Four-component reaction of isocyanides, acetylenic esters, and carboxylic acids for the synthesis of functionalised 2,5-diaminofurans

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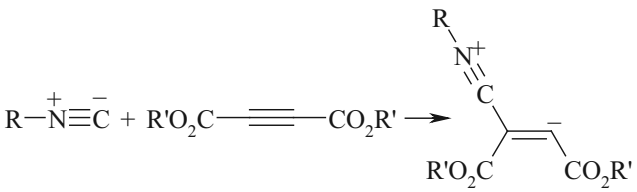
An improved four-component reaction of isocyanides is described. The reaction between two equivalents of an isocyanide, dialkyl acetylenedicarboxylates and aliphatic carboxylic acids at room temperature leads to 2,5-diaminofuran derivatives in good yields.

Keywords: isocyanide, four-component reaction, dialkyl acetylenedicarboxylates, carboxylic acids, diaminofurans

An important subject that has gained a great deal of attention from organic and bioorganic chemists during the last few decades has been the developing of new strategies for the synthesis of complex molecular structures from easily available substrates by short and effective routes. The most important of these strategies has been the developing of multi-component reactions (MCRs), a reaction in which three or more compounds connect together by covalent bonds to produce a complex molecule contains the main structure of all the starting materials. As MCRs are one-pot reactions, they are easier to carry out than multistep syntheses. Coupled with high-throughput library screening, this strategy was an important development in the drug discovery in the context of rapid identification and optimisation of biologically active lead compounds.[1-9]

Among the MCRs, isocyanide based multi-component reactions (IMCRs) have gained the most attention by the organic chemists. Ugi four component reaction(U-4CR)[6-8] and Passerini three component reaction (P-3CR)[10] are among the most important IMCRs. U-4CR and P-3CR describe the reaction of isocyanides with carboxylic acids in the presence of imines or aldehydes, respectively.

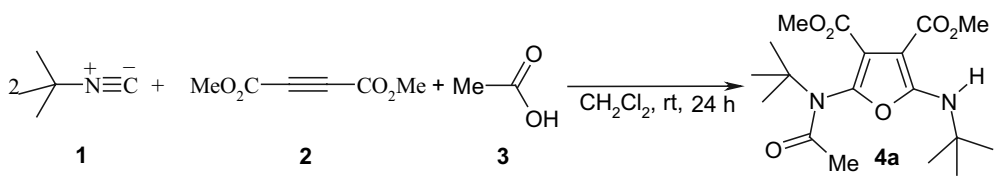
Recently, another kind of IMCRs has been developed and extensively investigated. Isocyanides react easily with electron-deficient acetylene diesters such as dimethyl acetylenedicarboxylate (DMAD) to produce a reactive zwitterionic intermediate (Scheme 1), which can be trapped by an electrophile. In recent years, a wide variety of electrophiles have been applied to trap isocyanide–DMAD intermediate, among them are carbon electrophiles such as aldehydes, imines, quinonids,[11] 1,2-diketones,[12] 1,2,3-tricarbonyl compounds,[13] isocyanates,[14] and hydrogen electrophiles such as pyrrole,[15] amides,[16] hydroxy coumarine,[17] phenoles,[18] phthalic anhydride,[19] and isatoic anhydride.[20] Treatment of isocyanide–DMAD zwitterion with aromatic carboxylic acids has been reported to produce unsaturated amides.[21] Reaction of isocyanide-DMAD adduct with aromatic-substituted acetic acids has been reported to afford 2,5-diaminofuran derivatives in the presence of two equivalents of an isocyanide.[22] In the context of our previous work on IMCRs,[15-17, 23] we now report the results of our investigations on the reaction of isocyanides and dialkyl acetylenedicarboxylates (DAADs) in the presence of aliphatic carboxylic acids such as acetic acid, propionic acid, trifluoroacetic acid, formic acid and succinic acid.



Scheme 1 Isocyanide–acetylene diester zwitterion.

Treatment of *tert*-butyl isocyanide (2 equiv.) with DMAD (1 equiv.) and acetic acid (1 equiv) in dichloromethane for 24 h at room temperature, after silica gel column chromatography afforded dimethyl 2-(acetyl-*tert*-butylamino)-5-(*tert*-butylamino)-furan-3,4-dicarboxylate (**4a**) in 97% yield (Scheme 2). The structure of compound **4a** was deduced form its elemental and spectral data. The ^{1}H NMR spectrum of compound **4a** was completely simple and exhibited six sharp single lines, which are respectively due to two *tert*-butyl groups (δ = 1.17 and 1.22 ppm), one methyl group (δ = 1.76), two methoxy groups (δ = 3.61 and 3.68 ppm) and one NH group (δ = 6.70 ppm, disappeared with addition of D_2O). The ^{13}C NMR spectrum of compound **4a** showed 14 distinct resonances in agreement with the proposed structure. The signals at 114.5, 139.5, 159.9, 163.4, 161.3 and 172.9 ppm are related to furan ring carbons and two carbonyl groups. The IR spectrum showed an absorption band at 3410 cm^{-1} for NH group. The carbonyl stretching vibrations observed as strong absorption bands at 1734, 1685 and 1676 cm^{-1}. The molecular ion peak at 368 in the mass spectrum of compound **4a** supported the 2:1:1 adduct of *tert*-butyl isocyanide, acetic acid and DMAD.

Similar reactivity was observed with other acetylene diesters, such as diethyl acetylenedicarboxylate (DEAD) and di(*tert*-butyl) acetylenedicarboxylate (DTAD), which underwent facile reaction with *tert*-butyl isocyanide and acetic acid yielding the 2,5-diaminofuran derivatives **4b–c** in good yields (enties 2–3, Table 1). The reaction was also compatible with cyclohexyl isocyanide, instead of *tert*-butyl isocyanide affording 2,5-diaminofuran derivative **4d**. Propionic acid was also found to be as reactive as acetic acid and 2,5-diaminofuran derivatives **4e–h** were obtained from its reaction with DAADs and isocyanides. The reaction was also examined with trifluoroacetic acid under similar conditions.



Scheme 2 Reaction of *tert*-butyl isocyanide, DMAD and acetic acid.

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Clean reactions took place with DMAD, trifluoroacetic and *tert*-butyl or cyclohexyl isocyanide and after silica gel column chromatography 2,5-diaminofuran derivatives **4i** and **4j** were obtained in good yields, respectively. From the reaction between *tert*-butyl isocyanide-DMAD zwitterion and formic acid a complex mixture was obtained and our efforts for separation of a pure product were unsuccessful. We also could not isolate any pure product from the complex mixture of the reaction between *tert*-butyl isocyanide-DMAD intermediate and succinic acid.

On the basis of the well established chemistry of isocyanides[6-8,22] it is reasonable to assume that compound **4** is produced by initial protonation of isocyanide–DAAD zwitterion intermediate by carboxylic acid followed by the addition of carboxylate anion **6** on nitrilium cation **5** to afford intermediate **7** which then rearranges to unsaturated imide **8**. Cycloaddition of another molecule of isocyanide to imide **8** leads to dihydrofuran intermediate **9** that tautomerises to furan derivative **4**.

In conclusion, the four-component reaction between isocyanides, aliphatic carboxylic acids and dialkyl acetylenedicarboxylates is a simple and efficient route for the synthesis of functionalised 2,5-diaminofuran derivatives. The advantages of the reported method are inexpensive and easily available starting materials, simple and neutral reaction conditions, high yields, single-product reaction and simple work-up processes.

Experimental

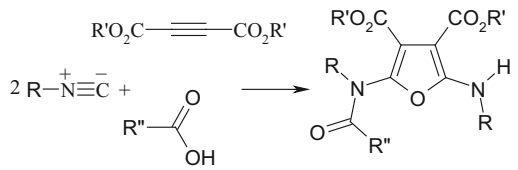
Elemental analyses were performed using a Heraeus CHN-O-Rapid analyser. Mass spectra were recorded on a FINNIGAN-MAT 8430 mass spectrometer operating at an ionisation potential of 70 eV. IR spectra were recorded on a Shimadzu IR-470 spectrometer. ^{1}H and ^{13}C NMR spectra were recorded on Bruker DRX-500 Avance spectrometer at solution in $CDCl_3$ using TMS as internal standard. The chemicals used in this work purchased from Fluka (Buchs, Switzerland) and were used without further purification.

General procedure

To a magnetically stirred solution of isocyanide (2 mmol) and carboxylic acid (1 mmol) in 10 mL dichloromethane was added a mixture of dialkyl acetylenedicarboxylate (1 mmol) in 1 mL dichloromethane at room temperature. The reaction mixture was then stirred for 24 h. The solvent was removed and the residue was purified by silica gel column chromatography using hexane-ethyl acetate (6 : 1) as eluent. The solvent was removed under reduced pressure to afford the product.

Dimethyl 2-[acetyl(tert-butyl)amino]-5-(tert-butylamino)furan-3,4-dicarboxylate (**4a**): Yellow oil, yield 0.36 g (97%); IR (KBr) (ν_{max}, cm^{-1}): 3410 (NH), 1734, 1685, 1676 (carbonyl groups). Anal. Calcd for $C_{18}H_{28}N_2O_6$: C, 58.68; H, 7.66; N, 7.60%. Found: C, 58.47; H, 7.50; N, 7.71%. MS (*m/z*,%): 368 (M^+, 10). ^{1}H NMR (500.1 MHz, $CDCl_3$): δ = 1.17 and 1.22 (18 H, 2 s, 2 *tert*-butyl), 1.76 (3 H, s, CH_3), 3.61 and 3.68 (6 H, 2 s, 2 OCH_3), 6.70 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 24.3 (CH_3), 28.5, 30.1 (methyl groups of 2 *tert*-butyl), 53.0 and 60.6 (2 NC), 52.7 and 55.8 (2 OCH_3), 86.6, 114.5, 139.5, 159.9, 163.4, 165.1 and 172.9 (Furan ring and carbonyl carbons).

Table 1 Four-component reaction of isocyanides, dialkyl acetylenedicarboxylates and aliphatic carboxylic acids

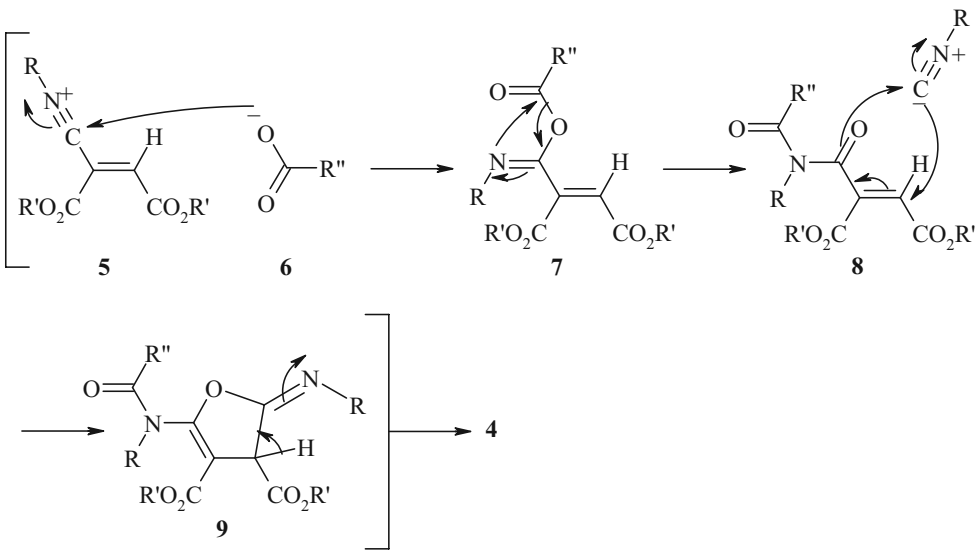


Entry	R	R'	R"	Product	Yield/%[a]
1	*tert*-Bu	Me	Me	**4a**	95
2	*tert*-Bu	Et	Me	**4b**	95
3	*tert*-Bu	*t*-Bu	Me	**4c**	90
4	*tert*-Bu	Me	Me	**4d**	94
5	*tert*-Bu	Me	Et	**4e**	90
6	*tert*-Bu	Et	Et	**4f**	93
7	*tert*-Bu	*t*-Bu	Et	**4g**	95
8	Cy	Me	Et	**4h**	90
9	Cy	Me	CF_3	**4i**	85
10	Cy	Me	CF_3	**4j**	88

[a]Isolated yields.

Diethyl 2-[acetyl(tert-butyl)amino]-5-(tert-butylamino)furan-3,4-dicarboxylate (**4b**): Yellow oil, yield 0.38 g (95%); IR (KBr) (ν_{max}, cm^{-1}): 3350 (NH), 1729, 1688, 1665 (carbonyl groups). Anal. Calcd for $C_{20}H_{32}N_2O_6$: C, 60.59; H, 8.14; N, 7.07%. Found: C, 60.77; H, 8.33; N, 6.78%. MS (*m/z*,%): 396 (M^+, 15). ^{1}H NMR (500.1 MHz, $CDCl_3$): δ = 1.29 and 1.35 (6 H, 2 t, *J* = 7 Hz, 2 CH_3), 1.39 and 1.45 (18 H, 2 s, 2 *tert*-butyl), 2.00 (3 H, s, CH_3), 4.28 (4 H, m, 2 CH_2), 6.88 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 14.5 and 14.7 (2 CH_3), 24.9 (CH_3), 28.5, 30.1 (methyl groups of 2 *tert*-butyl), 53.0 and 60.6 (2 NC), 60.2 and 61.8 (2 OCH_2), 86.6, 114.5, 139.5, 159.9, 163.4, 165.1 and 172.9 (Furan ring and carbonyl carbons).

Di-tert-butyl 2-[acetyl(tert-butyl)amino]-5-(tert-butylamino)furan-3,4-dicarboxylate (**4c**): Yellow oil, yield 0.41 g (90%); IR (KBr) (ν_{max}, cm^{-1}): 3400 (NH), 1725, 1689, 1663 (carbonyl groups). Anal. Calcd for $C_{24}H_{40}N_2O_6$: C, 63.69; H, 8.91; N, 6.19%. Found: C, 63.71; H, 8.80; N, 6.44%. MS (*m/z*,%): 452 (M^+, 12). ^{1}H NMR (500.1 MHz, $CDCl_3$): δ = 1.30, 1.32, 1.43 and 1.45 (36 H, 4 s, 4 *tert*-butyl), 1.90 (3 H, s, CH_3), 6.66 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 24.9 (CH_3), 28.5, 28.6, 28.9, 30.1 (methyl groups of 4 *tert*-butyl), 52.7 and 60.3 (2 NC), 80.7 and 82.3 (2 OC), 88.0, 115.9, 138.6, 159.5, 162.1, 164.7 and 173.0 (Furan ring and carbonyl carbons).



Dimethyl 2-[acetyl(cyclohexyl)amino]-5-(cyclohexylamino)furan-3,4-dicarboxylate (**4d**): Yellow oil, yield 0.39 g (94%); IR (KBr) (ν_{max}, cm^{-1}): 3340 (NH), 1732, 1690, 1675 (carbonyl groups). Anal. Calcd for $C_{22}H_{32}N_2O_6$: C, 62.84; H, 7.67; N, 6.66%. Found: C, 62.74; H, 7.33; N, 6.60%. MS (*m/z*,%): 420 (M^+, 9). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 0.96–1.96 (10 H, 5 CH_2 of cyclohexyl), 1.99 (3 H, m, CH_3), 3.75 and 3.79 (6 H, 2 s, 2 OCH_3), 4.29 (1 H, m, CH of cyclohexyl), 6.68 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 22.97 (CH_3), 24.8, 25.7, 25.8, 26.1, 32.9, 33.9 (10 CH_2 of cyclohexyl groups), 51.51 and 51.85 (2 NCH), 52.71 and 55.74 (2 OCH_3), 85.7, 114.9, 137.9, 160.1, 163.5, 165.3 and 172.2 (Furan ring and carbonyl carbons).

Dimethyl 2-[tert-butyl(propanoyl)amino]-5-(tert-butylamino)furan-3,4-dicarboxylate (**4e**): Yellow oil, yield 0.34 g (90%); IR (KBr) (ν_{max}, cm^{-1}): 3335 (NH), 1733, 1685, 1675 (carbonyl groups). Anal. Calcd for $C_{19}H_{30}N_2O_6$: C, 59.67; H, 7.91; N, 7.32%. Found: C, 59.44; H, 7.99; N, 7.10%. MS (*m/z*,%): 382 (M^+, 15). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 0.89 (3 H, t, *J* = 7 Hz, CH_3), 1.22 and 1.28 (18 H, 2 s, 2 *t*-butyl), 1.84 and 2.25 (2 H, 2 dq, *J* = 7 Hz, *J* = 16 Hz, CH_2), 3.62 and 3.68 (6 H, 2 s, 2 OCH_3), 6.76 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 9.4 (CH_3), 29.1 (CH_2), 28.4, 30.0 (methyl groups of 2 *tert*-butyl), 52.9 and 60.5 (2 NC), 51.3 and 52.5 (2 OCH_3), 86.3, 114.1, 139.5, 159.9, 163.6, 165.3 and 175.8 (Furan ring and carbonyl carbons).

Diethyl 2-[tert-butyl(propionyl)amino]-5-(tert-butylamino)furan-3,4-dicarboxylate (**4f**): Yellow oil, yield 0.38 g (93%); IR (KBr) (ν_{max}, cm^{-1}): 3330 (NH), 1729, 1691, 1665 (carbonyl groups). Anal. Calcd for $C_{21}H_{34}N_2O_6$: C, 61.44; H, 8.35; N, 6.82%. Found: C, 61.65; H, 8.50; N, 6.68%. MS (*m/z*,%): 410 (M^+, 14). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 0.96 (3 H, t, *J* = 7 Hz, CH_3), 1.21 and 1.55 (6 H, 2 t, *J* = 7 Hz, 2 CH_3), 1.31 and 1.34 (18 H, 2 s, 2 *tert*-butyl), 1.93 and 2.36 (2 H, 2 dq, *J* = 7 Hz, *J* = 16 Hz, CH_2), 4.22 (4 H, m, 2 CH_2), 6.81 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 9.4 (CH_3), 14.3 and 14.6 (2 CH_3), 29.3 (CH_2), 28.5, 30.7 (methyl groups of 2 *tert*-butyl), 52.9 and 60.5 (2 NC), 60.0 and 61.6 (2 OCH_2), 86.6, 114.4, 139.0, 159.8, 163.3, 165.0 and 175.9 (Furan ring and carbonyl carbons).

Ditert-butyl 2-[tert-butyl(propionyl)amino]-5-(tert-butylamino) furan-3,4-dicarboxylate (**4g**): Yellow oil, yield 0.44 g (95%); IR (KBr) (ν_{max}, cm^{-1}): 3400 (NH), 1717, 1694, 1663 (carbonyl groups). Anal. Calcd for $C_{25}H_{42}N_2O_6$: C, 64.35; H, 9.07; N, 6.00%. Found: C, 64.23; H, 9.26; N, 6.24%. MS (*m/z*,%): 466 (M^+, 12). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 0.95 (3 H, t, *J* = 7 Hz, CH_3), 1.31, 1.33, 1.43 and 1.45 (36 H, 4 s, 4 *tert*-butyl), 1.93 and 2.41 (2 H, 2 dq, *J* = 7 Hz, *J* = 17 Hz, CH_2), 6.75 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 9.3 (CH_3), 28.3 (CH_2), 28.5, 28.7, 28.9, 30.2 (methyl groups of 4 *tert*-butyl), 52.7 and 60.3 (2 NC), 80.7 and 82.2 (2 OC), 88.0, 116.0, 138.2, 159.5, 162.2, 164.8 and 176.3 (Furan ring and carbonyl carbons).

Dimethyl 2-[cyclohexyl(propionyl)amino]-5-(cyclohexylamino) furan-3,4-dicarboxylate (**4h**): Yellow oil, yield 0.39 g (90%); IR (KBr) (ν_{max}, cm^{-1}): 3340 (NH), 1732, 1691, 1675 (carbonyl groups). Anal. Calcd for $C_{23}H_{34}N_2O_6$: C, 63.57; H, 7.89; N, 6.45%. Found: C, 63.22; H, 7.91; N, 6.29%. MS (*m/z*,%): 434 (M^+, 17). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 0.96-1.96 (13 H, 5 CH_2 of cyclohexyl and CH_3), 1.95 and 2.39 (2 H, 2 dq, *J* = 7 Hz, *J* = 16 Hz, CH_2), 3.70 and 3.78 (6 H, 2 s, 2 OCH_3), 4.26 (1 H, m, CH of cyclohexyl), 6.65 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 9.3 (CH_3), 28.3 (CH_2), 24.8, 25.6, 25.8, 26.4, 32.9, 33.9 (10 CH_2 of cyclohexyl groups), 51.6 and 51.8 (2 NCH), 52.6 and 55.8 (2 OCH_3), 85.7, 114.7, 137.7, 160.1, 163.5, 165.5 and 172.2 (Furan ring and carbonyl carbons).

Dimethyl 2-[tert-butyl(trifluoroacetyl)amino]-5-(tert-butylamino) furan-3,4-dicarboxylate (**4i**):Yellow oil, yield 0.36 g (85%); IR (KBr) (ν_{max}, cm^{-1}): 3335 (NH), 1735, 1685, (carbonyl groups). Anal. Calcd for $C_{18}H_{25}F_3N_2O_6$: C, 51.18; H, 5.97; N, 6.63%. Found: C, 51.39; H, 5.66; N, 6.73%. MS (*m/z*,%): 422 (M^+, 10). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 1.44 and 1.48 (18 H, 2 s, 2 *tert*-butyl), 3.80 and 3.84 (6 H, 2 s, 2 OCH_3), 7.00 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 27.6, 30.6 (methyl groups of 2 *tert*-butyl), 53.2 and 64.4 (2 NC), 51.6 and 52.5 (2 OCH_3), 86.5, 114.8, 135.2, 157.8, 162.6 and 165.5 (Furan ring and two ester carbonyl carbons), 117.2 (d, *J* = 281 Hz, CF_3), 157.7 (q, *J* = 36 Hz, *C*OCF_3).

Dimethyl 2-[cyclohexyl(trifluoroacetyl)amino]-5-(cyclohexylamino) furan-3,4-dicarboxylate (**4j**): Yellow oil, yield 0.42 g (88%); IR (KBr) (ν_{max}, cm^{-1}): 3335 (NH), 1735, 1685, (carbonyl groups). Anal. Calcd for $C_{22}H_{29}F_3N_2O_6$: C, 55.69; H, 6.16; N, 5.90%. Found: C, 55.48; H, 6.12; N, 5.76%. MS (*m/z*,%): 474 (M^+, 9). ^1H NMR (500.1 MHz, $CDCl_3$): δ = 0.93-1.96 (10 H, 5 CH_2 of cyclohexyl), 3.82 and 3.85 (6 H, 2 s, 2 OCH_3), 4.21 (1 H, m, CH of cyclohexyl), 7.12 (1 H, s, NH). ^{13}C NMR (125.7 MHz, $CDCl_3$): δ = 24.8, 25.3, 25.4, 26.7, 32.6, 33.9 (10 CH_2 of cyclohexyl groups), 51.5 and 51.8 (2 NCH), 51.7 and 52.4 (2 OCH_3), 86.3, 114.2, 135.6, 157.9, 162.8 and 165.3 (Furan ring and two ester carbonyl carbons), 117.5 (d, *J* = 280 Hz, CF_3), 157.0 (q, *J* = 36 Hz, *C*OCF_3).

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