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ISSN 3030-3907

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{Mn(2,6-PDCA)(Cl)(H_2O)}n KOMPLEKS BIRIKMASI SINTEZI VA KRISTALL TUZILISHI

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Annotatsiya. 2,6-piridindikarbon kislotasi (2,6-PDCA)ning metall ionlari bilan ta’sirlashishi va uning metallokompleks birikmalarining xossalarini tadqiq qilish maqsadida, marganes(II) atsetat va xlorid kislota bilan M:L:L 1:1:1 nisbatdagi koordinatsion soni yettiga teng bo‘lgan, polimer tuzilishli aralash ligandli yangi kompleks birikmasi olinib, uning monokristali o‘stirildi. Uning tarkibi va tuzilishi RTT yordamida aniqlandi. Strukturada 2,6-PDCA ligandining karboksil guruhining ikkita O atomi va 2,6-PDCA dagi piridinning N atomi orqali marganesga ion va donor akseptor bog‘lanish orqali bog‘lanib, ligandning tridentant holatda birikishini ta’minlagan. Shu bilan birgalikda xlorid ioni birikkan bo‘lib, bir molekula suv donor-akseptor bog‘lanish orqali bog‘langan. Hirshfeld sirtini Crystal Explorer 17.5 dasturi yordamida tahlil qilindi. IQ-spektri tahlili o‘tkazilib, ligand bilan tebranish chastotalari taqqoslandi.UB-spektri tahlili o‘tkazildi.

Kalit so‘zlar. 2,6-piridindikarbon kislotasi (2,6-PDCA, dipic), marganes(II) atsetat, xlorid kislota

Аннотация. Для изучения взаимодействия 2,6-пиридиндикарбоновой кислоты (2,6-PDCA) с ионами металлов и свойств её металлокомплексных соединений получено новое комплексное соединение со смешанным лигандом полимерного строения с координационным числом семь в соотношении M:L:L = 1:1:1 с ацетатом марганца(II) и соляной кислотой, а также выращен его монокристалл. Состав и структура определены методом РСА. В структуре два атома кислорода карбоксильной группы лиганда 2,6-PDCA и атом азота пиридина в 2,6-PDCA связаны с марганцем посредством ионной и донорно-акцепторной связи, обеспечивая тридентатное

ISSN 3030-3907

связывание лиганда. Одновременно связывается ион хлора и одна молекула воды посредством донорно-акцепторной связи. Поверхность Хиршфельда была проанализирована с помощью программы Crystal Explorer 17.5. Был проведен ИК-спектральный анализ и сравнены колебательные частоты лиганда. Был проведен анализ УФ-спектра.

Ключевые слова: 2,6-пиридиндикарбоновая кислота (2,6-PDCA, дипик), ацетат марганца(II), соляная кислота

Abstract. To study the interaction of 2,6-pyridinedicarboxylic acid (2,6-PDCA) with metal ions and the properties of its metal complex compounds, a new complex compound with a mixed ligand of polymeric structure with a coordination number of seven in the ratio M:L:L = 1:1:1 with manganese(II) acetate and hydrochloric acid was obtained, and its single crystal was grown. The composition and structure were determined by X-ray method. In the structure, two oxygen atoms of the carboxyl group of the 2,6-PDCA ligand and the pyridine nitrogen atom of 2,6-PDCA are bound to manganese via ionic and donor-acceptor bonds, providing tridentate binding of the ligand. A chloride ion and one water molecule are simultaneously bound via donor-acceptor bonds. The Hirshfeld surface was analyzed using Crystal Explorer 17.5. IR spectral analysis was performed, and the vibrational frequencies of the ligand were compared. UV spectrum analysis was carried out.

Keywords: 2,6-pyridinedicarboxylic acid (2,6-PDCA, dipic), manganese(II) acetate, hydrochloric acid

KIRISH.

2,6-piridindikarbon kislotasi (2,6-PDCA, dipic) bir yoki bir nechta karboksilat kislorod atomlari orqali barqaror komplekslar hosil qiluvchi ko‘p qirrali N, O-xelatlovchi, bidentat va tridentat xelatlovchi liganddir [1-4]. Qiziqishning sababi shundaki, ligand H2pda va markaziy piridin halqasi va ikkita karboksilat guruhi o‘rtasida qattiq 120 ° burchakka ega va shuning uchun hosil bo'lish uchun turli xil bog‘lanish usullarini ta'minlay oladi [5].

So‘nggi paytlarda monoetanolaminlar va oddiy bir o‘rnini bosuvchi benzoy kislotalar asosidagi aralash ligandlardan tayyorlangan oltita muvofiqlashtirilgan mono- va ikki yadroli mis komplekslari, xususan, monoetanolamin (MEA) va p-nitroben kislotasi (p-nitrobenzo) va yuqori darajadagi mualliflar tomonidan nashr etilgan $[Cu^{2+}(MEA)_2]$ va $[2Cu^{2+}\ (MEA)_2(H_2O)_2)]$. Yann-Teller effekti tufayli buzilgan oktaedrik tuzilishli komplekslar. Bitta $[Ni(dipic)_2](mea)_2$ tarkibli yangi kompleks birikmaning monokristali piridin-2,6-dikarboksilik kislota (PDC yoki dipic) bilan (MEA) aralashgan holatda o‘stirildi [6,7].

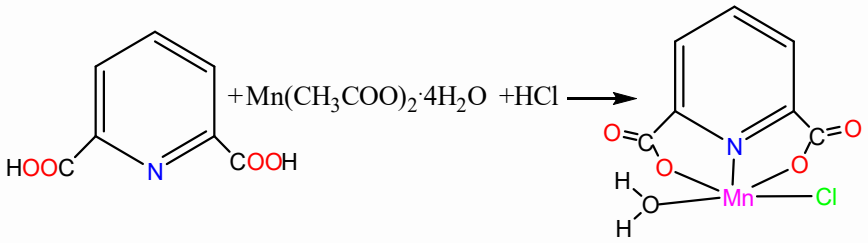
Hozirgi vaqtda kimyogarlar metall ionlariga biriktirilgan donor atomlarining soniga qarab di, tri, tetra yoki ko‘p ligandli komplekslar bo‘lishi

ISSN 3030-3907

mumkin bo'lgan qiziqarli fizik-kimyoviy xossalari tufayli aralash ligand komplekslari bo'yicha keng qamrovli tadqiqotlar olib bormoqda [8-11].

TAJRIBAVIY QISM. Yuqori sifatli kristallarni olish muhimligini hisobga olib, barcha ishlatiladigan kimyoviy moddalar va reagentlar tijorat manbalaridan (Sigma-Aldrich) olingan. $Mn(CH_3COO)_2{\cdot}4H_2O$ (98%) va 2,6 -PDCA (99%) qo'shimcha tozalashsiz ishlatilgan. Ushbu sintezda xlorid kislota va distillangan suv ham ishlatilgan.

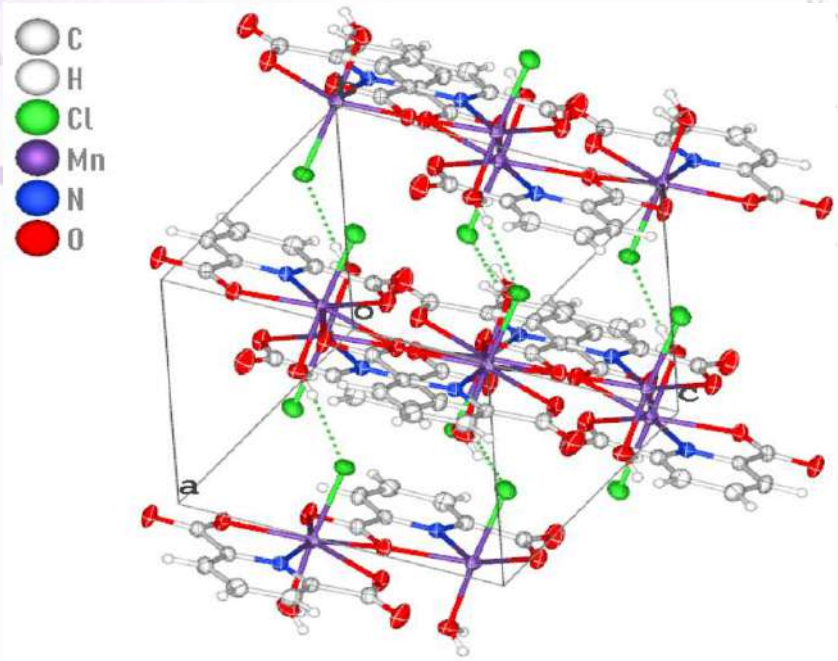
Marganes(II)atsetat kristallogidratidan $Mn(CH_3COO)_2{\cdot}4H_2O$ 0.245 gr (1 mmol), 0.167 gr (1 mmol) 2,6 -PDCA ni tegishlicha suvda hamda xlorid kislotaning suyultirilgan eritmasini 1;1;1 mol nisbatdagi eritmalari tayyorlandi. Eritmalar aralashtirildi. Hosil bo‘lgan cho‘kmani xlorid kislotaning suyultirilgan eritmasidan tomizildi. So‘ngra magnitli aralashtirgich yordamida 60 ^{0}C da 30 minut davomida intensiv aralashtirildi. Eritma xona haroratida qoldirildi. Natijada 10 kundan so‘ng idish tubida sariq rangli kompleks birikma kristali o‘sganligi kuzatildi. RTT analizi uchun yaroqli kristallar ajratilib, tekshirilganda [Mn(2,6-PDCA)(Cl)(H_2O)]n tarkibli ekanligi aniqlandi. Unumi 86 % {Mn(2,6-PDCA)(Cl)(H_2O)}n (Mr=220.5 g/mol) $C_7H_7NO_5Cl$ tahlili nazariy jihatdan: C 38.09, H 3.17, N 6.35,O 36.28 Cl 16.1 % ni ko‘rsatdi: ma’lum bo‘ldiki C 37.86, H 3.08, N 6.16, O 36.16, Cl 15.90 %. Reaksiya tenglamasi quyidagicha(1-sxema).



1-sxema. {Mn(2,6-PDCA)(Cl)(H_2O)}n kompleksi sintezi

TAHLIL NATIJALARI. Ushbu kompleks birikma {Mn(2,6-PDCA)(Cl)(H_2O)}$_n$ (($C_7H_5ClMnNO_5$)$_n$) tarkibli, monoclinic Singoniyaga ega va fazoviy guruhi P21/c.

ISSN 3030-3907

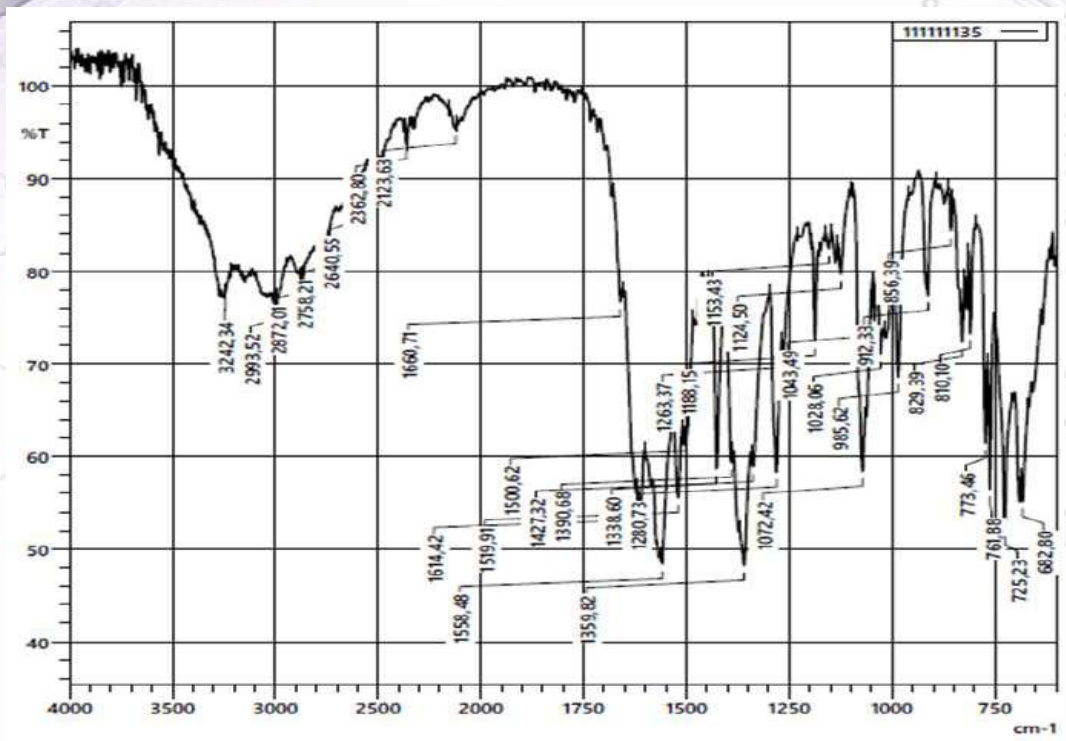


1-rasm. {Mn(2,6-PDCA)(Cl)(H_2O)}$_n$ kompleksining (a)molekulyar tuzilishi, (b) polimer zanjir hosil qilishi

Strukturada 2,6-PDCA ligandining karboksil guruhining ikkita O atomi va 2,6-PDCA dagi piridinning N atomi orqali marganesga ion va donor akseptor bog‘lanish orqali bog‘lanib, ligandning tridentant holatda birikishini ta’minlagan. Shu bilan birgalikda xlorid ioni birikkan bo‘lib, bir molekula suv donor-akseptor bog‘lanish orqali bog‘langan. Mn(II) ioni qo‘shni 2,6-PDCA ning karboksil ioni porqali bog‘lanishi marganesning koordinatsion yetti bo‘lishini ta’minlagan, shuning hisobiga kompleks polimer tuzilishli bo‘lgan. π - π stascing orasidagi mosafa 3.772 Å ekanligi pluton dasturi yordamida aniqlangan. Bog‘lanish uzunligi Mn1 -Cl1= 2.4404(10), Mn1 -O1= 2.250(2), Mn1-N1= 2.266(2) va Mn1 -O3 = 2.423(2) , Mn1 -O5=2.156(3) Å. Bog‘ burchaklarining ham turli xil ekanligi kuzatiladi. Cl1-Mn1 -O1 94.53(6), Mn1-N1 -C5 123.47(18), Cl1 -Mn1 -O3 90.67(7), Cl1 -Mn1 -O5 176.56 (7). Ligand geometriyasi tomonidan qo‘yilgan cheklovlar tufayli sezilarli darajada egilgan eksenel pozitsiyalarni kislorod atomlari egallaydi.

Ushbu kompleks birikmaning IQ-spektri tahliliga ko‘ra OH-guruhiga xos keng intensiv valent tebranish 3242 sm^{-1} sohada kuzatilgan (2-rasm).

ISSN 3030-3907



2-rasm. {Mn(2,6-PDCA)(Cl)(H_2O)}$_n$ kompleksining IQ-spektri

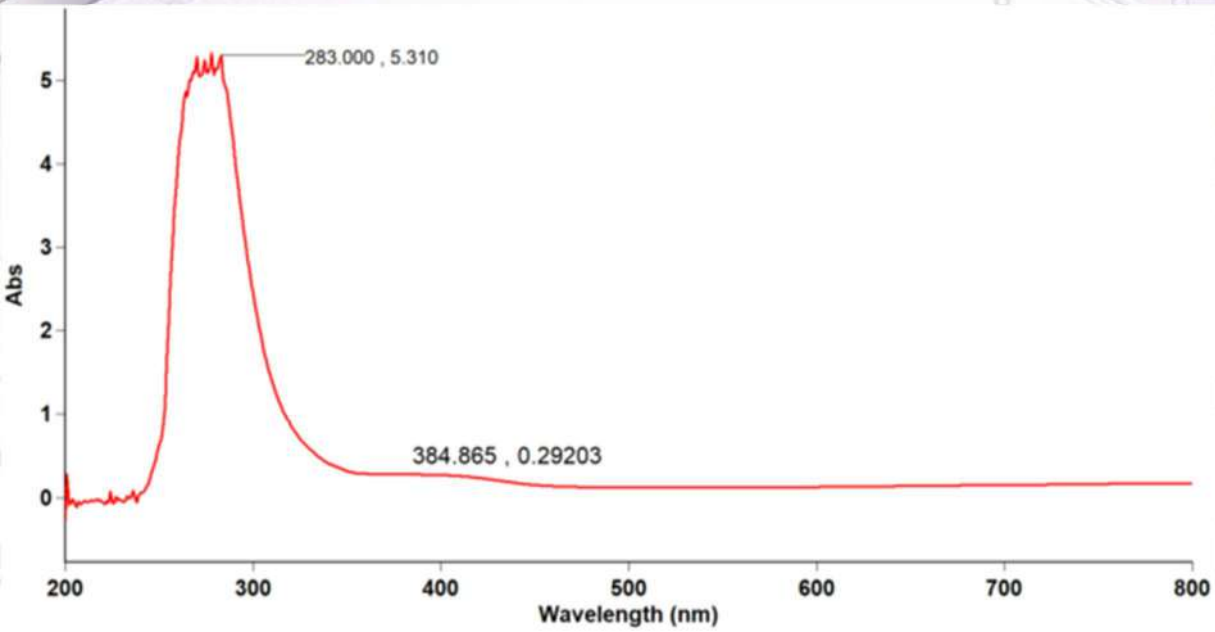
Bundan tashqari kompleksda C-O guruhiga xos tebranish 1558 sm^{-1} da paydo bo'ldi. Kompleks uchun neytral karboksilat guruhining n(COO−) va n(COO−) tebranishlari mos ravishda 1519 va 1421 sm^{-1} da paydo bo‘lgan. 1660-1427 sm^{-1} oralig‘ida C = N guruhiga xos tebranish, M-O va M-N bog‘lanishi uchun 682- 628 sm^{-1} kuzatildi (1-jadval).

1-jadval.

2,6-PDCA ligandi va {Mn(2,6-PDCA)(Cl)(H_2O)}$_n$ kompleksining IQ-spektri tahlii sm^{-1}

Tebranish	OH-	-C=N	Piridin halqasi	–C-H	Me-O	Me-N
2,6-PDCA	3063-3103	1687.71	1573	852	-	-
{Mn(2,6-PDCA)(Cl)(H_2O)}$_n$	3242	1660	1558	856	682	628

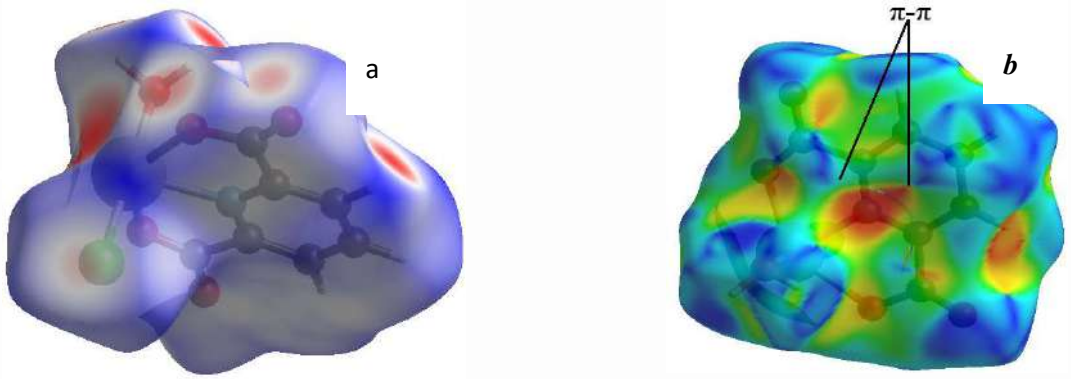
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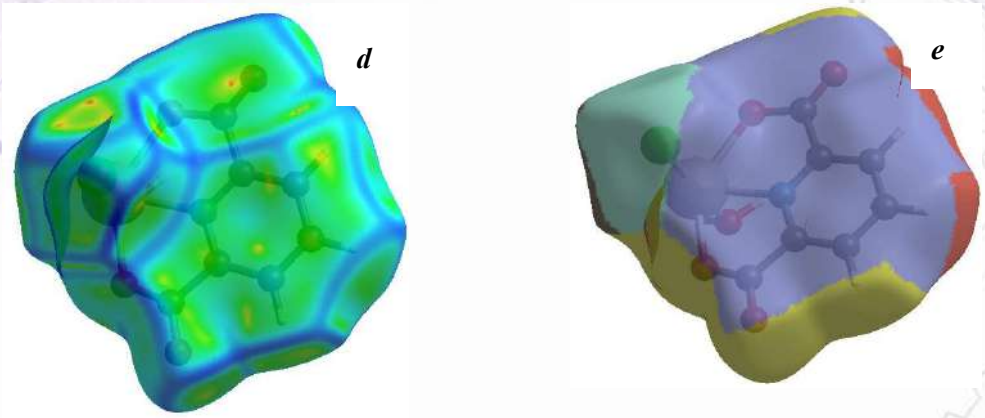
3-rasm. {Mn(2,6-PDCA)(Cl)(H_2O)}$_n$ kompleksning UB-spektri

3-rasmda ko'rsatilgan. Mn(II) kompleksining elektron spektrlari xona haroratida $1–10^{-3}$ M konsentratsiyali H_2O_2 erituvchisida qayd etilgan. Yutilish polosalarining intensivligi ma'lum bir mintaqada mavjud edi. Mn(II) kompleksi uchun cho‘qqilar mos ravishda 283 nm, 384 nm da kuzatilgan. Kompleks uchun ham π-π o‘tishi va metalldan ligandga zaryad o‘tishi tufayli 283 dan 384 nm gacha xarakteristikalarning o‘tishi kuzatilgan.

Hirshfeld sirtini Crystal Explorer17.5 dasturi yordamida tahlil qilindi. Qizil va ko‘k dog‘larning d_{norm} bo‘yicha standart o‘lchamlari mos ravishda -0,6476 va 1,1469.



ISSN 3030-3907



4-rasm. Hirshfeld surface (a), shape index(*b*), curvedness (*d*) and fragment patches(*e*).

Bu yerda qizil rang van der Waals radiuslari yig‘indisiga yaqinroq aloqalarni ko’rsatadi. Ko‘k dog‘lar esa van der Waals radiuslari yig‘indisidan uzoqroq aloqalarni ko‘rsatadi.Ushbu kompleks birikmaning d_{norm} bo‘yicha molekulaning Hirshfeld sirtida qizil dog‘larning asosiy qismini vodorod bog‘lar tashkil qiladi (*4-rasm*). Hirshfeld sirt yuzasi S= 222,94 Å² va hajmi V= 227,52 Å³ ga teng. Kristall tarkibida ichki zarrachalarning o‘zaro ta’siri *di* va kristall sirtidagi o’zaro ta’sirlarni *de* ikki o‘lchamli funksiyalari yordamida aniqlandi. Molekula tarkibidagi atomlarning asosiy o‘zaro ta’sirlashuvlarni O…H/H…O (21.4%), H…Cl/Cl…H (16.6%), H…H (13.6%), O…O 10.3%, H…C/C…H 9.7%, Cl…O/O…Cl 7.7%, Mn…O/O…Mn 7.0 %, O…C/C…O 6.6 %, C…C 4.7%,H…N/N…H 1.1 %, O…N/N…O 0.8%, Cl…C/C… Cl 0.5%, N…C/C…N 0.2%, Mn…C/C…Mn 0.1% hissasiga to‘g‘ri keladi.

XULOSA. Tadqiqotlar natijasida 2,6-piridindikarbon kislotasining marganes(II) atsetat va xlorid kislotaning M:L:L 1:1:1 nisbatdagi polimer tuzilishli kompleks birikmasi sintez qilindi. Sintez qilingan kompleksning tarkibi va tuzilishi RTT yordamida aniqlandi. Kompleks birikmaning kristallografik ma’lumotlari Kembridj kristallografik ma’lumotlar bazasiga (Cambridge Crystallographic Data Center) ga kiritildi(CCDC2403742). https://www.ccdc.cam.ac.uk/structures/Search?Ccdc=2403742&Author=nazarov&Access=referee. Hirshfeld sirti tahliliga ko‘ra molekula tarkibidagi atomlarning asosiy o‘zaro ta’sirlashuvlarni O…H/H…O (21.4%), H…Cl/Cl…H (16.6%), H…H (13.6%). UB-spektri cho‘qqilari aniqlandi.

FOYDALANILGAN ADABIYOTLAR RO‘YXATI.

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