SUPPORTING INFORMATION for

The combination of *tert*-butoxycarbonyl and triphenylphosphonium protecting groups in the synthesis of substituted hydrazines

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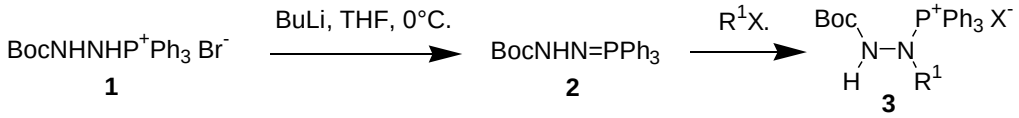
1. General methods

All melting points were measured on a Gallencamp melting point apparatus. Tetrahydrofuran was distilled from $LiAlH_4$ under argon immediately before use. The reaction vessels were dried in an oven at 200°C if a reaction is done under argon. TLC analyses were carried out on 0.25 mm thick precoated silica plates (Merck DC-Fertigplatten Kieselgel 60 F_{254}. TLC spots were visualized under UV light or by alcoholic phosphomolybdenic acid with subsequent heating (blue spots). Column chromatography was carried out on Merck Kieselgel 70-230 mesh. ^1H were recorded at 200 MHz and ^{13}C NMR spectra at 50 MHz on a Bruker AC 200P spectrometer in $CDCl_3$ or CD_3OD solution. All chemical shifts are given in ppm using TMS as reference and coupling constants are given in Hz. Chemical shifts of conformers are given in decreasing order of intensity and separated by slashes.

2. Spectral data and melting points of phosphonium salts 3



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All the phosphonium salts **(3)** were obtained as white solids. For the melting point measurements they all were recrystallized from acetonitrile.

Spectral data and melting points

3a. (R^1 = methyl) was isolated as THF solvate, mp=141-144°C (dec) (solvate), mp=110-112°C (recrystallized from MeCN).

^1H NMR ($CDCl_3$): δ=1.21 (s, 9H, Boc), 1.86 (m, 4H, CH_2 of THF), 3.30 (d, N-Me, J_{PH}=6.2), 3.74 (m, 4H, CH_2O of THF), 7.6-8.1 (m, 15H, 3×Ph), 9.19 (s, 1H, NH).

^{13}C NMR ($CDCl_3$): δ=25.5 (s, CH_2 of THF), 27.9 (s, Boc), 40.7 (d, N-Me, J_{PC}=12.5), 67.9 (s, CH_2O of THF), 81.6 (s, C_q, Boc), 119.3 (d, Ph, J_{PC}=105.4), 129.8 (d, Ph, J_{PC}=13.6), 134.9 (d, Ph, J_{PC}=11.1), 135.3 (d, Ph, J_{PC}=2.9), 155.4 (s, CO).

3b. (R^1 = benzyl), mp=164-165°C (dec)

^1H NMR ($CDCl_3$): δ=0.99 (s, 9H, Boc), 4.05-4.45 (broad signal, 1H, N-CH_2Ph), 5.0-5.6 (broad signal, 1H, N-CH_2Ph), 7.2-8.2 (2×m, 20H, 4×Ph), 9.98 (s, 1H, NH).

^{13}C NMR ($CDCl_3$): δ=27.8 (s, Boc), 56.3 (d, N-CH_2Ph, J_{PC}=11.7), 80.6 (s, C_q, Boc), 119.8 (d, Ph, J_{PC}=101.1), 128.4 (d, Ph, J_{PC}=7.8), 129.7 (d, Ph, J_{PC}=13.2), 130.8 (s, Ph), 133.2 (d, Ph, 5.0), 135.2-135.4 (overlapped signals, Ph), 155.5 (s, CO).

3c. (R^1 = 4-nitrobenzyl), mp=165-166.5°C (dec)

^1H NMR ($CDCl_3$): δ=0.99 (s, 9H, Boc), 4.1-4.5 (broad signal, 1H, N-CH_2Ph), 5.3-5.6 (broad signal, 1H, N-CH_2Ph), 7.6-8.2 (2×m, 19H, 3×Ph and $NO_2C_6H_4$), 10.13 (s, 1H, NH).

^{13}C NMR ($CDCl_3$): δ=27.7 (s, Boc), 55.1 (d, N-CH_2Ph, J_{PC}=13.0), 81.2. (s, C_q, Boc), 119.4 (d, Ph, J_{PC}=100.6), 123.3 (s, Ph), 130.0 (d, Ph, J_{PC}=13.4), 131.7 (s, Ph), 135.2 (d, Ph, J_{PC}=10.8), 135.6 (s, Ph), 140.5 (d, Ph, J_{PC}=5.5), 148.1 (s, Ph), 155.5 (s, CO).

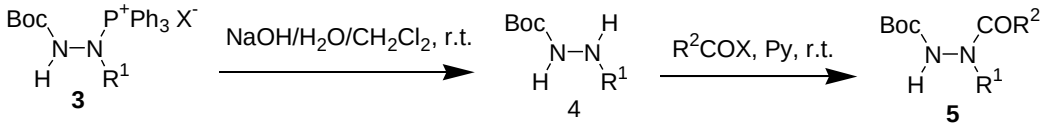
3d. (R^1 = *n*-butyl) mp=169-170°C (dec)
^{1}H NMR ($CDCl_3$): δ=0.78 (t, 3H, CH_3 of *n*-butyl), 1.0-1.3 (overlapped signals, 11H, Boc and $\underline{CH_2}$-CH_3), 1.75 (broad signal, 2H, $\underline{CH_2}$-Et), 3.2-3.8 (broad signal, 2H, N-CH_2), 7.6-8.1 (m, 15H, 3×Ph), 9.23 (s, 1H, NH).
^{13}C NMR ($CDCl_3$): δ=13.6 (s, CH_3 of *n*-butyl), 19.8 (s, $\underline{CH_2}$-CH_3), 28.0 (s, Boc), 29.3 (d, $\underline{CH_2}$-Et, J_{PC}=2.4), 53.4 (d, N-CH_2, J_{PC}=10.8), 81.3 (s, C_q, Boc), 119.6 (d, Ph, J_{PC}=101.6), 129.9 (d, Ph, J_{PC}=13.2), 135.3 (s, Ph, J=3.6), 155.8 (s, CO).

3e. (R^1 = propargyl) mp=170-171°C (dec)
^{1}H NMR ($CDCl_3$): δ=1.20 (s, 9H, Boc), 2.37 (s, 1H, C≡CH), 4.0-4.9 (broad signal, 2H, N-CH_2), 7.6-8.1 (m, 15H, 3×Ph), 9.81 (s, 1H, NH).
^{13}C NMR ($CDCl_3$): δ=28.0 (s, Boc), 43.9 (d, N-CH_2, J_{PC}=15.7), 76.1 ($\underline{HC}$≡C), 76.8 (d, HC≡$\underline{C}$, J_{PC}=3.3), 81.3 (s, C_q, Boc), 119.4 (d, Ph, J_{PC}=101.9), 129.8 (d, Ph, J_{PC}=13.4), 135.1 (d, Ph, J_{PC}=11.2), 135.4 (d, Ph, J_{PC}=2.9), 155.5 (s, CO).

3f. (R^1 = ethoxycarbonylmethyl) mp=162-163°C (dec)
^{1}H NMR ($CDCl_3$): δ=1.05-1.3 (12H, Boc and $\underline{CH_3}CH_2$), 4.13 (q, 2H, $CH_3\underline{CH_2}$), 4.30 (d, 2H, N-CH_2, J_{PH}=5.2), 7.6-8.1 (m, 15H, 3×Ph), 9.52 (s, 1H, NH).
^{13}C NMR ($CDCl_3$): δ=14.0 (s, $CH_2\underline{CH_3}$), 28.0 (s, Boc), 54.6 (d, N-CH_2, J_{PC}=12.5), 62.0 (s, O-CH_2), 81.3 (s, C_q, Boc), 119.2 (d, Ph, J_{PC}=101.8), 129.9 (d, Ph, J_{PC}=13.3), 135.2 (d, Ph, J_{PC}=11.3), 135.5 (d, Ph, J_{PC}=2.8), 155.2 (s, COOEt), 167.7 (s, CO, Boc).

3g. (R^1 = allyl) mp=174-174.5°C (dec)
^{1}H NMR ($CDCl_3$): δ=1.17 (s, 9H, Boc), 3.8-4.1 (broad signal, 1H, N-CH), 4.3-4.5 (broad signal, 1H, N-CH), 5.15-5.35 (m, 2H, CH_2=), 5.9-6.2 (m, 1H, CH=), 7.6-8.1 (m, 15H, 3×Ph), 9.80 (s, 1H, NH).
^{13}C NMR ($CDCl_3$): δ=28.0 (s, Boc), 56.2 (d, N-CH_2, J_{PC}=12.5), 80.9 (s, C_q, Boc), 119.7 (d, Ph, J_{PC}=101.5), 121.6 (s, CH=), 129.8 (d, Ph, J_{PC}=13.3), 131.0 (d, CH_2-$\underline{CH}$=, J_{PC}=3.0), 135.0 (d, Ph, J_{PC}=11.4), 135.3 (d, Ph, J_{PC}=2.6), 156.1 (s, CO).

3. Synthesis of hydrazines 5



Compound's number	R^1	R^2CO	Yield, %	Comments	mp, °C
5a	CH_3	CH_3CO	80		85.5-87.0
5b	Bzl	CH_3CO	82		50-52
5b	Bzl	CH_3CO	72	Et_3N/DMAP/AcCl	-

				as acylating mixture	
5b	Bzl	CH_3CO	95	Neat Ac_2O as acylating agent	-
5c	Bzl	PhCO	81		120-121
5d	Allyl	CH_3CO	86		53-54.5
5e	Allyl	COCH=CHCOOH	80		Obtained as a viscous oil
5f	CH_2COOEt	PhCO	70		67.5-69.5

Bzl = benzyl

Synthesis of 5a, 5b, 5c, 5d and 5f

Typical procedure is given below using **5d** as an example. 293 mg of **3g** (0.571 mmol) was dissolved in dichloromethane (1 mL). Under the stirring, 2M aqueous NaOH (2 mL) was added. The reaction can be monitored by TLC ($EtOH/CH_2Cl_2$ 1:7). After 3 min dichloromethane (10 mL) was added and the reaction mixture was saturated with solid NaCl. The aqueous layer was extracted with dichloromethane (10 mL, 3×6 mL). The combined organic extracts were dried over Na_2SO_4 and the solvent was evaporated. The obtained mixture of **4** and triphenylphosphine oxide was dissolved in pyridine (0.5 mL) and acetylchloride (50 μl, 1.2 eq) was added. The reaction was monitored by TLC (EtOAc/hexane 2:1). The reaction was essentially complete after 10 min. After 20 min the pyridine was evaporated in vacuo and the reaction mixture was partitioned between 0.4 M citric acid solution (3 mL) and ethyl ether (20 mL). The water phase was saturated with NaCl, extracted with ethyl ether (3×15 mL) and the combined organic extracts were dried over Na_2SO_4. After the solvent was evaporated the resulting mixture was chromatographed on silica column (EtOAc/hexane 1:2 as the mobile phase). 105 mg (86%) of **5d** was obtained, pure by TLC and NMR spectra.

The compound **5b** was also synthesized using another procedures for the acylation step. In both cases the equimolar mixture of $PhCH_2NHNHBoc$ and triphenylphosphine oxide was used as the starting material.

1). 0.362 g (0.722 mmol) of $PhCH_2NHNHBoc$ and Ph_3PO mixture was dissolved in the mixture of MeCN (1 mL) and Et_3N (1 mL). Then acetylchloride (54 μl, 1.05 eq) and DMAP (1.5 mg, 0.02 eq) was added. The reaction was monitored by TLC (EtOAc-hexane 2:1). As the reaction was not complete after 3 h, another 1 eq of acetylchloride was added. After 6.5 h the reaction was over and the solvents were evaporated. 0.4 M citric acid solution (2 mL) was added and the mixture was saturated with solid NaCl. The water phase was extracted with ethyl ether (6×10 mL) and the combined ether extracts were dried over Na_2SO_4. The solvent was evaporated and the product was chromatographed on silica (EtOAc/hexane 1:2). It was obtained 139 mg (72%) of pure **5b**.

2). 0.280 g (0.5585 mmol) of $PhCH_2NHNHBoc$ and Ph_3PO mixture was dissolved in ~0.5 mL of neat acetic anhydride under slight heating. The reaction was over after 5 min. The acetic anhydride was evaporated in vacuo and the resulting product was chromatographed on silica (EtOAc/hexane 1:2). It was obtained 141 mg (95%) of pure **5b**.

Synthesis of 5e.

The mixture of CH_2=CH-CH_2NHNHBoc and triphenylphosphine oxide was prepared from **3g** (285 mg, 0.555 mmol) as described above in the synthesis of **5a**. This mixture was dissolved in pyridine (0.5 mL) and maleic anhydride (57 mg, 1.05 eq) was added. The reaction was controlled by TLC (EtOAc/hexane 1:2) and it was over after 5 min. The pyridine was evaporated in vacuo. The resulting mixture was partitioned between 1 M $KHSO_4$ (6 mL) and ether (25 mL). The water phase was saturated with NaCl and extracted with ethyl ether (3×20 mL). The combined ether extracts were dried over Na_2SO_4 and the solvent was evaporated. For the purification the product was dissolved in dichloromethane (10 mL) and washed with 2 M NaOH solution (8 mL). The water phase was extracted with dichloromethane (3×10 mL). Then $KHSO_4$ (6 g) was added to the aqueous solution and the latter was extracted with ether (3×30 mL). The combined ether solutions were dried over Na_2SO_4 and the evaporation of ether gave 121 mg (80%) of **5e**, pure by TLC.

Spectral data

5a. ($R^1 = R^2 = CH_3$), white crystals

^{1}H NMR ($CDCl_3$): δ=1.49 (s, 9H, Boc), 2.08/2.15 (two signals, 3H, CH_3CO), 2.59/3.30 (two signals, 3H, N-CH_3), 7.09/7.46 (two broad signals, 1H, NH).

^{13}C NMR ($CDCl_3$): δ=20.3 (s, $\underline{CH_3}CO$), 28.3 (s, Boc), 35.7 (s, N-CH_3), 82.0 (s, C_q, Boc), 154.4 (s, CO, Boc), 173.8 (s, $CH_3\underline{CO}$).

5b. ($R^1 = PhCH_2$, $R^2 = CH_3$), white crystals

^{1}H NMR ($CDCl_3$): δ=1.42 (s, 9H, Boc), 2.10 (s, 3H, CH_3CO), 3.9-4.5 (broad signal, 1H, $PhCH_2$), 4.8-5.5 (broad signal, 1H, $PhCH_2$), 6.84 (s, 1H, NH), 7.2-7.5 (m, 5H, Ph).

^{13}C NMR ($CDCl_3$): δ=20.6 (s, $\underline{CH_3}CO$), 28.2 (s, Boc), 50.6 (s, N-CH_2), 82.0 (s, C_q, Boc), 127.9, 128.7, 129.1, 135.9 (Ph), 154.2 (s, CO, Boc), 173.4 (s, $CH_3\underline{CO}$).

5c. ($R^1 = PhCH_2$, $R^2 = Ph$), white crystals

^{1}H NMR ($CDCl_3$): δ=1.30 (s, 9H, Boc), 4.2-5.4 (broad signals, 2H, $PhCH_2$), 6.62 (s, 1H, NH), 7.2-7.6 (2×m, 10H, 2×Ph).

^{13}C NMR ($CDCl_3$): δ=28.0 (s, Boc), 52.3 (s, broad, N-CH_2), 81.8 (s, C_q, Boc), 127.4, 127.95, 128.00, 0, 128.8, 129.0, 130.2, 135.0, 135.8 (Ph), 154.0 (s, CO, Boc), 172.8 (s, Ph$\underline{CO}$).

5d. ($R^1 = CH_2$=CH-CH_2, $R^2 = CH_3$), white crystals

^{1}H NMR ($CDCl_3$): δ=1.48 (s, 9H, Boc), 2.11 (s, 3H, CH_3CO), 3.8-4.6 (broad signals, 2H, N-CH_2), 5.15-5.30 (m, 2H, CH_2=), 5.70-5.90 (m, 1H, CH=), 7.14 (s, 1H, NH).

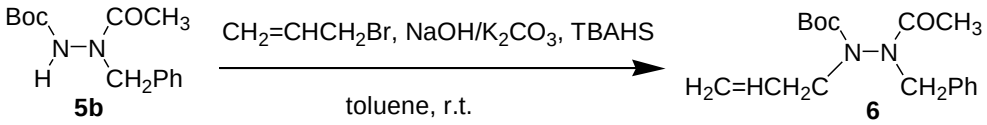
^{13}C NMR ($CDCl_3$): δ=20.6 (s, $\underline{CH_3}CO$), 28.2 (s, Boc), 50.1 (s, N-CH_2), 82.0 (s, C_q, Boc), 119.2 (s, CH=$\underline{CH_2}$), 132.1 (s, $\underline{CH}$=CH_2), 154.4 (s, CO, Boc), 173.3 (s, $CH_3\underline{CO}$).

5e. ($R^1 = CH_2$=CH-CH_2, R^2 = -CH=CH-COOH), obtained as yellowish viscous oil

^{1}H NMR ($CDCl_3$): δ=1.47 (s, 9H, Boc), 3.9-4.6 (broad signal, 2H, N-CH_2), 5.25-5.40 (m, 2H, CH_2=), 5.70-6.00 (m, 1H, $\underline{CH}$=CH_2), 6.25 (d, 1H, CH=CH, J_{HH}=12.4), 6.71 (d, 1H, CH=CH, J_{HH}=12.4).
^{13}C NMR ($CDCl_3$): δ=28.2 (s, Boc), 51.1 (N-CH_2), 83.3 (s, C_q, Boc), 120.9 (s, CH=$\underline{CH_2}$), 130.4 (s, $\underline{CH}$=CH_2), 131.1, 132.1 (CH=CH), 154.4 (s, CO, Boc), 166.1 (s, COOH), 169.2 (s, CO, COCH=CH).

5f. (R^1 = CH_2COOEt, R^2 = Ph), white crystals
^{1}H NMR ($CDCl_3$): δ=1.28 (t, 3H, $\underline{CH_3}CH_2$, J_{HH}=7.2), 1.35 (s, 9H, Boc), 4.19 (q, 2H, $\underline{CH_2}CH_3$, J_{HH}=7.2), 4.3-4.8 (broad signal, 2H, N-CH_2), 7.3-7.6 (m, 5H, Ph).
^{13}C NMR ($CDCl_3$): δ=14.1 (s, $\underline{CH_3}CH_2$), 28.1 (s, Boc), 49.7 (s, N-CH_2), 81.9 (s, C_q, Boc), 127.4, 128.0, 130.5, 134.1 (Ph), 153.9 (s, CO, Boc), 169.3 (s, COOEt), 173.2 (s, COPh).

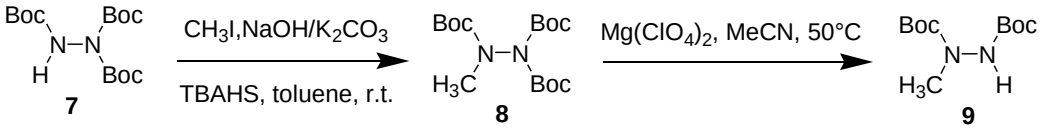
4. Synthesis of 1-Boc-1-allyl-2-acetyl-2-benzylhydrazine 6



110 mg (0.416 mmol) of **5b** was dissolved in toluene (0.5 mL). Under the stirring fine-grained NaOH (58 mg, 1.5 mmol), K_2CO_3 (117 mg, 0.85 mmol) and TBAHS (14 mg, 0.041 mmol) were added, followed by allylbromide (40 µl, 1.1 eq). The reaction was monitored by TLC (EtOAc/hexane 3:1). After 30 min the reaction was complete and the resulting mixture was partitioned between ethyl ether (20 mL) and water (6 mL). The ether layer was washed to neutral with brine (4×2 mL) and dried over $MgSO_4$. After the evaporation of solvent 123 mg (97%) of white solid **6** was obtained, pure by TLC.

6. (R^1 = $PhCH_2$, R^2 = CH_3, R^3 = CH_2CH=CH_2), mp=62.5-64°C
^{1}H NMR ($CDCl_3$): δ=1.28/1.36/1.47 (s, 9H, Boc), 2.05/2.19/2.21 (s, 3H, CH_3CO), 3.6-3.9 (broad signal, 1H, N-$\underline{CH_2}$CH=CH_2), 4.0-4.3 (broad signal, 1H, N-$\underline{CH_2}$CH=CH_2), 4.5-4.8 (broad signal, 2H, $PhCH_2$), 4.9-5.3 (broad signal, 2H, $\underline{CH_2}$=CH), 5.6-5.9 (broad signal, 1H, $\underline{CH}$=CH_2), 7.31 (m, 5H, Ph).
^{13}C NMR ($CDCl_3$): δ=20.7 (s, $\underline{CH_3}CO$), 28.1/29.7 (s, Boc), 50.6 (s, N-CH_2 of allyl), 52.5 (s, broad, N-CH_2Ph), 82.0 (s, C_q, Boc), 119.4 (s, CH=$\underline{CH_2}$), 127.7, 128.5, 129.7 (Ph), 132.2 (s, $\underline{CH}$=CH_2), 136.6 (s, Ph), 153.9 (s, CO, Boc), 173.5 (s, $CH_3\underline{CO}$).

5. Synthesis of 1,2-diBoc-1-methylhydrazine 9

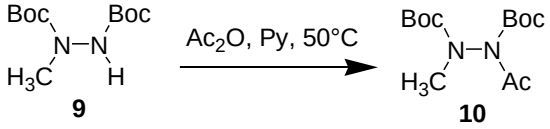


The synthesis of **8** from **7** was accomplished using previously described procedure (Mäeorg, U., Grehn, L., Ragnarsson, U. *Angew. Chem.*, 1996, 108, 2802-2803. *Angew. Chem. Int. Ed. Engl.*, 1996, 35, 22, 2626-2627).

1,1,2-triBochydrazine **7** (1.834 g, 5.518 mmol) was dissolved in toluene (5.5 mL). Under the stirring fine-grained NaOH (0.773 g, 19.3 mmol), K_2CO_3 (1.547 g, 11.2 mmol) and TBAHS (0.188 g, 0.554 mmol) were added, followed by MeI (0.41 mL, 1.2 eq). The reaction was monitored by TLC (EtOAc/light petroleum 1:3). After 45 min the most of the starting material was consumed and 1.5 eq of MeI was added to accelerate the process. After ~1h the reaction mixture was partitioned between ether (40 mL) and water (15 mL). The ether layer was washed by water (7 mL) and the water phase was extracted with ether (3×15 mL). The combined extracts were washed with water (10 mL) and then to neutral with brine. After drying over Na_2SO_4 and evaporation of solvent 1.906 g of colourless oil **8** was obtained (96%, contained trace amount of side product).
For the second step (synthesis of **9**) 1.906 g of **8** was dissolved in MeCN (13.7 mL). Under the stirring and flushing with argon the reaction flask was placed into oil bath, which was preceedingly heated to 50°C. Then $Mg(ClO_4)_2$ (248 mg, 1.11 mmol) was added. The reaction was monitored by TLC (EtOAc/light petroleum 1:3) and after 10 min most of the starting material was consumed. After 30 min the reaction mixture was partitioned between 0.2M citric acid solution (25 mL) and ether (10 mL). The water phase was extracted with ether (4×25 mL), the combined extracts were washed to neutral with brine (2×15 mL, 2×10 mL) and dried over Na_2SO_4. After the evaporation of solvent 1.222 g of white solid was obtained, containing two small impurities (TLC). The product was purified by column chromatography on silica (EtOAc/light petroleum/chloroform 1:4:1). It was obtained 1.045 g of pure crystalline solid **9** with 77% yield over two steps (including chromatography).

9. 1,2-diBoc-1-methylhydrazine, mp=55-56°C
^{1}H NMR ($CDCl_3$): δ=1.46, 1.48 (2 signals, 18H, 2×Boc), 3.11 (s, 3H, CH_3), 6.47/6.26 (two broad signals, 1H, NH).
^{13}C TMR ($CDCl_3$): δ=28.3 (2×Boc), 37.8 (CH_3), 81.1, 81.2 (two signals, C_q, 2×Boc), 155.2, 155.8 (two signals, 2×CO).

6. Synthesis of 1,2-diBoc-1-acetyl-2-methylhydrazine 10



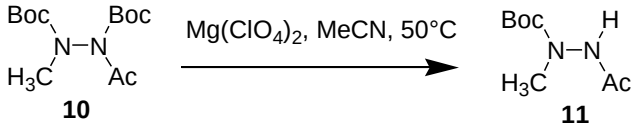
9 **10**

To **9** (1.045 g, 4.25 mmol) the acetic anhydride (7 mL, 74 mmol) and pyridine (3 mL, 37 mmol) were added, followed by DMAP (45 mg, 0.08 eq). The reaction was monitored by TLC (EtOAc/light petroleum/chloroform 1:4:1). The mixture was heated at 45-50°C for 1.5 days and the total reaction time was 5.5 days. The reaction mixture was diluted with ether (30 mL), poured into 1 M $NaHCO_3$ solution (100 mL) and stirred for 2 h. Then 1 M $NaHCO_3$ (25 mL) was added, the ether layer was isolated and washed with 1 M $NaHCO_3$ solution (45 mL). The water phase was extracted with ether (2×25 mL, 3×20 mL). The combined ether extracts were washed with 1 M $NaHCO_3$ solution (20 mL), then with 0.2 M citric acid solution (5×20 mL) and to neutral with brine (2×20 mL, 2×10 mL). The ether solution was dried over Na_2SO_4

and the solvent was evaporated. It was obtained 1.051 g of yellowish oil, pure by TLC. The product was purified by filter chromatography (EtOAc/light petroleum 1:4), furnishing 1.030 g (84%) of colourless viscous oily **10**.

10. 1,2-diBoc-1-acetyl-2-methylhydrazine
^{1}H NMR ($CDCl_3$): δ=1.42/1.49/1.52/1.54 (four signals together 18H, 2×Boc), 2.45/2.48 (two signals together 3H, CH_3CO), 3.06/3.08 (two signals together 3H, CH_3).
^{13}C NMR ($CDCl_3$): δ=25.1/25.3 (two signals, $\underline{C}H_3CO$), 28.0/28.2/28.3 (three signals, CH_3, 2×Boc), 35.6/37.0 (two signals, N-CH_3), 81.1/81.5, 83.9/83.9 (four signals, C_q, 2×Boc), 151.5, 151.6, 153.9 (three signals, CO, 2×Boc), 169.7/169.8 (two signals, CO, $CH_3\underline{CO}$).

7. Synthesis of 1-Boc-1-methyl-2-acetylhydrazine 11



10 **11**

104 mg (0.360 mmol) of **10** was dissolved in MeCN (1 mL). Under the stirring and flushing with argon the reaction flask was placed into oil bath, which was preceedingly heated to 50°C. Then $Mg(ClO_4)_2$ (16 mg, 0.2 eq) was added and the reaction was monitored by TLC (EtOAc/light petroleum 1:4 and 1:1). Most of the starting material was consumed after 15 min and after 1 h the additional amount of $Mg(ClO_4)_2$ (0.1 eq) was added to accelerate the reaction. After 1.5 h 0.2 M citric acid solution (5 mL), saturated NaCl solution (5 mL) and ether (10 mL) were added to the reaction mixture. The water phase was extracted with ethyl ether (10 mL, 2×7 mL), the combined ether solutions were washed to neutral with brine (4×5 mL) and dried over Na_2SO_4. The evaporation of solvent yielded 65 mg (96%) of colourless oily **11**. It contained miserable amount of impurity, which was identified by ^{1}H NMR spectra as *tert*-butyl derivative of product (it was formed due to the side reaction).

11. 1-Boc-1-methyl-2-acetylhydrazine
^{1}H NMR ($CDCl_3$): δ=1.46/1.48 (two signals, 9H, Boc), 1.97 (s, 3H, CH_3CO), 3.12/3.15 (two signals, 3H, N-CH_3), 7.81, 8.4-8.5 (two broad signals, 1H, NH).
^{13}C NMR ($CDCl_3$): δ=20.7/18.8 (two signals, $\underline{CH_3}CO$), 28.2/29.7 (two signals, CH_3, Boc), 37.7/38.3 (two signals, N-CH_3), 81.4/82.1 (two signals, C_q, Boc), 155.7 (CO, Boc), 169.3, 175.5 (two signals, $CH_3\underline{CO}$).