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SYNTHESIS OF THE DI(2-IZOPROPYL-5-METHYLPHENYLKARBOKSIMETHYLEN) OXALATE

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The purpose of the research is study of the reaction between natrium salt of oxalic acid and O-chloracetyl tymole in presence of dimethylformamide. High yields have been achieved by the reaction of di(2-isopropyl-5-methylphenylcarboxymethylene)oxalates in the presence of dimethylformamide as a solvent. The structure of the prepared compounds are installed by means of IR and NMR spectroscopy.

Keywords: O-chloracetyl tymole, tymole, chloroacetylchloride, chloroacetylation, natrium oxalate, nucleophilic substitution, dimethylformamide, chromatography, spectroscopy

СИНТЕЗ ДИ(2-ИЗОПРОПИЛ-5-МЕТИЛФЕНИЛКАРБОКСИМЕТИЛЕН) ОКСАЛАТА

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Целью исследования является изучение реакций хлорацетилирования тимола и оксалата натрия с О-хлорацетилтимолом в присутствии диметилформамида. Высокие выходы достигнуты реакцией ди(2-изопропил-5-метилфенилкарбоксиметилен) оксалатав в присутствии диметилформамида в качестве растворителя. Строение полученных веществ установлено методами ИК и ЯМР спектроскопии.

Ключевые слова: О-хлорацетилтимол, тимол, хлорацетилхлорид, хлорацетилирование, оксалат натрия, нуклеофильная замещения, диметилформамид, хроматография, спектроскопия

DI(2-IZOPROPIL-5-METILFENILKARBOKSIMETILEN) OKSALAT SINTEZI

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Tadqiqotning maqsadi timolni xloratsetillash va O-xloratsetiltimolning natriy oksalat bilan dimetilformamid erituvchida reaksiyalari asosida sintezlar olib borishdan iborat. Dimetilformamid erituvchi sifatida ishlatilganda di(2-izopropil-5-metilfenilkarboksimetilen) oksalat yuqori unum bilan olingan. Olingan moddalarning tuzilishlari IQ va YaMR spektroskopiya usullari bilan o'rnatilgan.

Kalit so'zlar: O-xloratsetiltimol, timol, xloratsetilxlorid, xloratsetillash, natriy oksalat, nukleofil almashinish, dimetilformamid, xromatografiya, spektroskopiya

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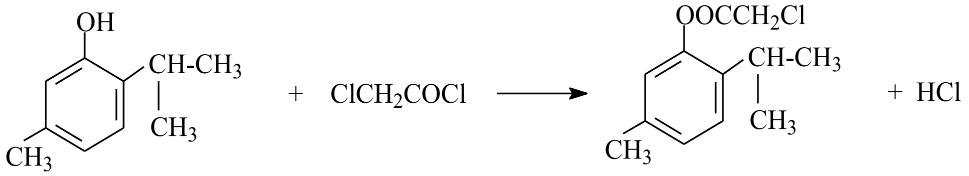
Kirish

Timol ko'plab tabiiy birikmalarning asosini tashkil qiladi. Tarkibida aromatik halqa tutgan, siklik fenol terpenoidlari o'simliklardan ajratib olingan. Ularning hosilalari yuqori biologik xossalari tufayli katta ahamiyatga ega. Timol asosidagi moddalar molekulasida bir necha reaksion markazlar borligi bu birikmalarni elektrofil va nukleofil almashinish reaksiyalarini tahlil qilish, ularni kimyoviy o'rganish qiziqligidan dalolat beradi. Shuning uchun timolni reaksion qobiliyatini o'rganish, ularning yo'nalishi qonuniyatlarini umumiy va farqlanuvchi tomonlarini aniqlash, yangi sintez qilingan moddalarni xromatografiya usulida tahlil qilish va ular orasida biologik faol birikmalarni izlash dolzarb masala hisoblanadi.

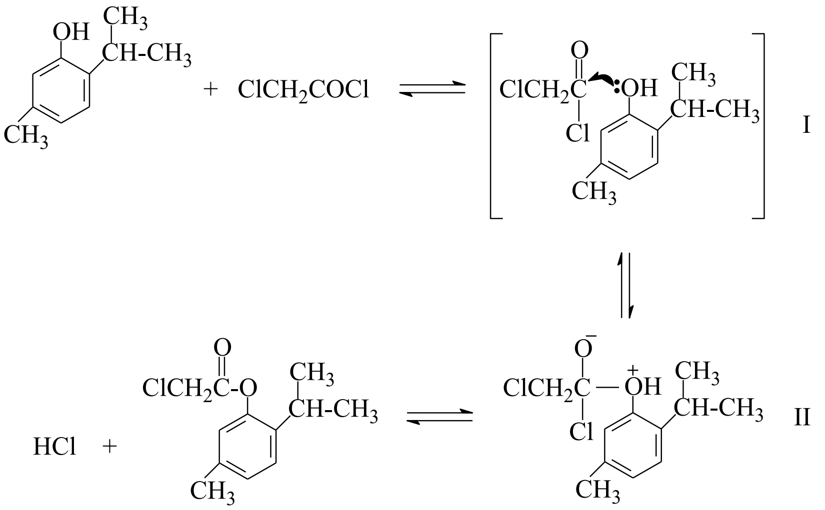
Maqoladan ko'zlangan asosiy maqsad - timol qatorida ko'plab birikmalar sintez qilinganligi, ularning reaksion qobiliyatlari va biologik faolligi o'rganilgan bo'lishiga qaramasdan O-xloratsetiltimol sintezi, uning kimyoviy xossalari hamda biologik faolligi to'g'risida adabiyotlarda ma'lumotlar deyarli yo'q. Shulardan kelib chiqib mazkur maqola di(2-izopropil-5-metilfenilkarboksimetilen) oksalat sintezini amalga oshirish, olingan birikmani zamonaviy spektroskopiya va yupqa qavatli xromatografiya yordamida tahlil qilishga qaratilgan.

Timolni xloratsetillash reaksiyasi borishini chuqurroq o'rganish maqsadida uni katalizatorsiz turli organik erituvchilar muhitida olib borildi.

Timolni katalizatorsiz organik erituvchi muhitida xloratsetillanganda reaksiya regioselektiv borib, tegishli murakkab efirlar hosil bo'lishi ko'rsatib berildi. Masalan, timolni xloratsetillash reaksiyasi xloroform muhitida olib borilganda faqat O-xloratsetillash reaksiyasi borib, xloratsetiltimol 95% unum bilan hosil bo'ladi:

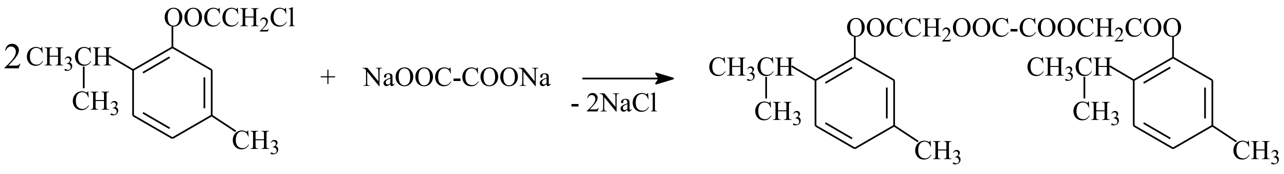


Timol va xloratsetilxloridning reaksiyasi vaqtida xloratsetilxlorid molekulasida elektron zichlik elektromanfiy kislorod tomon siljigan bo'ladi va kislorod qisman manfiy zaryadga ega bo'ladi. Elektromanfiy xlor va kislorod atomlarining ta'siri natijasida uglerod atomi esa qisman musbat zaryadga ega bo'ladi va timol molekulasidagi gidroksil guruhning juft elektronlari bilan ta'sirlashib, quyidagi I kompleksni hosil qiladi [1-5]. Reaksiya davomida kislorod va uglerod o'rtasida valent bog'i hosil bo'lib, II kompleks hosil bo'ladi va undan vodorod xlorid bilan reaksiya mahsuloti ajralib chiqadi:



Yuqoridagi reaksiya organik erituvchi (xloroform) da va katalizator ishtirokisiz borganligi uchun atsil kationi (ion juftlardagi) hosil bo'lmaydi. Shuning uchun bu reaksiya xloratsetilxlorid molekulasi karbonil guruhida nukleofil almashinish tarzida boradi va (masalan, timol bilan xloratsetilxloridning xloroform eritmasidagi reaksiyasida) efir mahsuloti hosil bo'ladi. Nukleofil reagent vazifasini esa timol molekulalaridagi kislorod atomi bajaradi [6-10].

Organik reaksiyalarning eng muhim va keng tarqalgan reaktsiya turlaridan biri to'yingan uglerod atomida boradigan nukleofil almashinish reaksiyasidir. Shuni aytish lozimki, nukleofil almashinish reaktsiyasi organik moddalarni sintez qilishda ko'p qo'llaniladi. Organik kimyo fanida reaktsiya mexanizmi haqidagi fundamental tushunchalarni paydo bo'lishida va rivojlanishida to'yingan uglerod atomida boradigan nukleofil almashinish reaksiyalari muhim rol o'ynaydi [11-15]. Ma'lumki, bipolyar aproton erituvchilar (DMSO, DMFA, TGF, atseton, dioksan) karbon kislota tuzidagi kationni solvatlash xisobiga alkilgalogenidlar bilan bimolekulyar nukleofil almashinish reaksiyasini osonlashtiradi. O-xloratsetiltimol bilan oksalat kislotaning dinatriyli tuzini dimetilformamid ishtirokidagi reaksiya tenglamasiga bog'liq holda reaksiya sxemasini quyidagicha taklif qilish mumkin:



Bu sxemani boshqa karbon kislotalarning reaksiyalarga ham qo'llash mumkin[16-20].

Tadqiqotning maqsadi timolni O-xloratsetillash reaksiyalari asosida sintezlar olib borishdan iborat.

Tadqiqot usullari

Reaktsiya mahsulotlarining yupqa qatlam xromatografiyasi Silufol - 254 plastinkasida o'tkazildi. Sintez qilingan birikmalarning IQ spektrlari Carl-Siess firmasining Specord belgili uskunasida (diapazoni 400-4000 sm^{-1}, o'lchamlari 4 sm^{-1}) olindi. Sintez qilingan birikmalarning namunalari 1H- va 13C-YaMR- spektrlari UNITY 400 plus (Varian) uskunasida o'rganildi. Spektrlarning talqini spektrlarni avtomatik ravishda o'lchashni amalga oshiradigan, spektrlarni va ularning parchalarini grafik tarzda namoyish etish vositalariga ega bo'lgan va foydalanuvchi spektrlari bilan ishlashni ta'minlaydigan asosiy dasturiy ta'minot yordamida amalga oshirildi [21-25].

O-xloratsetiltimolni olish. Vodorod xlorid chiqishiga moslangan naycha va qaytarma sovutkich bilan jihozlangan dumaloq tubli kolbaga 15g (0,1 mol) timol solinib, uni 50 ml geptanda eritildi. Timol to'liq erib ketgandan so'ng eritmaga 11.3g (0,1 mol) xloratsetilxlorid tomchilatib quyiladi va reaksiya aralashmasi 20 soat davomida 100 ^{o}C haroratda qizdirildi. Reaksiya tugaganligini vodorod xlorid gazining ajralishi to'xtashidan aniqlandi, erituvchi haydaldi, reaksiya mahsuloti ajratib olindi. Reaksiya unumi 21.5g (95%). Rf=0,60 (geksan-etilatsetat-etanol 10:20:1).

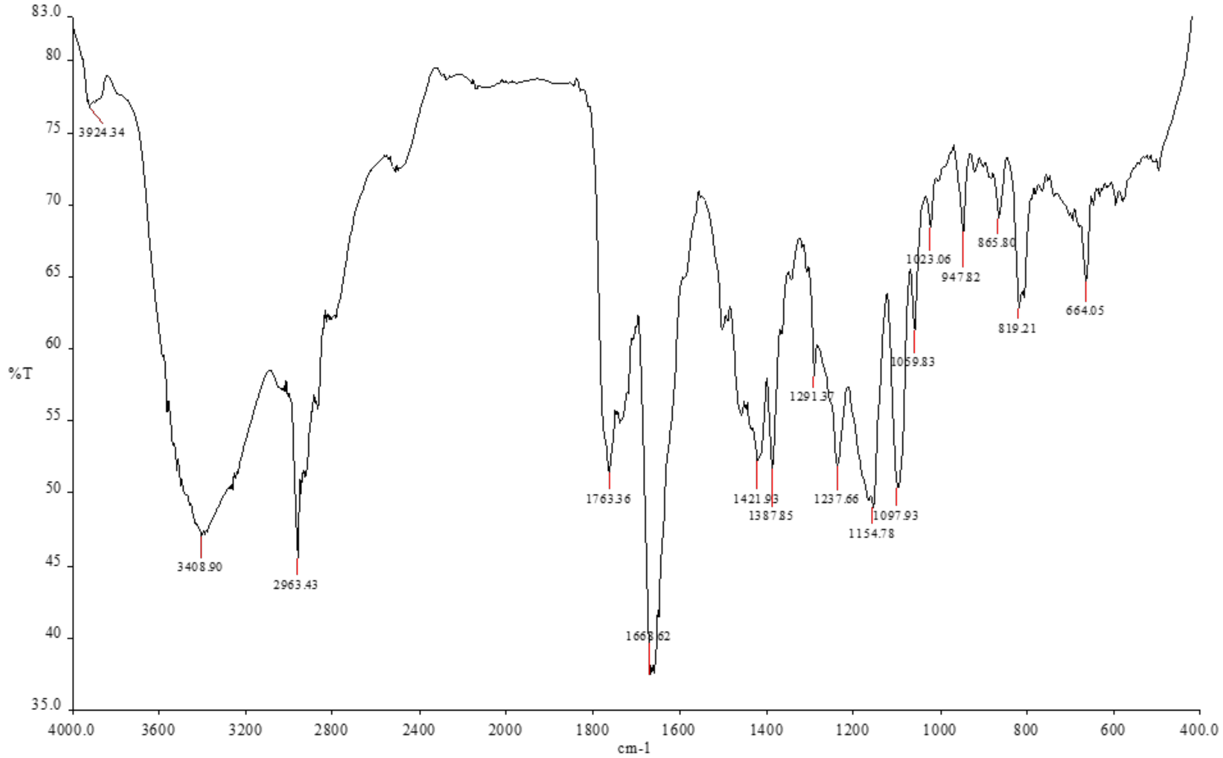
O-xloratsetiltimol va natriy oksalat reaksiyasidan di(2-izopropil-5-metilfenilkarboksimetilen) oksalatni olish. Keyingi tajriba aralashmani qaynatish bilan olib borildi: teskari sovutkichga ulangan, hajmi 100 ml bo'lgan tubi yumaloq kolbaga 1,9 g (0,01 mol) natriy oksalat, 2,63 g (0,01 mol) O-xloratsetiltimol va 15 ml dimetilformamid solib, reaksiya aralashmasi 1 soat davomida qaynatildi. Reaksiya tugagach, reaksiya aralashmasi sovutildi va suv bilan yuvildi hamda etilatsetat yordamidi ekstraksiya qilinib, efirli qism $CaCl_2$ bilan quritildi. Suv nasosida etilatsetat haydaldi va mahsulot ajratildi. Suyuqlanish harorati 53 ^{o}C (etanol). Mahsulot unumi 3,8 g (78 %), Rf=0,72 (elyuent benzol-etanol 3:1).

Natijalar va muhokama

Timolni regioselektiv O-xloratsetillash reaksiyasi o'rganilmagan. Reaksiya faqat erituvchi muhitida amalga oshirilganda atsil mahsulot O-xloratsetiltimol hosil bo'lib, mahsulot unumi 95% ni tashkil etdi.

Timolni xloratsetillash reaksiyasini sistematik tarzda o'rganish uchun, reaksiya turli organik erituvchilar yordamida olib borildi. Bu reaksiyada erituvchining tabiatini, miqdorini, harorat, reagentlar nisbatlarining reaksiya unumiga va mahsulot tarkibiga ta'siri o'rganildi [26-30].

Timolni xloratsetillash mahsuloti va xloratsetil mahsulot asosida sintez qilingan moddalarning IQ spektrlari ularning tuzilishini tasdiqlaydi.



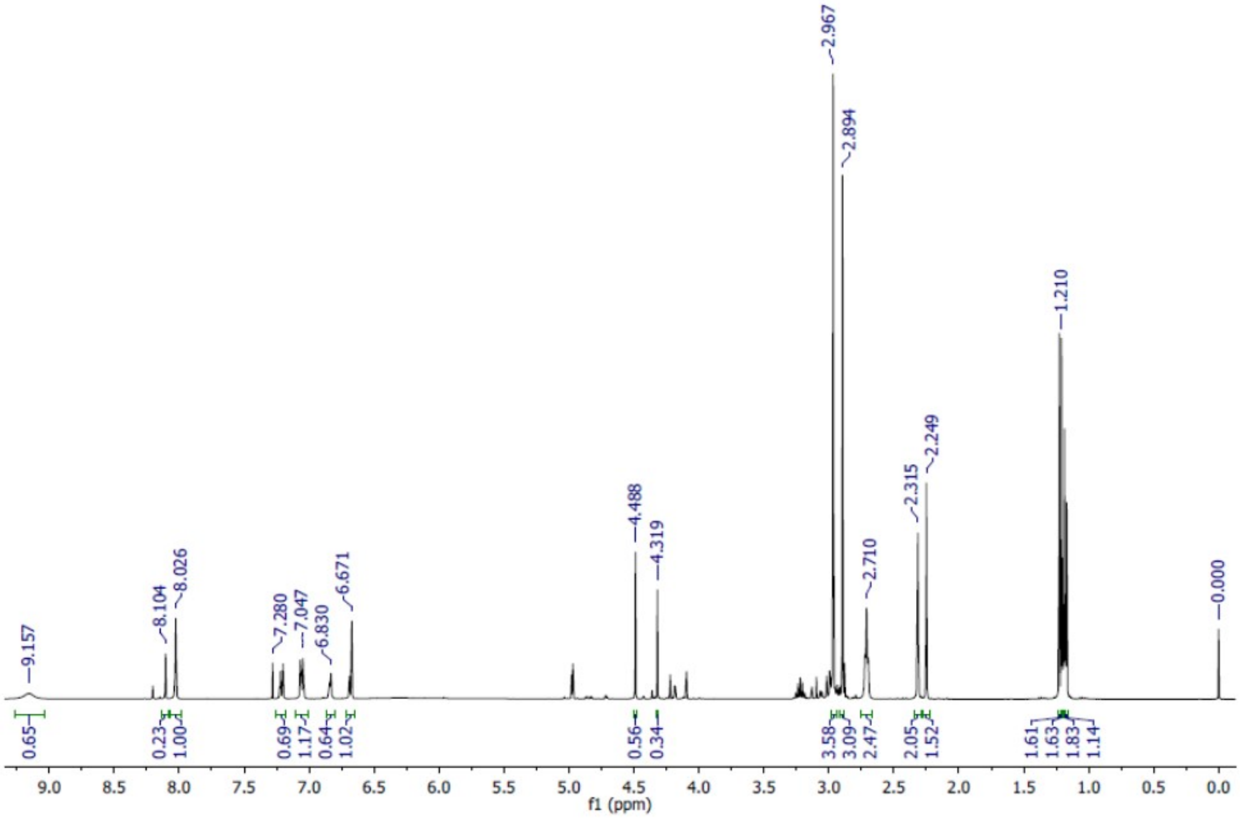
1-rasm. Di(2-izopropil-5-metilfenilkarboksimetilen) oksalatning IQ spektri.

1-Rasmda keltirilgan di(2-izopropil-5-metilfenilkarboksimetilen) oksalatning IQ spektri birikmadagi aromatik halqa, karbonil guruhi, C-Cl, =C-O-C, -CH$_2$CO- bog'larning halqadagi almashinishi tiplarini ko'rsatuvchi tebranish sohalarini ko'rsatadi. Sintez qilingan moddaning IQ spektrida 1218-1089 sm^{-1} sohada C-O-C atsetoksi guruhining valent tebranishi, 3002-2963 sm^{-1} sohada aromatik birikmadagi CH guruhining valent tebranishlari kuzatildi. 1505-1406 sm^{-1} sohada aromatik halqaning C=C bog'ining valent tebranishi, 1308 sm^{-1} sohada metilen guruhining simmetrik deformatsion tebranishi, 1021 sm^{-1} da murakkab efir bog'larining assimetrik valent tebranishi, 737 sm^{-1} da orto-almashgan benzol halqasining yassi bo'lmagan deformatsion tebranishi namoyon bo'ldi. 1768 sm^{-1} sohada esa karbonil guruhidagi C=O bog'ining valent tebranishi, 1505-1597 sm^{-1} o'tish sohasida esa aromatik halqa skeletining tebranish chiziqlari paydo bo'ldi. Metilen guruhidagi C-H bog'ining deformatsion tebranishi ($\delta_{C\text{-}H}$) 1447 sm^{-1} kuzatiladi. Monoxlor almashgan alkil- va arilgalogenidlarda C-Cl bog'ining valent tebranishlari 812-559 sm^{-1} sohada intensivligi o'rtacha tebranishlar hosil qildi.

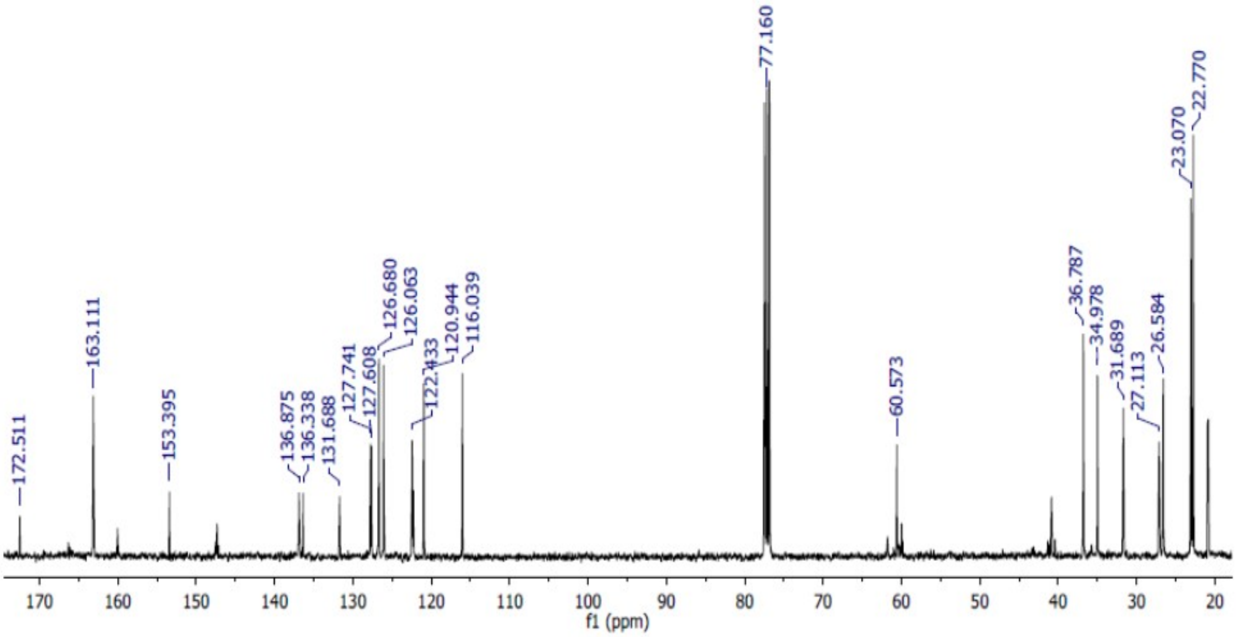
Shunday qilib, sintez qilib olingan moddalarning IQ spektrlari tahlili asosida, ularning kimyoviy tuzilishlari to'g'risidagi taxminlar o'zining ma'lum miqdordagi tasdig'ini topdi deyish mumkin. Olingan ma'lumotlar yanada ishonchli va to'laroq bo'lishi uchun sintez qilingan moddaning YaMR-spektri olindi va tahlili amalg oshirildi.

Di(2-izopropil-5-metilfenilkarboksimetilen) oksalatning 1H-YaMR - spektrida metilen guruhidagi vodorod protonlari 4.48 m.u. da singlet kimyoviy siljish, aromatik halqadagi vodorod protonlari esa 6.671, 6.83, 7.047, 7.28 m.u. larda kimyoviy siljish namoyon qiladi (2-Rasm). Chunki, benzol halqasida 1,4- holatda xlor va karboksil guruhi joylashgan, natijada aromatik halqadagi vodorod protonlarining kimyoviy siljishi bir-biridan farq qiladi. Metil (-CH3) guruhidagi vodorod atomlari 1.21 m.u. da kimyoviy siljish beradi.

3-Rasmda keltirilgan di(2-izopropil-5-metilfenilkarboksimetilen) oksalatning C-H gurux 13C- spektrida dublet signali hosil qiladi, uning konstantasi JCH=126 Gs ga teng. Agar uglerod



2 rasm. Di(2-izopropil-5-metilfenilkarboksimetilen) oksalatning ^{1}H-YaMR spektri.



3-rasm. Di(2-izopropil-5-metilfenilkarboksimetilen) oksalatning ^{13}C-YaMR spektri.

atomi qo'shbog' bilan bog'langan bo'lsa, xuddi shunga o'xshash C=C-H guruxida konstanta qiymati taxminan 163 Gs ga teng bo'ladi.

Xulosa

Tahlillardan shuni xulosa qilish mumkinki, O-xloratsetiltimolning natriy oksalat bilan dimetilformamid erituvchida olib borilganda di(2-izopropil-5-metilfenilkarboksimetilen) oksalatni yuqori unum bilan olish mumkin. Olingan birikmalarning kimyoviy tarkibi va taklif qilingan tuzilishi IQ spektroskopiyada karbonil guruhining intensiv valent tebranishining namoyon bo'lishi va 3000-3600 sm^{-1} sohada oberton tebranishning kuzatilishi, shuningdek YaMR spektroskopiya usulida metilen hamda aromatic halqa protonlarining kimyoviy siljishlarini taqqoslash natijasida aniqlandi.

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