ISSN 1070-3632, Russian Journal of General Chemistry, 2020, Vol. 90, No. 4, pp. 703–709. © Pleiades Publishing, Ltd., 2020.

Mercuration and Telluration of 2-Fluoro-5-nitroaniline: Synthesis, Antibacterial, and Computational Study

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Received February 1, 2020; revised April 7, 2020; accepted April 10, 2020

Abstract—New derivatives of organotellurium and organomercury compounds have been synthesized in the reaction of 2-fluoro-5-nitroaniline with mercuric acetate and further with tellurium(IV) tetrabromide. Reaction of (4-amino-5-fluoro-2-nitrophenyl)tellurium(IV) tribromide with 4-hydroxyphenyl mercury(II) chloride gives asymmetrical diaryltellurium(IV) dibromide, whereas reaction of (4-amino-5-fluoro-2-nitrophenyl)mercury(II) chloride with phenol and 4-hydroxy benzaldehyde produces a new aryl mercury(II) chloride that contains the azomethine and azo groups. Reaction of aryl mercury(II) chloride with tellurium(IV) tetrabromide results in aryaltellurium(IV) tribromide furnished by the azomethine and azo groups. Reduction of unsymmetrical diaryltellurium(IV) dibromide and aryltellurium(IV) tribromide by hydrazine hydrate leads to the corresponding asymmetrical diaryltelluride and diaryldittelluride. Structures of the new synthesized compounds are supported by FT-IR and ^{1}H NMR spectra. Anti-bacterial activity of the new products has been tested against *Klebsiella pneumonia Proteus*, *Escherichia coli*, *Pseudomonas spp.*, and *Staphylococcus aureus*. The compounds are characterized as highly active. The molecular structure level and energies of compounds have been computed through the Density Functional Theory (DFT).

Keywords: organotellurium, organomercury, antibacterial, computational study

DOI: 10.1134/S1070363220040222

INTRODUCTION

Mercury and tellurium organic compounds are characterized by exceptional nucleophilic reactivity and demonstrate high biological activity [1–5]. Organotellurium compounds exhibit strong antioxidant activity, pronounced cathepsin and caspase inhibiting, and act as anti-tumour agents [6–10]. Ammonium trichloro(dioxo ethylene-*O*,*O*)tellurate and octa-*O*-bis-(*R*,*R*)-tartrate ditellurane are used in treatment of AIDS and cancer [11–13].

Several studies were devoted to synthesis of organotellurium by organomercury compounds tansmetallation [14, 15]. Bis[2-(3-nitrobenzylideneamino)-5-nitrophenyl] telluride was synthesized and used as a ligand in complexes of Ni^{2+}, Mn^{2+} and Co^{2+} ions [16]. Schiff bases $ArTeBr_3$ and Ar_2Te (where Ar =[2-(3,4-dihydroxy benzylideneamino)-5-sulfamoylphenyl] and [2-(3,4-dihydroxybenzylideneamino)-5-acetyl phenyl]) demonstrated antioxidant and inhibitory activity against cell tumour [17].

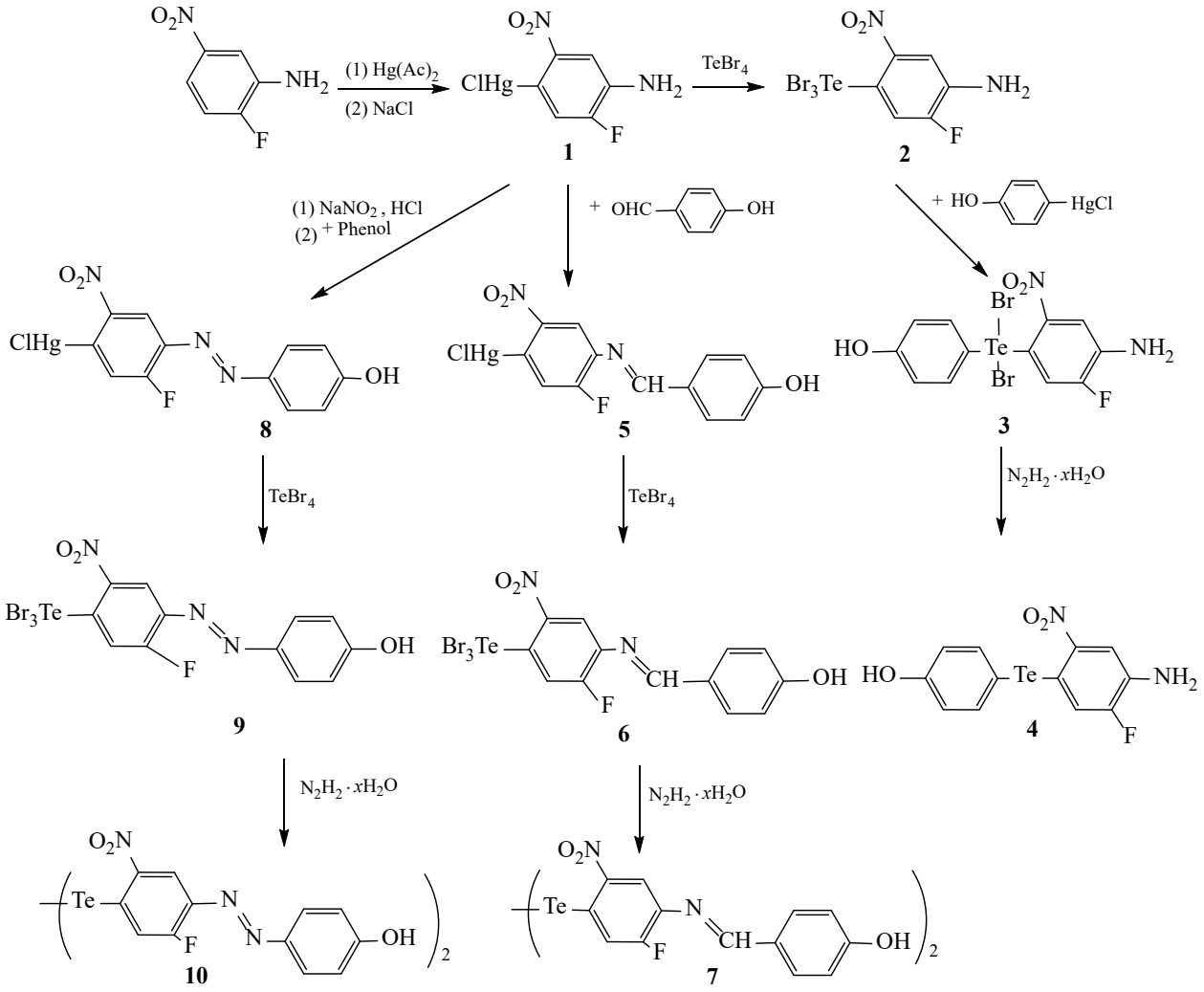
A series of chiral organotellurium compounds containing azomethine group in ortho position of tellurium atom was formed by reaction of bis(2-formylphenyl) telluride and 2-butyltelluro benzaldehyde with chiral amines (*R*)-(+)-(1-pheylethylamine) and (1*R*,2*S*)-(–)-norephedrine, respectively [18]. Azobenzene tellurated derivatives were synthesized by transmetallation of mercurated azobenzene with tellurium tetrachloride [19].

The objective of this study was synthesis of new organotellurium and organomercury compounds and their antibacterial study as well as their molecular configuration and energies study by DFT calculations.

RESULTS AND DISCUSSION

The reaction of 2-fluoro-5-nitroaniline with mercuric acetate and sodium chloride gave (4-amino-5-fluoro-2-nitrophenyl)mercury(II) chloride **1**, following transmetallation of which gave *para*-tellurated derivative **2** (Scheme 1). Reaction of compound **2** with 4-hydroxyphenyl mercury(II) chloride led to asymmetrical diaryldibromotelluride **3**. The new organomercury compound containing the azomethine group **5** was formed in the reaction of compound **1** with 4-hydroxy benzaldehyde. The new organomercury compound **8** that contained the azo group was formed in the reaction of the precursor **1** with phenol under highly acidic conditions. Aryltellurium tribromide compounds

Scheme 1. Synthetic approaches to organomercury and organotellurium compounds.



6 and **9** were derived from intermediates **5** and **8**, respectively, and tellurium tetrabromide. Asymmetrical telluride **4** and diaryl ditellurides **7** and **10** were the corresponding products of reduction of compounds **3**, **6**, and **9** by hydrazine (Scheme 1).

IR spectra of compounds **1**–**4** demonstrated two weak bands in the ranges of 3550–3355 and 3350–3330 cm^{-1} that were attributed to symmetrical and asymmetrical amino groups, accordingly. Presence of the azomethine (–C=N–) group in compounds **5**, **6**, and **7** was confirmed by the bands in the range of 1580–1570 cm^{-1}. The bands in the range of 1606–1570 cm^{-1} were attributed to the azo group (–N=N–) of compounds **8**, **9** and **10**. ^1H NMR spectra supported the structures of the synthesized compounds [20].

Antibacterial activity. Antibacterial activity of the synthesised compounds was tested against G-positive bacteria (*Staphylococcus aureus*) and four G-negative bacteria (*E. coli*, *Pseudomonas spp.*, *Klbsiella pneumonia and proteus*) (Table 1). The synthesized compounds exhibited activity against all bacterial species involved in the study. However, the highest activity was demonstrated against *E. coli*, *Klebsiella pneumoniae*, *Pseudomonas spp*. Thus, a plausible explanation of such results could be attributed to the chemical structure of bacterial cell wall's that contain key ligands that provided the receptor sites for drugs and antibiotics. This was evident from the inhibition zone values. Based on the data presented in Table 1, compounds **3**, **6**, **9**, and **10** demonstrated higher activity, induced by bromine and tellurium tetravalent atoms that influenced upon chemical reactivity and high electronegativity [21].

Computational study. According to the calculated structural parameters of the synthesized compounds

Table 1. Antibacterial activity data of the synthesized compounds

Compound	Diameter of inhibition zone (mm) for different microbial species				
	E. coli	*Pseudomonas. spp.*	*Klebsiella pneumonia*	*Staphylococcus aureus*	*Proteus spp.*
1	5	13	16	11	10
2	11	15	15	18	11
3	31	25	28	23	14
4	29	15	19	11	10
5	9	11	0	8	0
6	17	26	30	19	18
7	17	14	28	18	12
8	6	8	11	13	9
9	31	30	29	22	15
10	25	20	22	20	17
Ampicillin	30	32	38	42	19

Table 2. Some structural parameters of the studied molecules

Compound	Bonds length, Å				
	Hg–Cl	Hg–C	Te–Br	Te–C	Te–Te
1	2.43	2.25	–	–	–
2	–	–	2.68, 3.60, 5.46	2.11	–
3	–	–	2.80, 2.61	2.16, 2.28	–
4	–	–	–	2.16, 2.15	–
5	2.43	2.25	–	–	–
6	–	–	3.27, 2.89, 4.52	2.13	–
7	–	–	–	2.16, 2.16	2.79
8	2.33	2.09	–	–	–
9	–	–	2.71, 3.56, 5.43	2.09	–
10	–	–	–	2.16, 2.16	2.79
Compound	Angle, deg				
	Cl–Hg–C	Br–Te–C	Te–Te–C	C–Te–C	
1	172.29	–	–	–	
2	–	94.13, 148.18, 168.26	–	–	
3	–	90.86, 95.16, 176.27, 90.35	–	89.13	
4	–	–	–	100.90	
5	171.95	–	–	–	
6	–	105.84, 155.87, 73.81	–	–	
7	–	–	105.00, 98.91	–	
8	176.63	–	–	–	
9	–	93.87, 147.75, 168.63	–	–	
10	–	–	105.65, 97.76	–	

(Table 2), the distance (Te–C) in molecules of **2** and **9** was found to be shorter than in other structures due to π-conjugation [22]. In molecules of compounds **1**, **5**, and **8** the bonds angles of mercury atoms (C–Hg–Cl) were close to linear, 172.29°, 171.95°, and 176.63°, respectively.

The small LUMO–HOMO energy gaps calculated for compounds **3**, **9**, and **10** (Table 3) indicated their highest reactivity among all products, contrary to compounds **1**, **5**, and **8** with the highest energy gaps, hence, more stable than the other compounds. These results correlated well

Table 3. The calculated total energy, dipole moments, E_{HOMO}, E_{LUMO}, and energy gaps

Comp. no.	E_{tot}, kJ	μ, D	E_{HOMO}, eV	E_{LUMO}, eV	E_g, eV
1	–19463.164	6.618	–5.982	–3.921	2.061
2	–14925.713	5.417	–5.749	–4.223	1.526
3	–12658.468	9.180	–5.472	–4.181	1.291
4	–7510.769	2.547	–4.839	–3.222	1.617
5	–19807.233	6.430	–5.831	–3.920	1.911
6	–15269.764	9.209	–5.972	–4.490	1.482
7	–15096.517	8.985	–4.854	–3.475	1.379
8	–15643.022	7.251	–5.681	–3.928	1.753
9	–9980.732	11.699	–5.893	–4.618	1.275
10	–15128.542	8.427	–5.001	–3.785	1.216

with biological activity determined for the synthesized compounds.

Dipole moments of compounds **3**, **6**, and **9** had higher values than other compounds due to pronounced influence by Br and Te atoms, that also supported their relatively high chemical reactivity, and was consistent with our earlier study [23].

EXPERIMENTAL

All chemicals used in this study were obtained from highly regarded firms and used without further purification. IR spectra (KBr discs) were recorded on a Shimadzu model IR Affinity-1 spectrophotometer at the Chemistry Department, Education for Pure Sciences Faculty, Basrah University, Iraq. ^{1}H NMR spectra were measured on a Bruker 400 MHz spectrometer at Kashan University, Iran, using DMSO-d_6 as a solvent and TMS as an internal reference. CHN analysis was carried out on an elemental analyser at the Tarbiat Modares analytical laboratory, Tehran University, Iran.

(4-Amino-5-fluoro-2-nitrophenyl)mercury(II) chloride (1). The mixture of 2-fluoro-5-nitroaniline (20 mmol, 3.12 g) with mercuric acetate (24 mmol, 7.63 g) and ethanol (80 mL) was refluxed for 14 h. Sodium chloride (24 mmol, 1.40 g) was dissolved in boiling methanol and added to the above mixture after cooling it down, and the mixture was stirred vigorously for 1 h. The precipitate was filtered off, rinsed with water and ethanol to give pure compound **1** as white solid crystals, yield 86%, mp 236–238°C (decomp.). IR spectrum, ν, cm^{-1}: 3355 and 3350 (NH), 3228 (ArH), 1622 (C=C), 1502 and 1311 (N–O). ^{1}H NMR spectrum, δ, ppm: 5.84 s (2H, NH_2), 6.75 and 7.72 s (2H, Ar-H). Found, %: C 19.50; H 1.47; N 7.35. $C_6H_4ClFHgN_2O_2$. Calculated, %: C 18.42; H 1.03; N 7.16.

2-Fluoro-5-nitro-4-(tribromo-λ^4-tellanyl)aniline (2). A mixture of $TeBr_4$ (5.00 mmol, 2.23 g) with compound **1** (5.00 mmol, 2.60 g) and dry dioxane (40 mL) was refluxed for 6 h in the atmosphere of Ar. After cooling the mixture down, the precipitate was filtered off. The filtrate was distilled to dryness using a rotary evaporator. The product was recrystallized from MeOH – CH_2Cl_2 (2 : 1) giving the pure product **2** as brown crystals. Yield 63%, mp 165–168°C (decomp.). IR spectrum, ν, cm^{-1}: 3550 (NH), 3200, 3229 (ArH), 1614 (C=C), 1554 and 1379 (N–O). ^{1}H NMR spectrum, δ, ppm: 6.07 s (2H, NH_2), 6.56 and 7.67 s (2H, Ar-H). Found, %: C 15.0; H 1.00; N 4.79. $C_6H_4Br_3FN_2O_2Te$. Calculated, %: C 13.79; H 0.77; N 5.36.

4-[(4-Amino-5-fluoro-2-nitrophenyl)dibromo-λ^4-tellanyl]phenol (3). A mixture of 4-hydroxyphenyl mercury(II) chloride (3.00 mmol , 0.98 g) with compound **2** (3.00 mmol, 1.56 g) and 40 mL of dry 1,4-dioxane was refluxed upon stirring for 4 h in the atmosphere of argon. Then the mixture was cooled down, and the precipitate was separated. The filtrate was distilled in a rotary evaporator to dryness and recrystallized from ethanol–chloroform mixture (2 : 1) to give light brown crystals of pure compound **3**. Yield 63%, mp 187–190°C (decomp.). IR spectrum, ν, cm^{-1}: 3560 (OH), 3421 and 3330 (NH), 3089 and 3005 (ArH), 1622 (C=C), 1588 and 1341 (N–O). ^{1}H NMR spectrum, δ, ppm: 6.09 br. s (2H, NH_2), 6.59–7.44 m (6H, H-Ar), 9.36 s (1H, O–H). Found, %: C 29.56; H 2.57; N 4.75. $C_{14}H_{15}Br_2FN_2O_3Te$. Calculated, %: C 29.73; H 2.67; N 4.95.

4-[(4-Amino-5-fluoro-2-nitrophenyl)tellanyl]-phenol (4). To the mixture of compound **3** (2.00 mmol, 1.13 g) with 20 mL of ethanol was added hydrazine hydrate solution (20.00 mmol, 1.00 g) in ethanol dropwise upon refluxing till nitrogen stopped evolving. The mixture was cooled down to room temperature, the precipitate was

filtered off and recrystallized from ethanol – chloroform mixture (4 : 1) giving brown crystals of pure compound **4**. Yield 75%, mp 183–185°C (decomp.). IR spectrum, ν, cm^{-1}: 3559 (OH), 3450 and 3350 (NH), 3213 and 3074 (ArH), 1620 (C=C), 1496 and 1315 (N–O). ^1H NMR spectrum, δ, ppm: 5.95 br. s (2H, NH_2), 6.73–7.70 m (6H, H-Ar), 9.36 s (1H, O–H). Found, %: C 39.16; H 2.52; N 7.65. $C_{14}H_{15}FN_2O_3Te$. Calculated, %: C 38.35; H 2.41; N 7.45.

(*E*)-{5-Fluoro-4-[(4-hydroxybenzylidene)amino]-2-nitrophenyl}mercury(II) chloride (5). A mixture of 4-hydroxy benzaldehyde (5.00 mmol, 0.61 g) with compound **1** (5.00 mmol, 2.60 g), 30 mL of ethanol and 2–3 drops of sulphuric acid was refluxed upon stirring for 2 h. Then, it was cooled down, and the precipitate was filtered off, rinsed repeatedly with ethanol and recrystallized from a mixture ethanol–benzene (3 : 2) giving a pure product **5** as yellowish solid. Yield 82%, mp 221–223°C (decomp.). IR spectrum, ν, cm^{-1}: 3485 (OH), 1633 (C=C), 1571 (C=N), 1540 (N–O). ^1H NMR spectrum, δ, ppm: 7.05–7.23 m (6H, Ar-H), 9.28 s (1H, N=CH), 11.00 s (1H, O–H). Found, %: C 29.99; H 2.07; N 5.97. $C_{13}H_8ClFHgN_2O_3$. Calculated, %: C 31.53; H 1.63; N 5.66.

(*E*)-4-({[2-Fluoro-5-nitro-4-(tribromo-λ^4-tellanyl)-phenyl]imino}methyl phenol) (6). A mixture of tellurium tetrabromide (3.00 mmol, 1.34 g) with compound **5** (3.00 mmol, 1.48 g) in 40 mL of dry 1,4-dioxane was refluxed for 6 h in the atmosphere of argon. After cooling down the mixture, the precipitate was filtered off. The following evaporation of the filtrate in a rotary evaporator was carried out to dryness. The product was recrystallized from methanol–dichloromethane (2 : 1) mixture giving brown crystals of the pure product **6**. Yield 63%, mp 185–188°C (decomp.). IR spectrum, ν, cm^{-1}: 3445 (OH), 3199 and 3113 (ArH), 1624 (C=C), 1570 (C=N), 1516 and 1317 (N–O). ^1H NMR spectrum, δ, ppm: 7.25–7.52 m (6H, Ar-H), 9.85 s (1H, N=CH), 11.13 s (1H, O–H). Found, %: C 23.88; H 1.35; N 5.05. $C_{13}H_8Br_3FN_2O_3Te$. Calculated, %: C 24.92; H 1.29; N 4.47.

4,4'-(1*E*,1'*E*)-{[Ditellanediylbis(2-fluoro-5-nitro-4,1-phenylene)]bis(azanylylidene)bis-(methanylylidene)}diphenol (7). A solution of hydrazine hydrate (0.76 g, 15.00 mmol) in 10 mL of ethanol was added dropwise to a solution of compound **6** (2.00 mmol, 1.25 g) in 20 mL of ethanol upon refluxing. When evolving of nitrogen was over, the mixture was cooled down, and the precipitate was filtered off, washed with methanol and vacuum dried. A dark reddish-brown solid was recrystallized from ethanol to give pure product **7**. Yield 53%, mp 251–254°C (decomp.). IR spectrum, ν, cm^{-1}: 3394 (OH), 3209 (Ar-CH), 1618 (C=C), 1585 (C=N), 1511 and 1369 (N–O). ^1H NMR spectrum, δ, ppm: 7.62–8.66 m (6H, Ar–H), 9.38 s (1H, N=CH), 11.22 s (1H, O–H). Found, %: C 39.33; H 2.16; N 8.05. $C_{26}H_{14}F_2N_4O_6Te_2$. Calculated, %: C 40.37; H 2.08; N 7.24.

(*E*)-{5-Fluoro-4-[(4-hydroxyphenyl)diazenyl]-2-nitrophenyl}mercury(II) chloride (8). In an ice bath, compound **1** (5.00 mmol, 2.60 g) was dissolved in 20 mL of concentrated hydrochloric acid and the mixture was maintained at 0–5°C (first solution). Sodium nitrite (5.00 mmol, 0.34 g) was dissolved in 15 mL of cold water (second solution). Upon mixing both solutions a yellow mixture was formed. The mixture of NaOH (40%, 20 mL) with phenol was added dropwise to yellow diazonium salts solution, and the mixture was stirred for 30 min at 5°C. Then NaOH solution was added till reddish-brown solid was precipitated. The precipitate was filtered off and recrystallized from ethanol–chloroform (4 : 1) mixture to give pure compound **8**. Yield 73%, mp 231–233°C (decomp.). IR spectrum, ν, cm^{-1}: 3450 (OH), 3100 and 3050 (ArH), 1643 (C=C), 1602 (N=N), 1537 and 1388 (N–O). ^1H NMR spectrum, δ, ppm: 7.62–8.20 m (6H, Ar–H), 9.73 s (1H, O–H). Found, %: C 30.66; H 1.87; N 7.55. $C_{12}H_7ClFHgN_3O_3$. Calculated, %: C 29.04; H 1.42; N 8.47.

(*E*)-4-{[2-Fluoro-5-nitro-4-(tribromo-λ^4-tellanyl)-phenyl]diazenyl}phenol (9). The solution of tellurium tetrabromide (3.00 mmol, 1.34 g) in 40 mL of dry 1,4-dioxane was mixed with compound **8** (3.00 mmol, 1.48 g) and refluxed for 6 h under the atmosphere of argon. Upon cooling down, the white precipitate was filtered off. The filtrate was poured into 100 mL of ice-water and brown precipitate was formed. The precipitate was recrystallized from methanol–dichloromethane (2 : 1) mixture to give red-brown crystals of pure compound **9**. Yield 56%, mp 212–215°C (decomp.). IR spectrum, ν, cm^{-1}: 3412 (OH), 3062 (ArH), 1625 (C=C), 1606 (N=N), 1544 and 1359 (N–O). ^1H NMR spectrum, δ, ppm: 7.14–8.18 m (6H, Ar–H), 10.34 s (1H, O–H). Found, %: C 24.06; H 1.17; N 6.75. $C_{12}H_7Br_3FN_3O_3Te$. Calculated, %: C 22.97; H 1.12; N 6.70.

4,4'-{(1*E*,1'*E*)-[Ditellanediyl bis(2-fluoro-5-nitro-4,1-phenylene)]bis(diazene-2,1-diyl)}diphenol (10). To a solution of compound **9** (2.00 mmol, 1.25 g) in ethanol

(20 mL) was added dropwise hydrazine hydrate (0.76 g, 15.00 mmol) solution in ethanol (10 mL) upon refluxing until nitrogen stopped evolving. Upon cooling down the reaction mixture, the precipitate was filtered off and recrystallized from ethanol to give dark reddish-brown of pure product **9**. Yield 59%, mp 243–247°C (decomp.). IR spectrum, ν, cm^{-1}: 3452 (OH), 3049 (ArH), 1649 (C=C), 1570 (N=N), 1520 and 1381 (N–O). ^1H NMR spectrum, δ, ppm: 7.03–8.32 m (6H, Ar–H), 10.32 s (1H, O–H). Found, %: C 35.73; H 1.66; N 9.67. $C_{24}H_{14}F_2N_6O_6Te_2$. Calculated, %: C 37.17; H 1.82; N 10.84.

Antibacterial activity. Antibacterial activity of the synthesized compounds was tested in vitro against *Escherichia coli*, *Pseudomonas spp.*, *Staphylococcus aureus*, *Klebsiella pneumonia*, and *proteus* by the paper disc-agar diffusion method using Muller Hinton agar as a culture media. DMSO was used as a solvent. The disc-agar diffusion method was used for the solutions of 200 μg/mL. Ampicillin was used as a control. For all bacteria tested, petri plates that contained 20 ml of Muller Hinton Agar were employed. The Sterile Whatman no. 1 filter paper disks impregnated with the solution of the test compounds in DMSO were placed on the petri dishes (60 mm). Paper disks impregnated with DMSO were used as negative control. Incubation of the plates was carried out at 37°C for 24 h. Inhibition zone diameters were measured in mm.

Computational study. The density functional theory (DFT) method [24] was employed for computing the geometry and thermodynamic parameters of the synthesized compounds **1**–**10** at the PBE level theory. The standard DND basis set, Material studio-DMol3 Version 5.5 program [25], was used.

CONCLUSIONS

The transmetallation reaction between tellurium and mercury atoms can be regarded as a successful chemical approach to new organotellurium compounds. The synthesized organotellurium compounds demonstrate higher antibacterial activity than organomercury compounds. The compounds of Te(IV) are characterized by higher biological activity than Te(II) compounds, which is consistent with the theoretical studies. Based on calculated molecular energies values, organomercury compounds are characterized by higher stability than organotellurium compounds.

ACKNOWLEDGMENTS

The authors express their gratitude to the Department of Chemistry, Faculty of Education of Pure Sciences, Basrah University, Iraq for extending all facilities including laboratories and measurements of FT-IR spectra.

CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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