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o-Nitrophenyl selenocyanate, a valuable reagent in organic synthesis: Application to one of the most powerful routes to terminal olefins from *prim*-alcohols (the Grieco–Sharpless olefination reaction) and to the regioselective isomerisation of allyl alcohols

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Summary — *o*-Nitrophenyl selenocyanate allows the transformation, in neutral media, of alcohols into *o*-nitrophenyl selenides (the Grieco reaction). On oxidation, alkyl *o*-nitrophenyl selenides produce olefins (the Sharpless olefination reaction) whereas allylic compounds undergo a smooth [2,3] sigmatropic shift that leads to 'rearranged' allyl alcohols. Because of its mildness, efficiency and chemoselectivity, the olefination reaction has proved to be the best method so far described for the synthesis of terminal C,C double bonds from primary alcohols (the Grieco–Sharpless reaction).

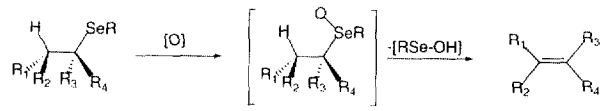
alcohol / alkyl selenide / olefin synthesis / oxidation / selenoxide / allyl selenide / [2,3] sigmatropic rearrangement / allyl alcohol synthesis

Résumé — Le sélénocyanate d'*o*-nitrophényle, un réactif précieux en synthèse organique. Le sélénocyanate d'*o*-nitrophényle permet la transformation en milieu neutre d'alcools en *o*-nitrophenyl séléniures (réaction de Grieco). Les alkyl *o*-nitrophenyl séléniures sont aisément transformés, après oxydation, en oléfines (réaction de Sharpless) tandis que les dérivés allyliques subissent un réarrangement sigmatropique [2,3] qui engendre des alcools allyliques. L'utilisation des deux premières réactions, permet de synthétiser des oléfines terminales à partir d'alcools primaires dans des conditions particulièrement douces. Cette suite de réactions est de loin la meilleure disponible actuellement pour effectuer cette transformation.

alcool / alkyl séléniure / synthèse d'oléfine / oxydation / sélénoxide / séléniure allylique / réarrangement sigmatropique [2,3] / synthèse d'alcool allylique

Introduction

Although organoselenium chemistry is almost as old as organic chemistry it is only about twenty years ago that organoselenium reagents became used routinely for the transformation of organic compounds [1–9]. Two barriers had to be overcome: the novelty and the bad reputation organoselenium compounds had. (i) The first members of the series had a very bad smell (nevertheless not so different from their sulfur analogs) and (ii) organoselenium compounds were presumed to be highly toxic [10], although it was already well established that the toxicity of a compound depends usually on its whole structure and not on the presence of a specific atom (Se) at any place in the molecule. It became rapidly evident that some organoselenium reagents possess unique properties and at the same time it was found that selenium is required as a trace element in many living organisms. For example glutathione peroxidase, which plays a crucial role in the protection of mammals against radicals, possesses four molecules of selenocysteine at its active site [4, 11]. Selenium dioxide was in fact for long the only selenium reagent commonly used by organic chemists since the early forties [12–14]. The recognition that alkyl selenoxides possess a very high propensity to produce olefins in the seventies encourages chemists to use this reaction for the synthesis of complex molecules (scheme 1) [15–19].



Scheme 1

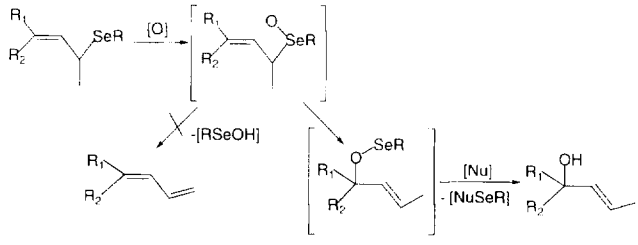
The selenoxide elimination reaction takes usually place on alkyl phenylselenoxides and involves the *syn* β-elimination of phenylselenenic acid which occurs at around 20 °C under very mild, non-basic, conditions. These are much milder than those of related sulfoxides

* Correspondence and reprints

and aminoxides which require a much higher temperature (160 °C and 220 °C respectively) [19, 20]. The reaction is less efficient when it is expected to produce a strained olefin or with primary alkyl selenides which possess a high propensity to be hydrated. In such cases the presence of an electron-withdrawing group, such as the *o*-nitro one, on the aromatic ring, proved to greatly facilitate the synthesis of the olefin [21, 22].

The reaction involving *o*-nitrophenyl selenoxides proved to be particularly valuable for the synthesis of terminal olefins because (i) only one type of hydrogen is available for the elimination and therefore it is regiospecific and does not involve stereochemical problems, (ii) other methods which require primary alkyl halides or sulfonates and a base suffer dramatically from competitive substitution reactions.

Allylic selenoxides behave differently and usually have a higher propensity to rearrange to allyl alcohols via a [2,3]-sigmatropic shift than to produce dienes via the elimination reaction reported above (scheme 2). Again the *o*-nitrophenyl selenoxides possess a much higher propensity to rearrange than, for example, the related phenyl derivatives.



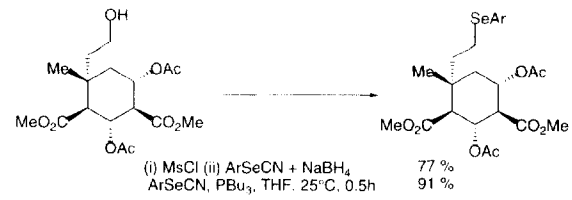
Scheme 2

The seleno moiety is not a functional group often encountered in organic molecules. Alkyl selenides can nevertheless be synthesized from commercially available selenium reagents by substitution of an alkyl halide, an alkyl sulfonate or an alcohol. They can be readily oxidized chemoselectively to the corresponding selenoxides by a large array of oxidants in the presence of a series of other functional groups, which may be, but are not oxidized in the presence of a selenide. Since we have shown that the *o*-nitrophenylseleno group offers several advantages over the others, we would like to focus our discussion specifically on it and show how *o*-nitrophenyl selenides are produced and how they can be transformed to olefins and allyl alcohols.

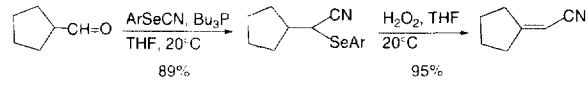
o-Nitrophenyl selenocyanate (ArSeCN, Ar = *o*-NO_2Ph, [51694-22-5], MW 228.088, Mp = 139–141 °C) is in fact the most valuable reagent to introduce the *o*-nitrophenylseleno moiety in organic molecules. It is a stable, easily handled, commercially available, light brown crystalline solid which reacts with:

– sodium borohydride in ethanol or DMF to produce almost quantitatively sodium *o*-nitrobenzeneselenolate (scheme 3) [21, 22] or *o*-nitrobenzeneselenol on acid hydrolysis ((i) $NaBH_4$, EtOH, 20 °C, (ii) excess $HClO_4$) [23];

– aldehydes to deliver *α*-(*o*-nitrophenylseleno)nitriles in good yields (76–90%, scheme 4) [24];



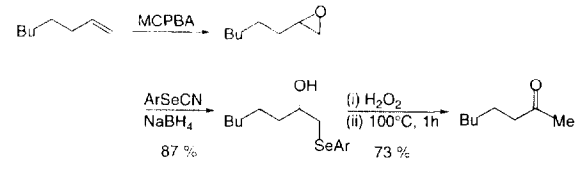
Scheme 3



Scheme 4

– carboxylic acids to produce selenol esters in modest yields (30%) [25].

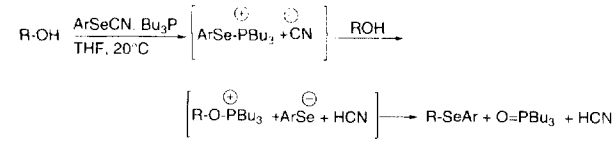
Sodium *o*-nitrobenzeneselenolate is a good nucleophile which reacts efficiently with (i) alkyl halides and alkyl sulfonates to produce the corresponding *o*-nitrophenyl selenides in reasonably good yields (60–90%, scheme 3) [21, 22], and (ii) epoxides to provide *β*-hydroxyalkyl selenides, precursors of methyl ketones (scheme 5) [26].



Scheme 5

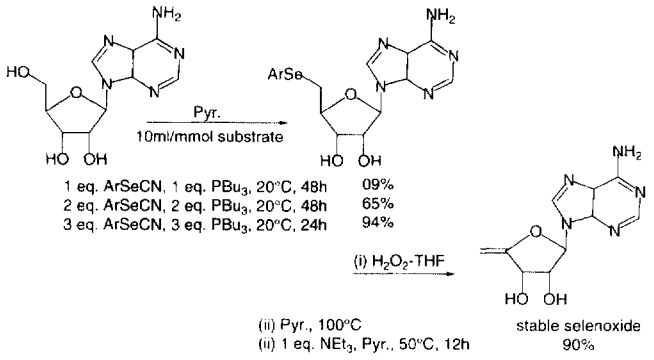
The Grieco reaction

Nevertheless the most easy way to synthesize *o*-nitrophenyl selenides is without doubt the one, discovered by Grieco, which involves the reaction of alcohols with *o*-nitrophenyl selenocyanate in the presence of tributyl phosphine (ArSeCN, PBu_3, THF, 20 °C, 2.5 h), the mechanism of which is presented in scheme 6. It is faster and often more efficient than the selenolate method, reported above, which requires the transformation of the hydroxyl group of the alcohol to a better leaving group (scheme 3) [21].



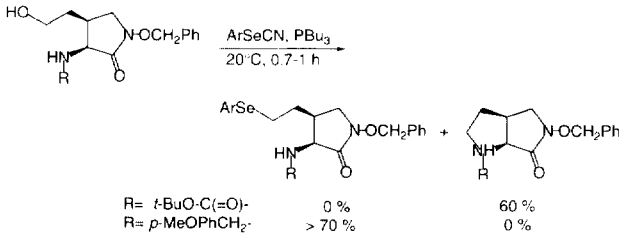
Scheme 6

The Grieco reaction is usually performed by mixing, in THF or better in pyridine [27–29], the alcohol with a slight excess of reagents (1.1 equiv ArSeCN and 1.1 equiv PBu_3). In some difficult cases, use of an excess of reagents (1.5 to 3 equiv ArSeCN and PBu_3) [29] (scheme 7) proved to be beneficial. The reaction has been successfully achieved on primary (the great majority) and secondary alcohols [21] belonging to the



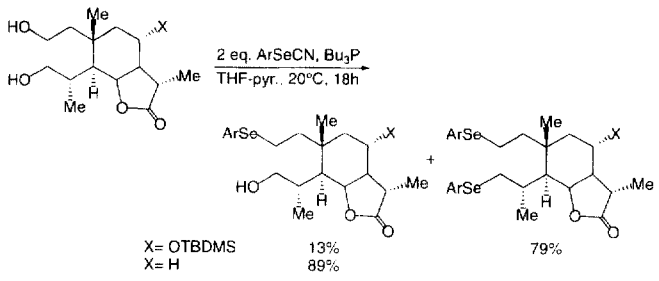
Scheme 7

saturated [21, 30], benzylic [21] and allylic [28, 31–33] series. It is quite general and only in a very few cases deviation occurs. It has for example once been reported that a five- [34] (scheme 8) or a seven-membered [35] heterocycle is formed instead of the expected selenide, by trapping the intermediate alkoxy phosphonium salt by an internal nucleophile placed in a suitable position. Even so, the reaction proceeds efficiently if the nucleophilicity of that group is lowered (scheme 8) [34]. The reaction is highly chemoselective and has been achieved on primary alcohols in the presence of (i) another primary alcohol whose side chain is more alkyl-substituted on the β-carbon (scheme 9) [35–37] or (ii) secondary [29, 38, 39] (scheme 7) or tertiary [40, 41] (scheme 10) alcohols as well as their protected forms such as TBDMS ethers (scheme 9) [37, 42, 43]. The reaction proceeds also very selectively on primary alcohols leaving unaffected the amino (NH_2) group of adenine (scheme 7) [29] or Boc-protected amino groups (scheme 8) [34], acetals [44] or thioacetals [45], esters (scheme 3) [21, 43] and lactones (scheme 9) [35, 37, 46], olefins [31, 35, 37, 42, 46, 47], epoxides [38, 42], enamides [48], indoles [36] and a carbamate function [49]. A competing reaction has however been reported once between primary and *sec*-allyl alcohols [50].

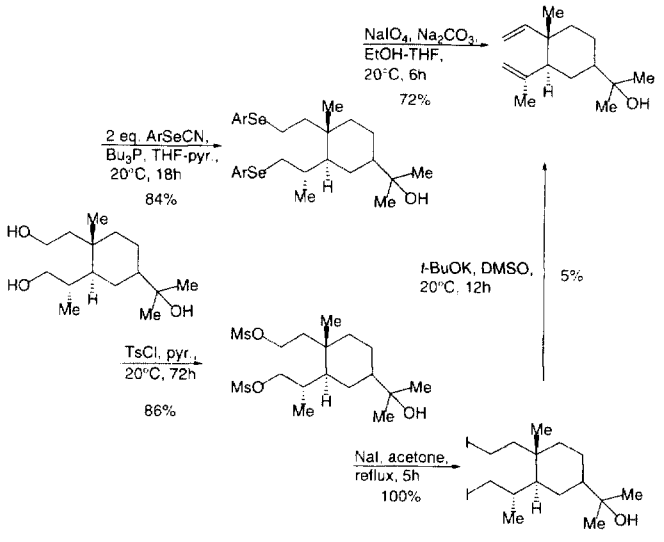


Scheme 8

Reaction of the diols, described in scheme 10, with equimolar amount [35] or with an excess of reagent (up to 4-fold) [46] usually leads to the formation of the corresponding bis-alkylselenides. These have then been transformed to dienes on a further selenoxide formation–elimination reaction [35, 46]. In a few cases, however, small changes on the basic skeleton lead to the formation of a seven-membered cyclic ether (1 equiv ArSeCN and 1 equiv Bu_3P) or to the exclusive production of the least hindered primary alkyl selenide [35]. In such cases the desired dienes have been obtained by



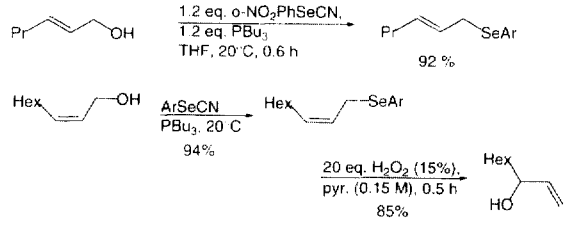
Scheme 9



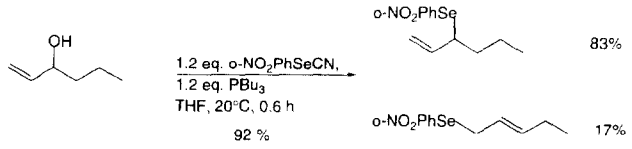
Scheme 10

stepwise reactions which involve (i) production of one of the two selenides and elimination of this seleno moiety via the usual oxidation–elimination reaction and then repeating the whole process on the remaining alcohol [35].

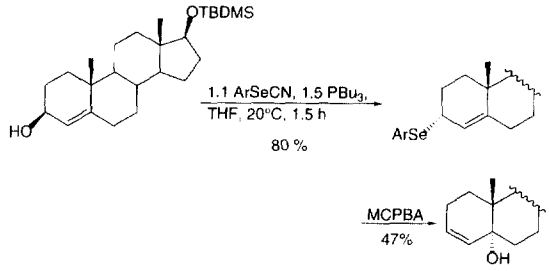
The Grieco reaction is completely regioselective with allyl alcohols (scheme 11) [28, 31, 32] except with those bearing a terminal C,C double bond [33]. The latter ones lead to a mixture of allyl selenides in which those resulting from direct substitution prevail (scheme 12) [33]. In general substitution occurs on the carbon bearing the hydroxyl group, maintaining the stereochemical integrity of the C,C double bond (scheme 11) [28, 31] and proceeding stereospecifically, in the case of secondary alcohols with complete inversion of the configuration at the substituted carbon (scheme 13) [32, 51].



Scheme 11



Scheme 12



Scheme 13

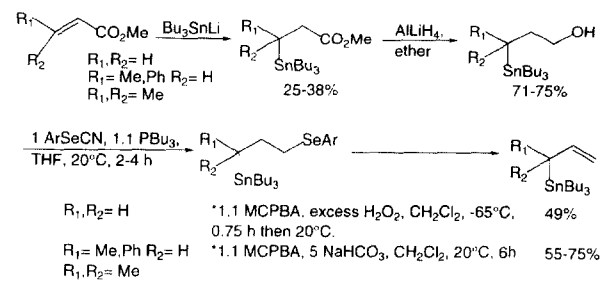
Synthesis of olefins and allyl alcohols from *o*-nitrophenyl selenides

Alkyl and allyl selenides are valuable precursors of olefins and allyl alcohols, respectively (scheme 1) [3, 52–56]. These transformations take advantage of the facile formation of the corresponding selenoxides (hydrogen peroxide, ozone, *m*-chloroperbenzoic acid or sodium periodate, 20 °C, 1–4 h) and the aptitude of: (i) alkyl selenoxides to eliminate selenenic acid (around 20 °C, *syn* elimination) to produce olefins, and (ii) allyl selenoxides to rearrange to the corresponding selenenate, around 20 °C, via a [2,3] sigmatropic shift. The *o*-nitrophenylselenoxy moiety proved to be by far better than the other selenoxy groups for these purposes [21, 22].

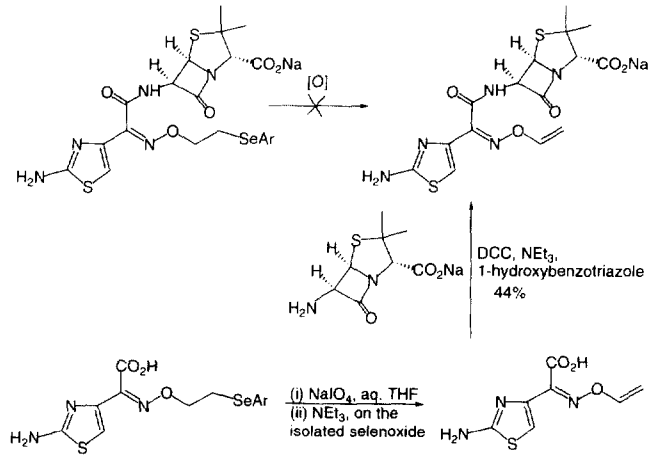
The Sharpless reaction: the synthesis of olefins

The oxidation of alkyl *o*-nitrophenyl selenides has mainly been achieved with hydrogen peroxide [21, 22, 24, 26, 27, 29, 34, 35, 37, 40, 42–47, 50, 57–59] as well as with *tert*-butyl hydroperoxide [60], ozone [60, 61], *m*-chloroperbenzoic acid [30, 36, 38] or sodium metaperiodate in alkaline media (usually 0–20 °C, 1–4 h) [41, 48, 62]. In one case concomitant use of *m*-chloroperbenzoic acid and hydrogen peroxide in methylene chloride at −65 °C was required (scheme 14) [30] and none of the oxidants used was able to selectively oxidize the selenide present on the penicillin derivative shown in scheme 15 [63]. The synthesis of the desired compound has nevertheless been achieved successfully by an alternative route, which still uses the 'Grieco-Sharpless olefination reaction' as depicted in scheme 15 [63].

The elimination reaction is particularly facile and usually occurs very rapidly in the reaction medium at room temperature. In some cases the selenoxide first formed is decomposed by heating in the presence of DBU [60] or triethylamine [29, 61] (scheme 7). Use of pyridine instead of triethylamine does not at least in one case allow the olefin synthesis [29]. Anyhow the 'Grieco–Sharpless olefination reaction' permits the synthesis of

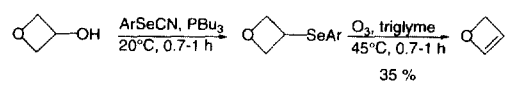


Scheme 14



Scheme 15

a large variety of olefins including strained and very unstable ones such as the parent unsubstituted oxete (scheme 16) [60], allyl stannanes (scheme 14) [30] and homoallyl stannanes [30], *O*-vinyl oximes (scheme 15) [63], enol ethers [29, 61] and enols which immediately rearrange to the corresponding carbonyl compounds (scheme 5) [26]. In some of the most difficult cases the elimination reaction requires 45 °C (scheme 16) [60] or even 100 °C (scheme 5) [26] to proceed. The selenoxide elimination reaction is usually not regioselective [30] except with primary alkyl selenides which produce terminal olefins.



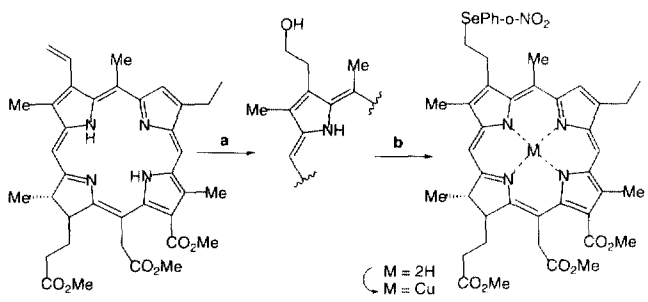
Scheme 16

The 'Grieco-Sharpless olefination reaction' takes advantage of the different features already disclosed. It allows the synthesis of terminal olefins from primary alcohols via the corresponding *o*-nitrophenyl selenides. It uses very mild, neutral conditions, is highly chemoselective and replaces the more conventional transformations, which require the use of strong bases on related primary alkyl halides. The latter reactions are often incompatible with the presence of other functional groups. A specific comparison of the two methods is illustrated in scheme 10 [41].

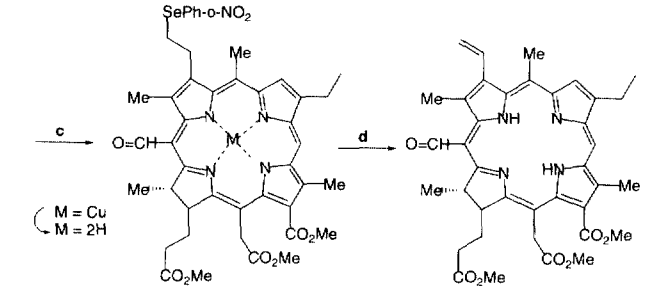
The 'Grieco-Sharpless olefination reaction' has been successfully used:

– as a key step in a large number of syntheses of complex natural products (over 50) especially those of: terpenes and related derivatives such as (+)-temisin [37, 46] and (+)-melitensin [46], vernolepin and vernomenin [50], (*R*)-santolinatriene [47], (±)-aphidicolin and (±)-β-chamigrene [64], β-elemol [41], (+)-costunolide [35], sativene [27], verrucarol [38], isocomene [62], linalyl oxides [31], germacranes pheromone (±)-periplanone-B [42], pseudo guaianolides aromatin and confertin [44]; alkaloids such as decahydroquinoline (+)-*trans*-219A [49]; (−)-stenine [59]; yomhimbinoid (±)-antirhine [36] or lycorine [48]; steroid estradiol [45] and vitamin D3 metabolites [43]; milbemycin spiroketals [58]; glycine/NMDA antagonist [34]; asteltoxin [40]; nucleosides [29, 61] and erythomycin [57] antibiotics as well as for the synthesis of functionalized porphyrins [65].

– as a key step in the protection of vinyl groups in porphyrin synthesis [65]. The set of reactions described in scheme 17 formally allows the selective formylation of the porphyrin nucleus without competing formylation on the vinyl group. It involves, at the protection stage, the synthesis of a primary alkyl aryl selenide by chemoselective hydroxylation of the vinylic moiety using thallium nitrate, followed by Grieco's reaction using *o*-nitrophenyl selenocyanate [65]. After introduction of the formyl group, the deprotection stage uses the Sharpless selenoxide elimination reaction which is carried out with hydrogen peroxide (scheme 17) [65]. This transformation is by far more efficient than the one involving a β-elimination reaction on the related primary alkyl chloride. The latter process often produces a base-promoted cyclized product instead (scheme 18).

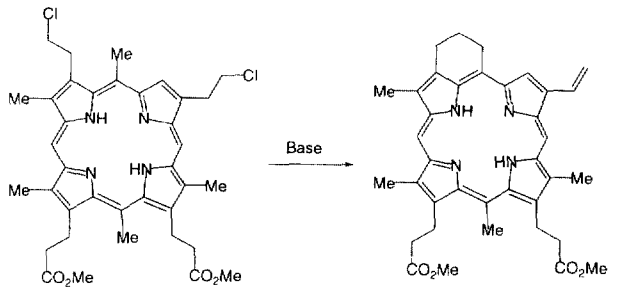


a : (i) $TlNO_2$ (ii) HCl (iii) CH_2N_2 (4) $NaBH_4$, **b** : *o*-NO_2PhSeCN, *n*-Bu_3P, CH_2Cl_2, 20°C, 0.5h
c : $Cu(OAc)_2$, **d** : (i) DMF-$POCl_3$ (ii) $NaHCO_3$, **e** : c H_2SO_4-TFA, **f** : H_2O_2



Scheme 17

– for the synthesis of α,β-unsaturated nitriles from aldehydes, which involves a one-carbon elongation [24] (scheme 4) and in a synthesis of methyl ketones from terminal olefins (scheme 5) [26] which has been applied to the synthesis of sugar derivatives [26].



Scheme 18

Synthesis of allyl alcohols

Allyl *o*-nitrophenyl selenides are efficiently oxidized to the corresponding *o*-nitrophenyl selenoxides [28, 31, 32] using hydrogen peroxide [28, 31] or *m*-chloroperbenzoic acid [32]. These allylic selenoxides are very unstable and rearrange *in situ* to allyl alcohols with very high regio- and stereocontrol (schemes 11, 13) [28, 32]. This reaction has been successfully used for the contrathermodynamic isomerisation of alk-2-enols to alk-1-enols (scheme 11) [28]. Epoxidation of remote double bonds catalyzed by the perseleninic acid formed in the medium have once been reported when the reaction is performed with excess hydrogen peroxide on allyl selenides bearing other C,C double bonds on the side chain (30% aqueous H_2O_2, CH_2Cl_2-pyr, 20 °C, 13 h) [31].

Typical procedures

Synthesis of alkyl o-nitrophenyl selenides from alcohols [21]

A solution of dodecanol (0.62 mmol, 0.84 equiv) in 2.0 mL of THF containing *o*-nitrophenyl selenocyanate (0.74 mmol, 1 equiv) under nitrogen was treated dropwise with tri-*n*-butylphosphine (0.74 mmol, 1 equiv) at room temperature. After the reaction was stirred for 0.5 h, the solvent was removed in vacuo leading to the desired dodecyl *o*-nitrophenyl selenide (94% yield).

Synthesis of olefins from selenides (scheme 10) [41]

$NaIO_4$ (0.3 mmol, 3 equiv) was added, with vigorous stirring, to a solution of selenide (0.2 mmol, 1 equiv) in a mixture of methanol, THF, water (0.9 mL), $NaHCO_3$ (3 equiv) maintained at 20 °C. After 6 h at that temperature, the reaction mixture was evaporated in vacuo, then diluted with saturated $NaHCO_3$ (5 mL) and extracted with ether (3 × 10 mL). The organic layer was washed with water, NaCl solution, and dried ($MgSO_4$). Usual work-up leads to the olefinic compound in 72% yield.

Synthesis of allyl alcohols from allyl o-nitrophenyl selenides (scheme 11) [32]

The allyl selenide (0.4 mmol) and *m*-chloroperbenzoic acid (0.2 mmol, 1 equiv) were reacted in THF at 0 °C for

0.2 h. It produces, after quenching the reaction medium with aqueous K_2CO_3, the allyl alcohol (47%).

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