New Method of Access to Selenoacetal Chemistry: Use of Tris(Phenylseleno)borane

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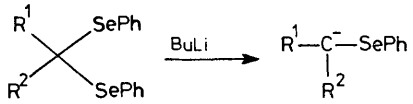
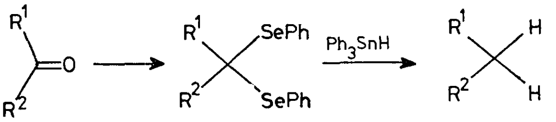
Summary The crystalline reagent, tris(phenylseleno)-borane, converts aldehydes and ketones into selenoacetals, the presence of trifluoroacetic acid being usually advantageous.

SELENOACETALS are an important compound class, in part because they provide a route to synthetically useful selenium-stabilized carbanions[1] [equation (1)] and, in part because of their involvement in an efficient method for ketone reduction[2] [equation (2)]. The selenoacetals needed for these processes can be made from carbonyl compounds by treatment with PhSeH.[3] However, this liquid reagent is extremely sensitive to air and small-scale work with it is correspondingly difficult. We have found that $(PhSe)_3B$, which is simple to prepare in high yield,[4] behaves as a crystalline (m.p. 152—153 °C), storable, and more easily handled carrier for the PhSe-group. Addition of a carbonyl compound to a solution (usually in $CHCl_3$) of $(PhSe)_3B$ (1 equiv.) affords the selenoacetal. Frequently, addition of a small amount (*ca.* 10 mole %) of trifluoroacetic acid (TFA) is advantageous. Our results are collected in the Table.



(1)



(2)

TABLE[a]

Carbonyl compound	Additive	Yield of phenyl-selenoacetal (%)
Cholestan-3-one	—	89[b]
Cholestan-3-one	TFA	88
Adamantanone	—	84[c]
Adamantanone	TFA	67
Nonan-5-one[d]	TFA	75
Nonan-5-one[d]	e	76[c]
Undecanal[d]	TFA	79
Undecanal[d]	e	78[f]
Acetophenone[d]	TFA	52

[a] In each case 1 equiv. of $(PhSe)_3B$ was used and reactions were conducted at room temperature. Reactions were carried out in dry $CHCl_3$ for 1 h except where indicated. The reaction mixture was originally 0·1—0·3 M in carbonyl compound. [b] Reaction run in dry CH_2Cl_2 for 2 h. [c] Reaction was carried out overnight. [d] In absence of an acid reaction is very slow. [e] A trace of toluene-*p*-sulphonic acid was used. [f] Reaction was carried out for 4 h.

New compounds had both correct mass measurements on $M^{+\cdot}$ or M^{+} — PhSe and satisfactory (±0·3%) combustion analytical data.

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