Three-component reaction between alkyl(aryl) isocyanides and dialkyl acetylenedicarboxylates in the presence of ethyl trifluoroacetate Khatereh Khandan–Barani, Malek Taher Maghsoodlou*, Sayyed Mostafa Habibi-Khorasani, Nourallah Hazeri and Seyyed Sajad Sajadikhah

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The 1:1 intermediate generated by the addition of alkyl(aryl) isocyanides to dialkyl acetylenedicarboxylates is trapped by ethyl trifluoroacetate in the absence of a catalyst to form highly functionalized dialkyl 5-[alkyl(aryl)imino]-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran–3,4-dicarboxylates in good yields.

Keywords: ethyl trifluoroacetate, furan derivatives, isocyanide, multicomponent reaction

The development of new syntheses of useful organic compounds from readily available reagents is one of the major aims of organic synthesis.¹ At the same time, the increasing environmental awareness of the chemical community has led to the search for more efficient methods of chemical synthesis.^{2,3} Multicomponent reactions (MCRs), by virtue of their convergence, productivity, facile execution and generally high yields of products, have attracted attention in synthetic chemistry.^{4,5}

The development of new MCRs is an interesting research topic in chemistry.^{6–8} In particular, isocyanide-based multicomponent reactions have been applied to the synthesis of various furan and furan derivatives.^{9–13} Polyfunctionalised furan derivatives are versatile synthetic starting materials for the preparation of a variety of heterocyclic and acyclic compounds,^{14–19} such as iminolactones which can be easily hydrolysed to spirolactones, a structural motif, present in a number of biologically active natural products.^{20,21} There are a number of reports of iminolactones synthesis.^{22–24}

In continuation of our previous works on IMCRs,^{25–33} we now report the result of our investigation of the reaction between alkyl(aryl) isocyanides **1** and dialkyl acetylenedicarboxylates **2** in the presence of ethyl trifluoroacetate **3** in refluxing CH_2Cl_2 .

Results and discussion

The one-pot three-component condensation reaction was carried out at 38 °C and completed after 24 h to afford dialkyl 5-[alkyl(aryl)imino]-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran–3,4-dicarboxylate **4** in good yields in the absence of a catalyst (Scheme 1).

The structure of compounds **4a–j** was deduced from their IR, ¹H NMR, ¹³C NMR, ¹⁹F NMR, mass spectral data and elemental analysis. The mass spectra of these compounds **4a–j** displayed M⁺ or M⁺+1 peaks at the appropriate m/z values. The ¹H NMR spectrum of compound **4a** exhibited a triplet for the methyl group at $\delta = 1.29$ ppm (³J = 7.0 Hz), a multiplet for the cyclohexyl ring $\delta = 1.21-1.75$ ppm, a multiplet for the N–CH cyclohexyl proton $\delta = 3.55$ ppm, a quartet for the O–CH₂ group $\delta = 3.62$ ppm, (³J = 7.0 Hz) and two singlets for the methoxy groups $\delta = 3.86$ and 3.91 ppm.

The ¹³C NMR spectrum of each compound displayed resonances in agreement with proposed structure. The ¹⁹F NMR spectrum of **4a** exhibited a single sharp line at $\delta = 80.52$ ppm. Partial assignments of these resonances are given in the experimental data. The IR spectra of **4a** showed strong absorptions at 1744 and 1697 cm⁻¹ due to the ester carbonyls. The ¹H NMR, ¹³C NMR and ¹⁹F NMR spectra of **4b–j** are similar to **4a**. These data are given in the experimental section.



Scheme 1 Synthesis of compounds 4a-j.

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Scheme 2 proposed mechanism for the formation of compound 4.

On the basis of the well established chemistry of isocyanides,^{34,35} it is reasonable to assume that compounds **4** result from initial addition of alkyl(aryl) isocyanides **1** to the dialkyl acetylenedicarboxylates **2** and concomitant addition to ethyl trifluoroacetate **3** leading to iminolactones **4** (Scheme 2).

In conclusion, we have described a convenient and efficient synthesis of functionalised dialkyl 5-[alkyl(aryl)imino]-2ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran–3,4-dicarboxylates from alkyl(aryl) isocyanides and dialkyl acetylenedicarboxylates in the presence of ethyl trifluoroacetate. The present procedure has the advantage that not only the reaction is performed under neutral conditions but also the reactants can be mixed without any activation or modification.

Experimental

Tert-butyl, benzyl, 2,6-dimethylphenyl and cyclohexyl isocyanides were purchased from Fluka. Dialkyl acetylenedicarboxylates and ethyl trifluoroacetate were obtained from Merck and Fluka. All of the compounds were used without further purification. Melting points and IR spectra were measured on an Electrothermal 9100 apparatus and a Shimadzu IR–470 spectrometer respectively. The ¹H, ¹³C and ¹⁹F NMR spectra were recorded on a Bruker DRX–300 Avance instrument with CDCl₃ as solvent at 300.1, 75.5 and 282.4 MHz, respectively. The mass spectra were recorded on a Shimadzu GC/MS QP 1100 EX mass spectrometer operating at an ionization potential of 70 eV. Elemental analyses for C, H and N using a Heraeus CHN–O-Rapid analyzer were carried out at the research laboratory of Tarbiat Moallem University of Tehran.

General procedure

The process for the preparation of dimethyl 5-(cyclohexylimino)-2ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran–3,4-dicarboxylates **4a** is described as an example. The solution of cyclohexyl isocyanide (0.11 g or 1 mmol) in CH₂Cl₂ 3 mL solvent was slowly added dropwise to a mixture of ethyl trifluoroacetate (0.14 g or 1 mmol) and dimethyl acetylenedicarboxylate (0.14 g or 1 mmol) in CH₂Cl₂ 20 mL solvent over 3 min at room temperature. After the addition, the solution was refluxed at 38 °C for 24 h. Then the solvent was removed under reduced pressure, and the residue was washed with mixture of cold diethyl ether and N–hexane with 1: 3 ratio (2×3 mL) to afford the pure product.

Dimethyl 5-(cyclohexylimino)-2-ethoxy-2-(trifluoromethyl)-2,5dihydrofuran–3,4-dicarboxylate (**4a**): Dark orange powder (0.350 g, 89%); m.p. 61–63 °C; IR (KBr) (ν_{max} , cm⁻¹): 2933, 2858, 1744 and 1697 (2C=O), 1647 (C=N); ¹H NMR (300.1 MHz, CDCl₃): $\delta_{\rm H}$ 1.29 (3H, t, ³*J* = 7.0 Hz, CH₃), 1.21–1.75 (10H, m, 5CH₂), 3.62 (2H, q, ³*J* = 7.0 Hz, OCH₂), 3.66 (1H, m, N–CH), 3.86 and 3.91 (6H, 2s, 20CH₃); ¹³C NMR (75.5 MHz, CDCl₃): $\delta_{\rm C}$ 14.7 (CH₃), 24.4, 24.5, 25.8, 33.0 and 33.2 (5CH₂ of cyclohexyl), 53.1 and 53.2 (2OMe), 57.2 (N–CH), 60.9 (OCH₂), 105.7 (q, ²*J* = 35.2 Hz, CCF₃), 120.5 (q, ¹*J* = 286.1 Hz, CF₃), 136.5 and 141.1 (C_{olefin}), 149.6 (C_{imine}), 159.9 and 160.6 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): $\delta_{\rm F}$ 80.52; MS (*m*/z, %): 394 (M⁺+1, 7), 393 (M⁺, 9), 361 (89), 347 (45), 334 (49), 252 (36), 236 (100), 97 (38), 55 (95), 41 (80). Anal. Calcd for C₁₇H₂₂F₃NO₆ (393.35): C, 51.91; H, 5.64; N, 3.56. Found: C, 52.35; H, 5.75; N, 3.88%.

Diethyl 5-(cyclohexylimino)-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran–3,4-dicarboxylate (**4b**): Orange oil (0.396 g, 94%); IR (KBr) (v_{max} , cm⁻¹): 2985, 2935, 1741 and 1698 (2C=O), 1659 (C=N); ¹H NMR (300.1 MHz, CDCl₃): $\delta_{\rm H}$ 1.26 (3H, t, ³*J* = 7.0 Hz, CH₃), 1.17– 1.74 (10H, m, 5CH₂), 1.30 and 1.36 (6H, 2t, ³*J* = 7.1 Hz, 2CH₃), 3.61) 2H, q, ³*J* = 7.1 Hz, OCH₂(, 3.64 (1H, m, N–CH), 4.30 and 4.38 (4H, 2q, ³*J* = 7.1 Hz, 2OCH₂); ¹³C NMR (75.5 MHz, CDCl₃): $\delta_{\rm C}$ 13.8 and 13.9 (2CH₃), 14.7 (CH₃), 24.4, 25.0, 25.6, 33.0 and 33.2 (5CH₂ of cyclohexyl), 57.0 (N–CH), 60.8 (OCH₂), 62.3 and 62.5 (2 OCH₂), 105.7 (q, ²*J* = 35.2 Hz, CCF₃), 120.6 (q, ¹*J* = 286.1 Hz, CF₃), 136.4 and 140.9 (C_{olefin}), 149.8 (C_{imine}), 159.5 and 160.2 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): $\delta_{\rm F}$ 80.21; MS (*mz*, %): 422 (M⁺+1, 96), 380 (100), 352 (6), 334 (435), 267 (12), 97 (18), 83 (17), 41 (35). Anal. Calcd for C₁₉H₂₆F₃NO₆ (421.41): C, 54.15; H, 6.22; N, 3.32. Found: C, 54.15; H, 6.45; N, 3.54%.

Di-tert-butyl 5-(cyclohexylimino)-2-ethoxy-2-(trifluoromethyl)-2,5dihydrofuran–3,4-dicarboxylate (4c): Orange oil (0.405 g, 85%); IR (KBr) (v_{max} , cm⁻¹): 2981, 2933, 1733 and 1699 (2C=O), 1657 (C=N); ¹H NMR (300.1 MHz, CDCl₃): $\delta_{\rm H}$ 1.29 (3H, t, ${}^{3}J$ = 7.0 Hz, CH₃), 1.31–1.75 (10H, m, 5CH₂), 1.52 and 1.55 (18H, 2s, 2C(CH₃)₃), 3.59 (2H, q, ${}^{3}J$ = 7.0 Hz, OCH₂), 3.69 (1H, m, N–CH); ¹³C NMR (75.5 MHz, CDCl₃): $\delta_{\rm C}$ 14.7 (CH₃), 24.1, 24.2, 25.7, 33.1 and 33.3 (5CH₂ of cyclohexyl), 28.0 and 28.1 (2C(CH₃)₃), 56.4 (N–CH), 60.5 (OCH₂), 84.1 and 84.2 (2O-C(Me)₃), 105.7 (q, ${}^{2}J$ = 34.6 Hz, CCF₃), 120.7 (q, ${}^{1}J$ = 285.6 Hz, CF₃), 136.8 and 140.3(C_{olefin}), 149.8 (C_{imine}), 158.9 and 159.4 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): $\delta_{\rm F}$ 80.11; MS (*m*/z, %): 478 (M⁺+1, 83), 461 (2), 422 (47), 347 (50), 319 (30), 301 (11), 275 (4), 98 (21), 57 (100), 41 (39). Anal. Calcd for C₂₃H₃₄F₃NO₆ (477.51): C, 57.85; H, 7.18; N, 2.93. Found: C, 58.33; H, 7.74; N, 3.38%.

Dimethyl 5-(tert-butylimino)-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran–3,4-dicarboxylate (**4d**): Orange oil (0.330 g, 90%); IR (KBr) (v_{max} , cm⁻¹): 2973, 2907, 1745 and 1693 (2C=O), 1659 (C=N); ¹H NMR (300.1 MHz, CDCl₃): $\delta_{\rm H}$ 1.26 (3H, t, ³*J* = 7.1 Hz, CH₃), 1.29 (9H, s, C(CH₃)₃), 3.56 (2H, q, ³*J* = 7.1 Hz, OCH₂), 3.83 and 3.88 (6H, 2s, 2OCH₃); ¹³C NMR (75.5 MHz, CDCl₃): $\delta_{\rm C}$ 14.7 (CH₃), 29.6 (C(CH₃)₃), 53.0 and 53.1 (2OMe), 55.8 (N–C(Me)₃), 60.9 (OCH₂), 106.0 (q, ²*J* = 35.1 Hz, CCF₃), 120.5 (q, ^{*J*} *J* = 286.1 Hz, CF₃), 135.0 and 142.9 (C_{olefin}), 147.7 (C_{imine}), 159.8 and 160.9 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): $\delta_{\rm F}$ 80.61; MS (*m*/z, %): 368 (M⁺+1, 94), 352 (100), 306 (34), 274 (23), 84 (14), 57 (56). Anal. Calcd for C₁₃H₂₀F₃NO₆ (367.32): C, 49.05; H, 5.49; N, 3.81. Found: C, 48.46; H, 5.64; N, 3.90%

Diethyl 5-(tert-butylimino)-2-ethoxy-2-(trifluoromethyl)-2.5-dihydrofuran-3,4-dicarboxylate (4e): Orange oil (0.363 g, 92%); IR (KBr) (v_{max}, cm⁻¹): 2983, 2940, 1740 and 1698 (2C=O), 1656 (C=N); ¹H NMR (300.1 MHz, CDCl₃): δ_{H} 1.26 (3H, t, ³J=7.0 Hz, CH₃), 1.29 $(9H, s, C(CH_3)_3)$, 1.31 and 1.34 (6H, 2t, ${}^{3}J = 7.1$ Hz, 2CH₃), 3.57 (2H, q, ${}^{3}J$ = 7.1 Hz, OCH₂), 4.26 and 4.35 (4H, 2q, ${}^{3}J$ = 7.1 Hz, 2OCH₂); ^{13}C NMR (75.5 MHz, CDCl_3): δ_{C} 13.7 and 13.9 (2CH_3), 14.7 (CH_3), 29.4 (C(CH₃)₃), 55.7 (N-C(Me)₃), 60.8 (OCH₂), 62.3 and 62.4 $(2OCH_2)$, 106.5 (q, ²J = 35.2 Hz, CCF₃), 120.6 (q, ¹J = 286.2 Hz, CF₃), 135.0 and 142.6 (C_{olefin}), 147.7 (C_{imine}), 159.4 and 160.5 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): δ_F 80.40; MS (*m*/*z*, %): 396 (M⁺+1, 71), 380 (100), 334 (39), 306(3), 260 (16), 84 (8), 57 (31). Anal. Calcd for C₁₇H₂₄F₃NO₆ (395.37): C, 51.64; H, 6.12; N, 3.54. Found: C, 51.52; H, 6.31; N, 3.59%.

Di-tert-butyl 5-(tert-butylimino)-2-ethoxy-2-(trifluoromethyl)-2,5dihydrofuran-3,4-dicarboxylate (4f): Pale orange oil (0.401 g, 89%); IR (KBr) (v_{max}, cm⁻¹): 2981, 2936, 1726 and 1701 (2C=O), 1661 (C=N); ¹H NMR (300.1 MHz, CDCl₃): $\delta_{\rm H}$ 1.28 (3H, t, ³J = 7.1 Hz, CH₃), 1.29 (9H, s, N-C(CH₃)₃), 1.49 and 1.54 (18H, 2s, 2OC(CH₃)₃), 3.56 (2H, q, ${}^{3}J$ = 7.1 Hz, OCH₂); ${}^{13}C$ NMR (75.5 MHz, CDCl₃): δ_{C} 14.7 (CH₃), 27.8 and 28.0 (2C(CH₃)₃), 29.5 (C(CH₃)₃), 55.3 (N- $C(Me)_3$, 60.5 (OCH₂), 83.9 and 84.1 (2O- $C(Me)_3$), 106.0 (q, ²J = 34.9 Hz, CCF₃), 120.8 (q, ${}^{1}J$ = 286.1 Hz, CF₃), 135.2 and 142.2 (C_{olefin}), 147.9 (Cimine), 158.8 and 159.8 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): $\delta_{\rm F}$ 80.49; MS (*m*/*z*, %): 452 (M⁺+1, 100), 436 (18), 380 (4), 324 (41), 278 (5), 84 (4), 57 (85). Anal. Calcd for $C_{21}H_{32}F_3NO_6$ (451.48): C, 55.87; H, 7.14; N, 3.10. Found: C, 56.27; H, 7.63; N. 2.88%.

Dimethyl 5-(2,6-dimethylphenylimino)-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran-3,4-dicarboxylates (4g): Dark orange powder (0.373 g, 90%); m.p. 70–72 °C; IR (KBr) (v_{max} , cm⁻¹): 2926, 2855, 1764 and 1703 (2C=O), 1655 (C=N); ¹H NMR (300.1 MHz, CDCl₃): $\delta_{\rm H}$ 1.27 (3H, t, ${}^{3}J$ = 7.0 Hz, CH₃), 2.10 (6H, s, 2CH₃), 3.64 (2H, q, ${}^{3}J = 7.0$ Hz, OCH₂), 3.92 and 3.99 (6H, 2s, 2OCH₃), 6.93–7.10 (3H, m, Ar-H); ¹³C NMR (75.5 MHz, CDCl₃): δ_C 14.6 (CH₃), 17.8 $(2CH_3)$, 53.4 and 53.5 (2OCH₃), 61.3 (OCH₂), 105.6 (q, ²J = 35.1 Hz, CCF₃), 120.8 (q, ¹J = 286.5 Hz, CF₃), 127.6, 129.0, 132.6, 134.0 $(C_{arom}),\ 138.9$ and 142.9 $(C_{olefin}),\ 149.4$ $(C_{imine}),\ 159.7$ and 160.3 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): δ_F 80.13; MS (*m*/*z*, %): 416 (M⁺+1, 29), 415 (M⁺, 100), 400 (2), 386 (18), 370 (6), 354(14), 296 (22), 146 (11), 118 (10), 91 (11), 77 (13), 59 (9). Anal. Calcd for C₁₉H₂₀F₃NO₆ (415.36): C, 54.94; H, 4.85; N, 3.37. Found: C, 55.23; H, 4.89; N, 3.31%

Diethyl 5-(2,6-dimethylphenylimino)-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran-3,4-dicarboxylates (4h): Dark orange (0.416 g, 94%); IR (KBr) (v_{max}, cm⁻¹): 2985, 2941, 1740 and 1709 (2C=O), 1661 (C=N); ¹H NMR (300.1 MHz, CDC₃): $\delta_{\rm H}$ 1.27 (3H, t, ³*J* = 7.1 Hz, CH₃), 1.33 and 1.40 (6H, 2t, ³*J* = 7.1 Hz, 2CH₃), 2.11 (6H, s, $2CH_3$, 3.65 (2H, q, ${}^{3}J = 7.1$ Hz, OCH_2), 4.37 and 4.46 (4H, 2q, ${}^{3}J = 7.1$ Hz, 2OCH₂), 6.93–7.05 (3H, m, Ar-H); ¹³C NMR (75.5 MHz, CDCl₃): δ_c 13.8 and 14.0 (2CH₃), 14.6 (CH₃), 17.8 (2CH₃), 61.3 (OCH₂), 62.7 and 62.9 (20CH₂), 61.3 (OCH₂), 108.7 (q, ²J = 28.5 Hz, CCF₃), 120.3 $(q, {}^{I}J = 286.4 \text{ Hz}, \text{ CF}_3)$, 127.4, 127.6, 128.8, 130.9 (C_{arom}), 138.6 and 143.0 (Colefin), 149.9 (Cimine), 159.2 and 159.9 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): $\delta_{\rm F}$ 80.08; MS (*m*/*z*, %): 444 (M⁺+1, 82), 443 (M⁺, 100), 414 (10), 370 (16), 340(9), 282 (12), 149 (5), 103 (6), 77 (6). Anal. Calcd for C₂₁H₂₄F₃NO₆ (443.41): C, 56.88; H, 5.46; N, 3.16. Found: C, 57.32; H, 5.79; N, 3.23%.

Di-tert-butyl 5-(2,6-dimethylphenylimino)-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran-3,4-dicarboxylates (4i): Orange powder (0.439 g, 88%); m.p. 72-74 °C; IR (KBr) (v_{max}, cm⁻¹): 2982, 2935, 1729 and 1702 (2C=O), 1655 (C=N); ¹H NMR (300.1 MHz, CDCl₃): δ_H 1.26 $(3H, t, {}^{3}J = 7.0 \text{ Hz}, \text{ CH}_{3}), 1.55 \text{ and } 1.60 (18H, 2s, 2C(\text{CH}_{3})_{3}), 2.09$ (6H, s, 2CH₃), 3.64 (2H, q, ${}^{3}J$ = 7.0 Hz, OCH₂), 6.94–7.04 (3H, m, Ar-H); ¹³C NMR (75.5 MHz, CDCl₃): δ_C 14.6 (CH₃), 17.9 (2CH₃), 27.9 and 28.1 (2C(CH₃)₃), 60.9 (OCH₂), 84.7 and 84.8 (2OC(Me)₃), 105.7 (q, ${}^{2}J$ = 35.2 Hz, CCF₃), 125.5 (q, ${}^{1}J$ = 230.0 Hz, CF₃), 127.0, 127.5, 132.1, 133.5 (C_{arom}), 139.0 and 144.6 (C_{olefin}), 151.9 (C_{inine}), 159.4 and 159.9 (2C=O); ¹⁹F NMR (282.4 MHz, CDCl₃): δ_F 80.13; MS (*m/z*, %): 500 (M⁺+1, 7), 499 (M⁺, 18), 443 (59), 414 (8), 387 (100), 370 (27), 250 (31), 149 (34), 105 (15), 57 (84). Anal. Calcd for C₂₅H₃₂F₃NO₆ (499.52): C, 60.11; H, 6.46; N, 2.80. Found: C, 60.54; H, 7.04; N, 3.34%.

Dimethyl 5-(benzylimino)-2-ethoxy-2-(trifluoromethyl)-2,5-dihydrofuran-3,4-dicarboxylates (4j): Dark orange powder (0.369 g, 92%); m.p. 78–80 °C; IR (KBr) ($\nu_{max},\,cm^{-1})$: 2963, 2889, 1749 and 1695 (2C=O), 1654 (C=N); ¹H NMR (300.1 MHz, CDCl₃): δ_H 1.28 $(3H, t, {}^{3}J = 7.0 \text{ Hz}, \text{CH}_{3}), 3.60 (2H, q, {}^{3}J = 7.0 \text{ Hz}, \text{OCH}_{2}), 3.89 \text{ and}$ 3.93 (6H, 2s, 2OCH₃), 4.74 (2H, s, N-CH₂), 7.26-7.35 (5H, m, Ar-H); 13 C NMR (75.5 MHz, CDCl₃): δ_{C} 14.7 (CH₃), 52.3 (NCH₂), 53.3 and 53.4 (2OMe), 61.2 (OCH₂), 105.7 (q, ${}^{2}J$ = 35.1 Hz, CCF₃), 120.3 (q, ${}^{1}J = 276.6$ Hz, CF₃), 127.0, 127.7, 128.4, 138.0 (C_{arom}), 138.5 and 140.2 (C_{olefin}), 152.2 (C_{imine}), 159.8 and 160.4 (2C=O); ¹⁹F NMR (282.4) MHz, CDCl₃): δ_F 80.37; MS (*m*/*z*, %): 402 (M⁺+1, 6), 401 (M⁺, 9), 369 (12), 355 (67), 340 (11), 296 (26), 236 (100), 132 (14), 105 (8), 91 (89), 77 (7), 59 (20). Anal. Calcd for C₁₈H₁₈F₃NO₆ (401.33): C, 53.87; H, 4.52; N, 3.49. Found: C, 54.26; H, 4.55; N, 3.65%.

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