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Abstract

A series of organotellurium(IV) dithiocarbomate compounds have been synthesized in good yields under mild reaction conditions by reacting sodium telluromorpholine-4-carbodithioate and disodium homopiperazine-1,4-bis-carbothionate with various organotellurium(IV) compounds (*i.e.* Ar_2TeCl_2, and $Ar_2(CH_3)TeI$ where Ar = C_6H_5, 4-BrC_6H_4. 4-$CH_3C_6H_4$, and 4-$CH_3OC_6H_4$). The new complexes have been structurally characterized by analytical (CHN) and various spectral techniques, such as FT-IR, and ^1H and ^{13}C NMR. The coordination mode of the ligands and the geometry of all compounds were proposed according to their spectroscopic data.

INTRODUCTION

Dithiocarbamates ($R_2NCS_2^-$ or $RNHCS_2^-$) are consider as an example of a general class of monoanionic 1,1-dithiolate ligands. Dithiocarbamate complexes exhibited various modes of coordination, such as classical monodentate, symmetric bidentate, and asymmetric bidentate modes. The dithiocarbamates and their metal complexes find use in lots of application such as radical precursors and intermediates in organic synthesis [1], antibacterial,[2] anthelmintic,[3] fungicidal[4], catalysts for as accelerators in rubber vulcanization [5], stabilizers for polypropylene [6] and for the important pharmacological implications[7].
Many complexes of dithiocarbamate with the transition elements [8] and main group elements [9] are well documented in literature. Tellurium(IV)-dithiocarbamate complexes have been reported much less than those of main group elements[10-13].

The coordination chemistry of the six-membered, telluromorpholine-4-carbodithioate, ligand and the seven-membered, homopiperazine-1,4-bis-carbothionate ligand, with organotellurium compounds are unknown and to the best of our knowledge and no example has been reported to date. Thus, the present work describes the synthesis of new organotellurium(IV)-dithiocarbamate complexes.

EXPERIMENTAL

a) Synthesis

sodium homopiperazine-1,4-bis-carbodithioate [14], telluromorpholine [15], diphenyltellurium dichloride [16], bis(4-bromophenyl)tellurium dichloride[17], bis(4-methylphenyl)tellurium dichloride[16], bis(4-methoxyphenyl)tellurium dichloride[16], diphenyl methyl telluronium iodide [18], bis(4-bromophenyl) methyl telluronium iodide[18], bis(4-methylphenyl) methyl telluronium iodide[18], and bis(4-methoxyphenyl) methyl telluronium iodide[18,19] were prepared according to literature methods. The products were purified by recrystallization and verified by their melting points and IR spectra.

Synthesis of sodium telluromorpholine-4-carbodithioate

Sodium hydroxide (0.8 g, 20 mmol) was dissolved in minimum amount of cold water. To this solution was added 25 mL of cold ethanol. The resulting solution was stirred in an ice bath and telluromorpholine (3.98 g, 20 mmol) was added. Carbon disulfide (20 mmol, 1.22 mL) was added drop-wise with continuous stirring. The reaction mixture was stirred in ice-bath for I h. The yellow solid was collected and recrystallized from a methanol/ether (1:1) giving sodium telluromorpholine-4-carbodithioate as a yellowish-white solid in 78% yield, m.p. 125-127^{o}C.

Anal. calc. for $C_5H_8NNaS_2Te$: C, 20.23; H, 2.72; N, 4.72; S, 21.60. Found: C, 20.15; H, 2.55; N, 4.27; S, 21.13.

IR (KBr, cm^{-1}): 2978m, 2934s, 2851s, 2102b, 1623s, 1483s, 1448s, 1409s, 1365s, 1258s, 1187s, 1095m, 1047s, 975s, 953s, 904m, 850s, 762m, 538m, 454m.

^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ 4.09 (t, 4 H, *J* Hz, ***CH$_2$***N), 2.96 (t, 4 H, ***CH$_2$***Te).

^{13}C NMR ($CDCl_3$, ppm): δ 202.5 (***CS$_2$***), 53.1(***CH$_2$***N), 27.1 (**CH$_2$**Te).

Compounds diphenyltellurium bis(telluromorpholine-4-carbodithioate) (**1**), bis(4-bromophenyl)tellurium bis(telluromorpholine-4-carbodithioate) (**2**), bis(4-methylphenyl)tellurium bis(telluromorpholine-4-carbodithioate) (**3**) and bis(4-methoxyphenyl)tellurium bis- (telluromorpholine-4-carbodithioate) (**4**) were prepared by the following general method and as described for the preparation of complex **1**:

Diphenyltellurium bis(telluromorpholine-4-carbodithioate) (**1**)

Diphenyltellurium dichloride (0.35 g; 1 mmol) in dichloromethane (25 mL) was mixed with a solution of sodium telluromorpholine-4-carbodithioate (0.60 g; 2 mmol) in dry ethanol (25 mL). The mixture was stirred under nitrogen for 3 h, after which the solvent was removed under reduced pressure. Dichloromethane was added to the residue with vigorous stirring, filtered to remove sodium iodide and then the solvent was removed. Methanol (10 mL) was added to the product with stirring and filtered. The crude product was recrystallized from ethanol to give pale yellow solid.

Diphenyl methyl telluronium telluromorpholine-4-carbodithioate (**5**), bis(4-bromophenyl) methyl telluronium telluromorpholine-4-carbodithioate (**6**), bis(4-methylphenyl) methyl telluronium telluromorpholine-4-carbodithioate (**7**), and bis(4-methoxyphenyl) methyl telluronium telluromorpholine-4-carbodithioate (**8**) were prepared by the following example:

Diphenyl methyl telluronium telluromorpholine-4-carbodithioate (**5**)

To a solution of diphenyl methyl telluronium iodide (0.42g; 1 mmol) in dichloromethane was added with stirring under nitrogen, a solution of sodium telluromorpholine-4-carbodithioate (0.30 g; 3 mmol) in methanol (25 mL) at room temperature. The stirring was continued for 1 h, after which the solvent was removed by evaporation. The product was treated with

dichloromethane and filtered to remove sodium iodide. Removal of solvent and recrystallization from ethanol gave a pale-yellow precipitate.

Bis(methyldiphenyl-λ^4-tellaneyl) 1,4-diazepane-1,4-bis(carbodithioate) **(9)**
This compound was prepared by a procedure identical to that for compound **5** by using diphenyl methyl telluronium iodide (0.42g; 1 mmol) and sodium homopiperazine-1,4-bis-carbodithioate (0.15 g; 0.5 mmol). A pale yellow precipitate was obtained in good yield.
Compounds bis(bis(4-bromophenyl)(methyl)-l4-tellaneyl) 1,4-diazepane-1,4-bis(carbodithioate) (**10**), bis(methyldi-p-tolyl- λ^4-tellaneyl) 1,4-diazepane-1,4-bis(carbodithioate) (**11**) and bis(bis(4-methoxyphenyl)(methyl)-l4-tellaneyl) 1,4-diazepane-1,4-bis(carbodithioate) (**12**) were prepared by the same above method.
Physical and analytical data for compounds **1**-**12** are presented in Table 1.

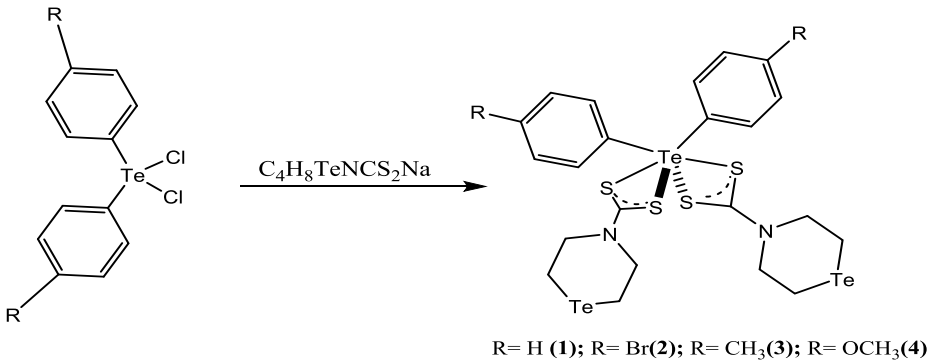
b) Physical measurements

The IR spectra were recorded in the range 4000-200 cm^{-1} on a Pye-Unicam SP3-300 spectrometer using KBr discs. ^1H and ^{13}C NMR spectra were measured on a Varian VXR-300 (300 MHz) with TMS as internal reference. Mass spectra of the complexes were measured at 70 eV with a MAT-1125 Finnigan mass spectrometer; the peaks shown relate to ^{130}Te. Microanalyses for carbon, hydrogen and nitrogen were carried out on a Perkin-Elmer 240B elemental analyzer or on a CHN Corder-MT3-Yanaca. Conductivity data were measured with a WTW conductivity meter LBR, using a standard conductivity cell with cell constant of 0.81
cm^{-1}. Melting points were measured by a Gallenkamp melting point apparatus and are uncorrected.

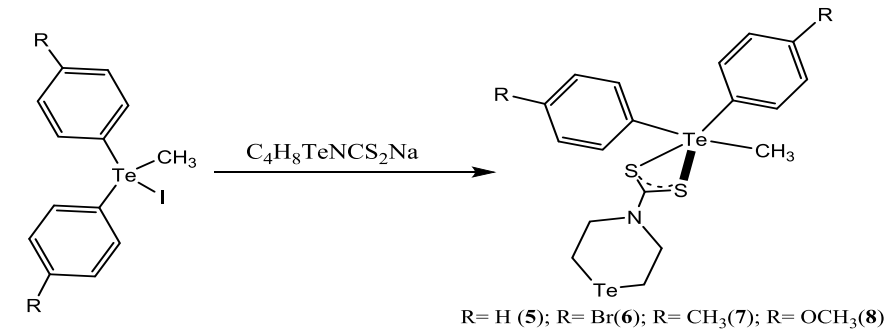
RESULTS AND DISCUSSION

The new ligand sodium telluromorpholine-4-carbodithioate was prepared as yellowish-white in good yield. Isolated yields, melting points and analytical data for the newly synthesized complexes (**1**-**12**) are listed in Table 1. The organotellurium to carbodithioate ligand molar ratios were 1: 1 and 2:1 for complexes **1**-**8** and **10**-**12**, respectively, Table 1 and Scheme 1. All the new complexes are pale yellow solids and soluble in common organic solvents. It is worth noting that complexes **1**-**8** decomposed on storage for a long time and liberated tellurium, while complexes **9**-**12** are stable for long time storage. The conductivity measurements of all complexes in DMSO indicated the non-electrolyte nature of these complexes, Table 1.
Some important IR frequencies of these complexes are listed in Table 2. The spectra showed that the bands due to the ν N–CS_2 are in the range of 1465–1480 cm^{-1}, which may suggest a bond order between a single and double carbon-nitrogen bond. Furthermore, the presence of a single and strong band due to a υ(CS_2) mode in the range 965-1010 cm^{-1} in the spectra of all complexes is strongly indicative of bidentate behavior of the 1,1-dithiolate in these complexes [20-22], otherwise a doublet is expected in the region 1000 ±70 cm^{-1} in the case of monodentate type coordination [23, 24]. Since all crystallographic data to date [25-27] show unsymmetrically bidentate dithiocarbamate groups to be coordinated to tellurium, it is clearly unwise to speculate more from our IR data than the bidentate behavior of the dithiocarbamate ligands in these complexes (*i.e.* **1**-**12**).

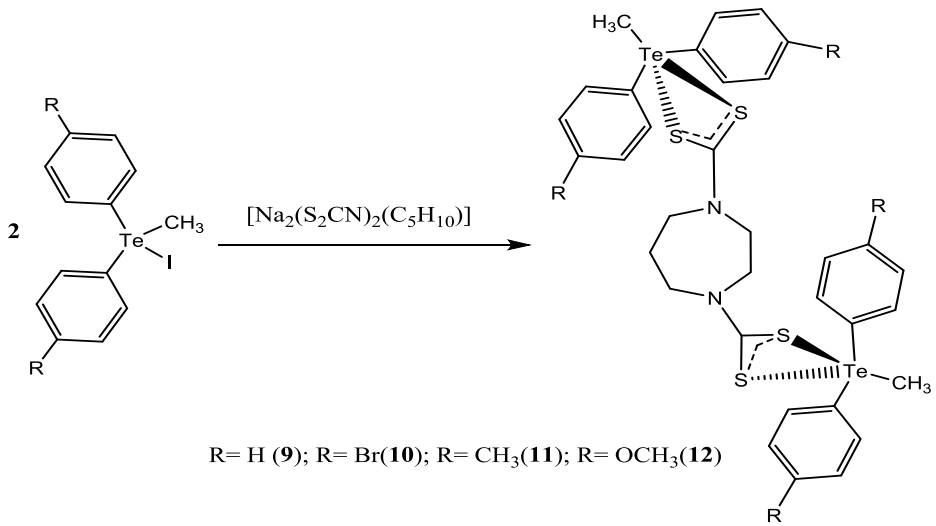
^{1}H NMR data are given in Table 2. The spectra showed proton resonances related to the ligand backbone: telluromorpholine-4-carbodithioate, and homopiperazine-1,4-bis-carbothionate, as well as those for the organotellurium entities.



R= H **(1);** R= Br**(2);** R= CH_3**(3);** R= OCH_3**(4)**



R= H (**5**); R= Br(**6**); R= CH_3(**7**); R= OCH_3(**8**)



R= H (**9**); R= Br(**10**); R= CH_3(**11**); R= OCH_3(**12**)

Scheme 1. Preparative methods for compounds **1-12**.

The methylene protons for Te*CH*$_2$ and N*CH*$_2$ appeared as triplets at ~ 3.0 ppm and at ~ 3.5 ppm, respectively for the complexes **1**-**8**, Table 2.
The peak integration is in good agreement with the proposed formulation of complexes 1-**8**, Table 2. ^{1}H NMR data for complexes **9**-**12**, all of the aromatic and aliphatic protons were observed at the estimated chemical shifts. The NCH$_2$***CH$_2$***CH$_2$N proton of the homopiperazine group gave a quintet signal at ~ 2.5 ppm. The N***CH$_2$***CH$_2$***CH$_2$***N protons were found at ~ 4.0 ppm while N***CH$_2$CH$_2$***N proton found at ~ 4.2 ppm for the homopiperazine moieties, Tabl 2. The aromatic protons of phenyl ring were found at 6.87–7.45 ppm, Table 2.
The ^{13}C NMR spectra of **1**–**12** show the expected patterns, with only one signal due to the CS_2 groups in the range of 191–200 ppm, that is shifted upfield with respect to the starting sodium salts of the ligands. Thus, the signal for the carbon atom of the dithiocarbamate group confirmed the tellurium– carbodithioate complex formation. The signals for Te- C_{phenyl} found around 121.0 ppm, Table 3. The large variation for carbon atoms bearing tellurium may be attributed to the polarity of Te–C bond [28]. The signals due to the carbon atoms of the organotellurium moieties do not present a significative variation with respect to the starting compounds, Table 3. The However, according to the spectroscopic data octahedral and trigonal bipyramidal geometries for these new compound may propose, Scheme 1.

In conclusion, the synthesis and characterization of three new series of organotellurium(IV) complexes with six- and seven-membered dithiocarbamates ligands is described and considering a bidentate coordination of these ligands in the three new series. Thus, it is possible to describe a geometry around the tellurium atom, octahedral for complexes **1**-**4**, and trigonal bipyramidal for complexes **8**-**12**.

Table 1. Analytical and physical properties of the new complexes **1** – **12**.

Complex	Yield (%)	M.p. (^{o}C)	Analysis[a]				Λ_M[b] (Ω^{-1} cm^2 mol^{-1})
			C	H	N	S	
1	78	144-146	31.76 (31.86)	2.96 (3.16)	3.21 (3.38)	15.12 (15.46)	6.85
2	69	213-215	26.33 (26.76)	2.45 (2.45)	2.27 (2.84)	12.68 (12.99)	5.78
3	83	186-187	33.73 (33.61)	3.45 (3.53)	3.14 (3.27)	14.68 (14.95)	4.33
4	80	201-203	31.87 (32.41)	3.21 (3.40)	2.87 (3.15)	14.21 (14.42)	5.21
5	83	157-159	33.54 (33.88)	3.58 (3.71)	1.97 (2.45)	11.05 (11.24)	6.36
6	86	114-117	29.42 (29.68)	2.57 (2.63)	1.76 (1.92)	8.57 (8.80)	4.22
7	74	203-205	39.79 (40.12)	4.03 (4.21)	1.98 (2.34)	10.34 (10.71)	6.10
8	67	131-133	37.83 (38.09)	3.86 (4.00)	1.86 (2.22)	9.82 (10.17)	6.35
9	82	156-158	47.15 (46.96)	4.11 (4.30)	3.02 (3.32)	14.86 (15.19)	5.22

10	64	163-166	34.10 (34.18)	2.43 (2.78)	2.03 (2.42)	10.74 (11.06)	6.03
11	72	192-193	49.24 (49.37)	4.57 (4.93)	2.89 (3.11)	13.86 (14.25)	4.73
12	80	173-175	45.83 (46.09)	4.54 (4.60)	2.57 (2.91)	12.84 (13.30)	5.05

[a] Calculated values are in parentheses. [b] 10^{-3} M DMSO solution.

Table 2. Selected IR and ^{1}H NMR data for the complexes **1**-**12**.

Complexes	ν(N–CS)	ν(CS_2)	^{1}H NMR (ppm)
1	1465s	975s	3.05(t, 4H, *J* = 7.1 Hz, Te***CH₂***),3.54(t, 4H, *J* = 6.8 Hz N***CH₂***), 7.27(m, 5H, ***Ar-H***).
2	1480s	980s	2.96(t, 4H, *J* = 7.0 Hz, Te***CH₂***),3.46(t, 4H, *J* = 7.1 Hz, N***CH₂***), 7.19 (d, 2H, *J*= 7.2Hz, Ar-***H***), 7.43(d, 2H, *J*= 6.9Hz, Ar-***H***).
3	1430s	992s	3.15(t, 4H, *J* = 7.3 Hz, Te***CH₂***), 3.28(s, 3H, Ar-***CH₃***), 3.66(t, 4H, *J* = 6.8 Hz, N***CH₂***), 7.02 (d, 2H, *J*= 6.9Hz,Ar-***H***), 7.52(d, 2H, *J*= 7.9Hz, Ar-***H***).
4	1475s	975s	3.15(t, 4H, Te***CH₂***), 3.76(s, 3H, O***CH₃***), 3.72(t, 4H, N***CH₂***), 7.12 (d, 2H, *J*= 7.3Hz Ar-***H***), 7.19(d, 2H, *J*= 6.9Hz, Ar-***H***).
5	1482s	985s	2.43(s, 3H, ***CH₃***), 3.12(t, 4H, Te***CH₂***), 3.36(t, 4H, N***CH₂***), 7.17-7.28 (m, 4H, Ar-***H***), 7.30 – 7.41 (m, 2H, Ar-***H***).
6	1455s	1002s	2.79(t, 4H, *J* = 6.9 Hz, Te***CH₂***),3.52(t, 4H, *J* = 6.8 Hz, N***CH₂***), 7.41 – 7.50 (m, 4H, Ar-***H***), 7.53 – 7. 62 (m, 4H, Ar-*H*).
7	1485s	975s	2.38(s, 3H, Ar-CH_3), 3.33(t, 4H, *J* = 7.3 Hz, TeCH_2), 3.59(t, 4H, *J* = 6.8 Hz, NCH_2), 6.98 (dq, *J* = 7.6, 0.7 Hz, 4H, Ar-*H*), 7.46 – 7.37 (m, 4H, Ar-*H*).
8	1490s	980s	2.91(t, 4H, Te***CH₂***), 3.61(s, 3H, O***CH₃***), 3.69(t, 4H, N***CH₂***), 6.85 – 6.75 (m, 4H, Ar-***H***), 7.60 – 7.50 (m, 4H, Ar-***H***).
9	1470s	1010s	2.41(q, 2H, *J* 6.4, 12.1Hz, NCH_2***CH₂***CH_2N), 2.91(s, 6H, ***CH₃***), 4.28(s, 4H, N***CH₂CH₂***N), 4.01(t, 4H, *J*=6.8Hz, N***CH₂***CH_2***CH₂***N), 7.50 – 7.41 (m, 4H, Ar-*H*), (m, 10H, Ar-***H***).
10	1482s	985s	2.39(q, 2H, *J* 6.4, 12.5Hz, , NCH_2***CH₂***CH_2N), 2.87(s, 6H, ***CH₃***), 4.23(s, 4H, N***CH₂CH₂***N), 4.03(t, 4H, *J*=6.8Hz, N***CH₂***CH_2***CH₂***N), 7.37 (d, 4H, *J*= 6.9Hz, Ar-***H***), 7.51(d, 4H, *J*= 7.5Hz, Ar-***H***).
11	1485s	1005s	2.35(s, 12H, Ar-***CH₃***), 2.53(q, 4H, *J* 6.4, 12.1Hz, , NCH_2***CH₂***CH_2N), 2.83(s, 6H, ***CH₃***), 4.21(s, 4H, N***CH₂CH₂***N), 4.11(t, 4H, *J*=6.8Hz, N***CH₂***CH_2***CH₂***N), 7.12 (d, 4H, *J*= 7.7Hz, Ar-***H)***, 7.19 (d, 4H, *J*= 7.2Hz, Ar-***H***).
12	1482s	995s	2.41(q, 4H, *J* 6.4, 12.1Hz, , NCH_2***CH₂***CH_2N), 2.77(s, 6H, ***CH₃***), 3.78(s, 12H, ***OCH₃***), 4.28(s, 4H, N***CH₂CH₂***N), 4.01(t, 4H, *J*=6.8Hz, N***CH₂***CH_2***CH₂***N), 6.83(d, 4H, *J*= 7.8Hz, Ar-***H***), 7.17(d, 4H, *J*= 8.1Hz, Ar-***H***).

Table 3. ^{13}C NMR data for complexes **1**-**12** in $CDCl_3$.

Compounds	δ ppm
1	19.5(Te*CH*$_2$),53.9(N*CH*$_2$), 121.7 (Te-C$_{Ar}$), 130. 6(C$_{Ar}$), 130.7(C$_{Ar}$), 133.3 (C$_{Ar}$), 192.4(CS$_2$).
2	18.8(Te*CH*$_2$), 55.0 (N*CH*$_2$), 122.5(Te-C$_{Ar}$), 122.3(Br-C$_{Ar}$), 134.3(C$_{Ar}$), 135.2(C$_{Ar}$), 192.4(CS$_2$).
3	19.8 (Te*CH*$_2$), 21.3 (CH$_3$-C$_{Ar}$), 53.2(N*CH*$_2$), 120.7 (Te-C$_{Ar}$), 133.4 (C$_{Ar}$), 133.5(C$_{Ar}$), 138.1(O-C$_{arom}$), 197.1(CS$_2$).
4	21.1(Te*CH*$_2$), 54.1(N*CH*$_2$), 55.3(O*CH*$_3$), 121.3(Te-C$_{Ar}$), 116.2(C$_{Ar}$), 134.5(C$_{Ar}$), 159.1(O-C$_{Ar}$), 195.2(CS$_2$).
5	20.1(Te*CH*$_2$), 21.9(Te*CH*$_3$), 53.9 (N*CH*$_2$),122.1(Te-C$_{Ar}$), 131.2(C$_{Ar}$),131.3(C$_{Ar}$), 137.1(C$_{Ar}$), 194.6(CS$_2$).
6	19.8(Te*CH*$_2$), 21.6(Te*CH*$_3$), 54.1 (N*CH*$_2$),121.4(Te-C$_{Ar}$), 122.9((Br-C$_{Ar}$), 136.1(C$_{Ar}$), 136.2(C$_{Ar}$), 193.8(CS$_2$).
7	19.8(Te*CH*$_2$), 21.3(Te*CH*$_3$), 54.2 (N*CH*$_2$),120.2(Te-C$_{Ar}$), 137.6 (CH$_3$-C$_{Ar}$), 132.1(C$_{Ar}$), 136.4(C$_{Ar}$), 197.1(CS$_2$).
8	20.1(Te*CH*$_2$), 21.9(Te*CH*$_3$), 53.2 (N*CH*$_2$), 54.2(O*CH*$_3$), 117.2(C$_{Ar}$),120.2(Te-C$_{Ar}$), 134.2(C$_{Ar}$), 158.9 (O-C$_{Ar}$), 197.1(CS$_2$).
9	20.9(Te*CH*$_3$), 27.3(NCH$_2$*CH*$_2$CH$_2$N), 44.6(N*CH*$_2$CH$_2$*CH*$_2$N), 54.3(N*CH*$_2$*CH*$_2$N), 122.5(Te-C$_{Ar}$), 127.4(C$_{Ar}$), 131.6(C$_{Ar}$), 132.2(C$_{Ar}$), 191.3(CS$_2$).
10	20.9(Te*CH*$_3$), 27.3(NCH$_2$*CH*$_2$CH$_2$N), 44.6(N*CH*$_2$CH$_2$*CH*$_2$N), 54.3(N*CH*$_2$*CH*$_2$N), 120.5(Te-C$_{Ar}$), 122.5((Br-C$_{Ar}$), 131.6(C$_{Ar}$), 132.2(C$_{Ar}$), 195.8(CS$_2$).
11	20.3(Te*CH*$_3$), 21.5 (CH$_3$-C$_{Ar}$), 26.8(NCH$_2$*CH*$_2$CH$_2$N), 45.2(N*CH*$_2$CH$_2$*CH*$_2$N), 53.8(N*CH*$_2$*CH*$_2$N), 119.9(Te-C$_{Ar}$), 133.6 (C$_{Ar}$), 133.8(C$_{Ar}$), 137.9(O-C$_{arom}$), 195.8(CS$_2$).
12	20.8(Te*CH*$_3$), 22.1 (CH$_3$-C$_{Ar}$), 27.0(NCH$_2$*CH*$_2$CH$_2$N), 44.9(N*CH*$_2$CH$_2$*CH*$_2$N), 54.2(N*CH*$_2$*CH*$_2$N), 120.1(Te-C$_{Ar}$), 133.2 (C$_{Ar}$), 133.4(C$_{Ar}$), 138.0(O-C$_{arom}$), 195.9(CS$_2$).

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