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5-ARIL-2-AMINO-1,3,4-TIADIAZOLLARNING SHIFF ASOSLARI SINTEZI

Annotatsiya

2-(R-fenil)gidrazin-1-karbotioamidlar asosida olingan 5-(fenil, 2,4-dixlorfenil)-2-amino-1,3,4-tiadiazollarning aromatik aldegidlar bilan reaksiyalari qulay sharoitlari topildi. Sintez qilingan 5-(R-fenil)-2-amino-1,3,4-tiadiazollar va azometin (Shiff asoslari) hosilalari tuzilishi spektral usullar (IQ, ^{1}H YAMR, ^{13}C YAMR) yordamida tasdiqlangan, moddalarning fizik-kimyoviy konstantalari aniqlandi.

Kalit so‘zlar: 2-(R-fenil)gidrazin-1-karbotioamidlar, 5-(R-fenil)-2-amino-1,3,4-tiadiazollar va azometin (Shiff asoslari), sintez, tuzilishi, IQ, ^{1}H YAMR, ^{13}C YAMR spektrlar.

СИНТЕЗ ОСНОВАНИЙ ШИФФА 5-АРИЛ-2-АМИНО-1,3,4-ТИАДИАЗОЛОВ

Аннотация

Найдены оптимальные условия реакций 5-(фенил, 2,4-дихлорфенил)-2-амино-1,3,4-тиадиазолов с ароматическими альдегидами. Строение синтезированных 5-(R-фенил)-2-амино1,3,4-тиадиазолов и азометиновых (Шиффовы основания) производных подтверждены спектральными (ИК, ^{1}H ЯМR, ^{13}C ЯМR) методами, определены физико-химические константы веществ.

Ключевые слова: 2-(R-фенил)гидразин-1-карботиоамиды, 5-(R-фенил)-2-amino-1,3,4-тиадиазолы и азометины (Шиффовы основания), синтез, спектры ИК, ^{1}H ЯМR, ^{13}C ЯМR.

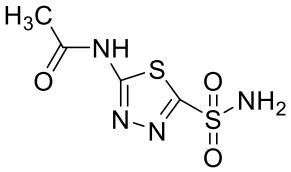
SYNTHESIS OF SCHIFF BASES 5-ARYL-2-AMINO-1,3,4-THIADIAZOLES

Annotation

Optimal conditions for reactions of 5-(phenyl, 2,4-dichlorophenyl)-2-amino-1,3,4-thiadiazoles with aromatic aldehydes were found. The structure of the synthesized 5-(R-phenyl)-2-amino1,3,4-thiadiazoles and azomethine (Schiff bases) derivatives was confirmed by spectral (IR, ^{1}H NMR, ^{13}C NMR) methods, the physicochemical constants of the substances were determined.

Key words: 2-(R-phenyl)hydrazine-1-carbothioamides, 5-(R-phenyl)-2-amino-1,3,4-thiadiazoles and azomethines (Schiff bases), synthesis, IR spectra, ^{1}H NMR, ^{13}C NMR.

Kirish. Tarkibida azot, kislorod, oltingugurt kabi geteroatomlarni tutgan besh azoli geterohalqali birikmalar kimyosi organik kimyoning katta va muhim qismlaridan biri. Tiadiazollar, xususan, 5-almashingan-1,3,4-tiadiazollar va ularning 1,3,4-tiadiazol-2-tion, 1,3,4-tiadiazol-2-aminlar kabi analoglari ushbu geterotsiklik birikmalar sinfining jadal rivojlanayotgan hamda keng o‘rganilayotgan vakillari hisoblanadi. 5-Almashingan-1,3,4-tiadiazollar tadqiqotchilar e’tiborini nafaqat kimyoviy o‘zgarishlarni o‘tkazish uchun keng imkonyatlari mavjudligi bilan [1-3], balki ularning ko‘plab hosilalari orasida turli biologik faolliklarga ega birikmalarning borligi bilan ham jalb qiladi [4-8]. Hozirgi vaqtda 5-almashingan-1,3,4-tiadiazollar asosida yaratilgan ko‘plab preparatlar amaliyotda qo‘llab kelinmoqda, bir qanchalari esa so‘nggi klinik sinovlar o‘tkazilmoqda. Ular orasidan tibbiyot va qishloq xo‘jaligida keng qo‘llanilayotgan quyidagi preparatlarni misol qilib keltirish mumkin: diuretik - atsetazolamid 1, antibakterial - sulfametazol 2, sefazolin 3, antidepressant - atibepron 4 [9], yalpi ta’sirli gerbitsidlar - etidibimuron 5, butiuron 6 [10]:



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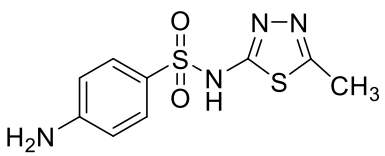

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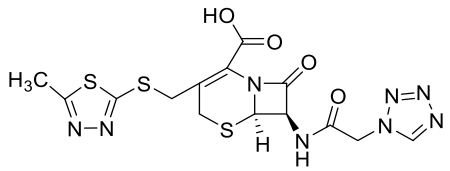

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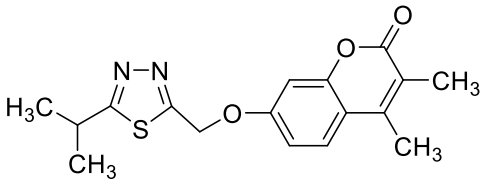

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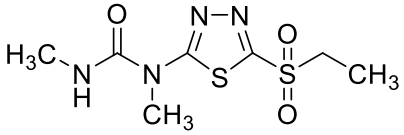

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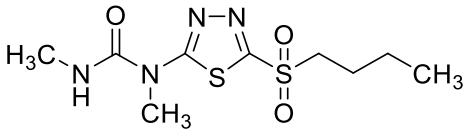
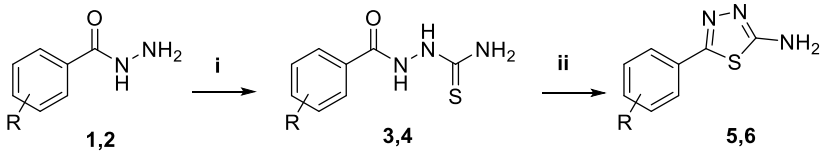


6

Adabiyotlarda 1,3,4-tiadiazollar va ular ishtirokidagi turli reaksiyalar haqidagi ma’lumotlar XIX asrning oxirlaridan boshlab paydo bo‘la boshlagan. Tiadiazol to‘g‘risidagi birinchi ma’lumot 1894 yili Bush tadqiqotlarida uchraydi [11]. U gidrazin sulfatning uglerod disulfid bilan kaliy gidroksidining spirtli eritmasi ishtirokidagi ta’sirini amalga oshirgan. Tiadiazollar asosidagi tadqiqotlar ko‘p yillardan beri olib borilishiga qaramay, ushbu geterotsiklni, hususan, 5-almashingan-2-amino-1,3,4-tiadiazollarni turli hosilalari sintezi va ulani keyingi kimyoviy o‘zgarishlarini o‘rganish xozirgi kunda ham dolzarb masalalardan biri.

Natijalar va ularning tahlili. Yuqoridagi fikrlardan kelib chiqib, ushbu tadqiqot ishimizda biz oldingi yillarda [12-16] boshlagan 5-almashingan-1,3,4-tiadiazollarni o‘rganishni davom etdirib, bu gurux birikmalariga turdosh bo‘lgan 2-amino-5-aril-1,3,4-tiadiazollar va ularning xosilalarini sintezi hamda kimyoviy o‘zgarishlarini amalga oshirishni davom ettirdik. 5-Aril-2-amino-1,3,4-tiadiazollarni sintez qilib olishda adabiyotlarda yoritilgan almashingan benzoy kislotalar va tiosemikarbazidni sulfat kislota muxitida qizdirish bilan olib borilgan tajribalarimiz kutilgan natijani bermadi. Bu xolatdan kelib chiqqan xolda biz tadqiqotlarimiz uchun tanlab olingan tiadiazollarni boshqa usul bilan sintezini amalga oshirdik. Sintezning birinchi bosqichida almashingan benzoy kislotalarining gidrazidlari (benzoy va 2,4-dixlorbenzoy kislotalarining gidrazidlari) va ammoniy rodanidning (NH_4SCN) ekvimolyar miqdorlarini etanolda (HCl ishtirokida) 7-8 soat davomida qaynatish bilan reaksiyalari amalga oshirildi. Natijada tegishli 2-(aril)gidrazin-1-karbotioamidlar mos ravishda **3**,**4** 78 va 85% unumlarda sintez qilib olindi:



1,2 **3,4** **5,6**

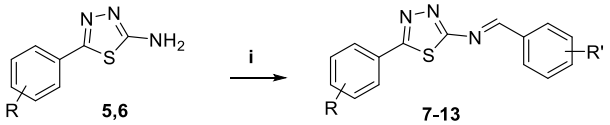
R = H (**1,3,5**), 2-4Cl_2 (**2,4,6**). **i** = NH_4SCN, C_2H_5OH, HCl, 78 °C. **ii** = H_2SO_4, 20-25 °C.

Olingan 2-(aril)gidrazin-1-karbotioamidlar 3,4 och qaymoq rangli kristallar bo‘lib, ularning tuzilishi ^1H va ^{13}C YAMR spektlari natijalari bilan tasdiqlandi. Masalan, 2-(2,4-dixlorbenzoil)gidrazin-1-karbotioamid 4 ^1H YAMR spektrida 7.24, 7.34 va 7.78 m.u.da aromatik xalqa protonlariga tegishli singlet xamda dublet ko‘rinishidagi signallar, 7.43 m.u. da esa, NH_2 guruxi protonlari singlet signali, 9.37 va 10.25 m.u. larda mos ravishda moddaning gidrazin guruxidagi NH protonlari singlet signallari kuzatildi. ^{13}C YAMR spektri ma’lumotlari ham olingan birikmaning tuzilishiga mos keladi: 127.15 m.u. (S-5′), 129.60 (C-6′), 131.86 (C-3′), 132.72 (C-1′), 133.23 (C-2′), 135.93 (C-4′), 165.09 (C-1), 182.39 (C-4).

Keyingi bosqichda 2-(aril)gidrazin-1-karbotioamidlarni **3**,**4** uzoq vaqt (20 soat) xona xaroratida konsentrlangan H_2SO_4 da uzluksiz aralashtirish bilan olib borilgan geteroxalqalash natijasida maqsaddagi 5-(fenil, 2,4-dixlorfenil)-2-amino-1,3,4-tiadiazollar 82 va 85 % unumlarda olindi. Sintez qilingan 5-(2,4-dixlorfenil)-2-amin-1,3,4-tiadiazolning ^1H YAMR spektrida 7.24 m.u. soxasida keng singlet ko‘rinishidagi NH_2 guruxi protonlari signallari xamda aromatik xalqadagi tegishli protonlarning 7.36 (1H, d, J=9.6, Ar-H-5′), 7.49 (1H, s, Ar-H-3′), 8.03 (1H, d, J=8.6, Ar-H-6′) dublet va singlet signallari mavjud. Bu birikmaning ^{13}C YAMR spektridagi uglerod atomlari signallari ma’lumotlari uning tuzilishini to‘liq isbotlaydi: 127.88 m.u. (S-5′), 129.43 (C-6′), 130.07 (C-3′), 131.55 (C-1′), 131.67 (C-2′), 135.02 (C-4′), 150.91 (C-5), 170.78 (C-2).

Shunday qilib, 5-aril-2-amino-1,3,4-tiadiazollarni yaxshi unumlarda sintez qilishning qulay usuli tegishli benzoiltiosemikarbazidlarni xona xaroratida sulfat kislotada geteroxalqalash ekanligi topildi.

Ma’lumki, 5-almashingan-2-amin-1,3,4-tiadiazollarning ko‘plab xosilalari turli biologik faolliklarga ega [17-19]. Ana shunday biologik faolliklarga ega bo‘lgan 5-almashingan-1,3,4-tiadiazollarning ko‘plab xosilalarini olish va ularning turli xsusiyatlarini, shu jumladan, farmakologik faolliklarini o‘rganish xozirgi vaqtda anchagini dolzarb vazifa hisoblanadi. Tadqiqotlarimizni sintez qilib olingan 5-(fenil, 2,4-dixlorfenil)-2-amino-1,3,4-tiadiazollarning 5,6 bir qator xosilalarini olish maqsadida ularning aromatik aldegidlar bilan reaksiyalarini o‘rganish bilan davom ettirdik:



5,6 **7-13**

R = H, R' = 3-OH (**7**), 4-OH (**8**), 4-NO_2 (**9**), 4-$N(CH_3)_2$ (**10**), 3-4$(OCH_3)_2$ (**11**), 4-Br (**12**). R = 2-4 Cl_2, R' = 4-$N(CH_3)_2$ (**13**). **i** = C_2H_5OH, C_6H_6, CH_3COOH, R' - C_6H_5COH.

Reaksiyalarni olib borishni qulay sharoitlarini topish maqsadida tajribalar 5-fenil-2-amino-1,3,4-tiadiazol 5 va 4-dimetilamino-benzaldegidlar misolida o‘tkazildi. Tajribalar reagentlarning teng miqdorlarini etil spirtida va quruq benzolda xona (22-25°C) va erituvchilarning qaynash xaroratlarida (78, 80°C) katalitik miqdordagi muz sirka kislotasi (CH_3COOH), uchftor sirka kislotasi (CF_3COOH) yoki sulfat kislotasi (H_2SO_4) ishtirokida olib borildi. Bunda ko‘zlangan maqsaddagi 10 birikmani faqat benzolda muz sirka kislotasi ishtirokida 8 soat qaynatish natijasida 60% unum bilan ajratib olishga erishildi. Boshqa sharoitlarda

maxsulotlar ajratib olinmadi – ba’zi xollarda olingan xosilalar saqichlanib (osmolenie) qoldi, yoki individual ko‘rinishda ajratib olishni imkoni bo‘lmagan bir nechta moddalar aralashmasi olindi. Sintez qilingan (E)-N,N-dimetil-4-(((5-fenil-1,3,4-tiadiazol-2-il)imino)metil)anilin 10 birikmasini etil spirtida qayta kristallanganda och sariq rangli kristallar ajratib olindi (R_f 0.61 (xlf: spirt 10:1), T_{suyuql} 199-200 °C). Birikmaning IQ- spektrida C=N guruxiga xos bo‘lgan soxada $\nu_{S=N}$ 1614 sm^{-1} yutilish signali borligini ko‘rishimiz mumkin. Moddaning ^{1}H YAMR spektrida dastlabki 5-fenil-2-amino-1,3,4-tiadiazolning 7.24 m.u. soxasida keng singlet ko‘rinishidagi NH_2 guruxi signali yo‘qolib, yangi azometin (N=CH-) guruxining bitta protoni 8.70 m.u. soxasida singlet signali paydo bo‘lishi amino gurux bo‘yicha yangi xosila olinganini ko‘rsatadi. SHu 10 birikmaning -N-$(CH_3)_2$ guruxi protonlari singlet ko‘rinishida 3.11 m.u. soxada namoyon bo‘lishi, ikki aromatik xalqa protonlari ularga xos bo‘lgan soxalarda tegishli signallar berishi - 6.74 m.u. (2H, d., J=9, Ar-H-3″,5″), 7.43-7.49 (3H, m., Ar-H-3′,5′,4′), 7.81 (2H, d., J=8.9, Ar-H-2″,6″), 7.85-7.90 (2H, m., Ar-H-2′,6′) hamda ^{13}C YAMR spektri ma’lumotlari - 40,30 m.u. (S-9,10), 111.82 (S-3″,5″), 122.46 (C-2′,6′), 126.71 (C-1″), 127.54 (C-3′,5′), 129.41 (S-2″,6″), 130.89 (S-4′), 132.79 (C-1′), 154.17 (S-4″), 164.73 (C-5), 166.88 (C-7), 174.77 (S-2) sintez qilib olingan 10 hosilaning tuzilishiga to‘liq isbotlaydi.

Analogik reaksiya sharoitlarida 5-(fenil, 2,4-dixlorfenil)-2-amino-1,3,4-tiadiazollarning boshqa aromatik aldegidlar (3-gidroksi-, 4- gidroksi-, 4-brom-, 4-nitro-, 3,4 dimetoksibenzaldegid) bilan tegishli 7-13 xosilalari (reagentlar nisbati 1:1, benzol, CH_3COOH, 80°S) 55-65% unum bilan sintez qilindi. Bunda eng yuqori unumlar 4-nitrobenzaldegid (67%), 3-gidroksi-, 4-gidroksi-, 4-dimetilamino-benzaldegidlar (65% dan) bilan olindi. 4-Brom benzaldegid va 3,4 dimetoksi-benzaldegid bilan olingan hosilalarning unumi nisbatan kamroq bo‘lib, ular 55% ni tashkil etdi. Olingan barcha hosilalar etanolda qayta kristallangandan keyin och sariq yoki limon rangli kristal moddalar, ularning tuzilishi IQ-, ^{1}H va ^{13}C YAMR spektri ma’lumotlari asosida tasdiqlandi.

Sintez qilingan moddalarning ba’zi fizik-kimyoviy konstantalari jadvalda keltirilgan.

Tajriba qismi. 5-(2,4-Dixlorfenil)gidrazin-1-karbotioamid sintezi. Ekvimolyar miqdordagi almashingan 2,4-dixlorbenzoy kislota gidrazidi va ammoniy rodanidni (NH_4SCN) etanolda HCl ishtirokida 7-8 soat davomida qaynatildi. Reaksiya borishi YUQX bilan kuzatib borildi. Reaksion aralashma xona xaroratigacha sovitilganda tushgan cho‘kma filtrlab olindi, 1-2 marotaba sovuq etanol va suv bilan yuvildi, olingan och qaymoq rangli kristallar ochiq havoda quritildi.

5-(Aril)-2-amino-1,3,4-tiadiazollar sintezi

2-(Aril)gidrazin-1-karbotioamidlar 3,4 (0,05 mol) va 20 ml. H_2SO_4 aralashmasini xona xaroratida magnit aralashtirgich bilan 24 soat aralashtirildi. Reaksion aralashmani maydalangan muz ustiga quyib, ammiak eritmasi bilan ishqoriy muxitgacha keltirildi. Xosil bo‘lgan oq rangli cho‘kma filtirlab olindi, suv bilan bir necha marotaba yuvili. Olingan oq rangli moddalar ochiq xavoda quritildi.

5-(Fenil, 2-4 dixlorfenil)-2-amino-1,3,4-tiadiazollarning aromatik aldegidlar (3-gidroksi-, 4-gidroksi-, 4-brom-, 4-nitro-, 3-4 dimetoksi-benzaldegid) bilan kondesatsiyasi reaksiyalari (umumiy usul)

5-(Fenil, 2-4dixlorfenil)-2-amino-1,3,4-tiadiazol 5,6 (0,001 mol) va tegishli aromatik aldegid (0,001 mol) aralashmasi 0.2 ml muz sirka kislotasi ishtirokida benzolda (8 ml) 8-10 soat davomida qaynatildi. Reaksiya aralashmasi sovitib, hosil bo‘lgan cho‘kma filtrlab olindi va bir necha marotaba suv bilan yuvili. Olingan sarg‘ish rangli kukunsimon moddalar ochiq havoda quritildi va etanolda qayta kristallandi.

Sintez qilingan moddalarning fizik-kimyoviy konstantalari

T/p	Модда номи	Ранги	$T_{суюк}$, °C	R_f	Унум, %
1	Бензогидразид	оқ	112-114	0.46	78
2	2-4-Дихлорбензогидразид	оқ	163-166	0.48	70
3	2-(фенил)гидразин-1-карботиоамид	оқ	153-157	0.47	78
4	2-(Дихлорфенил)гидразин-1-карботиоамид	оқ	89-91	0,61	85
5	5-фенил-2-амино-1,3,4-тиадиазол	оқ	233-234	0,63	78
6	5-(Дихлорфенил)-2-амино-1,3,4-тиадиазол	оқ	102-103	0.48	86
7	(E)-3-((5-фенил-1,3,4-тиадиазол-2-ил)имино)метил)фенол	оч сариқ	218-220	0,46	65
8	(E)-4-((5-фенил-1,3,4-тиадиазол-2-ил)имино)метил)фенол	оч сариқ	259-260	0,48	65
9	(E)-1-(4-нитрофенил)-N-(5-фенил-1,3,4-тиадиазол-2-ил)метанимин	оч сариқ	238-239	0,64	67
10	(E)-N,N-диметил-4-(((5-фенил-1,3,4-тиадиазол-2-ил)имино)метил)анилин	оч сариқ	199-200	0,61	65
11	(E)-1-(3,4-диметоксифенил)-N-(5-фенил-1,3,4-тиадиазол-2-ил) метанимин	оч сариқ	159-160	0,51	55
12	(E)-1-(4-бромофенил)-N-(5-фенил-1,3,4-тиадиазол-2-ил)метанимин	оч сариқ	200-201	0,62	55
13	(E)-4-(((5-(2,4-дихлорофенил)-1,3,4-тиадиазол-2-ил) имино) метил)-N, N-диметиланилин	тўқ сариқ	219-220	0,66	72

Xromatografik sistema: xloroform-etanol, 24-1

Xulosa. Shunday qilib, 2-(R-fenil)gidrazin-1-karbotioamidlar asosida olingan 5-(fenil, 2,4-dixlorfenil)-2-amino-1,3,4-tiadiazollarning bir qator aromatik aldegidlar bilan reaksiyalari qulay sharoitlari topildi. Sintez qilib olingan 5-(R-fenil)- 2-amino-1,3,4-tiadiazollar va azometin (SHiff asoslari) hosilalari tuzilishi spektral usullar (IQ, ^{1}H YAMR, ^{13}C YAMR) yordamida to‘liq tavsiflandi, moddalarning fizik-kimyoviy konstantalari aniqlandi.

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