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Efficient and selective deprotection method for *N*-protected 2(*3H*)-benzoxazolones and 2(*3H*)-benzothiazolones

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Abstract—Cyclic carbamate flanked with heterocyclic or aliphatic moieties are frequently used in medicinal chemistry. The synthesis of derivatives bearing a free NH often requires the use of a protection method. A literature search reveals very few protection/deprotection methods for cyclic carbamates. In this paper, we described different methods applicable to 2(*3H*)-benzoxazolone and 2(*3H*)-benzothiazolone.

1. Introduction

2(*3H*)-Benzoxazolones and 2(*3H*)-benzothiazolones derivatives have attracted considerable attention as a result of their medicinal properties. Several potentially useful drugs and pharmacological tools based on these pharmacophores have been developed in recent years.[1–6] *N*-methyl-2(*3H*)-benzoxazolones and 2(*3H*)-benzothiazolones have been largely used in medicinal chemistry, but surprisingly their N–H homologues are less accessible although in many cases, a free N(3)–H group is an essential structural requirement for activity and receptor selectivity of these 2(*3H*)-benzazolones derivatives. Moreover, the NH heterocycle can serve as a pivotal structure for the constitution of a library N-derivatized analogues.

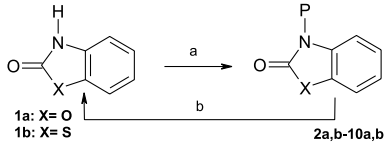
Indeed, in many cases encountered in our own research, reactions that are successful in the N-methyl series cannot be applied in the N–H series: C(6)-formylation,[7,8] C(6)-tributyltin derivatization, photohalogenation,[9] crotonisation, etc.[10,11] Obviously, the use of N-protected 2(*3H*)-benzazolones are in order for the success of these reactions. However, close inspection of the literature reveals very few indications concerning protecting groups of cyclic carbamates that can be easily introduced and subsequently smoothly removed. Indeed, benzyl protecting group[12] was found in this series of heterocycle but reaction of formylation, acylation or the cleavage of N-benzyle derivative with NBS, AIBN could induced secondary products. Therefore, in an effort to fill this gap, we explored various protecting groups and examined their ease of deprotection.

2. Results and discussion

2.1. Protection/deprotection of cyclic carbamate on phenyl ring (1a,b) via different methods

The compounds **2a**,**b**–**10a**,**b**, in Scheme 1, were synthetized by methods A or B, according the reagent desired, with 2(*3H*)-benzoxazolone (**1a**) or 2(*3H*)-benzothiazolinone (**1b**). Various deprotection methods, described in Table 1, were then tested.

As expected, deprotection of derivatives **2a** and **2b** did take place under mild acid (methods C and D), basic (method F)



Scheme 1. Protection and deprotection of 2(*3H*)-benzazolones derivatives (**2a**,**b**–**10a**,**b**) via different methods. (a): method A: ClP (P: COR or SO_2R), Bu_4NBr, K_2CO_3, CH_2Cl_2; method B: ClP (P: MOM or MEM), K_2CO_3, DMF (b) method C: TFA; method D: HCl 12 N (3 équiv), MeOH; method E: $TiCl_4$ (3 équiv), CH_2Cl_2; method F: KOH (3 équiv), MeOH; method G: Bu_4NF (1 M in solution in THF, 3 équiv), THF.

Keywords: 2(*3H*)-Benzoxazolone; 2(*3H*)-Benzothiazolone; Protecting groups.
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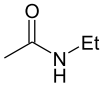
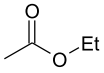
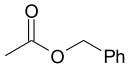
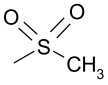
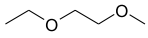
Table 1. results of deprotection attempts of 2(*3H*)-benzazolones derivatives (**2a**,**b**–**10a**,**b**)

Entry	X	Protecting groups	TFA (C)	HCl–MeOH (D)	$TiCl_4$–CH_2Cl_2 (E)	KOH–MeOH (F)	Bu_4NF–THF (G)
2a	O		1 h rt (85%)	2 h rt (79%)	N	0.5 h rt (89%)	1 h rt (91%)
2b	S		1 h rt (81%)	1.5 h rt (89%)	N	0.5 h rt (68%)	1 h rt (95%)
3a	O		N	N	N	0.5 h rt (30%)	1 h rt (95%)
3b	S		N	N	N	0.5 h rt (91%)	1 h rt (97%)
4a	O		N	N	N	0.5 h rt (78%)	0.25 h rt (98%)
4b	S		N	N	N	0.5 h rt (94%)	0.25 h rt (95%)
5a	O		N	N	N	0.5 h rt (39%)	0.25 h rt (89%)
5b	S		N	N	N	0.5 h rt (90%)	0.25 h rt (94%)
6a	O		N	N	2 h rt (90%)	0.5 h rt (20%)	0.5 h rt (96%)
6b	S		N	N	2 h rt (86%)	0.5 h rt (95%)	0.5 h rt (98%)
7a	O		N	N	N	0.5 h rt (10%)	0.5 h rt (96%)
7b	S		N	N	N	0.5 h rt (90%)	0.5 h rt (93%)
8a	O		N	N	N	Ring opening	1 h rt (93%)
8b	S		N	N	N	N	1 h rt (97%)
9a	O		4 h reflux (92%)	N	N	N	N
9b	S		4 h reflux (96%)	N	N	N	N
10a	O		4 h reflux (95%)	N	N	N	N
10b	S		4 h reflux (97%)	N	N	N	N

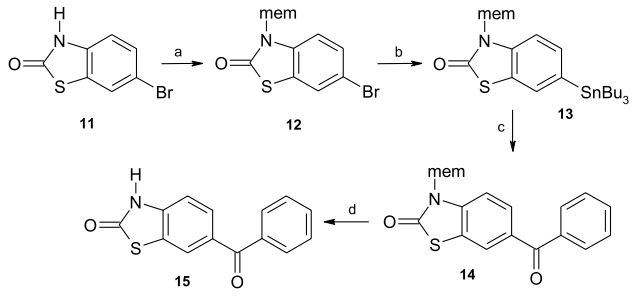
N: unsuccessful test performed 1 day at room temperature and then 1 day at reflux; rt: room temperature.

and neutral (method G) conditions and therefore the *N*-acetyl cannot be considered as a good protecting group. Compounds **3a**,**b**–**5a**,**b** and **7a**,**b**, respectively with COPh, CONHEt, CO_2Et and SO_2Me, resisted in acid medium (methods C, D and E). Application of the method F, in basic media, to the compounds **3b**–**5b** and **7b** give the desired derivative **1b** with good yield (90–95%), but in the 2(*3H*)-benzoxazolone series (**3a**–**5a** and **7a**) we observed that deprotected compounds were accompanied by important amounts of ring opening products (such as 2-aminophenol) which reduced the yield (10 to 78%). Deprotection method G, for compounds **3a**,**b**–**5a**,**b** and **7a**,**b**, with Bu_4NF in THF,[13,14] however gives excellent yields (89–98%).

Compound **6a**,**b** (Cbz protected) was not deprotected in acid media (method C and D) but in mild basic media (X=S: 95% and X=O: 20%) with method F (30 min at rt) and also by methods E and G with excellent yields (86–98%). Another attempt of deprotection of **6a**,**b** was performed by hydrogenolysis in THF using Pd/C (5 h, rt), which gave the deprotected derivatives **1a**,**b** with excellent yields (X=O: 94% and X=S: 97%). Method G with Bu_4NF in THF was found to be a very good alternative (**6a**: 96% and **6b**: 98%) specially because we did not observe any ring opening products.

Surprisingly, with compound **8b** we did not observe any deprotection either in acid or basic medium (methods C, D, E and F). In the corresponding 2(*3H*)-benzoxazolone series (**8a**) we observed ring opening, which gave 2-amino-(*N*-phenylsulfonyl) phenol. Compounds **8a**,**b** were successfully cleaved with Bu_4NF (THF, rt, 1 h, 93 and 97%). Deprotection of compounds **9a**,**b** and **10a**,**b** was realized only in TFA at reflux for 4 h, nevertheless with very good yields (92 to 97%).

In order to validate the interest of the protecting group for 2(*3H*)-benzazolone, we applied our results to a benchmark, i.e. the synthesis of 6-benzoyl-2(*3H*)-benzothiazolone. In our laboratory we observed indeed that the introduction of tributyltin in the 6-position of the 2(*3H*)-benzothiazolone could be performed only on N-methyl compounds and not on the free NH series. In Scheme 2, we introduced the MEM protecting group on 6-bromo-2(*3H*)-benzothiazolone (**12**) to synthetise the corresponding tributyltin derivative. The benzoyl moiety was then easily introduced and the resulting



Scheme 2. Protection of 2(*3H*)-benzothiazolone. (a): MEM-Cl, K_2CO_3, DMF (b): $(Bu_3Sn)_2$, $Pd(PPh_3)_4$, toluene (c): ClCOPh, $PdCl_2(PPh_3)_2$, toluene (d): TFA.

compound was then deprotected to afford the free NH derivative (**15**).

More specifically, 6-bromobenzothiazolinone[15] (**11**) was protected in DMF with MEM group to afford compound **12**. Tributyltin derivative **13** was obtained from the bromo precursor **12** via Stille's reaction with $(Bu_3Sn)_2$ and $Pd(PPh_3)_4$ in toluene. Compound **13** can then embarked in coupling reactions with various aryl or cycloalkylcarbonyl chlorides. In Scheme 2, to exemplify the validity of our approach, we introduced a benzoyl group in 6-position to give derivative **14**. The MEM protection group was then cleaved in refluxed TFA for 2 h to furnish the expected compound **15**.[16]

3. Conclusion

In conclusion, we found different protecting groups which can be used in acid or basic medium reaction conditions and subsequently removed without major problems. The COPh, CONHEt, CO_2Et, CO_2CH_2Ph, SO_2CH_3 derivatives (**3a**,**b**–**7a**,**b**) resisted in acid conditions and were cleaved in basic medium. MOM and MEM protection (**9a**,**b**–**10a**,**b**) resisted in basic media and were cleaved in TFA. The SO_2Ph protection (**8b**) resisted in acid and basic media and was cleaved with Bu_4NF in THF (method G).

Method G in particular constitutes a mild and selective method of deprotection for *N*-protected-2(*3H*)-benzazolones (derivatives **3a**,**b**–**8a**,**b**) with excellent yields (89–98%) and was compatible both with acid or basic-sensitive groups. To exemplify the validity of our approach, we synthesized compounds **15** via *N*-MEM protection. These deprotection methods could be extended to other cyclic carbamates, such as oxazolo[4,5]pyridin-2(*3H*)-ones, hydantoins, and barbiturates.

4. Experimental

4.1. General methods of protection

Method A (**2a**,**b**–**8a**,**b**). To a solution of 2(*3H*)-benzazolones (22 mmol) in CH_2Cl_2 (30 mL), K_2CO_3 (66 mmol), Bu_4NBr (1 mmol) and the desired acid chloride reagent (66 mmol) were added. The reaction was refluxed for 4 h. The solvent was evaporated under reduce pressure. The solution was hydrolyzed with water (30 mL) and stirred for 1 h. The precipitate was filtered and recrystallized from the appropriate solvent.

Method B (**9a**,**b**–**10a**,**b**). To a solution of 2(*3H*)-benzazolones (22 mmol) in DMF (30 mL), K_2CO_3 (66 mmol) was added. The reaction was stirred at 80 °C for 1 h and the desired chlororeagent (66 mmol) added. The solution was stirred for 3 h at the same temperature. The solvent was evaporated under reduced pressure. The solution was hydrolyzed with water (30 mL) and stirred for 1 h. The precipitate was filtered and recrystallized with the appropriate solvent.

4.1.1. 3-Acetyl-2(*3H*)-benzoxazolone (2a). Yield 90% (cyclohexane). Mp 90–91 °C. IR (KBr) 2870, 1726, 1688, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=2.75$ (s, 3H), 7.15–7.29 (m, 3H), 8.25 (m, 1H).

4.1.2. 3-Acetyl-2(*3H*)-benzothiazolone (2b). Yield 84% (cyclohexane). Mp 103–104 °C. IR (KBr) 2930, 1695, 1676, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=2.80$ (s, 3H), 7.03–7.24 (m, 3H), 8.15 (m, 1H).

4.1.3. 3-Benzoyl-2(*3H*)-benzoxazolone (3a). Yield 70% (cyclohexane). Mp 178–179 °C. IR (KBr) 1805, 1697, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=7.25$–7.38 (m, 3H), 7.48–7.59 (m, 2H), 7.67 (m, 1H), 7.81–7.92 (m, 3H).

4.1.4. 3-Benzoyl-2(*3H*)-benzothiazolone (3b). Yield 81% (cyclohexane). Mp 165–166 °C. IR (KBr) 1702, 1724, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=7.32$–7.48 (m, 3H), 7.65–7.78 (m, 2H), 7.84 (m, 1H), 7.95–8.14 (m, 3H).

4.1.5. 3-Ethylaminocarbonyl-2(*3H*)-benzoxazolone (4a). Yield 80% (cyclohexane). Mp 104–106 °C. IR (KBr) 3350, 2970, 1737, 1658, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=1.30$ (t, $J=7.30$ Hz, 3H), 3.20 (q, $J=7.30$ Hz, 2H), 5.00 (m, 1H), 7.23–7.30 (m, 3H), 8.05 (m, 1H).

4.1.6. 3-Ethylaminocarbonyl-2(*3H*)-benzothiazolone (4b). Yield 88% (cyclohexane). Mp 112–113 °C. IR (KBr) 3340, 2954, 1701, 1665, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=$ 1.35 (t, $J=7.20$ Hz, 3H), 3.20 (q, $J=7.20$ Hz, 2H), 5.05 (m, 1H), 7.11–7.23 (m, 3H), 8.10 (m, 1H).

4.1.7. 3-Ethoxycarbonyl-2(*3H*)-benzoxazolone (5a). Yield 70% (petroleum ether). Mp 70–71 °C. IR (KBr) 2975, 1852, 1747, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=1.51$ (t, $J=7.34$ Hz, 3H), 4.55 (q, $J=7.34$ Hz, 2H), 7.26–7.38 (m, 3H), 7.80 (m, 1H).

4.1.8. 3-Ethoxycarbonyl-2(*3H*)-benzothiazolone (5b). Yield 90% (petroleum ether). Mp 65–66 °C. IR (KBr) 2971, 1845, 1746, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=1.50$ (t, $J=7.30$ Hz, 3H), 4.50 (q, $J=7.30$ Hz, 2H), 7.31–7.53 (m, 3H), 8.10 (m, 1H).

4.1.9. Benzyloxycarbonyl-2(*3H*)-benzoxazolone (6a). Yield 70% (cyclohexane). Mp 130–131 °C. IR (KBr) 1809, 1749, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=5.50$ (s, 2H), 7.16–7.28 (m, 3H), 7.36–7.46 (m, 3H), 7.50–63–7.24 (m, 2H), 7.75 (m, 1H).

4.1.10. Benzyloxycarbonyl-2(*3H*)-benzothiazolone (6b). Yield 88% (cyclohexane). Mp 62–63 °C. IR (KBr) 1739, 1709, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=5.55$ (s, 2H), 7.26–7.45 (m, 8H), 7.55 (m, 1H).

4.1.11. 3-Methylsulfonyl-2(*3H*)-benzoxazolone (7a). Yield 75% (cyclohexane). Mp 142–143 °C. IR (KBr) 1733, 1600, 1185 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=3.50$ (s, 3H), 7.18–7.31 (m, 3H), 7.70 (m, 1H).

4.1.12. 3-Methylsulfonyl-2(*3H*)-benzothiazolone (7b). Yield 79% (cyclohexane). Mp 148–149 °C. IR (KBr) 1697, 1600, 1185 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=3.60$ (s, 3H), 7.28–7.43 (m, 3H), 8.10 (m, 1H).4.1.

4.1.13. 3-Phenylsulfonyl-2(*3H*)-benzoxazolone (8a). Yield 96% (cyclohexane). Mp 145–146 °C. IR (KBr) 1733, 1600, 1190 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=7.25$ (m, 1H), 7.35–7.48 (m, 2H), 7.60 (m, 2H), 7.70 (m, 1H), 8.10 (m, 2H), 8.25 (m, 1H).

4.1.14. 3-Phenylsulfonyl-2(*3H*)-benzothiazolone (8b). Yield 96% (cyclohexane). Mp 136–137 °C. IR (KBr) 1717, 1620, 1190 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=7.30$ (m, 1H), 7.32–7.45 (m, 2H), 7.60 (m, 2H), 7.70 (m, 1H), 8.10 (m, 2H), 8.25 (m, 1H).

4.1.15. 3-Methoxymethyl-2(*3H*)-benzoxazolone (9a). Yield 90% (cyclohexane). Mp 90–91 °C. IR (KBr) 1762, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=3.40$ (s, 3H), 5.20 (s, 2H), 7.12–7.29 (m, 4H).

4.1.16. 3-Methoxymethyl-2(*3H*)-benzothiazolone (9b). Yield 95% (cyclohexane). Mp 114–115 °C. IR (KBr) 1695, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=3.35$ (s, 3H), 5.10 (s, 2H), 7.02–7.24 (m, 4H).

4.1.17. 3-Methoxyethoxymethyl-2(*3H*)-benzoxazolone (10a). Yield 90% (cyclohexane). Mp 28–29 °C. IR (KBr) 2882, 1782, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=3.30$ (s, 3H), 3.50 (t, $J=7.00$ Hz, 2H), 3.75 (t, $J=7.00$ Hz, 2H), 5.40 (s, 2H), 7.15–7.36 (m, 4H).

4.1.18. 3-Methoxyethoxymethyl-2(*3H*)-benzothiazolone (10b). Yield 93% (cyclohexane). Mp 75–76 °C. IR (KBr) 2898, 1709, 1600 cm^{-1}. ^1H NMR ($CDCl_3$) $\delta=3.30$ (s, 3H), 3.50 (t, $J=7.05$ Hz, 2H), 3.70 (t, $J=7.05$ Hz, 2H), 5.35 (s, 2H), 7.06–7.28 (m, 4H).

4.1.19. 6-Bromo-3-methylethoxymethyl-2(*3H*)-benzothiazolone (12). To a mixture of 6-bromo-2(*3H*)-benzothiazolone (**11**) (5 g, 21.7 mmol) in DMF (50 mL), potassium carbonate (9 g, 65.2 mmol) and MEM-Cl (9.9 mL, 86.8 mmol) were added. The reaction was stirred at 90 °C for 2 h. The solution was evaporated under reduced pressure and 50 mL of water added. The solution was extracted with CH_2Cl_2, then the organic layer evaporated under reduced pressure. The residue was recrystallized in cyclohexane. Yield 92%. Mp 97–98 °C. IR 1692, 1600. ^1H NMR ($CDCl_3$) $\delta=3.30$ (s, 3H), 3.50 (m, 2H), 3.70 (m, 2H), 5.50 (s, 2H), 7.15 (d, 1H, $J=8.30$ Hz), 7.45 (dd, 1H, $J=8.30$ Hz, $J=1.50$ Hz), 7.55 (d, 1H, $J=1.50$ Hz).

4.1.20. 6-Tributyltin-3-methylethoxymethyl-2(*3H*)-benzothiazolinone (13). To a mixture of 6-bromo-3-methylethoxymethyl-2(*3H*)-benzothiazolone (**12**) (5 mmol) in toluene (20 mL) under argon, tetrakis(triphenyl phosphine) palladium (0.5 mmol) and bis(tributyltin) (10 mmol) were added. The reaction was refluxed for 16 h. The solution was evaporated under reduced pressure. The oily residue was purified by flash column chromatography with petroleum ether/EtOAc (9.5/0.5) to give an oily product. Yield 63%. IR 1692, 1605. ^1HNMR ($CDCl_3$): $\delta=0.90$ (t, 9H, $J=5.90$ Hz), 1.10 (t, 6H, $J=6.10$ Hz), 1.35 (m, 6H), 1.55 (m, 6H), 3.35 (s, 3H), 3.55 (m, 2H), 3.75 (m, 2H), 5.45 (s, 2H), 7.30 (d, 1H, $J=7.90$ Hz), 7.40 (dd, 1H, $J=7.90$ Hz, $J=1.05$ Hz), 7.50 (s, 1H).

4.1.21. 6-Benzoyl-3-methylethoxymethyl-2(*3H*)-benzothiazolone (14). 6-Tributylstannic-3-methylethoxymethyl-2(*3H*)-benzothia-zolone (**13**) (1.9 mmol) in toluene (10 mL) was placed under argon, dichlorobis(triphenylphosphine) palladium (0.18 mmol) and benzoylchloride (2.8 mmol) were added. The reaction was refluxed for 16 h. The solution was evaporated under reduced pressure. The residue was purified by flash column chromatography with CH_2Cl_2/EtOAc (9/1) and recrystallized in cyclohexane. Yield 86%. Mp 101–102 °C; IR 1696, 1649. ^1HNMR ($CDCl_3$): $\delta=3.35$ (s, 3H), 3.50 (m, 2H), 3.75 (m, 2H), 5.50 (s, 2H), 7.40 (d, 1H, $J=8.70$ Hz), 7.50 (m, 2H), 7.60 (m, 1H), 7.80 (m, 2H), 7.85 (dd, 1H, $J=8.70$ Hz, $J=1.10$ Hz), 8.00 (d, 1H, $J=1.10$ Hz).

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