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Chemoselective Substitution in 4-Toluenesulfonamides and Carbamates by Di-*tert*-butyl Dicarbonate in the Presence of 4-Dimethylaminopyridine

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Three novel derivatives of 1,2-ethylenediamine **1**, **3** and **5** with 4-toluenesulfonyl and/or carbamate moieties have been prepared and submitted to reaction with a limiting amount of di-*tert*-butyl dicarbonate in the presence of catalytic amounts of 4-dimethylaminopyridine. All the reagents underwent completely selective substitution under these conditions and gave rise to the products **2a**, **4** and **6** in 93–100% yields and the results were rationalized in terms of higher NH-acidity at the reaction sites. The products might be useful in synthesis of substituted 1,2-ethylenediamines. A similar approach might be applicable to many other diamines.

Flynn, Zelle and Grieco first demonstrated that secondary amides and lactams undergo facile reaction with di-*tert*-butyl dicarbonate (Boc_2O) in the presence of 4-dimethylaminopyridine (DMAP) to give derivatives of Boc-*amide* type, i.e. acylcarbamates, and subsequently cleaved the products regioselectively to carbamates.[1] Independently of these authors we used similar conditions to introduce Boc-groups into *N*-heterocycles.[2] DMAP thereby functions as a catalyst,[3] in the absence of which generally no reaction takes place. Since Boc is an important NH-protecting group, originally introduced and exclusively used in peptide synthesis but more recently also applied in various other synthetic contexts,[4] several other types of NH-compound have subsequently been investigated and shown to undergo this substitution reaction.[5] Among these are sulfonamides and carbamates,[6] sulfonamides thereby giving rise to sulfonylcarbamates and carbamates to imidodicarbonates. Even nitridotricarbonic acid tri-*tert*-butyl ester can be synthesized by this method.[7]

Obviously this novel chemistry is of potential synthetic interest and we would therefore like to extend its scope to compounds derived from substrates containing two or more amino groups. To this end we have prepared a number of simple 4-toluenesulfonyl and carbamate derivatives of 1,2-ethylenediamine and used them as model substances in reactions with a limiting amount of Boc_2O to explore chemoselective substitution with respect to these moieties. To the best of our knowledge this topic has not been addressed previously in the chemical literature.

Results and discussion

Starting from 1-tosyl-1,2-ethylenediamine,[8] using a well-known method we first converted it into the corresponding novel benzyl carbamate (**1**) and submitted it in essence to our original exhaustive *tert*-butoxycarbonylation procedure involving *catalytic*, not stoichiometric,[1] amounts of DMAP, no extra base and only a modest excess of Boc_2O in acetonitrile.[2,6] The product **2a** was thereby obtained in quantitative yield (Scheme 1). Although benzyl carbamates are known to undergo substitution on the NH,[6] it is remarkable that in this case no impurity could be detected due to the small excess of Boc_2O applied. On the other hand, this fact may also have a negative side insofar as a stronger base and/or a more reactive acylating agent is required to convert this otherwise attractive, orthogonally triply protected intermediate into other, more highly substituted derivatives. Until very recently the tosyl group would not have been considered attractive in this context owing to the severe conditions required for its subsequent removal, but we have come up with a reductive method that works exceedingly well when a Boc-group is present on the same nitrogen.[9]

Bordwell *et al.* recently correlated substrate acidities in DMSO and reactivity towards Boc_2O–DMAP in acetonitrile within a series of cyclic nitrogen compounds.[10] The authors thus assumed acidities in the latter

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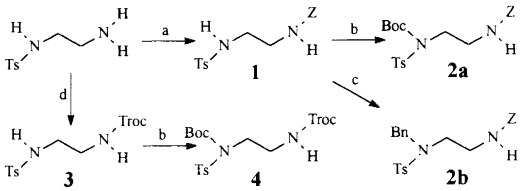
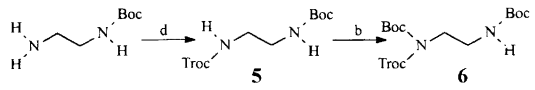


Scheme 1. Reagents and conditions: (a) Z-Cl, pyridine (91%); (b) Boc_2O, DMAP, MeCN (100%); (c) BnOH, Ph_3P, diethyl azodicarboxylate, THF (60%); (d) Troc-Cl, pyridine (77%). Abbreviations: Boc, *tert*-butoxycarbonyl; DMAP, 4-dimethylaminopyridine; Troc, trichloroethoxycarbonyl; Ts, tosyl; Z, benzyloxycarbonyl.

solvent to be about 10 pK_a units higher than in DMSO.[11] Provided available pK_a-data related to NH acidity in DMSO can also be applied in this case, we find that benzenesulfonamide (16.1) is 8.5 units more acidic than ethyl carbamate (24.6).[11] Further assuming on the one hand benzene- and 4-toluenesulfonamides, and on the other methyl and ethyl carbamates to have roughly similar acidities and benzyl carbamate to be less than 0.5 units more acidic than the latter,[12] one is led to conclude that the acidities of the two groups in **1** may differ by up to 8 units. A direct comparison between the acidities of benzyl tosylcarbamate and dibenzyl imidodicarbonate in DMSO indicates a difference of 6.7 units[13] which is considered to be high enough to rationalize the results in the synthesis of **2a**.

At this stage we therefore considered it logical to prepare a new derivative of 1,2-ethylenediamine with closer pK_a values than those of **1** and react it similarly. We next chose to make **3** for this purpose, since it should have a smaller (by 1.3–1.8 units) difference in pK_a than **1** and consequently an estimated difference in acidity between its Ts- and Troc-NH of 4.9–5.4 units.[12,13] Compound **4** was also obtained from **3** in quantitative yield but in this case we noticed a characteristic difference insofar as an excess of Boc_2O had to be avoided to prevent substitution in the carbamate moiety.

Since the Troc-group gives rise to the most acidic, practically useful carbamate we know, we chose not to make and explore any further mixed sulfonamide/carbamate derivatives. Instead, starting from the known Boc-derivative,[14] we decided to make **5** (Scheme 2) with two *carbamate* groups with a much smaller estimated difference in pK_a values (1.7–3.1). When this compound was reacted with Boc_2O–DMAP under standard conditions, a surprisingly pure product **6** was obtained in nearly quantitative yield. In this case the high selectivity may also be due to steric reasons, since derivatives with two Boc-groups on the same nitrogen are bulky and their formation is resisted by additional bulky groups.[15,16] Although high NH acidity and, as a consequence, facilitated anion formation should be essential for reactivity with a hypothetical, positively charged Boc-DMAP complex, in species with two functions of comparable pK_a in the presence of a limiting component, steric factors may ultimately control product formation.



Scheme 2. For reagents, conditions and abbreviations, see legend to Scheme 1. (b) (93%); (d) (85%).

The three triply protected products **2a**, **4** and **6** should be applicable in the synthesis of derivatives of 1,2-ethylenediamine by various alkylation/acylation procedures. The three protecting groups in **2a** appear to be orthogonal and **2a** should, in principle, allow tetrasubstituted derivatives to be made by a stepwise procedure, whereas for **4** at this stage problems are foreseen in the selective cleavage of tosyl and Troc.[9] Compound **6** is probably inferior to **2a** with respect to both selectivity and reactivity.

This approach to selectively protected derivatives of 1,2-ethylenediamine should be applicable to many other diamines, too.

A final comment concerns compounds **1** and **2b**. Some years ago Weinreb *et al.* demonstrated that Ts-NH-Me could be reacted with alcohols under Mitsunobu conditions to give the corresponding sulfonamides.[17] All yields reported were, with one exception, in the range 50–62%. When we reacted benzyl alcohol with **1** we obtained **2b** in 60% yield after chromatography. No interference by the benzyl carbamate moiety could be detected in this case.

Experimental

All reaction vessels were flame-dried immediately before use and the subsequent reactions performed under argon. Pyridine and acetonitrile were of analytical grade and dried over molecular sieves (4 Å). Commercial Ts-Cl was purified according to a standard procedure whereas commercial Z-Cl and Troc-Cl were used as such. Preparative chromatography was carried out on silica (Merck Kieselgel 60, 70–230 mesh) and TLC analyses on 0.25 mm thick precoated silica plates (Merck Fertigplatten Kieselgel $60F_{254}$) with the mobile phases toluene–acetonitrile 2:1 (A) and dichloromethane–diethyl ether 19:1 (B). TLC spots were visualized, after brief heating, by exposure to Cl_2 followed by dicarboxidine spray (violet–blue spots). ^1H and ^{13}C NMR spectra were recorded for ≈5% solutions in $CDCl_3$ at 25 °C on a JEOL JMN EX 400 spectrometer. All shifts are given in ppm using $\delta_H(TMS)=0$ and δ_C $(CDCl_3)=77.02$, respectively, as references. Assignments were made by comparison of chemical shifts and peak multiplicities, and in the case of more than one conformer, only data for the major one is given. All melting points were measured on a Gallenkamp melting point apparatus and are uncorrected. All analytical samples were chromatographically pure in both systems unless otherwise stated. Elemental analyses of crystalline products were carried out by Astra Draco AB, Lund, Sweden.

N-*Tosyl*-N′-*benzyloxycarbonyl-1,2-ethylenediamine* (**1**). 1-Ts-1,2-ethylenediamine[8] (2.15 g, 10.0 mmol) in pyridine (30 ml) was chilled to −30 °C and Z-Cl (2.04 g, ⩽12 mmol) was introduced dropwise with rapid stirring over 30 min. After a further 1 h the cooling was interrupted and the pale yellow mixture was left with stirring overnight. Most of the solvent was stripped off at reduced pressure and the remaining oil was redissolved in dichloromethane (100 ml) and the solution was partitioned between diethyl ether (200 ml) and 1 M $KHSO_4$ (100 ml). The organic phase was washed consecutively with 1 M $KHSO_4$, 1 M $NaHCO_3$ and sat. NaCl (3 × 50 ml each) and dried ($MgSO_4$) in the presence of decolourizing carbon. Removal of the solvent left a white solid which was thoroughly triturated with cold dry diethyl ether. The insoluble white powder was collected by filtration, rinsed repeatedly with more cold solvent and dried. The yield of crude but chromatographically pure product was 3.18 g (91%). Recrystallization from diethyl ether–dichlorometane 2:1 (30 ml g^{-1}, decolourizing carbon) afforded an analytical specimen as white shiny crystals; m.p. 109.5–110.5 °C. ^{1}H NMR: δ_H 2.39 (s, 3 H, Ts-CH_3), 3.03 (perturbed q, 2 H, CH_2NHZ), 3.26 (perturbed q, 2 H, CH_2NHTs), 5.05 (s, 2 H, Z-CH_2), 5.31 (perturbed t, 1 H, NHZ), 5.42 (perturbed t, 1 H, NHTs), 7.24–7.35 (complex signal, 7 H, Ph-H + $Ts_{3,5}$), 7.71 (d, J = 8.1 Hz, 2 H, $Ts_{2,6}$). ^{13}C NMR: δ_C 21.47 (Ts-CH_3), 40.71 (CH_2NHZ), 43.24 (CH_2NHTs), 66.85 (Z-CH_2), 127.00, 127.99, 128.12, 128.50, 129.75, 136.25, 136.66 (Ar-C), 143.50 (Ts-C_1), 156.87 (Z-CO). Found: C, 58.60; H, 5.79; N, 8.04. $C_{17}H_{20}N_2O_4S$ (348.4) requires C, 58.83; H, 5.91; N, 8.12.

N-tert-*Butoxycarbonyl*-N-*tosyl*-N′-*benzyloxycarbonyl-1,2-ethylenediamine* (**2a**). Recrystallized **1** (350 mg, 1.00 mmol) and recrystallized DMAP (6 mg, 0.05 mmol) were dissolved in MeCN (12 ml), chilled to −15 °C and treated with Boc_2O (240 mg, 1.10 mmol) in MeCN (6 ml) dropwise over 30 min. The clear solution was stirred for 1 h at −10 °C and 4 h at rt, whereupon TLC (A) indicated that all starting material had been consumed. The light brown mixture was partitioned between diethyl ether (200 ml) and 0.2 M citric acid (80 ml), and the organic extract was washed successively with citric acid, 1 M $NaHCO_3$ and sat. NaCl (3 × 40 ml each) and dried ($MgSO_4$). Removal of the solvent left, after drying *in vacuo*, 450 mg (quant.) of a colourless viscous oil, which solidified with prolonged standing in the cold. The analytical specimen was obtained by recrystallization from light petroleum (20 ml g^{-1}, −20 °C) as white tiny crystals; m.p. 76–77 °C. ^{1}H NMR: δ_H 1.33 (s, 9 H, Boc-CH_3), 2.42 (s, 3 H, Ts-CH_3), 3.54 (q, 2 H, CH_2NHZ), 3.99 [t, 2 H, CH_2N(Boc)Ts], 5.12 (s, 2 H, Z-CH_2), 5.21 (perturbed t, 1 H, NH), 7.26–7.35 (complex signal, 7 H, Ph-H + $Ts_{3,5}$), 7.77 (d, J = 8.3 Hz, 2 H, $Ts_{2,6}$). ^{13}C NMR: δ_C 21.59 (Ts-CH_3), 27.80 (Boc), 41.17 (CH_2NHZ), 45.98 [CH_2N(Boc)Ts], 66.66 (Z-CH_2), 84.73 (Boc-C_q), 127.92, 127.99, 128.02, 128.44, 129.30, 136.59, 136.96 (Ar-C), 144.32 (Ts-C_1), 151.11 (Boc-CO), 156.41 (Z-CO). Found: C, 58.9; H, 6.4; N, 6.2. $C_{22}H_{28}N_2O_6S$ (448.5) requires C, 58.91; H, 6.29 N, 6.25.

N-*Benzyl*-N-*tosyl*-N′-*benzyloxycarbonyl-1,2-ethylenediamine* (**2b**). Recrystallized **1** (699 mg, 2.00 mmol) and benzyl alcohol (220 µl, 2.20 mmol) were dissolved in THF (6 ml, freshly distilled from sodium–benzophenone) and the solution was chilled to −30 °C under argon. Recrystallized Ph_3P (630 mg, 2.40 mmol) was introduced in small portions with stirring and when all had dissolved, diethyl azodicarboxylate (405 µl, 2.60 mmol) was added dropwise with rapid stirring over 30 min. After 1 h at this temperature and 16 h at rt, maintaining dry conditions, the solvent was evaporated off and the resulting light brown oil was chromatographed on silica using CH_2Cl_2–Et_2O 20:1 as the eluent. A central fraction consisting of pure **2b** was obtained as a pale yellow oil weighing 550 mg (60%), which could be crystallized from hexane–Et_2O 3:1 (80 ml g^{-1}; decolourizing carbon) to give a microcrystalline fluffy product; m.p. 65.5–66 °C. ^{1}H NMR: δ_H 2.42 (s, 3 H, Ts-CH_3), 3.11 (q, 2 H, CH_2NHZ), 3.20 [t, 2 H, CH_2N(Bn)Ts], 4.30 (s, 2 H, N-CH_2), 4.92 (perturbed t, 2 H, NH), 5.01 (s, 2 H, Z-CH_2), 7.25–7.37 (complex signal, 7 H, Ph-H + $Ts_{3,5}$), 7.71 (d, J = 8.2 Hz, 2 H, $Ts_{2,6}$). ^{13}C NMR: δ_C 21.51 (Ts-CH_3), 39.78 (CH_2NHZ), 47.97 [CH_2N(Bn)Ts], 53.28 (N-CH_2), 66.56 (Z-CH_2), 127.24, 127.97, 128.02, 128.06, 128.39, 128.47, 128.77, 129.85, 136.04, 136.08, 136.52 (Ar-C), 143.63 (Ts-C_1), 156.22 (Z-CO). Found: C, 65.97; H, 6.12; N, 6.46. $C_{24}H_{26}N_2O_4S$ (438.6) requires C, 65.73; H, 5.98; N, 6.39.

N-*Tosyl*-N′-*trichloroethoxycarbonyl-1,2-ethylenediamine* (**3**). Prepared from 1-Ts-1,2-ethylenediamine[8] and Troc-Cl on a 10 mmol scale essentially as described for **1**. The yield of crude, chromatographically pure (A) **3**, after rinsing with cold ether, was 3.01 g (77%). Crystallization from dichloromethane–diethyl ether 1:9 (15 ml g^{-1}, decolourizing carbon) gave an analytical sample as a white microcrystalline solid after seeding and cooling to −20 °C; m.p. 102–103 °C. ^{1}H NMR: δ_H 2.43 (s, 3 H, Ts-CH_3), 3.10 (perturbed q, 2 H, CH_2NHTroc), 3.36 (perturbed q, 2 H, CH_2NHTs), 4.69 (s, 2 H, Troc-CH_2), 5.38 (t, 1 H, NHTroc), 5.60 (perturbed t, 1 H, NHTs), 7.31, 7.75 (AB system, 4 H, J = 8.2 Hz, Ts). ^{13}C NMR: δ_C 21.53 (Ts-CH_3), 40.97 (CH_2NHTroc), 42.95 (CH_2NHTs), 74.55 (Troc-CH_2), 95.40 (CCl_3), 127.05 ($Ts_{3,5}$), 129.86 ($Ts_{2,6}$), 136.45 (Ts_4), 143.75 (Ts_1), 155.05 (Troc-CO). Found: C, 37.28; H, 3.94; N, 7.30. $C_{12}H_{15}Cl_3N_2O_4S$ (389.6) requires C, 37.00; H, 3.88; N, 7.19.

N-tert-*Butoxycarbonyl*-N-*tosyl*-N′-*trichloroethoxycarbonyl-1,2-ethylenediamine* (**4**). Synthesized from **3** by DMAP-catalyzed reaction with Boc_2O as described above for the corresponding Z-derivative **2a**. As this substrate was more prone to excessive reaction on the

remaining Troc-NH, only a 1% excess of Boc_2O was used in this case. An analogous work-up provided crude **4** in quantitative yield. Recrystallization from diethyl ether (20 ml g^{-1}, −20 °C) afforded white fluffy crystals; m.p. 132–132.5 °C. ^1H NMR: δ_H 1.35 (s, 9 H, Boc-CH_3), 2.44 (s, 3 H, Ts-CH_3), 3.58 (q, 2 H, CH_2NHTroc), 4.02 [t, 2 H, CH_2N(Boc)Ts], 4.74 (s, 2 H, Troc-CH_2), 5.46 (perturbed t, 1 H, NH), 7.31, 7.78 (AB system, J= 8.3 Hz, 4 H, Ts-H). ^{13}C NMR: δ_C 21.62 (Ts-CH_3), 27.85 (Boc), 41.43 (CH_2NHTroc), 45.74 [CH_2N(Boc)Ts], 74.59 (Troc-CH_2), 84.99 (Boc-C_q), 95.51 (CCl_3), 127.96 ($Ts_{3,5}$), 129.36 ($Ts_{2,6}$), 136.86 (Ts_4), 144.47 (Ts_1), 151.17 (Boc-CO), 154.67 (Troc-CO). Found: C, 41.61; H, 4.75; N, 5.67. $C_{17}H_{23}Cl_3N_2O_6S$ (489.8) requires C, 41.69; H, 4.73; N, 5.72.

N-*Trichloroethoxycarbonyl*-N′-tert-*butoxycarbonyl-1,2-ethylenediamine* (**5**). Obtained from 1-Boc-1,2-ethylenediamine[14] and Troc-Cl in pyridine by the procedure described for **3**. Conventional work-up furnished the chromatographically pure product **5** as a white solid in 85% yield in a 4 mmol run. Recrystallization from diethyl ether–light petroleum 1:4 (20 ml g^{-1}, decolourizing carbon) afforded an analytical specimen as white tiny crystals; m.p. 100.5–101.5 °C. ^1H NMR: δ_H 1.44 (s, 9 H, Boc-CH_3), 3.29 (perturbed q, 2 H, CH_2NHBoc), 3.36 (perturbed q, 2 H, CH_2NHTroc), 4.72 (s, 2 H, Troc-CH_2), 4.92 (br signal, 1 H, NHBoc), 5.57 (br signal, 1 H, NHTroc). ^{13}C NMR: δ_C 28.35 (Boc-CH_3), 40.32 (CH_2NHBoc), 41.78 (CH_2NHTroc), 74.52 (Troc-CH_2), 79.78 (Boc-C_q), 95.54 (CCl_3), 155.02 (Boc-CO), 156.50 (Troc-CO). Found: C, 36.24; H, 5.19; N, 8.22. $C_{10}H_{17}Cl_3N_2O_4$ (335.6) requires C, 35.79; H, 5.11; N, 8.35.

N,N′ - *Di*(tert - *butoxycarbonyl*) - N - *trichloroethoxycarbonyl-1,2-ethylenediamine* (**6**). This compound was made from **5** with 1.1 equiv. of Boc_2O as described for **2a** and obtained as an almost colourless oil after the usual extractive work-up. The yield was 93% in a 0.6 mmol run. Chromatography on silica using CH_2Cl_2–Et_2O 20:1 as the eluent furnished very pure material as a pale yellow oil which failed to crystallize even after prolonged standing in the cold. ^1H NMR: δ_H 1.43 (s, 9 H, Boc-CH_3), 1.55 (s, 9 H, NH-Boc-CH_3), 3.40 (q, 2 H, CH_2NHBoc), 3.87 [t, 2 H, CH_2N(Troc)Boc], 4.81 (br signal, ≈1 H, NH), 4.83 (s, 2 H, Troc- CH_2). ^{13}C NMR: δ_C 27.94 (Boc-CH_3), 28.37 (NH-Boc-CH_3), 39.78 (CH_2NHBoc), 46.09 [CH_2N(Troc)Boc], 75.75 (Troc-CH_2), 79.33 (NH-Boc-C_q), 84.11 (Boc-C_q), 94.49 (CCl_3), 151.78 (Boc-CO), 152.00 (Boc-CO), 156.80 (Troc-CO).

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