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# PROTECTIVE GROUPS IN ORGANIC SYNTHESIS

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THIRD EDITION

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and

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Pharmacia and Upjohn Company



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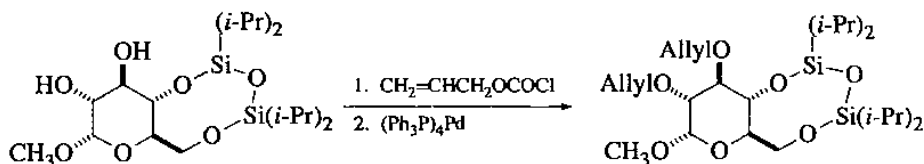
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#### Allyl Ether (Allyl-OR): $\text{CH}_2=\text{CHCH}_2\text{-OR}$ (Chart 1)

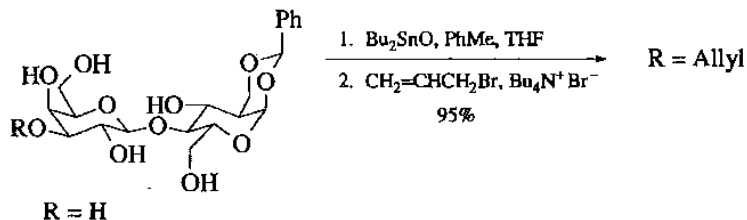
The use of allyl ethers for the protection of alcohols is common in the literature on carbohydrates because allyl ethers are generally compatible with the various methods for glycoside formation.<sup>1</sup> Obviously, the allyl ether is not compatible with powerful electrophiles such as bromine and catalytic hydrogenation, but it is stable to moderately acidic conditions (1 *N* HCl, reflux, 10 h).<sup>2</sup> The ease of formation, the many mild methods for its cleavage in the presence of numerous other protective groups, and its general stability have made the allyl ether a mainstay of many orthogonal sets. The synthesis of perdeuteroallyl bromide and its use as a protective group in carbohydrates has been reported. The perdeutero derivative has the advantage that the allyl resonances in the NMR no longer obscure other, more diagnostic resonances, such as those of the anomeric carbon in glycosides.<sup>3</sup> The use of the allyl protective group primarily covering carbohydrate chemistry has been reviewed.<sup>4</sup>

#### Formation

1.  $\text{CH}_2=\text{CHCH}_2\text{Br}$ , NaOH, benzene, reflux, 1.5 h,<sup>5</sup> or NaH, benzene, 90–100% yield.<sup>6</sup>
2.  $\text{CH}_2=\text{CHCH}_2\text{OC(=NH)CCl}_3$ ,  $\text{H}^+$ .<sup>7</sup>
3. Allyl carbonates have been converted to allyl ethers.<sup>8</sup> In the following situation, acid- and base-catalyzed procedures failed because of the sensitivity of the  $[(i\text{-Pr})_2\text{Si}]_2\text{O}$  group.

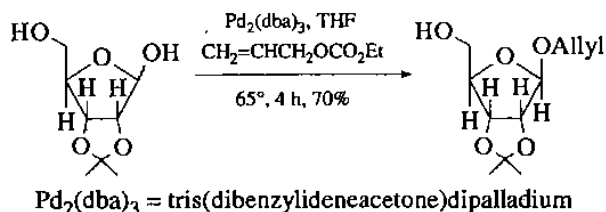


4.  $\text{Bu}_2\text{SnO}$ , toluene, THF;  $\text{CH}_2=\text{CHCH}_2\text{Br}$ ,  $\text{Bu}_4\text{N}^+\text{Br}^-$ , 96% yield.<sup>9</sup>



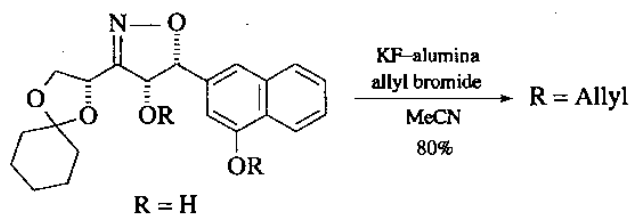
The crotyl ether has been introduced using similar methodology.<sup>10</sup>

5.  $\text{CH}_2=\text{CHCH}_2\text{OCO}_2\text{Et}$ ,  $\text{Pd}_2(\text{dba})_3$ , THF,  $65^\circ$ , 4 h, 70–97% yield.<sup>11</sup>



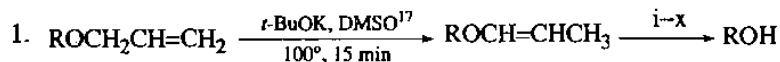
Note the preferential reaction at the anomeric hydroxyl. The method is also effective for the protection of primary and secondary alcohols.

6.  $\text{MeO}_2\text{COCH}_2\text{CH}=\text{CH}_2$ ,  $\text{Pd}_2(\text{dba})_3$ ,  $\text{CHCl}_3$ ,  $\text{Ph}_2\text{P}(\text{CH}_2)_4\text{PPh}_2$ , THF,  $65^\circ$ , 95% yield.<sup>12</sup>
7. Allyl bromide,  $(\text{RO})_2\text{Mg}$ .<sup>13</sup>
8. KF–alumina, allyl bromide, 80% yield. These conditions were developed because the typical strongly basic metal alkoxide-induced alkylation led to Beckmann fragmentation of the isoxazoline.<sup>14</sup>



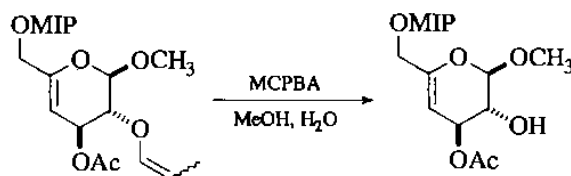
9. Allyl bromide, DMF, BaO, rt.<sup>15</sup>
10. Allyl bromide,  $\text{Al}_2\text{O}_3$ , days. These conditions were developed to alkylate selectively an alcohol in the presence of an amide.<sup>16</sup>

### Cleavage



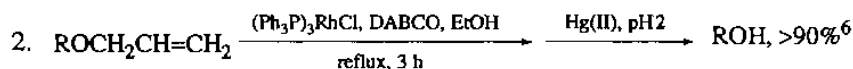
- i. 0.1 N HCl, acetone–water, reflux, 30 min.<sup>8</sup>
- ii.  $\text{KMnO}_4$ ,  $\text{NaOH-H}_2\text{O}$ ,  $10^\circ$ , 100% yield. These basic conditions avoid acid-catalyzed acetonide cleavage.<sup>2</sup>
- iii.  $\text{HgCl}_2/\text{HgO}$ , acetone– $\text{H}_2\text{O}$ , 5 min, 100% yield.<sup>18</sup>
- iv. Ozonolysis.<sup>17,19</sup>

- v.  $\text{SeO}_2$ ,  $\text{H}_2\text{O}_2$ , 92% yield.<sup>20</sup>
- vi. 0.1 eq.  $\text{TsOH}$ ,  $\text{MeOH}$ ,  $25^\circ$ , 2.5 h, >86% yield.<sup>21</sup>
- vii.  $\text{Me}_3\text{NO}$ ,  $\text{OsO}_4$ ,  $\text{CH}_2\text{Cl}_2$ , >76% yield.<sup>22</sup>
- viii.  $\text{MCPBA}$ ,  $\text{MeOH}$ ,  $\text{H}_2\text{O}$ .<sup>23</sup>



When the OAc group was a hydroxyl, the epoxidation selectivity was not very good, presumably because of the known directing effect of hydroxyl groups in peracid epoxidations.

- ix.  $\text{NIS}$ ,  $\text{CH}_2\text{Cl}_2$ ,  $\text{H}_2\text{O}$ .<sup>24</sup>
- x.  $\text{BF}_3 \cdot \text{Et}_2\text{O}$ ,  $\text{Bu}_4\text{N}^+\text{F}^-$ ,  $0^\circ$ , 52–88% yield.<sup>25</sup>

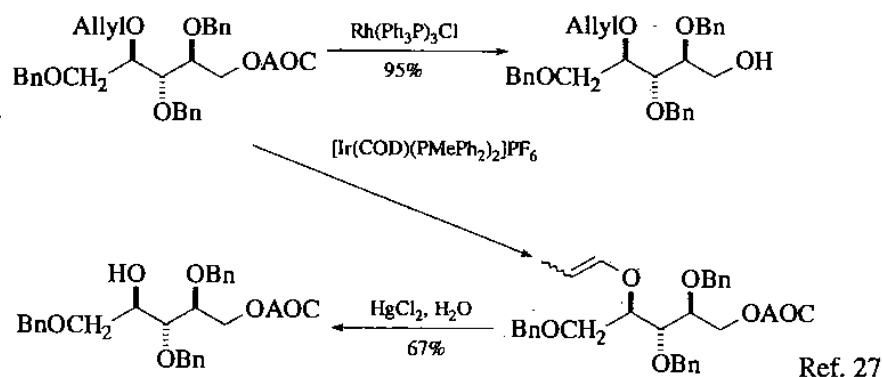


Allyl ethers are isomerized by  $(\text{Ph}_3\text{P})_3\text{RhCl}$ , and  $t\text{-BuOK/DMSO}$  in the following order:<sup>26</sup>

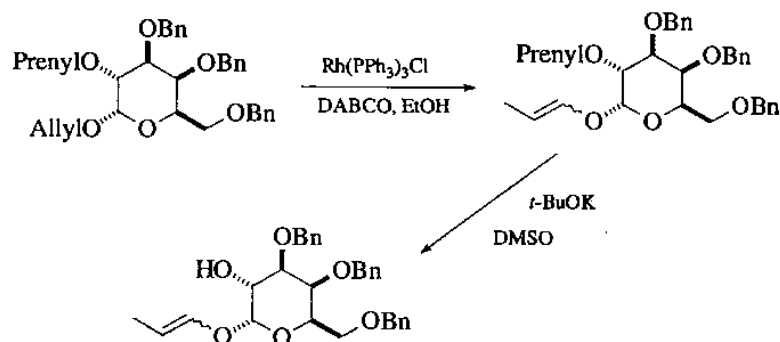
$(\text{Ph}_3\text{P})_3\text{RhCl}$ : allyl > 2-methylallyl > but-2-enyl

$t\text{-BuOK}$ : but-2-enyl > allyl > 2-methylallyl

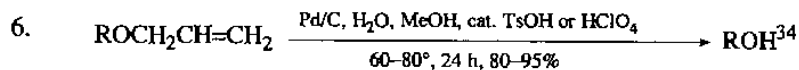
3. It is possible to remove the allyl group in the presence of an allyloxycarbonyl (AOC, Alloc, or Aloc) group using an  $[\text{Ir}(\text{COD})(\text{Ph}_2\text{MeP})_2]\text{PF}_6$ -catalyzed isomerization, but the selectivity is not complete. The allyloxy-carbonyl group can be removed selectively in the presence of an allyl group using a palladium or rhodium catalyst.<sup>27</sup> Hydrogen-activated  $[\text{Ir}(\text{COD})(\text{Ph}_2\text{MeP})_2]\text{PF}_6$  is a better catalyst for allyl isomerization (91–100% yield) because there is no reduction of the alkene, as is sometimes the case with  $(\text{Ph}_3\text{P})_3\text{RhCl}$ .<sup>28,29</sup> When Wilkinson's catalyst is pre-reduced with  $\text{BuLi}$ , alkene reduction is not observed and high yields of enol ethers are obtained.<sup>30</sup> This method can be used for isomerization of but-2-enyl ethers.<sup>31</sup> The iridium catalyst is also compatible with acetylenes.<sup>32</sup> Because the iridium catalyst can effect isomerization at room temperature, adjacent azides do not cycloadd to the allyl group during the isomerization reaction, as is the case when the isomerization must be performed at reflux.<sup>22</sup>



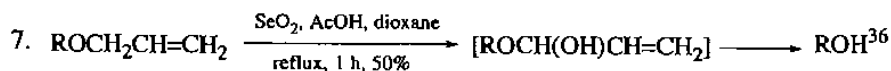
4. Useful selectivity between allyl and 3-methylbut-2-enyl (prenyl) ethers has been achieved.<sup>26</sup>



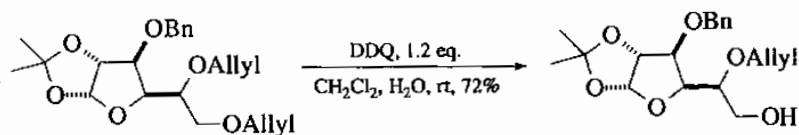
5. *trans*-Pd(NH<sub>3</sub>)<sub>2</sub>Cl<sub>2</sub>/*t*-BuOH isomerizes allyl ethers to vinyl ethers that can then be hydrolyzed in 90% yield, but in the presence of an  $\alpha$ -hydroxy group the intermediate vinyl ether cyclizes to an acetal.<sup>33</sup> Benzylidene acetals are not affected by this reagent.



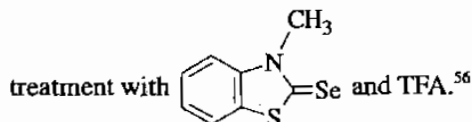
When TsOH is omitted, the reaction gives the vinyl ether.<sup>35</sup>



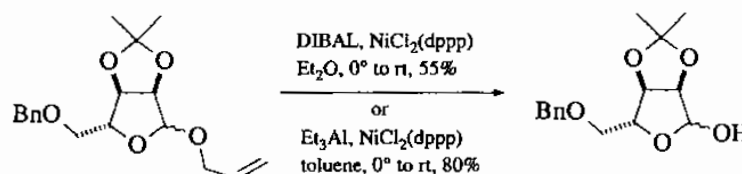
8.  $\text{ROCH}_2\text{CH}=\text{CH}_2 \xrightarrow[\text{reflux, 2 h, 85-99\%}]{\text{NaTeH, EtOH, AcOH}} \text{ROH}^{37}$
9.  $\text{Ac}_2\text{O}$ ,  $\text{BF}_3 \cdot \text{Et}_2\text{O}$ , then  $\text{MeONa/MeOH}$  to hydrolyze the acetate.<sup>38</sup>
10.  $\text{RhCl}_3$ , DABCO, EtOH,  $\text{H}_2\text{O}$ ;  $\text{H}_3\text{O}^+$ , EtOH.<sup>39</sup>
11.  $\text{RhH}(\text{Ph}_3\text{P})_4$ , TFA, EtOH,  $50^\circ$ , 30 min, 98% yield.<sup>40</sup>
12.  $\text{PdCl}_2$ ,  $\text{CuCl}$ , DMF,  $\text{O}_2$ , 4 h, rt, 88-93% yield.<sup>41</sup>
13.  $\text{Pd}(\text{Ph}_3\text{P})_4$ , AcOH,  $80^\circ$ , 10-60 min, 72-98% yield.<sup>42</sup>
14.  $\text{TiCl}_3$ , Mg, THF, 28-96% yield.<sup>43</sup>
15. Electrolysis, DMF,  $\text{SmCl}_3$ ,  $(n\text{-Bu})_4\text{N}^+\text{Br}^-$ , Mg anode, Ni cathode, 60-90% yield.<sup>44</sup>
16. Electrolysis,  $[\text{Ni}(\text{bipy})_3](\text{BF}_4)_2$ , Mg anode, DMF, rt, 25-99% yield.<sup>45</sup> Aryl halides are reduced.
17.  $\text{AlCl}_3$ - $\text{PhNMe}_2$ ,  $\text{CH}_2\text{Cl}_2$ , 73-100% yield.<sup>46</sup> Benzyl ethers are also cleaved.
18. NBS,  $h\nu$ ,  $\text{CCl}_4$ ; base, 78-99% yield.<sup>47</sup>
19.  $\text{CoCl}_2$ , AcCl,  $\text{CH}_3\text{CN}$ , rt, 8-12 h, 71-84% yield. Benzyl ethers are among those that are also cleaved.<sup>48</sup>
20.  $\text{Cp}_2\text{Zr}$ ;  $\text{H}_2\text{O}$ , 50-98% yield. Allyl ethers are cleaved faster than allyl amines that are also cleaved (66%).<sup>49</sup>
21.  $\text{H}_2\text{Ru}(\text{PPh}_3)_4$ , EtOH,  $95^\circ$ ; 1.5 h, TsOH, MeOH, 2.5 h, 86% yield.<sup>21</sup>
22. Other catalysts that have been used to isomerize allylic ethers are Polystyrene- $\text{CH}_2\text{NMe}_3$ - $\text{RhCl}_4$  (EtOH,  $\text{H}_2\text{O}$ ),<sup>50</sup>  $\text{RuCl}_2(\text{PPh})_3$  ( $\text{NaBH}_4$ , EtOH),<sup>51</sup>  $\text{Rh}(\text{diphos})(\text{acetone})_2[\text{ClO}_4]_2$  (acetone,  $25^\circ$ ),<sup>52</sup> and  $\text{Fe}(\text{CO})_5$  (xylene,  $135^\circ$ , 8-15 h, 97% yield).<sup>53</sup>
23.  $\text{PdCl}_2$ , AcOH,  $\text{H}_2\text{O}$ , NaOAc, 89% yield.<sup>19</sup>
24.  $\text{Ti}(\text{O-}i\text{-Pr})_4$ ,  $n\text{-BuMgCl}$ , THF, rt, 69-97% yield. Methallyl and other substituted allyl ethers are not cleaved, but ester groups are partially removed, as expected.<sup>54</sup>
25. DDQ, wet  $\text{CH}_2\text{Cl}_2$ , 70-92% yield. Anomeric and secondary allylic ethers could not be cleaved under these conditions.<sup>55</sup>



26. Protection for the double bond in the allyl protecting group may be achieved by epoxidation. Regeneration of the allyl group occurs upon



27. Allyl groups are subject to oxidative deprotection with Chromiapiillared Montmorillonite Clay, *t*-BuOOH, CH<sub>2</sub>Cl<sub>2</sub>, isooctane, 85% yield.<sup>57</sup> Allylamines are cleaved in 84–90% yield, and allyl phenyl ethers are cleaved in 80% yield.
28. NaBH<sub>4</sub>, I<sub>2</sub>, THF, 0°, 53–96% yield.<sup>58</sup>
29. Pd(Ph<sub>3</sub>P)<sub>4</sub>, RSO<sub>2</sub>Na, CH<sub>2</sub>Cl<sub>2</sub> or THF/MeOH, 70–99% yield. These conditions were shown to be superior to the use of sodium 2-ethylhexanoate. Methallyl, crotyl, and cinnamyl ethers, the Alloc group, and allylamines are all efficiently cleaved by this method.<sup>59</sup>
30. DIBAL, NiCl<sub>2</sub>(dppp), toluene, CH<sub>2</sub>Cl<sub>2</sub>, THF, or ether, 80–97% yield.<sup>60</sup> These conditions are chemoselective for simple alkyl and phenolic allyl ethers. More highly substituted allyl ethers are unreactive.



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#### Propargyl Ethers: $\text{HC}\equiv\text{CCH}_2\text{OR}$

##### Cleavage

1. Propargyl ethers are cleaved with  $\text{TiCl}_3\text{-Mg}$  in THF, 54–92% yield. Allyl and benzyl ethers were not cleaved; phenolic propargyl ethers are also cleaved.<sup>1</sup>
2.  $(\text{BnNEt}_3)_2\text{MoS}_4$  (benzyltriethylammonium tetrathiomolybdate).<sup>2</sup>

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#### *p*-Chlorophenyl Ether: $p\text{-ClC}_6\text{H}_4\text{-OR}$

##### Formation/Cleavage<sup>1</sup>

