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STEPWISE SYNTHESIS OF ENAMINONE DERIVATIVES

Annotation

In this study, new enaminone derivatives belonging to the class of 5-N-((3,4-dimethoxyphenethyl)amino)-2,4-diethyl ester-1-methyl-3-phenylcyclohex-4-en-1-ol were synthesized via a three-component condensation reaction involving 3,4-dimethoxyphenethylamine (homoveratrylamine), aromatic aldehydes, and ethyl acetoacetate. The reactions were carried out in a stepwise manner through intermediate imines. The structures of the synthesized compounds were confirmed by IR, ^{1}H NMR, and ^{13}C NMR spectroscopic analysis.

Keywords: enaminones, homoveratrylamine, three-component condensation, imines, ethyl acetoacetate, aromatic aldehydes, organic synthesis.

ПОСТАДИЙНЫЙ СИНТЕЗ ЕНАМИНОНОВ

Аннотация

В данной работе методом трехкомпонентной конденсации между 3,4-диметоксифенилэтиламином (гомовератриламином), ароматическими альдегидами и ацетоуксусным эфиром синтезированы новые производные енаминонов, относящиеся к ряду 5-N-((3,4-диметоксифенилэтил)амино)-2,4-диэтилэфир-1-метил-3-фенилциклогексен-4-ол-1. Реакции проведены по стадийному методу через промежуточные имины. Структуры синтезированных соединений подтверждены методами ИК, ^{1}H и ^{13}C ЯМР спектроскопии.

Ключевые слова: енаминоны, гомовератриламин, трехкомпонентная конденсация, имины, ацетоуксусный эфир, ароматические альдегиды, органический синтез.

YENAMINON HOSILALARINING BOSQICHLI SINTEZI

Annotatsiya

Ushbu ishda 3,4-dimetoksifeniletilamin (gomoveratrilamin), aromatik aldegidlar va asetosirka efiri ishtirokida uch komponentli kondensatsiya reaksiyasi orqali yangi 5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-fenil-siklogeksen-4-ol-1 turkumiga mansub yenaminon hosilalari sintezi amalga oshirildi. Reaksiyalar bosqichli (oraliq iminlar orqali) usullarda olib borildi. Sintezlangan mahsulotlarning tuzilmalari IQ, ^{1}H, ^{13}C YaMR spektroskopik tahlillar orqali tasdiqlandi.

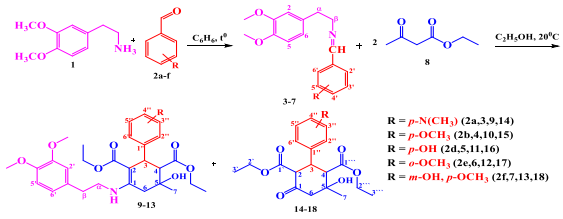
Kalit so‘zlar: yenaminonlar, gomoveratrilamin, uch komponentli kondensatsiya, iminlar, asetosirka efiri, aromatik aldegidlar, organik sintez.

Kirish. Organik sintezning zamonaviy yo‘nalishlaridan biri sifatida biologik faol moddalarning yangi sinflarini olishga qaratilgan izlanishlar alohida ahamiyat kasb etmoqda. Shu jihatdan, yenaminon va uning hosilalari diqqatga sazovor bo‘lib, ular tuzilmasida bir vaqtning o‘zida enamin va karbonil guruhlarini saqlagani sababli turli reaksion markazlarga ega, kimyoviy modifikatsiyalarga oson uchraydi [1,2]. Bunday moddalarning reaktivligi yuqori bo‘lishi, ularni turli farmakologik jihatdan ahamiyatli birikmalarga aylantirish imkonini beradi [3].

Yenaminonlar antibiotiklar, virusga qarshi preparatlar, yallig‘lanishga qarshi vositalar hamda antitumor faol moddalarning sintezida oraliq mahsulotlar sifatida keng qo‘llaniladi [4, 5]. Oxirgi yillarda gomoveratrilamin, β-diketonlar (masalan, asetilaseton, asetosirka efiri) va aromatik aldegidlar ishtirokida uch komponentli kondensatsiya reaksiyalari orqali yenaminon hosilalari sintez qilish bo‘yicha bir qator samarali usullar ishlab chiqildi. Bu yo‘nalishda olib borilgan oldingi tadqiqotlarimizda gomoveratrilaminning aromatik aldegidlar bilan reaksiyalari, karbosikllanish jarayonlari, shuningdek, sintetik mahsulotlarning sitotoksik faolligi o‘rganilgan [6-15].

Natijalar muhokamasi. Ushbu reaksiyaning muvaffaqiyatli borishi ikkala reagentning ham tabiatiga birdek bog’liq bo‘ladi. Aminning nukleofilligi qanchalik yuqori bo‘lsa, kondensatsiya tezligi shunchalik yuqori bo‘ladi (bizning misolimizda

gomoveratrilamin qo‘llanildi). Neytral muhitda iminlar (**3-7**) hosil bo‘lish tezligini belgilovchi bosqich karbinolaminning suvsizlanishi, kislotali muhitda esa - uning hosil bo‘lishidir. Muvozanatni o‘ngga siljitish va imin hosil bo‘lishi uchun suvni azeotropik distillash lozim bo‘ladi. Buning uchun biz Dina-Starka nasadkasidan foydalandik. Shiff asoslari benzolda barcha aldegidlar uchun deyarli oson olinadi. 1-1,5 soat qaynatilganda hosil bo‘lgan (**3-7**) birikmalar ajratilmagan holda, imin reaksiya muhitiga (benzol haydalganidan so‘ng) uchinchi komponent sifatida aktiv metilen birikmasi sifatida 2 mol asetosirka efiri qo‘shildi. Ushbu jarayonda imin ta’sirida asetosirka efiri C-H protonlar chiqariladi va C=C bog‘lar hosil bo‘ladi. Hosil bo‘lgan yangi hosilalar qayta tuzilib, yenaminon birikmasi hosil bo‘ladi.



1-Sxema. 5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-fenil-siklogeksen-4-ol-1 yenaminon hosilalari sintezi

Mahsulot unumi benzol halqasidagi o‘rinbosarlarning turiga bog'liq bo‘ldi. Xuddi bir bosqichli usulda kuzatilgani kabi, birinchi turdagi o‘rinbosarlar yenaminonlarning unumini oshirdi va nitroguruhning mavjudligi esa siklogeksan(en) tipidagi yangi mahsulotlarning shakllanishiga yordam berdi. (**9-13**) birikmalar yuqori unum bilan “one-pot” reaksiyalarda olingan [13-15]. Ikki bosqichli usulning yagona afzalligi - reaksiya vaqtini qisqaligi bo‘ldi.

1-Jadval. 9-13 birikmalarning hosil bo’lish vaqti va unumi

Birikma	Vaqt, soat	Reaksiya unumi, %
9	48	61
10	48	70
11	48	54
12	48	51
13	48	72

Olingan mahsulotlar – 5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-fenil-siklogeksen-4-ol-1 yenaminon hosilalarining (4-13) tuzilishi zamonaviy fizik-tadqiqot usullari: IQ-, ^{1}H YaMR va ^{13}C YaMR –spektroskopiyasi usullari yordamida yenaminon strukturasi to‘liq isbotlandi.

^{1}H YaMR spektrlarda 6-holatdagi metilen (CH_2) protonlari signalari 2.04–2.74 m.u., etoksi guruhlarining metilen va metil protonlari esa mos ravishda 3.67–4.29 m.u. va 0.67–1.36 m.u. oralig‘ida triplet va multipletlar ko‘rinishida aniqlangan. NH guruhining keng singlet signali va 3,4-holatdagi metoksi guruhlarining 3H, s (3.75–3.89 m.u.) signallari molekuladagi amin fragmentini tasdiqlaydi.

Shuningdek, aromatik protonlarning signallari 6.72–7.58 m.u. oralig‘ida multipletlar sifatida kuzatildi. Siklogeksen yadrosidagi xiral markaz protonlari signallarining aniqlanishi esa ichki halqalanish reaksiyalarining muvaffaqiyatli amalga oshganligini ko‘rsatdi.

Tajribaviy qismi. Mahsulotlarning chiqish unumi va tozaligi Shimadzu LC-20 HPLC (Yaponiya) va C18 kolonka (Shimadzu LC-20, Yaponiya) asboblarida aniqlangan. IR spektrlari FT-IR/NIT Spectrum 3 spektrometri yordamida ATR tizimida yozib olingan. ^{1}H va ^{13}C YaMR spektrlari JNM-ECZ400R va JNM-ECZ600R (Jeol, Yaponiya) spektrometrlarda, mos ravishda 400 va 600 MHz chastotalarda, $CDCl_3$ eritmasida yozilgan. ^{1}H YaMR spektrlari uchun tetrametilsilan (TMS, 0 ppm) ichki standart sifatida ishlatilgan. ^{13}C YaMR spektrlari uchun erituvchi $CDCl_3$ (77.16 ppm, TMSga nisbatan) ichki standart sifatida qabul qilingan. HR-ESI-MS spektrlari Agilent Technologies 6420 (uchli LS/MS tizimida, elektropurkovka ionlanish – ESI TIC Scan va CAMAG TLC-MS tizimi, ACQUITY QDa detektori bilan) asbobida yozib olingan. IR spektrlari, shuningdek, Perkin-Elmer kompaniyasining FTIR System 2000 asbobida KBr tabletkalari yordamida qayd etilgan.

5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-fenil-siklogeksen-4-ol-1 yenaminon hosilalari sintezining umumiy usuli: Aromatik aldegid (2 mmol) va gomoveratrilamin (2 mmol) 25 ml suvsiz benzolda aralashtirildi. Aralashma qaynatiladigan sharoitda Din-Stark nasadkasi yordamida 1.5 soat davomida azeotrop haydash usuli orqali kondensatsiya reaksiyasi olib borildi. Reaksiya davomida suv ajralib chiqishi vizual nazorat qilindi. Qaynatish tugaganidan so‘ng, eritma sovitilib, benzol to‘liq haydaldi. Hosil bo‘lgan oraliq mahsulotlar – Shiff asoslari (iminlar) – ajratilmasdan keyingi bosqichga o‘tkazildi. Yuqorida olingan reaksiya muhitiga 2 mmol asetosirka efiri qo‘shildi, so‘ngra 10 ml suvli etanol eritmasi solindi. Aralashma xona haroratida magnitli aralashtirgich yordamida 12–24 soat davomida aralashtirildi. Reaksiya tugagach, reaksiya massasi muzlatildi va cho‘kma ajratib olindi. Hosil bo‘lgan birikmalar etanol bilan kristallantirildi va quritildi.

5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-p-dimetilaminofenil-siklogeksen-4-ol-1, $C_{31}H_{42}N_2O_7$ sintezi (9). Yuqorida keltirilgan usul bilan 0.4 g (0.39 ml, 1.0254 g/ml, 0.0025 mol) *p*-dimetilaminobenzaldegid, 0.68 g (0.66 ml, ρ=1.0284 g/ml, 0.005 mol) asetosirka efiri, 0.47 g (0.437 ml, ρ=1.074 g/ml, 0.0025 mol) gomoveratrilamin va 10 ml etanol ishtirokida 10 sutka davomida olib borilgan reaksiyadan 1.13 g (82.5%) mahsulot **(9)** olindi. T_{suy}= yog’simon (xloroform), R_f 0.73 (benzol:methanol, 8:1).

^{1}H NMR spektri (600 MHz, $CDCl_3$, δ, m.u., J/Hz): 1.01 (3H, t, J=7.0, Et-CH3), 1.23 (3H, t, J=7.1, Et-CH3), 1.77 (3H, s, CH3-1'), 2.03 (1H, d, J=8.6, H-6'a); 2.24 (1H, d, J=7.3, H-6'e)), 2.81 (2H, m, H-α), 3.16 (1H, d, J=1.4, H-2'), 3.51 (2H, m, H-β), 3.84 (3H, s, OCH3), 3.85 (3H, s, OCH3), 3.87 (3H, s, OCH3), 3.89 (1H, d, J=3.7, H-3'), 4.12 (2H, m, Et-CH2), 4.18 (2H, m, Et-CH2), 6.15 (1H, keng.s., OH), 6.74-6.78 (3H, m, ArH-2, 5, 6), 6.82 (1H, dd, J=1.2, 8.1, ArH-3"), 6.91 (1H, dd, J=2.4, 7.6, ArH-6"), 7.12 (1H, dt, J=1.8, 8.0, ArH-4"), 7.22 (1H, dt, J=2.4, 8.1, ArH- 5"), 9.01 (1H, keng.s., NH).

5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-p-metoksifenil-siklogeksen-4-ol-1, $C_{30}H_{39}NO_8$ sintezi (10). Yuqorida keltirilgan usul bilan 0.385 g (0.344ml, 1,119 g/ml, 0.0028 mol) *p*-metoksibenzaldegid, 0.737 g (0.716 ml, ρ=1.0284 g/ml, 0.005 mol) asetosirka efiri, 0.513g (0.477 ml, ρ=1.074 g/ml, 0.0028 mol) gomoveratrilamin va 10 ml etanol

ishtirokida 10 sutka davomida olib borilgan reaksiyadan 1.27 g (85%) mahsulot **(10)** olindi. T_{suy}= yog’simon (xloroform), R_f 0.6 (benzol:methanol, 6:1).

^{1}H NMR spektri (600 MHz, $CDCl_3$, δ, m.u., J/Hz): 1.08 (3H, t, J=7.1, Et-CH3), 1.23 (3H, t, J=7.1, Et-CH3), 1.83 (3H, s, CH3-1'), 2.04 (1H, d, J=10.1, H-6'a); 2.31 (1H, d, J=10.5, H-6'e)), 2.81 (2H, m, H-α), 3.07 (1H, d, J=1.4, H-2'), 3.49 (2H, kv, J=7.1, H-β), 3.75 (3H, s, OCH3), 3.76 (3H, s, OCH3), 3.84 (3H, s, OCH3), 3.86 (1H, d, J=2.3, H-3'), 3.98 (2H, kv, J=7.1, Et-CH2), 4.15 (2H, kv, J=7.1, Et-CH2), 6.18 (1H, d, OH), 6.69 (1H, d, J=8.8, H-5), 6.73 (2H, dd, J=2.2, 8.9, H-2, 6), 6.99 (1H, d, J=8.8, H-2"), 7.09 (2H, dt, J=2.3, 8.8, ArH-3", 5"), 7.18 (1H, d, J=8.8, ArH- 6"), 8.96 (1H, keng.s., NH).

5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-p-gidroksifenil-siklogeksen-4-ol-1, $C_{29}H_{37}NO_8$ sintezi (11). Yuqorida keltirilgan usul bilan 0.27 g (0.22 ml, 1.226 g/ml, 0.002 mol) *p*-gidroksibenzaldegid, 0.575 g (0.56 ml, ρ=1.0284 g/ml, 0.004 mol) asetosirka efiri, 0.4 g (0.37 ml, ρ=1.074 g/ml, 0.002 mol) gomoveratrilamin va 10 ml etanol ishtirokida 10 sutka davomida olib borilgan reaksiyadan 0.83 g (79%) mahsulot **(11)** olindi. T_{suy}=168-171°C (xloroform), R_f 0.81(benzol:methanol, 4:1).

^{1}H NMR spektri (600 MHz, $CDCl_3$, δ, m.f., J/Hz): 1.17 (3H, t, J=7.1, Et-CH_3), 1.36 (3H, t, J=7.1, Et-CH_3), 2.30 (3H, s, CH_3-1'), 2.75 (1H, m, H-6'a), 2.78 (2H, t, J=7.1, H-α), 2.92 (1H, d, J=7.1, H-6'e), 3.48 (1H, d, J=7.0, H-2'), 3.52 (2H, kv, J=7.1, H-β), 3.75 (1H, s, OH), 3.85 (3H, s, OCH_3), 3.86 (3H, s, OCH_3), 3.87 (1H, d, J=2.2, H-3'), 4.25 (2H, kv, J=7.1, Et-CH_2), 4.29 (2H, kv, J=7.1, Et-CH_2), 4.47 (1H, s, OH), 6.67 (1H, d, J=2.0, ArH-2), 6.69 (2H, dd, J=2.1, 7.3, ArH-5, 6), 6.71-6.74 (4H, m, ArH-2", 3", 5", 6"), 9.91 (1H, t, J=5.8, NH).

5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-o-metoksifenil-siklogeksen-4-ol-1, $C_{30}H_{39}NO_8$ sintezi (12). Yuqorida keltirilgan usul bilan 0.25 g (0,22 ml, 1,127 g/ml, 0,0018 mol) *o*-metoksibenzaldegid, 0,477 g (0,46 ml, ρ=1.0284 g/ml, 0,0036 mol) asetosirka efiri, 0,332 g (0,3 ml, ρ=1.074 g/ml, 0,0018 mol) gomoveratrilamin va 10 ml etanol ishtirokida 10 sutka davomida olib borilgan reaksiyadan 0.77 g (80.5%) mahsulot **(12)** olindi. T_{suy}=155-157°C (xloroform), R_f 0.8 (benzol:methanol, 6:1).

^{1}H YaMR spektri: (400 MHz, $CDCl_3$, δ, m.u., J/Hz) (12): 1.01 (3H, t, J=7.0, Et-CH_3), 1.23 (3H, t, J=7.1, Et-CH_3), 1.77 (3H, s, CH_3-1'), 2.03 (1H, d, J=8.6, H-6'a); 2.24 (1H, d, J=7.3, H-6'e)), 2.81 (2H, m, H-α), 3.16 (1H, d, J=1.4, H-2'), 3.51 (2H, m, H-β), 3.84 (3H, s, OCH_3), 3.85 (3H, s, OCH_3), 3.87 (3H, s, OCH_3), 3.89 (1H, d, J=3.7, H-3'), 4.12 (2H, m, Et-CH_2), 4.18 (2H, m, Et-CH_2), 6.15 (1H, keng.s., OH), 6.74-6.78 (3H, m, ArH-2, 5, 6), 6.82 (1H, dd, J=1.2, 8.1, ArH-3"), 6.91 (1H, dd, J=2.4, 7.6, ArH-6"), 7.12 (1H, dt, J=1.8, 8.0, ArH-4"), 7.22 (1H, dt, J=2.4, 8.1, ArH- 5"), 9.01 (1H, keng.s., NH).

5-N-((3,4-dimetoksifeniletil)amino)-2,4-dietilefir-1-metil-3-*m*-gidroksi-*p*-metoksifenil-siklogeksen-4-ol-1, $C_{30}H_{39}NO_9$ sintezi (13). Yuqorida keltirilgan usul bilan 0.19 g (0,18 ml, 1,056 g/ml, 0.0012 mol) izovanilin aldegidi, 0.33 g (0.32 ml, ρ=1.0284 g/ml, 0.0024 mol) asetosirka efiri, 0.23 g (0.21 ml, ρ=1.074 g/ml, 0.0012 mol) gomoveratrilamin va 10 ml etanol ishtirokida 10 sutka davomida olib borilgan reaksiyadan 0.51 g (77%) mahsulot **(13)** olindi. T_{suy}=yog’simon (xloroform), R_f = 0.76 (benzol:metanol – 6:1).

IQ (KBr, ν_{max}, cm^{-1}): 2953, 2836 (Ar), 1733, 1710 (C=O), 1642, 1591 (C=C), 1514 (C-C), 1441 (-CH_2-), 1365 (C-N), 1262, 1235 (C-O-C).

^{13}C YaMR spektri: (400 MHz, $CDCl_3$, δ, m.u.) (13): 14.27 (Et-CH_3), 14.59 (Et-CH_3), 18.51 (CH_3-1'), 33.50 (C-β), 35.26 (C-6'), 37.12 (C-2'), 41.99 (C-3'), 42.45 (C-α), 55.88 (OCH_3), 55.93 (OCH_3), 56.12 (OCH_3), 58.51 (Et-CH_2), 58.88 (Et-CH_2),111.34 (C-1'), 120.48 (C-4'), 120.64 (C-2), 120.86 (C-5), 129.71 (C-5"), 130.35 (C-2"), 131.63 (C-6), 148.02 (C-6"), 149.22 (C-1), 158.09 (C-1"), 161.18 (C-3"), 163.39 (C-4"), 164.13 (C-4), 164.51 (C-3), 164.93 (C-5'), 171.67 (C=O), 191.11 (C=O).

Xulosa. Ushbu ishda gomoveratrilamin, aromatik aldegidlar va asetosirka efiri ishtirokidagi uch komponentli kondensatsiya reaksiyasi orqali yangi yenaminon hosilalari sintezi amalga oshirildi. Reaksiya mexanizmi bosqichma-bosqich tahlil qilinib, iminlar hosil bo‘lishining tezligi va barqarorligiga amin va aldegidlarning xossalari muhim rol o‘ynashi aniqlangan. Azeotropik distillash orqali suvni olib tashlash reaktsiyaning muvaffaqiyatli borishini ta’minladi. Olingan mahsulotlarning tuzilmalari IQ, ^{1}H, ^{13}C YaMR usullari yordamida aniqlanib, ularning yuqori tozaligi va barqarorligi tasdiqlandi.

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