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SYNTHETIC APPLICATIONS OF ORGANOTELLURIUM CHEMISTRY

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Abstract Newly developed applications of organotellurium chemistry, including carbon-carbon bond-forming reactions and functional group interconversions, are discussed.

INTRODUCTION

Organic tellurium compounds have a history dating back to 1840 when Wöhler prepared diethyl telluride, the first organic compound of tellurium. During the next 130 years many investigations were performed which enriched the knowledge in the organotellurium area. However, until 1970 most of the studies were concerned with chemical, physical and structural properties of organotellurium compounds. Very few attempts were made to develop organotellurium reagents of possible utility in organic synthesis.

During the last fifteen years the search for synthetic applications of organotellurium compounds has benefitted by developments in related fields, especially that of organoselenium chemistry. It soon became clear that there were both similarities and dissimilarities in the chemical behaviour of organic compounds of the two elements. When the tellurium chemistry has been pursued simply as an extension of well-established organoselenium chemistry the results have often (but not always) been disappointing in terms of synthetic applications. However, when the unique behaviour of tellurium in some of its reactions has been recognized and taken advantage of, some quite spectacular organic transformations have resulted.

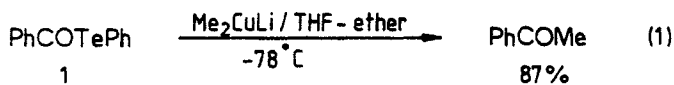
The newly developed applications of organotellurium chemistry include carbon-carbon bond forming reactions as well as various types of functional group interconversions. Two review articles are already available on the earlier aspects of the topic^{1,2} one of them² including also experimental procedures for the preparation of the most commonly used organotellurium reagents.

In the following the most recent (2-3 years) synthetic applications of organotellurium chemistry will be discussed.

CARBON-CARBON BOND-FORMING REACTIONS

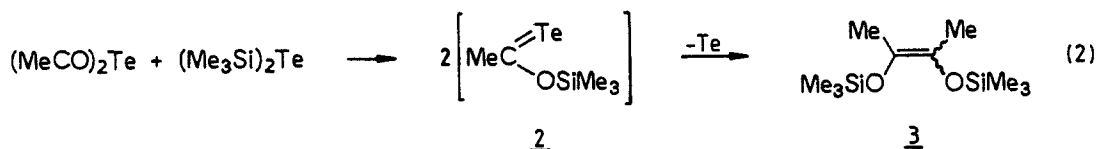
Early work in the field of organotellurium chemistry has produced a variety of readily available organotellurium compounds in which tellurium is bonded to one, two, three or four carbon atoms. Obviously, it should be possible for the inventive synthetic chemist to design methods and reagents which transform the weak carbon-tellurium bonds of these materials into new useful C-C bonds. However, the last few years have seen surprisingly little development along these lines.

Tellurol ester **1** has been found to undergo a smooth substitution reaction with lithium dialkyl cuprates to produce the corresponding ketone in high yield (eq. 1)³. The required tellurol ester was conveniently obtained from the reaction of phenyltelluro-trimethylsilane with benzoyl chloride.

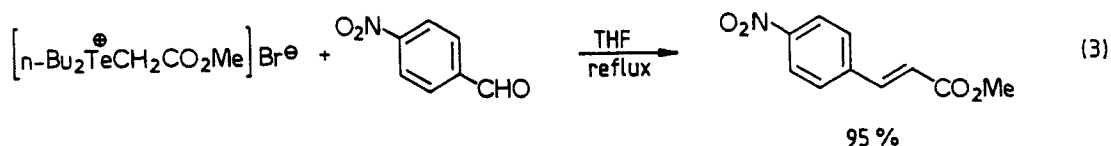


When diacetyl telluride was allowed to react with bis(trimethylsilyl)telluride without solvent at -20 °C, the mixture rapidly turned intense blue and elemental tellurium started to separate out (eq 2).⁴ After 12-15 h at room temperature an E/Z-mixture of olefin **3** was isolated in good yield. The intermediacy of a telluroester **2** with a carbon-tellurium doublebond has been suggested and is supported by mass spectrometry. With sterically more demanding

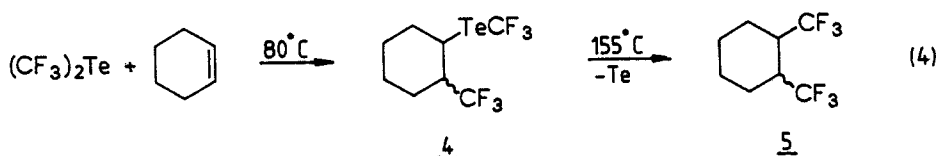
diacyl tellurides (e.g. dipivaloyl telluride) the corresponding telluroester was stable with no sign of tellurium extrusion.



Telluronium ylides such as dialkyltelluronium carbethoxymethylide have previously been used for the synthesis of α, β -unsaturated esters from aldehydes at low temperature in tetrahydrofuran. In a further development of this reaction⁵ it was shown that even telluronium salts undergo condensation reactions with certain carbonyl compounds in neutral medium at elevated temperature (eq.3).

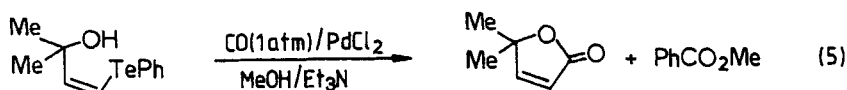


A comparative study of the trifluoromethylating properties of $(\text{CF}_3)_2\text{Hg}$, CF_3I and $(\text{CF}_3)_2\text{Te}$ showed that the tellurium compound was far superior to the other reagents in both the thermal and the photochemical reaction.⁶ Cyclohexene underwent addition to produce the telluride 4 at 80 °C in the thermal process (eq.4). At elevated temperature the chalcogen atom was efficiently extruded to afford only the trifluoromethylated cycloalkane 5.



In an attempt to use the weak carbon-tellurium bond of organotelluriums for carbon monoxide insertion reactions, various aryl- and alkenyltellurium compounds were converted to their corresponding carboxylic acids by treatment with CO in the presence of a stoichiometric amount of a palladium(II)-salt.⁷ Improved yields of carboxylic acid methyl esters from aryl-, alkenyl- and alkynyltellurium compounds were recently obtained⁸ by using the

system PdCl_2 /methanol/triethylamine. The reaction was also extended to a butenolide synthesis as shown in eq.5. In the presence of a suitable reoxidant (CuCl_2), most of the reactions were catalytic in PdCl_2 . Furthermore, the formation of methyl benzoate which was always a side-reaction in the stoichiometric process was essentially suppressed in the catalytic one. Aryl- and alkenyl sulfides are totally inactive under the present carbonylation conditions and the corresponding organoselenium compounds react very sluggishly.



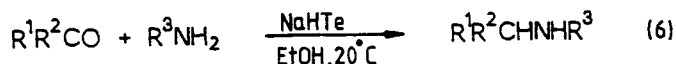
It was recently reported that when alkyl phenyl tellurides are treated with excess *m*-chloroperbenzoic acid (MCPBA) in alcohol solvents, they undergo a substitution reaction with displacement of the PhTe -moiety to afford dialkyl ethers. The reaction is sometimes accompanied by phenyl migration as demonstrated in the synthesis of 2-arylpropanoic acids from the ethylene glycol acetals of α -phenyltelluropropiophenones.⁹

FUNCTIONAL GROUP INTERCONVERSIONS

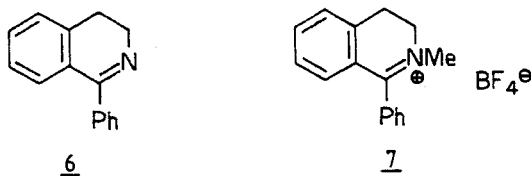
An overwhelming majority of recently reported tellurium-mediated functional group manipulations involve oxidation or reduction reactions. Although some of the reagents used are inorganic and do not always involve an organotellurium intermediate in their reactions (NaHTe , Na_2Te , TeO_2) they have been included in this article. The tellurium-based oxidants and reducing agents are all very mild, thus making them highly selective for certain types of transformations.

Tellurium in the oxidation state -II has been used for a variety of reduction reactions.¹ Depending on the pH of the solution, the reductant is present as H_2Te , HTe^- or Te^{2-} ($\text{pK}_{a1} = 2.64$; $\text{pK}_{a2} = 11.80$ for H_2Te^{10}). Yamashita¹¹ has recently demonstrated that carbonyl compounds can be reductively aminated to yield secondary

amines in the presence of NaHTe (eq.6). This reaction was further



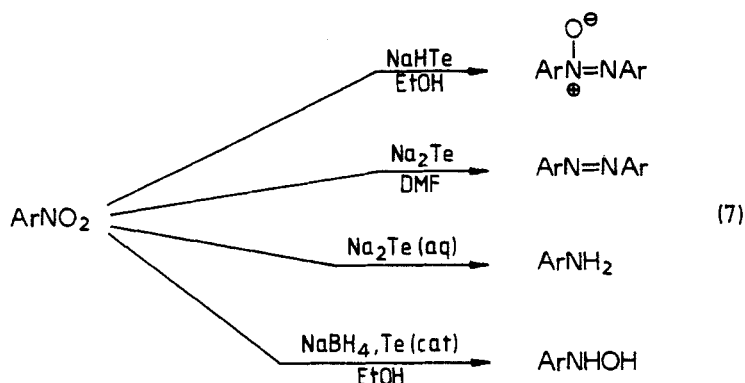
studied by Barton and coworkers¹² who demonstrated the importance of pH control with the reagent. Under standard NaHTe-conditions¹³ (EtOH, pH 6) the imine 6 was smoothly reduced to a secondary amine, but at pH 10-11 no reaction occurred. Apparently, the protonated imine is the reducible species in this and the Yamashita reaction. The iminium salt 7 was reduced to a tertiary amine at all pH.



N-oxides were reduced to the corresponding amines by NaHTe at pH 6¹⁴ and nitrones were similarly reduced by NaHTe to imines at alkaline pH and to sec.-amines at pH 6.

Previous work with NaHTe has shown that the reagent is capable of reducing nitrobenzenes to azoxybenzenes (eq.7). Sodium telluride, prepared by heating tellurium and sodium hydride in a 1:2 molar ratio in dry DMF has now been found to reduce a variety of aromatic nitro compounds selectively to azo compounds.¹⁵ On the other hand, sodium telluride generated in alkaline aqueous medium from Rongalite ($HOCH_2SO_2Na \cdot 2H_2O$) was found to reduce aromatic nitro compounds to anilines without any indication of the formation of intermediate reduction products (eq.7).¹⁶ Polynitro compounds were reduced with similar ease to polyamines. When two nitro groups were ortho or para to each other, the reduction could be stopped at the nitroamine stage by controlling the amount of reducing agent. A future study of the behaviour of aqueous Na_2Te towards azobenzene, nitrosobenzene and N-phenylhydroxylamine, respectively, would hopefully indicate if these compounds are intermediates in the reduction of nitrobenzenes to anilines.

Surprisingly, N-arylhydroxylamines were obtained in



synthetically useful yields when aromatic nitro compounds were treated at ambient temperature with sodium borohydride and a catalytic amount of tellurium in ethanol (eq.7).¹⁷ The identity of the actual reducing species (NaHTe or Na_2Te) is not known for this process. When NaOH was added to create basic conditions where Na_2Te should clearly predominate, *N*-phenylhydroxylamine was obtained in 60% yield from nitrobenzene.

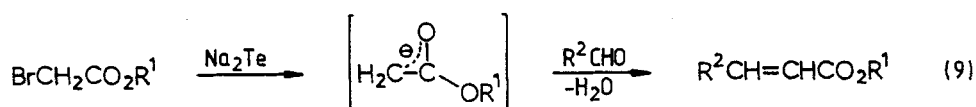
Alkyl and aryl azides are known to be mildly reduced to the corresponding amines by a reagent formulated as NaHTe .¹⁸ However, since the reagent was prepared by heating elemental tellurium with 2.4 eq. of NaBH_4 in ethanol (without acidification as described in the original preparation of NaHTe ¹³), Na_2Te might very well be the actual reductant in the process. Nevertheless, in an extension of the earlier findings a number of α -azidoketones were reduced to α -aminoketones which underwent self-condensation during workup to afford pyrazines.¹⁹

When tertiary nitro compounds were treated with the same reductant, a rapid displacement of the nitro group was observed (e.g. 8a+8b). This reductive removal was not restricted to nitro groups at the α -position of electron withdrawing groups, but occurred also with β -nitrocarbonyl compounds and α -nitrocumene derivatives (compounds 9 and 10, respectively).²⁰ However, tertiary nitro groups situated γ to a ketone, ester or nitrile functionality were untouched by the reagent.

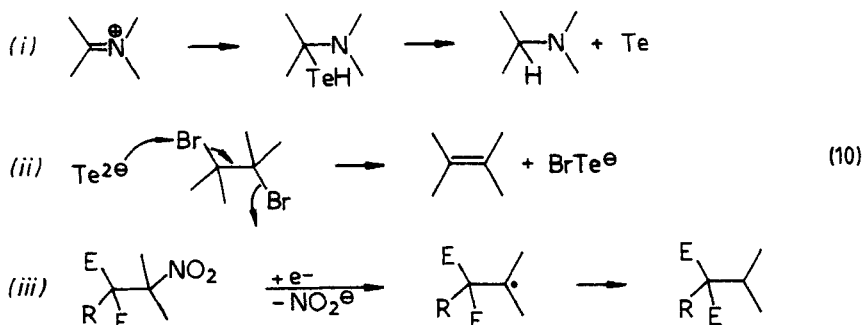
Vicinal dibromoalkanes have been known for some time to be debrominated by the action of NaHTe. Sodium telluride, prepared from Rongalite and elemental tellurium, has now been found to execute the similar type of transformation.²³ The reaction proceeded to completion at ambient temperature and occurred stereospecifically in an anti-fashion. The dehalogenation of vic-dibromides with diphenyl tellurium to yield diphenyltellurium dibromide and the corresponding olefin is the first reported transformation of an organic molecule by means of an organotellurium reagent. By using aqueous K₂S₂O₅ as the stoichiometric reductant in a two-phase system containing

benzene and bis(4-methoxyphenyl)telluride the reaction has now been turned into a catalytic one with respect to the tellurium reagent.²⁴ The maximum turnover number of the catalyst was estimated to be 30.

When α -halocarbonyl compounds are treated with lithium 2-thiophenotellurolate a reduction reaction occurs to afford, via protonation of an enolate anion, the hydrodehalogenated carbonyl compound. Suzuki and co-workers have now, in an extension of this process, reacted α -bromoacetic esters with anhydrous sodium telluride and condensed the resulting ester enolate anions with aldehydes.²⁵ This allowed the α,β -unsaturated esters to be isolated after acidic workup with no need for a separate dehydration step as shown in eq.9.

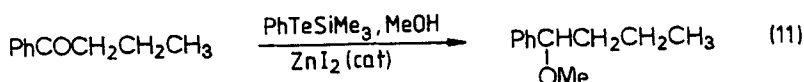


Several mechanisms would seem possible for the many tellurium(-II)-reductions which do not involve an isolable organotellurium intermediate (eq.10). The first type (i) involves a covalent carbon-tellurium bond which fragments to give a radical which could be further reduced by hydrogen atom transfer. This kind of mechanism appears likely for the reaction described in eq.6. The second type of mechanism (ii) involves nucleophilic attack by tellurium on a heteroatom. This process seems favoured when an electron recipient (like a β -bromide leaving group or an electron withdrawing group) is also present in the molecule. The third mechanism (iii) involves electron transfer from tellurium to form a radical intermediate which is subsequently reduced to the product.

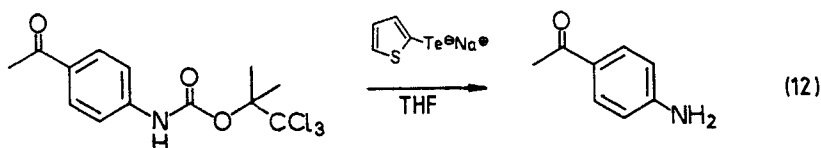


This mechanism would seem most probable for the hydrodenitration reactions of compounds 8a, 9 and 10 but could also account for most of the reduction results with the tellurium(-II)-reagents.

The reducing capacity of benzenetellurol, PhTeH , generated in situ by the methanolysis of phenyltellurotrimethylsilane, has been investigated in some detail.²⁶ In addition to the previously known transformations brought about by the reagent it was found that secondary alkyl aryl tellurides were reduced to alkanes, and acetals were reduced to the corresponding ethers in the presence of ZnI_2 . A variety of carbonyl compounds were reductively converted to unsymmetrical ethers by in situ generated benzenetellurol and ZnI_2 as exemplified in eq.11.²⁷



Cava has recently reported the efficient catalytic deprotection of 2,2,2-trichloro-tert-butyloxycarbonyl (TCBOC) protected amines by sodium 2-thiophenetellurolate under mild alkaline conditions (eq.12).²⁸ This reagent, generated in situ by reduction of bis(2-thienyl)ditelluride, has been previously used for the catalytic debromination of vicinal dibromides and for the debromination of 1,4-dibromo-2-enes to 1,3-dienes.

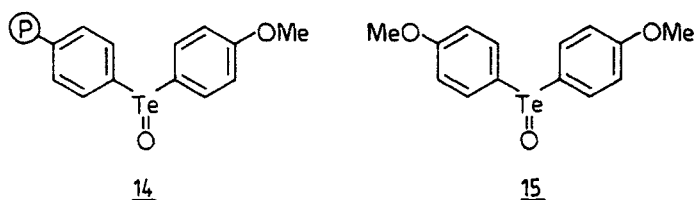


α, α' -Dihalo-o-xylenes were dehalogenated on treatment with sodium benzenetellurolate and the o-quinodimethane products trapped in moderate yields as Diels-Alder adducts.²⁹

Various tellurium(IV)-reagents have attracted attention as mild selective oxidants in organic synthesis. Barton found that aryltellurinic acid anhydrides oxidized hydroquinones to quinones in high yields and converted thiols to disulphides under conditions where simple phenols were untouched.³⁰

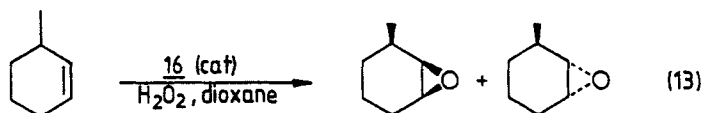
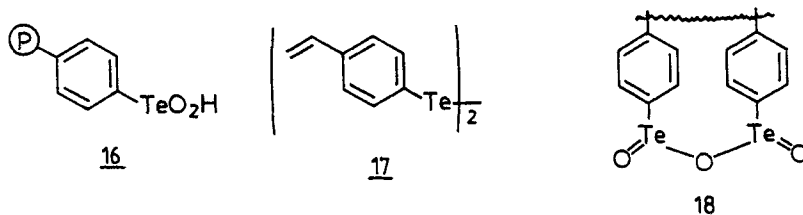
Ogura made similar observations with *p*-methoxybenzene-tellurinic acid anhydride but he also reported oxidations of phosphines, thioamides, thioureas and benzylic alcohols.³¹

Polymer-supported tellurium-based oxidants have appeared in the literature for the first time. The polymer-bound diaryl telluroxide 14³² showed much the same oxidizing properties as its monomeric counterpart, compound 15. Thus, thiols were oxidized to disulphides, phosphines to phosphineoxides, hydroquinones to quinones and thiocarbonyl compounds to the corresponding carbonyl compounds. For these reactions the polymer-bound reagent offers only practical advantages (easier separation/reoxidation) as compared with the soluble one. The reagent also effected a novel solvent-dependent conversion of thioamides to nitriles or 1,2,4-thiadiazoles at ambient temperature. The selectivity for nitrile formation was usually excellent in dichloromethane and other non-acidic solvents whereas the oxidative dimerization pathway to yield 1,2,4-thiadiazoles was competitive in only a few cases in acetic acid.



Another polymer-supported tellurium oxidant, formulated as a benzenetellurinic acid 16, has been found to catalyze the epoxidation of olefins with hydrogen peroxide (eq.13).³³ Catalysts prepared from highly cross-linked polystyrene gave the best conversions. However, the amount of tellurium which could be introduced was inversely proportional to the cross-linking. Since arenetellurinic acids are known to readily form anhydrides, it has been hypothesized that these species, linked to the polymer, should in fact be the active catalysts in the process. In support of this idea, bis(4-vinylphenyl)ditelluride (17) was copolymerized with divinylbenzene to produce, after oxidation, an active oxidation catalyst 18. On the other hand, when 4-vinylbenzenetellurinic acid was copolymerized with divinylbenzene to

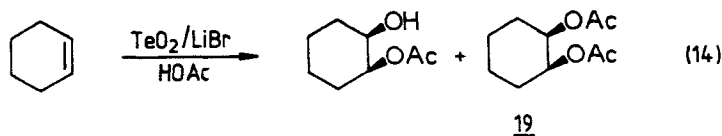
produce a polymer with randomly placed tellurinic acid groups, very little catalytic activity was observed.



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The use of TeO_2 as an oxidant in organic synthesis was explored already in the 1940's. Thus, benzil was obtained from benzoin and TeO_2 in 95% yield at 190°C in the absence of a solvent. A recent report describes the oxidation of stilbene dibromides to benziles using TeO_2 in acetic acid.³⁴

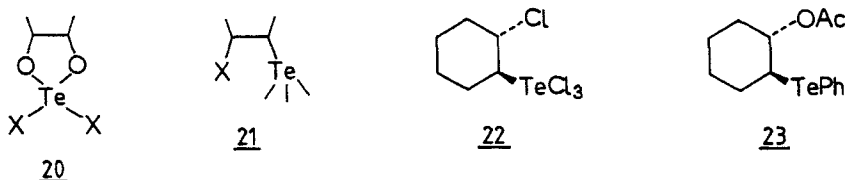
Tellurium dioxide in acetic acid containing a source of bromide ion has long been known to convert olefins into a mixture of 1,2-alkanediol monoacetates and diacetates. Early studies indicated dominant cis-diacetoxylation of Z-olefins but little or no selectivity with E-olefins and terminal olefins.³⁵ Uemura³⁶ has confirmed these results and also shown that cyclic olefins were stereospecifically cis diacetoxylation via cis-1,2-cycloalkanediol monoacetates, the primary products of the process (eq.14). As to the



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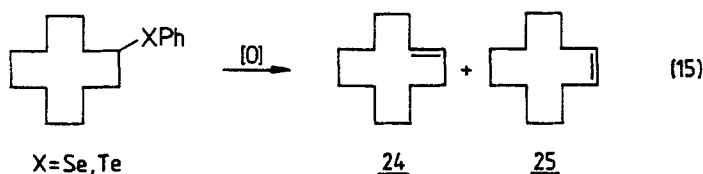
reaction mechanism of these oxidations, three different pathways have been considered. The first one,³⁷ involving a cyclic telluroester 20, was dismissed mainly for stereochemical reasons.³⁵ A second one, involving TeO_2 -oxidation of bromide ion, followed by bromination/solvolysis³⁸ appeared highly unlikely since tellurium dioxide is

incapable of oxidizing Br^- under the conditions used.³⁹ The third mechanism,³⁵ involving an acetoxy or halotelluration intermediate 21 has now received additional support.³⁶ Thus, organotelluriums 22 and 23 with well-defined stereochemistry were stereospecifically converted to diacetate 19 when treated in acetic acid containing TeO_2 and LiBr .



Benzenetellurinic acid anhydride has also been found to oxidize olefins to 1,2-alkanediol diacetates.⁴⁰ The long induction period of the reaction (5 h) was drastically reduced in the presence of a mild reducing agent. This result seems to indicate that the active oxidant is a reduced form of benzenetellurinic acid anhydride rather than the anhydride itself. The reaction pathway from olefins to 1,2-alkanediol diacetates (cis-stereochemistry) may involve acetoxytellurenylation followed by displacement of the phenyltelluro group by an acetoxy group.

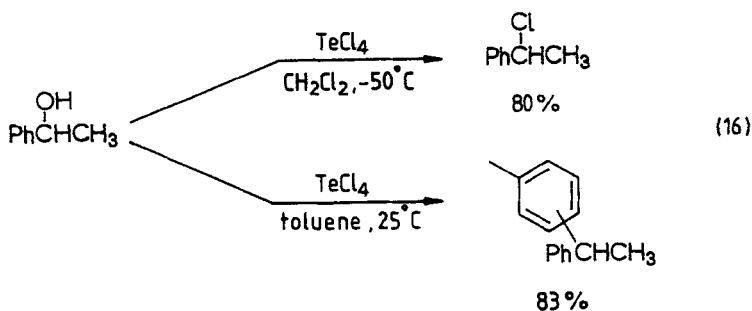
Uemura⁴¹ has found that the double bond geometry of alkenes produced by oxidative elimination of some sec.-alkyl phenyl selenides and tellurides varies with the amount of oxidant used for the tellurium compound but not for the selenium compound (eq. 15). Thus, the molar ratio 24/25 was 100/0 when one eq. of NaIO_4 was used but as low as 30/70 when a five-fold excess of the oxidant was employed. The formation of the cis-isomer was tentatively explained assuming the formation of a tellurone or a tellurinic acid ester $\text{PhTe}(\text{O})\text{OR}$ (R = cyclododecyl).



4-Methoxybenzenetellurinic acid anhydride catalyzed the

hydration of alkynes in a very selective way.³¹ Thus, terminal alkynes were smoothly converted to ketones whereas non-terminal alkynes are unaffected. The method nicely complements the conventional mercury-assisted hydrations which are applicable to all kinds of acetylenes.

Tellurium tetrachloride has been found to convert alcohols to the corresponding alkyl chlorides in various non-polar solvents (eq.16).⁴² Experiments with optically pure 1-phenylethanol showed that the hydroxyl group was replaced with racemization. This seems to indicate an S_N1 -like mechanism of the substitution reaction. $TeCl_4$ also functioned as a mild Friedel-Crafts alkylation catalyst with certain benzylic or tertiary alcohols in the presence of an aromatic solvent (eq.16). The intermediacy of alkyl chlorides and the catalytic action of $TeCl_4$ in the alkylation process were shown.



Tosyl azide is usually inert towards benzaldehyde and other carbonyl compounds. However, in the presence of diisobutyl telluride nitrogen was slowly evolved and benzaldehyde N-tosylimine formed.⁴³ A postulated tellurilimine intermediate was independently synthesized and reacted with benzaldehyde to give the corresponding N-tosylimine.

In addition to its reducing properties, $NaHTe$ has also been reported to promote deacylation of aryl acetates and benzoates⁴⁴ and to deallylate allyl carboxylates and allyl aryl ethers.⁴⁵

ACKNOWLEDGEMENTS

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