikmalarini sintez qilish jarayonida, $\text{Ni}(\text{NO}_3)_2 \cdot 2\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, $\text{Ni}(\text{NO}_3)_2$ kabi tuzlari ishlatildi. 2-gidroksibenzamid (L_1), dastlab etil spirtida eritilib, uning 0,02 mol/l, 100ml li eritmasi tayyorlab olindi. Keyin $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ tuzi suvda eritish orqali uning 0,01 mol/l, 100 ml li eritmasi tayyorlandi. Eritmalar tayyor bo'lgach, tubi dumaloq kolba olinib ($V = 200 \text{ ml}$ li) unga dastlab tuz eritmasidan 20 ml solinib, uning ustiga ligand eritmasidan 40 ml ni asta sekin to'rt marta har 15 minutdan qo'shib borildi. Bu kolba shtativga mahkamlanib, uni magnitli mishalka ustiga qo'yiladi va 60-80 minut davomida aralashtirib turildi. Keyin hosil bo'lgan aralashma xona haroratida biroz vaqt tindirish uchun tinch holatda qoldiriladi. Keyin olingan kompleks birikmaning monokristalini olish uchun, tayyorlangan aralashma to'rtta stakanga teng bo'lib solingan holda, 30°C haroratda termostatda 5-7 kun saqlanadi [8]. Olingan kompleks birikma monokristallari dimetilformamidda qayta kristallab ajratib olindi. Reaksiya unumi 78,6 %.



1-sxema. $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ tarkibli koordinatsion birikmani olish reaksiyasi

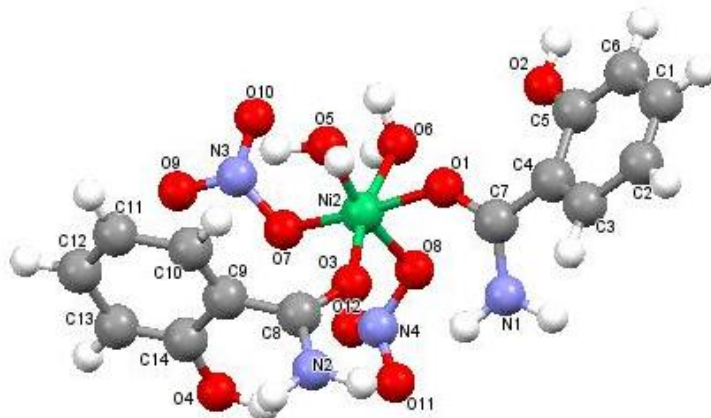
NATIJALAR VA MUHOKAMA. Sintezlangan nikelli kompleks birikmani $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ tarkibli koordinatsion birikmaning monokristali o'stirildi va rentgen strukturaviy tahlil yordamida analiz qilindi hamda tegishli parametrlari aniqlanib quyidagi jadvallarda ifodalab berildi [9,10].

2-Jadval

Nikel salitsilamidli-kompleks birikmaning kristallografik ma'lumotlari va strukturasiga aniqlik kirituvchi parametrlar

Parametr	Qiymatlar	Parametr	Qiymatlar
$[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$			
Formula	$\text{NiC}_{14}\text{H}_{18}\text{N}_4\text{O}_{12}$	Kristall o'lchami, [mm]	0.18×0.14×0.12
Molekulyar massa	493.01	Harorat T, °K	294

Singoniya	triklinik	Skanerlash oralig‘i $\theta, ^\circ\text{grad.}$	2,18; -32,18
Fazoviy guruh	A1	Interval h,k,l	-32:28; -15:13; -12:10
$a, \text{\AA}$	15.54	Jami reflekslar	6124
$b, \text{\AA}$	18.82	Mustaqil reflekslar soni	2064
$c, \text{\AA}$	22.46	R_{int}	0.056
$\alpha ^\circ$	96.2	$F^2 \geq 2\sigma(F^2)$ Kriteriy	882
$\beta ^\circ$	101.345		
$\gamma ^\circ$	105.846		
$V, \text{\AA}^3$	7514	Aniqlagan parametrlar	164
Z	2	Stukturani aniqlash sifati	1.18
$D_x, \text{g/cm}^{-3}$	0.218	$R_1, wR_2(I > 2\sigma(I))$	0.842, 0.952, 0.1234
$\mu(\text{CuK}\alpha), \text{mm}^{-1}$	0.142	$\Delta\rho_{\text{min/max}}, e\text{\AA}^{-3}$	-0.832, 0.946



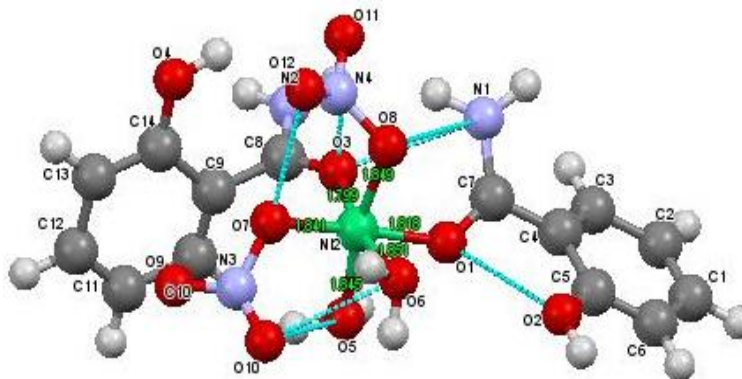
3-rasm. Sintezlangan $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ tarkibli kompleks birikma molekulasining tasviri

3-jadval

Kompleks birikmaning bog‘ uzunliklari va bog‘lanish burchaklari qiymatlari

Bog‘	$d, \text{\AA}$	Bog‘	$d, \text{\AA}$
Ni(1)-O(1)	1.8183	O(1)-Ni(1)-O(3)	89.21
Ni(1)-O(3)	1.7989	O(1)-Ni(1)-O(5)	80.63
Ni(1)-O(5)	1.8448	O(1)-Ni(1)-O(6)	78.92
Ni(1)-O(6)	1.8515	O(1)-Ni(1)-O(7)	172.38
Ni(1)-O(7)	1.8409	O(1)-Ni(1)-O(8)	89.12

Ni(1)-O(8)	1.8494	O(3)-Ni(1)-O(5)	93.37
O(1)-C(7)	1.2356	O(3)-Ni(1)-O(6)	165.54
O(2)-C(5)	1.3574	O(3)-Ni(1)-O(7)	98.27
O(3)-C(8)	1.2344	O(3)-Ni(1)-O(8)	91.34
O(4)-C(14)	1.3557	O(5)-Ni(1)-O(6)	92.79
O(7)-N(3)	1.3409	O(5)-Ni(1)-O(7)	102.31



4-rasm. Olingan $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ tarkibli kompleks birikmaning atomlararo bog‘lanishi va bog‘ uzunligi

Kristalning elementar yacheykasi parametrlari quyidagicha: fazoviy guruhi A1, $a=15.54 \text{ \AA}$, $b=18.82 \text{ \AA}$, $c=22.46 \text{ \AA}$, $\alpha=90^\circ$, $\beta=135.62^\circ$, $\gamma=90^\circ$, $V=7514 \text{ \AA}^3$, $Z=2$. $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ kompleksi monoyadroli bo‘lib Ni^{2+} ionining 2-gidroksobenzamid molekulası bilan hosil qilingan, neytral tabiatga ega [11,12]. Kompleks tarkibidagi Ni-O(1), Ni-O(3), Ni-O(5), Ni-O(6), Ni-O(7) va Ni-O(8) bog‘lari orasidagi masofasi qiymati mos ravishda $1.8183 \text{ \AA}$, $1.7989 \text{ \AA}$, $1.848 \text{ \AA}$, $1.8515 \text{ \AA}$, $1.8409 \text{ \AA}$ va $1.8 \text{ \AA}$ ga teng O(1)-Ni-O(3), O(1)-Ni-O(5), O(1)-Ni-O(6), O(1)-Ni-O(7), O(1)-Ni-O(8), O(3)-Ni-O(5), O(3)-Ni-O(6), O(3)-Ni-O(7) va O(3)-Ni(1)-O(8) ning burchak kattaliklari mos ravishda 89.21 , 80.63 , 78.92 , 172.38 , 89.12 , 93.37 , 165.54 , 98.27 va 91.34 ga teng ekanligini ko‘rish mumkin.

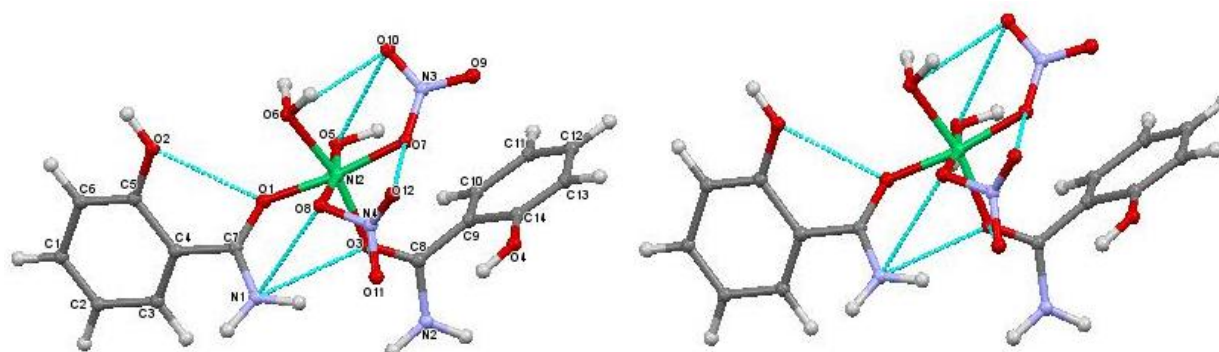
4-jadval

Kristall tuzilishidagi vodorod bog‘lar (A°)

Bog‘lanish D—H···A	Masofa, Å			Burchak D—H···A, grad.	Atom koordinatalari, A(i)
	D—H	H···A	D···A		
[Ni(Sal) ₂ (NO ₃) ₂]·2H ₂ O					
N(1)---H(1)...O(3)	1.07	2.31	2.954	118	x,-1/2+y,-1/2+z
N(1)---H(1)...O(8)	1.09	2.47	2.839	102	x,1/2+y,1/2+z
N(1)--- H(1)...O(11)	1.12	2.57	3.414	143	1/2+x,1/2+y,1/2+z
C(14)--- H(6)...N(8)	1.09	2.33	3.163	132	1/2+x,1/2+y,1/2+z

O(5)--- H(10)...O(10)	0.97	2.56	2.882	134	-1/2+x,-1/2+y, -1/2+z
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D-donor, A- akseptor. i-simmetriya



5-rasm. Olingan kompleks birikmaning atomlari orasida vodorod bog'larning vujudga kelishi

XULOSA. Sintez qilingan kompleks birikmaning tarkibi, tuzilishi va xossalari zamonaviy fizik-kimyoviy tadqiqot usullari bilan o'rganilganda, birikma tarkibidagi salitsilamidning kislorod atomi va markaziy atom nikel orasidagi bog' tabiati, mustahkam ion bog'lanish ekanligi aniqlandi. Kompleks birikmadagi markaziy atom nikel triklinik tipida, ikkita molekula salitsilamidni kislorod atomlari orqali va ikki molekula nitrat anioni hamda ikki molekula suv bilan kislorod atomi orqali koordinatsiyalanib, $[\text{Ni}(\text{Sal})_2(\text{NO}_3)_2] \cdot 2\text{H}_2\text{O}$ tarkibli kompleks birikmani xosil qiladi. Yaratilgan sintez metodi o'xshash koordinatsion birikmalarni keyinchalik sintez qilishda qo'llanilishi mumkin. Markaziy atom nikelning koordinatsion soni 6 ga teng bo'lib, sp^3d^2 xolatda gibridlangan. Sintezlangan koordinatsion birikma, 0,002% li eritma holida qishloq xo'jaligida yangi stimulyator sifatida qo'llanilishi mumkin.

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