THALLIUM; ANNUAL SURVEY COVERING THE YEAR 1980[*]

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The number of papers dealing with organothallium compounds during the year 1980 remained almost the same as in the previous year. Reaction chemistry and synthetic application of both thallium(III) and thallium(I) compounds continued to draw more attention than preparative and structural chemistry. This survey will deal with 1) synthesis, structure and spectroscopy of organothallium(III) compounds, 2) thallium-metal bonded compounds, 3) reactions and kinetics, 4) organic synthesis with thallium(III) compounds and 5) organothallium(I) compounds.

1. Synthesis, Structures and Spectroscopy of Organothallium(III) Compounds

Matthews and co-workers have determined the structures of various diorganothallium derivatives (1-3). The dimeric structure of chlorine-bridged bis(trimethylsilylmethyl)thallium chloride **1**, previously proposed from molecular weight and IR data [Bull. Chem. Soc. Jpn., 51 (1978) 1397], was established crystallographically (1). The C-Tl-C angle observed (168°) is close to linearity, with the coordination geometry around thallium being approximated by a distorted trigonal bipyramid with one vacant equatorial site. Also determined were the structures of diphenylthallium chelate compounds **2** and **3** (2). **2** is monomeric with a four-coordinate thallium atom, and **3** dimeric with a five-coordinate thallium atom. The C-Tl-C angle in the former (148°) is considerably smaller than that in the latter (163°). The latter structure may be compared with that of dimethylthallium tropolonate which has an oxygen-bridged polymeric chain involving a six-coordinate thallium atom [see Acta Cryst., 31B (1975) 1929].

The structures of several bis(pentafluorophenyl)thallium complexes were proposed in the late sixties mainly on the basis of molecular weights and far IR spectral data. The structure of a related compound, $(2,3,5,6\text{-}C_6HF_4)_2TlBr$

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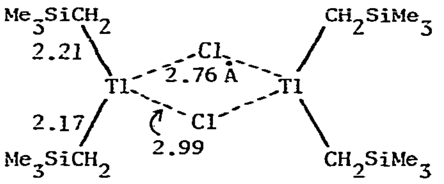
[*] Thallium; Annual Survey covering the year 1979 see J. Organometal. Chem., 203(1980) 313-325.



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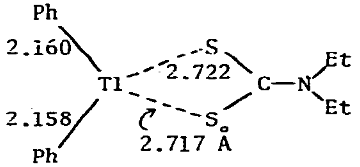

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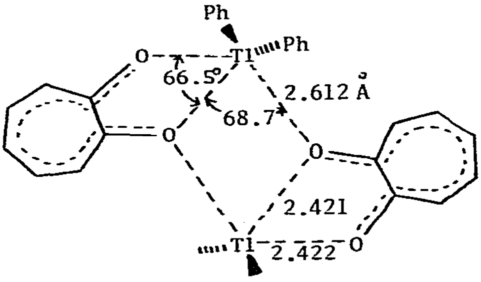

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was determined recently and the compound was shown to be polymeric [Inorg.Chim. Acta, 35 (1979) L335]. Now the X-ray crystal study of $(2,3,5,6\text{-}C_6HF_4)_2TlCl\cdot OPPh_3$ has established a dimeric structure with unsymmetrical chlorine bridging as shown in Fig. 1 (3). The stereochemistry about each thallium is that of a distorted trigonal bipyramid, with the two carbons and one chlorine occupying the equatorial positions. This structure is closely related to that of $(2,3,5,6\text{-}C_6HF_4)_2TlBr$ which possesses a similar distorted trigonal bipyramidal thallium atom,

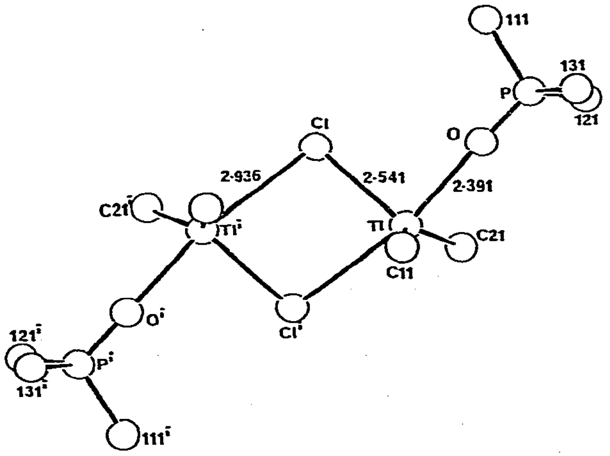
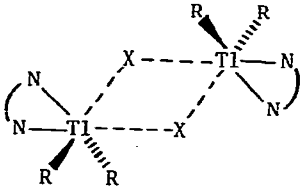


Fig. 1. The structure of $(2,3,5,6\text{-}C_6HF_4)_2TlCl\cdot OPPh_3$. Only the first carbon atom of each phenyl ring is shown. [Reproduced from J. Organometal. Chem., 193 (1980) 13].

These crystallographic results then prompted Deacon and Phillips to examine or reinvestigate solution properties and structures of a series of complexes, R_2TlX and $R_2TlX{\cdot}L$ (R= C_6F_5, 2,3,5,6-C_6HF_4, 2,3,4,5-C_6HF_4; X= Br, Cl; L= $OPPh_3$, bipy, Ph_3P) in more detail (4). In acetone solution R_2TlX were monomeric, and $R_2TlX{\cdot}L$ underwent partial dissociation by loss of L. There was also slight ionization into $[R_2Tl{\cdot}L]^+$ and $[R_2TlX_2]^-$ when L= bipy. In benzene solution R_2TlX were predominantly dimeric, and $R_2TlX{\cdot}L$ showed slight but significant association. On the basis of these results, as well as related X-ray structural results, possible structures of these complexes have been proposed, including polymeric halides and dimeric adducts such as one shown in Fig. 1 and 4.



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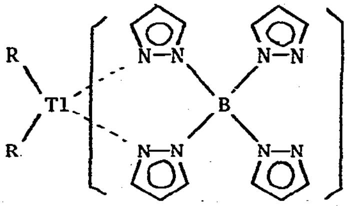
Some novel organothallate complexes containing the perfluoro- and perchlorophenyl groups were synthesized by the reaction of R_2TlX, TlX_3 and TlX_4^- with RLi (R= C_6F_5, C_6Cl_5) (5). Thus isolated as salts of bulky cations and characterized by conductivity and IR spectra were: $[(C_6F_5)_6Tl]^{3-}$, $[R_4Tl]^-$, $[(C_6F_5)_2(C_6Cl_5)_2Tl]^-$, $[R_3TlX]^-$ (X= Cl, Br, NCS), $[RTlCl_3]^-$, $[\{(C_6F_5)_3Tl\}_2Cl]^-$ and $[\{(C_6F_5)_2TlCl\}_2Cl]^-$. Complexes of dimethyl- and diphenylthallium cations with 1,2-dicyanoethylenedithiolate ion (mnt^{2-}) and 2,2',2''-triaminotriethylamine (tren), $[Me_2Tl]_2(mnt)$, $[Ph_2Tl(mnt)]^-$ and $[Me_2Tl(tren)]^+$, were prepared from the corresponding diorganothallium halides and the chelating ligands (6). On the other hand, similar treatment of $PhTlCl_2$ with Na_2mnt resulted in production of Ph_2TlCl. IR and ^1H NMR spectral data of the new complexes have been discussed in terms of different coordination abilities of sulfur and nitrogen atoms to thallium. In another study of complexes of diorganothallium cation, Onishi et al. synthesized dialkylthallium pyrazoylborates, 5 from the organothallium

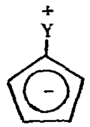


5 R= Et, n-Bu

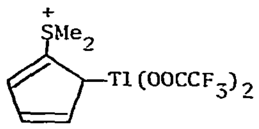
halides and sodium pyrazoylborate (7). They were monomeric in dichloromethane, and non-electrolytes in this solvent as well as in THF, acetone and ethanol. Variable temperature NMR spectra have been discussed, assuming essentially ionic character of the Tl-N bonds.

Variations in the ^{13}C and ^{1}H NMR spectral parameters, in particular the coupling constants involving thallium nucleus, in a series of $RTlX_2$ and R_2TlX (R= neopentyl, Me_3SiCH_2) have been interpreted in terms of the Fermi contact spin coupling mechanism and the nature of the atoms attached to the α-carbon (1).

Roberts examined reactions of phosphonium and sulfonium cyclopentadienylides 6 with $Tl(OOCCF_3)_3$ in CF_3COOH by ^{1}H NMR spectroscopy (8). Addition, rather than substitution, occurred to give 7 and 8. These compounds did not exhibit rapid fluxional behavior as was found in η^1-cyclopentadienylmetallic compounds. Similar reactions of these ylides with Hg(II) salts also have been described.



6 Y= SMe_2 , PPh_3



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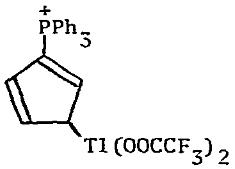

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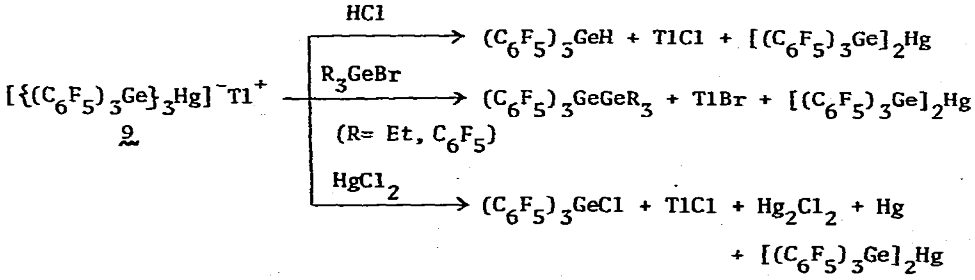
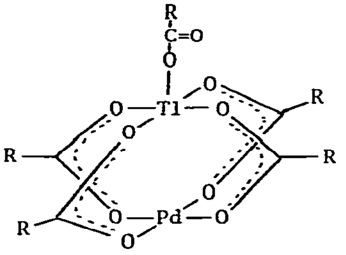
2. Thallium-Metal Bonded Compounds

Reaction of metallic thallium with $[(C_6F_5)_3Ge]_2Hg$ in 1,2-dimethoxyethane to give colorless crystals of the formula, $[(C_6F_5)_3Ge]_2Hg\cdot Tl[Ge(C_6F_5)_3]\cdot 1.5\,DME$ 9 was described by a Russian group (9). Some reactions of 9 (Scheme 1) and IR data suggested, when compared with those of known lanthanide metal analogues, an ionic structure for 9.

Scheme 1



Although no convincing evidence for the presence of Pd-Tl bonds was presented, a series of interesting clusters containing Tl and Pd atoms were prepared (10). Thus, reactions of $Tl(OOCR)_3$ with $Pd(OOCR')_2$ (R,R'= Me, Et, i-Pr, Ph) in toluene gave $PdTl(OOCR)_3(OOCR')_2$. On the basis of ^{1}H and ^{13}C NMR data, the authors suggested a possible structure as shown in 10, rather than 11. Variable temperature NMR aspects have been interpreted in terms of an apical ligand dissociation and exchange of bridging and apical carboxylate ligands.



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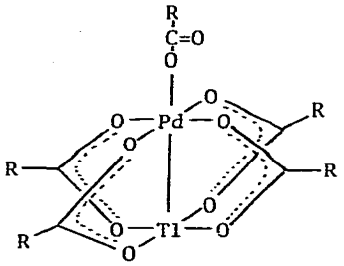

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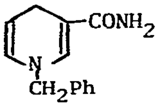
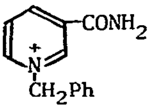
3. Reactions and Kinetics

Oxidative addition reactions of $(C_6F_5)_2TlBr$ with low valent metal complexes are well known. Spanish workers now have reported preparation and reactions with gold(I) complexes and $SnCl_2$ of several bis(polyfluorophenyl)thallium chlorides, R_2TlCl (R= 2,3,5,6-C_6HF_4, 2,3,4,6-C_6HF_4, 2,3,6-$C_6H_2F_3$, 4-C_6H_4F, 3-$C_6H_4CF_3$) (11). The gold(III) and tin(IV) compounds thus obtained were of type $R_2AuCl \cdot L$ [L= PPh_3, tetrahydrothiophen (tht)], $R_2(C_6F_5)Au(tht)$ and $[R_2SnCl_3]^-$.

Monoorganothallium compounds of the type, $RTlX_2$ (R= alkyl, p-tolyl; X= OAc, Cl) reacted with N-benzyl-1,4-dihydronicotinamide to give RH and thallium(I) compounds as shown in eq. 1 (12). High yield formation of some ether products from oxythallated adducts of styrene, isobutene and o-allylphenol through eq. 1 may be contrasted to predominant regeneration of the parent olefins in the reduction of these organothallium compounds with $NaBH_4$. Examination of the

$$RTlX_2 + BNAH \longrightarrow RH + TlX + [BNA]X \qquad (1)$$

BNAH=


BNA$^+$=


hydrogen source which replaced thallium in eq. 1 and spin trapping experiments using dioxygen and perdeuterionitrosodurene led the authors to suggest the following scheme involving one-electron transfer to $RTlX_2$ and subsequent Tl-C bond homolysis.

$$BNAH + RTlX_2 \longrightarrow [BNAH]^+ + RTlX + X^- \qquad (2)$$

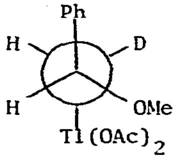
$$RTlX \longrightarrow R\cdot + TlX \qquad (3)$$

$$R\cdot + [BNAH]^+ \longrightarrow RH + [BNA]^+ \qquad (4)$$

A mechanistic study of copper(I) halide mediated halodethallation of alkylthallium compounds (eq. 5) was reported (13). Experiments using a spin trap (perdeuterionitrosodurene) and stereospecific organothallium reagents 12 and 13 indicated occurrence of two competing pathways as shown in Scheme 2. In this scheme an ionic path which predominates at lower temperatures proceeds through anchimeric assistance by the phenyl group to afford the substitution products with net retention of configuration, whereas a radical path which predominates at higher temperatures gives the products with epimerization. These two pathways also compete in the reaction of erythro-n-OcCH(OMe)CHDTl(OAc)$_2$ with CuCl/KCl, although the ionic path in this case results in inversion of configuration.

$$RTl(OAc)_2 + KX \xrightarrow{CuX} RX + TlOAc + KOAc \qquad (5)$$

$$X = Cl,\ Br$$



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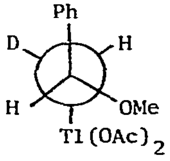

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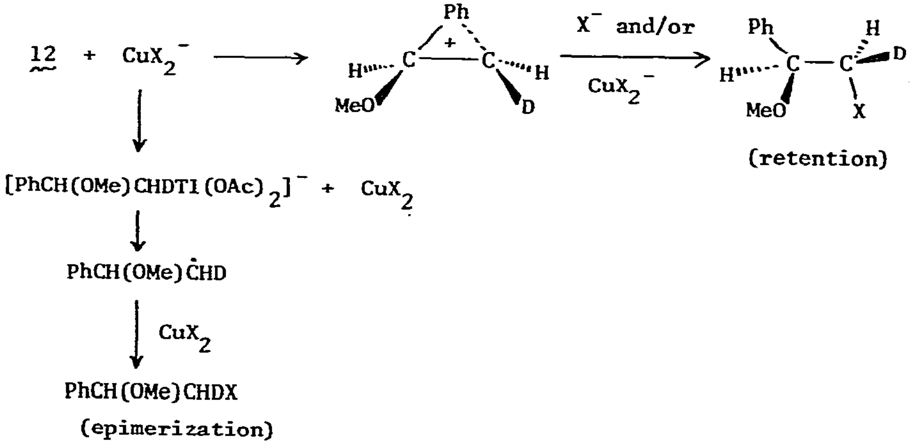
Scheme 2



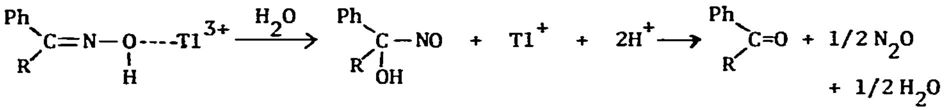
Phenyl and tolylthallium (o : m : p= 9 : 5 : 86) bis(trichloroacetate) were prepared via thallation from Tl_2O_3, trichloroacetic acid and benzene or toluene. These compounds underwent several known transformations of monoarylthallium compounds (14); typical examples include 1) substitution reaction to give ArX [X/reagent: I/KI, Cl/$CuCl_2$, CN/CuCN, SeCN/$Cu(SeCN)_2$, NO_2/$NaNO_2$],

2) coupling reaction to give Ar-Ar with $PdCl_2$, and 3) olefin substitution to give ArCH=CHR with $PdCl_2$ and CH_2=CHR (R= CN, Ph, COOEt).

Product selectivity (ketones, aldehydes, diols) in the oxidation of branched alkenes by $Tl_2(SO_4)_3$ was examined as a function of alkene structure, temperature and solvents (15). With increasing temperature the quantity of ketones decreased in the case of internal alkenes, but increased in the case of terminal alkenes (RR'C=CH_2). In the former case there was observed the isoselective temperature at which three different olefins exhibit the same selectivity. Use of the linear free energy analysis suggested that in the case of oxidation of terminal olefins carried out in H_2O, steric effects have an influence on the ketone/aldehyde ratio, but in methanol polar effects are decisive in determining this selectivity. Polar effects are also important in determining the carbonyl/diol ratio.

Kinetic studies of the thallation of benzene, toluene and t-butylbenzene with $Tl(OOCCF_3)_3$ were performed by the aid of ^{1}H NMR analysis, rather than conventional analysis via I^- quenching of arylthallium products [cf. J. Org. Chem., 44 (1979) 2309] (16). A primary isotope effect of 5.0 was found for toluene thallation, indicating rate-determining proton transfer. The inductive order of the reactivity (t-Bu$>$Me$\gg$H) was found to contrast to the hyperconjugative order in the case of mercuration reaction.

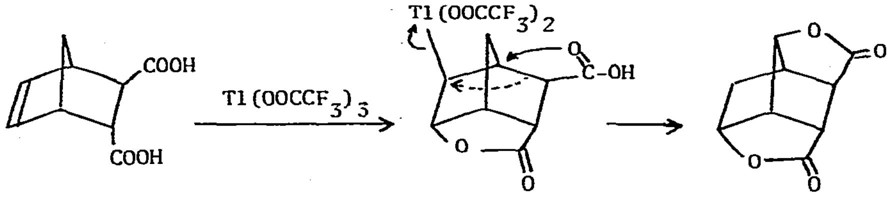
A kinetic study of oxidation of oximes of benzaldehyde and acetophenone by Tl(III) ion in water was reported (17). The rate equation obtained suggested a complex formation step between Tl(III) ion and the oxime. The oxime then was converted to the nitroso compound which eventually decomposed to the carbonyl product as shown in eq. 6.



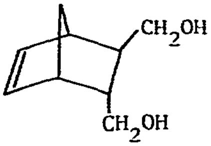
(6)

4. Organic Synthesis with Thallium(III) Compounds

Treatment of the dicarboxylic acid 14 with $Tl(OOCCF_3)_3$ in the presence of $BF_3 \cdot OEt_2$ in CH_2Cl_2/CF_3COOH gave a previously unknown lactone 16 as shown in eq. 7 (18). The reaction may proceed through an intermediate 15, the diacetoxy analogue of which has previously been isolated. Similarly, the reaction of the biscarbinol 17 gave the bisether 18.



14 15 16 (7)



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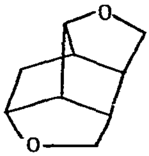

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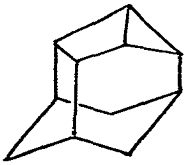
Oxidative cleavage of the cyclopropane ring in 3,5-dehydronoriceane 19 with $Hg(OAc)_2$, $Tl(OAc)_3$ and $Pb(OAc)_4$ was investigated by a Japanese group (19). In the first reaction in aqueous THF it was possible to isolate an organomercury compound 20 (M= HgCl) after treatment with aqueous NaCl. The main products from thallium and lead oxidation in acetic acid were 21∼23. Possible routes to 21 and 22 involving intermediates 20 [M= $Tl(OAc)_2$ and $Pb(OAc)_3$] and 24 have been suggested. The formation of 23 may involve the Wagner-Meerwein rearrangement at certain stages of the reaction, one possibility being shown in eq. 8.



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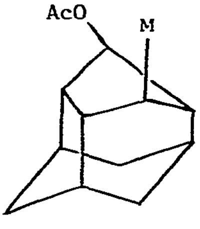

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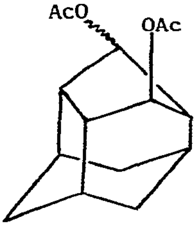
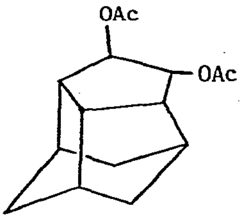


21 exo-exo
22 endo-exo



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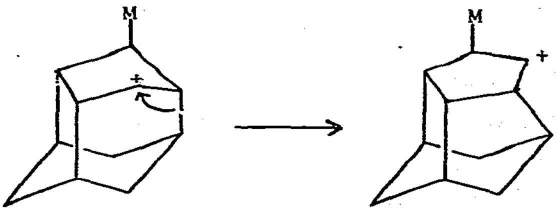
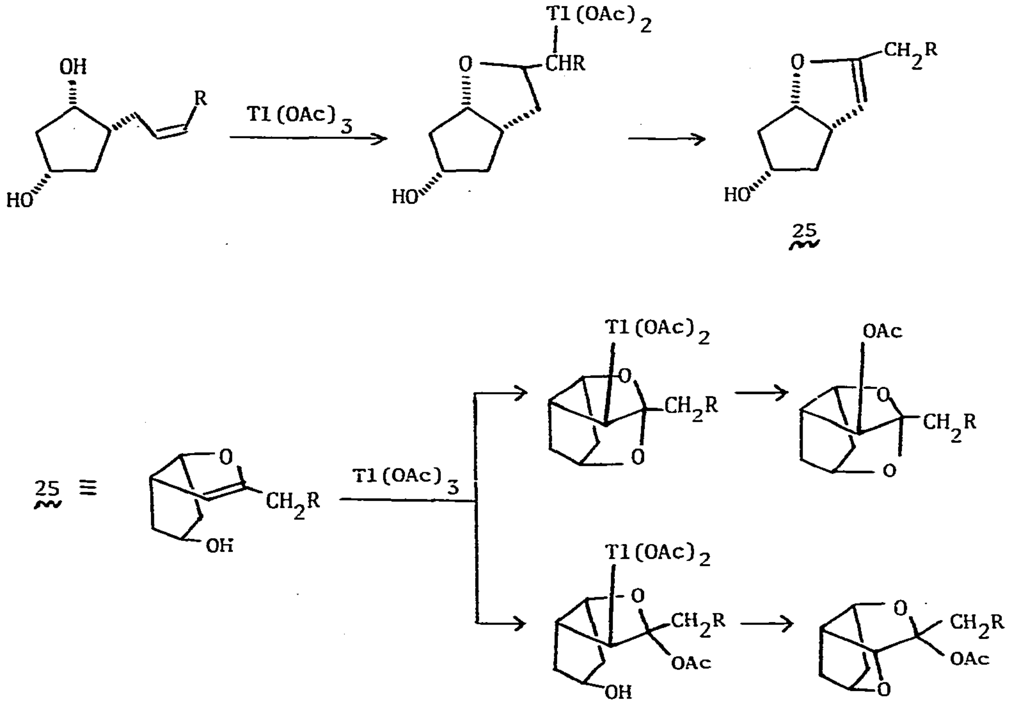


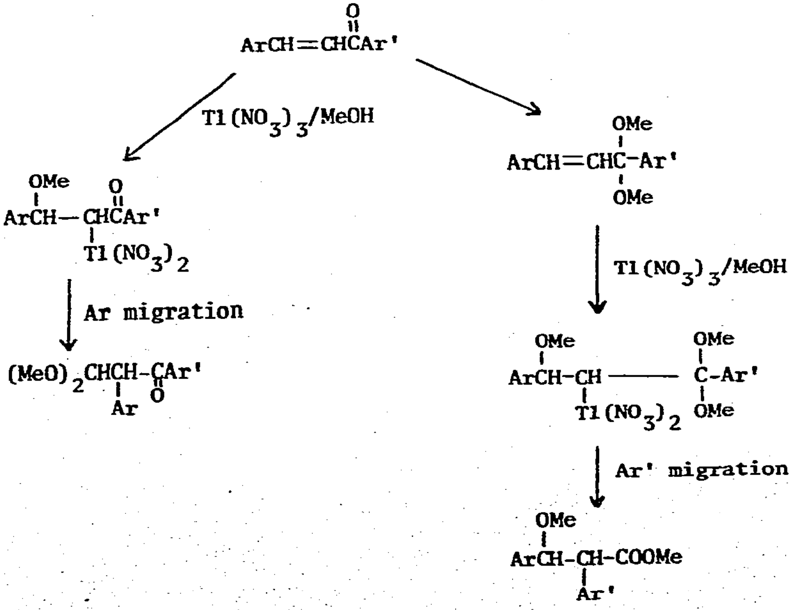
24 (8)

A study of the oxidative rearrangement of prostaglandin derivative $PGF_{2\alpha}$ methyl ester by $Tl(OAc)_3$ [J. Am. Chem. Soc., 100 (1978) 6756] was extended to other prostaglandins (20). A reaction scheme central in the present work is summarized in Scheme 3.

Scheme 3

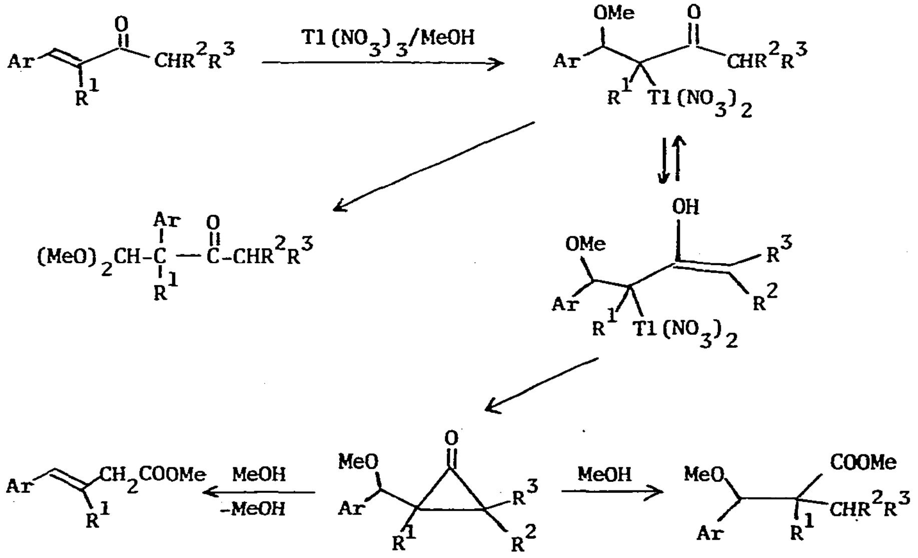


Scheme 4

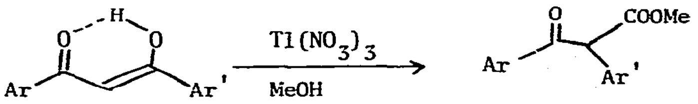


Taylor, McKillop and co-workers previously reported that reactions of chalcones (ArCH=CHCOAr') with $Tl(NO_3)_3$ give either keto acetals in methanol or the propanoate derivatives in trimethyl orthoformate (TMOF) [J. Am. Chem. Soc., 95 (1973) 3641; J. Org. Chem., 42 (1977) 4167] (Scheme 4). They now have examined the effect of substituents on Ar and Ar' rings on the ratio of these competing rearrangements involving Ar and Ar' migration in acidic methanol or TMOF (21). As expected, increasing the electron-donating ability of the para-substituent in Ar resulted in higher amounts of the keto acetal products. Related work involving oxidative rearrangement of arylideneacetone derivatives with $Tl(NO_3)_3$ in methanol was described by Antus and co-workers (22). A general scheme proposed to account for various products is shown in Scheme 5. The same authors also described another oxidative rearrangement involving 1,3-diaryl-1,3-propanedione derivatives with $Tl(NO_3)_3$ in methanol as shown in eq. 9 (23).

Scheme 5



The similar reaction with 1-(2-hydroxyphenyl)-3-aryl-1,3-propanediones gave oxidative cyclization products as shown in eq. 10.



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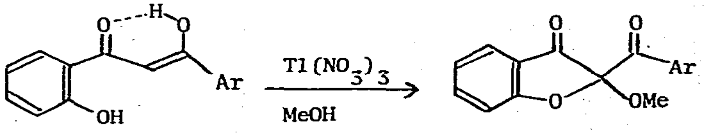
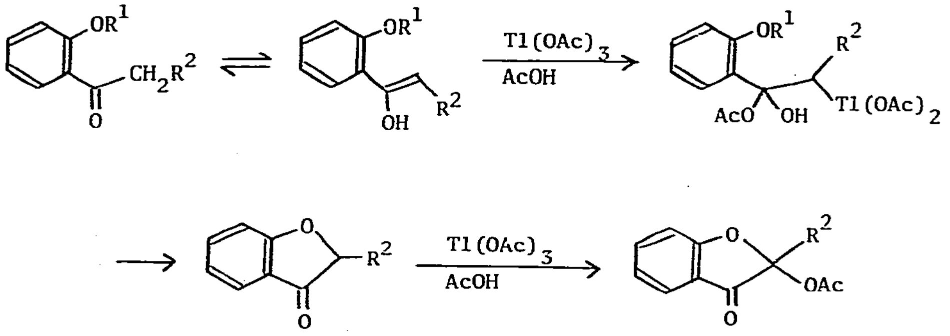


(10)

Oxidative cyclization of enolizable o-hydroxy and o-methoxyphenyl ketones with $Tl(OAc)_3$ gave predominantly benzofuranone derivatives as shown in Scheme 6 (24).

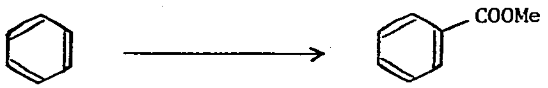
Scheme 6



R^1 = H, Me; R^2 = H, Me, CH_2Ph

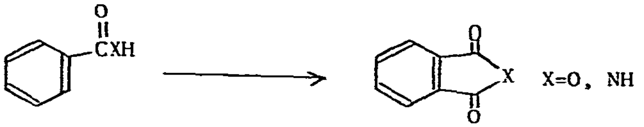
A method of enhancing reactivity of thallium(III) compounds in thallation of polyfluoroarenes was described (25). Thus, addition of $BF_3 \cdot OEt_2$ to the reaction mixture containing $Tl(OOCCF_3)_3$ and 2,3,5,6-tetrafluoroanisole gave, after cleavage with I^- ion, a 48 % yield of 2,3,5,6-tetrafluoro-4-iodoanisole, whereas no thallation was observed if $BF_3 \cdot OEt_2$ was absent. Similarly, SbF_5 effected thallation of 1,2,3,5-tetrafluorobenzene with $Tl(O_3SCF_3)_3$. Also found to be effective in such acid enhanced thallation were 4,4'-dihydrooctafluorobiphenyl, 1,2,3,5-tetrafluoro-4-nitrobenzene and pentafluorobenzene.

Carbonylation of arylthallium intermediates, obtained by thallation of various arenes with $Tl(OOCCF_3)_3$, proceeded under mild conditions in the presence of a catalytic amount of $PdCl_2$ to give good yields of esters, lactones, acid anhydrides and heterocycles (26). Typical examples are shown in eq. 11-14. The ease of this transformation may particularly be contrasted to the direct carbonylation of $ArTlX_2$ compounds which required much higher temperatures and pressures [J. Chem. Soc. A, (1968) 1616]. A possible reaction sequence involving an organopalladium(II) intermediate has been suggested.



(11)

(12)



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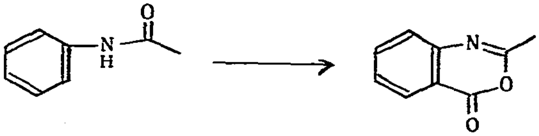
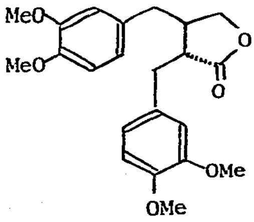


(14)

A full account of Tl(OOCCF$_3$)$_3$ mediated biaryl synthesis [J. Org. Chem., 42 (1977) 764; A.S. 1977, ref. 41] appeared (27). Thus, treatment of a variety of aromatic substrates with Tl(OOCCF$_3$)$_3$ in the presence of BF$_3$·OEt$_2$ resulted in smooth, rapid and direct regiospecific oxidative dehydrodimerization to give symmetrical biaryls in good to excellent yields. The reaction was thought to proceed through generation and reaction of the radical cation, ArH$^{+\cdot}$. The scope and limitations of the reaction and reactivity comparisons with other metal mediated oxidative coupling have been described. The same authors also reported full details of some isoquinoline alkaloid synthesis [Chem. Comm., (1977) 538; A.S. 1977, ref. 42] which makes use of the intramolecular version of the above aromatic coupling reaction (28). Related aryl coupling with Tl(OOCCF$_3$)$_3$/BF$_3$·OEt$_2$ by a British group [Synth. Comm., 8 (1978) 245] was extended to a tricyclic lignan lactone 26 to give a tetracyclic dibenzocyclooctadiene 27 in high yield (29).



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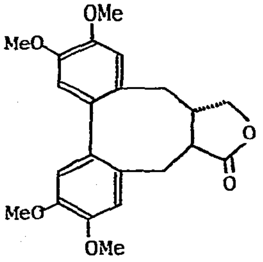
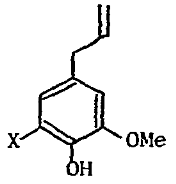


27

Oxidation of allylphenol 28 (X= H) with Tl(NO$_3$)$_3$ in methanol gave three products, 29 (X= H), 30 and 31 (30). The compounds 30 and 31 may have been formed from the diene 32. Similarly, oxidation of 28 (X= OMe) with Tl(NO$_3$)$_3$ gave 29 (X= OMe) and 33. On the other hand, the reaction of the nitro analogue 28 (X= NO$_2$) resulted in attack of Tl(NO$_3$)$_3$ at the side chain C=C bond to afford the usual diol and ketone derivatives.



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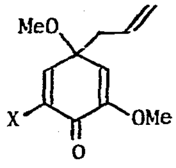

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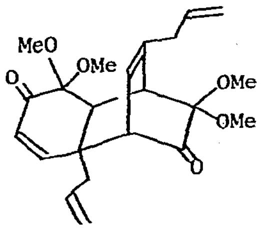

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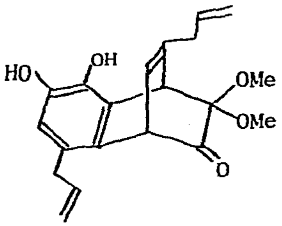

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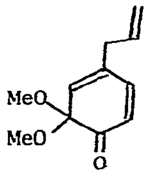

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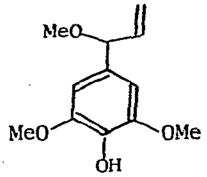
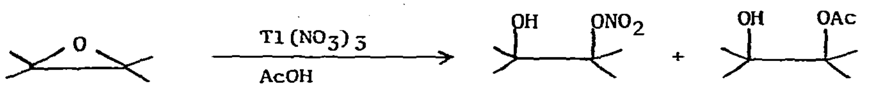


33

Reactivity patterns in the oxidation of aryldialkylamines by $Tl(NO_3)_3$ were compared with those observed with cerium(IV) nitrate (33). The reaction products were those derived from oxidative dealkylation and aromatic nitration, with the distribution of these being a function of the oxidizing power and electrophilicity of the reagent and the coordination property of the solvent. Thallium(III) nitrate was also used in a non-oxidative reaction, namely cleavage of epoxides and ethers as typically shown in eq. 15 and 16 (32).



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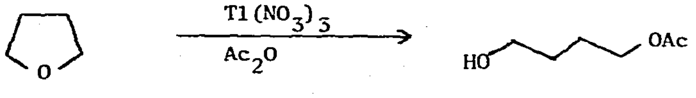
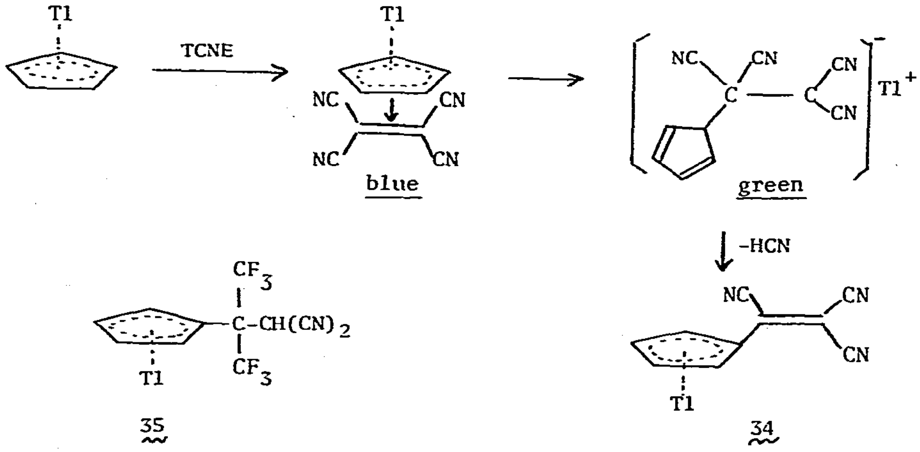


(16)

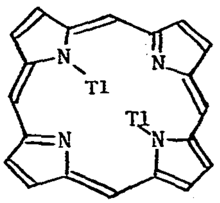
5. Organothallium(I) Compounds

A method of preparing indenylthallium(I) was reported which involved high temperature (90°C) treatment of aqueous basic Tl_2SO_4 solution with indene (33). The compound was a pale yellow solid, stable in air and more soluble in polar organic solvents than C_5H_5Tl. A full account of the study on a new organothallium(I) compound 34 derived from the reaction of tetracyanoethylene (TCNE) with C_5H_5Tl [J. Am. Chem. Soc., 99 (1977) 5194] appeared (34). The reaction scheme to form 34 was suggested to proceed through 1) formation of a blue πcomplex, 2) nucleophilic attack to give a green intermediate, and 3) elimination of HCN to afford the red compound 34 (Scheme 7). A similar reaction of 1,1-dicyano-2,2-bis(trifluoromethyl)ethylene gave 35. Use of 34 and 35 in making some substituted cyclopentadienyl derivatives of Fe, Mn and Cu was described.

Scheme 7



Stable thallium(I) derivatives of octaethyl- and tetraphenylporphyrins of the type 36 (ring substituents not shown) were prepared from the parent porphyrins and TlOEt (35). These compounds showed spectroscopic (NMR, UV-visible, mass) properties different from the corresponding thallium(III) porphyrin complexes of type 37 [J. Chem. Soc. Perkin I, (1973) 2142]. We now take this opportunity to add to the 1977 Thallium Annual Survey one related work of great significance which was erroneously missing in that survey. This is the structural and ^{13}C NMR study of the thallium(III)-porphyrin complexes 37 (X= Me, Cl) (36). In each complex the thallium atom of five-coordination is somewhat displaced from the mean plane of the four nitrogen atoms.



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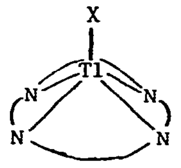
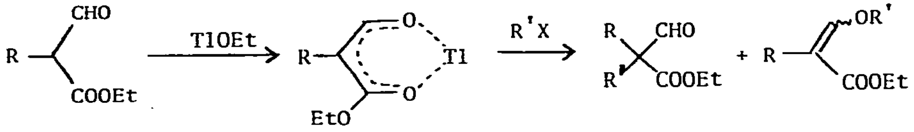


37

The ^{205}Tl NMR spectra of eleven thallium alkoxide derivatives were recorded in toluene solution (37). The observed ^{205}Tl-^{203}Tl coupling patterns were consistent with the predominance of tetrameric species, $[Tl(OR)]_4$, in solution.

The use of thallium(I) β-diketonates in synthetic reactions is well known. These compounds have now been applied to the C-alkylation of α-formyl esters as shown in eq. 17 (38). However, exclusive C-alkylation was not attained in each case, the method representing a useful procedure only when the α-formyl ester has a relatively low enolization tendency, as in the case of R= CH_2COOEt and Bu, and when the steric demand of the electrophile is small.



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