SCIENTIFIC LETTER 19

Edito

Pour cette 19ème lettre scientifique d'Atlanchim Pharma, nous tenions à mettre en lumière les chimistes qui œuvrent chaque jour à la réalisation de vos projets. Ainsi vous retrouverez en dernière page la présentation de notre chef de projet, **Thibaut MARTINEZ** qui a travaillé lors sa thèse sur la « Cyclisation de 2-pyridylallènes : vers de nouveaux dérivés d'indolizines et ligands carbéniques chiraux » (Louis Fensterbank, Sorbonne Université, IPCM, Paris, 2019).

Nous sommes heureux de pouvoir vous les présenter car grâce à cette équipe de scientifiques nous enrichissons le savoir-faire de notre belle société de prestation de services.

Chez Atlanchim Pharma nous avons pour objectif d'être réactifs dans le retour de vos demandes, de vous offrir un suivi projet et client régulier et de qualité, une communication scientifique renforcée pour mieux vous servir. C'est aussi et surtout plus de 18 ans d'expérience reconnue pour notre forte expertise en synthèse de petites molécules, à l'échelle du laboratoire, et dans les domaines suivants : Synthèse à façon, que ce soit de la recherche à la reproduction de vos documents ; le marquage à froid de molécules ; et synthèse d'impureté : de l'identification à l'isolement.

L'équipe Atlanchim Pharma a le plaisir de vous annoncer qu'en 2023 nous apportons du changement du côté de la communication avec un nouveau format de nos WEBINAIRES en anglais, toujours présenté par notre Directeur Scientifique, **Jacques Lebreton**. Mais aussi un nouveau site internet sur lequel vous retrouverez toute notre expertise, nos publications scientifiques et bien plus encore…

Restez connecté et n'hésitez pas à vous inscrire à notre newsletters sur notre site internet pour ne pas manquer la thématique de notre prochain Webinaire du 13 Juin 2023:
A VERSATILE REDUCING AGENT.

Bonne lecture !

Aurélie et Héléna

Editorial

For this 19th scientific letter of Atlanchim Pharma, we wanted to highlight the chemists who work every day to realize your projects. You will find on the last page the presentation of our project manager, **Thibaut MARTINEZ** who worked during his thesis on the "Cyclisation of 2-pyridylallenes: towards new indolizine derivatives and chiral carbenic ligands" (Louis Fensterbank, Sorbonne University, IPCM, Paris, 2019)

We are happy to present them to you and thanks to this team of scientists we are enriching the know-how of our beautiful Contract Research Organization.

At Atlanchim Pharma our objective is to be reactive in the return of your requests, to offer you a regular and quality project and customer follow-up, a reinforced scientific communication to better serve you. It is also and above all more than 18 years of experience recognized by our strong expertise in small molecules synthesis, on a laboratory scale, and in the following fields such as : Custom synthesis, from research to reproduction of your documents; site-specific stable isotope labeling; and impurity synthesis: from identification to isolation.

Atlanchim Pharma's team is pleased to announce that in 2023 we are also bringing change in the communication with a new format of our WEBINARS in English still presented by our Scientific Director, **Jacques Lebreton**. But also a new website where you will find all our expertise, our scientific publications and much more...

Stay tuned and feel free to subscribe to our newsletters on our website so that you don't miss the theme of our next Webinar on June, 13th:
A VERSATILE REDUCING AGENT .

Enjoy the reading !

Aurélie et Héléna

About silyl ether protecting groups

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The right choice of protecting groups is crucial in organic synthesis, especially for the preparation of complex functionalized molecules[1]. In this context, silyl ether protecting groups have a predominant place[2]. First introduced in 1944 by R. O. Sauer[3], and popularized by E. J. Corey[4] with his seminal 1972 paper, cited over 7,000 times, silyl ethers have had a very strong impact in the field of organic synthesis, and more particularly in total synthesis. Silyl ethers, such as trimethylsilyl (TMS), triethylsilyl (TES), tert-butyldimethylsilyl (TBDMS, also abbreviated TBS), tri-iso-propylsilyl (TIPS), and tert-butyldiphenylsilyl (TBDPS)[5] (See Figure 1), have been widely used as protecting groups of hydroxyl functions with success over the past decades.

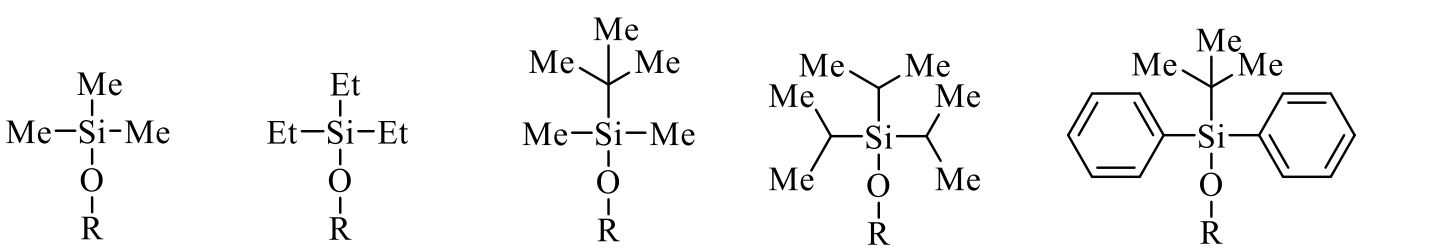


Figure 1. Some commonly used silyl ether protecting groups

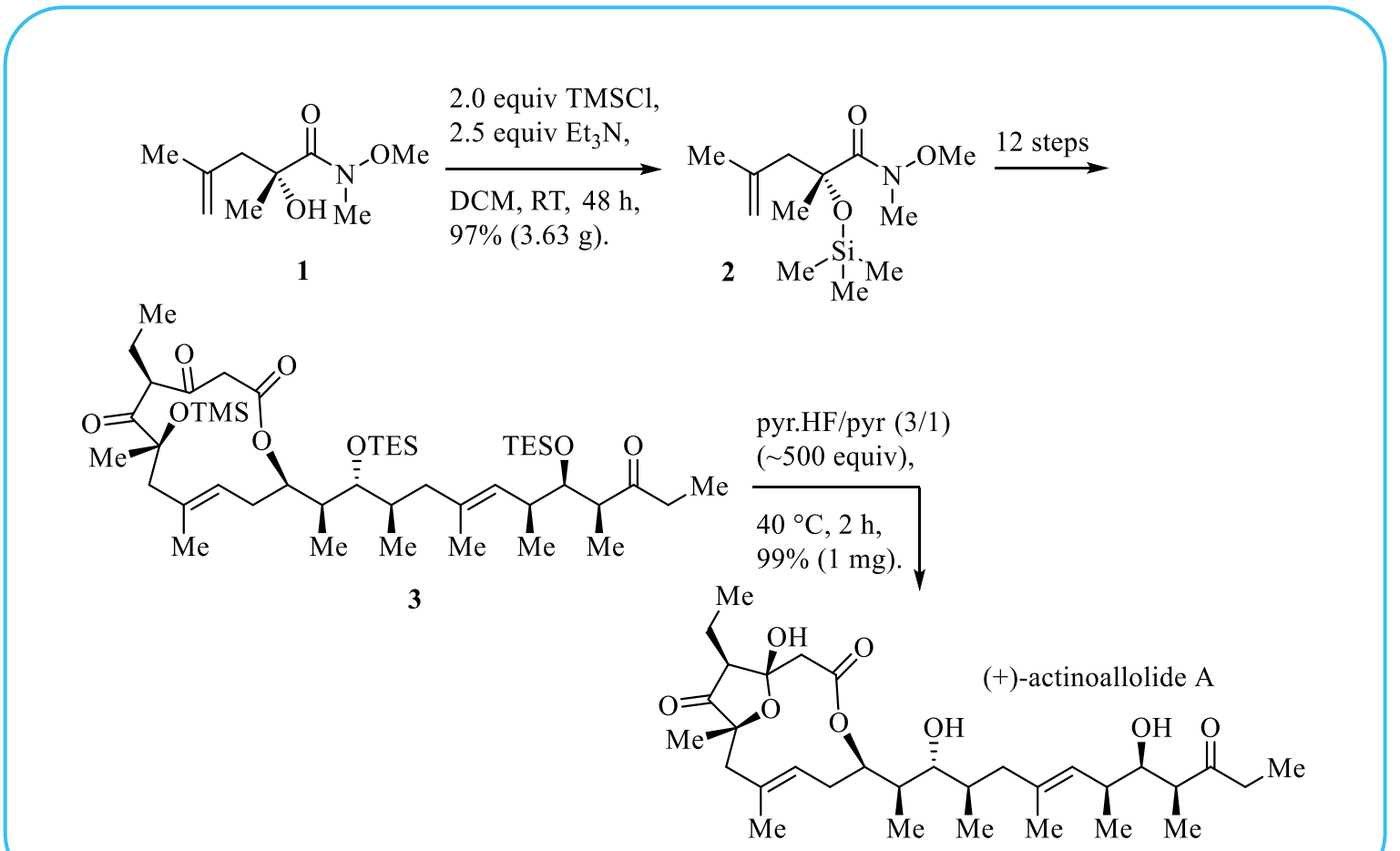
Substituents on the silicon atom, depending on their steric and electronic properties, greatly affect the reactivity of silyl ethers, resulting in differences in stability depending on reaction conditions. In acidic conditions, stability of silyl ethers increases in general, in the following order:
TMS (1) < TES (64) < TBDMS (20,000) << TIPS (700,000)) << TBDPS (5,000,000).
In basic conditions, the following order of increasing stability is reported as following:
TMS (1) < TES (10-100) << TBDMS ~ TBDPS (20,000) << TIPS (100,000).

[1] E. J. Corey, X.-M. Cheng, The Logic of Chemical Synthesis, Wiley: New York, **1995**.
[2] (a) P. J. Kocienski, Protecting Groups, 3rd Edition, Thieme, 2005. (b) P. G. M. Wuts, Greene's Protective Groups in Organic Synthesis, 5th Edition, Wiley: New York, 2014. (c) For an excellent review on the silyl ether protecting groups, see: R. D. Crouch, *Tetrahedron* **2013**, 69, 2383-2417 and references cited.
[3] R. O. Sauer, *J. Am. Chem*. Soc. **1944**, *66*, 1707-1710.
[4] E. J. Corey, A. Venkateswarlu, *J. Am. Chem*. Soc. **1972**, *94*, 6190-6191.
[5] S. Hanessian, P. Lavallée, Can. *J. Chem*. **1975**, *53*, 2975-2977.

This microreview presents selected examples of silyl protecting groups manipulation from recent literature. These examples illustrate the most common problems encountered in AtlanChim Pharma with silyl protecting groups, in the past ten years, and the solutions we have provided.

TMS protecting group

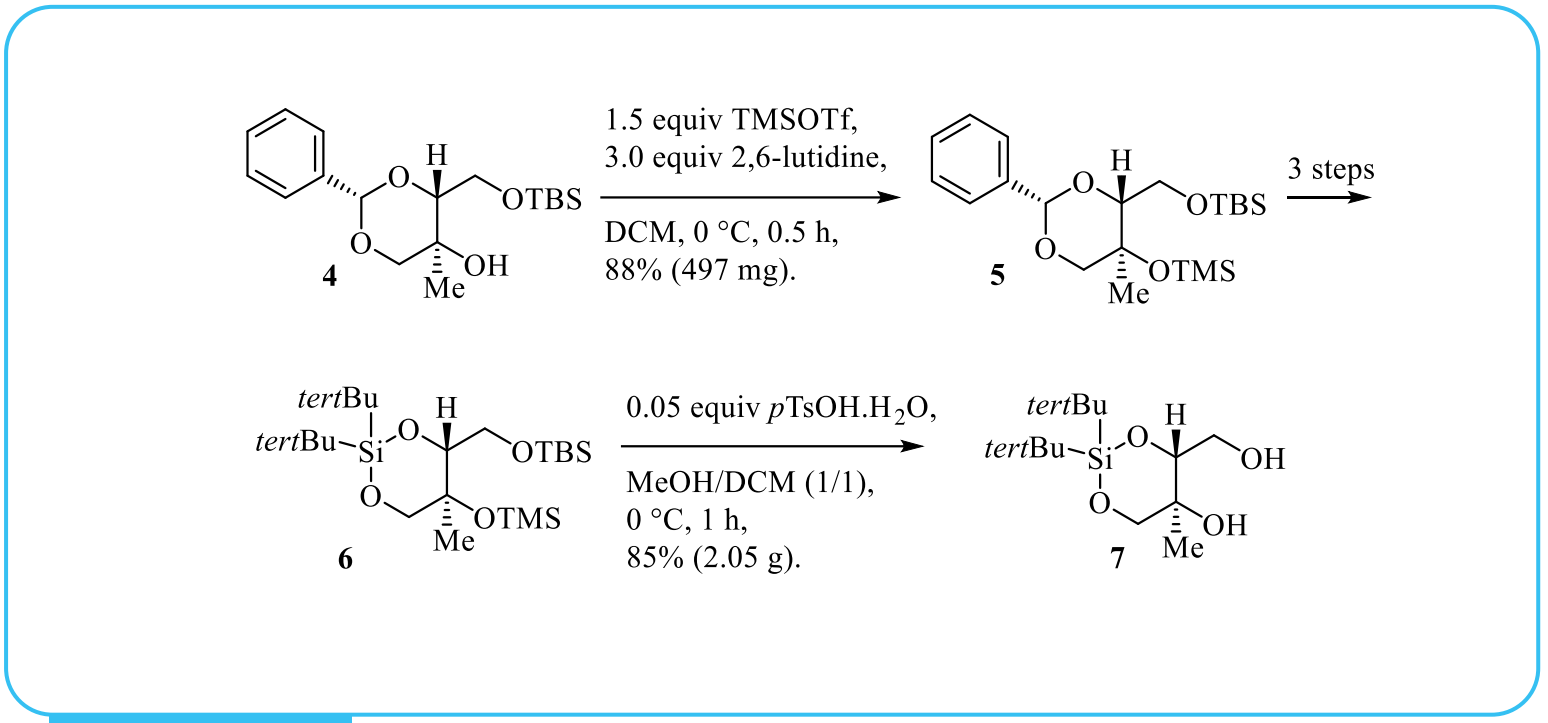
In general, the cleavage of the TMS protecting group is so easy (*vide supra*) that it is unsuitable for most alcohol functions requiring long-term protection. Nevertheless, TMS protecting group has been used with success on tertiary alcohols as reported in the following examples. In the first total synthesis of the potent and selective *in vitro* anti-trypanosomal macrolide (+)-actinoallolide A, the Weinreb amide intermediate **1** was reacted under standard conditions with an excess of TMSCl and Et_3N to afford in nearly quantitative yield on a multigram scale the corresponding TMS ether **2**, as outlined in Scheme 1.[6] From this latter intermediate **2**, the TMS protecting group was sufficiently stable to allow the next twelve steps to provide the triketone **3**, which was treatmented with HF-Pyridine buffered with pyridine giving (+)-actinoallolide A in excellent yield. In these later conditions both TES groups on secondary alcohols and the TMS group were cleaved and the release of the tertiary alcohol inducing the controlled hemiacetal moiety formation.



SCHEME 1

[6] M. J. Anketell, T. M. Sharrock, I. Paterson, *Angew. Chem. Int. Ed*. **2020**, *59*, 1572-1576.

Sakai and Mori used the TMS protecting group in the synthesis of the ABCD fragment of gymnocin-B, a cytotoxic ladder-shaped polyether, as reported in Scheme 2.[7] Tertiary alcohol **4** was protected with TMS triflate in the presence of an excess of 2,6-lutidine to afford the intermediate **5** in 88% yield. After three steps, treatment of protected compound **6** under acidic conditions led to the cleavage of both TMS and TBS groups affording the diol **7** in 85% yield.



SCHEME 2

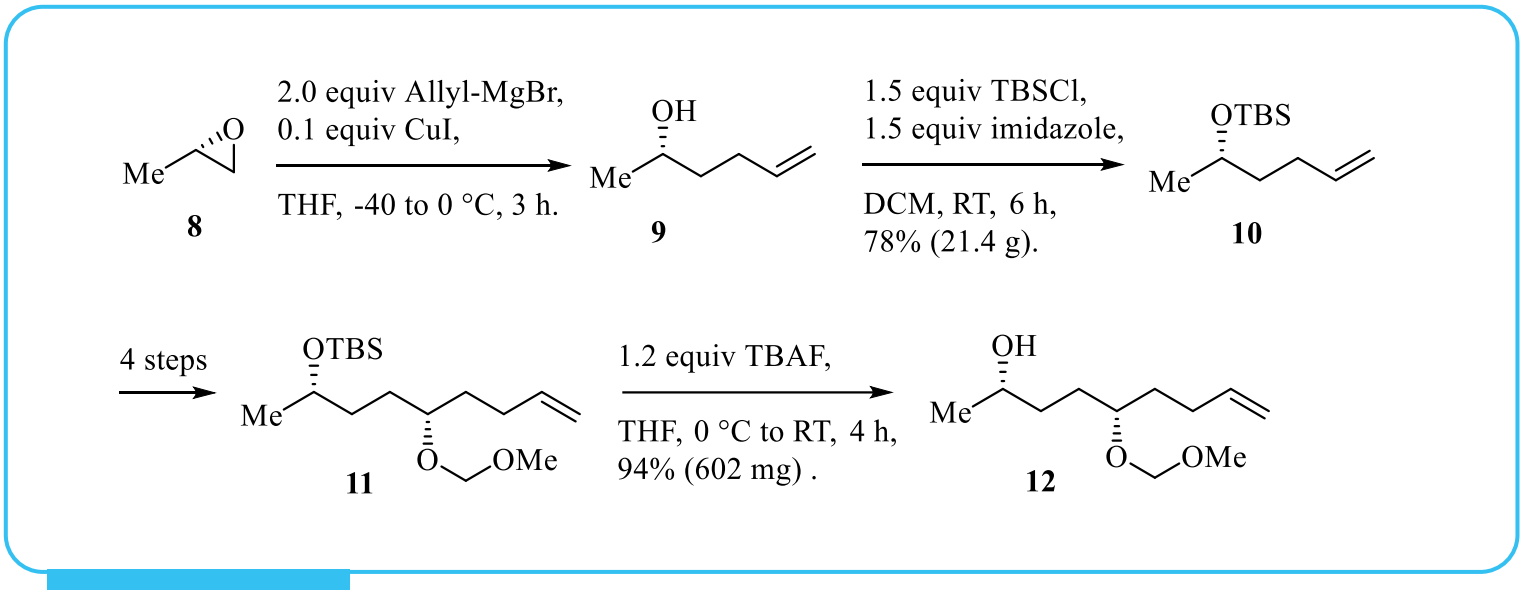
As shown in these later examples with tertiary alcohols, steric factors play a major role in the relative stability of TMS protecting groups.

TBS and TBDPS protecting groups

The most used silyl ethers are TBS and TBDPS due to their good stability towards various reagents and conditions. The most common procedures to introduce these protecting groups are the treatment of alcohol with TBS-Cl using imidazole or Et_3N in presence of catalytic amounts of DMAP, as highlighted in Schemes 3 and 4. As a starting point of a total synthesis and structural revision of greensporone F and dechlorogreensporone F recently published by Mohapatra, the secondary homoallylic alcohol **9**, prepared from (*S*)-propylene oxide **8** by treatment with the allyl Grignard reagent and 10 mol% of CuI, was reacted with TBS-Cl in the presence of imidazole in DCM at 0 °C to provide the TBS ether **10** in 78% yield over two steps.[8] Four steps later, the TBS group in intermediate **11** was removed smoothly with tetrabutylammonium fluoride (TBAF) to give **12** in 94% yield. For the cleavage of silyl ethers, TBAF is one of the most popular fluoride-based reagent, commercially available in 1 M THF solution containing ca. 5% water or as white solid trihydrate (*vide infra*).

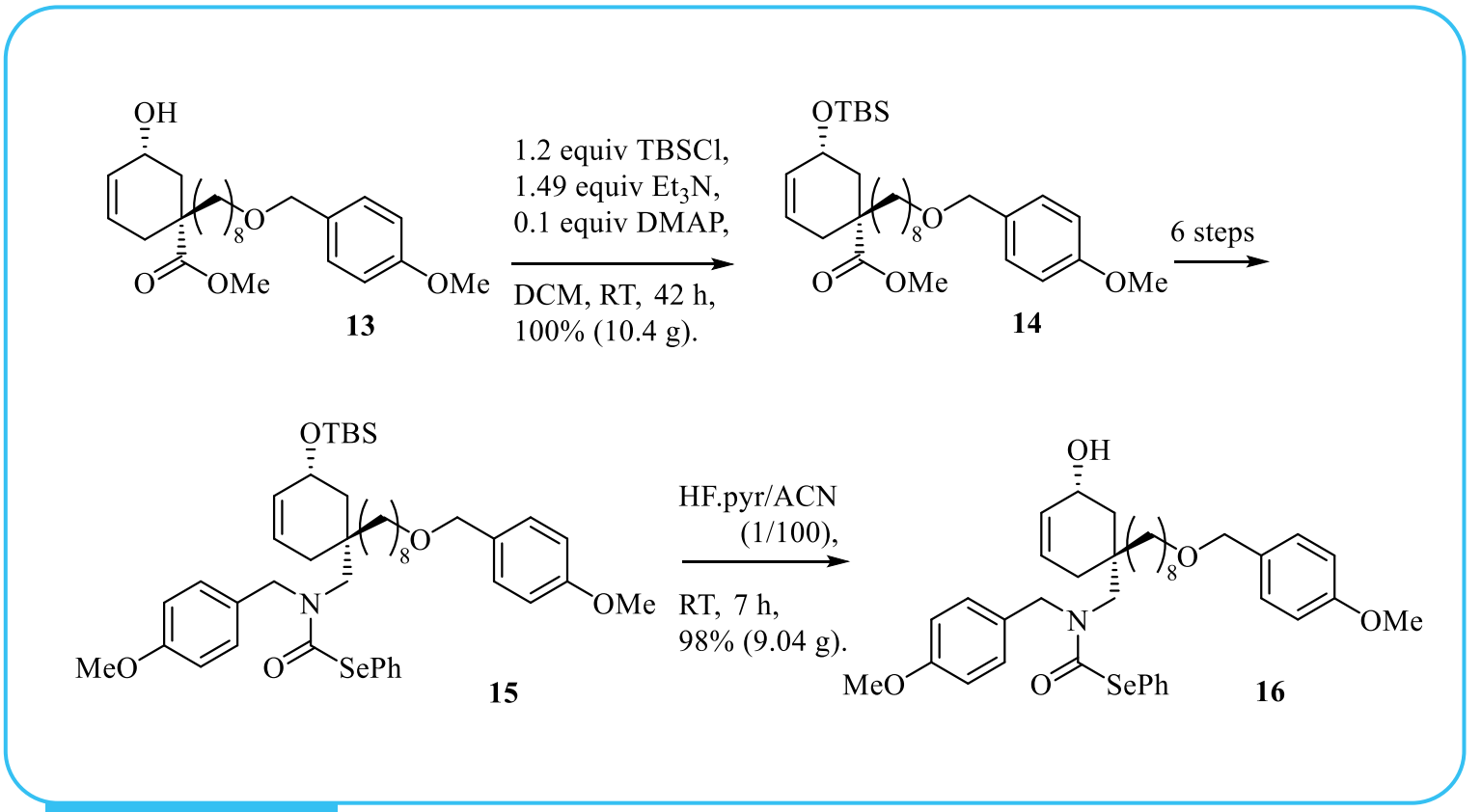
[7] T. Sakai, K. Hata, Y. Kitamura, R. Ishibashi, Y. Mori, *Tetrahedron Lett*. **2019**, *60*, 151261.

[8] J. Gaddam, Aedula V. V. Reddy, A. V. S. Sarma, J. S. Yadav, D. K. Mohapatra, *J. Org. Chem*. **2020**, *85*, 12418-12429.



SCHEME 3

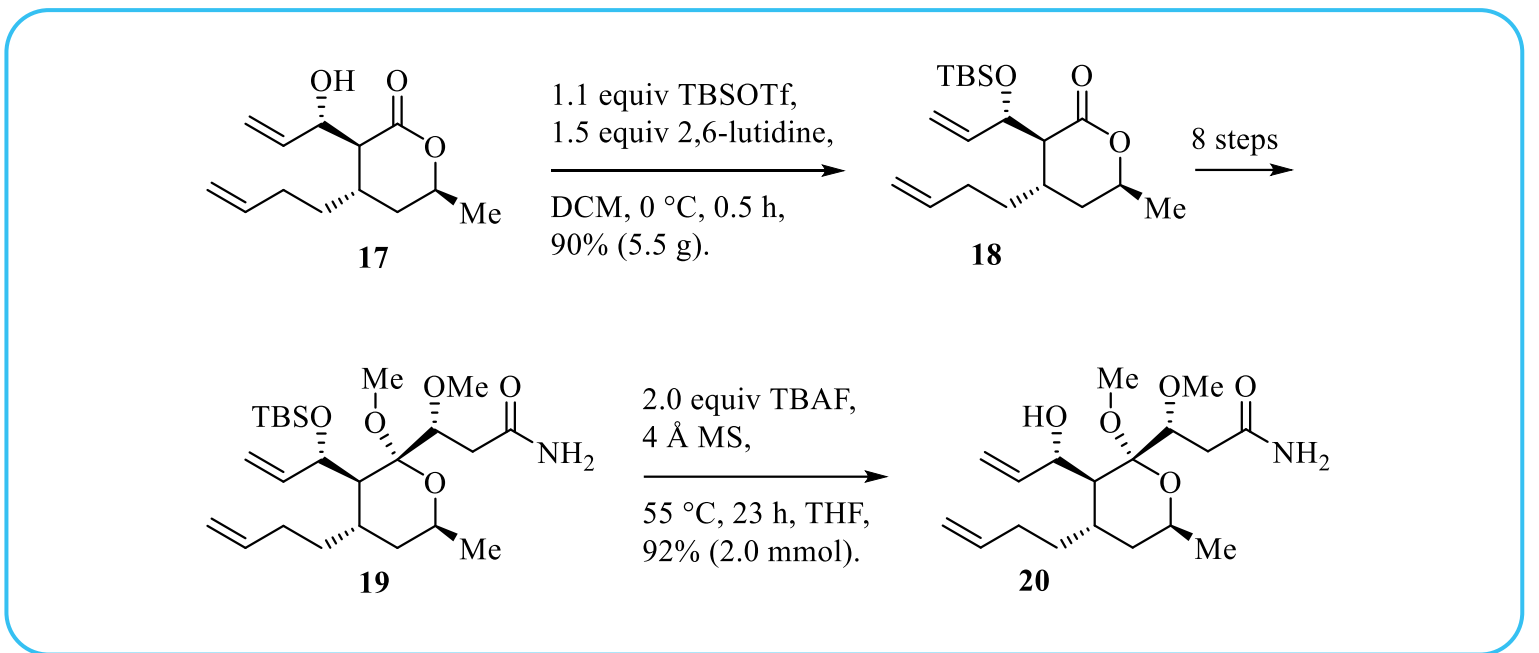
In the context of a formal synthesis of (-)-haliclonin A, the chiral cyclic allylic alcohol **13** was protected with TBS-Cl in the presence of Et_3N and a catalytic amount of DMAP to give the TBS silyl ether **14** in 100% yield on ten-gram-scale, as reported in Scheme 4.[9] Few steps later, desilylation of **15** was performed by treatment with HF-Pyridine at RT in ACN to give the selenocarbamate intermediate **16** in an excellent yield of 98%.



SCHEME 4

[9] K. Komine, Y. Urayama, T. Hosaka, Y. Yamashita, H. Fukuda, S. Hatakeyama, J. Ishihara, *Org. Lett.* **2020**, *22*, 5046-5050.

With more hindered alcohols, TBS-triflate in the presence of 2,6-lutidine is employed as reported in Scheme 5 for the preparation of intermediate **18** from alcohol **17**, in the total synthesis of the proposed structure of marineosin A.[10] After eight steps, the removal of silyl protecting group of amide **19** was carried out with TBAF in the presence of 4 Å molecular sieves to provide the free alcohol **20** in 92% yield on 2.0 mmol scale. Thus, it is important to note that fluoride forms strong hydrogen bonds with water, so moisture absorption by the molecular sieves considerably increases the reagent's nucleophilicity and basicity and, therefore, the reaction rate and yield of silyl ether cleavage.[11]

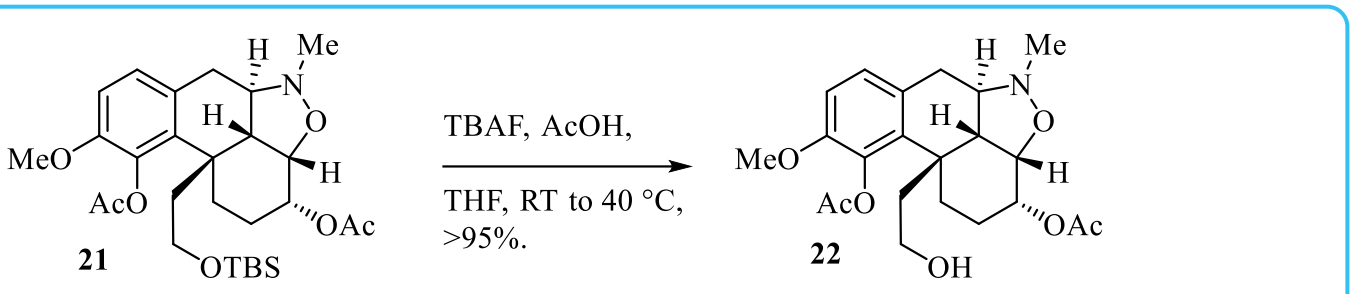


SCHEME 5

It is also well-established that with TBAF-induced removal of silyl ethers, the basicity of the resulting reaction mixture, due to the inevitable presence of water which reacts with the strongly basic tetrabutylammonium alkoxide intermediate releasing a hydroxide, may be incompatible with base-sensitive substrates.[12] In particular, migration and/or cleavage of acetyl and more generally acyl groups are well-documented phenomena observed with TBAF during desilylation. It should be pointed out that attempts to remove water from commercial TBAF trihydrate salts by heating under high vacuum resulted in decomposition to tetrabutylammonium bifluoride (HF_2^-), tributylamine, and 1-butene.[13]

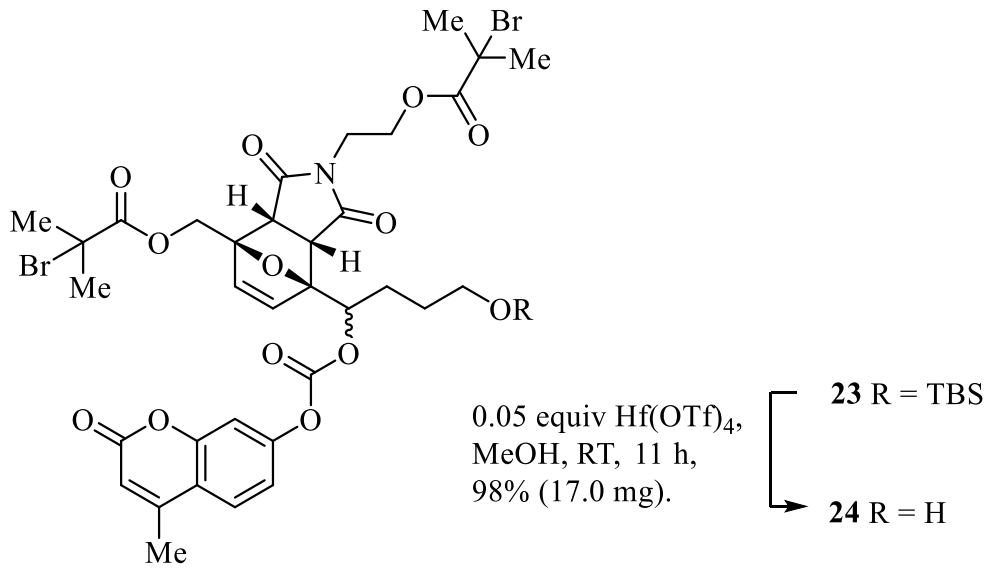
In this context, TBAF buffered with HOAc is typically used to avoid degradation and to improve yields. It is worth noting that HF-pyridine-mediated deprotection is also an alternative for substrates exhibiting base-sensitivity, as mentioned in Scheme 4. A representative example of TBAF buffered with HOAc desilylation on the advanced diacetate intermediate **21** to provide primary alcohol **22**, in the total synthesis of (±)- thebainone A, is highlighted in Scheme 6.[14] During our research, we often used this procedure and we noticed that the desilylation reaction rates, on the same substrates, with TBAF buffered with AcOH were much slower than those carried out with TBAF alone.

[10] B. Xu, G. Li, J. Li, Y. Shi, *Org. Lett.* **2016**, *18*, 2028-2031.
[11] (a) H. C. Kolb, S. V. Ley, A. M. Z. Slawin, D. J. J. Williams, *Chem. Soc.*, *Perkin Trans*. 1 **1992**, 2735-2761.
(b) T. Sakai, K. Hata, Y. Kitamura, R. Ishibashi, Y. Mori, *Tetrahedron Letters* **2019**, *60*, 151261.
[12] (a) A. S. Pilcher, H. L. Ammon, P. DeShong, *J. Am. Chem. Soc*. **1995**, *117*, 5166-5167.
(b) B. Doboszewski, P. Herdewijn, *Tetrahedron Letters* **2012**, *53*, 2253-2256.
(c) Z. A. Morrison, M. Nitz, *Org. Lett*. **2020**, *22*, 1453-1457.
[13] D. P. Cox, J. Terpinski, W. Lawrynowicz, *J. Org. Chem*. **1984**, *49*, 3216-3219.
[14] Y. Wang, A. Hennig, T. Küttler, C. Hahn, A. Jäger, P. Metz, *Org. Lett.* **2020**, *22*, 3145-3148.



SCHEME 6

For efficient selective cleavage of silyl ethers, besides acidic and fluoride-based reagents (*vide supra*), metal Lewis acid-based methodologies have been described using $Hf(OTf)_4$[15] or $NaAuCl_4 \cdot 2H_2O$[16], in particular, as outlined in Schemes 7 and 8. For the preparation of the mechanophore (stress or strain activated molecules) scaffold **24**, various attempts to cleave the TBS group of **23** with TBAF led to partial release of the hydroxycoumarin moiety (see Scheme 7).[17] Alternatively, $Hf(OTf)_4$-catalyzed desilylation of **23** in methanol at room temperature provided the desired target **24** in 98% yield.

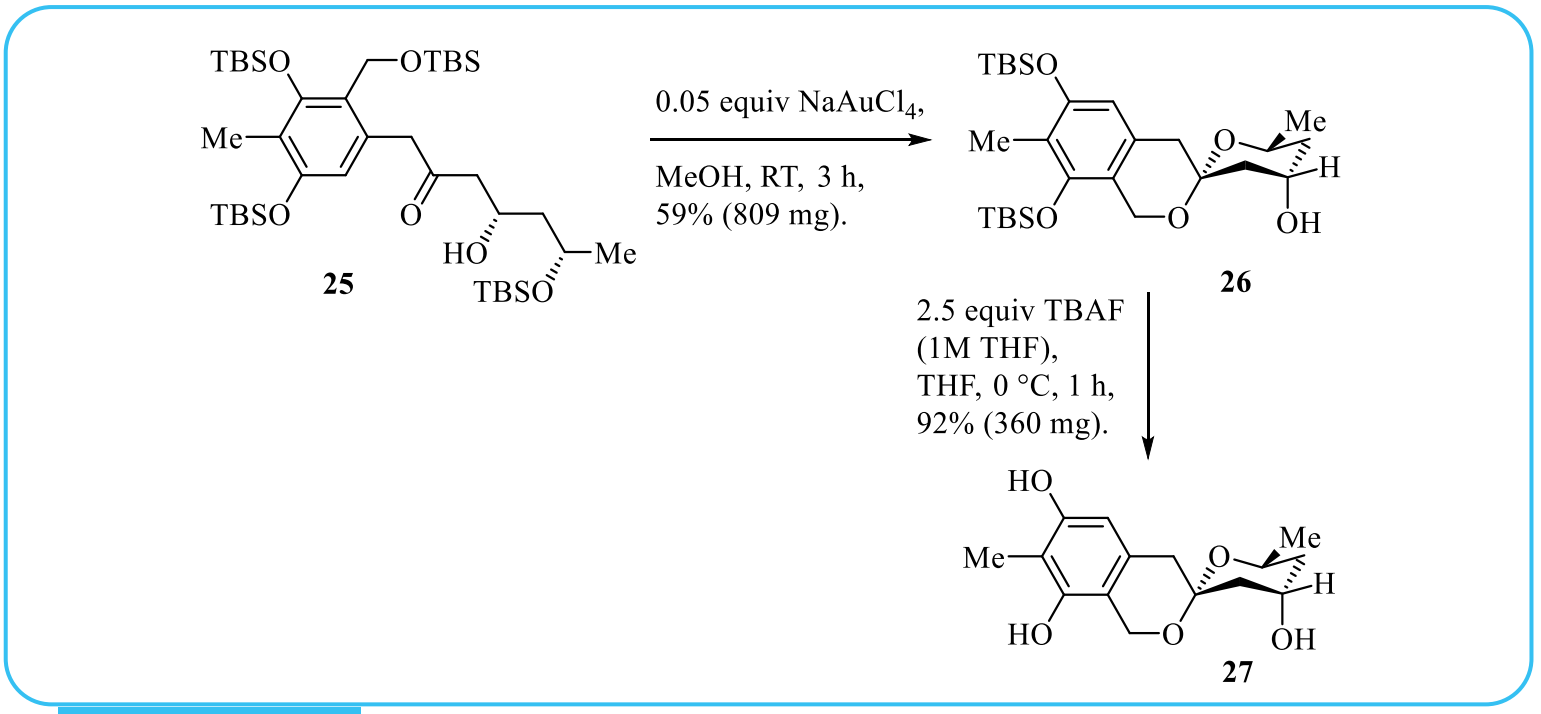


SCHEME 7

In the course of the total syntheses of (+)-peniciketals A and B, and (-)-diocollettines A, all attempts with TBAF, TBAF buffered in AcOH, HF.pyridine, and acidic conditions with PTSA or CSA in methanol, to remove all four TBS groups of adduct **25** in one single step failed, leading to significant degradation (Scheme 8)[18].

Gold(III)-catalyzed desilylation of the two aliphatic TBS groups followed by a high diastereoselective spiroketalization provided intermediate **26** in 59% yield as a single diastereomer. This later protected benzo-fuzed spiroketal **26** treated with TBAF led to the cleavage of both phenolic TBS groups to afford the key intermediate **27** in 92% yield (360 mg).

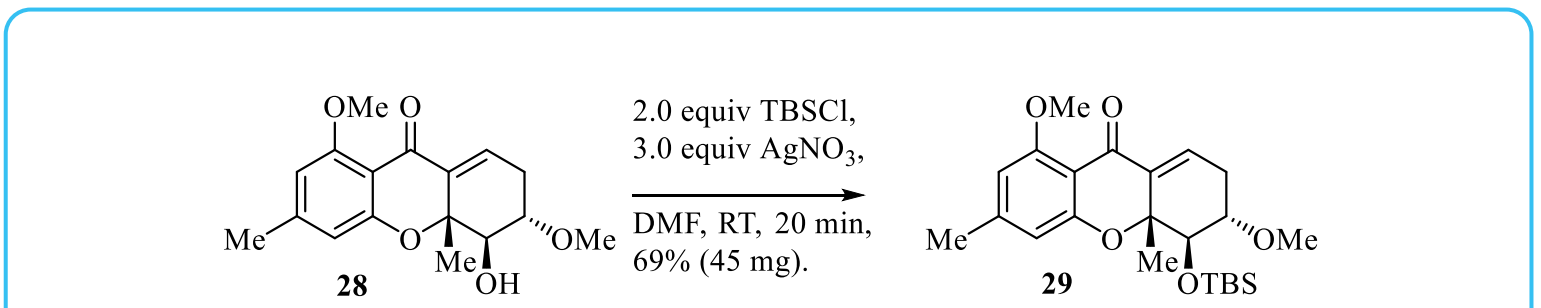
[15] X.-A. Zheng, R. Kong, H.-S. Huang, J.-Y. Wei, J.-Z. Chen, S.-S. Gong, Q. Sun, *Synthesis* **2019**, *51*, 944-952.
[16] Q. Zhang, X. Kanga, L. Longa, L. Zhua, Y. Chai, *Synthesis* **2015**, *47*, 55-64.
[17] C. C. Husic, X. Hu, M. J. Robb, *ACS Macro Lett*. **2022**, *11*, 948-953.
[18] (a) Y. Deng, Y. Zou, C.-P. H. Yang, K. N. Houk, A. B. Smith, *J. Org. Chem*. **2021**, *86*, 13583-13597.
(b) Y. Deng, C.-P. H. Yang, A. B. Smith, *J. Am. Chem. Soc.* **2021**, *143*, 1740-1744.



SCHEME 8

In 1993, Hardinger[19] published an original and efficient silylation procedure of alcohols using TBDPS-Cl and $AgNO^3$ or NH^4NO^3 in DMF, *via in situ* formation of the corresponding silyl nitrate (TBDPS-O-NO_2) as highlighted in the following Schemes 9 and 10. It should be pointed out that trimethylsilyl nitrate in presence of catalytic amount of BF_3OEt_2, in nonpolar solvents, has been described as new nitrating agent.[20]

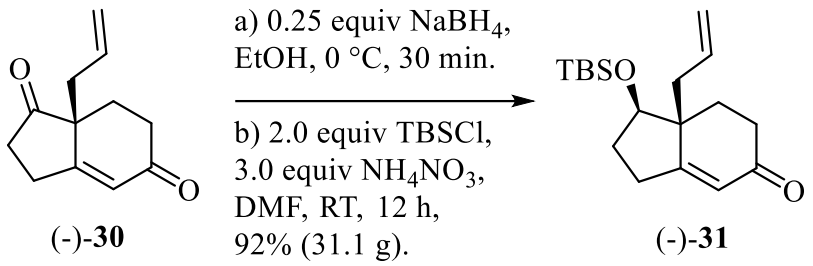
This method has been particularly efficient for the silylation of hindered alcohols, as described in Scheme 9. The protection of the hydroxyl group of **28** using TBS-Cl and $AgNO_3$ in DMF at RT, furnished the silylated intermediate **29** in 69% yield.[21]



SCHEME 9

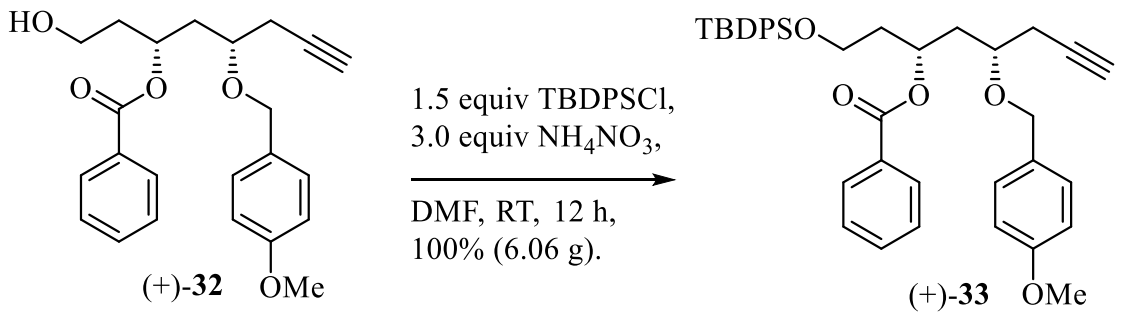
In the first steps of the enantioselective total synthesis of (-)-jiadifenolide, chemo- and diastereoselective reduction of the chiral bicyclic dione 30 with $NaBH_4$ followed by treatment of the resulting crude sterically hindered secondary alcohol with TBS-Cl and NH_4NO_3 provided the desired building block 31 in 92% yield on 0.1 mol scale over the two steps (see Scheme 10).[22]

[19] S. A. Hardinger, N. Wijaya, *Tetrahedron Lett*. **1993**, *34*, 3821-3824.
[20] M. Kimura, K. Kajita, N. Onoda, S. Morosawa, *J. Org. Chem*. **1990**, *55*, 4887-4892.
[21] Z. Xiao, Y. Li, S. Gao, *Org. Lett*. **2017**, *19*, 1834-1837.
[22] J. Xu, L. Trzoss, W. K. Chang, E. A. Theodorakis, *Angew. Chem*. Int. Ed. **2011**, *50*, 3672-3676.



SCHEME 10

Hardinger's method (*vide supra*) has been successfully applied in the total synthesis of fully functionalized ABC ring systems of the unnatural enantiomer of cochleamycin A published by Paquette.[23] To avoid possible 1,5-migration of benzoyl group under basic conditions, silylation of the primary alcohol in intermediate **32** was carried out using TBDPS-Cl and NH_4NO_3 in DMF at room temperature to give after purification on silica gel column the fully protected triol **33** in 100% yield, as presented in Scheme 11.



SCHEME 11

TASF

TASF (tris(dimethylamino)sulfonium difluorotrimethylsilicate) is a crystalline anhydrous commercially available (around 160 €/1.0 g) source of fluoride anion with a bulky non-coordinating counter ion which has been widely used for deprotection of silyl ethers, in particular. It should be pointed out that the sulfonium salt containing dimethylamino groups is sometimes abbreviated as TASF(Me) and its congener with diethylamino groups is denoted TASF(Et). If TASF(Me) is easier to prepare[24] and use, TASF(Et) has greater solubility in organic solvents. A set of examples of TASF-mediated deprotection of base-sensitive substrates is shown in the following Schemes 12-14, showing the efficiency of this reagent over TBAF (NB: TASF(Me) is noted TASF).

[23] L. A. Paquette, J. Chang, Z. Liu, *J. Org. Chem.* **2004**, *69*, 6441-6448..

[24] W. J. Middleton, *Org. Synth.* **1990**, *64*, 221-225.

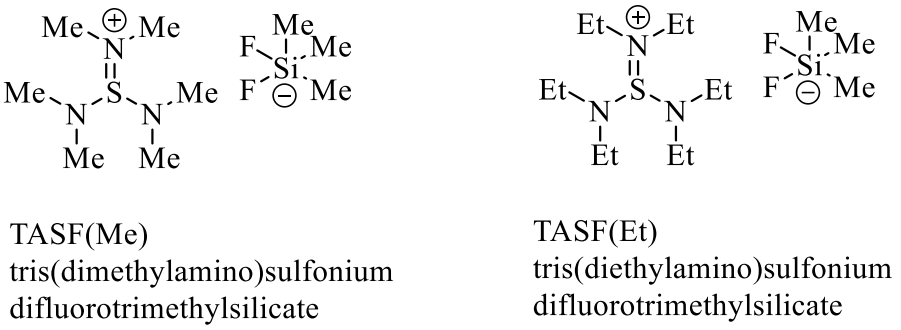
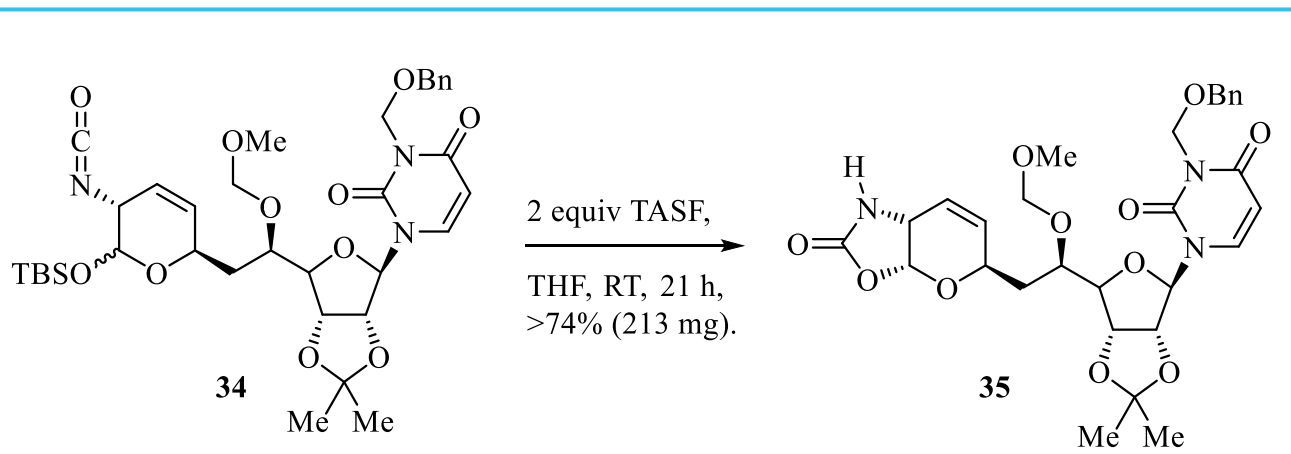


Figure 2. Structure Structures of TASF(Me) and TASF(Et).

In the course of the total synthesis of tunicamycin V, removal of the TBS group adjacent to isocyanate moiety in **34**, upon treatment with TASF in dry THF afforded, by intramolecular cyclization of the resulting alkoxide intermediate, the cyclic carbamate **35** in good yield (Scheme 12).[25]

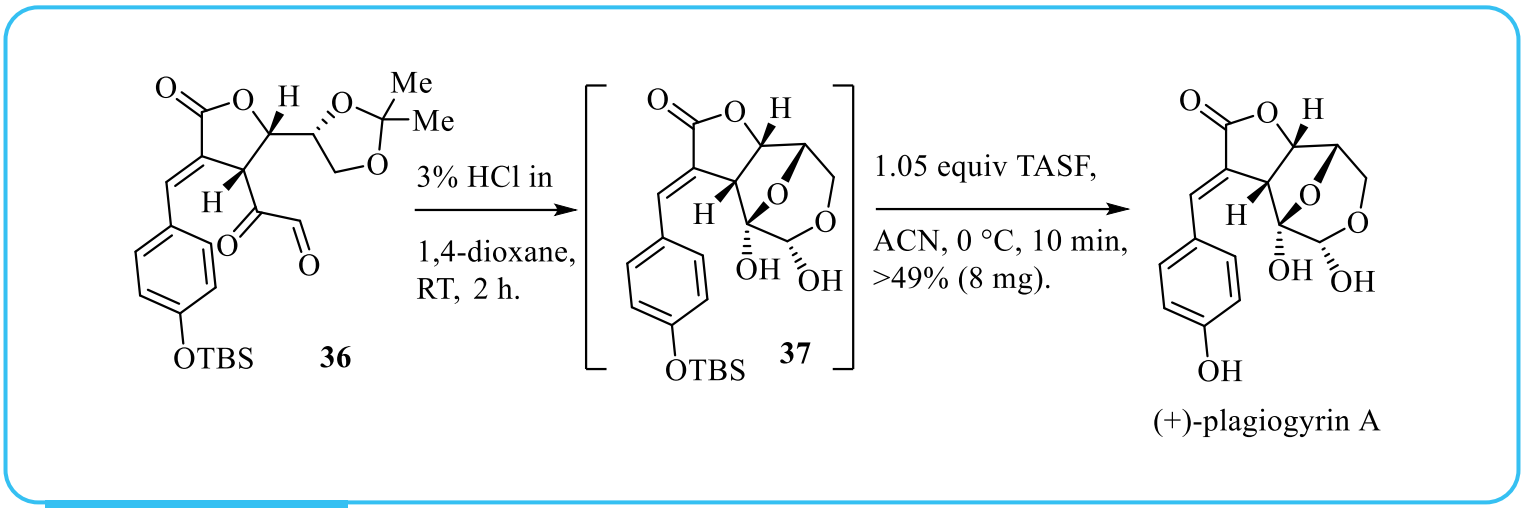


SCHEME 12

At the final stage of the total synthesis of (+)-plagiogyrin A, anhydrous acidic conditions were required to cleave the acetonide group releasing the protected diol which can then form by cyclization the required hemiacetal moiety (see Scheme 13).[26] Treatment of acetonide **36** with 3% HCl in dioxane at room temperature, led to the formation of the desired hemiacetal motif in intermediate **37**, however TBS group remained intact, even upon heating. At this point, various fluoride sources, including TBAF, CsF, and HF·pyridine were evaluated without success. Finally, cleavage of the TBS silyl ether of intermediate **37** using stoichiometric amount of TASF delivered the (+)-plagiogyrin A in correct yield (49% yield for the last three steps).

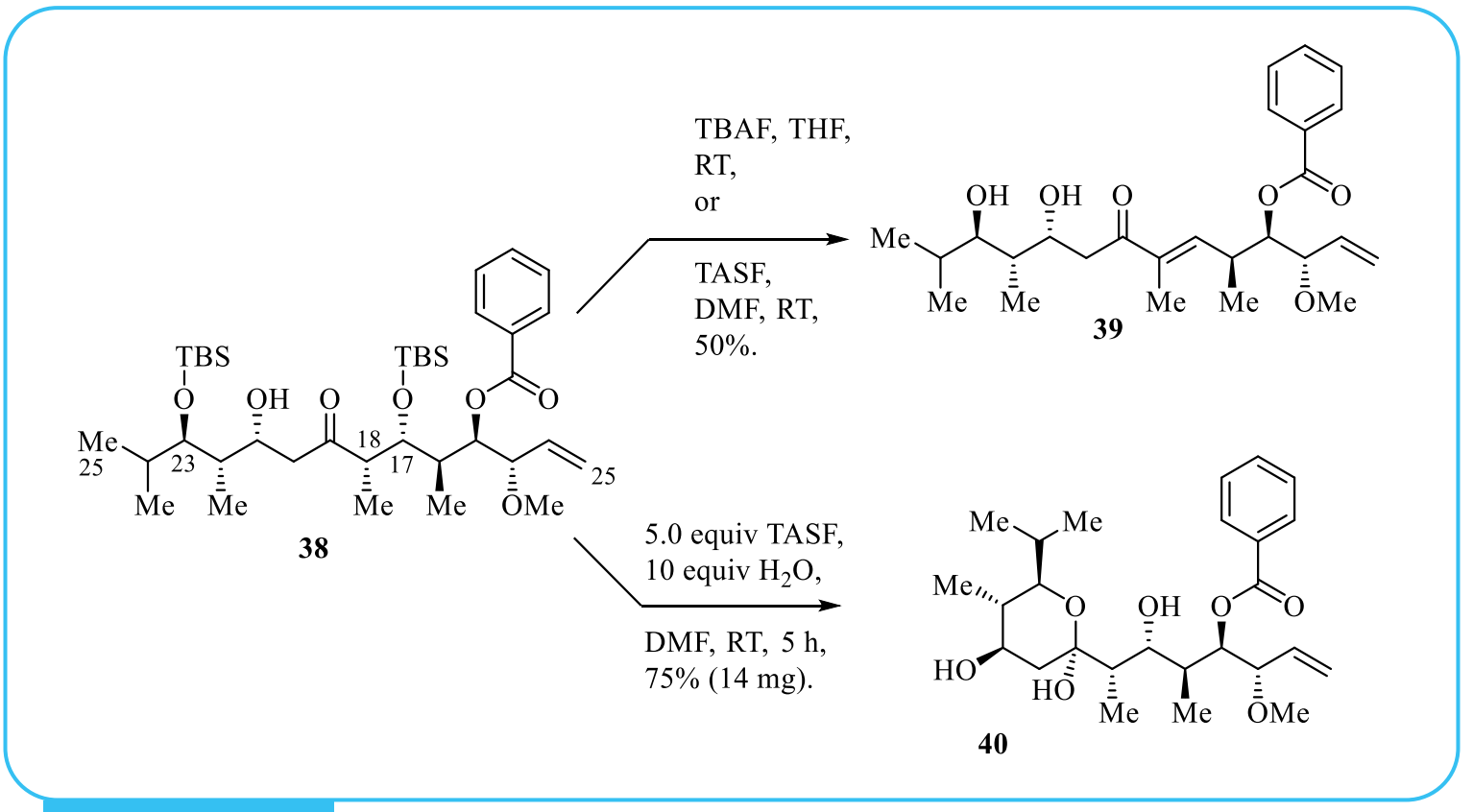
[25] K. Yamamoto, F. Yakushiji, T. Matsumaru, S. Ichikawa, *Org. Lett*. **2018**, *20*, 256-259.

[26] Y. Shi, J. G. Pierce, *Org. Lett*. **2016**, *18*, 5308-5311.



SCHEME 13

During the completion of his elegant total synthesis of (-)-bafilomycin A1, Roush studied the last step on model subunit C14-C25, as depicted in Scheme 14.[27] For the removal of the C17 and C23 TBS ethers, all attempts using HF-pyridine left the starting material **38** unchanged, even over long reaction times (24 hours). While TBAF allowed the isolation of enone **39**, as major adduct, formed by concomitant deprotonation at C18, adjacent to carbonyl, and then elimination of the C(17)-OTBS group. Essentially the same results, as observed with TBAF, were noticed when the cleavage was performed with TSAF in anhydrous DMF. However, using these later conditions with addition of water, provided the desired hemiketal **40** in 75% yield (14 mg). These previous optimized experimental conditions for complete deprotection of the silyl protecting groups were applied with success, leading to (-)-bafilomycin A1 in 93% yield (3.7 mg) (not shown).

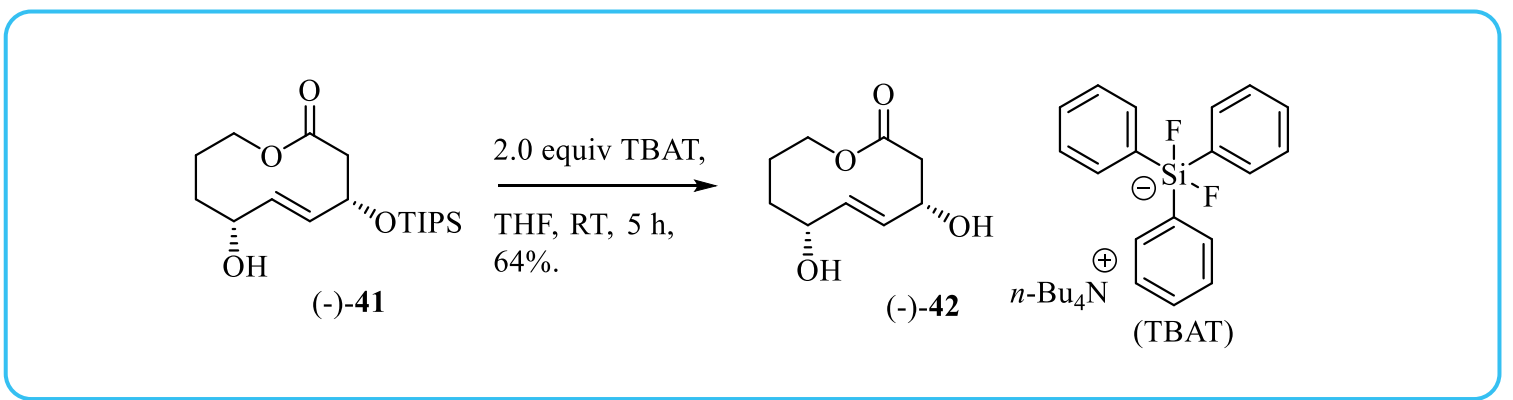


SCHEME 14

[27] K. A. Scheidt, T. D. Bannister, A. Tasaka, M. D. Wendt, B. M. Savall, G. J. Fegley, W. R. Roush, *J. Am. Chem. Soc* **2002**, *124*, 6981-6990.

TASF should be regarded as an excellent alternative to TBAF for the mild deprotection of base-sensitive silyl ethers, with the possibility of adding water as a protic additive to attenuate the nucleophilicity and basicity of this reagent.[28]

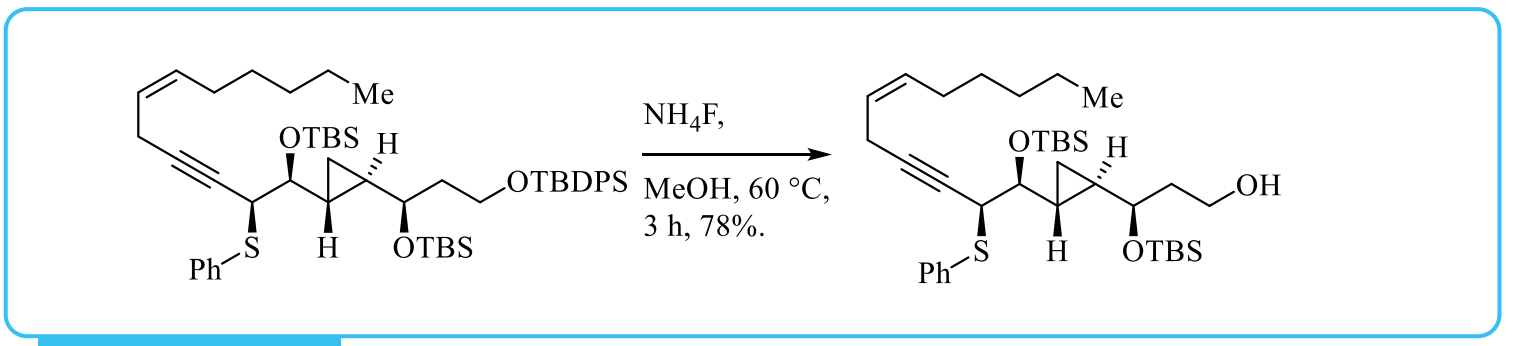
To end this part, it should be noted that tetrabutylammonium triphenyldifluorosilicate (TBAT, 1.0 M solution in methanol around 100 mL/80 €) has been reported by DeShong as anhydrous non-hygroscopic fluoride source.[12a] However, to the best of our knowledge, this reagent has been rarely used to cleave silyl ethers. Nevertheless, an example is described in Scheme 15, in which the removal of the sterically bulky TIPS silyl ether group in intermediate **41** was performed with TBAT to afford the novel decanolide **42** in 64% yield.[29] In contrast, when using TBAF the yield dropped significantly to around 24%.



SCHEME 15

NH_4F

Treatment of silyl ethers with NH_4F in methanol is an economical and practical alternative to TBAF.[30] In any case, it should be kept in mind that with TBAF-mediated silyl deprotection, removal of the tetrabutylammonium cation during the work-up, mainly due to its phase transfer properties, can be difficult and complicated.[31] Also, in a very interesting way, upon exposure to NH_4F in methanol, primary silyl ethers such as TBS or TBDPS are more labile than the TBS group on secondary alcohols. This chemoselective silyl deprotection of primary silyl ethers in the presence of secondary silyl ethers has been successfully used in total synthesis of complex natural products as reported in Schemes 16-18.



SCHEME 16 [32]

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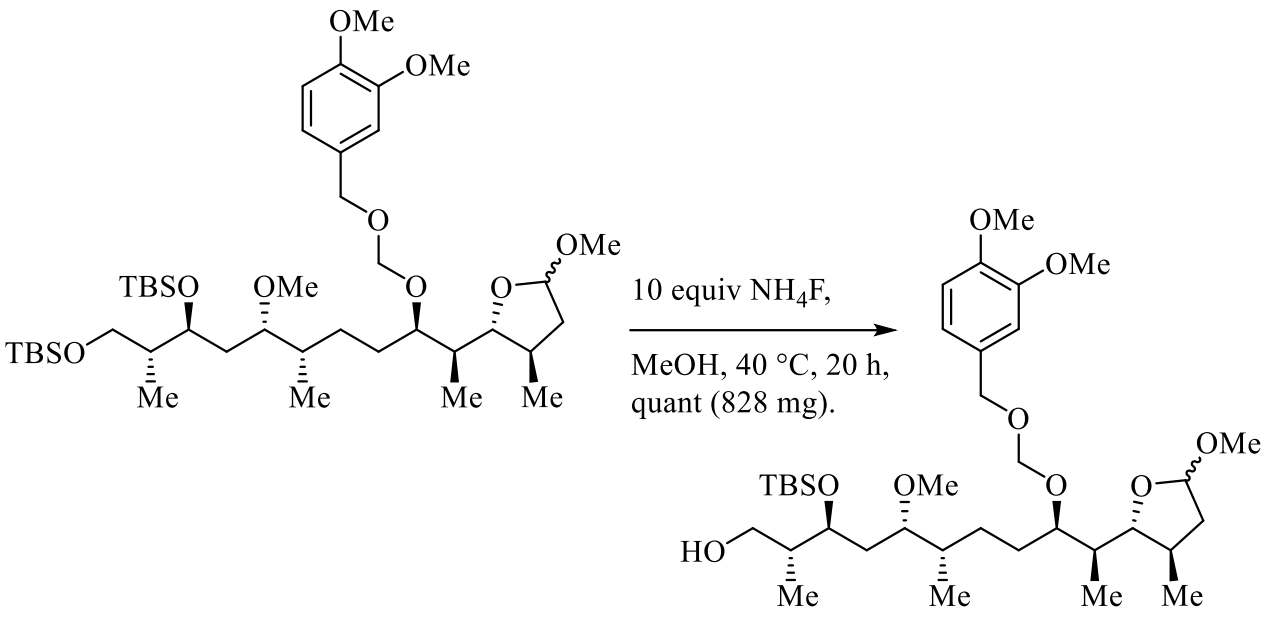
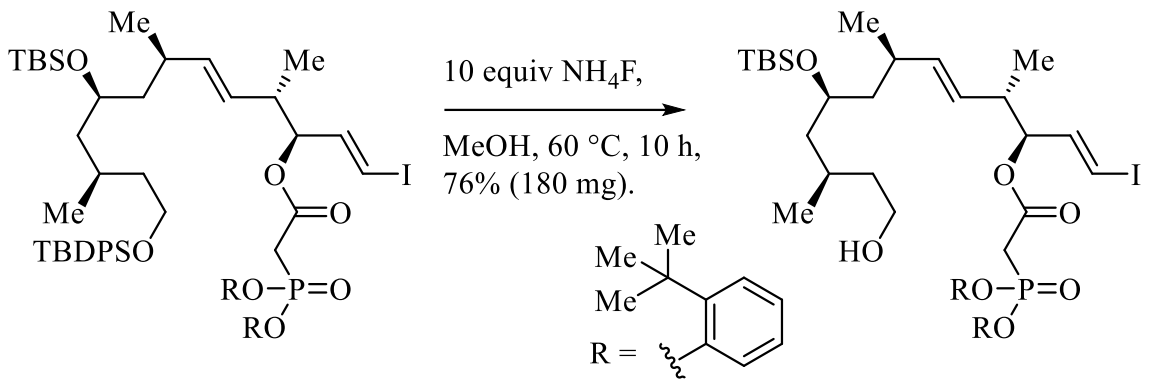
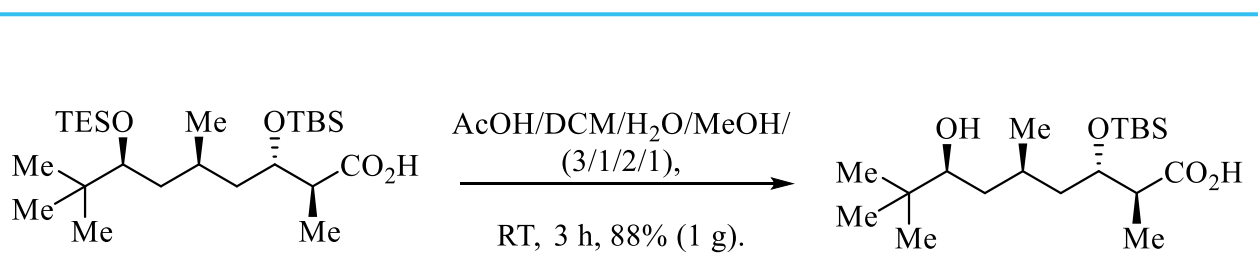


SCHEME 17 [33]



SCHEME 18 [34]

Removal of silyl ethers under acidic conditions can be mediated by various Brønsted or Lewis acids. In general, the rate of these cleavages is faster for less sterically encumbered silyl ethers, as presented in Scheme 19 in the course of a supported synthesis of oxoapratoxin A.[35]



SCHEME 19

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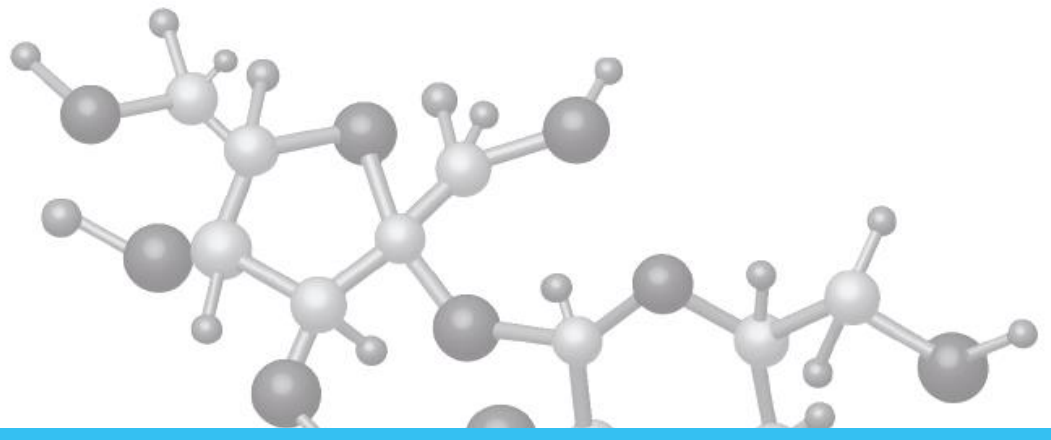
Conclusion

Trisubstituted silyl ether protecting groups will continue to provide organic synthesis, in particular the synthesis of complex molecules, with effective solutions.

Acknowledgement

I would like to thank Dr Ronan Gealageas for fruitful discussions and comments. I also warmly thank my colleagues, Dr. Sylvie Archambaud, Dr. Aude Vibert, Dr. Thibaut Martinez and Dr. Thomas Glachet from AtlanChim Pharma for their valuable suggestions during the preparation of this manuscript. A special thanks to Lionel Bidet for linguistic assistance.



AtlanChim Pharma

PORTRAIT

Thibaut MARTINEZ, Chef de Projet chez Atlanchim Pharma depuis 2020 est titulaire d'un diplôme d'ingénieur de l'Ecole Nationale Supérieur de Chimie de Paris, d'un master en Chimie Moléculaire de l'Université Pierre et Marie Curie ainsi que d'un Doctorat en chimie organique de la Sorbonne Université à Paris.

Lors d'une première expérience en société de prestation de services avant de rentrer chez ATLANCHIM PHARMA, il a acquis un savoir-faire en synthèse des dérivés biologiquement actifs.

Au sein d'Atlanchim Pharma, nos chimistes ont l'opportunité de travailler sur une grande diversité de molécules et de réactions chimiques, mais grâce à sa thèse, Thibaut possède une spécialisation en chimie organométallique et en catalyse asymétrique à Au (I).

N'hésitez pas à nous contacter, nous serons ravis de pouvoir répondre à vos sollicitudes.

Thibaut MARTINEZ, Project Manager at Atlanchim Pharma since 2020, holds an engineering degree from the Ecole Nationale Supérieur de Chimie de Paris, a master's degree in Molecular Chemistry from the Pierre et Marie Curie University and a PhD in Organic Chemistry from the Sorbonne University in Paris.

Before joining ATLANCHIM PHARMA, he had a first experience in a CRO company, where he acquired a know-how in the synthesis of biologically active derivatives.

At Atlanchim Pharma, our chemists have the opportunity to work on a wide variety of molecules and chemical reactions, but thanks to his thesis, Thibaut has a specialization in organometallic chemistry and asymmetric Au (I) catalysis.

Feel free to contact us, we will be delighted to answer your requests.