Isocyanatophosphoric Acid Dichloride: A Novel Reagent for the Introduction of a Cyano Group into the Molecules of Electron-Rich Heterocycles and Enamines

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Abstract: A new synthetic method is developed which enables a direct one-step introduction of a cyano group into electron-rich heterocyclic systems of the indole, pyrrole, and indolizine series, and also to enamines using isocyanatophosphoryl dichloride.

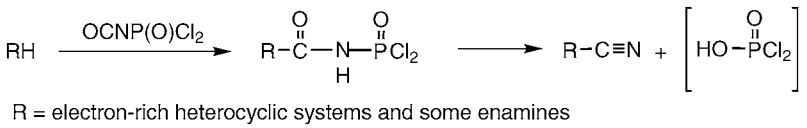
Key words: nitriles, isocyanatophosphoryl dichloride, indoles, pyrroles, indolizines, enamines

New chemical agents enabling functional groups to be selectively introduced into organic compounds under mild conditions attract the active interest of synthetic practitioners. Among such reagents, one can quote triphenylthiocyanogen (TPPT)[1] and chlorosulfonyl isocyanate (the Graf isocyanate)[2] which made possible a direct one-step introduction of a cyano group into a number of electron-rich heterocycles and enamines.[1,3] These chemicals, however, are not free of certain disadvantages: TPPT is relatively difficult to prepare and unstable under storage, and the reaction mixture resulting on using it contains an equimolar quantity of hard-to-separate triphenylthiophosphine oxide[1]; as for the Graf isocyanate, it reacts does not react selectively with more active heterocycles than indole and gives rise to side reactions[4].

Herein we report isocyanatophosphoryl dichloride (the Kirsanov isocyanate)[5] as an easily accessible and convenient reagent for the introduction of a cyano group into electron-rich heterocycles and enamines. As was reported by us previously, the Kirsanov isocyanate dissolved in octane readily acylates electron-rich heterocycles to furnish, in good yields, *N*-dichlorophosphorylheterylcarboxamides.[6] Here we have found that the latter compounds decompose in more polar solvents [CH_2Cl_2, $Cl(CH_2)_2Cl$, CH_3CN] at 20 °C to give the desired nitriles and a mixture of phosphorus-containing inorganic products easily removable with water. As a result, we have elaborated a one-step method for the introduction of a cyano group into electron-rich heterocycles and enamines by reacting them with the Kirsanov isocyanate in polar solvents (Scheme 1); conditions and results of the synthesis are listed in Table 1. The nitrile formation rate is limited by the first reaction step, acylation of the substrate.

In most cases, heterocyclic nitriles are formed under mild conditions over a period of 1-4 h at 20 °C, except for nitriles **2a**, **5a, 10a**, and **12a** (Table 1). Thus, in the synthesis of nitrile **10a**, the reaction lasts for 10-12h, which is due to the electron-acceptor substituent in the substrate.[6] The intermediate *N*-dichlorophosphorylhetarylcarboxamides formed in the preparation of **2a** and **5a** are rather stable thermally and need heating to release nitriles. To obtain nitrile **12a**, the reaction mixture should be boiled for 1 hour so as to destroy the adduct of the isocyanate and the heterocycle.

The cyano group is introduced selectively into heterocycles **1b**, **3**–**7b**, **10**–**12b**. Highly reactive 2-methylindolizine reacts with the isocyanate to provide a mixture of nitriles **8a** and **9a** readily separable by crystallization. *N*-Phenylpyrrole **2b** undergoes the nonselective acylation[6] resulting in a mixture of α- and β-isomeric nitriles from which we have managed to isolate the predominant component, the nitrile **2a**.



Scheme 1

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Table 1 Introduction of a Cyano Group with Isocyanatophosphoryl Dichloride

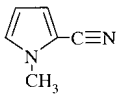
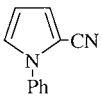
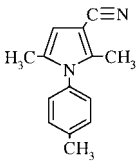
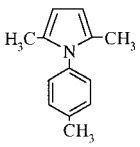
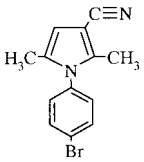
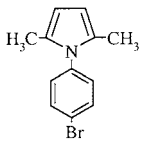
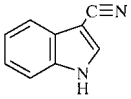
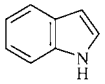
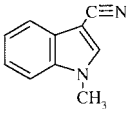
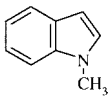
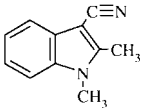
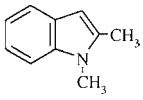
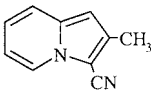
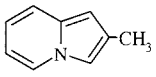
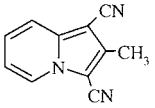
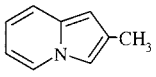
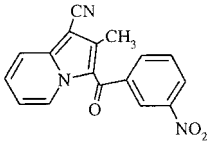
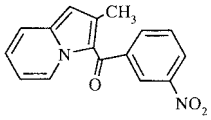
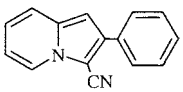
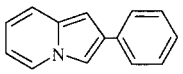
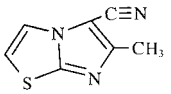
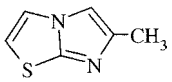
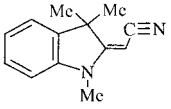
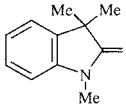
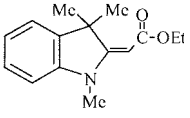
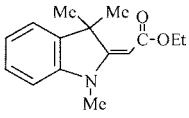
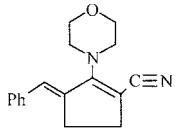
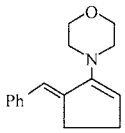
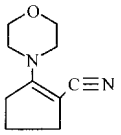
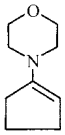
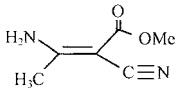
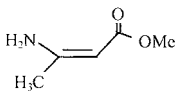
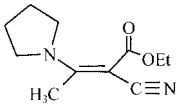
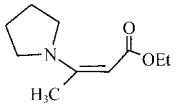
Compound No.	Nitrile **a**[a]	Substrate **b**	Solvent	Reaction Time (h); Temperature (°C)	Yields (%)[b]
1			$Cl(CH_2)_2Cl$	1; 20	64
2			CH_2Cl_2	2; reflux	45
3			$Cl(CH_2)_2Cl$	4; 20	92
4			$Cl(CH_2)_2Cl$	4; 20	95
5			CH_2Cl_2–CH_3CN (1:1)	1; reflux	89
6			$Cl(CH_2)_2Cl$	2; 20	93
7			$Cl(CH_2)_2Cl$	2; 20	95
8			$Cl(CH_2)_2Cl$	2; 20	50
9			$Cl(CH_2)_2Cl$	2; 20	20
10			$Cl(CH_2)_2Cl$	12; 20	90
11			$Cl(CH_2)_2Cl$	4; 20	80
12			CH_2Cl_2	1; reflux	67
13			CH_2Cl_2	4; 20	46
14			$Cl(CH_2)_2Cl$	2; 20	95

Table 1 Introduction of a Cyano Group with Isocyanatophosphoryl Dichloride (continued)

Compound No.	Nitrile **a**[a]	Substrate **b**	Solvent	Reaction Time (h); Temperature (°C)	Yields (%)[b]
15			CH_2Cl_2	12; 20	57
16			$Cl(CH_2)_2Cl$	1; reflux	15
17			$Cl(CH_2)_2Cl$	2; 20	30
18			$Cl(CH_2)_2Cl$	2; 20	70

[a] Compounds **1a**, 2a, **5a**–**7a**, **12a**, **13a**, **15a**–**18a** were previously described in the literature.
[b] Isolated yields.
[c] Reaction is run in the presence of Et_3N.

The Kirsanov isocyanate makes it also possible to introduce, in one step, a cyano group into enamine molecules. Nitriles **13a**, **14a**, **17a**, and **18a** are synthesized under mild conditions like heterocyclic nitriles. To obtain **15a** and **16a**, the reaction should be run in the presence of excess triethylamine in order to avoid the hydrolytic cleavage of the enamines into the ketonitriles. The low yield of nitrile **17a** is attributed to the nonselective reaction of the Kirsanov isocyanate with aminocrotonic acid, an ambident nucleophile.

In summary, good yields, the simple work-up and the comparatively easy accessibility of the Kirsanov isocyanate enable us to regard this compound as a convenient and promising reagent for the direct one-step introduction of a cyano group in the molecules of electron-rich heterocycles and enamines.

^{1}H NMR spectra were recorded on Varian Gemini – 300 spectrometer at 300 MHz using TMS as an internal standard. IR spectra were recorded on a UR–20 spectrophotometer using KBr discs for solid samples and films on NaCl plates for oils. IR spectra of the compounds obtained exhibit the characteristic signal for the C≡N bond (2200 cm^{-1}). Solvents were dried and purified prior to use.

Nitriles 1a–18a; General Procedure
To a stirred solution of the substrate (10 mmol) in the respective solvent (20 mL) was added isocyanatophosphoryl dichloride (1.6 g, 10 mmol). The mixture was left at r.t. or refluxed for the time given in Table 1. The solvent was removed in vacuo and the residue was purified.

1-Methyl-1*H*-pyrrole-2-carbonitrile (1a)
The general procedure was followed starting from **1b** (0.81 g). The residue was distilled in vacuo; yield: 0.68 g (64%); yellow oil; bp 60–62 °C/20 mmHg.

^{1}H NMR (DMSO-d_6): δ = 3.90 (s, 3 H, N-CH_3), 6.20 (t, *J* = 3 Hz, 1 H, H-4), 7.0 (d, *J* = 2.5 Hz, 1 H, H-3), 7.20 (br s, 1 H, H-5).

Anal. Calcd for $C_6H_6N_2$: C, 67.91; H, 5.70; N, 26.40. Found: C, 67.85; H, 5.74; N, 26.37.

1-Phenyl-1*H*-pyrrole-2-carbonitrile (2a)
The general procedure was followed starting from **2b** (1.43 g). To the residue was added H_2O (20 mL) and the precipitate was filtered. The product was extracted from residue with heptane (3 × 10 mL) and the solvent was removed in vacuo. The product was purified by recrystallization from heptane; yield: 0.76 g (45%); colorless solid; mp 58–59 °C.

^{1}H NMR (DMSO-d_6): δ = 6.40 (t, *J* = 7.5 Hz, 1 H, H-4), 7.12 (d, *J* = 7.5 Hz, 1 H, H-5), 7.50 (m, 6 H, C_6H_5, H-3).

Anal. Calcd for $C_{11}H_8N_2$: C, 78.55; H, 4.79; N, 16.65. Found: C, 78.49; H, 4.81; N, 16.61.

2,5-Dimethyl-1-(4-methylphenyl)-1*H*-pyrrole-3-carbonitrile (3a)
The general procedure was followed starting from **3b** (1.85 g). To the residue was added H_2O (20 mL) and the precipitate was filtered and dried in vacuo. The product was purified by recrystallization from Et_2O; yield: 1.93 g (92%); yellow solid; mp 110–111 °C.

^{1}H NMR ($CDCl_3$): δ = 2.0 (s 3 H, 5-CH_3), 2.19 (s, 3 H, 2-CH_3), 2.44 (s, 3 H, Ar-CH_3), 6.12 (s, 1 H, H-4), 7.16 (d, *J* = 7.5 Hz, 2 H, *m*-Ar), 7.31 (d, *J* = 7.5 Hz, 2 H, *o*-Ar).

Anal. Calcd for $C_{14}H_{14}N_2$: C, 79.97; H, 6.71; N, 13.32. Found: C, 79.90; H, 6.76; N, 13.29.

1-(4-Bromophenyl)-2,5-dimethyl-1*H*-pyrrole-3-carbonitrile (4a)
Prepared according to the method described for **3a** starting from **4b** (2.5 g). The product was purified by recrystallization from 70% aq MeOH; yield: 2.6 g (95%); colorless solid; mp 140–141 °C.

^{1}H NMR ($CDCl_3$): δ = 1.98 (s, 3 H; 2-CH_3), 2.14 (s, 3 H, 5-CH_3), 6.15 (s, 1 H, H-4), 7.26 (d, *J* = 8.3 Hz, 2 H, *m*-ArH), 7.70 (d, *J* = 8.3 Hz, 2 H, *o*-ArH).

Anal. Calcd for $C_{13}H_{11}BrN_2$: C, 56.75; H, 4.03; N, 10.18. Found: C, 56.70; H, 4.10; N, 10.12.

1*H*-Indole-3-carbonitrile (5a)
The general procedure was followed starting from **5b** (1.17 g). To the residue was added H_2O (20 mL) and the mixture was stirred for 2 h. The precipitate was filtered, dried in vacuo and purified by recrystallization from 70% aq MeOH, yield: 1.26 g (88.7%); pink solid; mp 178–180 °C.

^{1}H NMR ($CDCl_3$): δ = 7.22 (m, 2 H, H-5,6), 7.51 (d, *J* = 7.5 Hz, 1 H, H-4), 7.61 (d, *J* = 7.5 Hz, 1 H, H-7), 8.12 (d, *J* = 3 Hz, 1 H, H-2), 12.0 (s, 1 H, N).

Anal. Calcd for $C_9H_6N_2$: C, 76.04; H, 4.25; N, 19.71. Found: C, 76.00; H, 4.27; N, 19.70.

1-Methyl-1*H*-indole-3-carbonitrile (6a)
The general procedure was followed starting from **6b** (1.31 g). To the residue was added H_2O (20 mL) and the mixture was stirred for 20 min. The product was extracted from the crude residue with octane (3 × 10 mL) and the solvent was removed in vacuo; yield: 1.45 g (93%); yellow oil; bp 135 °C/20 mmHg.

^{1}H NMR ($CDCl_3$): δ = 3.89 (s, 3 H, N-CH_3), 7.30 (m, 2 H, H-5,6), 7.52 (m, 2 H, H-4,7), 8.15 (s, 1 H, H-2).

Anal. Calcd for $C_{10}H_8N_2$: C, 76.90; H, 5.16; N, 17.94. Found: C, 76.87; H, 5.17; N, 18.00.

1,2-Dimethyl-1*H*-indole-3-carbonitrile (7a)
This compound was prepared according to the method described for **6a** starting from **7b** (1.45 g); yield: 1.62 g (95%); yellow solid; mp 104–106 °C.

^{1}H NMR ($CDCl_3$): δ = 2.57 (s, 3 H, 2-CH_3), 3.73 (s, 3 H, N-CH_3), 7.31 (m, 2 H, H-5,6), 7.50 (m, 2 H, H-4), 7.56 (m, 4 H, H-7).

Anal. Calcd for $C_{11}H_{10}N_2$: C, 77.62; H, 5.92; N, 16.46. Found: C, 77.60; H, 5.95; N, 16.45.

2-Methyl-3-indolizinecarbonitrile and 2-methyl-1,3-indolizinedicarbonitrile (9a)
To a stirred solution of 2-methylindolizine (1.31 g, 10 mmol) in 1,2-dichloroethane (20 mL) at 0 °C was added isocyanatophosphoryl dichloride (1.6 g, 10 mmol). The mixture was left at r.t. for 2 h. The solvent was removed in vacuo. To the residue was added H_2O (20 mL) and the precipitate was filtered and dried in vacuo. The precipitate was dissolved in MeOH and cooled to 3 °C and the precipitated **9a**, was filtered. The mother liquor was concentrated in vacuo and the residue of compound **8a** was recrystallized from Et_2O.

8a
Yield: 0.78 g (50%); yellow solid; mp 93–94 °C.

^{1}H NMR ($CDCl_3$): δ = 3.03 (s, 3 H, CH_3), 6.27 (s, 1 H, H-1), 6.72 (t, *J* = 6.8 Hz, 1 H, H-6), 6.95 (t, *J* = 8.6 Hz, 1 H, H-5), 7,30 (d, *J* = 8.9 Hz, 1 H, H-7), 8.13 (d, *J* = 6.9 Hz, 1 H, H-4).

Anal. Calcd for $C_{10}H_8N_2$: C, 76.90; H, 5.16; N, 17.94. Found: C, 76.98; H, 5.17; N, 18.02.

9a
Yield: 0.36 g (20%); yellow solid; mp 222–223 °C.

^{1}H NMR ($CDCl_3$): δ = 3.10 (s, 3 H, CH_3), 7.06 (t, *J* = 6.9 Hz, 1 H, H-6), 7.36 (t, *J* = 7.8 Hz, 1 H, H-5), 7.70 (d, *J* = 9.2 Hz, 1 H, H-7), 8.29 (d, *J* = 6.9 Hz, 1 H, H-4).

Anal. Calcd for $C_{11}H_7N_3$: C, 72.92; H, 3.89; N, 23.19. Found: C, 72.90; H, 3.95; N, 23.16.

2-Methyl-3-(3-nitrobenzoyl)indolizine-1-carbonitrile (10a)
Compound **10a** was prepared according to the method described for **3a** starting from **10b** (2.8 g), by leaving the reaction mixture at r.t. for 12 h. The product was purified by recrystallization from MeOH; yield: 2.75 g (90%); yellow solid; mp 229–230 °C.

^{1}H NMR ($CDCl_3$): δ = 2.15 (s, 3 H, CH_3), 7.20 (t, *J* = 8.1 Hz, 1 H, H-5), 7.40 (t, *J* = 8.7 Hz, 1 H, H-6), 7.69 (t, *J* = 8.1 Hz, 1 H, *m*-ArH), 8.0 (d, *J* = 6.6 Hz, 1 H, H-7), 8.10 (d, *J* = 8.7 Hz, 1 H, *o*-ArH), 8.41 (d, *J* = 7.5 Hz, 1 H, *p*-ArH), 8.50 (s, 1 H, *o*-ArH), 9.56 (d, *J* = 6.9 Hz, 1 H, H-4).

Anal. Calcd for $C_{17}H_{11}N_3O_3$: C, 66.88; H, 3.63; N, 13.76. Found: C, 66.90; H, 3.65; N, 13.72.

2-Phenylindolizine-3-carbonitrile (11a)
Compound **11a** was prepared according to the method described for **3a** starting from **11b** (1.93 g). The product was purified by recrystallization from EtOH; yield: 1.74 g (80%); green solid; mp 90–93 °C.

^{1}H NMR ($CDCl_3$): δ = 6.69 (s, 1 H, H-1), 6.83 (t, *J* = 6.9 Hz, 1 H, H-5), 7.03 (t, *J* = 7.8 Hz, 1 H, H-6), 7.43 (m, 1 H, H-7), 7.60 (m, 5 H, C_6H_5), 8,30 (d, *J* = 6.3 Hz, 1 H, H-4).

Anal. Calcd for $C_{15}H_{10}N_2$: C, 82.55; H, 4.62; N, 12.83. Found: C, 82.51; H, 4.64; N, 12.77.

6-Methylimidazo[2,1-*b*][1,3]thiazole-5-carbonitrile (12a)
The general procedure was followed starting from **12b** (1.38 g). To the stirred residue were added first H_2O (15 mL) and then the adjusted to 7.5 with 15% aq NaOH. The mixture was stirred for 30 min, the precipitate was filtered and dried in vacuo. The product was purified by recrystallization from MeOH; yield: 1.1 g (66.5%); colorless or pink solid; mp 135–136 °C.

^{1}H NMR ($CDCl_3$): δ = 2.39 (s, 3 H, CH_3), 7,50 (d, *J* = 4.2 Hz, 1 H, H-1), 8,1 (d, *J* = 4.2 Hz, 1H, H-6).

Anal. Calcd for $C_7H_5N_3S$: C, 51.52; H, 3.09; N, 25.75. Found: C, 51.48; H, 3.10; N, 25.70.

2-(1,3,3-Trimethyl-1,3-dihydro-2*H*-indol-2-ylidene)acetonitrile (13a)
To a stirred solution of **13b** (1.73 g, 10 mmol) in CH_2Cl_2 (20 mL) at –50 °C was added isocyanatophosphoryl dichloride (1.6 g, 10 mmol). The mixture was left at r.t. for 4 h. The reaction mixture pH was adjusted to 7 with 10% aq NaOH. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2 (30 mL). The organic layers were combined and the solvent was removed in vacuo. The product was purified by silica gel column chromatography using CCl_4–benzene, 1:1 as eluent; yield: 0.91 g (46%); pink solid; mp 115–116 °C.

^{1}H NMR ($CDCl_3$): δ = 1.576 (s, 6 H, 2 × CH_3), 3.4 (s, 3 H, N-CH_3), 4.39 (s, 1 H C-H) 6.92 (m, 2 H, ArH) 7.22 (m, 2 H, ArH).

Anal. Calcd for $C_{13}H_{14}N_2$: C, 78.75; H, 7.12; N, 14.13. Found: C, 78.71; H, 7.17; N, 14.11.

Ethyl 2-Cyano-2-(1,3,3-trimethyl-1,3-dihydro-2*H*-indol-2-ylidene)acetate (14a)
Compound **14a** was prepared according to the method described for **3a** starting from **14b** (2.45 g), by leaving the reaction mixture to stir at r.t. for 2 h. The product was purified by recrystallization from octane; yield: 2.57 g (95%); yellow solid; mp 105–106 °C.

^{1}H NMR ($CDCl_3$): δ = 1.25 (t, *J* = 9 Hz, 3 H, CH_3), 1.62 (s, 6 H, 2 × CH_3), 3.52(s, 3 H, N-CH_3), 4.18 (q, *J* = 9 Hz, 2 H, CH_2), 7.23 (m, 4 H, ArH).

Anal. Calcd for $C_{16}H_{18}N_2O_2$: C, 74.97; H, 6.71; N, 11.66. Found: C, 74.95; H, 6.74; N, 11.71.

2-(4-Morpholinyl)-3-(phenylmethylidene)cyclopent-1-ene-1-carbonitrile (15a)

To a stirred solution of **15b** (2.41 g, 10 mmol) and Et_3N (5.05g, 50 mmol) in CH_2Cl_2 (20 mL) was added isocyanatophosphoryl dichloride (1.6 g, 10 mmol). The mixture was left at r.t. for 12 h. The solvent was removed in vacuo. The product was extracted from the crude residue with heptane–Et_2O (1:1, 10 × 6 mL) afford the product **16a** in 24 h; yield: 1.52 g (57%); brown solid; mp 105–106 °C.

^{1}H NMR ($CDCl_3$): δ = 2.5 (m, 2 H, CH_2,), 2.82 (m, 2 H, CH_2), 3.36 (m, 4 H, CH_2N-CH_2), 3.74 (m, 4 , OCH_2-CH_2), 6.65 (s, 1 H, CH), 7,33 (m, 5 H, C_6H_5).

Anal. Calcd for $C_{17}H_{18}N_2O$: C, 76.66; H, 6.81; N, 10.52. Found: C, 76.61; H, 6.85; N, 10.50.

2-(4-Morpholinyl)cyclopent-1-ene-1-carbonitrile (16a)

To a stirred solution of **16b** (1.53 g, 10 mmol) and Et_3N (5.05g, 50 mmol) in dichloroethane (20 mL) was added isocyanatophosphoryl dichloride (1.6 g, 10 mmol). The mixture was refluxed for 1 h and the solvent was removed in vacuo. The product was extracted from the residue with benzene (3 × 20 mL) and the solvent was removed in vacuo; yield: 0.267 g (15%); yellow oil.

^{1}H NMR ($CDCl_3$): δ = 2.45 (m, 2 H, CH_2,), 2.74 (m, 2 H, CH_2),3.19 (br s, 4 H, CH_2N-CH_2), 3.45 (m 6 H, CH_2, OCH_2-CH_2).

Anal. Calcd for $C_{10}H_{14}N_2O$: C, 67.39; H, 7.92; N, 15.72. Found: C, 67.35; H, 7.90; N, 15.75.

Methyl 3-Amino-2-cyanobut-2-enoate (17a)

Compound **17a** was prepared according to the method described for **3a** starting from **17b** (1.15 g), by leaving the reaction mixture at r.t. for 2 h. The product was purified by recrystallization from octane; yield: 0.42 g (30%); yellow solid; mp 182–183 °C.

^{1}H NMR ($CDCl_3$): δ = 2.15 (s, 3 H, CH_3), 3.63 (s, 3 H, OCH_3), 8.9 (br s, 2 H, NH_2).

Anal. Calcd for $C_6H_8N_2O_2$: C, 51.42; H, 5.75; N, 19.99. Found: C, 51.39; H, 5.78; N, 20.01.

Ethyl 2-Cyano-3-(1-pyrrolidinyl) but-2-enoate (18a)

Compouind **18a** was prepared according to the method described for **3a** starting from **18b** (1.83 g). The product was extracted from the residue with octane (3 × 10 mL) and the solvent was removed in vacuo; yield: 1.36 g (70%); yellow oil.

^{1}H NMR ($CDCl_3$): δ = 1.20 (t, *J* = 9 Hz, 3 H, CH_3), 1.89 (s, 3 H, CH_3), 2.42 (s, 4 H, pyrrolidinyl), 3.63 (s, 4 H, pyrrolidinyl), 4.04 (q, *J* = 9 Hz, 2 H, CH_2).

Anal. Calcd for $C_{10}H_{16}N_2O_2$: C, 61.84; H, 7.27; N, 14.42. Found: C, 61.81; H, 7.30; N, 14.40.

References

(1) Tamura, Y.; Kawasaki, T.; Adachi, M.; Tanio, M.; Kita, Y. *Tetrahedron Lett.* **1977**, *50*, 4417.
(2) Graf, R. *Chem. Ber.* **1956**, *89*, 1071.
(3) (a) Tamura, Y.; Adachi, M.; Kawasaki, T.; Yasuda, H.; Kita, Y. *J. Chem. Soc., Perkin Trans. 1* **1980**, 1132. (b) Lohaus, G. *Cem. Ber.* **1967**, *100*, 2719. (c) Mehta, G.; Dhar, D.; Suri, S. *Synthesis* **1978**, 374.
(4) Barnet, G.; Anderson, H.; Loader, C. *Can. J. Chem.* **1980**, *58*, 409.
(5) Kirsanov, A. V. *Zh. Obshch. Chem.* **1954**, *24*, 1033; *Chem. Abstr.* **1955**, *49*, 8787.
(6) Chaykovskaya, A. A.; Smaliy, R. V.; Tolmachev, A. A.; Kudrya, T. N.; Pinchuk, A. M. *Heteroatom Chem.* **1999**, *10*, 343.