

Ortho-tellurated derivatives of some arylamines and imines

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Abstract. A range of novel bi-, tri- and multidentate organotellurium ligands containing Te and N donor atoms and their derivatives have been synthesised. The synthetic strategy involves telluration of orthochelating, monoanionic substrates derived from the following arylamines: N,N-dimethylbenzylamine, (*S*)-(–)-N,N-dimethyl-1-phenethylamine, N,N-dimethylnaphthylamine, (N,N-di-methylamino-methyl)ferrocene, tricarbonyl (N,N-dimethylbenzenemethanamine)chromium and 2-(3-thienyl)-pyridine. In addition novel chiral hybrid Schiff base ligands have been prepared by the condensation reaction of *bis*(*o*-formylphenyl) telluride and *o*-butyltellurobenzaldehyde with (*R*)-(+)-1-phenethylamine and (1*R*, 2*S*)-(–)-norephedrine.

Keywords. Organotellurium; hybrid ligands; orthometallation; ferrocene; chiral Schiff bases.

1. Introduction

Organotellurium chemistry has attracted considerable current interest in the areas of organic synthesis and ligand chemistry. The use of organoselenium reagents in organic synthesis, in particular organoselenyl halides (RSeX) and organoselenolates (RSe[–]), is well documented (Liotta 1987). The stimulus for the upsurge of activity in the synthesis and structure of tellurium analogues is due to their potential applications as reagents in organic synthesis. Recently a number of stable monomeric aryltellurenyl halides stabilised by intramolecular coordination due to inbuilt donor groups such as carbonyl-, nitro-, azo-, azomethine- and substituted pyridines have been isolated and characterised (Sudha and Singh 1994). In these derivatives the donor atoms are generally *sp*² hybrid nitrogen or oxygen atoms.

The in-built donor groups not only stabilise the otherwise unstable organotellurenyl derivatives but also afford hybrid bi-, tri- and multidentate ligands containing both 'hard' (N or O) and 'soft' (Te) bases. Though examples of simple hybrid ligands have recently become available (Singh and Srivastava 1992; Hope and Levason 1993) very few optically active hybrid tellurium ligands have been reported. This paper describes synthesis, structure and reactions of a variety of novel hybrid chiral and achiral ligands containing tellurium and *sp*³/*sp*² nitrogen. In the past few years we have also been interested in the synthesis and structure of the related orthotellurated derivatives of the type RTeX, RTeX₃, RR'TeX₂ etc. (where R is an orthochelating monoanionic group and X = Cl, Br, I). A brief review of recent results is presented here.

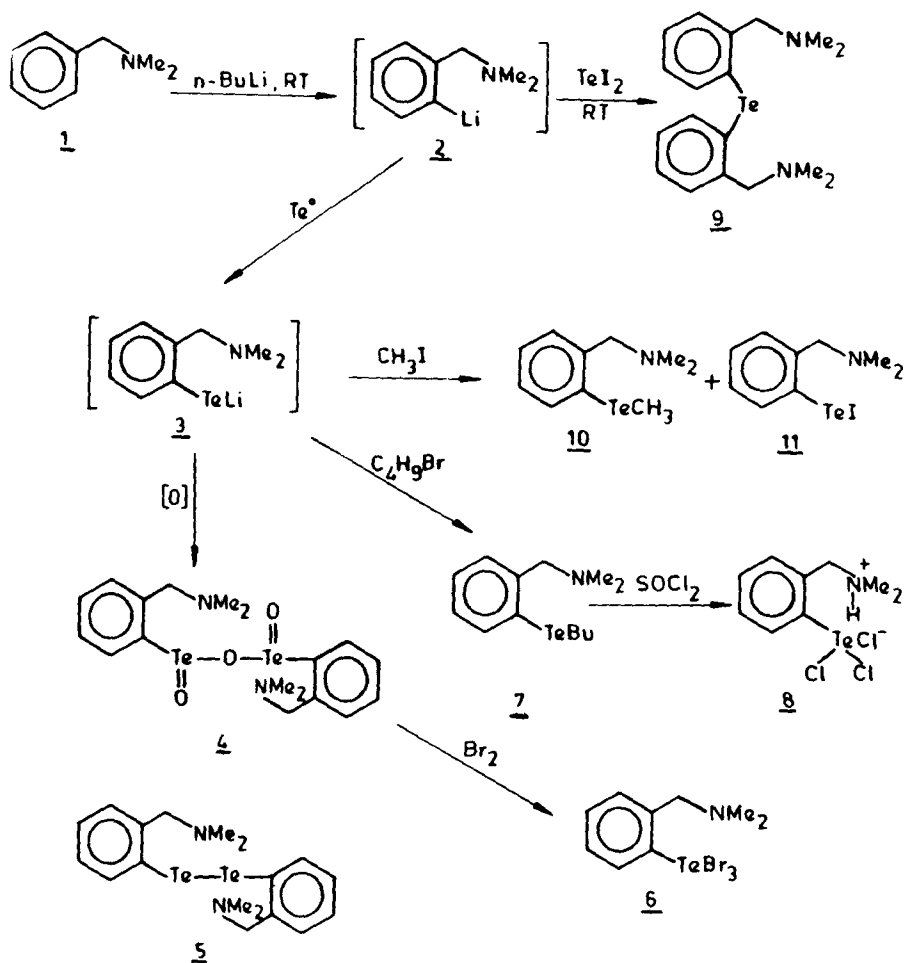
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2. Results and discussion

2.1 Orthotellurated derivatives of *N,N*-dimethylbenzylamine

Gysling and co-workers attempted the orthotelluration of dimethylbenzylamine (**1**) by the reaction of *o*-lithiodimethylbenzylamine (**2**) with tellurium tetrachloride in THF at -78°C (Gysling *et al* 1980). The work-up of the reaction led to decomposition to elemental tellurium. We have recently demonstrated that the reaction of *o*-lithio-*N,N*-dimethylbenzylamine with elemental tellurium affords the versatile 'synthon' (**3**) for the synthesis of intramolecularly coordinated tellurium derivatives (scheme 1) (Singh *et al* 1990). Initial attempts to isolate pure hybrid ligands proved unsuccessful and the ligands were characterised as halogenated derivatives.

The "ill-characterised" ditelluride (**4**) was brominated to yield the tellurium tribromide (**6**) which was characterised by single crystal X-ray diffraction study. The Te-N distance, [2.422(14) Å] is significantly longer than the sum of Pauling's single bond covalent radii (3.7 Å). If the Te...N interaction is considered to be significant, then the



Scheme 1.

environment around tellurium can be considered to be as pseudooctahedral. The bidentate ligand (**7**) was characterised by derivatising with SOCl_2 to give dichloride (**8**). The single crystal X-ray study revealed no interaction between the protonated nitrogen and the tellurium atom, as the nitrogen is twisted away from the central tellurium atom.

We have now found that by using either ethylacetate or ether as the solvent for elution during column chromatography of the crude products, the pure ligands (**5**, **9**, **10**) could be easily isolated in very good yields (Kaur *et al* 1994). The ligands and the related derivatives obtained as side products, have been isolated in the pure form and fully characterised by spectroscopic and X-ray crystallography techniques. For (**9**) the X-ray study showed the bond configuration around Te to be V-shaped. The structure is unsymmetrical and a more pronounced interaction of one nitrogen with tellurium is indicated. The two N's exist in the *cis*-conformation. In (**11**) the tellurium atom is T-shaped (3 coordinate) bonded to a carbon atom, an iodine atom and intramolecularly coordinated to N (N...Te distance is 2.366 Å). Weak intermolecular Te...I interactions are also observed.

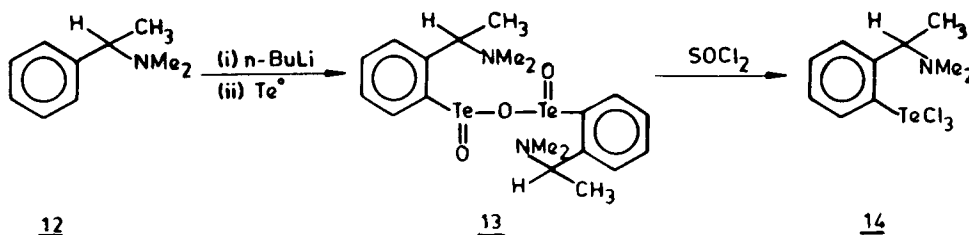
2.2 Chiral orthotellurated derivatives of [(*S*)-1-(dimethylamino)ethyl]benzene

The optically active orthotellurated compounds were readily obtained by the organolithium route (scheme 2). Intramolecular Te...N interaction in these compounds renders the NMe_2 diastereotopic, leading to anisochronous resonances. For the complex (**14**) even at as high as 140°C, the singlets due to NMe_2 do not coalesce, indicating a strong Te...N interaction. The structure of complex (**14**) has been determined by X-ray diffraction study. The co-ordination about tellurium is pseudooctahedral with a lone pair of electrons occupying the fourth equatorial site (Singh *et al* 1992).

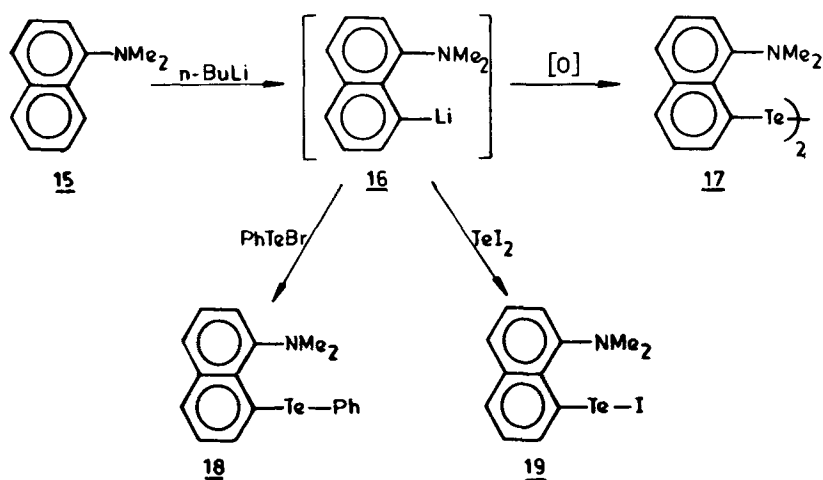
2.3 Orthotellurated derivatives of *N,N*-dimethylnaphthylamine

In the preceding two instances (§ 2.1 and 2.2), the five-membered ring formed by Te...N interaction was flexible. In order to determine whether flexibility is an important factor in deciding the structure of these molecules, we have synthesised a range of orthotellurated derivatives of rigid and flat 8-(dimethylamino)-1-naphthyl ligand (scheme 3) (Menon *et al* 1995a). Unexpectedly the *transmetallation* of (**16**) with TeI_2 afforded novel tellurenyl iodide (**19**) instead of the telluride. The identity of the new ligands and related derivatives was confirmed by mass, NMR (^1H , ^{13}C and ^{125}Te) and in the case of (**18**) by X-ray crystallography.

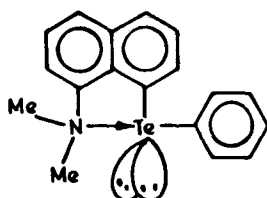
Only four structures of the type $\text{RR}'\text{Te}$ (Mcwhinnie 1992) have been structurally characterised and all show weak Te...N contacts in the range 2.69–2.77 Å. Molecule



Scheme 2.



Scheme 3.

Figure 1. Structure of 8-(dimethylamino)-1-phenyltelluronaphthalene (**18**).

18 exhibits a $\text{Te}\cdots\text{N}$ distance of 2.713 \AA . The electronic structure about Te atom can be depicted (figure 1) in terms of the VSEPR model (Barton *et al* 1993). The two lone pairs occupy the equatorial positions of a trigonal bipyramidal geometry.

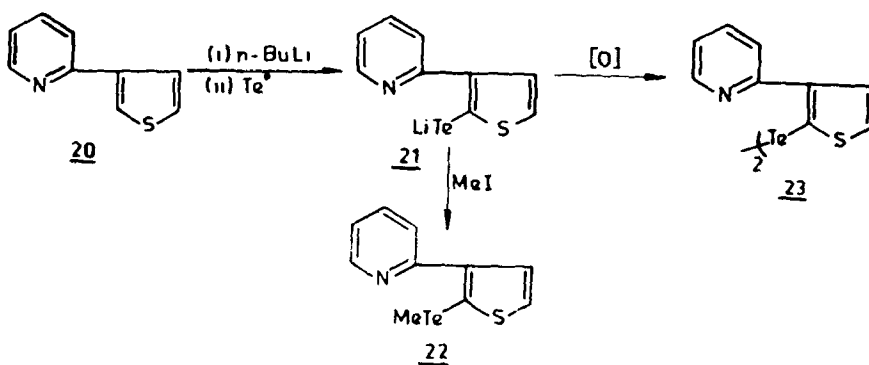
2.4 Cyclotellurated derivatives of 2-(3-thienyl)pyridine

Tellurated derivatives of substituted pyridine substrate, 2-(2-pyridyl)phenyl and 2-(quinolin-2-yl)phenyl have been reported (Al-Salim *et al* 1988). Isolation of novel tritelluride of type R_2Te_3 has generated considerable interest in the synthesis of such compounds.

Amongst the substituted pyridine substrates, 2-(3-thienyl)-pyridine is also known to give intramolecularly stabilised organometallic compounds. The substrate has been orthotellurated for the first time to afford the tridentate and tetradentate ligands (**22**), (**23**) (scheme 4) (Singh and Sudha 1992).

2.5 Orthotellurated imine derivatives

Renson and coworkers (Piette and Renson 1970) were the first to report orthotellurated compound (**24**) stabilised by orthocarbonyl functional group (aldehyde). More recently Minkin *et al* (1988) have reported the synthesis of *bis(o-formylphenyl)telluride* (**25**) by the treatment of *o*-lithiobenzaldehydeacetal with TeI_2 followed by the removal of the protecting group. Whereas a range of orthotellurated imine derivatives from these aldehydes have recently been reported, examples of tellurated imines bearing chiral



Scheme 4.

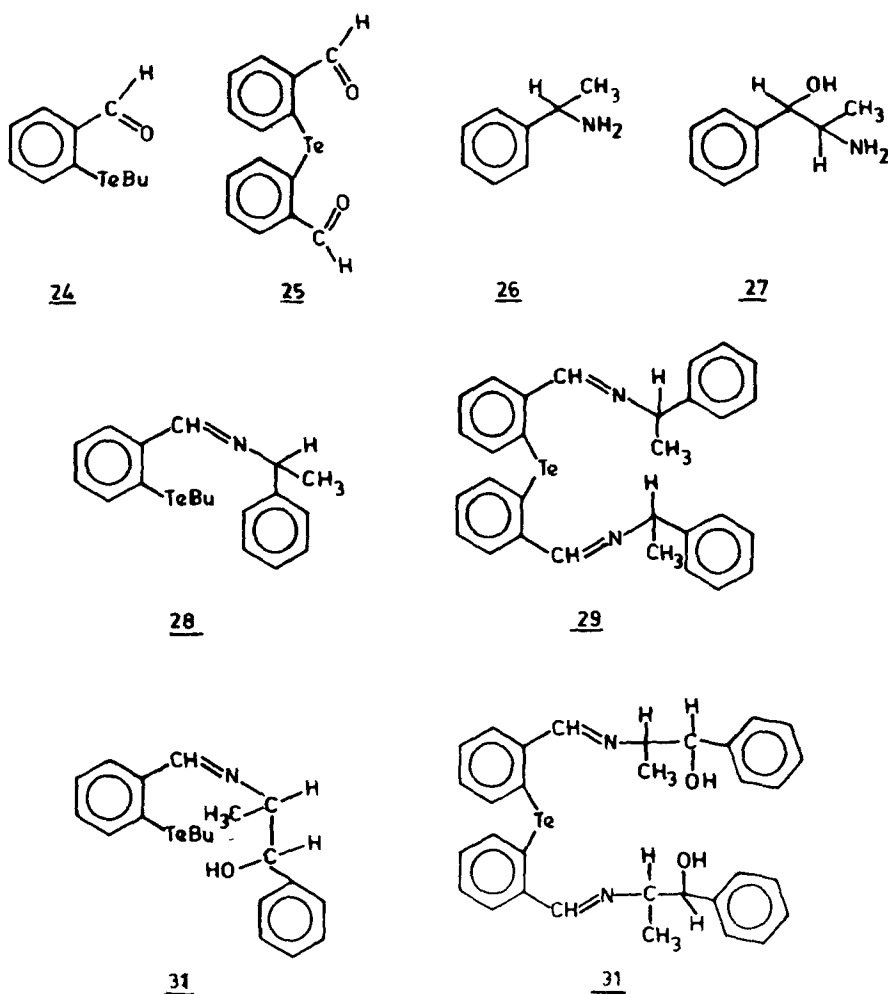


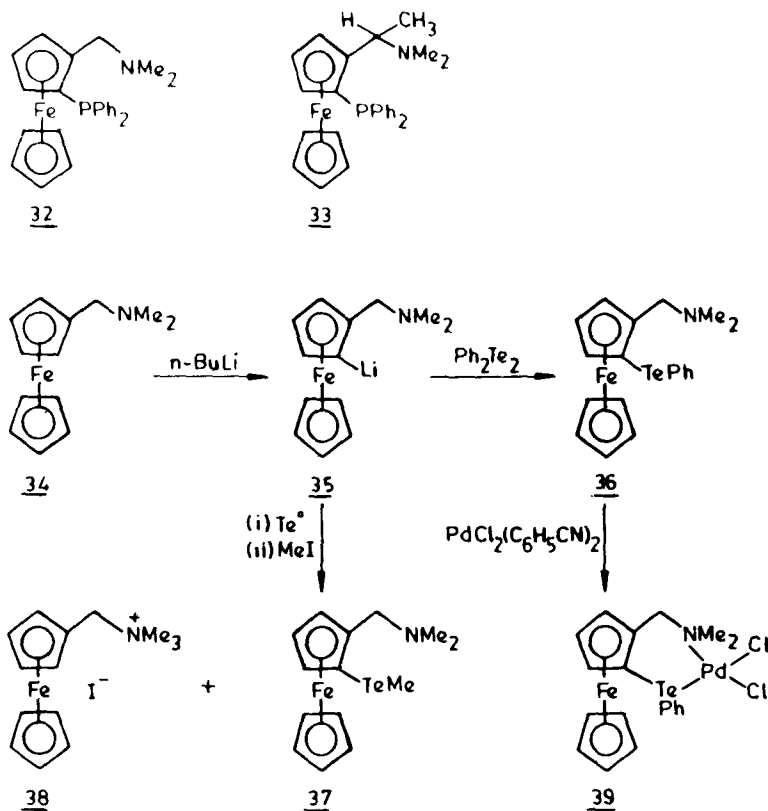
Figure 2. Structure of tellurium containing chiral Schiff bases.

substituents have not been described in the literature. We have now succeeded in synthesising chiral tellurium containing bidentate, tridentate and pentadentate Schiff bases (figure 2) (Menon *et al* 1995b).

The optically active tellurium azomethines are obtained by the condensation of (24) and (25) with (*R*)-(+)-1-phenylethylamine (26) and (*R,S*)-(–) norephedrine (27) respectively. All these compounds were characterised by elemental analyses, MS and detailed NMR studies. In ^1H NMR spectra the signals from methine protons ($-\text{CH}=\text{N}-$) were observed at $\approx \delta 8.5-8.7$ as sharp singlets, shifted 1.57–1.65 ppm upfield in comparison with the signals of the formyl protons ($-\text{CHO}$) in the precursor. The ligands (29) and (31) showed molecular ion peak at m/e 546 and m/e 604 respectively in the mass spectra while (28) and (30) showed the highest mass peaks (336) and (368) for ($M-\text{C}_4\text{H}_9$).

2.6 Orthotellurated derivatives of *N,N*-dimethylferrocenylamine

Optically and redox-active phosphine ligands, 1-[(dimethylamino)methyl]-2-(diphenylphosphino)ferrocene (*S*-FCPN, 32) and *N,N*-dimethyl-1-[(2-diphenylphosphino)ferrocenyl]ethylamine ((*S*)-(*R*) and (*R*)-(*S*)-PPFA, 33) are useful ligands in asymmetric Grignard cross coupling catalysed by nickel or palladium complexes (Blystone 1989). It seems that the ferrocene planar chirality plays a more important role than the carbon centre chirality on the side chain of the ferrocene. More recently the



Scheme 5.

S and Se analogues (Okoroafor *et al* 1988) of the phosphine ligands have been reported. The palladium complexes of the S ligand are good selective homogeneous and heterogeneous catalysts for the reduction of conjugated dienes to monoenes at room temperature. In this work, we report two examples of the tellurium analogues of these ligands. During the course of our work the synthesis of diferrocenyl dichalcogenides (Gornitzka *et al* 1992; Nishibayashi *et al* 1994) has been reported.

Ortholithiation of ferrocenylamine (**34**) was carried out following the method developed by Okoroafor *et al* (1988). Telluration was achieved either by treating the lithiated ferrocene (**35**) with diphenyl ditelluride or tellurium powder (scheme 5). Both the reactions were quite sluggish and gave only very poor yield of the tellurides (**36**) and (**37**). Interestingly the quenching of the intermediate with MeI afforded large quantities (85%) of the quaternary ammonium salt, $[\text{C}_5\text{H}_5\text{FeC}_5\text{H}_3\text{CH}_2\text{NMe}_3]^+\text{I}^-$ (**38**). This result is in sharp contrast to the reaction of *o*-LiTeC₆H₅CH₂NMe₂ with MeI (scheme 1). The mass spectrum of (**36**) shows molecular ion peak at *m/e* 449 as the highest mass in the spectrum.

The ¹H NMR data of the ligands are significantly different from that of the free amine. The aminomethylene protons (N-CH₂) in (**36**) become diastereotopic with a shift of 0.11 ppm between the protons leading to an anisochronous resonance even at room temperature.

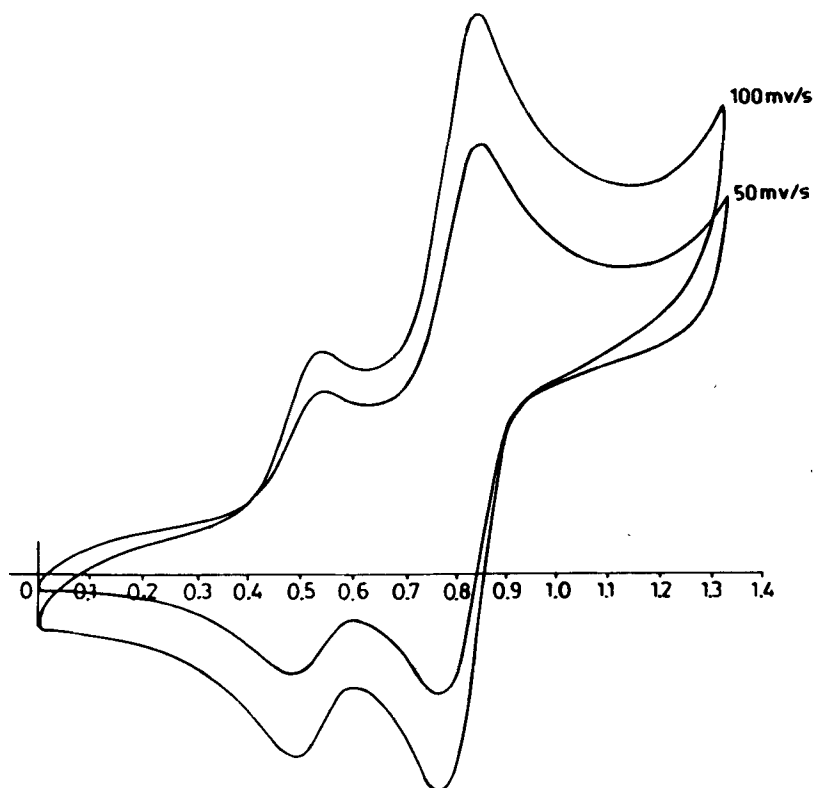
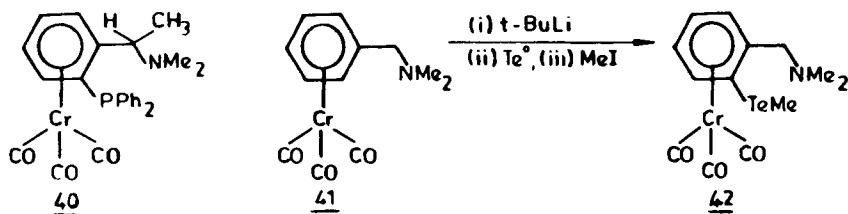


Figure 3. Cyclic voltammogram of (**36**) in dichloromethane at 50 mV/s and 100 mV/s.



Scheme 6.

Compound (36) is electrochemically well behaved. Well defined two reversible redox peaks ($^1E_{1/2} = 0.49$, $^2E_{1/2} = 0.8$ eV) (figure 3) were observed. This observation indicates that the substitution of the ferrocenylamine aids the stabilisation of the cation radical.

The palladium complex (39) was prepared by the reaction of ligand (36) with bis(benzonitrile)palladium dichloride in benzene. The NMe_2 protons which appear as a singlet in the ^1H NMR spectrum of the ligand give rise to a doublet in the complex (39). Furthermore, the methylene protons appear as a pair of widely separated doublets.

2.7 Orthotellurated (η^6 -arene)chromium derivatives

Tricarbonyl (η^6 -arene)chromium complexes, can exist in two enantiomeric forms when the phenyl ring has two different substituents at the ortho or meta positions. Recently Uemura *et al* (1992) have reported the synthesis of optically active (η^6 -arene)chromium phosphines (40) which were used as ligands for the palladium or nickel catalysed asymmetric cross coupling. The asymmetric cross-coupling of 1-phenylethylmagnesium or -zinc reagent with vinyl bromides in the presence of the catalysts gave the coupling products up to 61% ee. Here we wish to report the attempted synthesis of tellurium containing (η^6 -arene)chromium ligands.

The precursor amine complex (41) could easily be prepared in 80% yield following a reported method. Ortholithiation and telluration was attempted at -78°C in THF (scheme 6). The work-up gave a greenish-yellow material with poor solubility in common organic solvents. Although the elemental analysis was close to that of the desired product (42), poor solubility hindered the complete characterisation. The compound was found to be nonconducting. Alternatively the synthesis of 42 was attempted by the thermal reaction of $\text{Cr}(\text{CO})_6$ and 2- $\text{NMe}_2\text{CH}_2\text{C}_6\text{H}_4\text{TeMe}$. Refluxing led to decomposition and precipitation of tellurium.

3. Experimental

3.1 Synthesis of [1-(dimethylamino)methyl-2-(phenyltelluro)ferrocene (36)]

A 1.6 M solution of *n*-BuLi in hexane (2.90 ml, 475 mmol) was added over a half an hour period to a solution of [(dimethylamino)methyl]ferrocene (0.98 g, 0.8 mL, 4.03 mmol) in dry ether (100 ml) at -78°C under argon in a 250 ml round bottomed flask. The suspension was stirred for 24 h at room temperature and a solution of diphenylditelluride (1.92 g, 4.70 mmol) in 20 mL of ether was added dropwise over 30 minutes at -78°C . The reaction mixture was further stirred for 24 hours at room temperature and then refluxed for 8 hours.

After this, the mixture was slowly added to aqueous NaHCO_3 by cooling in an ice bath. The resulting organic layer and the extracts from the aqueous layer were combined, washed with ice water, dried over sodium sulphate and concentrated in vacuo to afford a brown oil which contained some diphenylditelluride and unreacted amine in addition to the desired product. The crude product was dissolved in minimum amount of dichloromethane and adsorbed on silica gel. The adsorbed material was loaded on top of a silica gel column and eluted with petroleum ether (60–80°) alone to isolate diphenyl ditelluride. Further elution with a mixture of petroleum ether/dichloromethane mixture (60:40) afforded the desired product (33). The unreacted amine moved very slowly even when the elution was carried out with CH_2Cl_2 only. The new product is found to be a reddish brown oil, yield 20%.

UV-Vis(λ_{max}): 231, 258, 431 nm

IR (neat): 710, 750, 840, 850, 1040, 1120, 1400, 1580, 2850, 2950, 3080 cm^{-1} .

MS, (*m/e*, relative intensity, %): 449(18.43, M^+), 405(100), 339(12), 275(31), 242(18), 199(28).

$^1\text{H NMR}$ (CDCl_3): δ 2.0(s, NMe_2), 3.45(*dd*, NCH_2), 4.1(s, C_5H_5), 4.9(*t*, C_5H_3), 7.6–6.8(*m*, aromatic C-H).

$^{13}\text{C NMR}$ (CDCl_3): δ 44.6(NMe_2), 59.3(NCH_2), 69.8(C_5H_5), 89.1(C1), 79.5(C2), 71.1(C3, C4, C5), 137.0, 128.9, 127.0, 117.3 (aromatic-C).

3.2 Synthesis of 1-[(dimethylamino)methyl]-2-(methyltelluro)ferrocene (37)

n-BuLi in hexane (7.5 ml, 12 mmol) was added over a half an hour period to a solution of [(dimethylamino)methyl]ferrocene (2.188 g, 1, 78 ml, 9 mmol) in dry ether (50 ml) under argon in a 100 ml three-necked round bottom flask at -78°C . After the suspension was stirred for 20 hours at room temperature, the powder (1.148 g, 9 mmol) was added under a brisk flow of argon at -78°C . The reaction mixture was warmed to room temperature over 3–4 hours and then addition of CH_3I (0.56 ml, 9 mmol) was carried out at 0°C . After additional stirring for 4 hours, the reaction mixture was poured into a solution of aqueous NaHCO_3 by cooling in an ice bath and extracted with ether. Most of the compound remained in the aqueous layer and hence a second extraction with chloroform was carried out. Both the ether and chloroform extracts were concentrated separately. The ether extract gave a pale yellow viscous oil which corresponded to the desired compound.

Yield 0.18 g, 5%.

$^1\text{H NMR}$ (CDCl_3 , 300 MHz); δ : 2.02(s, 6H, NMe_2), 2.40(s, 3H, Te-Me) 3.76(s, 2H, CH_2), 4.15(s, 5H, *cp* unsubstituted), 4.21–4.24(*m*, 3H, *cp* substituted).

3.3 Synthesis of 1-[(dimethylamino)methyl]-2-(phenyltelluro)ferrocene]-palladium dichloride(39)

The complex (39) was prepared by reacting a benzene solution of $(\text{PhCN})_2\text{PdCl}_2$ (0.019 g, 0.05 mmol) with (36) (0.26 g, 0.06 mmol). The reaction mixture was stirred for 2 hours and the precipitated complex washed with benzene followed by petroleum ether and recrystallized from dichloromethane-hexane mixture (2:1) to give a reddish-brown solid. Yield: 18% m.p.: $149\text{--}153^\circ\text{C}$ (*d*)

Analysis: Calculated C, 35.5, H, 2.9, N, 2.3%. Found C, 35.5, H, 2.7, N, 2.1%.

UV-Vis(λ_{max}): 236, 328.2, 406.8 nm.

$^1\text{H NMR}$ (CDCl_3): 2.05(*s*, NMe_2), 2.1(*s*, NMe_2), 3.41(*d*, NCH_2), 3.52(*d*, NCH_2), 4.1(*s*, C_5H_5), 4.25(*m*, C_3H_3), 4.6(*m*, C_3H_3), 7.7–7.76(*m*, Ph)

3.4 Characteristic data for [1-(dimethylamino)methyl]ferrocene (34)

UV-Vis(λ_{\max}): 237, 436 nm

IR (neat): 750, 820, 84, 925, 1005, 1020, 1040, 1110, 1130, 1170, 1240, 1260, 1350, 1380, 1410, 1460, 2780, 2800, 2840, 2940, 3090 cm^{-1} .

$^1\text{H NMR}$ (CDCl_3): δ 2.1(s, NMe_2), 3.2(s, NCH_2), 4.01(s, C_5H_5)

$^{13}\text{C NMR}$ (CDCl_3): δ 44.6(NMe_2), 59.0(NCH_2), 83.2(C1), 69.95(C2), 67.86(C3 , C4 , C5), 68.3(C_3H_5).

3.5 Synthesis of (2-Me₂NCH₂C₆H₅)Cr(CO)₃ (41)

In a 100 ml three-necked flask fitted with a gas inlet and a simple reflux condenser was added *n*-dibutylether (60 ml) and THF (5 ml). The solvents were deoxygenated thoroughly by the passage of a slow stream of N_2 for approximately 15 min, after which $\text{Cr}(\text{CO})_6$ (2.5 g, 11.4 mmol) and the amine (1.5 ml, 10.2 mmol) were added. The nitrogen stream was then stopped and the mixture heated at reflux for 30 hours. A bubbler was placed on top of the condenser to prevent the entry of air. A yellow solution was obtained which was cooled and filtered under nitrogen over a pad of Celite, which was then washed with a little additional solvent. The solvents were distilled off on a rotary evaporator from a water bath held at 60°C. To the residual deep yellow oil was added pentane. Cooling for 20 hours produced yellow crystals of the desired compound. A small amount of $\text{Cr}(\text{CO})_6$ is recovered from the condenser, the remainder distilled off with the solvent. Recrystallisation from benzene/petroleum ether afforded yellow crystalline product.

Yield: 2.2 g, 80%, m.p.: 41°C.

$^1\text{H NMR}$ (60 MHz, CDCl_3): δ 2.3(s, 6H, NMe_2), 3.1(s, 2H, CH_2), 5.3(s, 5H, Aromatic H)

Analysis: Calculated for $\text{C}_{12}\text{H}_{13}\text{CrNO}_3$, C, 53.14, H, 4.79, N, 5.6% Found C, 52.86, H, 4.53, N, 4.99%. IR(KBr): 1950, 1890, 1880 cm^{-1} .

3.6 Attempted synthesis of 2-NMe₂CH₂C₆H₄TeMeCr(CO)₃ (42) Method A:

Synthesis of (42) was attempted by a method similar to that used for of (41). Refluxing 2-MeN₂NCH₂C₆H₄TeMe (0.11 g, 0.395 mmol) with an excess of $\text{Cr}(\text{CO})_6$ (0.10 g, 0.45 mmol) in *n*-dibutylether and THF resulted in decomposition of the tellurium ligand and precipitation of tellurium.

Method B: In a three-neck flask fitted with a N_2 inlet, a septum, and a stopper was added (41) (0.20 g, 0.74 mmol) dissolved in THF. The system was deoxygenated for 10 minutes, after which it was cooled to -78°C and *t*-BuLi was syringed into the reaction vessel (0.4625 ml, 0.74 mmol). An orange coloured solution was obtained to which was added tellurium powder (0.094 g, 0.74 mmol). After stirring for half an hour CH_3I (0.46 ml, 0.74 mmol) was syringed in dropwise. A greenish yellow precipitate was obtained which was washed with methanol over a small column of silica. Yellow precipitate was obtained after evaporation of the solvent.

m.p.: 205°C (d). Analysis calculated for $\text{C}_{13}\text{H}_{15}\text{NO}_3\text{CrTe}$ C, 37.8 H, 3.36 N, 3.39%. Found C, 36.78 H, 3.26 N, 2.67%.

3.7 Materials and methods

Reagents used were tellurium powder (Fluka), iodomethane (Fluka), *n*-BuLi (Fluka), palladium chloride (SRL), benzonitrile (EMERK), ferrocenylamine (Fluka), benzylamine (Fluka), $\text{Cr}(\text{CO})_6$ (Strem). Diphenyl ditelluride was prepared by a reported procedure.

Measurements of UV-visible spectra in solution (CH_2Cl_2) were performed on a Shimadzu UV-260 spectrophotometer. IR spectra in the range $4000\text{--}400\text{ cm}^{-1}$ were recorded on a Nicolet 170-5 \times FT infrared spectrometer. The solid samples were examined as KBr pellets. ^1H and ^{13}C NMR spectra were recorded on Varian V \times R 300S spectrometer. C, H and N analysis was carried out using Carbo-Erba 1106 analyser. Mass spectra were recorded on a Varian MAT 1125 mass spectrometer.

3.8 Electrochemistry

A bioanalytical system model CV-18 electrochemical analyzer was used in a standard three-electrode configuration (a platinum bead working electrode, a platinum wire counter electrode and Ag/AgCl reference electrode). The electrolyte was 0.1 M tetrabutylammonium perchlorate in dichloromethane and the compound concentration was 5×10^{-4} M in CH_2Cl_2 .

4. Conclusion and outlook

We have attempted to synthesise a range of hybrid bidentate, tridentate and multidentate tellurium ligands and synthetically important stable organotellurylhalides (RTeX). Of particular interest are the chiral ligands and tellurenyl halides. Our future studies will be directed towards application of these ligands for the preparation of stable monomeric precursors for the MOCVD of HgCdTe, chiral catalysts and their utility in homogeneous catalysis.

Acknowledgements

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