TELLURIUM
LITERATURE SURVEY COVERING THE YEAR 1979*

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*Tellurium; Literature Survey covering the year 1978 see J. Organometal. Chem., Vol. 189(1980) p. 65-128.

I. Introduction

This annual survey of the organic chemistry of tellurium covers the literature abstracted in Chemical Abstracts Volume 90, No. 11 through Volume 92, No. 14 and papers which had appeared earlier, but had not been included in previous surveys. The symbol "R", for which the term "organyl" has been adopted, is used for alkyl as well as aryl groups. When reference is made to earlier work, the original papers are generally not cited; instead, the reader is referred to previous surveys [1-6] which will provide access to the pertinent literature. The tellurium compounds, which have been claimed to be useful as components in photosensitive materials, are listed in section XIV.

One page of references was inadvertently omitted from the review covering the year 1976 (J. Organometal. Chem., 158 (1978) 235-266) [4]. The missing references (number 54 through 74 and P-1 through P-3) are listed separately in the reference section of this review.

The assistance of the investigators, who provided reprints and preprints of their papers, and the partial financial support of this endeavor by the Robert A. Welch Foundation of Houston, Texas is gratefully acknowledged.

II. Reviews

The following reviews, which were published during the survey period, are of interest to organic tellurium chemists.

Organic Selenium and Tellurium Compounds (Magnus, P. D., 1979) [7]: This exposition covers the organic chemistry of selenium and tellurium on 33 and 3 pages, respectively.

Aliphatic Organic Sulphur, Selenium and Tellurium Compounds: Compounds with Exocyclic Sulphur Functional Groups and Their Selenium and Tellurium Analogues (Barret, G. C., 1979) [8]: This review covers the literature from April 1976 through March 1978.

Ylides and Carbionic Compounds of Sulphur, Selenium and Tellurium (Block, E., 1979) [9]: The major part of this review is devoted to sulfur compounds with a few pages taken up by selenium derivatives. The only tellurium compound mentioned is phenyltelluromethyl lithium. The literature from April 1976 through March 1978 is covered.

Ylides of Sulphur, Selenium and Tellurium: Compounds with S=N Functional Groups (Haake, M., 1979) [10]: This literature review covering the period from April 1976 through March 1978 discusses mostly sulfur compounds, a few selenium compounds and no tellurium derivatives.

Systems with Heteroatoms Other Than Nitrogen, Oxygen or Sulphur (Livingstone, R., 1979) [11]: The five- and six-membered heterocyclic compounds with one tellurium atom are reviewed on six pages. The remaining seventy pages are devoted to heterocycles containing selenium, phosphorus, arsenic, antimony, bismuth, silicon, germanium, tin, lead, aluminum, boron, bromine or iodine.

A ^{125}Te-Moessbauer Study of Organotellurium Compounds (Jones, C.H. W., McWhinnie, W. R., and Berry, F. J., 1976) [12]: This review summarizes the results of Te-125 Moessbauer studies on organic tellurium compounds.

Synthesis and Structures of Telluranes (Sadekov, I. D., Bushkov, A. Ya., and Minkin, V. I., 1979) [13]: This review covers the syntheses and structures of tri- and tetra-coordinated organic tellurium compounds and provides tables containing their melting points, yields and methods of preparation.

Thiophens and Their Selenium and Tellurium Analogues (Gronowitz, S., 1979) [14]: This review contains a summary of the publications dealing with tellurophenes, which appeared during 1976.

NMR and the Periodic Table: Group VI - Oxygen, Sulphur, Selenium and Tellurium (Rodger, C., Sheppard, N., McFarlane, C., and McFarlane, W., 1977) [15]: The results of Te-125 nmr investigations of organic and inorganic tellurium compounds are summarized. The Te-125 chemical shifts relative to dimethyl telluride and Te-H, Te-C-13, Te-F-19, Te-P-31, Te-Se-77, Te-Sn-119, Te-Xe-129 and Te-Pt-195 coupling constants are tabulated.

Selenium and Tellurium (Wason, J. R., 1978) [16]: The inorganic chemistry of selenium and tellurium is discussed in ten pages.

Water Analysis (Fishman, M. J., and Erdmann, D. E., 1979) [17]: The literature of analytical chemistry applied to water analysis covering the period October 1976 through September 1978 is reviewed. Methods for the determination of selenium and tellurium are included.

Environmental Health Aspects of Selenium and Tellurium (Glover, J. R., 1976) [18]: The industrial uses and the occupational health and hygiene problems of selenium and tellurium, their modes of entry into the body, reported cases of occupational exposure and therapeutic measures are discussed.

Biosynthesis of Organometallic and Organometalloidal Compounds (Challenger, F., 1978) [19]: The biological methylation of tellurite is mentioned in context of the metabolic processes affecting sulfur, selenium, arsenic and other elements.

III. Methods for the Introduction of Tellurium into Organic Molecules

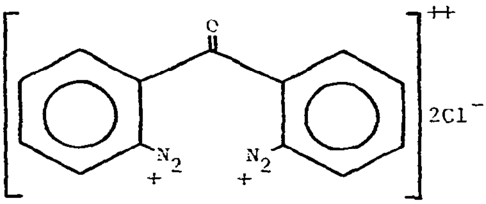
Tellurium, disodium telluride, disodium ditelluride, tellurium tetrahalides, tellurium dioxide, Sb_2Te_3, In_2Te_3 and $Te_n(AsF_6)$ (n=4,6) were employed during the survey period to prepare organic tellurium compounds.

Tellurium heated in a solution of potassium hydroxide in water/dimethyl sulfoxide with vinylacetylene at 100° gave bis(buta-1,3-dien-1-yl) telluride in three percent yield [20]. The same compound was obtained under similar conditions from acetylene and tellurium [P-13]. Trifluoromethyl radicals generated from hexafluoroethane reacted with tellurium at liquid nitrogen temperatures to yield bis(trifluoromethyl)telluride and ditelluride [21]. 1-Lithio-2-benzylbenzene [22] and 1,8-dilithionaphthalene [23] and tellurium provided aryl lithium tellurides as intermediates in the preparation of heterocyclic compounds.

Tellurium and sodium in liquid ammonia [24-27,67] and tellurium and sodium borohydride in ethanol [26a,28,29] were employed to prepare disodium ditelluride. Tellurium and Rongalite [30], sodium borohydride [26a,31], potassium borohydride [32] or $Na_2S_2O_4/(H_2N)_2CSO_2$ [33] in aqueous medium yielded disodium telluride. Disodium ditellurides labeled with Te-123m were also synthesized *via* these methods [25-28].

Tellurium was used as a catalyst for the preparation of unsaturated esters by acyloxylation of C-4 fractions [P-5] and for the conversion of amines to ureas and formamides through reactions with carbon monoxide [34]. Organic tellurium compounds are probably intermediates in these reactions.

Disodium ditelluride was alkylated to produce dialkyl ditellurides [24,25,26,26a,28,69,68] and bis(carboxyalkyl) ditellurides [27,29]. Disodium telluride and alkyl halides produced dialkyl tellurides [26a,32,33]. Dimethyl tin dichloride and Na_2Te yielded cyclic $[(CH_3)_2SnTe]_3$ [31]. The diazonium salt **1** stirred with Na_2Te in ethanol/water gave 10-tellura-9-oxo-9,10-dihydroanthracene **2** [30].



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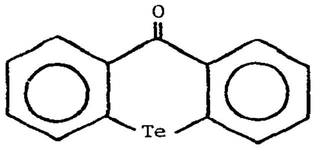


2

The reactions of tellurium tetrachloride, which were carried out during the survey period, are summarized in Fig. 1.

The addition of tellurium tetrachloride to carbon-carbon double bonds is a well-known reaction. When cyclic olefins were employed the expected 2-chlorocycloalkyl tellurium trihalides were first formed and subsequently transformed by additional tellurium tetrachloride to aromatic, tellurium-free, in some cases chlorinated aromatic compounds. These reactions proceeded in CCl_4, $CHCl_3$ or CH_3CN solutions at 76°. The following products were obtained (starting material, products): 9,10-dihydroanthracene → 9,10-dichloroanthracene; decalin or tetralin, neat at 200° → naphthalene, 1-chloronaphthalene, 1,2-dichloronaphthalene; 2,2'-bicyclohexene → biphenyl; cyclohexene → benzene; 4-methylcyclohexene →

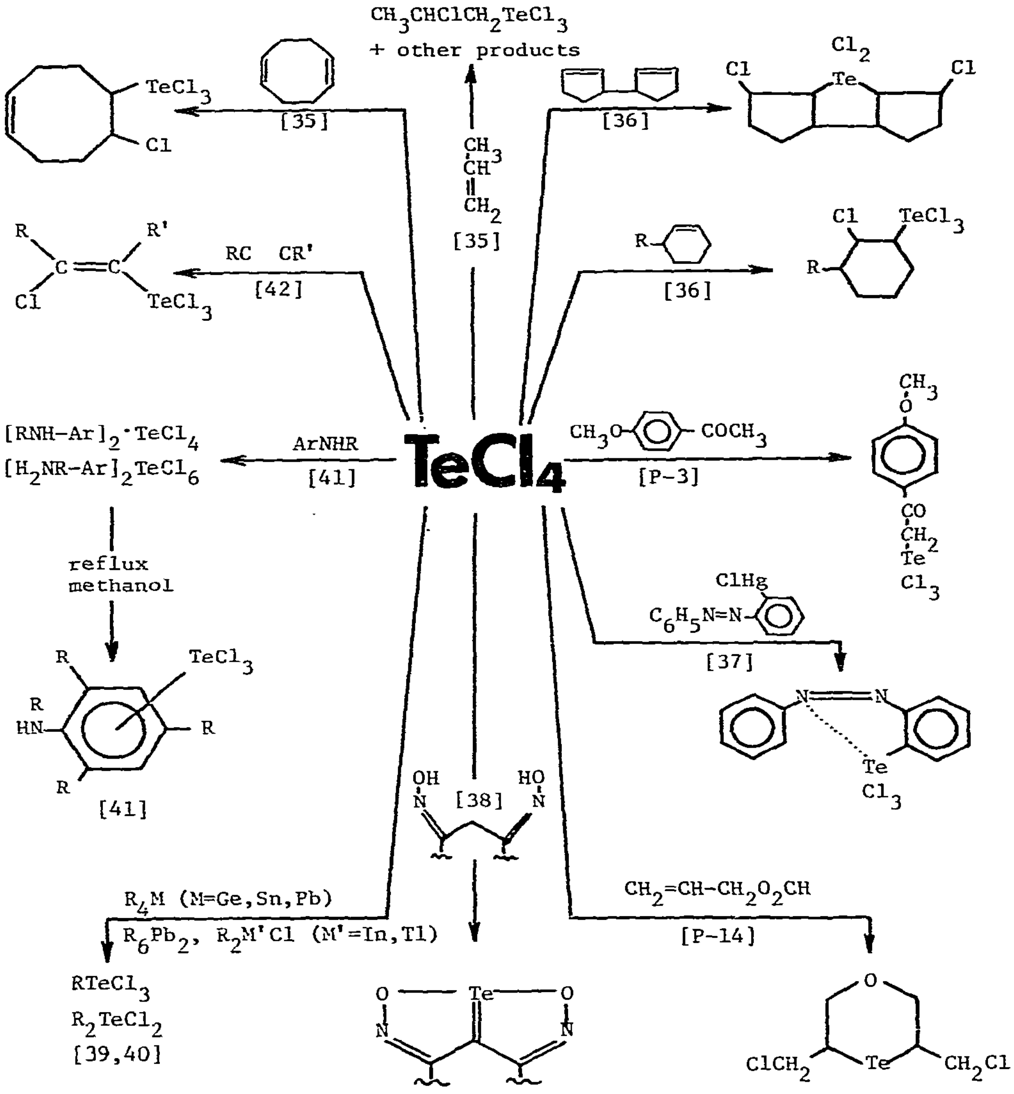


Fig. 1 : Reactions Employing Tellurium Tetrachloride as the Reagent for the Introduction of Tellurium into Organic Molecules

toluene; 1-methylcyclohexene → toluene; 3-chlorocyclohexene → benzene; cyclohexyl chloride or bromide → benzene; limonene → p-cymene. These reactions proceed by a combination of steps including dehydrohalogenation, addition of tellurium tetrachloride and loss of tellurium. Hydrogen chloride and tellurium were formed in all of these reactions. Bicyclohexene and tellurium tetrachloride gave an addition product which has not yet been identified. Only aromatic compounds and no additional products were formed from 9,10-dihydroanthracene, decalin, tetralin, 1-methylcyclohexene and limonene. Tellurium tetrabromide reacted similarly but slower than tellurium tetrachloride with cyclehexene but was unreactive toward cyclohexyl chloride [36].

Tellurium tetrachloride and triphenylchloromethane yielded $[(C_6H_5)_3C]^+[Te_3Cl_{13}]^-$ [43], a yellow-brown solid which hydrolyzed upon contact with moisture. Tellurium tetrabromide was claimed to be a catalyst for the decomposition of peroxides resulting from the autoxidation of unsaturated aldehydes [P-11].

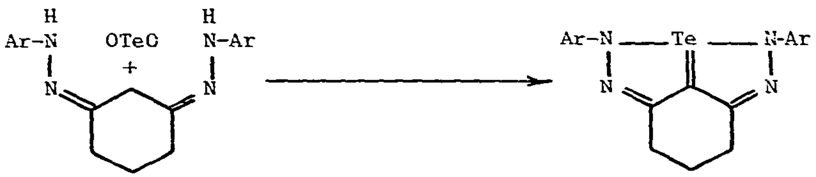
Tellurium dioxide in acetic acid in the presence of lithium bromide oxidized *cis*- and *trans*-2 butene and 1-decene to the corresponding alkanediol acetates [35]. Ethylene was converted to ethylene glycol by air with tellurium dioxide and HBr serving as catalysts [P-4]. Organic tellurium compounds are very likely intermediates in these oxidations.

Tellurium dioxide reacted with dioximes [38] and diphenylhydrazones [44] in xylene/amyl alcohol to yield dihydropentalene derivatives 3 and 4, respectively (eqns. 1,2).

(1)

3

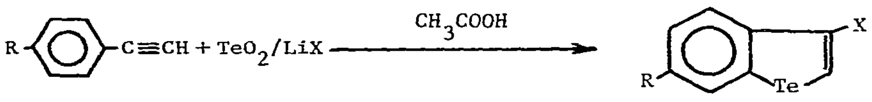
(2)



4

When phenylacetylenes and tellurium dioxide reacted in the presence of a lithium halide substituted benzotellurophenes 5 were obtained (eqn. 3) [45].

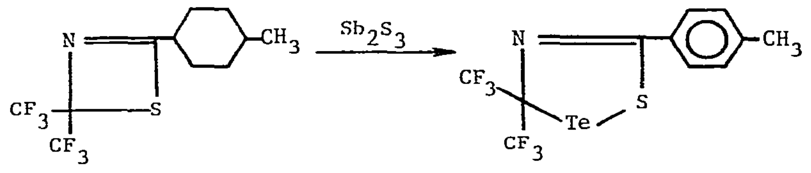
(3)



5

Antimony tritelluride and 2-(p-methylphenyl)-4,4-bis(trifluoromethyl)-1-aza-3-thiacyclobut-1-ene 6 kept in toluene at 80-90° in the dark for five weeks produced the tellurium heterocycle 7 (eqn. 4) [46]. Indium tritelluride, In_2Te_3, and tellurium reacted similarly, but gave 7 in lower yields.

(4)



6 7

Tetrafluoroethylene and $Te_n(AsF_6)_2$ (n=4,6) produced $(C_2F_5)_2Te_n$ (n=1,2), $C_4F_9TeC_2F_5$ and $C_2F_5Te(CF_2)_3COF$ [47] as reported earlier in several publications [2,3].

The reactions of organic tellurium compounds which were employed to produce other organic tellurium derivatives are summarized in Fig. 2. All of these reactions are discussed in the sections devoted to the particular tellurium compounds.

IV. Tellurocyanates

No synthetic papers on ionic tellurocyanate compounds appeared during the survey period. The structure of $[(C_6H_5)_3P{=}N{=}P(C_6H_5)_3]^+TeCN^-$ is discussed in section XI-G. Organyl tellurocyanates, RTeCN are treated in section V-B.

V. Compounds Containing a Single Carbon-Tellurium Bond

Tellurols, RTeH, aryl tellurium halides, RTeX, benzyl tellurocyanate phenyldihalotellurates(II), organyl tellurium trihalides and diorganyl ditellurides were investigated during the survey period. Tellurium derivatives, in which the second tellurium valence is satisfied by a group I to V element atom, sulfur or selenium are discussed in section IX.

A. Tellurols

The reduction of diorganyl ditellurides by sodium borohydride in tetrahydrofuran, ethanol/benzene, absolute ethanol or ethanol/tetrahydrofuran in the presence or absence of aqueous 1 M NaOH solution is widely practiced. The products of the reduction, which are generally not isolated, are claimed to be organyl sodium tellurides, RTeNa, or tellurols, RTeH.

It is known that the acidity of hydrogen chalcogenides, H_2X, increases from oxygen to tellurium. Therefore, tellurols can be expected to be quite acidic. In the presence of sodium hydroxide, they will be neutralized and in the absence of base, they probably react with excess

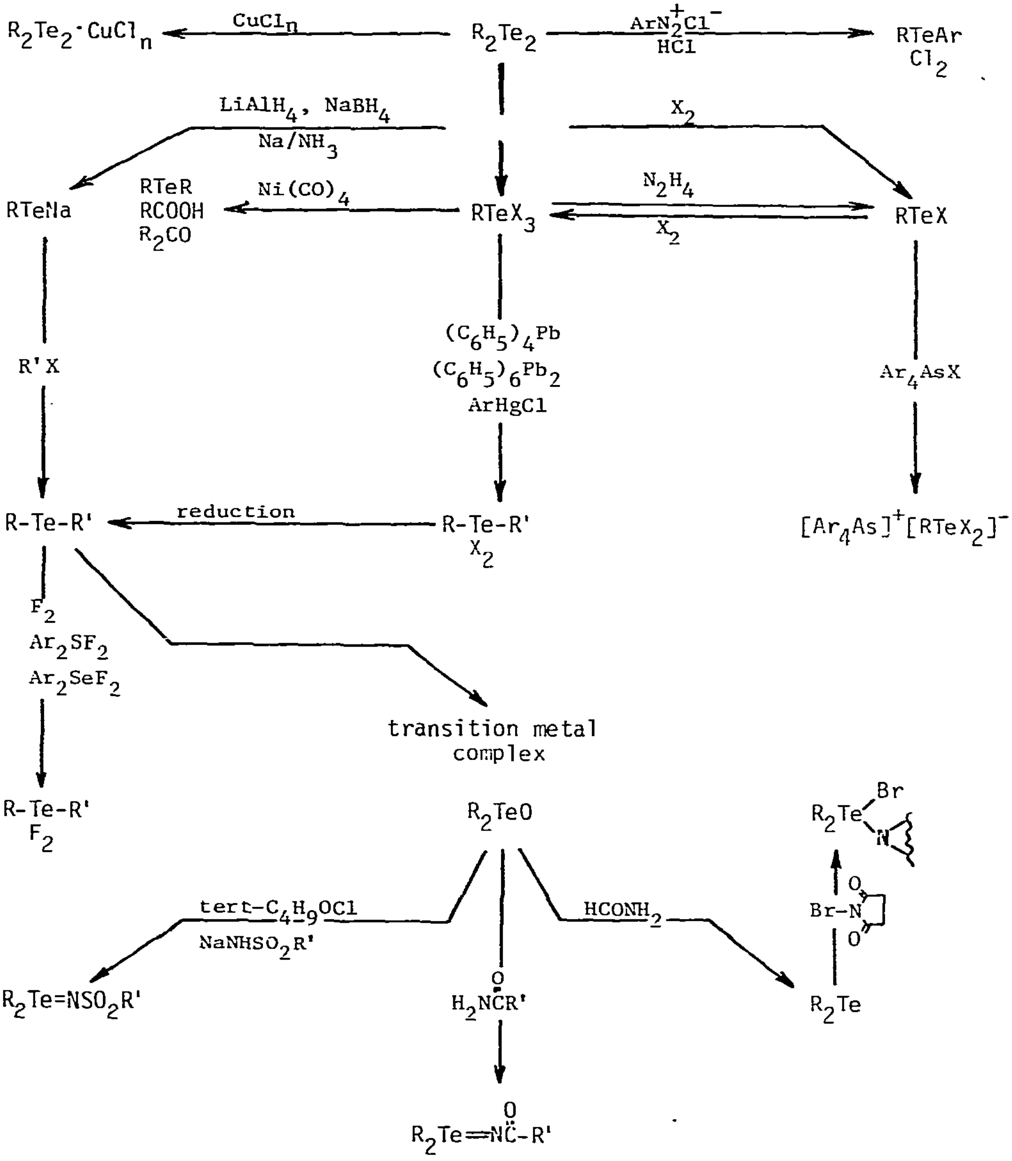
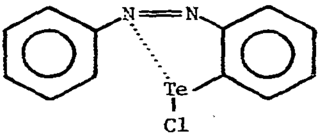


Fig. 2: Transformations of Organic Tellurium Compounds

sodium borohydride forming sodium salts in both instances. A certain probably small fraction of the organyltellurolate will always be present in protonated form. The concentration of tellurol in a solution will be determined by the unknown pK_a of the tellurol and the properties of the medium. It might well be that the tellurol is the species actually reacting, e.g., in addition reactions with carbon-carbon multiple bonds. However, experimental proof for such a mechanism is not available. Therefore, all reactions of the products obtained by reduction of diorganyl ditellurides are discussed in other pertinent sections and summarized in section IX-A.

B. Tellurenyl Compounds, RTeX

2-Nitrophenyl tellurium bromide, a reddish-black, crystalline solid with a melting point of 76°, was obtained in 90 percent yield by reacting bis(2-nitrophenyl) ditelluride or 2-nitrophenyl benzyl telluride with an equimolar amount of bromine in carbon tetrachloride solution. The reduction of 2-nitrophenyl tellurium tribromide with two molar equivalents of sodium hydrogen sulfite in water at 0° also produced the 2-nitrophenyl tellurium bromide [50]. Refluxing 2-phenylazophenyl tellurium trichloride and hydrazine hydrate in methanol yielded brown crystals of the tellurenyl halide 8 [37].



mp. 80°; 80% yield

8

The reactions of phenyl tellurium bromide and iodide, obtained from diphenyl ditelluride and bromine or iodine with arsonium halides [2,3,51] are described in section V-C. The conversion of aryl tellurium halides to trihalides [37,50] (section V-D), the reduction of compound 8 to bis(2-aminophenyl) ditelluride [50] (section V-E) and the reaction of benzyl tellurocyanate with 2-nitrophenyl lithium to produce diorganyl tellurides [50] (section VI-A) are treated in the indicated section.

C. Organyldihalotellurium Compounds, $[RTeX_2]^-M^+$

Several compounds of the general formula $[RTeXY]^-M^+$ (X, Y = Br, I, CN, SCN, SeCN; M = tetraorganylammonium, -phosphonium, or -arsonium) have been reported [2]. Klaboe and coworkers [51] prepared tetraphenylarsonium phenyldihalotellurates(II) according to eqn. (5). The unit cell dimensions of the orange-red crystals were determined.

(5) $$(C_6H_5)_2Te_2 \xrightarrow[CH_3CN]{1\ X_2} \underset{\textit{not isolated}}{C_6H_5TeX} \xrightarrow{(C_6H_5)_4AsY} [(C_6H_5)_4]^+[(C_6H_5TeXY]^-$$

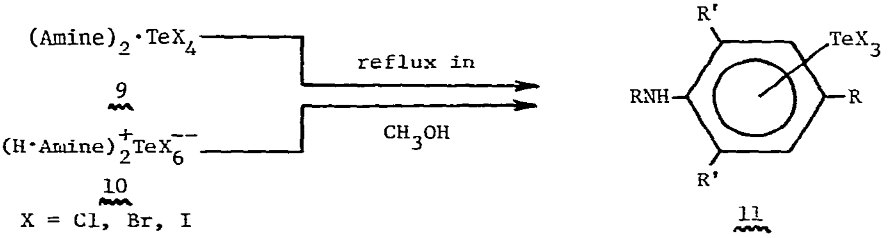
X, Y, mp. °C, yield %: Br, Cl, 138-40°, 80%;
Br, I, 168-70°, 86%;
Br, Br, 158-60°, 85%;
I, I, 174-76°, 88%.

D. Organyl Tellurium Trihalides

A number of new organyl tellurium trihalides were prepared from aromatic amines and tellurium tetrahalides, by arylation of tellurium tetrachloride with aromatic organometallic compounds, by addition of tellurium tetrachloride to olefins and acetylenes and by reaction of ditellurides or organyl tellurium halides with elemental halogen.

When the amine-tellurium tetrachloride adducts 9 or the hexahalotellurate salts 10 were heated in methanol the aminoaryl tellurium trihalides 11 were obtained (eqn. 6) [41]. These trihalides are crystalline solids, which are fairly stable in dry air but are easily hydrolyzed by moisture to tellurium dioxide and the amine hydrohalides. They are soluble in solvents of high dielectric constant, insoluble in nonpolar solvents and behave as 1:1 electrolytes in acetone, acetonitrile or nitrobenzene solution. Neither yields nor melting points are reported.

(6)

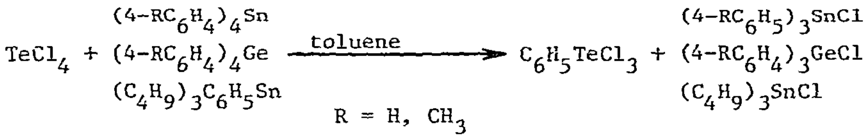


Amine = aniline, N-methylaniline, N,N-dimethylaniline,
4-methylaniline, 2-methylaniline, 2,4-dimethylaniline,
2,6-dimethylaniline.

The TeX_3 groups were postulated to occupy the position para to the amino groups [41]. The position of the TeX_3 group in the 4-methylaniline derivatives is uncertain.

Aryl tin and aryl germanium compounds transferred one aryl group to convert tellurium tetrachloride to aryl tellurium trichlorides (eqn. 7) [40] in 70 to 90 percent yield. Tetraaryl germanium had to be refluxed in toluene for the reaction to occur, whereas tetraaryl tin reacted with tellurium tetrachloride at room temperature to produce aryl tellurium trichlorides. Refluxing equimolar mixtures of tetra(tolyl) tin and $TeCl_4$ produced ditolyl tellurium dichloride. The experiments with tributyl phenyl tin showed that phenyl groups are preferentially transferred [40]. Attempts to prepare aryl tellurium trichlorides by reacting equimolar amounts of tellurium tetrachloride and diaryl indium chloride afforded only diaryl tellurium dichlorides [40].

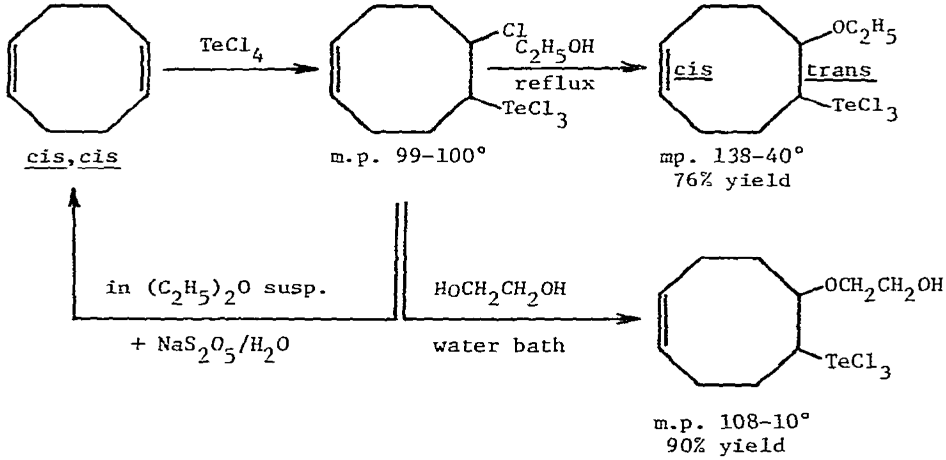
(7)



$$TeCl_4 + \begin{array}{l}(4\text{-}RC_6H_4)_4Sn\\(4\text{-}RC_6H_4)_4Ge\\(C_4H_9)_3C_6H_5Sn\end{array} \xrightarrow[R\ =\ H,\ CH_3]{\text{toluene}} C_6H_5TeCl_3 + \begin{array}{l}(4\text{-}RC_6H_5)_3SnCl\\(4\text{-}RC_6H_4)_3GeCl\\(C_4H_9)_3SnCl\end{array}$$

Tellurium tetrachloride and cis,cis-1,5-cyclooctadiene kept for 12 hours in dry, cold, ethanol-free chloroform formed 2-chloro-cyclooct-5-en-1-yl tellurium trichloride in 64 percent yield. The compound can be stored at -15° for several months, but decomposes slowly at room temperature in air to a black tar [35]. The reactions of this compound are summarized in eqn. (8).

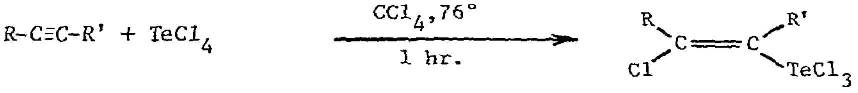
(8)



4-R-Cyclohexene (R = H [1]; R = CH_3, new compound) and equimolar amounts of tellurium tetrachloride formed 4-R-cyclohexyl tellurium trichlorides in quantitative yields. The trichlorides reacted with additional tellurium tetrachloride to give benzene or toluene [36].

The addition of tellurium tetrachloride to organylacetylenes produced *cis*-2-chlorovinyl tellurium trihalides, which reacted with iodine or N-bromosuccinimide to give *cis*-2-chloro-1-haloethylene [42] (eqn. 9).

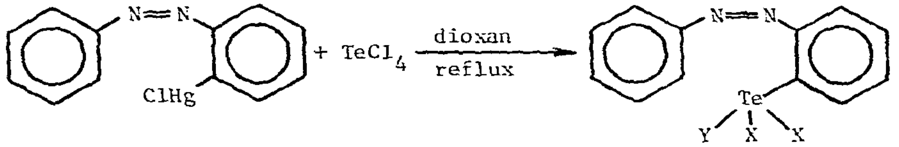
(9)



R, R', mp. °C, yield %:			
CH_2OH,	H,	–,	–;
C_6H_5,	H,	200°(dec),	86%;
C_6H_5,	CH_3,	oil,	>90%;
C_6H_5,	C_2H_5,	oil,	>90%;
C_6H_5,	C_6H_5,	97–103°,	76%.

4-Methoxybenzoylmethyl tellurium trichloride [P-3] was isolated when tellurium tetrachloride was condensed with 4-methoxyacetylbenzene in chloroform at 0°. Orange 2-phenylazophenyl tellurium trichloride 12 was synthesized as described in eqn. 10 [37].

(10)



12

X = Y = Cl: mp. 235°; 100% yield

The tribromide of compound 12 (X = Y = Br) (mp. 245°) was obtained by refluxing 2-phenylazophenyl 4-ethoxyphenyl telluride and bromine in carbon tetrachloride. The chloride dibromide 12 (Y = Cl, X = Br) (mp. 145°) was formed from the monochloride and bromine [37].

2-Nitrophenyl tellurium tribromide (mp. 165°) was synthesized from 2-nitrophenyl tellurium bromide and bromine in CCl_4 (75% yield) and by heating 2-nitrophenyl benzyl telluride and bromine in CCl_4 (95% yield) [50].

2-Benzylphenyl tellurium trichloride, yellowish crystals melting at 199-200°, was formed in quantitative yield from the ditelluride and chlorine in CH_2Cl_2 [22]. The cyclization of this compound [22] and of 2-phenyl-2-chlorovinyl tellurium trichloride [45] is discussed in section X.

Refluxing 4-methoxyphenyl tellurium trichloride or 2-naphthyl tellurium trichloride and nickel tetracarbonyl in dry dimethylformamide yielded RCOOH, R_2CO and R_2Te [52].

The reactions of aryl tellurium trichlorides with tetraphenyl lead, hexaphenyl dilead [39] or aryl mercury chloride [37] are treated in section VI-B2. The reduction of aryl tellurium trihalides to tellurenyl compounds [37,50] and to ditellurides [50,52] is presented in sections V-B and V-E, respectively.

E. Diorganyl Ditellurides

The dialkyl ditellurides, R_2Te_2 (R = CH_3 [24,26a,28], i-C_3H_7 [26a,67,68], C_6H_{13} [25,26]; C_8H_{17} [25,26,68], $C_{16}H_{33}$ [26a], $HOOCCH_2$ [27], $HOOC(CH_2)_7CH=CH(CH_2)_6$ [29], labeled with gamma-active Te-123m were prepared from disodium ditelluride and the appropriate alkyl halide in liquid ammonia [24-26] or in ethanol [28,29].

Bis(4-methoxyphenyl) ditelluride was isolated in 40 percent yield when the aryl tellurium trichloride was heated with diiron nonacarbonyl in dry dimethylformamide at 70° for 24 hours [52]. Trifluoromethyl radicals generated from C_2F_6 and vaporized tellurium yielded bis(trifluormethyl) ditelluride [21]. As reported earlier in several publications [2,3], $Te_n(AsF_6)_2$ (n = 4,6) and tetrafluoroethylene at 100° or in liquid sulfur dioxide formed bis(pentafluoroethyl) ditelluride [47].

Bis(2-nitrophenyl) ditelluride (mp. 204-6°) was obtained in 91 percent yield when the aryl tellurium tribromide was reduced by sodium hydrogen sulfite [50]. The reduction of 2-phenylazophenyl tellurium chloride by sodium borohydride in boiling methanol afforded red, crystalline bis(2-aminophenyl) ditelluride (mp. 100°) [37]. The product of the reaction between tellurium and 2-benzylphenyl lithium was transformed into bis(2-benzylphenyl) ditelluride upon treatment with hydrochloric acid in the presence of air. The red, crystalline compound melted at 101° and was obtained in 78 percent yield [22].

The diorganyl ditellurides were subjected to the following reactions which are discussed in the indicated sections: conversion to organyl tellurium halides, RTeX, [50,51] (section V-B); halogenolysis to aryl tellurium trihalides [22] (section V-D); reduction by sodium borohydride to organyl sodium tellurides [24,25,26,28,29,49,53,54,67,68, P-1], by lithium borohydride to organyl lithium tellurides [55], by sodium in liquid ammonia [56] and by dithiothreitol in alkaline medium [27] (section IX-A); reaction with diazonium salts in the presence of copper acetate to give unsymmetric diaryl tellurium dichlorides [57] (section VI-B2); and formation of copper chloride complexes [37] (section IX-E).

VI. Compounds Containing a Carbon-Tellurium-Carbon Moiety

Symmetric and unsymmetric diorganyl tellurides, symmetric and unsymmetric diorganyl tellurium dihalides, telluracyclopentane dipseudohalides and diorganyl telluroxides were investigated.

A. Diorganyl Tellurides

Several new diorganyl tellurides, which are with a few exceptions of the unsymmetric type R-Te-R', were prepared. Among these compounds are telluroamino acids, tellurohydantoins, telluracarboxylic acids and tellurium-containing steroids.

1. Symmetric diorganyl tellurides

Several new tellurides, marked by asterisks, were synthesized and a few previously reported compounds were obtained by new methods.

Kozyrkin [33] prepared the already known dialkyl tellurides [1], $(C_nH_{2n+1})_2Te$ (n = 1, 2, 3, 5] in 70 percent yield from disodium telluride and the alkyl halides and investigated their purification in relation to their use in the production of semiconductors. Dihexadecyl telluride was synthesized similarly from sodium hydrogen telluride [26a].

The trimethylsilylalkyl chlorides, $(CH_3)_3Si(CH_2)_nCl$ (n = 1, 3), refluxed in methanol with disodium telluride produced the bis(trimethylsilylalkyl) tellurides* in 95 percent yield. The compounds boiled at 33°/3 torr (n = 1) and 51°/0.003 torr (n = 3). They deposited tellurium on prolonged storage in air [32].

Bis(1,3-butadien-1-yl) telluride* was obtained in four percent yield as a mixture of isomers when tellurium and but-3-en-1-yne [20] or acetylene [P-13] were reacted with each other.

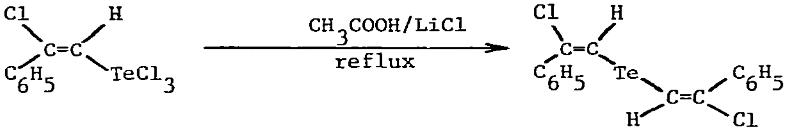
The reactions described by eqn. 11-18 were employed to prepare diorganyl tellurides.

$$(11)\qquad Te \xrightarrow[\text{gas phase}]{CF_3\cdot} (CF_3)_2Te \qquad [21]$$

10% yield

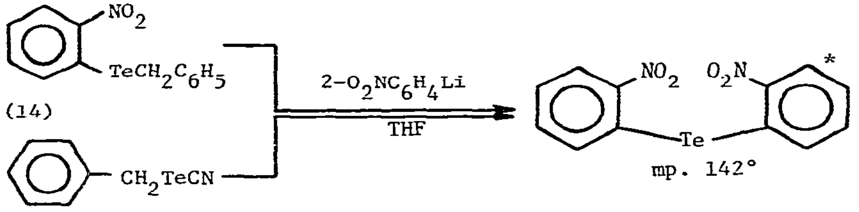
$$(12)\qquad Te_n(AsF_6)_2 \xrightarrow[n\,=\,4,\ 6]{C_2F_4} (C_2F_5)_2Te \qquad [47]$$

(13)



[45]

(14)



20% yield

[50]

2% yield

(15) $C_6H_5TeNa + C_6H_5X \xrightarrow[NH_3(\ell)]{h\nu} (C_6H_5)_2Te$ [56]

X = I, 90% yield,; X = Br, yield not reported.

(16) $RTeCl_3 \xrightarrow[DMF,70°]{Ni(CO)_4} R_2Te$ [52]

R, yield %: 4-$CH_3OC_6H_4$, 15%; 2-naphthyl, trace.

(17) $(4\text{-}RC_6H_4)_2TeCl_2 \xrightarrow[DMF,70°]{Ni(CO)_4} (4\text{-}RC_6H_4)_2Te$ [52]

R, yield %: H, 16%; CH_3O, 15%.

(18) $(4\text{-}RC_6H_4)_2TeO \xrightarrow[-CO_2,-NH_3]{HC(O)NH_2,150°} (4\text{-}RC_6H_4)_2Te$ [58]

R, mp. °C, yield %: H, –, 100%; CH_3, –, 100%; CH_3O, –, 100%; $(CH_3)_2N$,* 67°, 95%.

The claim that $(CH_3)_2X$ molecules, among them dimethyl telluride, have a linear skeleton [60] has been refuted and called untenable. It should be disregarded because the conclusion was based on the misinterpretation of poor-quality vibrational spectra [59].

According to proton nmr data diethyl telluride and tellurium dibromide reacted in $CDCl_3$ to produce some C_2H_5TeBr [61]. Diethyl telluride did not react with nickel tetracarbonyl [52].

The reactions of diorganyl tellurides with fluorine, $C_6H_5)_2SF_2$ or $(C_6H_5)_2SeF_2$ [62] (section VI-B1), with N-haloimides [63] (section IX-C), with tert-butyl hypochlorite followed by N-sodium alkylsulfonamide [64] (section VIII) and with transition metal compounds [32,65,66] (section IX-E) are described in the indicated sections..

2. Unsymmetric diorganyl tellurides

Most of the unsymmetric diorganyl tellurides prepared during this survey period were synthesized by reacting organyl alkali metal tellurides, which are easily obtained from diorganyl ditellurides (sections V-E, IX-A), with alkyl halides. The new compounds are marked by an asterisk.

When aryl alkyl tellurides are to be prepared, the preferred starting materials are aryl alkali metal tellurides and alkyl halides. Several new tellurides were obtained in this manner (eqn. 19). The aryl heptafluoropropyl tellurides are light-yellow liquids, which are not hydrolyzed by water, but are unstable in the presence of oxygen [55].

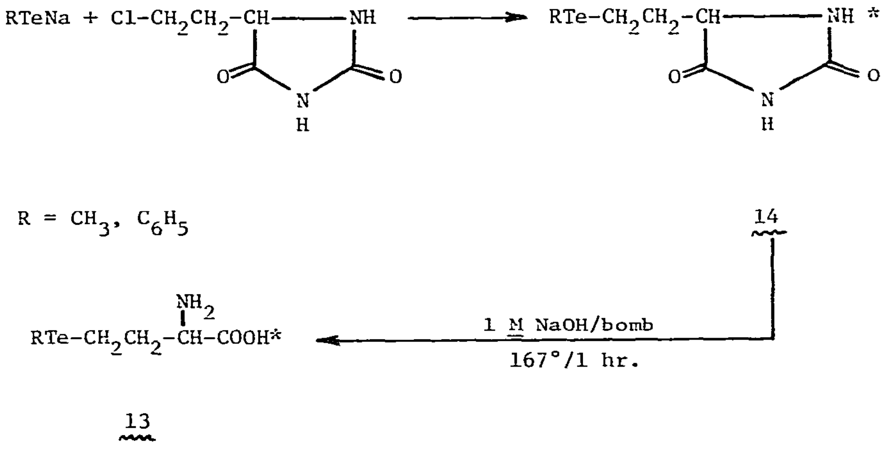
(19) $RC_6H_4Te^-M^+ + R'X \longrightarrow RC_6H_4\text{-}Te\text{-}R'$

R, M, R', X, bp. °C/torr, yield %: H, Na, CH_3, I, –, 80% [56];
H, Na, $CH_2CH(OCH_3)_2$*, Br, oil, 87% [48,49,53];
H, Li, C_3F_7,* I, 80°/15, 44% [55];
3-F, Li, C_3F_7,* I, 84°/16, 41% [55];
4-F, Li, C_3F_7,* I, 84°/16, 42% [55];
4-Br, Li, C_3F_7,* I, 84°/1, 59% [55];

Treatment of 2,2-dimethyoxyethyl phenyl telluride with ethanol or CD_3OD in the presence of 1 M HCl afforded 2,2-diethoxyethyl* (33% yield) and 2,2-bis(trideuteriomethoxy)ethyl phenyl telluride* (25% yield) [53]. Attempts to hydrolyze these acetals to the free aldehyde caused decomposition [48,49].

A route to telluroamino acids 13 via organyltellurohydantoins 14 was discovered (eqn. 20) [24,48,49]. The hydantoin 14 (R = CH_3) was obtained

(20)



in 55 percent yield. It melted at 132°. Hydrolysis of the hydantoins by 1 M sodium hydroxide in a bomb at 167° gave the telluroamino acids, which could not be obtained entirely pure [24].

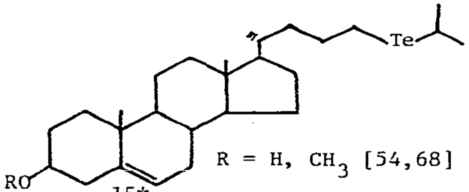
Telluraalkanoic acids and telluraalkanoic esters labeled with Te-123m were prepared according to eqn. 21. The esters were purified by chromatography.

(21) $RTeNa + R'X \longrightarrow R\text{-}Te\text{-}R'$

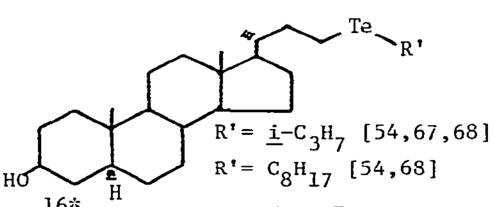
R, X, R': C_6H_{13}, Br, $(CH_2)_7COOCH_3$;* C_8H_{17}, Br, $(CH_2)COOCH_3$* [25,26]; $(CH_2)_5CH{=}CH(CH_2)_7COOH$, I, CH_3* [29].

The oily compounds were stable under argon at 0° for several weeks. Refluxing the esters with 1 M $NaOH/H_2O/C_2H_5OH$ yielded the telluraalkanoic acids, which are relatively unstable [25,26].

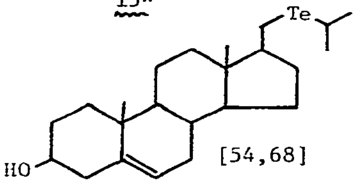
Tellurosteroids 15-20 labeled with Te-123m were similarly obtained in chemical yields ranging from 10 to 50 percent from alkyl sodium tellurides and bromosteroids. The tellurosteroids were purified by column chromatography.


R = H, CH_3 [54,68]

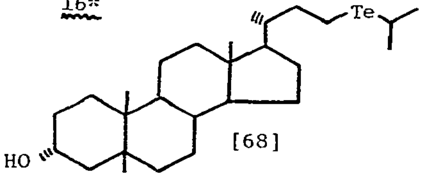
15*


R'= i-C_3H_7 [54,67,68]
R'= C_8H_{17} [54,68]

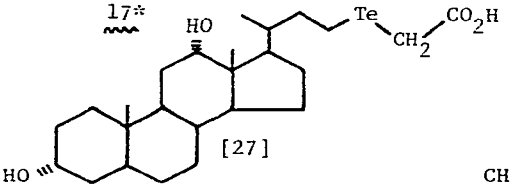
16*


[54,68]

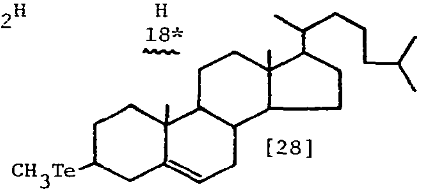
17*


[68]

18*


[27]

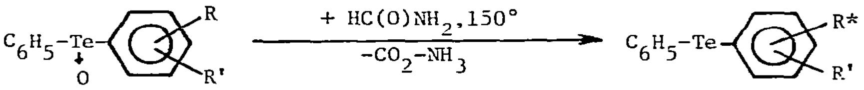
19*


[28]

20*

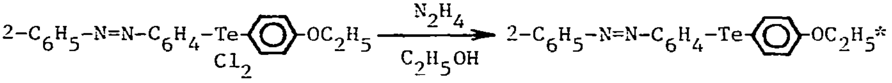
Several other unsymmetric diorganyl tellurides were prepared as shown in eqn. 22-27.

(22)



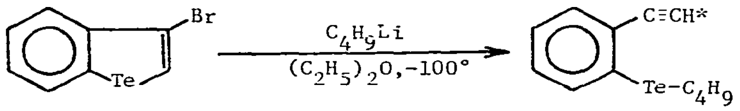
R, R', mp. °C, yield %: 3-CH_3O, 4-CH_3O, 63°, 100%;
2-CH_3O, 5-CH_3O, 60°, 100% [58].

(23)



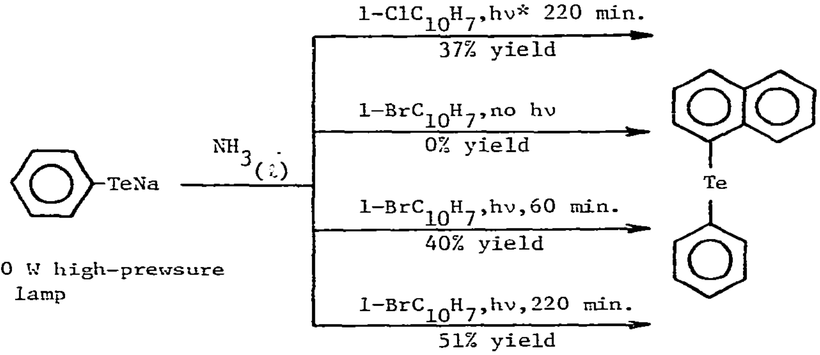
mp. 120° [37]

(24)



[45]

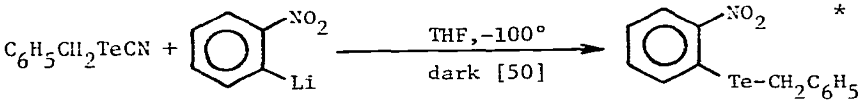
(25)



[56]

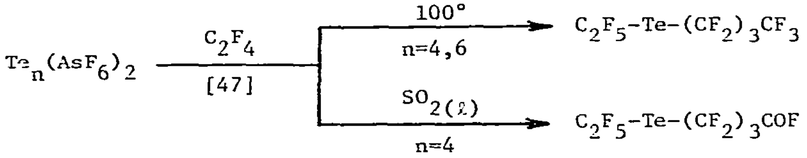
*250 W high-prewsure uv lamp

(26)



mp. 85-6°; 72% yield

(27)



The electrochemical oxidation of the tellurides 4-RC_6H_4-Te-C≡C-C_6H_5 (R = H, mp. 26°; R = CH_3, mp. 69°; R = Cl,* mp. 105°) in acetonitrile [0.08 M $(C_2H_5)_4NClO_4$] proceeded via an unstable radical cation. These acetylenyl tellurides formed charge-transfer complexes with tetracyanoethylene [69].

Heating phenyl 2-hydroxy-2-phenylethyl telluride in refluxing toluene or treating the compound in methanolic solution at 20° with perchloric acid gave vinylbenzene in approximately 16 percent yield [70].

The reactions of unsymmetric diorganyl tellurides with fluorine [62] (section VI-B2), with bromine [32,50] (section V-D), with N-haloimides [63] (section IX-C), and with 2-nitrophenyl lithium [50] (section VI-A1, eqn. 14) are discussed in the indicated sections.

B. Diorganyl Tellurium Compounds, R_2TeX_2

The arylation of tellurium tetrachloride with aryl lead, aryl tin, aryl indium or aryl thallium compounds produced diaryl tellurium dihalides. The fluorination of diorganyl tellurides with fluorine or $(C_6H_5)_2XF_2$ (X = S, Se) yielded diorganyl tellurium difluorides. Heterocyclic compounds containing the TeX_2 group are discussed in section X. The compounds RR'TeX(N<) are treated in section IX-C.

1. Symmetric diorganyl tellurium dihalides, R_2TeX_2 (X = F, Cl, Br, I)

New diorganyl tellurium dihalides were not reported during the survey period, but several known compounds were prepared by new routes.

Dimethyl tellurium difluoride and diphenyl tellurium difluoride were synthesized using fluorine diluted by argon and the respective diorganyl telluride [62] (eqn. 28). Diphenyl tellurium difluoride was also formed when the telluride was treated with $(C_6H_5)_2XF_2$ (X = S, Se) in chloroform solution [62].

$$(28)\quad R_2Te \xrightarrow[CFCl_3,\,-78°]{+\ F_2/Ar(1:5)} R_2TeF_2$$

R, mp. °C, yield %: CH_3, 82°, 22%; C_6H_5, 147°, 94%.

The preparation of diorganyl tellurium dichlorides from tellurium tetrachloride and aryl metal compounds in toluene is described by eqn. (29). The reactions with the organic lead compounds proceeded at room temperature; the other reactions were carried out in refluxing toluene. The methyl lead compounds reacted slower than the aryl lead derivatives.

$$(29)\quad TeCl_4 + n\ \text{aryl metal compound} \longrightarrow Ar_2TeCl_2$$

Aryl metal compd., n, yield %: $(C_6H_5)_2TlCl$, 2, 94% [40]; $(CH_3)_4Pb$, 1, 80% [39]; $(4\text{-}CH_3C_6H_4)_2InCl$, 2, 86% [40]; $(CH_3)_6Pb_2$, 0.5, 78% [39]; $(C_6H_5)_2InCl$, 2, 96% [40]; $(C_6H_5)_6Pb_2$, 0.5, 94% [39]; $(C_6H_5)_2(C_4H_9)_2Sn$, 1, 84% [40]; $(4\text{-}CH_3C_6H_4)_6Pb_2$, 0.5, 86% [39]; $(4\text{-}CH_3C_6H_4)_4Sn$, 1, 90% [40].

Treatment of diphenyl ditelluride in acetone with a hydrochloric acid solution of benzenediazonium chloride in the presence of copper(II) acetate gave diphenyl tellurium dichloride in 86 percent yield [57] (see section VI-B2). The diaryl tellurium dichlorides, $(4\text{-}RC_6H_4)_2TeCl_2$ ($R = H, CH_3O$), were obtained when their sulfonimides, $(4\text{-}RC_6H_4)_2Te{=}NSO_2R'$, were reacted with dry hydrogen chloride [64].

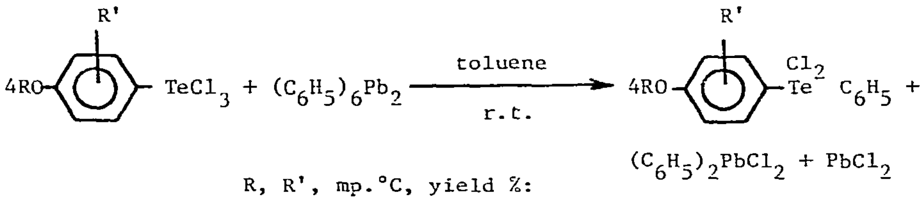
The reactions of $(4\text{-}RC_6H_4)_2TeCl_2$ ($R = H, CH_3O$) with $Ni(CO)_4$ in dimethylformamide at 70° produced diaryl tellurides (∿15%), $4\text{-}RC_6H_4COOH$ (∿65%) and diaryl ketones, nickel telluride, carbon monoxide and hydrogen chloride. A similar reaction with bis(2-chloropropyl) tellurium dichloride gave a black precipitate of NiTe, but no acid, ketone or telluride. The starting tellurium dichloride seems to be unstable under these conditions decomposing into propene, $NiCl_2$ and NiTe [52].

2. Unsymmetric diorganyl tellurium dihalides, $RR'TeX_2$ ($X = F, Cl, Br, I$)

Four new unsymmetric diorganyl tellurium dihalides, marked by asterisks in the equations and in the text below, were synthesized during the survey period.

The reactions of hexaphenyl dilead with aryl tellurium trihalides produced phenyl aryl tellurium dihalides in high yields (eqn. 30) [39].

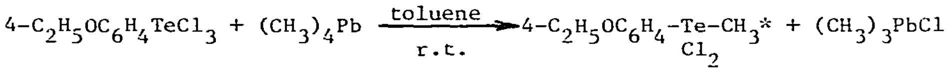
(30)



R, R', mp.°C, yield %:
CH_3, H, 113°, 89%;
C_2H_5, H, 114°, 92%;
CH_3, CH_3O*, 148°, 73%;
CH_3, CH_3*, 108°, 78%.

4-Ethoxyphenyl methyl tellurium dichloride* was similarly obtained in 75% yield employing tetramethyl lead (eqn. 31).

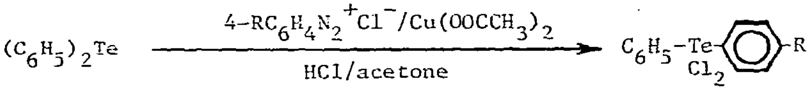
(31)



$$4\text{-}C_2H_5OC_6H_4TeCl_3 + (CH_3)_4Pb \xrightarrow[\text{r.t.}]{\text{toluene}} 4\text{-}C_2H_5OC_6H_4\text{-}\underset{Cl_2}{Te}\text{-}CH_3^{*} + (CH_3)_3PbCl$$

mp. 93-95°

Hot solutions of diphenyl ditelluride and copper(II) acetate in acetone mixed with hydrochloric acid solutions of arenediazonium chlorides yielded diaryl tellurium dihalides (eqn. 32). It has been postulated that the initially formed diaryl tellurides were oxidized by copper(II) chloride (from copper acetate and HCl) to diaryl tellurium dichlorides [57].

(32) 

R, mp. °C, yield %: CH_3O, 139°, 83%; F,* 134°, 85%.

Methyl phenyl tellurium difluoride,* which melted at 73° and sublimed at 80°/0.001 torr, was obtained in 53 percent yield by fluorination of methyl phenyl telluride in $CFCl_3$ solution with fluorine/argon (1:5) at -78° [62].

The condensation of 4-ethoxyphenyl tellurium trichloride with 2-phenylazophenyl mercury chloride in refluxing dioxane gave 4-ethoxyphenyl 2-phenylazophenyl tellurium dichloride.* This compound was not purified. The crude material was reduced with hydrazine to the telluride (section VI-A2) [37].

3. Diorganyl tellurium compounds, R_2TeX_2 (X = NC, NCO, NCS, NCSe, N_3)

Telluracyclopentane dipseudohalides and telluracyclohexane dipseudohalides were prepared during this survey period. These compounds are discussed in sections X-A and X-H.

4. Diorganyl tellurium dicarboxylates

Only heterocyclic tellurium dicarboxylates were prepared during this survey period. These compounds are discussed in sections X-A and X-H.

C. Diorganyl Telluroxides

The known diaryl telluroxides, $(4\text{-}RC_6H_4)_2TeO$ (R = H, CH_3O), were obtained through hydrolysis of the sulfonimides, $(4\text{-}RC_6H_4)_2Te{=}NSO_2R'$, with aqueous alkali hydroxide solutions or with boiling water [64]. The reactions of diaryl telluroxides with acyl amides [64] and formamide [58] are discussed in sections VIII and VI-A, respectively.

VII. Triorganyl Telluronium Compounds, $[R_3Te]^+X^-$

Ziolo [71] prepared trimethyl telluronium tetraphenylborate and triphenyl telluronium tetraphenylborate as voluminous precipitates by adding an aqueous solution of sodium tetraphenylborate to the aqueous solutions of the telluronium chlorides. The structure of trimethyl telluronium tetraphenylborate was determined [71] (section XI-G).

VIII. Tellurium Ylides

Publications dealing with tellurium ylides, $R_2Te{=}CR'_2$, did not appear during the survey period. The compounds $R_2Te{=}NSO_2R'$ are discussed in section IX-C.

IX. Organic Tellurium Compounds Containing a Tellurium-Metal, Tellurium Metalloid, Tellurium-Nitrogen, Tellurium-Sulfur or Tellurium-Selenium Bond

During the period covered by this survey, organic tellurium compounds containing a Te-Li, Te-Na, Te-Ge, Te-Sn, Te-N, Te-S, Te-Se, Te-Ta, Te-Ru, Te-Rh, Te-Pd, Te-Cu, and Te-Hg bond were investigated.

A. Organic Compounds of Tellurium with Alkali Metals

Organyl alkali metal tellurides are oxygen-sensitive and difficult to handle and are, therefore, used *in situ*.

Treatment of the diaryl ditellurides, $(RC_6H_4)_2Te_2$ (R = H, 3-F, 4-F, 4-Br), with lithium aluminum hydride under an argon atmosphere in anhydrous diethyl ether generated aryl lithium tellurides [55], which were reacted with heptafluoropropyl iodide to give aryl heptafluoropropyl tellurides (section VI-A2). 2-Benzylphenyl lithium and tellurium in diethyl ether yielded 2-benzylphenyl lithium telluride, which was oxidized to the ditelluride [22] (section V-E).

Sodium borohydride converted diorganyl ditellurides to organyl sodium tellurides (eqn. 33) (see also section V-A). Ethanol, methanol, benzene-methanol or benzene-ethanol with or without sodium ethoxide or aqueous sodium hydroxide served as the reaction media. The organyl sodium tellurides were alkylated to yield unsymmetric diorganyl tellurides (section VI-A2) or phenyl triphenylgermyl telluride [P-1] (section IX-B).

$$(33)\quad R_2Te_2 \xrightarrow[\text{organic solvent}]{NaBH_4} 2\ RTeNa$$

R: CH_3 [24,28]; $\underline{i}\text{-}C_3H_7$ [54,67,68]; C_6H_{13} [25,26]; C_8H_{17} [25,26,54,68];
C_6H_5 [53,P-1]; $HOOC(CH_2)_7CH{=}CH(CH_2)_6$ [29]; 3-cholesteryl [28].

Phenyl sodium telluride, generated from diphenyl ditelluride and sodium in liquid ammonia, was reacted with organic halides to produce diorganyl tellurides [56] (section VI-A). Carboxymethyl sodium telluride was obtained by reduction of the ditelluride with alkaline dithiothreitol and subsequently used to prepare a tellurosteroid [27] (section VI-A2).

Alkylation of sodium hydrogen telluride with alkyl halides yielded RTeH(Na) (R = $\underline{i}$-C_3H_7, $C_{16}H_{13}$) which were converted to ditelluride (R = $\underline{i}$-C_3H_7) or the telluride (R = $C_{16}H_{13}$) [26a].

Tetraphenylphosphonium benzenetellurolate reacted with $[(C_6H_5)_4P]^+[Hg(TeC_6H_5)_3]^-$ to give $[(C_6H_5)_4P]_5[Hg_3(TeC_6H_5)_{11}]$ [72] (section IX-F).

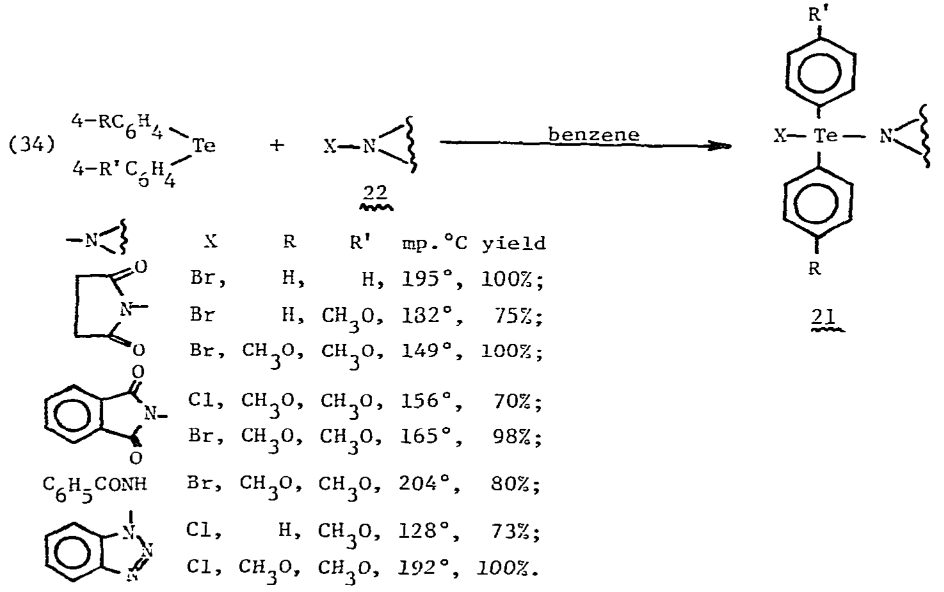
B. Organic Compounds of Tellurium Containing a Tellurium-Group IV Element Bond

Phenyl triphenylgermyl telluride was prepared in 61 percent yield from phenyl sodium telluride and triphenylgermyl chloride [P-1].

The scrambling of neat mixtures of bis(trimethylgermyl) telluride and digermyl telluride to give $(CH_3)_3Ge$-Te-GeH_3 was slow on the nmr time scale [73].

C. Organic Compounds of Tellurium Containing a Tellurium-Nitrogen Bond

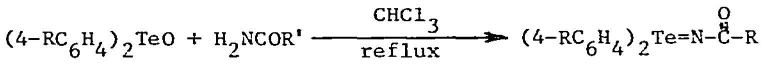
Srivastava [63] synthesized diorganyl tellurium halide imides 21 by reacting diorganyl tellurides with N-haloimides 22 (eqn. 34).



	X	R	R'	mp. °C	yield
	Br,	H,	H,	195°,	100%;
	Br	H,	CH_3O,	182°,	75%;
	Br,	CH_3O,	CH_3O,	149°,	100%;
	Cl,	CH_3O,	CH_3O,	156°,	70%;
	Br,	CH_3O,	CH_3O,	165°,	98%;
C_6H_5CONH	Br,	CH_3O,	CH_3O,	204°,	80%;
	Cl,	H,	CH_3O,	128°,	73%;
	Cl,	CH_3O,	CH_3O,	192°,	100%.

These compounds are monomeric in freezing nitrobenzene and behave as non-electrolytes in acetone. The tellurium-nitrogen bond is stable toward methanol [63].

Diaryl tellurium imides, $R_2Te{=}NCOR'$ 23, were obtained in quantitative yields by refluxing diaryl telluroxides and amides in chloroform (eqn. 35). Compound 23 [$R = (CH_3)_2N$, $R' = 4\text{-}O_2NC_6H_4$] formed the monohydrate on

(35) 

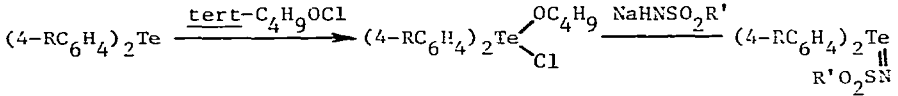
$$(4\text{-}RC_6H_4)_2TeO + H_2NCOR' \xrightarrow[\text{reflux}]{CHCl_3} (4\text{-}RC_6H_4)_2Te{=}N{-}\overset{\overset{O}{\|}}{C}{-}R$$

23

R, R', mp. °C: CH_3O, CCl_3, 152°; $(CH_3)_2N$, CCl_3, 162°;
$(CH_3)_2N$, $4\text{-}O_2NC_6H_4$, 213°; $(CH_3)_2N$, $3\text{-}O_2NC_6H_4$, 200°;
$(CH_3)_2N$, $4\text{-}ClC_6H_4$, 151° (isolated as monohydrate).

recrystallization from water or on standing in air. It was dehydrated by heating to 155°. The water could not be removed from compound 23 [$R = (CH_3)_2N$, $R' = 4\text{-}ClC_6H_4$].

The synthesis of diaryl tellurium sulfonimides was accomplished by treating diaryl tellurides first with tert-butyl hypochloride and then with N-sodium sulfonamides (eqn. 36).

(36) 

$$(4\text{-}RC_6H_4)_2Te \xrightarrow{tert\text{-}C_4H_9OCl} (4\text{-}RC_6H_4)_2Te\begin{matrix}OC_4H_9\\Cl\end{matrix} \xrightarrow{NaHNSO_2R'} (4\text{-}RC_6H_4)_2\underset{\underset{R'O_2SN}{\|}}{Te}$$

R, R', mp. °C, yield %: H, C_6H_5, 184°, 100%; CH_3O, C_6H_5, 96°, 100%;
$4\text{-}CH_3O$, $4\text{-}CH_3C_6H_4$, 152°, 100%;
$4\text{-}CH_3O$, $C_6H_5CH_2$, 167°, 100%.

A telluratetrazapentalene derivative [44] is described in section X-G.

D. Organic Compounds of Tellurium Containing a Tellurium-Sulfur or Tellurium-Selenium Bond

The reaction of phenyl tellurium 4-methylphenylthiosulfonate with sodium benzenethiolate yielded diphenyl sulfide telluride, $C_6H_5\text{-}S\text{-}Te\text{-}C_6H_5$, which melted at 58°. The attempt to prepare this compound from diphenyl ditelluride and benzenethiolate was not successful [74].

Bis(pentafluoroethyl) selenide telluride was formed from selenium impurities during the reaction of $Te_4(AsF_6)_2$ with C_2F_4 in liquid sulfur dioxide [47]. The heterocycle 1-aza-3-tellura-4-thiacyclopent-5-ene is discussed in section X-C.

E. Organic Tellurium Compounds as Ligands in Transition Metal Complexes

The ligand exchange reaction $TaX_5\cdot L + L^* \rightleftharpoons TaX_5\cdot L^* + L$ [$X = Cl, Br; L = (CH_3)_2Te$] was studied in methylene chloride as a function of pressure by proton FT nmr. These associative interchange reactions have a negative volume of activation [75].

The ruthenium(II) complex, $[(NH_3)_5Ru\cdot Te(CH_3)_2]^{++}$ was prepared from $[(NH_3)_5Ru(OH_2)](PF_6)_2$ and dimethyl telluride in deaerated acetone. The electrochemical behavior of this complex was investigated by cyclic voltammetry. Oxidation with oxygen, cerium(IV) or hydrogen peroxide yielded $[(NH_3)_5Ru\cdot Te(CH_3)_2]^{+++}$. Upon treatment of $[(NH_3)_5Ru\cdot Te(CH_3)_2]^{++}$ with aqueous 0.2 M HCl/0.1 M 4-cyanopyridine one of the NH_3 ligands was probably replaced by water, which was in turn exchanged for the 4-cyanopyridinium group [65].

Rhodium(III)-dimethyl telluride complexes were synthesized according to eqn. (37) [66].

(37)
$$\begin{tikzcd} K_3[RhX_6] \arrow[r, "(CH_3)_2Te", "\text{deaerated } C_2H_5OH/N_2"'] & \underline{mer}\text{-}\{RhX_3\cdot[Te(CH_3)_2]_3\} \arrow[dl, "X = Br", "+ NaI/acetone"'] \\ \underline{mer}\text{-}\{RhI_3\cdot[Te(CH_3)_2]_3\} & \end{tikzcd}$$

X, mp. °C: Cl, 159-61° (dec).
Br, dec.

$\underline{mer}$-{$RhI_3\cdot[Te(CH_3)_2]_3$} mp. (dec)

Palladium(II) complexes with bis(trimethylsilylaklyl) tellurides as ligands were prepared according to eqn. 38 and 39 [32]. The red

(38)
$$2[(CH_3)_3Si(CH_2)_n]_2Te + K_2Pd(SCN)_2 \xrightarrow[r.t.]{CH_3OH} Pd(SCN)_2\{Te[(CH_2)_nSi(CH_3)_3]_2\}_2$$

n, mp. °C, yield: 1, 143°, 88%;
3, 64°, 76%.

(39)
$$2[(CH_3)_3Si(CH_2)_n]_2Te \xrightarrow[n = 3;\ + Na_2PdCl_4/H_2O, CH_3OH, r.t.]{n = 1;\ + PdCl_2(NCC_6H_5)_2/C_6H_6, r.t.} PdCl_2\cdot\{Te[(CH_2)_nSi(CH_3)_3]_2\}_2$$

n, mp. °C, yield: 1, 99°, 56%;
3, 70°, 71%.

complexes have a trans-configuration according to ir and Raman data [32].

The copper chloride complexes, $(2\text{-}H_2NC_6H_4)_2Te_2\cdot CuCl_n$ [n = 1, mp. 140°; n = 2, mp. 90° (dec)], formed when either Cu_2Cl_2 or $CuCl_2$ dissolved in acetonitrile was mixed with diethyl ether solutions of the ditelluride. The complexes are 1:1 electrolytes in acetonitrile. When the $CuCl_2$ complex was reacted with a solution of ethylenediaminetetracetic acid, the ditelluride was set free [37].

F. Organic Compounds of Tellurium Containing a Tellurium-Mercury Bond

The reaction of bis(phenyltelluro) mercury with phenyl sodium telluride in liquid ammonia produced the anion $[(C_6H_5Te)_3Hg]^-$, which was isolated as the tetraphenylphosphonium salt (mp. 130°, dec.) in 80 percent yield. The anion has - according to ir and Raman data - a planar $\mathrm{Te{-}Hg}{<}^{\mathrm{Te}}_{\mathrm{Te}}$ skeleton. The amber-colored salt is stable in dry air, but decomposes in sunlight. With tetraphenyl phosphonium benzenetellurolate in chloroform the salt $[(C_6H_5)_4P]_5^+[Hg_3(TeC_6H_5)_{11}]^{5-}$ was formed [72].

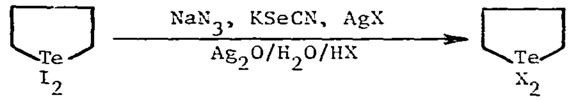
X. Heterocyclic Tellurium Compounds

Telluracyclopentane, [3,4-c]-benzo-2,5-dihydrotellurophene, tellurophene, benzotellurophene, five-membered heterocycles with Te-chalcogen groups in the ring, telluracyclohexane, 1-oxa-4-telluracyclohexane, 1-thia-4-telluracyclohexadiene, 10-tellura-9,10-dihydroanthracene, 1,3,5-tristanna-2,4,6-tritelluracyclohexane and phenoxtellurine (section XIII) were investigated.

A. Telluracyclopentane

1-Telluracyclopentane 1,1-dihalides, dipseudohalides and dicarboxylates were obtained when the diiodide was treated with NaN_3 or potassium selenocyanate in acetone, ground with Ag_2O/H_2O and the resulting product acidified with hydrofluoric or hydrochloric acid, or reacted in acetone at room temperature with freshly prepared silver bromide, silver cyanide, silver cyanate, silver thiocyanate or silver carboxylates (eqn. 40). The telluracyclopentane derivatives are monomeric in freezing

(40)



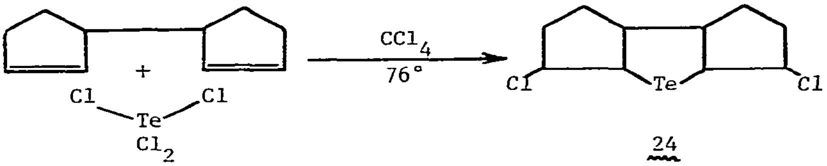
X, mp. °C, yield %: F, 72°, 82%; Cl, 113°, 80%; Br, 128-31°, 80%; N_3, 155° (dec), 100%; -NC, 180-2° (dec) 63%; -NCO, 108° (dec), 80%; -NCS, 87°, 40%; -NCSe, 110° (dec), 40%; CH_3COO, 81°, 60%; CCl_3COO, 130°, 84%; C_6H_5COO, 146°, 60%.

benzene. Upon heating they decompose smoothly at ~220° to telluracyclopentane, 1,4-dihalobutane and TeX_2 [76].

B. [2,3-b;4,5-d]-Dicyclopentano-1-telluracylcopentane

The reaction of tellurium tetrachloride with 2,2'-bicyclopentene produced [2,3-b;4,5-d]-dicyclopentano-1-telluracyclopentane 1,1-dichloride 24 (eqn. 41), which was characterized by elemental analyses and C-13 and proton nmr spectroscopy [36].

(41)

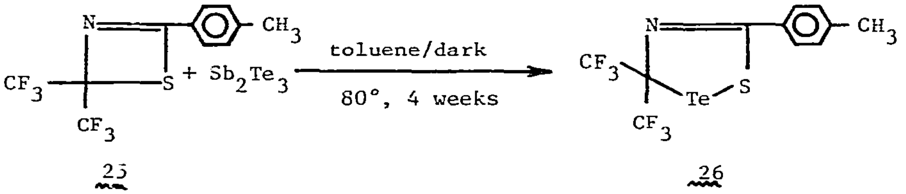


24

C. 1-Aza-3-tellura-4-thiacyclopent-5-ene

Antimony telluride and 1-aza-3-thiacyclobut-4-ene 25 combined to give 1-aza-3-tellura-4-thiacyclopent-5-ene 26 (eqn. 42). The violet solutions of this brown-red substance are extremely light sensitive. Compound 26 reacts with phosphites and multiple-bond systems with elimination of tellurium and can thus serve as a reagent equivalent to $(CF_3)_2C=N-C(S)Ar$ [46].

(42)



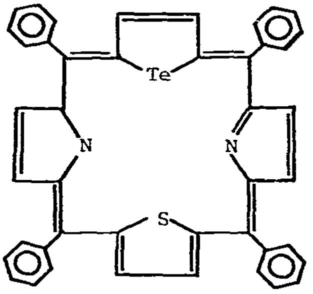
25

26

mp. 72-3°; 15% yield

D. Tellurophene

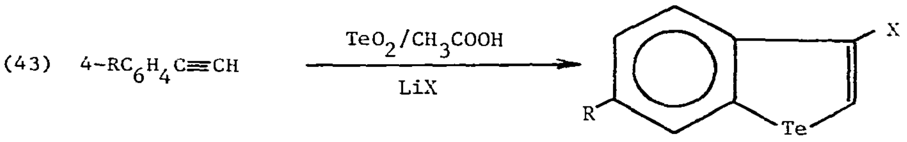
The tellurium-containing tetraphenylporphyrin 27 and related compounds were studied by proton nmr spectroscopy to investigate the inner and outer aromaticity employing protonation and complex formation to disrupt bonding interactions within the core of the molecule. The tellurium compound 27 decomposed immediately upon contact with CF_3COOD [77].



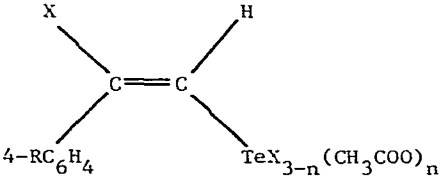
27

E. [4,5-d]-Benzotellurophene

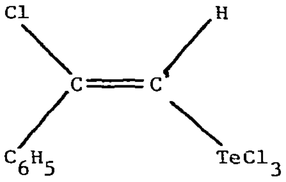
A one-step synthesis for 3-halobenzotellurophenes was developed [45] employing tellurium dioxide as the tellurium source (eqn. 43). In these reactions tellurium halide acetates, $TeX_{4-n}(CH_3COO)_n$ add to the triple bond. The resulting intermediate 28 cyclizes to a benzotellurophene derivative followed by a reductive elimination of acetate and halide. In



R, X: H, Cl; CH_3, Cl;
Br, Cl; H, Br;
H, I.



28



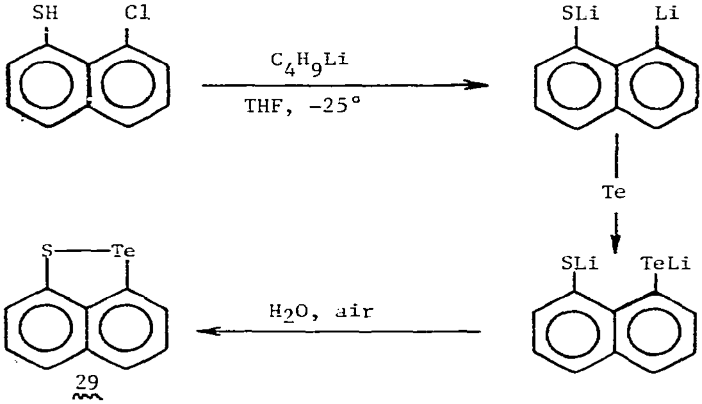
28a

line with this postulated mechanism compound 28a refluxed with $CH_3COOH/LiCl$ yielded 3-chlorobenzotellurophene. When LiCl was omitted only bis(2-phenyl-2-chlorovinyl) telluride was isolated [45].

F. 1-Chalcogena-2-telluraacenapthenes

An article in Ann. N.Y. Acad. Sci. [23] summarizes the synthesis of 1-chalcogena-2-telluraacenaphthene, discusses the formation of complexes with iodine and TCNQ and reports on the conductivity, magnetic susceptibility and vibrational spectroscopy. Much of this material was published earlier [5]. The previously reported synthesis employing 1,8-dibromonaphthalene as a starting material gave only a yield of two percent. The reaction sequence described in eqn. 44 provided the telluraacenaphthene 29 in 60 percent yield [23].

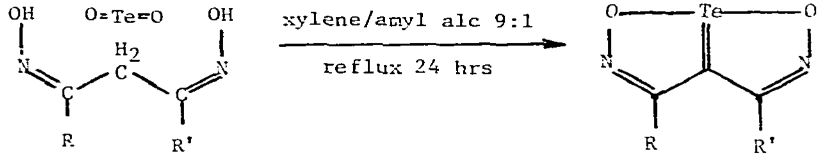
(44)



G. 2,5-Diaza-3,4-dioxa-3a-tellura-3,4-dihydropentalene and 2,3,4,5-Tetraaza-3a-tellura-3,4-dihydropentalene

A number of 2,5-diaza-3,4-dioxa-3a-tellura-3,4-dihydropentalenes 30 were synthesized [38] from tellurium dioxide and dioximes of 1,3-dioxo compounds (eqn. 45) according to a procedure published previously [3].

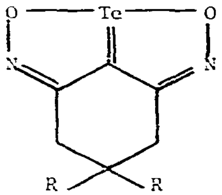
(45)



30

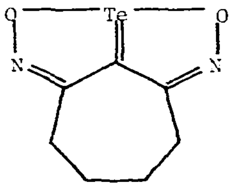
R, R', mp.°C, yield %: H, H, 200°, 25%;
CH_3, CH_3, 145°, 20%; CH_3, $C_2H_5OC(O)$, 99°, 5%;
H, C_6H_5, 128°, 5%; H, 4-$CH_3C_6H_4$, 188°, 5%;
H, 4-ClC_6H_4, 221°, 4%.

The pentalenes shown below were prepared similarly [38]. Compound 31 (R = CH_3) was also obtained (10 percent yield) from tellurium tetrachloride and the dioxime in chloroform solution. Reactions of compound 30 (R = R' = H) with halogens, nitric acid/sulfuric acid mixtures, dimethylformamide (Vilsmeyer reaction) or Friedel-Crafts reagents failed to replace the hydrogen atoms. Treatment of 31 (R = CH_3) with hydrazine or phenylhydrazine produced the 1,3-dioxime 2-hydrazone of 1,2,3-trioxo-5,5-dimethylcyclohexane. Hydroxylamine and compound 31 (R = H) in ethanol afforded the trioxime, whereas alcoholic potassium hydroxide or ethanol/sodium ethoxide yielded the 1,3-dioxime of 1,2,3-trioxocyclohexane [38].

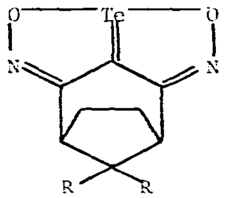


31

R = H: mp. 167°
30% yield
R = CH_3: mp. 167°
30% yield

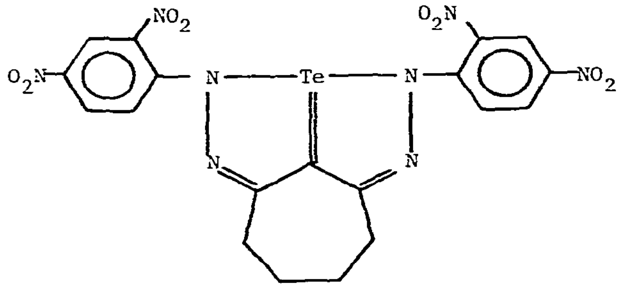


mp. 185°
45% yield



R = H: mp. 186°
60% yield
R = CH_3: mp. 125°
50% yield

2,3,4,5-Tetraaza-3a-tellura-3,4-dihydropentalene 32 (mp. 251°) was formed in two percent yield from tellurium dioxide and the bis(2,4-dinitrophenylhydrazone) of 1,3-dioxocycloheptane [44].

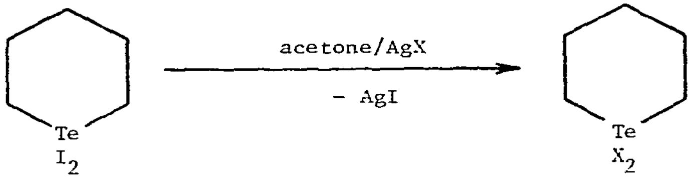


32

H. Telluracyclohexane

Several 1-telluracyclohexane 1,1-dihalides, dipseudohalides and dicarboxylates were prepared from the diiodide and silver halides, pseudohalides or carboxylates (eqn. 46). These compounds are non-electrolytes in acetonitrile solution [78].

(46)



X, mp. °C, yield %: F,* 210°(dec) 85%; Cl, 105°, 80%; Br, 106°, 80%;
CN, 180°(dec), 65%; N_3, 153°, 95%;
NCO, 180°(dec), 85%; NCS, 100°, 45%;
NCSe, 122°(dec), 40%; CH_3COO,** 52°, 65%;
CCl_3COO,** 178°, 82%; $C_6H_5CH_2COO$,** 135°, 70%;
$CH_3(CH_2)_{12}COO$,** 50°(dec), 50%;
C_6H_5COO,** 150°, 74%.

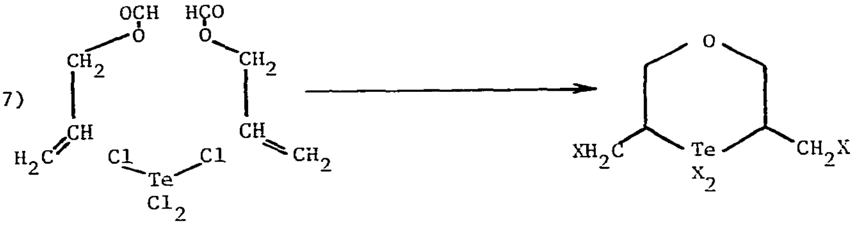
*from the diiodide and excess Ag_2O/H_2O; filtrate acidified with HF.
**potassium carboxylates can be used instead of the silver salts.

According to ir data, the cyanate, thiocyanate, and selenocyanate groups are bonded to the tellurium atoms via the nitrogen atoms, whereas C-Te bonding has been suggested for the cyanide groups [78].

I. 1-Oxa-4-telluracyclohexane

A Russian patent [P-14] claimed that 3,5-dihalomethyl-1-oxa-4-telluracyclohexane 4,4-dihalides were formed when tellurium tetrahalides and allyl formate (CH_2=$CHCH_2O_2CH$) were refluxed in diethyl ether (eqn. 47).

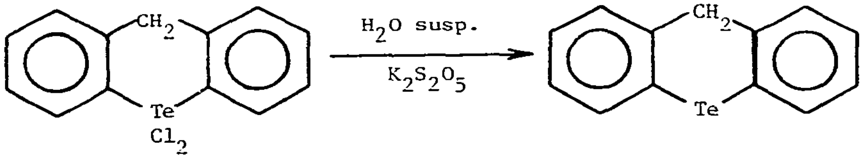
(47)



J. 10-Tellura-9,10-dihydroanthracene

Cyclization of 2-benzylphenyl tellurium trichloride in 1,2-dichlorobenzene at 50-70° in the presence of aluminum chloride produced 9-tellura-9,10-dihydroanthracene 9,9-dichloride 33 in 80 percent yield. The reduction of the dichloride by an aqueous solution of $K_2S_2O_5$ formed the parent heterocycle 34 quantitatively [22] (eqn. 48).

(48)

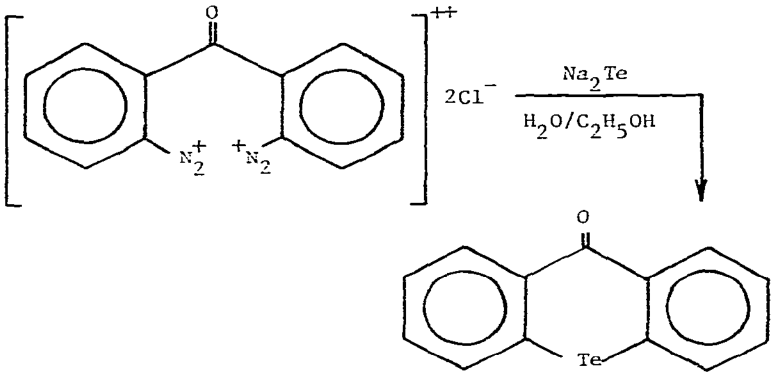


33 mp. 250-70°(dec)

34 mp. 151°

10-Oxo-9-tellura-9,10-dihydroanthracene was prepared [30] according to eqn. 49.

(49)



mp. 202°; 2% yield

K. 1,3,5-Tristanna-2,4,6-tritelluracyclohexane

1,1,3,3,5,5-Hexamethyl-1,3,5-tristanna-2,4,6-tritelluracyclohexane was previously prepared from tellurium and dimethylstannane [6], but could not be completely separated from the Sn_3Te_2 heterocycle which was formed as a by-product. The reaction of sodium hydrogen telluride, obtained from tellurium and sodium borohydride in aqueous medium, with dimethyldichlorostannane gave the yellow Sn_3Te_3 compound in 62 percent yield. The compound was stable in the dark for several days under an inert atmosphere [31].

XI. Physicochemical Investigations of Organic Tellurium Compounds

Infrared, Raman, ultraviolet-visible. proton, carbon-13, fluorine-19, tellurium-125, and rhodium-103 nuclear magnetic resonance, electron, Moessbauer, and mass spectroscopy, X-ray structure analysis and dipole moment measurements were employed to characterize organic tellurium compounds.

A. Infrared and Raman Spectroscopy

The infrared (ir) and Raman (R) spectral data for organic tellurium compounds reported during the survey period are summarized in this section. Frequencies (cm^{-1}) are listed only for modes involving the tellurium atom.

$[(C_6H_5)_3C]^+Te_3Cl_{13}^-$ [43]: ir(Nujol) 33-400; modes of Te_3Cl_{13} identified.

$C_6H_5TeCl\cdot$ethylenethiourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(STeCl) 254w(ir), 257s(R); ν_s(STeCl) 210m(ir), 208s(R).

$C_6H_5TeBr\cdot$ethylenethiourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(STeBr) 222w(ir), 228s(R); ν_s(STeBr) 193m(ir), 193s(R).

$C_6H_5TeI\cdot$ethylenethiourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(STeI) 265m(ir), 255w(R); ν_s(STeI) 172s(ir), 170vs(R).

$C_6H_5TeBr\cdot$ethyleneselenourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(SeTeBr) 190m(ir), 192w(R); ν_s(SeTeBr) 170s(ir), 169m (R).

$C_6H_5TeI\cdot$ethyleneselenourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(SeTeI) 180m(ir), 178w(R); ν_s(SeTeI) 150s(ir), 152vs(R).

$C_6H_5TeCl\cdot$thiourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(STeCl) 252m(ir), 257m(R); ν_s(STeCl) 244m(ir), 243m(R).

$C_6H_5TeBr\cdot$thiourea [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(STeBr) 242s(ir), 242vs(R); ν_s(STeBr) 164w(ir), 164w(R).

$2\text{-}O_2NC_6H_4TeBr$ [50]: ir(KBr) bands at 1590, 1490, 1325.

$2\text{-}C_6H_5N{=}NC_6H_4TeCl$ [37]: ir(Nujol, polyethylene) 200-4000; ν(TeN) 300m; ν(TeCl) 247s.

$[C_6H_5TeBrCl]^-[(C_6H_5)_4As]^+$ [51]: ir(polyethylene) 30-220; R-laser(solid); ν_{as}(BrTeCl) 188s(ir); ν_s(BrTeCl) 148m(ir), 150vs(R); δ(BrTeCl) 92vw(ir), 105m(R).

$[C_6H_5TeBr_2]^-[(C_6H_5)_4As]^+$ [51]: ir(polyethylene) 30-220; R-laser(solid); ν_{as}(BrTeBr) 138s(ir); ν_s(BrTeBr) 151w(ir), 150vs(R); δ(BrTeBr) 89m(ir), 92vw(R).

$[C_6H_5TeIBr]^-[(C_6H_5)_4As]^+$ [51]: ir(polyethylene) 30-220; R-laser(solid); ν_{as}(ITeBr) 144s(ir), 146w(R); ν_s(ITeBr) 121s(ir), 120m(R); δ(ITeBr) 70w(ir).

$[4\text{-}C_2H_5OC_6H_4TeIBr]^-[(C_6H_5)_3PCH_3]^+$ [51]: ir(polyethylene) 30-220; R-laser(solid); ν_{as}(ITeBr) 128m(ir), 126w(R); ν_s(ITeBr) 114vs(ir), 120m(R); δ(ITeBr) 65m(ir).

$[C_6H_5TeI_2]^-[(C_6H_5)_4As]^+$ [51]: ir(polyethylene) 30-220; R-laser(solid); ν_{as}(ITeI) 127m(ir), 120s(R); ν_s(ITeI) 110vs(R); δ(ITeI) 61m(ir).

$[4\text{-}C_2H_5OC_6H_4TeI_2]^-[(C_6H_5)_3PCH_3]^+$ [51]: ir(polyethylene 30-220; R-laser(solid); ν_{as}(ITeI) 114vs(ir), 119m(R); ν_s(ITeI) 103m(ir), 100m(R); δ(ITeI) 63m(ir).

$[C_6H_5Te(NCS)_2]^-[(CH_3)_4N]^+$ [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(STeS) 247w(ir), 257w(R); ν_s(STeS) 219vw(ir), 216vs(R).

$[C_6H_5Te(NCSe)_2]^-[(CH_3)_4N]^+$ [79]: ir(polyethylene) 50-500; R-laser(solid); ν_{as}(SeTeSe) 189w(ir), 188w(R); ν_s(SeTeSe) 138vw(ir), 136vs(R).

$2\text{-}O_2NC_6H_4TeBr_3$ [50]: ir(KBr); bands listed 850-1580.

aminoaryltellurium trihalides (see section V-D, eqn. 6) [41]: ir; far ir bands listed and ν(TeX-terminal), ν(TeX-bridging) and ν(TeC) assigned.

$2\text{-}C_6H_5N{=}NC_6H_4TeCl_3$ [37]: ir(Nujol, polyethylene) 200-4000; ν(TeCl) 353m; ν(TeCl) + ν(TeN) 307s.

$2\text{-}C_6H_5N{=}NC_6H_4TeBr_3$]37]: ir(Nujol, polyethylene) 200-4000; ν(TeN) 295; ν(TeBr) and azobenzene modes 260s.

$2\text{-}C_6H_5N{=}NC_6H_4TeClBr_2$ [3]: ir(Nujol, polyethylene) 200-4000; ν(TeCl) and ν(TeN) 303s; ν(TeBr) 205s; ν(TeBr) and azobenzene modes 254-264s.

2-R-cyclooct-5-en-1-yl tellurium trichloride (R = Cl, C_2H_5O, $HOCH_2CH_2O$) [35]: ir(KBr) bands 600-4000 listed.

$(C_2F_5)_2Te_2$ [47]: ir; vibrational assignments.

$(2\text{-}H_2NC_6H_4)_2Te_2$ [37]: ir(Nujol, polyethylene) 200-4000; ν(NH).

$(2\text{-}O_2NC_6H_4)_2Te_2$ [50]: ir(KBr); bands listed 850-1580.

$(C_2F_5)_2Te$ [47]: ir, vibrational assignments.

$C_6H_5TeCH_2CH(OCH_3)_2$ [53]: ir(thin film); bands listed 705-3060.

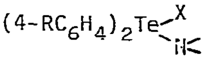
$CH_3(CH_2)_nTe(CH_2)_7COOCH_3$ (n = 5, 7) [25,26]: ir(neat); ν(CO).

5-(β-methyltelluroethyl)hydantoin [24]: ir(KBr); ν(CO).

$2\text{-}O_2NC_6H_4TeCH_2C_6H_5$ [50]: ir(KBr); bands listed 850-1580.

$(2\text{-}O_2NC_6H_4)_2Te$ [50]: ir(KBr); bands listed 715-1500.

$2\text{-}C_6H_5N{=}NC_6H_4TeC_6H_4\text{-}4\text{-}OC_2H_5$ [37]: ir(Nujol, polyethylene); γCH.


$(4\text{-}RC_6H_4)_2Te\begin{smallmatrix}\nearrow X\\ \searrow N<\end{smallmatrix}$ (see section IX-C) [63]: ir; ν(CO); ν(N=N).

$(4\text{-}RC_6H_4)_2Te{=}NR$ (see section IX-C) [64]: ir(vaseline oil); bands listed 600-4000.

$M_3(CO)_9H_2Te$; $M_3(CO)_9Te_2$ [80]: ir; ν(CO).

$PdX_2\{Te[(CH_2)_nSi(CH_3)_3]_2\}_2$ (X = Cl, NCS; n = 1, 3) [32]: ir(polystyrene); ν(PdCl); ν(CN), ν(CS), δ(SCN).

$(2\text{-}H_2NC_6H_4)_2Te_2\cdot CuCl_n$ (n = 1, 2) [37]: ir(Nujol, polyethylene); ν(NH).

$[(C_6H_5)_4P]^+[Hg(TeC_6H_5)_3]^-$ [72]: ir(polyethylene); ν_s(HgTe) 120.

telluracyclopentane difluoride [76]: ir, 400-4000, ν(TeF) 480.

telluracyclopentane dichloride [76]: ir, 400-4000, ν(TeCl) 296.

telluracyclopentane dicyanide [76]: ir, 400-4000, ν(CN).

telluracyclopentane $(RCOO)_2$ (R = CH_3, CCl_3, C_6H_5) [76]: ir, 400-4000; ν(TeO) 330.

telluracyclopentane X_2 (X = F, Cl, Br, N_3, CN, NCS, NCSe, CH_3COO, CCl_3COO, C_6H_5COO) [76]: ir, 400-4000; ν(TeC) 535-555.

2,2-bis(trifluoromethyl)-5-(*p*-tolyl)-1-aza-3-tellura-4-thiacyclopent-5-ene [46]: ir(KBr); 1607, 1594.

1,2-ditelluraacenaphthene [23]: R, 100-300; ν(TeC) 197.

1,2-ditelluraacenaphthene$\cdot I_x$ [23]: R, 100-300; ν(TeC) 204.

1-M-2-telluraacenaphthene (M = S, Se) [23]: R; bands listed 129-534.

telluracyclohexane difluoride [78]: ir; ν(TeF) 488.

telluracyclohexane dicarboxylates (see section X-H) [78]: ir, ν(COO), δ(COO).

telluracyclohexane dipseudohalides (NC, OCN, SCN, SeCN, N_3) [78]: ir; ν(pseudohalides).

telluracyclohexane X_2 (see section X-H) [78]: ir; ν(TeC) 520-550.

9-tellura-10-oxo-9,10-dihydroanthracene [30]: ir($CHCl_3$); 1720, 1645, 1595.

B. Ultraviolet-Visible Spectroscopy

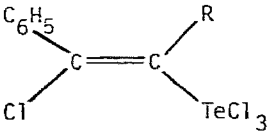
Ultraviolet-visible spectral data were reported for 2-$O_2NC_6H_4TeBr_3$ [50], $(C_6H_5)_2Te_2$ [74], (2-$O_2NC_6H_4)_2Te_2$ [50], $CH_3(CH_2)_nTe(CH_2)_7COOR$ (n = 5, 7; R = H, CH_3) [25,26], $(C_6H_5)_2TeS$ [74], 5-(β-methyltelluroethyl)hydantoin [24], telluromethionine [24], phenyl 2,2-dimethoxyethyl telluride [53], 2-nitrophenyl benzyl telluride [50], 4-R-phenyl phenylacetylenyl telluride-tetracyanoethylene [69] and $[(CH_3)_2Te\cdot Ru(NH_3)_5]^{n+}$ (n = 2, 3) [65].

C. Nuclear Magnetic Resonance Spectroscopy

Nuclear magnetic resonance spectroscopy employing the nuclei ^1H, ^{13}C, ^{19}F, ^{125}Te and ^{103}Rh was used to characterize organic tellurium compounds.

1. ^1H-NMR Spectroscopy

Proton-nmr data were reported for the following compounds:
4-$R_2NC_6H_4TeX_3$ (R = H, CH_3; X = Cl, Br, I) [41].



(R = H, CH_3) [42].

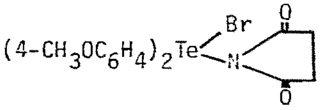
2-(β-hydroxyethoxy)cyclooct-5-en-1-yl tellurium trichloride [35].

$(C_{16}H_{13})_2Te_2$ [26a]; $(2\text{-}H_2NC_6H_4)_2Te_2$ [37]; $(2\text{-}C_6H_5CH_2C_6H_4)_2Te_2$ [22].

$CH_3(CH_2)_nTe(CH_2)_7COOR$ (n = 5, 7; R = H, CH_3) [25,26];
$CH_3TeCH_2CH_2CH(NH_2)COOH$ [24]; 2-(β-methyltelluroethyl)hydantoin [24];
$2\text{-}O_2NC_6H_4TeCH_2C_6H_5$ [50]; $C_6H_5TeCH_2CH(OCH_3)_2$ [53];
$3,4\text{-}(CH_3O)_2C_6H_3TeC_6H_5$ [39]; $[4\text{-}(CH_3)_2NC_6H_4]_2Te$ [58].
$(C_6H_5Te_2$, $(4\text{-}CH_3C_6H_4)_2Te_2$ [81]: conformation in solution; coupling constants.

$(4\text{-}CH_3C_6H_4)_2TeX_2$ (X = Cl, Br, I) [41]; $4\text{-}CH_3C_6H_4(C_6H_5)TeCl_2$ [39];
$4\text{-}C_2H_5OC_6H_4(C_6H_5)TeCl_2$ [39]; $3\text{-}CH_3\text{-}4\text{-}CH_3OC_6H_3(C_6H_5)TeCl_2$ [39];
$4\text{-}C_2H_5OC_6H_4(CH_3)TeCl_2$ [39].
$C_6H_5(CH_3)TeF_2$, $(CH_3)_2TeF_2$ [62]: coupling constants (FTeCH).

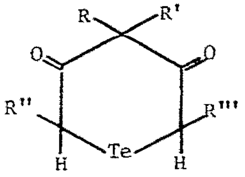
$(CH_3)_3SiTeSiH_3$ [73]; $(CH_3)_3GeTeGeH_3$ [73].



[63].

mer-$\{RhXYZ[Te(CH_3)_2]_3\}$ (X, Y, Z = Cl, Br, I) [66]: Te-H coupling constants.
$Pd(SCN)_2\{Te[(CH_2)_nSi(CH_3)_3]_2\}_2$ (n = 1, 3) [32]: $Eu(fod)_3\text{-}d_{27}$.

telluracyclopentane $(RCOO)_2$ (R = CH_3, C_6H_5) [76].
2,2-bis(trifluoromethyl)-5-(*p*-tolyl)-1-aza-3-tellura-4-thia-cyclopent-5-ene [46].
tellurium-containing tetraphenylporphyrin (see section X-D) [77].
1,2-ditelluraacenaphthene [23].
2,5-diaza-3,4-dioxa-3a-tellura-3,4-dihydropentalenes **30** (R = R' = H, CH_3) and **31** (R = CH_3) (see section X-G) [38].

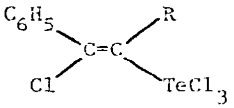


R, R', R'', R''': H, H, H, H; CH_3, CH_3, H, H; C_2H_5, H, H, H; CH_3, H, CH_3, H; H, H, CH_3, CH_3; CH_3, H, CH_3, CH_3 [82]: coupling constants HCH, Te-125CH, Te-125CCH

telluracyclohexane $(RCOO)_2^-$ (R = CH_3, $C_{13}H_{27}$, $C_6H_5CH_2$, C_6H_5) [78].
9-tellura-9,10-dihydroanthracene [22].
10-oxo-9-tellura-9,10-dihydroanthracene [30].

2. ^{13}C-NMR Spectroscopy

^{13}C-Nmr data were reported for the following compounds:



(R = H, C_6H_5) [42].

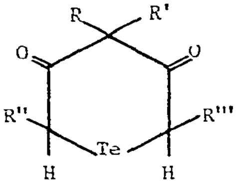
2-chlorocyclooct-5-en-1-yl tellurium trichloride [35].

$(C_6H_5)_2Te$ [8].

$(CH_3)_2TeF_2$, $C_6H_5(CH_3)TeF_2$, $(C_6H_5)_2TeF_2$ [62]: F-Te-C coupling constants.

$Pd(SCN)_2\{Te[(CH_2)_nSi(CH_3)_3]_2\}_2$ (n = 1, 3) [32].

2,2-bis(trifluoromethyl)5-(p-tolyl)-1-aza-3-tellura-4-thiacyclopent-5-ene [46].
1,6-R_2-2,5-diaza-3,4-dioxa-3a-tellura-3,4-dihydropentalenes (R = H, CH_3) [38].



R, R', R'', R''': H, H, H, H; CH_3, CH_3, H, H; C_2H_5, H, H, H; CH_3, H, CH_3, H; H, H, CH_3, CH_3; CH_3, H, CH_3, CH_3; coupling constants TeC, $TeCCH_3$ [82].

3. ^{19}F-NMR Spectroscopy

^{19}F-Nmr data were reported for the following compounds:
$(CF_3)_2Te_2$ [21]: coupling constant TeF 93 Hz.
$(CF_3)_2Te$ [21]: coupling constant TeF 22 Hz.
$3\text{-}FC_6H_4TeC_3F_7$, $4\text{-}FC_6H_4TeC_3F_7$ [55].
$(CH_3)_2TeF_2$, $(C_6H_5(CH_3)TeF_2$ [62]: coupling constants FTeCH.
$(C_6H_5)_2TeF_2$ [62]: coupling constant FTe 540 Hz.
2,2-bis(trifluoromethyl)-5-(p-tolyl)-1-aza-3-tellura-4-thiacyclopent-5-ene [46].

4. ^{125}Te-NMR Spectroscopy

^{125}Te-Chemical shifts were reported for Te_4^{++}, Te_6^{4+}, $Te_2Se_n^{++}$ (n = 2, 4), $TeSe_3^{++}$, $Te_3Se_n^{++}$ (n = 1, 3) [83], $Te_nSe_{4-n}^{++}$ (n = 1-4) [84] (coupling constants ^{125}Te-^{123}Te, ^{125}Te-^{77}Se); $(TeX_2)_n$ (X = Cl, Br, I; n = 1, 2), $TeCl_4$, $TeBr_4$, $Te(OH)_6$, $Te(OH)_5F$ [61].
$(CH_3)_2Te$ [61]: coupling constants TeC, TeCH.
$(C_2H_5)_2Te$ [61]: coupling constants TeC, TeCC, TeCH, TeCCH.
2,4,6-$(CH_3)_3C_6H_2Te$ [61]: coupling constants TeC, TeCC, TeCCC.
$(C_6H_5)_2TeBr_2$ [61].
mer-$\{RhX_3 \cdot [Te(CH_3)_2]_3\}$ (X = Cl, Br, I) [66]: coupling constants ^{125}Te-^{103}Rh.

5. ^{103}Rh-NMR Spectroscopy

The ^{103}Rh chemical shifts of the compounds *mer*-$\{RhXYZ \cdot [Te(CH_3)_2]_3\}$ (X = Cl, Br, I; all combinations) were reported relative to Ξ(Rh) = 3.16 MHz [66].

D. Electron Spectroscopy

The photoelectron (HeI) spectral bands of 1-thia-4-telluracyclohexadiene were assigned by comparison with published spectra of similar compounds and with the help of semi-empirical calculations [85].

E. ^{125}Te-Moessbauer Spectroscopy

The isomer shift values for materials commonly used as sources and absorber standards for the ^{125}Te-Moessbauer transition were evaluated [86,87]. Moessbauer data for thiourea complexes of tellurium dihalides were reported [88]. The present status of ^{125}Te-Moessbauer studies of organic tellurium compounds was reviewed [12].

F. Mass Spectroscopy

Mass spectral data were reported for the following compounds: 2-$O_2NC_6H_4TeBr$ [50]; 2-R-cyclooct-5-en-1-yl trichloride (R = Cl, C_2H_5O) [35]. 2-$O_2NC_6H_4TeBr_3$ [50]; 2-$C_6H_5N{=}NC_6H_4TeCl_3$ [37]; (2-$O_2NC_6H_4)_2Te_2$ [50]; $(CH_2{=}CHCH{=}CH)_2Te$ [20]; $CH_3(CH_2)_nTe(CH_2)_7COOCH_3$ (n = 5, 7) [25,26]; 5-(β-methyltelluroethyl)hydantoin [24]; telluromethionine [24]; $C_6H_5TeCH_2CH(OR)_2$ (R = CH_3, CD_3, C_2H_5) [53]; 2-$O_2NC_6H_4TeCH_2C_6H_5$ [50]; (2-$O_2NC_6H_4)_2Te$ [50]; 1,6-R_2-2,5-diaza-3,4-dioxa-3a-tellura-3,4-dihydropentalene (R = H, CH_3) [38]; 2,2-bis(trifluoromethyl)-5-(*p*-tolyl)-1-aza-3-tellura-4-thia-cyclopent-5-ene [46]; 1-M-2-telluraacenaphthene (M = S, Se, Te) [23]; 10-oxo-9-tellura-9,10-dihydroanthracene [30].

G. X-Ray Structure Analysis

In the crystals of $[C_6H_5)_3]^+[Te_3Cl_{13}]^-$ the anion has approximately C_{3v} symmetry consisting of three edge-sharing octahedra with a central triply bridging chlorine atom [43]. The complex $[\text{bis(benzimidazolethione)}\cdot TeCl_4]_3\cdot\text{dioxane}$ has a two-fold axis of symmetry with the tellurium atom in an octahedral environment and *cis*-thione ligands [89], whereas $[\text{bis(benzothiazolethione)}\cdot TeCl_4]_2\cdot\text{dioxane}$ has a *trans* configuration [90].

The tellurenyl compounds $2\text{-RNHC(O)}C_6H_4TeX$ (R, X: H, Br; CH_3, Cl) have *s-cis*-conformation with Te-O distances of 2.237 and 2.250 Å, respectively [91]. 2-Phenylazophenyl tellurium chloride [37] forms triclinic crystals. The coordination of the tellurium atom can be considered to be distorted trigonal-bipyramidal when the two lone electron pairs are included. The tellurium atom is bonded to a nitrogen atom (Te-N 2.19-2.23 Å) forming a five-membered ring.

In the compounds $C_6H_5TeCl\cdot[Y{=}C(NH_2)_2]_2$ (Y = S, Se) the tellurium atom is three-coordinated with bonds to one carbon and two chalcogen atoms. The tellurium-chalcogen bond lengths are 2.69 Å (Y = S) and 2.80 Å (Y = Se) [92].

In 2-ethoxycyclooct-5-en-1-yl tellurium trichloride the bonding about tellurium can be considered to be square pyramidal with the oxygen atom of the ethoxy group and the three chlorine atoms at the base and the carbon atom at the apex [35].

Bis(4-methylphenyl) ditelluride forms orthorhombic crystals. The molecule is characterized by a dihedral angle of 85.7° and Te-C and Te-Te bond lengths of 2.13 and 2.697 Å, respectively [95].

In 1-phenyl 1-telluroniacyclohexane bromide the telluracyclohexane ring is in the chair conformation with the phenyl group occupying an axial position. The Te-C distances are 2.14 Å ($Te\text{-}CH_2$) and 2.13 Å (Te-phenyl) [94].

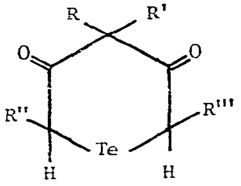
The trimethyl telluronium cation in $[(CH_3)_3Te]^+[B(C_6H_5)_4]^-$ is pyramidal with a Te-C distance of 2.14 Å and CTeC angles of 92° [71].

Bis(triphenylphosphoranylidene)ammonium tellurocyanate, $[(C_6H_5)_3P{=}N{=}P(C_6H_5)_3]^+TeCN^-$, has been shown to contain monomeric, essentially linear TeCN anions (TeCN angle 175°) with Te-C and C-N bond lengths of 2.02 Å and 1.07 Å, respectively [95].

The complex $Pd(SCN)_2\{Te[(CH_2)_3Si(CH_3)_3]_2\}_2$ has a *trans*-configuration with a Pd-Te distance of 2.606 Å and Te-C bond lengths of 2.23 and 2.16 Å [32].

The light orange α-modification of [3,4-c]-benzotellurophene diiodide has all nine ring atoms in one plane with a distorted octahedral environment for tellurium. The intramolecular Te-C (2.139, 2.145 Å) and Te-I distances (2.900, 2.928 Å) have the expected values. Two intermolecular Te-I interactions have distances of 3.653 and 3.878 Å [96].

X-Ray structural investigations of methyl-substituted 3,5-dioxo-1-telluracyclohexanes 35-37 showed that these molecules adopt the chair conformation with the methyl groups in compounds 36 and 37 occupying equatorial positions. The CTeC bond angles range from 86.4° to 89.7°. The TeC bond lengths have values between 2.168 and 2.184 Å.



	35	36	37
R	CH_3	H	CH_3
R'	CH_3	H	H
R'''	H	CH_3	CH_3
Ref.	[97]	[98]	[99]

The six-membered heterocycle $[(CH_3)_2SnTe]_3$ has approximately C_2 symmetry and Te-Sn distances ranging from 2.717 Å to 2.768 Å. The SnTeSn and TeSnTe angles are approximately 96° and 112°, respectively [31].

H. Dipole Moment Measurements

The dipole moments of telluracyclopentane [76] and telluracyclohexane [78] dihalides (F, Cl, Br), dipseudohalides (N_3, NCO, NCS, NCSe) and dicarboxylates (CH_3COO, CCl_3COO, C_6H_5COO, $C_6H_5CH_2COO$, $C_{13}H_{27}$) were reported.

XII. Analytical Techniques

A variety of metal trace impurities in dimethyl telluride were determined by emission spectrography [100].

XIII. Biology of Organic Tellurium Compounds

The inhibition of the growth of *Cryptococcus albidus* by sodium tellurite and tellurate [101], the formation of an Fe-Te albumin complex from K_2TeO_3 and bovine serum albumin [102] and the interaction of sodium tellurate with selenium, arsenic and mercury in rats [103] were investigated.

The telluracarboxylic acids, $CH_3(CH_2)_nTe(CH_2)_7COOH$ (n = 5, 7) [25,26] were concentrated in the hearts and livers of rats and allowed the heart to be imaged. Similarly, 16-methyltelluro-9-hexadecenoic acid was evaluated as an agent for myocardial perfusion [29].

The tissue distribution of steroids with tellurium atoms in the alkyl chains (see section VI-A2) was studied in female rats. High concentrations of the steroids were found in the adrenal gland [54,67,68].

Telluracyclopentane dichloride, dibromide, diazide and dicarboxylates were found to be inactive towards bacteria and fungi [76]. The relations between the chemical structure and the antibacterial activities of some phenoxtellurine compounds were investigated [104].

XIV. Organic Tellurium Compounds as Imaging Agents

The following tellurium compounds were claimed to be components of photosensitive, heat developable or charge sensitive recording materials: $Te[S_2CN(C_2H_5)_2]_2$ [P-12]; tellurium di(butylxanthate) [P-8]; $Te(S_2OCOC_3H_7)_2$ [P-9]; $4\text{-}CH_3OC_6H_4COCH_2TeCl_3$ [P-3]; $[4\text{-}CH_3OC_6H_4]_2Te$ [P-7]; $(C_6H_5COCH_2)_2TeCl_2$ [P-20, P-10]; $RR'TeBr_2$ [P-6]; $RTe\text{-}MR'_3$ (M = Si, Ge, Sn, Pb) [P-1].

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NOTE: The following references were inadvertently omitted from the review covering the year 1976, which appeared in J. Organometal. Chem., 158 (1978) 235-266. This page of references (No. 54 through P-3) should be inserted as p. 266A.

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