Synthetic Communications®, 36: 2087–2095, 2006

ISSN 0039-7911 print/1532-2432 online
DOI: 10.1080/00397910600636436

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Formation of 1,2,5-Oxadiazole, Isoxazole, Isothiazole, 1,2,3-Triazole, and Pyrrole Rings from *N*-(5,5-Dimethyl-3-oxocyclohexenyl)-*S,S*-diphenylsulfilimine

Masahiko Takahashi, Shinji Onda, and Nobuaki Matsumoto
Department of Materials Science, Faculty of Engineering,
Ibaraki University, Hitachi, Ibaraki, Japan

Abstract: Reactions of *N*-(5,5-dimethyl-3-oxocyclohexenyl)-*S,S*-diphenylsulfilimine, a kind of enaminone, with isopentyl nitrite, isocyanates, isothocyanates, benzenediazonium chloride, and 1,1,1-trifluoro-4-ethoxy-3-buten-2-one gave 1,2,5-oxadiazole, isoxazole, isothiazole, 1,2,3-triazole, and pyrrole derivatives condensed with cyclohexane, respectively, in one pot.

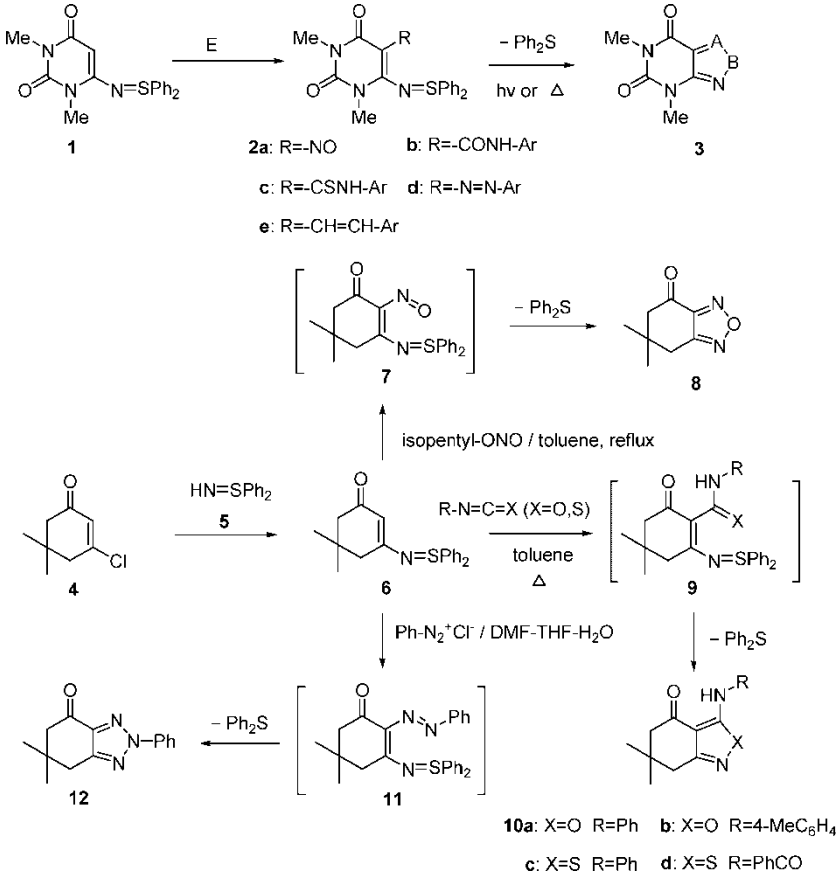
Keywords: Isothiazole, isoxazole, 1,2,5-oxadiazole, pyrrole, sulfilimine, 1,2,3-triazole

N-Unsubstituted sulfilimines (e.g., **5**) are useful building blocks for nitrogen heterocycles because they have a nucleophilic nitrogen atom and a good-leaving sulfonium atom.[1] These sulfilimine moieties could be readily introduced into organic molecules by a substitution reaction with olefinic halogens to give *N*-conjugated sulfilimines, which would be cyclized to heterocycles by elongation of the conjutated system, followed by extrusion of the sulfonium group by photolysis or thermolysis. According to this protocol, we have introduced a sulfilimine group into biological important uracils (**1**) to construct $\alpha,\beta,\gamma,\delta$-unsaturated sulfilimine systems (**2**) by the reaction with electrophiles and then cyclized them by photolysis or thermolysis to give uracils (**3**) condensed with 1,2,5-oxadiazole, isoxazole, isothiazole,[2] 1,2,3-triazole,[3]

Received in Japan October 7, 2005
Address correspondence to Masahiko Takahashi, Department of Materials Science, Faculty of Engineering, Ibaraki University, Hitachi, Ibaraki 316-8511, Japan. Fax: +81-294-38-5078; E-mail: takamasa@mx.ibaraki.ac.jp

and pyrrole rings[4] (Scheme 1). In these reactions, a modified enaminone structure, $-CO-C{=}C-N{=}SPh_2$, in the molecule (**1**) plays an important role. Therefore, we have studied similar reactions using a simpler enaminone system[5] and compared the reactivities.

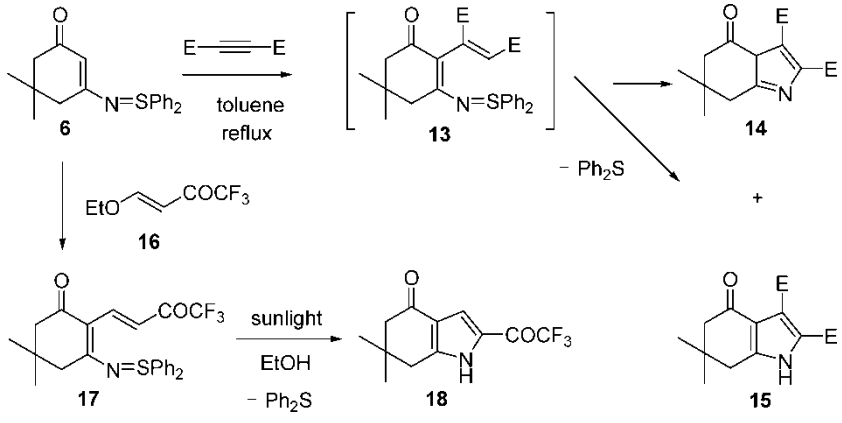
As an enaminone system simpler than **1**, we chose a cyclohexenone ring (**6**), which was readily prepared in 70% yield from 3-chloro-5,5-dimethylcyclohexen-3-one (**4**)[6] and *S,S*-diphenylsulfilimine (**5**)[7] using a method similar to the one reported.[8] At first, nitrosation by isopentyl nitrite was carried out. Refluxing a mixture of **6** and isopentyl nitrite in toluene gave 1,2,5-oxadiazole (furazan) (**8**) in 53% yield. The structures of **8** and the following compounds were determined on the basis of IR, ^{1}H NMR, ^{13}C NMR, and mass spectra and the elemental analysis. Although the nitroso intermediate (**2a**) was isolated in the case of the uracil derivatives (**1**),[2] the corresponding intermediate (**7**) was not obtained.



Scheme 1.

The reaction of **6** with phenyl isocyanate and *p*-tolyl isocyanate in refluxing toluene directly gave isoxazole rings (**10a**, 52%) and (**10b**, 46%). This is in marked contrast to the reaction of **1**, where the intermediate **2b** was isolated and converted into 3-substituted isoxazolo[3,4-*d*]pyrimidines (**3**) only by photolysis.[2] On the other hand, similar treatment of **6** with phenyl isothiocyanate and benzoyl isothiocyanate afforded condensed isothiazoles (**10c**, 21%) and (**10d**, 27%) in one step as observed in the reaction of **1**. The 1,2,3-triazole ring (**12**) was also prepared directly in 20% yield without isolation of the intermediate **11** by the reaction with benzenediazonium chloride prepared in a mixed solvent of DMF–THF–H_2O at 0°C to room temperature, and the isolation of azo compounds (**2d**) in high yields was reported in a previous paper.[3] The reaction of simple enaminones, Ph–CO–CH=C(Me)-NHR, with benzenediazonium tetrafluoroborate was recently reported to give azo-coupled enaminones and pyridazinium salts.[9]

The reaction of **6** with electron-deficient and unsaturated compounds appears to be a route to the pyrrole ring via the adduct (**13**). Indeed, the reaction with dimethyl acetylenedicarboxylate in refluxing toluene gave two pyrrole products (**14**, 28%) and (**15**, 27%) (Scheme 2). The structure of **14** was deduced on the basis of the spectral data; the NMR spectrum showed one singlet methine proton at δ 3.79, nonequivalent absorptions of methylene protons of C-5 and C-7, and two methyl protons at δ1.16 and 1.17. Furthermore, no NH absorption was observed in the IR spectrum. Additional heating of **14** in toluene did not afford the product (**15**), indicating that **14** would not be the intermediate to **15**. No intermediate (**13**) was isolated in this case. However, the reaction with 1,1,1-trifluoro-4-ethoxy-2-butenone (**16**)[10] yielded an isolable intermediate (**17**) in 58% yield, which was further transformed into the expected indole derivative (**18**) by photolysis in ethanol using a mercury lamp. Because photolysis of **2e** by sunlight to the



Scheme 2.

corresponding pyrrolo[2,3-*d*]pyrimidine[4] was achieved in high yields, the ethanolic solution of **17** was similarly irradiated by sunlight for 3 h to give **18** in the best yield of 98% (method A). The high yield of **18** by photolysis using sunlight prompted us to prepare **18** from **6** without isolation of the intermediate (**17**); a mixture of **6** and **16** in acetonitrile was stirred at room temperature for 5 days and then irradiated by sunlight for 4 h to give **18** in 49% yield after column chromatography (method B).

In conclusion, the elongation of the enaminone system (**6**) by electrophiles followed by cyclization occurred thermally in one pot without isolation of the intermediates **7**, **9**, **11**, and **13**, showing that the reactivity of these intermediates is higher than **2**. The rather low yields in some cases are attributable to the thermal unstability of the starting **6**, which caused decomposition during refluxing in the solvents. Cyclization of **17** to **18** in high yield by sunlight was also observed as in the case of **2e**, suggesting that the system of C═C–C═C–N═SR_2 is a very useful scaffold for the construction of pyrrole.

EXPERIMENTAL

Melting points were determined with MRK Mel-Temp II and are uncorrected. The IR spectra were measured on Jasco FT/IR-420 spectrophotometer. MS and NMR spectra were taken with JEOL JMS DX-300 and JEOL GSX-400 spectrometer, respectively. Microanalyses were performed with Yanaco CHN-Coder MT-5.

N-(5,5-Dimethyl-3-oxocyclohexenyl)-*S,S*-diphenylsulfilimine (6)

This compound was prepared according to the method for *N*-(3-oxocyclohexenyl)-*S,S*-diphenylsulfilimine.[8]A mixture of **4**[6] (574 mg, 3.6 mmol), **5**[7] (1.19 g, 5.4 mmol), and triethylamine (1.0 ml) in acetonitrile (5 ml) was refluxed for 12 h. After evaporation of the solvent, $CHCl_3$ (10 ml) and water (50 ml) were added to the residue. The mixture was extracted with $CHCl_3$ and the $CHCl_3$ solution was dried over $MgSO_4$. Evaporation of the solvent gave an oil, which was purified by column chromatography on silica gel using $CHCl_3$–AcOEt (1:1) to give yellow viscous oil (**6**) (817 mg, yield 70%). Because the product was somewhat labile to heat, it was used for the next step without further purification. IR (KBr): 2956, 1736, 1601, 1520, 1475, 1444, 1362, 1240, 1142 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.09 (s, 6H), 2.16 (s, 2H), 2.47 (s, 2H), 5.20 (s, 1H), 7.46–7.70 (m, 10H). ^{13}C NMR ($CDCl_3$): δ 28.33, 33.01, 47.44, 50.30, 101.05, 127.24, 129.87, 132.14, 135.37, 174.78, 196.44.

6,6-Dimethyl-4,5,6,7-tetrahydrobenzofurazan-4-one (8)

A mixture of **6** (258 mg, 0.80 mmol) and isopentyl nitrite (0.13 ml, 1.0 mmol) in toluene (4 ml) was refluxed for 2 h. After evaporation of the solvent, the

residue was purified by column chromatography on silica gel using $CHCl_3$ and washed with hexane to give **8** (70 mg, 53%), white needles, mp 65–66°C (MeOH). IR (KBr): 1728, 1570, 1468, 1242 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.16 (s, 3H), 2.65 (s, 2H), 2.99 (s, 2H). ^{13}C NMR ($CDCl_3$): δ 28.08, 33.36, 34.91, 53.84, 148.34, 155.00, 188.38. MS m/z (%): 166 (M^+, 31), 109 (88), 94 (100), 80 (58), 55 (96). Anal. calcd. for $C_8H_{10}N_2O_2$: C, 57.82; H, 6.07; N, 16.86. Found: C, 57.46; H, 5.84; N, 16.94.

6,6-Dimethyl-3-phenylamino-4,5,6,7-tetrahydrobenz[*c*]isoxazol-4-one (10a)

Typical Experimental Procedure

A mixture of **6** (380 mg, 1.18 mmol) and phenyl isocyanate (0.38 ml, 3.53 mmol) in toluene (4 ml) was refluxed for 8 h. After evaporation of the solvent, the product was isolated by column chromatography on silica gel using $CHCl_3$ and recrystallized from MeOH to give **10a** (156 mg, 52%), white needles, mp 156–157°C. IR (KBr): 3282, 1657, 1628, 1583, 1543, 1432, 1255 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.14 (s, 6H), 2.32 (s, 2H), 2.67 (s, 2H), 7.14–7.46 (m, 5H), 8.75 (br s, 1H). ^{13}C NMR ($CDCl_3$): δ 28.44, 34.75, 35.39, 51.22, 94.83, 118.68, 124.57, 129.44, 136.32, 163.17, 164.17, 192.06. MS m/z (%): 256 (M^+, 69), 228 (20), 187 (22), 120 (51), 93 (83), 77 (100). Anal. calcd. for $C_{15}H_{16}O_2N_2$: C, 70.29; H, 6.29; N, 10.93. Found: C, 70.27; H, 6.26; N, 11.00.

6,6-Dimethyl-3-(*p*-tolylamino)-4,5,6,7-tetrahydrobenz[*c*]isoxazol-4-one (10b)

Yield 46%, white plates, mp 130–131°C (MeOH). IR (KBr): 3292, 2958, 1655, 1626, 1581, 1550, 1466, 1252 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.13 (s, 6H), 2.32 (s, 2H), 2.34 (s, 3H), 2.66 (s, 2H), 7.18 (d, $J = 8.6$ Hz, 2H), 7.33 (d, $J = 8.6$ Hz, 2H), 8.67 (br s, 1H). ^{13}C NMR ($CDCl_3$) δ: 20.90, 28.48, 34.77, 35.42, 51.22, 94.66, 118.76, 129.96, 133.75, 134.42, 163.11, 164.22, 191.96. MS m/z (%): 270 (M^+, 53), 242 (16), 200 (25), 172 (16), 144 (16), 107 (100), 77 (64). Anal. calcd. for $C_{16}H_{18}N_2O_2$: C, 71.09; H, 6.71; N, 10.36. Found: C, 71.20; H, 6.73; N, 10.41.

6,6-Dimethyl-3-phenylamino-4,5,6,7-tetrahydrobenz[*c*]isothiazol-4-one (10c)

Yield 21%, white plates, mp 126–127°C (MeOH). IR (KBr): 3211, 2954, 1631, 1597, 1566, 1495, 1394, 1244 cm^{-1}. ^1H NMR ($CDCl_3$) δ: 1.12 (s, 6H), 2.45 (s, 2H), 2.73 (s, 2H), 7.13–7.45 (m, 5H), 10.69 (br s, 1H). ^{13}C NMR ($CDCl_3$): δ 28.35, 34.50, 42.76, 51.97, 113.05, 117.76, 124.00, 129.71, 139.32, 168.79, 171.32, 195.20. MS m/z (%): 272 (M^+, 100), 257

(19), 216 (20), 170 (10), 142 (12), 115 (18), 77 (95). Anal. calcd. for $C_{15}H_{16}N_2OS$: C, 66.15; H, 5.92; N, 10.29. Found: C, 66.14; H, 5.95; N, 10.27.

3-Benzoylamino-6,6-dimethyl-4,5,6,7-tetrahydrobenz[*c*]isothiazol-4-one (10d)

Yield 27%, orange needles, mp 141–142°C (MeOH). IR (KBr): 3228, 2960, 1670, 1649, 1552, 1469, 1257 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.13 (s, 6H), 2.52 (s, 2H), 2.83 (s, 2H), 7.55–8.05 (m, 5H), 11.92 (br s, 1H). ^{13}C NMR ($CDCl_3$): δ 28.36, 34.77, 42.35, 52.27, 115.89, 127.82, 129.12, 130.23, 133.56, 164.71, 165.09, 166.82, 196.55. MS m/z (%): 300 (M^+, 14), 283 (3), 195 (3), 105 (100), 77 (71). Anal. calcd. for $C_{16}H_{16}N_2O_2S$: C, 63.98; H, 5.37; N, 9.33. Found: C, 63.93; H, 5.43; N, 9.32.

6,6-Dimethyl-2-phenyl-4,5,6,7-tetrahydro-2*H*-benzotriazol-4-one (12)

A solution of **6** (384 mg, 1.2 mmol) in a mixed solvent of DMF (5 ml) and THF (1 ml) was added dropwise to a solution of benzenediazonium chloride prepared from aniline (0.14 ml, 1.5 mmol), hydrochloric acid (0.2 ml of concentrate hydrochloric acid in 5 ml of water), and sodium nitrite (104 mg, 1.5 mmol in 3 ml of water) below 5 °C during 20 min. After stirring for 1 h at 5 °C, the stirring was continued for 1 h at room temperature. Water (100 ml) was added to the mixture, and it was extracted with chloroform (5 ml × 3). The oily residue obtained after evaporation of the solvent was washed with diethyl ether–hexane to give a solid (**12**) (56 mg, 20%), white needles, mp 113–114 °C (MeOH). IR (KBr): 1691, 1597, 1518, 1483, 1462, 1342, 1325 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.18 (s, 6H), 2.59 (s, 2H), 2.92 (s, 2H), 7.39–8.17 (m, 5H). MS m/z (%): 241 (M^+, 35), 185 (33), 105 (28), 77 (100). Anal. calcd. for $C_{14}H_{15}N_3O$: C, 69.69; H, 6.27; N, 17.42. Found: C, 69.53; H, 6.34; N, 17.41.

Dimethyl 6,6-dimethyl-4-oxo-4,5,6,7-tetrahydro-3a*H*-indole-2,3-dicarboxylate (14) and Dimethyl 6,6-dimethyl-4-oxo-4,5,6,7-tetrahydro-1*H*-indole-2,3-dicarboxylate (15)

A mixture of **6** (390 mg, 1.2 mmol) and dimethyl acetylenedicarboxylate (596 mg, 4.2 mmol) in toluene (4 ml) was refluxed for 3 h. After evaporation of the solvent, the residue was purified by column chromatography on silica gel using $CHCl_3$. The products (**14**) (94 mg, 28%) and (**15**) (90 mg, 27%) were obtained as oily substances from the first and the second fractions, respectively. They were solidified by addition of diethyl ether. The product

14 is white needles, mp 106–107°C (diethyl ether-hexane). IR (KBr): 2954, 1738, 1726, 1655, 1437, 1269, 1254 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.16 (s, 3H), 1.17 (s, 3H), 2.47 (d, $J = 12.6$ Hz, 1H), 2.86 (d, $J = 17.2$ Hz, 1H), 2.93 (d, $J = 17.2$ Hz, 1H), 3.23 (d, $J = 12.6$ Hz, 1H), 3.79 (s, 3H), 3.88 (s, 3H), 6.94 (s, 1H). ^{13}C NMR ($CDCl_3$): δ 28.38, 31.73, 32.31, 34.23, 37.29, 51.97, 53.02, 53.05, 139.06, 141.77, 161.64, 165.59, 170.18, 201.19. MS m/z (%): 248 (M^+-31, 4), 220 (3), 164 (9), 136 (3), 83 (100). Anal. calcd. for $C_{14}H_{17}NO_5$: C, 60.21; H, 6.13; N, 5.02. Found: C, 60.21; H, 6.10; N, 5.01. The product (**15**) is white powder, mp 164–165°C (THF–hexane). IR (KBr): 3334, 1738, 1703, 1657, 1523, 1495, 1448, 1304, 1282, 1252, 1203, 1169 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.12 (s, 6H), 2.38 (s, 2H), 2.71 (s, 2H), 3.86 (s, 3H), 3.94 (s, 3H), 9.95 (br s, 1H). ^{13}C NMR ($CDCl_3$): δ 28.46, 35.53, 36.37, 51.98, 52.30, 52.81, 118.42, 119.64, 120.36, 144.30, 160.50, 165.73, 192.30. MS m/z (%): 279 (M^+, 37), 248 (23), 223 (100), 191 (76), 165 (14), 135 (14), 107 (22), 78 (38). Anal. calcd. for $C_{14}H_{17}NO_5$: C, 60.21; H, 6.13; N, 5.02. Found: C, 60.05; H, 6.07; N, 4.99.

N-[5,5-Dimethyl-2-(4,4,4-trifluoro-3-oxo-1-butenyl)-3-oxocyclohexenyl]-*S,S*-diphenylsulfilimine (17)

A mixture of **6** (311 mg, 0.96 mmol) and **16** (568 mg, 3.4 mmol) in acetonitrile (4 ml) was stirred at room temperature for 4 days. After evaporation of the solvent, the residue was purified by column chromatography on silica gel using $CHCl_3$ to give **17** (249 mg, 58%), yellow plates, mp 140 °C (dec.) (MeOH). IR (KBr): 2952, 1668, 1631, 1545, 1396, 1329, 1267, 1236, 1186, 1128, 1070, 1005 cm^{-1}. ^{1}H NMR ($CDCl_3$): δ 1.01 (s, 6H), 2.30 (s, 2H), 2.68 (s, 2H), 7.56–7.80 (m, 10H), 7.83 (d, $J = 15.6$ Hz, 1H), 8.80 (d, $J = 15.6$ Hz, 1H). ^{13}C NMR ($CDCl_3$): δ 28.40, 31.85, 46.50, 50.95, 111.39, 113.33, 117.43 (q, $J = 291$ Hz), 127.13, 130.49, 133.09, 135.61, 143.96, 178.95, 181.33 (q, $J = 32$ Hz), 194.64. MS m/z (%): 259 (M^+-186), 203 (68), 186 (100), 175 (45), 77 (32). Anal. calcd. for $C_{24}H_{22}F_3NO_2S$: C, 64.70; H, 4.98; N, 3.14. Found: C, 64.62; H, 5.09; N, 3.17.

2-Trifluoroacetyl-6,6-dimethyl-4,5,6,7-tetrahydro-1*H*-indol-4-one (18)

Method A

A solution of **17** (160 mg, 0.36 mmmol) in EtOH (30 ml) was irradiated by sunlight at room temperature for 3 h. After evaporation of the solvent, the residue was purified by column chromatography on silica gel using $CHCl_3$ to give **18** (91 mg, 98%).

Method B

A mixture of **6** (332 mg, 1.0 mmmol) and **16** (594 mg, 3.5 mmol) in acetonitrile (4 ml) was stirred at room temperature for 5 days. Acetonitrile (25 ml) was further added to the solution, and the mixture was irradiated by sunlight for 4 h. After the solvent was evaporated in vacuo, the residue was purified by column chromatography on silica gel using $CHCl_3$ to give **18** (132 mg, 49%), white plates, mp 199–200°C ($CHCl_3$–hexane). IR (KBr): 3248, 1676, 1649, 1514, 1214, 1194, 1147 cm^{-1}. ^1H NMR (DMSO-d_6): δ 1.04 (s, 6H), 2.35 (s, 2H), 2.78 (s, 2H), 7.32 (s, 1H). ^{13}C NMR (DMSO-d_6): δ 27.90, 34.93, 35.82, 51.63, 116.55 (q, $J = 287$ Hz), 117.35, 121.21, 125.22, 151.34, 169.19 (q, $J = 35$ Hz), 192.57. MS m/z (%): 259 (M^+, 32), 203 (100), 175 (49), 78 (39). Anal. calcd. for $C_{12}H_{12}F_3NO_2$: C, 55.60; H, 4.67; N, 5.40. Found: C, 55.72; H, 4.75; N, 5.41.

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