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Synthesis and complexation of dichalcogenoethers with cyclopropyl backbones, $(\overline{CH_2)_2}C(CH_2EMe)_2$ (E = Se or Te)

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ABSTRACT

The reaction of LiTeMe with $C(CH_2Br)_4$ in thf gives $(\overline{CH_2)_2}C(CH_2TeMe)_2$ irrespective of the ratio of reactants, in contrast to the reaction with LiSeMe, which gives either $C(CH_2SeMe)_4$ or $(\overline{CH_2)_2}C(CH_2SeMe)_2$ depending upon the reaction conditions. The synthesis and properties of $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$, $(\overline{CH_2)_2}C(CH_2TeMeI_2)_2$, $[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2EMe)_2\}]$ (E = Se or Te) and $[MCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2EMe)_2\}]PF_6$ (M = Ru or Os) are described. X-ray crystal structures are reported for $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$, $[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]$, $[MCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]PF_6$ (M = Ru, E = Se or Te and M = Os, E = Se). The effect of the cyclopropyl ring in the ligand backbone is to open up the C–C–C angle within the chelate ring, compared with trimethylene linked analogues. Selenium–carbon bond fission occurs on attempted quaternisation of $o\text{-}C_6H_4(CH_2SeMe)_2$ or $(\overline{CH_2)_2}C(CH_2SeMe)_2$ with MeI yielding $[Me_3Se]I$.

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1. Introduction

Detailed studies have shown that neutral selenium and tellurium donor ligands (chalcogenoethers R_2E, E = Se or Te) are better donors than the more familiar sulphur analogues. They also exhibit ready oxidation at the heteroatom, and cleavage of the weaker and more reactive E–C bonds, properties less commonly found with thioethers [1–3]. We recently reported the synthesis and some metal carbonyl complexes of the tetradentates 1,2,4,5-$C_6H_2(CH_2EMe)_4$ (E = S or Se) and $C(CH_2EMe)_4$ [4], and found that when E = Se, the reaction of MeSeLi with $C(CH_2Br)_4$ produces either $C(CH_2SeMe)_4$ or $(\overline{CH_2)_2}C(CH_2SeMe)_2$ depending upon the reaction conditions. The corresponding $(\overline{CH_2)_2}C(CH_2TeMe)_2$ was mentioned some years ago in a paper dealing with the synthesis of ditelluroethers [5], but the reaction was not investigated in detail. In the present paper we have explored the synthesis of the cyclopropyl-based telluroethers, and report some organotellurium derivatives, and a series of metal complexes of the 1,1-bis(methyl selenomethyl/telluromethyl)cyclopropanes. The use of selenium compounds, mostly based upon selenoalkenes, as reagents for the formation of cyclopropyl rings in organic synthesis, is well established [6].

2. Experimental

Infrared spectra were recorded as Nujol mulls between CsI plates over the range 4000–200 cm^{-1} or as chlorocarbon solutions in NaCl solution cells over the range 2200–1700 cm^{-1}, using Perkin–Elmer 983G or PE Spectrum100 instruments. ^1H and $^{13}C\{^1H\}$ NMR spectra were recorded at ambient temperatures unless stated otherwise, using a Bruker AV300 spectrometer and referenced internally to the solvent resonance, and $^{77}Se\{^1H\}$, $^{125}Te\{^1H\}$ and ^{55}Mn NMR spectra on a Bruker DPX400 spectrometer and are referenced to external neat $SeMe_2$, neat $TeMe_2$, and aqueous $KMnO_4$ respectively. Mass spectra were obtained using a VG Biotech platform. Microanalyses were undertaken by Medac Ltd. Electrochemical measurements were undertaken using an Ecochemie PGStat20 with 0.1 mol dm^{-3} $[NBu_4][BF_4]$ in dry MeCN as electrolyte. All preparations were carried out under a N_2 atmosphere. $(\overline{CH_2)_2}C(CH_2SeMe)_2$ [4], $[Mn(CO)_5Cl]$ [7], $[\{RuCl_2(\eta^6\text{-}p\text{-cymene})\}_2]$ and $[\{OsCl_2(\eta^6\text{-}p\text{-cymene})\}_2]$ [8] were made by literature methods.

2.1. 1,1-Bis(methyltelluromethyl)cyclopropane, $(\overline{CH_2)_2}C(CH_2TeMe)_2$

Freshly ground tellurium, powder (3.02 g, 29.3 mmol) was added to dry THF (25 mL) under nitrogen. The solution was then frozen with liquid nitrogen. MeLi solution (15.6 mL of 1.6 M solution in diethyl ether, 24.9 mmol) was then added via syringe and

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the mixture left to thaw for 40 min., then stirred at 0 °C for 2 h. The flask was then placed in an acetone slush bath (195 K) and pentaerythrityl tetrabromide (2.5 g, 6.5 mmol) in THF (30 mL) was added dropwise, and then stirred for a further 2 h. The reaction mixture was hydrolysed with saturated NaCl solution (25 mL). The product was extracted with diethyl ether (3 × 20 mL), the ether extracts dried over $MgSO_4$, filtered, and the solvent and Me_2Te_2 removed under high vacuum to yield a dark red oil. Yield: 1.2 g, (52% based on $C(CH_2Br)_4$). ^1H NMR ($CDCl_3$): 0.65 (s, [4H], $CH_{2cyclopropyl}$), 1.87 (s, [6H], TeMe), 2.80 (s, [4H], CH_2Te). $^{13}C\{^1H\}$ NMR ($CDCl_3$): −22.0 (TeMe), 16.4 (CH_2Te), 17.7 (CH_2C), 22.3 ($C_{quaternary}$). $^{125}Te\{^1H\}$ NMR (CH_2Cl_2): 63.1.

2.2. 1,1-Bis(dimethyltelluromethyl)cyclopropane di-iodide, $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$

$(\overline{CH_2)_2}C(CH_2TeMe)_2$ (0.10 g, 0.28 mmol) was stirred with an excess of iodomethane (1.5 mL) in CH_2Cl_2 (20 mL) for 30 min. The yellow solid was collected by filtration, washed with CH_2Cl_2 and dried *in vacuo*. Yield: 0.13 g (77%). *Anal.* Calc. for $C_9H_{20}I_2Te_2$: C, 17.0; H, 3.2. Found: C, 16.8; H, 3.1%. ^1H NMR (d^6-dmso): 0.92 (s, [4H], $CH_{2cyclopropyl}$), 2.25 (s, [12H], TeMe), 3.20 (s, [4H], CH_2Te). $^{125}Te\{^1H\}$ NMR (*N*,*N*-dimethylformamide/d^6-acetone): 470.8. ES^+-MS: *m/z* = 511 $[M-I]^+$. Crystals were grown by cooling a dmso/methanol solution of the compound at 255 K.

2.3. 1,1-Bis(methyldi-iodotelluromethyl)cyclopropane, $(\overline{CH_2)_2}C(CH_2TeMeI_2)_2$

$(\overline{CH_2)_2}C(CH_2TeMe)_2$ (0.19 g, 0.54 mmol) in dry degassed THF (20 mL) was stirred in a foil wrapped Schlenk tube, iodine (0.27 g, 1.1 mmol) in dry degassed THF (20 mL) was added via a syringe and stirred for a further 2 h. The solvent was then reduced in volume to 5 mL before dry diethyl ether (10 mL) was added. The dark red solid was collected via a filter cannula, washed with cold diethyl ether and dried in vacuo. Yield: 0.41 g (51%). *Anal.* Calc. for $C_7H_{14}I_4Te_2$: C, 9.8; H, 1.6. Found: C, 10.2; H, 1.6%. ^1H NMR (d^6-dmso, poorly soluble and solvent resonance partially obscures signal): 0.99 (s, [4H], $CH_{2cyclopropyl}$), 2.65 (s, [12H], TeMe), 3.12 (br, [4H], CH_2Te). $^{125}Te\{^1H\}$ NMR (d^6-dmso): 598.

2.4. Reaction of $(\overline{CH_2)_2}C(CH_2SeMe)_2$ with MeI

$(\overline{CH_2)_2}C(CH_2SeMe)_2$ (0.2 g, 0.78 mmol) was placed in a Schlenk tube and degassed acetone (10 mL) was added via syringe. An excess of iodomethane (2.5 mL) in degassed acetone (10 mL) was then added and the mixture was heated to 40 °C for 2 h. The white precipitate was collected *via* a filter cannula and dried under high vacuum to yield a white solid. Yield: 0.16 g (41% based upon Se). ^1H NMR (D_2O): 2.7 s. Lit [9] (D_2O) 2.7. $^{77}Se\{^1H\}$ NMR (dmso): 256. Lit [9] 253. ES^+-MS: *m/z* = 125 $[Me_3Se]^+$. The filtrate was refrigerated for 2 days when large yellow crystals formed. The crystal structure was determined and shown to be $[Me_3Se]I$ (orthorhombic, P*nma*, *a* = 13.960, *b* = 7.944, *c* = 6.158 Å), which is in excellent agreement with the literature data [10].

2.5. Reaction of *o*-$C_6H_4(CH_2SeMe)_2$ with MeI

o-$C_6H_4(CH_2SeMe)_2$ (0.2 g, 0.66 mmol) was placed in a Schlenk tube containing 10 mL of degassed acetone. An excess of iodomethane (2.5 mL) in degassed acetone (10 mL) was then added and the mixture was heated to 40 °C for 2 h. The resulting white precipitate was collected *via* a filter cannula and dried under high vacuum. Crystals were grown by placing the filtrate in the freezer for a week, shown to be $[C_9H_{11}Se]I$ (X-ray study – below). Yield: 0.12 g (88%) (yield of $[C_9H_{11}Se]I$). ^1H NMR ($CDCl_3$): 1.95 (s, [3H], Me), 4.25 (s, [4H], CH_2), 7.1 (m, [4H] $CH_{aromatic}$). $^{77}Se\{^1H\}$ NMR ($CDCl_3$): 406. ES^+-MS: *m/z* = 199 $[C_9H_{11}Se]^+$. NMR and mass spectrometry analysis of the filtrate from this reaction showed it also contains $[Me_3Se]I$ ($^{77}Se\{^1H\}$ NMR (acetone): 259; ES^+-MS: *m/z* = 125 $[Me_3Se]^+$).

2.6. [Chloro{1,1-bis(methylselenomethyl)cyclopropane}-(η^6-*p*-cymene)ruthenium(II)] hexafluorophosphate

$[Ru(\eta^6\text{-}p\text{-cymene})Cl_2]_2$ (0.12 g, 0.19 mmol) in dry ethanol (10 mL) was added to $(\overline{CH_2)_2}C(CH_2SeMe)_2$ (0.10 g, 0.39 mmol) in dry ethanol (5 mL) and refluxed for 2 h. $[NH_4][PF_6]$ (0.065 g, 0.4 mmol) in dry degassed ethanol (5 mL) was added, the solution was then allowed to cool and stirred overnight. The pale orange precipitate was filtered off, washed with cold ethanol (2 × 5 mL) and dried in vacuo. Yield: 0.17 g (65%). Crystals were grown by refrigerating the filtrate. *Anal.* Calc. for $C_{17}H_{28}ClF_6PRuSe_2$: C, 30.4; H, 4.2. Found: C, 30.3; H, 4.3%. ^1H NMR (CD_3CN): 0.62 (m, [2H], $CH_{2cyclopropyl}$), 0.80 (m, [2H], $CH_{2cyclopropyl}$), 1.32 (d, [6H], *J* = 7 Hz, $CH(CH_3)_2$), 2.24 (s, [3H], cymene-CH_3), 2.28, 2.45, 2.48 (major), 2.55 (4 × s, [6H], SeMe), 2.80 (m, [H], CH), 3.37, 3.41, 3.50 (major), 3.53 (major), 3.75, 3.79 (6 × s, [4H], CH_2Se), 5.60–5.84 (overlapping d, [4H], $CH_{aromatic}$). $^{77}Se\{^1H\}$ NMR (CH_2Cl_2): 147.8, 123.8, 117.7 (major), 71.8; (CH_3CN): 135.1, 113.7, 110.3 (major), 62.5. IR (Nujol, cm^{-1}): 839 υ(PF), 557 δ(PF). ES^+-MS: *m/z* = 527 $[C_{17}H_{28}ClRuSe_2]^+$.

2.7. [Chloro{1,1-bis(methyltelluromethyl)cyclopropane}-(η^6-*p*-cymene)ruthenium(II)] hexafluorophosphate

$[Ru(\eta^6\text{-}p\text{-cymene})Cl_2]_2$ (0.086 g, 0.14 mmol) in THF (10 mL) was added to a Schlenk tube containing $(\overline{CH_2)_2}C(CH_2TeMe)_2$ (0.10 g, 0.28 mmol) in dry degassed ethanol (10 mL). The dark red solution was then stirred at room temperature for 2 h. $[NH_4][PF_6]$ (0.05 g, 0.3 mmol) in dry degassed ethanol (5 mL) was added and the solution was stirred for a further 1 h before filtering to remove small amounts of insoluble material. The filtrate was then reduced in volume by 50% and placed in the freezer overnight. The bright red solid deposited was filtered off, washed with hexane (2 × 5 mL) and dried in vacuo. Yield 0.165 g (76%). Crystals were grown by refrigerating the filtrate. *Anal.* Calc. for $C_{17}H_{28}ClF_6PRuTe_2$: C, 26.6; H, 3.7. Found: C, 26.6; H, 3.4%. ^1H NMR (CD_3Cl_3): 0.51, 0.77, 1.00 (3 × m, [4H], $CH_{2cyclopropyl}$), 1.27 (overlapping d, [6H], $CH(CH_3)_2$), 2.19 (s, [3H], cymene-CH_3), 2.24, 2.25, 2.27, 2.30 (4 × s, [6H], TeMe), 2.76 (m, [H], CH), 3.10, 3.14, 3.46 (major), 3.50 (major), 3.67, 3.71 (6 × s, [4H], CH_2Te), 5.50–5.90 (overlapping m, [4H], $CH_{aromatic}$). $^{125}Te\{^1H\}$ NMR (CH_2Cl_2): 283.1, 265.8 (major), 264.0. IR (Nujol, cm^{-1}): 839 υ(PF), 557 δ(PF). ES^+-MS: *m/z* = 627 $[C_{17}H_{28}ClRuTe_2]^+$.

2.8. [Chloro{1,1-bis(methylselenomethyl)cyclopropane}-(η^6-*p*-cymene)osmium(II)] hexafluorophosphate

$[Os(\eta^6\text{-}p\text{-cymene})Cl_2]_2$ (0.15 g, 0.20 mmol) in dry degassed ethanol (30 mL) was added to a Schlenk tube containing $(\overline{CH_2)_2}C(CH_2SeMe)_2$ (0.10 g, 0.39 mmol) in dry degassed ethanol (10 mL). The dark yellow solution was then stirred at 70 °C for 1 h. $[NH_4][PF_6]$ (0.031 g, 0.20 mmol) in dry degassed ethanol (15 mL) was added, the solution was then stirred overnight before filtering to remove small amounts of insoluble material. The filtrate was reduced in volume by 50% and placed in the freezer overnight. The yellow solid deposited was filtered off, washed with hexane (2 × 5 mL) and dried in vacuo. Yield: 0.21 g (71%). Crystals were

Table 1
Crystal data and structure refinement details[a].

Complex	$[(CH_2)C(CH_2TeMe_2)_2]I_2$	$[o\text{-}C_6H_4(\overline{CH_2)_2}SeMe]I$	$[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]$
Formula	$C_9H_{20}I_2Te_2$	$C_9H_{11}ISe$	$C_{10}H_{14}ClMnO_3Te_2$
M	637.25	325.04	527.80
Crystal system	monoclinic	monoclinic	monoclinic
Space group	$P2_1/n$ (#14)	$P2_1/n$ (#14)	$P2_1/n$ (#14)
a (Å)	10.9852(15)	7.8050(10)	9.1254(15)
b (Å)	13.472(2)	11.2813(15)	15.567(4)
c (Å)	11.9328(15)	11.5214(15)	11.321(3)
α (°)	90	90	90
β (°)	114.696(10)	97.474(10)	107.847(15)
γ (°)	90	90	90
U (Å^3)	1604.5(4)	1005.8(2)	1530.7(6)
Z	4	4	4
μ(Mo Kα) (mm^{-1})	7.453	6.741	4.766
F(0 0 0)	1136	608	976
Total number of reflections	13490	11656	21722
Unique reflections	3583	2269	3508
R_{int}	0.102	0.024	0.038
Number of parameters, restraints	118, 17	100, 0	156, 0
R_1[b] [$I_o > 2\sigma(I_o)$]	0.088	0.023	0.030
R_1 (all data)	0.163	0.025	0.035
wR_2[b] [$I_o > 2\sigma(I_o)$]	0.220	0.051	0.063
wR_2 (all data)	0.278	0.052	0.065

Complex	$[Ru(p\text{-cymene})Cl\{(\overline{CH_2)_2}C(CH_2SeMe)_2\}][PF_6]$	$[Ru(p\text{-cymene})Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}][PF_6]$	$[Os(p\text{-cymene})Cl\{(\overline{CH_2)_2}C(CH_2SeMe)_2\}][PF_6]$
Formula	$C_{17}H_{28}ClF_6PRuSe_2$	$C_{17}H_{28}ClF_6PRuTe_2$	$C_{17}H_{28}ClF_6OsPSe_2$
M	671.80	769.08	760.93
Crystal system	orthorhombic	orthorhombic	orthorhombic
Space group	$P2_12_12_1$ (#19)	$P2_12_12_1$ (#19)	$P2_12_12_1$ (#19)
a (Å)	12.3668(15)	12.5305(15)	12.363(2)
b (Å)	12.4682(10)	12.5472(10)	12.4710(14)
c (Å)	15.028(2)	15.2251(10)	15.056(2)
α (°)	90	90	90
β (°)	90	90	90
γ (°)	90	90	90
U (Å^3)	2317.2(5)	2393.7(4)	2321.3(6)
Z	4	4	4
μ(Mo Kα) (mm^{-1})	4.051	3.270	8.862
F(0 0 0)	1312	1456	1440
Total number of reflections	15487	21520	20612
Unique reflections	5104	5219	5282
R_{int}	0.034	0.038	0.056
Number of parameters, restraints	254, 0	241, 6	258, 0
R_1[b] [$I_o > 2\sigma(I_o)$]	0.028	0.034	0.032
R_1 (all data)	0.031	0.038	0.037
wR_2[b] [$I_o > 2\sigma(I_o)$]	0.062	0.080	0.064
wR_2 (all data)	0.064	0.083	0.065

[a] Common items: temperature = 120 K; wavelength (Mo Kα) = 0.71073 Å; θ(max) = 27.5°.
[b] $R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$. $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma wF_o^4]^{1/2}$.

grown from the filtrate at −18 °C. *Anal.* Calc. for $C_{17}H_{28}ClF_6OsPSe_2$: C, 26.8; H, 3.7. Found: C, 25.4; H, 3.4%. ^{1}H NMR (CD_3CN): 0.58–0.72 (m, [4H], $CH_{2cyclopropyl}$), 1.27 (major), 1.30 (2 × d, [6H], $CH(CH_3)_2$), 2.16 (major), 2.19 (2 × s, [3H], cymene-CH_3), 2.27, 2.40 (major), 2.49, 2.58 (4 × s, [6H], SeMe), 2.90 (m, [H], CH), 3.46, 3.50, 3.62 (major), 3.65 (major), 3.82, 3.86 (6 × s, [4H], CH_2Se), 5.76–6.05 (overlapping d, [4H], $CH_{aromatic}$). ^{77}Se{^{1}H} NMR (CH_3CN): 92.9, 68.4, 65.8 (major), 47.2. IR (Nujol, cm^{-1}): 839 υ(PF), 557 δ(PF). ES$^+$-MS: m/z = 617 $[C_{17}H_{28}ClOsSe_2]^+$.

2.9. [Chloro{1,1-bis(methyltelluromethyl)cyclopropane}-(η^6-p-cymene)osmium(II)] hexafluorophosphate

$[Os(\eta^6\text{-}p\text{-cymene})Cl_2]_2$ (0.11 g, 0.14 mmol) in dry degassed ethanol (30 mL) was added to a Schlenk tube containing $(\overline{CH_2)_2}C(CH_2TeMe)_2$ (0.10 g, 0.28 mmol) in dry degassed ethanol (10 mL). The orange solution was then stirred at 50 °C for 1 h. $[NH_4][PF_6]$ (0.023 g, 0.14 mmol) in dry degassed ethanol (15 mL) was added, the solution was then stirred overnight before filtering to remove small amounts of insoluble material. The filtrate was then reduced in volume by 50% and placed in the freezer overnight. The orange solid was filtered, washed with hexane (2 × 5 mL) and dried in vacuo. Yield: 0.07 g (57%). ^{1}H NMR (CD_3CN): (see text) 0.50–0.91 (overlapping m, [4H], $CH_{2cyclopropyl}$), 1.24–1.29 (3 overlapping d, [6H], $CH(CH_3)_2$), 2.15, 2.16 (2 × s, [3H], cymene-CH_3), 2.25, 2.29, 2.37, 2.41 (4 × s, [6H], TeMe), 2.6–2.8 (m, [H], CH), 3.14, 3.18, 3.42, 3.52, 3.53 (major), 3.56 (major), 3.69, 3.73 (8 × s, [4H], CH_2Te), 5.67–6.10 (overlapping m, [4H], $CH_{aromatic}$). ^{125}Te{^{1}H} NMR (CH_3CN): 185.4, 164.5, 159.5 (major), 58.0. IR (Nujol, cm^{-1}): 839 υ(PF), 557 δ(PF). ES$^+$-MS: m/z = 715 $[C_{17}H_{28}ClOsTe_2]^+$.

2.10. Chlorotricarbonyl{1,1-bis(methylselenomethyl)cyclopropane}manganese(I)

$[Mn(CO)_5Cl]$ (0.09 g, 0.38 mmol) was dissolved in $CHCl_3$ (50 mL) and $(\overline{CH_2)_2}C(CH_2SeMe)_2$ (0.10 g, 0.38 mmol) in $CHCl_3$ (20 mL) added. The mixture was refluxed for 3 h, cooled, and the solvent removed in vacuo. The residue was washed with hot $CHCl_3$ (3×20 mL). The bright orange solid was dried in vacuo. Yield: 0.11 g (68%). *Anal.* Calc. for $C_{10}H_{14}ClMnO_3Se_2{\cdot}1/2CHCl_3$: C, 26.7; H, 3.1. Found: C, 26.2; H, 3.7%. ^{1}H NMR ($CDCl_3$): peaks too broad to assign. $^{13}C\{^1H\}$ NMR ($CH_2Cl_2/CDCl_3$): 10.88, 11.43, 12.77, 13.46, 14.61 ($CH_{2cyclopropyl}$), 15.16, 17.56, 18.00, 19.05 (MeSe), 21.65, 30.02 ($C_{quaternary}$), 32.50, 34.60, 35.10, 39.17 (CH_2Se), 219 (vbr) CO. $^{77}Se\{^1H\}$ NMR (CH_2Cl_2): 74.4, 76.7, 108.9, 136.5. ^{55}Mn NMR (CH_2Cl_2): −205, −230, −290. IR (CH_2Cl_2, cm^{-1}): υ(CO) 2028, 1948, 1916.

2.11. Chlorotricarbonyl{1,1-bis(methyltelluromethyl)cyclopropane}manganese(I)

$[Mn(CO)_5Cl]$ (0.09 g, 0.38 mmol) was dissolved in dry $CHCl_3$ (50 mL) and $(\overline{CH_2)_2}C(CH_2TeMe)_2$ (0.14 g, 0.38 mmol) in $CHCl_3$ (20 mL) was added. The mixture was heated at 40 °C for 3 h, cooled, and the solvent removed in vacuo. The residue was washed with hot $CHCl_3$ (3×20 mL). The resulting red solid was filtered and then dried in vacuo. Yield: 0.14 g (68%). *Anal.* Calc. for $C_{10}H_{14}ClMnO_3Te_2$: C, 22.8; H, 2.7. Found: C, 23.6; H, 2.3%. ^{1}H NMR ($CDCl_3$): very broad, see text. $^{13}C\{^1H\}$ NMR ($CH_2Cl_2/CDCl_3$): −21.73, −13.17, −10.67, −10.30 (TeMe), 16.66, 17.98, 19.17, 20.87, 22.77, 24.68 ($CH_2Te + CH_{2cyclopropyl}$), 220 (vbr) CO. $^{125}Te\{^1H\}$ NMR (CH_2Cl_2): 194.5, 183.8, 61.1 (major), 45.2. ^{55}Mn NMR (CH_2Cl_2): −615, −646, −688. IR (CH_2Cl_2. cm^{-1}): υ(CO) 2012, 1943, 1904.

2.12. X-ray crystallography experimental

Details of the crystallographic data collection and refinement parameters are given in Table 1. The crystallisation details are provided under the section for each compound. Data collection used a Nonius Kappa CCD diffractometer fitted with monochromated Mo Kα X-radiation (λ = 0.71073 Å), and with the crystals held at 120 K in a dinitrogen gas stream. Structure solution and refinement were straightforward [11–13], except as described below, and H atoms were introduced into the models in calculated positions using the default C–H distances. The diffraction data for $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$ were of modest quality and needed several SHELXL DELU commands to restrain the anisotropic atomic displacement parameters (adp). The data for $[Ru(p\text{-cymene})Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}][PF_6]$ were initially collected for a tetragonal system ($a \approx b$), but a solution only emerged in an orthorhombic space group. The anion showed large adp values for the F atoms, and this was modelled using the EADP command on *trans* F atoms and restraining all the P–F distances to be the same (DFIX/FVAR). Selected bond lengths and angles for the metal complexes are given in Tables 2 and 3.

Table 2
Selected bond lengths (Å) and angles (°) for *fac*-$[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]$.

Mn1–Te1	2.6394(7)	Mn1–C8	1.813(4)
Mn1–Te2	2.6285(8)	Mn1–C9	1.806(4)
Mn1–Cl1	2.4053(11)	Mn1–C10	1.785(4)
Te1···Te2	3.712(1)	C–O	1.139(5)–1.150(5)
Te1–Mn1–Te2	89.62(2)	Cl1–Mn1–Te1	88.65(3)
C8–Mn1–Cl1	91.03(13)	Cl1–Mn1–Te2	89.98(3)
C9–Mn1–Cl1	88.96(13)	C8–Mn1–Te2	89.13(12)
C9–Mn1–Te1	89.42(12)	C10–Mn1–Te2	90.26(12)
C10–Mn1–Te1	90.20(12)	C–Mn1–C	90.1(2)–91.8(2)

3. Results

3.1. Synthesis of the telluroether and organotellurium(IV) derivatives

We recently reported [4] that the reaction of MeSeLi with $C(CH_2Br)_4$ produces $C(CH_2SeMe)_4$ when the molar ratio of the reactants is 4.5:1, whilst increasing the amount of MeSeLi, progressively forms $(\overline{CH_2)_2}C(CH_2SeMe)_2$, the yield being ~90% with an 8:1 molar ratio. We have now re-examined the corresponding reactions of MeTeLi with $C(CH_2Br)_4$, which was reported [5] to give only $(\overline{CH_2)_2}C(CH_2TeMe)_2$, to establish whether the tetratelluroether, $C(CH_2TeMe)_4$, could be obtained. However, using varying molar ratios of MeTeLi to $C(CH_2Br)_4$ (~4:1 through 8:1) and reaction temperatures of 200 K to ambient gave only $(\overline{CH_2)_2}C(CH_2TeMe)_2$ (^{125}Te NMR: δ = 63.1) and Me_2Te_2 as significant tellurium-containing products. *In situ* $^{125}Te\{^1H\}$ NMR spectra taken from reaction mixtures at various stages also failed to show evidence for the tetratelluroether (since ^{125}Te chemical shifts are approximately additive with the substituents [5,14], the tetratelluroether is expected to have a resonance at δ ~30). The cyclopropyltelluroether was fully characterised spectroscopically and as the Te(IV) derivatives, $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$ and $(\overline{CH_2)_2}C(CH_2TeMeI_2)_2$, formed on reaction with excess MeI and I_2 respectively (Sections 2.2, 2.3). The conversion to the Te(IV) derivatives is accompanied by large, high-frequency shifts in the ^{125}Te NMR resonances as expected [5,15]. Rather poor quality crystals were obtained by refrigerating a dmso/MeOH solution of $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$ at 255 K. The structure (Fig. 1) provides conclusive proof of the ligand backbone and shows pyramidal TeC_3 units with longer Te···I (secondary) interactions in the range 3.803–3.568 Å completing a distorted octahedron about Te2 and a distorted square pyramid about Te1. The Te–C and Te–I distances are similar to those in other telluroni-

Table 3
Selected bond lengths (Å) and angles (°) for $[M(p\text{-cymene})Cl\{(\overline{CH_2)_2}C(CH_2EMe)_2\}]^+$ (M = Ru or Os, E = Se; M = Ru, E = Te).

M = Ru, E = Se		M = Ru, E = Te		M = Os, E = Se	
Ru1–Se1	2.4856(5)	Ru1–Te1	2.6222(6)	Os1–Se1	2.4954(7)
Ru1–Se2	2.4972(6)	Ru1–Te2	2.6231(7)	Os1–Se2	2.5034(7)
Ru1–Cl1	2.3960(10)	Ru1–Cl1	2.4097(15)	Os1–Cl1	2.4041(14)
$Ru1–C_{ring}$	2.178(4)–2.238(4)	$Ru1–C_{ring}$	2.188(7)–2.251(6)	$Os1–C_{ring}$	2.181(6)–2.231(6)
Se1–Ru1–Se2	87.87(2)	Te1–Ru1–Te2	88.17(2)	Se1–Os1–Se2	87.70(2)
Cl1–Ru1–Se2	88.58(3)	Cl1–Ru1–Te2	88.67(4)	Cl1–Os1–Se2	87.83(4)
Cl1–Ru1–Se1	88.36(3)	Cl1–Ru1–Te1	88.33(4)	Cl1–Os1–Se1	87.90(4)

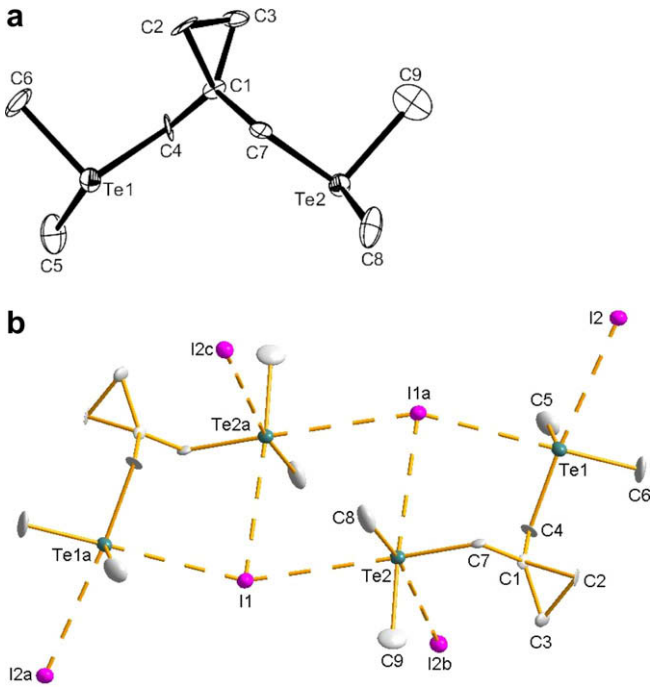


Fig. 1. (a) Structure of the cation in $[(\overline{CH_2)_2}C(CH_2TeMe_2)_2]I_2$ showing the numbering scheme adopted. Ellipsoids are drawn at the 60% probability level and H atoms have been omitted for clarity. Selected bond lengths (Å) and angles (°): Te–C 2.10(2)–2.18(2), Te1···Te2 4.925(2), C4–C1–C7 116.9(2), C2–C1–C3 58.7(12), Te–C–C 112.3(13), 115.1(12). (b) Shows the additional long range Te···I interactions 3.568–3.803 Å making Te1 [3 + 2] (approx. square pyramidal) and Te2 [3 + 3] (approx. octahedral) coordinate.

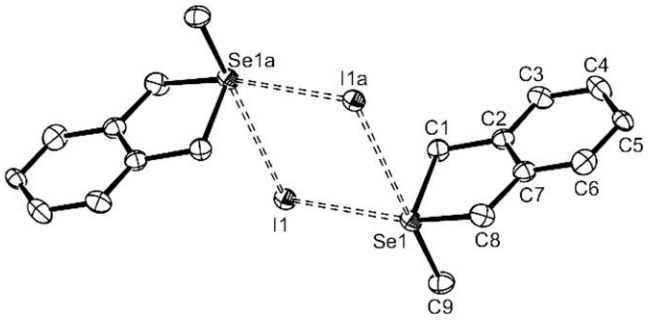


Fig. 2. Structure of $[o\text{-}C_6H_4(\overline{CH_2)_2}SeMe]I$ showing the weakly associated centrosymmetric dimer unit. Ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. Symmetry operation: $a = 1-x, -y, -z$. Selected bond lengths (Å) and angles (°): Se1–C1 1.949(3), Se1–C8 1.961(3), Se1–C9 1.940(3), C1–Se1–C8 91.00(12), C1–Se1–C9 97.28(13), C8–Se1–C9 97.62(14), Se1···I1 3.589(1), Se1···I1a 3.599(1).

um salts [16–18], although detailed comparisons are not justified due to the modest crystal quality.

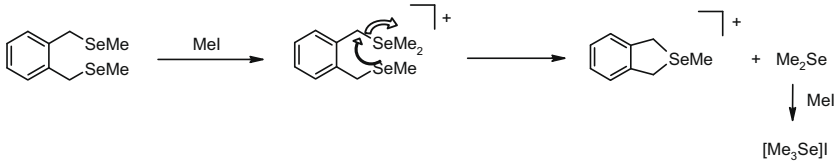
The formation of either the $C(CH_2SeMe)_4$ or $(\overline{CH_2)_2}C(CH_2SeMe)_2$ may be achieved depending upon the reaction conditions, whereas $(\overline{CH_2)_2}C(CH_2TeMe)_2$ is the only product in the $C(CH_2Br)_4$/LiTeMe reaction, shows the subtle balance between substitution and elimination in these systems. We note that the tritelluroethers $MeC(CH_2TeR)_3$ (R = Me or Ph) are prepared without difficulty [5,26]. Unsurprisingly, $C(CH_2SMe)_4$ is the only product of the reaction of $C(CH_2Br)_4$ with $\geqslant$4 molar equivalents of LiSMe [4], reflecting the stronger S–C bond which resists elimination of Me_2S_2. The fragility of the C–Se bonds in these systems was also shown in the reaction of $(\overline{CH_2)_2}C(CH_2SeMe)_2$ with MeI in warm acetone solution, which was expected to lead to the corresponding quaternary derivative $[(\overline{CH_2)_2}C(CH_2SeMe_2)_2]I_2$, as observed with other aliphatic diselenoethers [19]. Unexpectedly, the selenium-containing product of this reaction was identified as $[Me_3Se]I$ from the ^{77}Se NMR resonance at +256 (lit. [9] +253), and the major feature in the ES^+ mass spectrum at $m/z = 125$ $[Me_3Se]^+$. Crystals obtained from the solution were also confirmed to be $[Me_3Se]I$ by an X-ray study. The structure has previously been reported [10] and as our data are in excellent agreement, it is not discussed further. The $[Me_3Se]I$ is not a minor by-product since the ^{77}Se NMR spectrum of the bulk product showed no other significant selenium species.

A related rearrangement was observed on reaction of $o\text{-}C_6H_4(CH_2SeMe)_2$ with MeI. In this case the major products were the selenonium species $[o\text{-}C_6H_4(\overline{CH_2)_2}SeMe]I$ and $[Me_3Se]I$. The cation in the former species has been obtained previously from reaction of the diselenoether with $GaCl_3$ or $InCl_3$ [20,21], where it was isolated as $[o\text{-}C_6H_4(\overline{CH_2)_2}SeMe][MCl_4]$ (M = Ga or In) and characterised by an X-ray structure (Ga salt), its characteristic ^{77}Se chemical shift and by ES^+ mass spectrometry. In the present iodide salt the structure (Fig. 2) shows the cations are weakly linked into dimers via long Se···I···Se bridges (Se1···I1 3.589(1), Se1···I1a 3.599(1) Å). A possible mechanism for the rearrangement is that quaternisation of one selenium by MeI (or coordination to the Group 13 Lewis acid), polarises the $Se\text{–}C_\alpha H_2$ bond, which is then attacked by the second selenium, forming the cyclic selenonium cation and eliminating Me_2Se (Scheme 1). The Me_2Se formed is then quaternised to $[Me_3Se]I$. A different selenonium derivative $[C_{17}H_{19}Se]_2[TiCl_6]$ was obtained from the reaction of $o\text{-}C_6H_4(CH_2SeMe)_2$ with $TiCl_4$ [22], but the diselenoether ligand has produced a wide range of complexes with later transition metals in which it functions as a chelating bidentate as expected [23]. In contrast, the ditelluroether $o\text{-}C_6H_4(CH_2TeMe)_2$ quaternises "normally" to form $o\text{-}C_6H_4(CH_2TeMe_2I)_2$ which has a dimer structure composed of a Te_4I_4 cubane core [24]. It is clear that there is also a subtle balance between normal quaternisation and elimination in these systems, possibly the less electronegative Te centre makes the Te–C bond less polar and hence the mechanism in Scheme 1 is disfavoured.

3.2. Metal complexes of $(\overline{CH_2)_2}C(CH_2EMe)_2$ (E = Se or Te)

3.2.1. Manganese(I) carbonyl complexes

The reaction of $Mn(CO)_5Cl$ with $(\overline{CH_2)_2}C(CH_2EMe)_2$ (E = Se or Te) in hot chloroform gave orange-red *fac*-$[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2EMe)_2\}]$ as the sole product in each case. Pyramidal inversion at the chalcogen centres is slow on the NMR timescales for these complexes, both of which show three ^{55}Mn NMR resonances of disparate intensities, corresponding to the three invertomers *meso-1*, *meso-2* and *DL* with four corresponding ^{77}Se or ^{125}Te resonances (the *meso* forms have equivalent SeMe/TeMe groups either *syn* or *anti* to the Mn–Cl unit, whilst in the *DL* form two resonances of equal intensity are seen) [25,26]. As is common in complexes of manganese carbonyl halides, the resonances of the carbonyl groups in the $^{13}C\{^1H\}$ NMR spectra and the ^1H NMR resonances are extensively broadened by the quadrupolar manganese nucleus and the latter in particular provide little useful data. The two cyclopropyl-based ligands generate six-membered chelate rings and are directly comparable to the complexes of $MeE(CH_2)_3EMe$ (E = Se, Te), and comparison of the carbonyl stretching frequencies in the IR spectra, the ^{55}Mn chemical shifts, and the coordination shifts in the ^{77}Se and ^{125}Te NMR spectra, show very similar trends [25,26], the cyclopropyl unit in the ligand backbone having minimal spectroscopically identifiable effects at the metal centre, and there is no evidence in these systems of any reaction at the three-membered ring.



Scheme 1.

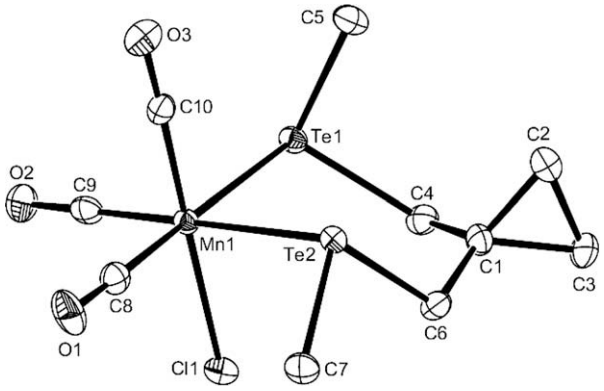


Fig. 3. Structure of $[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]$ showing the numbering scheme adopted. Ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

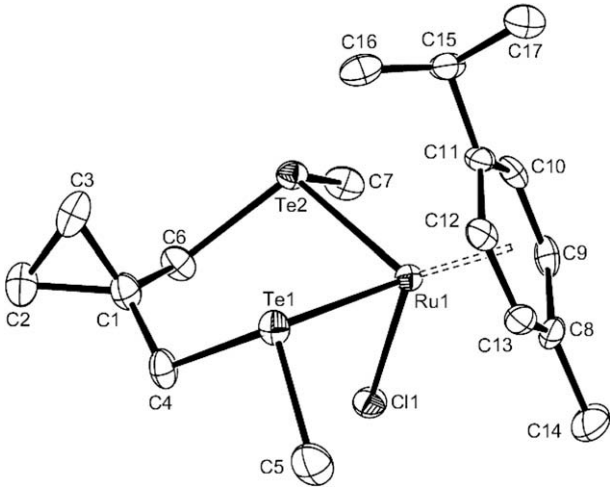


Fig. 4. Structure of the cation in $[Ru(p\text{-cymene})Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}][PF_6]$ showing the numbering scheme adopted. Ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. The numbering scheme adopted for Se-containing cyclopropyl ligand is the same in the Ru and Os compounds.

Red crystals of *fac*-$[Mn(CO)_3Cl\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]$ obtained from $CHCl_3$ solution were found to contain the *DL* form (Fig. 3, Table 2). The angles about the manganese centre are very close to the idealised 90°, with d(Mn–C) shorter *trans* to Cl (1.785(4) Å) than *trans* to Te (1.806(4), 1.813(4) Å). The Mn–Te bond lengths are very similar to those in other structurally characterised telluroether complexes with manganese carbonyl, e.g. *fac*-$[Mn(CO)_3Cl\{o\text{-}C_6H_4(TeMe)_2\}]$ and *fac*-$[Mn(CO)_3\{MeC(CH_2TeMe)_3\}][CF_3SO_3]$ [26]. The constrained geometry of the cyclopropane ring (e.g. C2–C1–C3 = 59.7°) is compensated for by a widening of the C4–C1–C6 angle within the chelate ring (116.7°), leading to Te–Mn–Te = 89.62(2)°.

3.2.2. Ruthenium and osmium complexes

The $[M(\eta^6\text{-}p\text{-cymene})Cl]^+$ (M = Ru or Os) units also proved to be suitable metal fragments to bind the chalcogenoethers, giving robust and soluble complexes and without reaction at the cyclopropyl rings. The reaction of two molar equivalents of $(\overline{CH_2)_2}C(CH_2EMe)_2$ (E = Se or Te) with $[\{MCl_2(\eta^6\text{-}p\text{-cymene})\}_2]$ in ethanol, followed by addition of $[NH_4][PF_6]$ gave good yields of orange (E = Se) or red (E = Te) complexes $[MCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2EMe)_2\}][PF_6]$. The complexes show ions with the correct isotope pattern for the $[MCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2EMe)_2\}]^+$ cation in the ES^+ mass spectra.

The ^1H NMR spectra are very complicated, however, both these and the $^{77}Se/^{125}Te$ NMR spectra are fully consistent with slow pyramidal inversion at the coordinated chalcogen donor atoms, revealing one major *meso* form (probably that seen in the X-ray structures – below), as well as minor amounts of the *DL* and sometimes the second *meso* form. For example, $[OsCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2TeMe)_2\}]^+$ shows four ^{125}Te resonances, a major peak at $\delta = 159$ corresponding to a *meso* invertomer and three much weaker peaks due to the *DL* (two singlets at 185.4 and 164.5 ppm) and the second *meso* form (58.0 ppm). The chemical shifts in the ^{77}Se and ^{125}Te NMR spectra for the stereoisomers span a considerable chemical shift range, reflecting the orientations of the EMe groups with respect to the *p*-cymene and Cl ligands, and are also moderately solvent dependent, for example in $[RuCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2SeMe)_2\}]^+$, the chemical shifts are ~7–10 ppm to lower frequency in CH_3CN compared to CH_2Cl_2 solution. The coordination shifts in the ^{77}Se and ^{125}Te spectra are greater for corresponding Ru complexes than for the Os analogues as expected [2].

Crystals of three Ru and Os complexes were obtained and the structures (Fig. 4, Tables 1 and 3) show they are isomorphous ($P2_12_12_1$) with *meso* coordinated chalcogenenoethers. The *i*-propyl group in the *p*-cymene lies on the opposite side of the ME_2 plane to the chalcogenoether methyl groups.

Cyclic voltammetry measurements were undertaken on solutions of $[MCl(\eta^6\text{-}p\text{-cymene})\{(\overline{CH_2)_2}C(CH_2EMe)_2\}][PF_6]$ in 0.1 mol dm^{-3} $^nBu_4NBF_4$ in dry MeCN. The voltammograms each show an irreversible oxidation at +0.90 (M = Ru, E = Se), +1.01 (M = Ru, E = Te) and +1.10 V (M = Os, E = Se) versus $[Fe(Cp)_2]/[Fe(Cp)_2]^+$ at a scan-rate of 0.1 V s^{-1} (the Os telluroether complex was not sufficiently soluble in the base electrolyte solution). These processes are attributed to irreversible $M^{II/III}$ couples. Electrochemical data are available for several Ru and Os complexes with selenoether and telluroether ligands. These are mostly of the form *trans*-$[MX_2(dichalcogenoether)_2]$ [27], and $[RuX_2(tetraselenoether)]$ [28]. Such species usually exhibit reversible II/III redox couples with $E_{1/2}$ values around 0 to +0.2 V versus $[Fe(Cp)_2]/[Fe(Cp)_2]^+$, with the $E_{1/2}$ values largely insensitive to chalcogen type and halide. The irreversibility observed for the *p*-cymene complexes here is also observed for $[Ru(p\text{-cymene})Cl(SMe_2)_2]^{+/2+}$ [29], possibly reflecting stabilisation of the low spin d^6 configuration by the arene co-ligand.

4. Conclusions

The formation of $(\overline{CH_2)_2}C(CH_2TeMe)_2$ and Me_2Te_2 as the only significant products of the reaction of $C(CH_2Br)_4$ with LiTeMe has been confirmed, and the reaction chemistry of this novel ligand and its selenium analogue has been explored with selected metal reagents. Some new organotellurium species have also been thoroughly characterised. The cyclopropyl unit seems unreactive in metal complexation chemistry, although the $(\overline{CH_2)_2}C(CH_2SeMe)_2$ (but not the telluroether) is cleaved upon quaternisation with MeI.

Acknowledgements

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Appendix A. Supplementary material

CCDC 755214, 755215, 755216, 755217, 755218 and 755219 contain the supplementary crystallographic data for the six structures reported in the order they appear in Table 1. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jorganchem.2010.02.010.

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