

Five-, six- and eight-membered tellurium-containing heterocycles with vicinal Te, O and Te, N heteroatoms

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Dedicated to Academician Mikhail G. Voronkov in recognition of his outstanding contribution to diverse areas of organic and organoelement chemistry and on the occasion of his 80th birthday

(received 20 Mar 01; accepted 08 Apr 02; published on the web 16 Apr 02)

Abstract

Previously undescribed tellurium-containing heterocycles (5H-oxatelluroles-1,2 and their benzoderivatives, 1,2,3-telluradiazines and 1,2,6-oxatellurazocines) were prepared by dehydrohalogenation of the aromatic or alkene compounds containing vicinal Te(R)Hal₂ or TeHal and hydroxy or NH groups. The same procedure was used for the synthesis of isotellurazoles. By oxidation of β-aryltelluroallylic alcohols and o-aryltellurobenzoic acids 5H-1,2-oxatelluroles and 3H-2,1-benzoxatelluro-3-ones have been prepared in good yields.

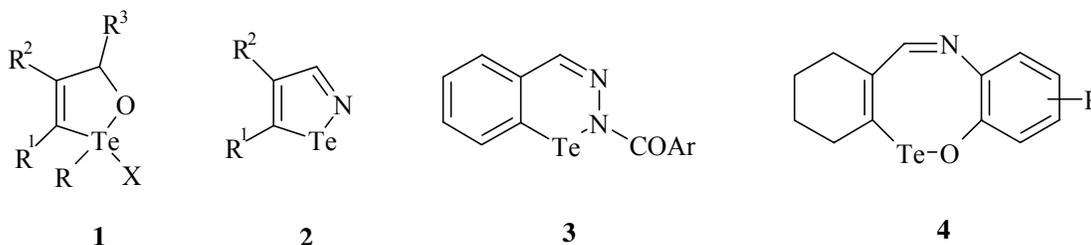
Keywords: 1-Aryl-1-chloro-2,1-benzoxatelluro-3-ones, N-arylisotellurazolium perchlorates, 2-aryloxy-1,2,3-telluradiazines, 7,8-benzo-1,2,6-oxatellurazocines, 3H-2,1-benzoxatelluroles, dehydrohalogenation, isotellurazoles, 5H-oxatelluroles-1,2, oxidation, spiro[3H-2,1-benzoxatellurole], tellurium-containing heterocycles

Introduction

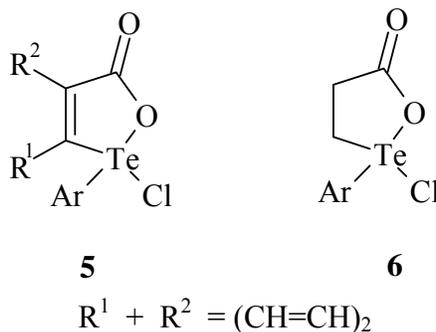
Derivatives of 1,2-telluroles represent the most thoroughly studied group of five-membered tellurium-containing heterocycles with two vicinal heteroatoms.¹ The interest in these compounds has been mostly because of their use as the donor components of conducting charge-transfer complexes and radical-ion salts. The heterocyclic compounds with Te, O centers in the positions 1,2 of a five-membered ring have been first described in our papers. Of Te, N-containing heterocycles of this type only benzisotellurazole² was known at the beginning of our work and some of the 3-substituted and 3,5-disubstituted isotellurazoles³ had been prepared in

very low yields. This paper gives a brief survey of our study in the area and contains also some recently obtained results.

The principal method employed for the synthesis of 1,2-Te,O- and 1,2-Te,N-heterocycles consists in dehydrohalogenation of the arene or vinylic compounds containing vicinal Te(R)Hal₂ or TeHal and OH or NHR groups. This approach has been used for the synthesis of 5H-1,2-oxatelluroles and their benzo analogues **1**, isotellurazoles **2**, 1,2,3-telluradiazines **3** and 1,2,6-oxatellurazocines **4**.



The synthesis of 5H-1,2-oxatelluroles **1** and 1,2-oxatellurole-5-ones **5** and their dihydro derivatives **6** has been performed by oxidation of 3-(aryltelluro)propen-2-ols-3 and 2-aryltellurobenzoic and β-aryltelluropropionic acids with *tert*-butyl hypochlorite.

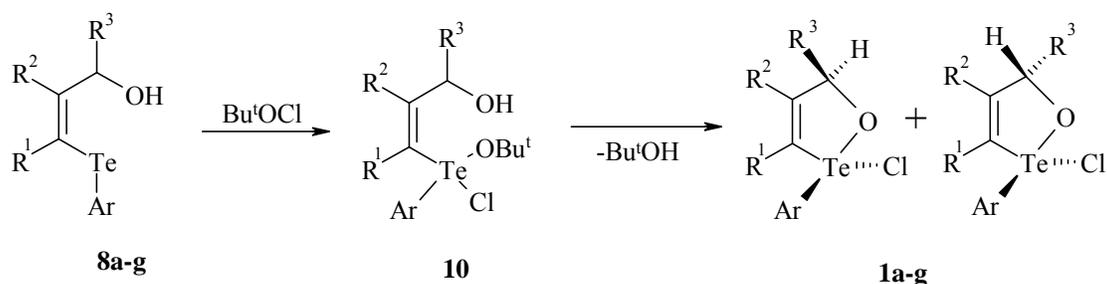


β-Organyltellurovinylaldehydes and ketones as well as their imines serve as useful synthons for the preparation of heterocycles **1-4**. The synthesis, reactions, spectral and structural characterization of these compounds are described elsewhere⁴⁻⁸.

1. 5H-1,2-Oxatelluroles and their benzo analogues

3-(Aryltelluro)propen-2-ols **8** and 2-butyltellurobenzylic alcohols **9** serves as precursors of 5H-oxatelluroles-1,2, **1** and their benzo analogues **7** (**1**, R¹ + R² = (CH=CH)₂). The synthesis of compounds **8**, which are obtained exclusively in *Z*-isomeric forms, and **9** has been previously described in much detail.⁹⁻¹³ Two different methods have been applied for the cyclization of the alcohols **8,9** into the heterocycles **1,7**. The first one applied mostly for the synthesis of 2-aryl-2-chloro-5H-oxatelluroles-1,2 **1** consists in the oxidation of **8** with *tert*-butylhypochlorite.⁹⁻¹⁰ The reaction affords the heterocycles **1** in 44-82% yield and involves σ-telluranes **10** as the

intermediates. The compounds **1a-g** thus obtained and also 2-aryl-2-iodo-5H-oxatelluroles **1h,i** prepared by the halogen exchange reaction from **1d,f** are characterized in Table 1.



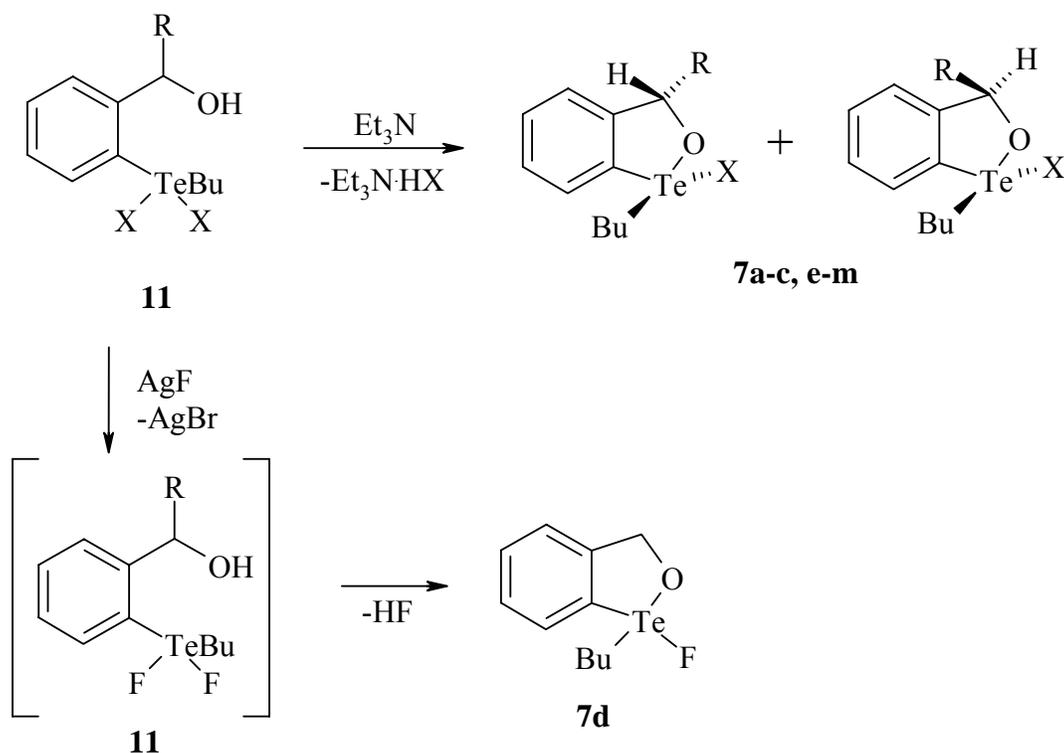
a-d Ar = Ph: $R^1 = \text{Ph}$, $R^2 = R^3 = \text{H}$ (**a**); $R^1 + R^2 = (\text{CH}_2)_4$, $R^3 = \text{H}$ (**b**); $R^1 = R^2 = \text{H}$, $R^3 = \text{Ph}$ (**c**); $R^1 = R^2 = R^3 = \text{H}$ (**d**);

e,f Ar = 4-EtOC₆H₄: $R^1 = \text{Ph}$, $R^2 = R^3 = \text{H}$ (**e**); $R^1 = R^2 = R^3 = \text{H}$ (**f**); Ar = 4-MeOC₆H₄: $R^1 = R^2 = R^3 = \text{H}$ (**g**)

Table 1. Characteristics of 1,2-Oxatelluroles **1a-i**

Compound	Molecular formula	Mp (°C)	¹ NMR, δ, ppm	Yield, %
1a	C ₁₅ H ₁₃ ClOTe	115		79
1b	C ₁₃ H ₁₅ ClOTe	118.5	2.13 (8H, m, 4CH ₂), 4.90 (2H, dd, <i>J</i> = 17.0 Hz, 3-H), 7.63 (5H, m, C ₆ H ₅)	82
1c	C ₁₅ H ₁₃ ClOTe	126	6.02 and 6.26 (1H, s, s, 3α or β-H), 7.45 (12H, m, 2C ₆ H ₅ + CH=CH)	78
1d	C ₉ H ₉ ClOTe	128	5.27 (2H, dd, <i>J</i> = 17.8 Hz, CH ₂), 7.54 (7H, m, C ₆ H ₅ + CH=CH)	44
1e	C ₁₇ H ₁₇ ClO ₂ Te	130.5	1.40 (3H, t, CH ₃), 4.05 (2H, q, OCH ₂), 5.18 (2H, dd, <i>J</i> = 18.0 Hz, 3-H), 7.44 (10H, m, C ₆ H ₅ + C ₆ H ₄ + 4-H)	75
1f	C ₁₁ H ₁₃ ClO ₂ Te	115	1.40 (3H, t, CH ₃), 4.04 (2H, q, OCH ₂), 5.18 (2H, dd, <i>J</i> = 18.1 Hz, 3-H), 6.96 (2H, d, 3'-, 5'-H), 7.32 (2H, m, CH=CH), 8.12 (2H, d, 2'-, 6'-H)	45
1g	C ₁₀ H ₁₁ ClO ₂ Te	118	3.81 (3H, s, OCH ₃), 5.17 (2H, dd, <i>J</i> = 18.1 Hz, 3-H), 6.97 (2H, d, 3'-, 5'-H), 7.30 (2H, m, CH=CH), 7.82 (2H, d, 2'-, 6'-H)	47
1h	C ₉ H ₉ IOTe	120	5.15 (2H, dd, <i>J</i> = 18.0 Hz, CH ₂), 7.59 (7H, m, C ₆ H ₅ + CH=CH)	82
1i	C ₁₁ H ₁₃ IO ₂ Te	104	1.40 (3H, t, CH ₃), 4.03 (2H, q, OCH ₂), 5.25 (2H, dd, <i>J</i> = 18.5, 3-H), 7.37 (6H, m, C ₆ H ₄ + CH=CH)	84

Dehydrohalogenation of primary and secondary 2-butyldihalogenotellurobenzylic alcohols **11** leads to 3*H*-2,1-benzoxatelluroles **7**^{9,12,13}. The advantage of this approach is a possibility of preparing benzoxatelluroles with various halogen atoms at the tellurium center. Conditions for the dehydrohalogenation of the alcohols **11** obtained in high yields by the oxidation-addition reaction of the tellurides **9** with dihalogens (Cl₂, Br₂, I₂),^{9,12,13} crucially depends on the origin of the halogen atoms attached to the tellurium center. Thus, 2-butyldifluorotellurobenzylic alcohol (prepared by the halogen exchange reaction between 2-butyldibromobenzylic alcohol and AgF) spontaneously forms 1-butyl-1-fluoro-2,1-benzoxatellurole **7d** (87%)^{9,12}. The cyclization of 2-butyldihalogenotellurobenzylic alcohols **11** occurs on treatment with triethylamine^{9,12,13}. Oxa-**1** and benzoxatelluroles **7** with a substituent in the 3 position are obtained as mixtures of two diastereomers as determined by ¹H NMR spectra^{9,12}. Table 2 contains some data on the compounds **7**.



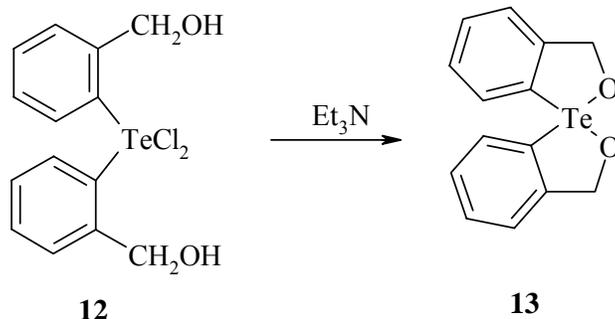
a-d R = H: X = Cl (**a**), Br (**b**), I (**c**), F (**d**); **e-g** R = Ph: X = Cl (**e**), Br (**f**), I (**g**); **h-j** R = 4-MeC₆H₄: X = Cl (**h**), Br (**i**), I (**j**); **k-m** R = Me: X = Cl (**k**), Br (**l**), I (**m**).

Dehydrohalogenation of the alcohol **12** prepared from the corresponding telluride by the oxidation-addition reaction with chlorine¹⁴ gives rise to 1,1'-spiro[3*H*-2,1-benzoxatellurole] **13**. The treatment of a benzene solution **12** with two equivalents of triethylamine affords **13** in almost quantitative yield. 3,3,3,3-Tetramethyl- and 3,3,3,3-tetra(trifluoromethyl)-1,1'-spiro[3*H*-

2,1-benzoxatelluroles] were also synthesized in 20-60% yields by coupling arylmagnesium chlorides containing *o*-CMe₂OMgBr and C(CF₃)₂OMgBr groups with tellurium tetrachloride¹⁵.

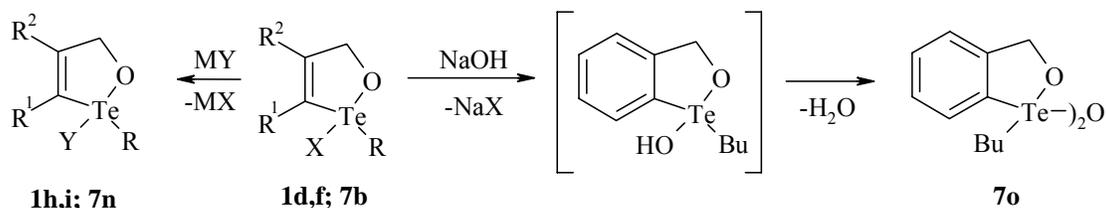
Table 2. Characteristics of 3*H*-2,1-Benzoxatelluroles **7a-o**

Compound	Molecular formula	Mp (°C)	¹ NMR, δ, ppm	Yield, %
7a	C ₁₁ H ₁₅ ClOTe	114-115	1.79 (9H, m, C ₄ H ₉), 5.32 (2H, dd, <i>J</i> = 15.3 Hz, 3-H), 7.68 (4H, m, C ₆ H ₄)	89
7b	C ₁₁ H ₁₅ BrOTe	113-114	1.96 (9H, m, C ₄ H ₉), 5.41 (2H, dd, <i>J</i> = 15.1 Hz, 3-H), 7.80 (4H, m, C ₆ H ₄)	78
7c	C ₁₁ H ₁₅ IOTe	110-112	2.19 (9H, m, C ₄ H ₉), 5.37 (2H, dd, <i>J</i> = 15.1 Hz, 3-H), 7.72 (4H, m, C ₆ H ₄)	75
7d	C ₁₁ H ₁₅ FOTe	Oil	1.83 (9H, m, C ₄ H ₉), 5.45 (2H, dd, <i>J</i> = 15.2 Hz, 3-H), 7.80 (4H, m, C ₆ H ₄)	87
7e	C ₁₇ H ₁₉ ClOTe	127-128	2.27 (9H, m, C ₄ H ₉), 6.40 and 6.71 (1H, s, s, 3α or β-H), 7.91 (9H, m, C ₆ H ₅ + C ₆ H ₄)	70
7f	C ₁₇ H ₁₉ BrOTe	138-139	2.11 (9H, m, C ₄ H ₉), 6.28 and 6.57 (1H, s, s, 3α or β-H), 7.75 (9H, m, C ₆ H ₅ + C ₆ H ₄)	82
7g	C ₁₇ H ₁₉ IOTe	140-142	1.99 (9H, m, C ₄ H ₉), 6.17 and 6.42 (1H, s, s, 3α or β-H), 7.00 (9H, m, C ₆ H ₅ + C ₆ H ₄)	76
7h	C ₁₈ H ₂₁ ClOTe	140-141	2.22 (9H, m, C ₄ H ₉), 2.42 (3H, s, 4-CH ₃), 6.38 and 6.78 (1H, s, s, 3α or β-H), 7.87 (8H, m, 2C ₆ H ₄)	79
7i	C ₁₈ H ₂₁ BrOTe	138-140	2.05 (9H, m, C ₄ H ₉), 2.33 (3H, s, 4-CH ₃), 6.29 and 6.70 (1H, s, s, 3α or β-H), 7.78 (8H, m, 2C ₆ H ₄)	84
7j	C ₁₈ H ₂₁ IOTe	100-102	1.98 (9H, m, C ₄ H ₉), 2.26 (3H, s, 4-CH ₃), 6.10 and 6.65 (1H, s, s, 3α or β-H), 7.60 (8H, m, 2C ₆ H ₄)	62
7k	C ₁₂ H ₁₇ ClOTe	124-125	2.12 (9H, m, C ₄ H ₉), 1.52 (3H, d, CH ₃), 5.64 and 5.92 (1H, s, s, 3α or β-H), 7.97 (4H, m, C ₆ H ₄)	81
7l	C ₁₂ H ₁₇ BrOTe	116-118	2.01 (9H, m, C ₄ H ₉), 1.47 (3H, d, CH ₃), 5.48 and 5.75 (1H, s, s, 3α or β-H), 7.84 (4H, m, C ₆ H ₄)	85
7m	C ₁₂ H ₁₇ IOTe	120-121	-	62
7n	C ₁₃ H ₈ O ₃ Te	68-71	1.78 (9H, m, C ₄ H ₉), 2.01 (3H, s, CH ₃), 5.40 (2H, dd, <i>J</i> = 15.1 Hz, 3-H), 7.53 (4H, m, C ₆ H ₄)	50
7o	C ₂₂ H ₃₀ O ₃ Te ₂	89-91	1.92 (9H, m, C ₄ H ₉), 5.40 (2H, dd, <i>J</i> = 15.2 Hz, 3-H), 7.65 (4H, m, C ₆ H ₄)	32



In contrast with similar sulfurane¹⁶, no diastereotopic splitting has been observed for the methylene protons of **13** at ambient temperature¹⁴. This fact indicates that the energy barrier to the polytopal rearrangement of the tellurane is lower than that of its sulfur analogue and is in accord with the predictions based on the quantum mechanical calculations¹⁷.

Oxatelluroles **1** and benzoxatelluroles **7** are susceptible to readily occurring exchange of the halogen atoms at the tellurium centers for other anionic groups⁹. When treated with potassium iodide in an acetone solution, the compounds **1d,f** are converted to the corresponding iodo derivatives (the yields are 80-85%). 1-Acetoxy-1-butyl-2,1-benzoxatellurole **7n** was obtained by the reaction of its bromo analogue with silver acetate. Contrary to expectations, the action of an ethanol solution of NaOH on **7b** leads not to 1-butyl-1-hydroxybenzotellurole-2,1, but to a product of its dehydration **7o**⁹.



1h,i $\text{R}^1 = \text{R}^2 = \text{H}$, $\text{X} = \text{I}$; $\text{R} = \text{Ph}$ (**h**), $4\text{-EtOC}_6\text{H}_4$ (**i**)

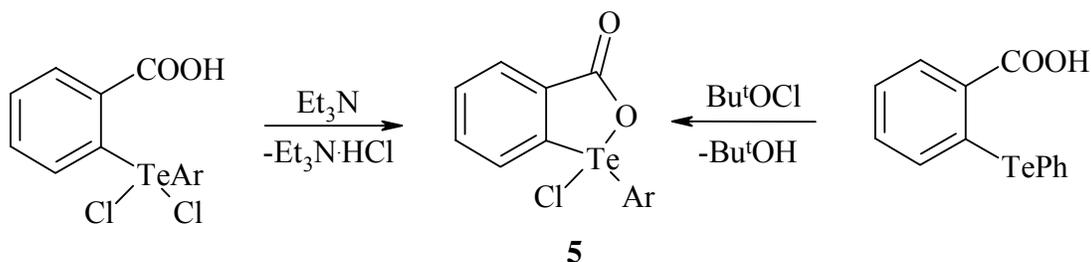
7n $\text{R}^1 + \text{R}^2 = (\text{CH}=\text{CH})_2$, $\text{X} = \text{OCOMe}$

The halogen exchange in the compounds **7** may also be realized through their reduction with zinc powder followed by a treatment of the reaction mixture with an equivalent amount of halogen⁹. Thus, the compound **7c** was prepared in almost quantitative yield from the bromo derivative **7b**.

o-Carbonyl derivatives of Te,Te-dibromophenylbutyl telluride **14a-c** are formed in 73-95% yields when chloroform or carbon tetrachloride solutions of 1-bromo-1-butyl-2,1-benzoxatelluroles **7b,f,i** and an equimolar amounts of bromine are heated at reflux^{9,12,13}. A possible mechanism of this Te-O bond cleavage reaction involves the derivatives of hexacoordinated tellurium **15** rearranging to the hypobromites **16**.

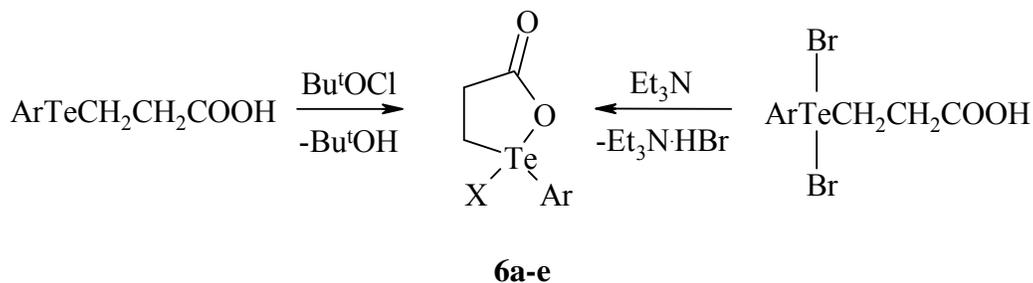
2. 2,1-Oxatellurol-3-ones

The methods developed for the synthesis of oxatellurolones and benzoxatellurolones have been found to be applicable for the synthesis of their oxo-derivatives **5**⁹, **6**¹⁹. Thus, 1-aryl-1-chloro-2,1-benzoxatellurolone-3 **5** were prepared in 75-90% yields by oxidation of 2-carboxydiphenyl telluride with *tert*-butylhypochlorite and the dehydrohalogenation of 2-(dichlorotelluroaryl)benzoic acids²⁰ under the action of triethylamine⁹.



Ar = Ph (**a**), 4-MeOC₆H₄ (**b**), 4-EtOC₆H₄ (**c**)

The dihydro derivatives of the oxatellurolones were obtained in high yields (more than 90%) by oxidation of β -aryltelluropropionic acids²¹ with *tert*-butylhypochlorite in chloroform solution. The bromo analogues of **6** are accessible through dehydrobromination of aryl(β -carboxyethyl)tellurium dibromides in toluene solution¹⁹.



X = Cl: Ar = Ph (**a**), 4-MeC₆H₄ (**b**), 4-EtOC₆H₄ (**c**);

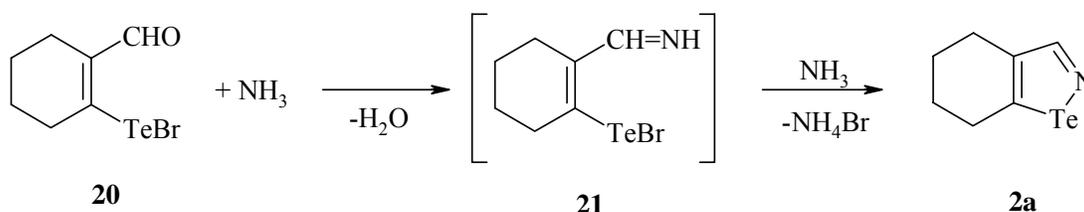
X = Br: Ar = 4-MeC₆H₄ (**d**), 4-EtOC₆H₄ (**e**)

Benzoxatellurol-3-ones **5** are isoelectronic to 2-phenyliodonium carboxylates. One may expect that by this analogy thermolysis of **5** would lead to the formation of dehydrobenzene and aryltellurenyl chlorides, which in their turn would disproportionate into diaryltellurium dichlorides and Te. However, no dehydrobenzene was generated in this reaction were performed under various conditions.

3. Isotellurazoles and *N*-arylisotellurazolium cations

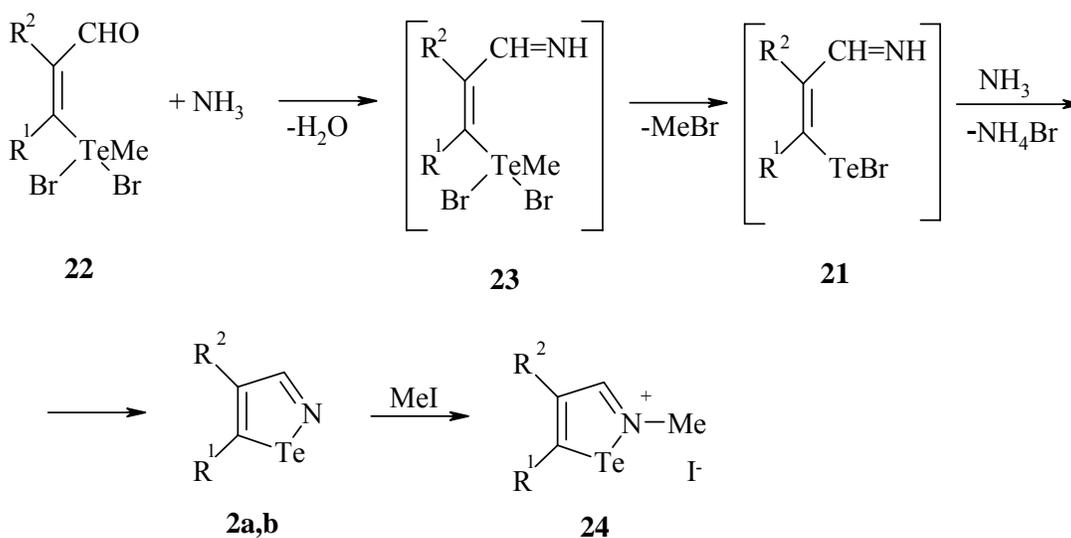
Isotellurazoles **2** were first prepared by the reaction between α -acetylenic ketones, potassium telluride and hydroxylamine-O-sulfonic acid occurring in an aqueous solution of potassium acetate³. The compounds **2** are formed in very low yields (less than 10%). The method cannot be applied for the synthesis of 3*H*-isotellurazoles because when α -acetylenic aldehydes are used in this reactions instead of the ketones, only di(β -cyanovinyl)tellurides are formed as the final products³.

We have found that β -bromotellurenylvinylaldehydes^{7,8} **20** may serve as the precursors of isotellurazoles when reacted with ammonia in a benzene solution. In this way, 4,5-tetramethyleneisotellurazole **2a** was obtained in 70% yield²². The reaction proceeds through dehydrobromination of the intermediate imine **21**.

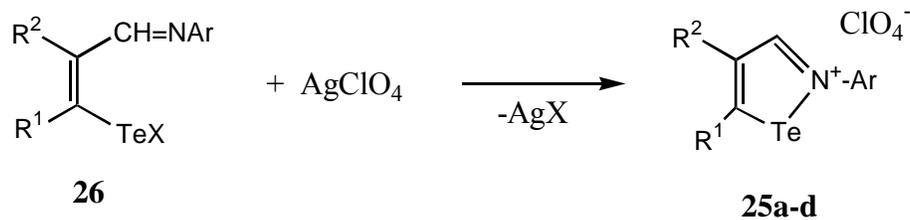


Preparation of the compounds **20** is difficult,^{7,8} however the readily accessible β -methyl dibromotellurovinylaldehydes **22** have been found to be useful precursors of isotellurazoles.

By bubbling ammonia through benzene solutions of the tellurium dibromides **22** isotellurazoles **2a,b** were obtained in about 70% yields. In accordance with these findings²³, the following mechanism has been suggested for this reaction.



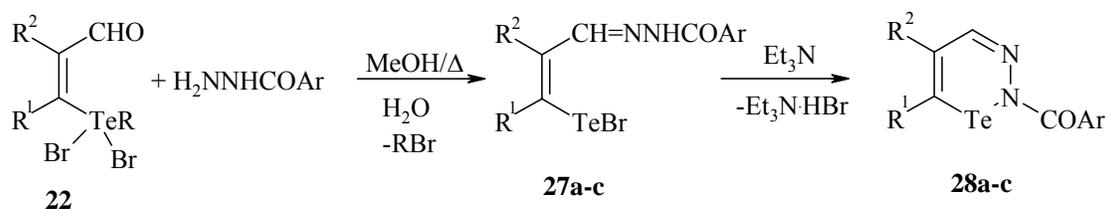
An analogous route has been previously used for the synthesis of isoselenazoles²⁴. However, due to low thermal stability of the tetracoordinated selenium analogues of **22** the reaction must be carried out at very low temperature (-70°C). Likewise benzoisotellurazole, isotellurazoles **2** react with methyl iodide under mild conditions to give the quaternary salts, e.g. **24**, in good yields²². N-Arylisotellurazolium perchlorates **25**²² and their benzoderivatives **25** ($\text{R}^1 + \text{R}^2 = (\text{CH}=\text{CH})_2$)^{23,25} were prepared in high yields by treating solutions of β -bromotellurenylvinyldimines **26** ($\text{X} = \text{Br}$)²² and 2-chlorotellurenylbenzaldimines **26** ($\text{26}, \text{R}^1 + \text{R}^2 = (\text{CH}=\text{CH})_2, \text{X} = \text{Cl}$)²⁵ with silver perchlorate in acetone or dimethylformamide.



$\text{R}^1 = 4\text{-BrC}_6\text{H}_4, \text{R}^2 = \text{H}; \text{Ar} = 4\text{-MeC}_6\text{H}_4$ (**a**), $4\text{-MeOC}_6\text{H}_4$ (**b**); $\text{R}^1 + \text{R}^2 = (\text{CH}_2)_4; \text{Ar} = 4\text{-MeC}_6\text{H}_4$ (**c**), $4\text{-MeOC}_6\text{H}_4$ (**d**); $\text{R}^1 + \text{R}^2 = (\text{CH}=\text{CH})_2; \text{Ar} = \text{Ph}$ (**e**), $4\text{-MeC}_6\text{H}_4$ (**f**), $4\text{-BrC}_6\text{H}_4$ (**g**)

4. 2-Aroyl-1,2,3-telluradiazines

Dehydrobromination of the arylhydrazons of β -bromotellurenylvinylaldehydes **27** gives rise to previously unknown 1,2,3-telluradiazines **28**²⁶. As shown by scheme below, the formation of **28** in the reaction of the aldehydes **22** with the hydrazides proceeds with elimination of a molecule of an alkyl bromide. The heterocycles **28** are obtained in rather high yields (about 80%).

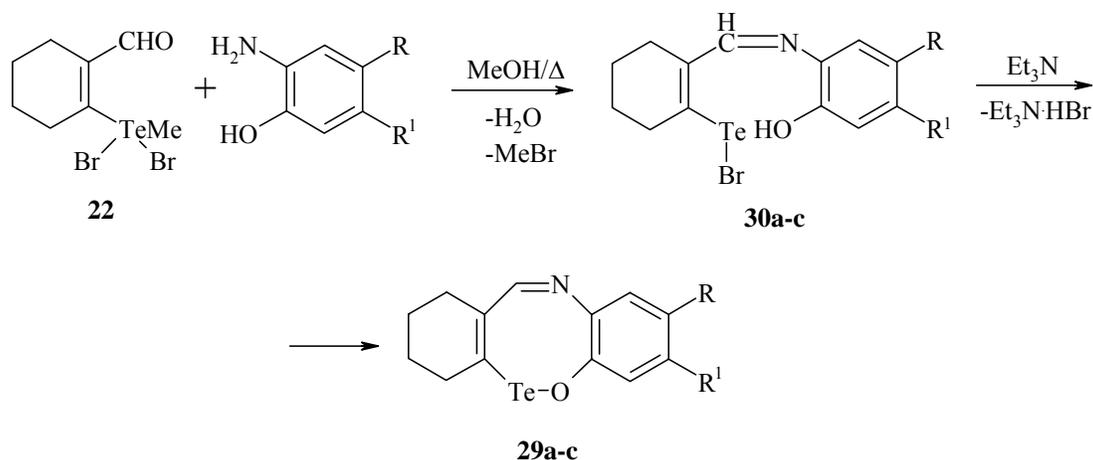


22 $\text{R} = \text{Me}, \text{R}^1 + \text{R}^2 = (\text{CH}_2)_4; \text{R} = \text{Bu}, \text{R}^1 + \text{R}^2 = (\text{CH}=\text{CH})_2$

27,28 $\text{R}^1 + \text{R}^2 = (\text{CH}_2)_4; \text{Ar} = 4\text{-MeOC}_6\text{H}_4$ (**a**), $4\text{-BrC}_6\text{H}_4$ (**b**); $\text{R}^1 + \text{R}^2 = (\text{CH}=\text{CH})_2; \text{Ar} = 4\text{-MeOC}_6\text{H}_4$ (**c**)

5. 1,2,6-Benzoxatellurazocines

Preparation of 1,2,6-benzoxatellurazocines **29**²⁷ is another example of the use of the reaction of dehydrobromination of tellurenylbromides in the synthesis of tellurium-containing heterocycles. 2-Bromotellurenylcyclohexenylidene-2'-aminophenols **30**, were used as the starting material. A treatment of suspensions of **30** in benzene with equimolar amounts of triethylamine followed by short reflux of the reaction mixtures resulted in the compounds **29** obtained in 80-90% yields.



29, 30 R = R¹ = H (**a**); R = Me, R¹ = H (**b**); R = H, R¹ = NO₂ (**c**)

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