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Synthesis, characterization and antioxidant activity of sulfonyl-1H-1,2,3-triazoles

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Abstract

The compounds were characterized by FT-IR (Fourier Transform Infrared Spectroscopy), ^{1}H-NMR (Proton Nuclear Magnetic Resonance Spectroscopy) and ^{13}C-NMR (Carbon Nuclear Magnetic Resonance Spectroscopyspectroscopic) methods. The antioxidant properties of the compounds were evaluated using two widely accepted methodologies assays DPPH (1,1-Diphenyl-2-picrylhydrazyl radical; $C_{18}H_{12}N_5O_6$) and FRAP (Determination of Ferric Reducing Antioxidant Power). Compounds **7** and **10** emerged as the most potent antioxidant candidates, displaying the strongest effects in both assays.

Keywords: 1,2,3-triazole, organic synthesis, antioxidant activity, FRAP assay

1. Introduction

Sulfonyl compounds and 1,2,3-triazoles are important and versatile structures in the field of organic chemistry, notable for their biological activities, chemical stability, and wide range of applications. These compounds are of great significance both in pharmaceutical research and industrial chemistry. Due to their unique structures, these two classes of compounds can undergo a variety of chemical reactions and interact with biological systems. The sulfonyl group refers to a structure in which a sulfur atom is bonded to two oxygen atoms and a carbon group. The compounds containing this group can be generally represented by the formula R-SO_2-. The sulfonyl group holds a significant place in organic chemistry due to its chemical bondability and reactivity. Sulfonyl compounds are widely used in many industrial products, such as pharmaceutical molecules, polymers, agricultural chemicals, detergents, dyes, and plastics. The biological effects of sulfonyl compounds are quite diverse. These compounds can exhibit antimicrobial, antitumor, anti-inflammatory, and analgesic properties. Especially in the pharmaceutical field, drugs containing sulfonyl groups are highlighted as effective therapeutic agents due to their high affinity for specific targets. Additionally, molecules containing sulfonyl groups are being researched as potential therapeutic agents for cancer, diabetes, and HIV treatment. The interaction of sulfonyl groups with biological systems often results in the activation or inhibition of proteins, making them an important building block in drug design. Furthermore, sulfonyl compounds can also act as catalysts in chemical reactions because these compounds, with their electrophilic properties, can interact with different reactants to carry out various chemical transformations [1–12]. 1,2,3-Triazoles are known for their biological activities, with many triazole derivatives demonstrating antimicrobial, antifungal, antiviral, anticancer, and anti-inflammatory effects. 1,2,3-Triazoles are part of the most well-known class of antifungal drugs, azoles, and are thus used in the treatment of fungal infections. Moreover, numerous studies have explored the potential of triazole derivatives as therapeutic agents, particularly in the treatment of cancer and neurological diseases. Sulfonyl compounds and 1,2,3-triazoles are frequently studied in combination with one another in organic chemistry. The advantage of such combinations lies in the merging of the biological and chemical activities of both structures. These combinations can be used to create new strategies in molecular design. While the triazole ring facilitates binding to biological targets, the sulfonyl group enhances the reactivity and stability of the molecule, increasing its overall efficacy. The combination of triazole and sulfonyl compounds can also be useful in catalytic processes, as both groups serve as active building blocks for various chemical transformations [13-21].

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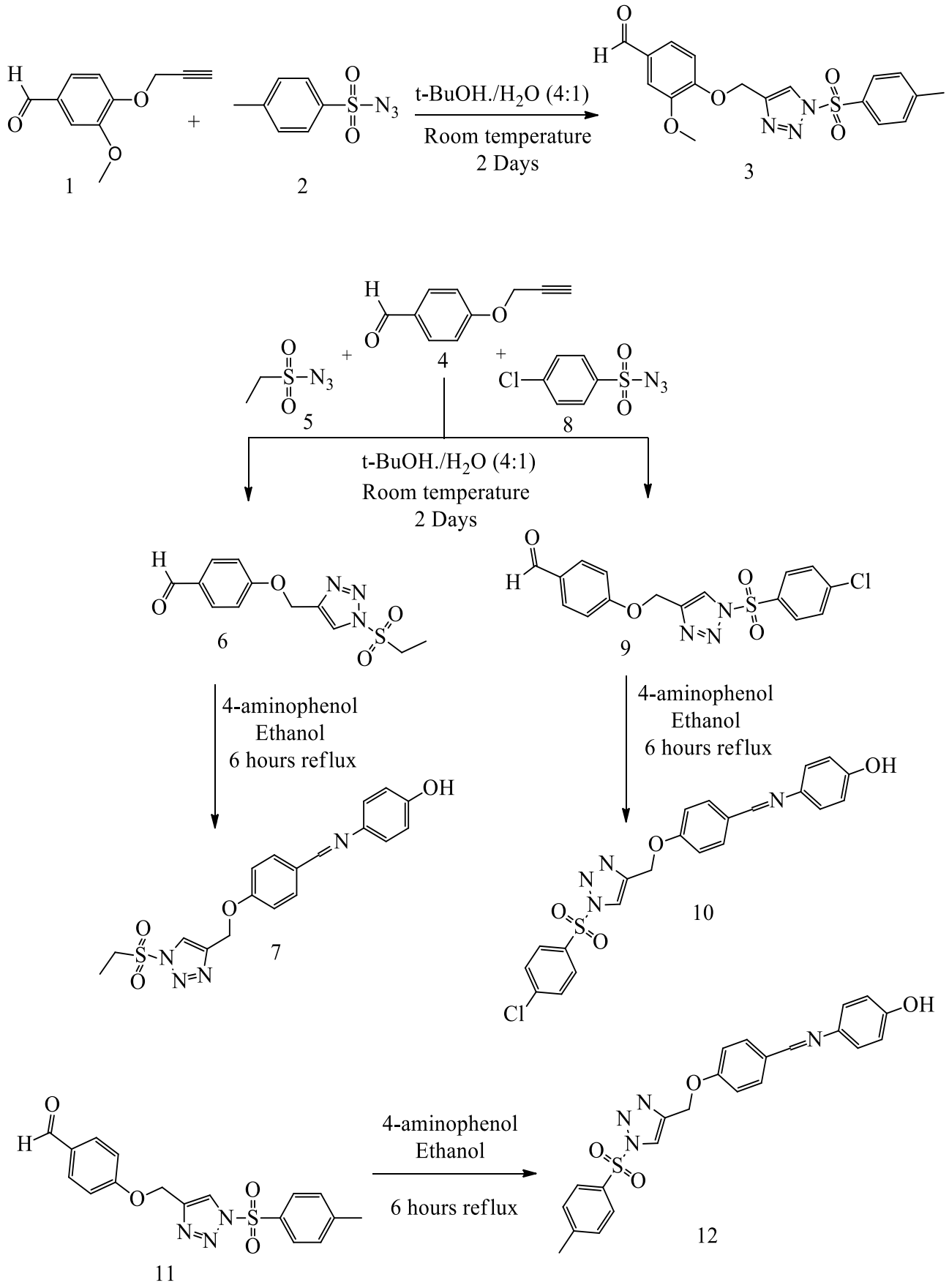

Scheme 1. Synthetic pathway for the preparation of compounds 3,6,7,9,10,12

2. Experimental

2.1. Instrumentation

IR spectra of the synthesized compounds were taken on a Perkin Elmer FT-IR 1600 FT-IR (4000-400 cm^{-1}) spectrophotometer device, and ^{1}H-NMR, ^{13}C-NMR spectra were taken on a Bruker brand 400 MHz NMR device with DMSO-d_6 (Dimethyl sulfoxide-d_6) solvent. The solvents and chemicals used in synthesis and structure elucidation were obtained from Fluka, Merck and Aldrich companies, and all solvents were subjected to appropriate purification and drying processes. Compounds **1** and **2** were synthesized literature [22,23].

2.2. Synthesis of 3-methoxy-4-((1-tosyl-1H-1,2,3-triazol-4-yl)methoxy)benzaldehyde (3):

3-Methoxy-4-(prop-2-yn-1-yloxy)benzaldehyde (1) (1 mmol) and 4-methylbenzenesulfonyl azide (2) (1 mmol) was mixed in water/t-butanol (1:4) Then copper sulfate pentahydrate %98 (1/20 mol) and sodium ascorbate %98 (1/10 mol) were added to the reaction mixture. The reaction mixture was stirred at room temperature for 2 days. At the end of the reaction the contents of the flask were poured into the ice-water mixture, the resulting solid was filtered and washed with water, crystallized with N,N-Dimethylformamide (DMF)-water (Scheme 1).

Yield: 88.02%; m.p.170-172^{o}C; IR (ν,cm^{-1}): 1742 (C=O), 1587 (C=C), 1265 (C-O),1123 (C-SO_2) ; ^{1}H-NMR (δ ppm): 2.39 (s, 3H, CH_3), 3.78 (s, 3H, OCH_3), 4.20 (s, 2H, OCH_2), Arom. [7.12 (bs, 1H, CH), 7.40 (bs, 3H, CH), 7.50 (bs, 1H, CH), 7.80 (bs, 2H, CH)], 9.84 (s, 1H,1,2,3-trz.CH), 12.26 (s,1H, HC=O); ^{13}C-NMR (δ ppm): 21.53 (CH_3), 56.06 (OCH_3), 64.14 (OCH_2), Arom. C [110.39 (CH), 126.43 (CH), 127.98 (CH), 149.58(C), 153.56(C) 169.47(C) 4-CH_3-Ph C (122.73 (CH), 130.00 (CH), 136.89 (C), 144.71 (C))], 112.73 1,2,3-trz.(CH), 130.29 1,2,3-trz.(C), 191.87 (HC=O).

2.3. Synthesis of compounds (6,9)

Compound 4-(prop-2-yn-1-yloxy)benzaldehyde (4) (1 mmol) separately with ethanesulfonyl azide (5) and 4-chlorobenzenesulfonyl azide (8) (1 mmol) water/t-butanol (1: 4) mixed in. Then, copper sulfate pentahydrate %98 (1/20 mol) and sodium ascorbate %98 (1/10 mol) were added to the reaction mixture. The reaction mixture was stirred at room temperature for 2 days. At the end of the reaction, the contents of the flask were poured into the ice-water mixture, and the solid obtained was filtered and washed with water. It was crystallized with DMF-water.

2.3.1. 4-((1-(Ethylsulfonyl)-1H-1,2,3-triazol-4-yl)methoxy)benzaldehyde (6):

Yield: 89.08%; m.p.160-162^{o}C; IR (ν,cm^{-1}): 1705 (C=O), 1600 (C=C), 1255 (C-O) ,1130 (C-SO_2) ; ^{1}H-NMR (δ ppm): 1.20 (t, 3H, CH_3) 2.85 (q, 2H,SCH_2) , 4.32 (s, 2H, OCH_2), Arom. [7.14 (d, 2H, CH), 7.88 (d, 2H, CH)], 9.88 (s, 1H,1,2,3-trz.CH), 11.81 (s,1H, HC=O); ^{13}C-NMR (δ ppm): 8.22 (CH_3), 47.01(SCH_2) , 63.98 (OCH_2), Arom. C [115,54 (CH), 132.31 (CH), 163.49 (C), 170.57(C)], 115.70 (1,2,3-trz.(CH)), 130.14 (1,2,3-trz.(C)), 191.88 (HC=O).

2.3.2. 4-((1-((4-Chlorophenyl)sulfonyl)-1H-1,2,3-triazol-4-yl)methoxy)benzaldehyde (9):

Yield: 90.12%; m.p.185-187^{o}C; IR (ν,cm^{-1}): 1729 (C=O), 1604 (C=C), 1258 (C-O) 1130 (C-SO_2); ^{1}H-NMR (δ ppm): 4.22 (s, 2H, OCH_2), Arom. [7.01 (d, 2H, CH), 7.72 (d, 2H, CH), 7.85 (d, 2H, CH), 7.94 (d, 2H, CH)], 9.96 (s, 1H,1,2,3-trz.CH), 12.14 (s,1H, HC=O); ^{13}C-NMR (δ ppm): 63.83 (OCH_2), Arom. C [115,70 (CH), 129.79 (CH), 163.51 (C), 169.73(C), 4-Cl-Ph C (129.99 (CH), 132.22 (CH), 138.52 (C), 139.15 (C))], 115.70 (1,2,3-trz.(CH)), 130.20 (1,2,3-trz.(C)), 191.96 (HC=O).

2.4. Synthesis of compounds (7,10,12)

Compounds 7,10,12 (1 mmol) were mixed separately in 4-aminophenol and ethanol a round bottom flask and refluxed for 6 hours. At the end of the reaction, the contents of the balloon were kept in the refrigerator overnight. The solid formed as a result of standing was filtered, washed with water and crystallized with DMF-water and dried.

2.4.1. 4-((4-((1-(Ethylsulfonyl)-1H-1,2,3-triazol-4-yl)methoxy)benzylidene)amino)phenol (7):

Yield: 91.25%; m.p.180-182^{o}C; IR (ν,cm^{-1}): 3221 (OH), 1577 (N=CH), 1606 (C=C), 1254 (C-O), 1138 (C-SO_2); ^{1}H-NMR (δ ppm): 1.20 (t, 3H, CH_3) 2.82 (q, 2H,SCH_2) , 4.28 (s, 2H, OCH_2), Arom. [6.79 (d, 2H, CH), 7.04 (d, 2H, CH), 7.16 (d, 2H, CH), 7.84 (d, 2H, CH)], 8.52 (s, 1H,1,2,3-trz.CH), 9.45 (s,1H, N=CH), 11.63 (s,1H, OH); ^{13}C-NMR (δ ppm): 8.24 (CH_3), 46.99 (SCH_2) , 63.60 (OCH_2), Arom. C [115.12 (CH), 132.50 (CH), 156.00 (C), 160.00(C), 4-OH-Ph C(116.12 (CH), 130.45 (CH), 143.41 (C), 170.00(C)], 122.74 (1,2,3-trz.(CH)), 130.05 (1,2,3-trz.(C)), 157.00 (N=CH).

2.4.2. 4-((4-((1-((4-Chlorophenyl)sulfonyl)-1H-1,2,3-triazol-4-yl)methoxy)benzylidene)amino) phenol (10):

Yield: 93.32%; m.p.201-203^{o}C; IR (ν,cm^{-1}): 3384 (OH), 1573 (N=CH), 1605 (C=C), 1241 (C-O), 1158 (C-SO_2); ^{1}H-NMR (δ ppm): 4.15 (s, 2H, OCH_2), Arom. [6.79 (d, 2H, CH), 6.92 (bs, 2H, CH), 7.14 (bs, 2H, CH), 7.72 (bs, 2H, CH) 7.79 (bs, 2H, CH), 7.94 (bs, 2H, CH)], 8.51 (s, 1H,1,2,3-trz.CH), 9.45 (s,1H, N=CH), 11.96 (s,1H, OH); ^{13}C-NMR (δ ppm): 63.93 (OCH_2), Arom. C [115.07 (CH), 129.97 (CH), 156.37 (C), 160.77(C), 4-OH-Ph C(116.12 (CH), 130.38 (CH), 143.38 (C), 170.09(C)), 4-Cl-Ph

C(129.74 (CH), 132.23 (CH), 138.72(C), 139.01(C))], 122.74 (1,2,3-trz.(CH)), 138.72 (1,2,3-trz.(C)), 157.00 (N=CH).

2.4.3. 4-((4-((1-Tosyl-1H-1,2,3-triazol-4-yl) methoxy) benzylidene)amino) phenol (12):

Yield: 95.41%; m.p.211-213°C; IR (ν,cm^{-1}): 3383 (OH), 1574 (N=CH), 1605 (C=C), 1241 (C-O), 1156 (C-SO_2); ^{1}H-NMR (δ ppm): 2.39 (s, 3H, CH_3) 4.36 (s, 2H, OCH_2), Arom. [6.79 (d, 2H, CH), 6.93 (bs, 2H, CH), 7.15 (bs, 2H, CH), 7.42 (bs, 2H, CH) 7.81 (bs, 4H, CH)], 8.51 (s, 1H,1,2,3-trz.CH), 9.46 (s,1H, N=CH), 12.01 (s,1H, OH); ^{13}C-NMR (δ ppm): 21.54 (CH_3), 63.57 (OCH_2), Arom. C [115.09 (CH), 128.00 (CH), 156.36 (C), 160.92(C), 4-OH-Ph C(116.12 (CH), 130.37 (CH), 144.24 (C), 169.70(C)), 4-CH_3-Ph C(129.99 (CH), 132.23 (CH), 137.20(C), 140.26(C))], 122.74 (1,2,3-trz.(CH)), 134.24 (1,2,3-trz.(C)), 157.01 (N=CH).

2.5. Antioxidant activity

The antioxidant properties of the compounds were evaluated using two widely accepted methodologies: the DPPH and FRAP assays, renowned for their effectiveness in gauging the antioxidant potential of diverse compounds. The DPPH assay, adapted from [24] measures the compounds' ability to scavenge the DPPH radical. This method relies on the decolorization of the purple DPPH solution (0.1 mM) upon interaction with antioxidants. Absorbance changes at 517 nm, recorded spectrophotometrically, indicate the degree of radical scavenging activity. Results, reported as SC_{50} values (μg of sample per mL), delineate the concentration required for a 50% reduction in the DPPH radical compared to the standard Trolox.

Concurrently, the antioxidant capacity was determined through the FRAP method, following the protocol described by [25]. This approach involves the reduction of the Fe^{3+}-TPTZ complex to the Fe^{2+}-TPTZ complex in the presence of antioxidants. Spectrophotometric readings at 593 nm after a 4-minute incubation period elucidate the compounds ability to reduce ferric ions. Results are expressed as μM Trolox equivalent per milligram of compound, where higher Trolox equivalent values denote elevated FRAP and hence increased antioxidant efficacy.

Both assays serve as robust tools for assessing the antioxidant prowess of compounds, providing valuable insights into their capacity to neutralize free radicals and reduce ferric ions. These standardized methodologies offer a comparative analysis of diverse compounds, enabling a comprehensive evaluation of their antioxidant capabilities. All experiments were conducted in three independent repetitions.

3. Results and discussion

3.1. Synthesis

Sulfonyl-1H-1,2,3-triazoles (6,7,9,10,12) 3-methoxy-4-((1-tosyl-1H-1,2,3-triazol-4-yl)methoxy)benzaldehyde (**3**), 4-((1-(ethylsulfonyl)-1H-1,2,3-triazol-4-yl) methoxy) benzaldehyde (**6**), 4-((4-((1-(ethylsulfonyl)-1H-1,2,3-triazol-4-yl) methoxy) benzylidene) amino)phenol (**7**), 4-((1-((4-chloro phenyl) sulfonyl)-1H-1,2,3-triazol-4-yl) methoxy) benzaldehyde (**9**), 4-((4-((1-((4-chlorophenyl) sulfonyl)-1H-1,2,3-triazol-4-yl) methoxy) benzylidene) amino) phenol (**10**), 4-((4-((1-tosyl-1H-1,2,3-triazol-4-yl) methoxy) benzylidene)amino) phenol (**12**) were synthesized. The compounds were characterized by FTIR, ^{1}H-NMR and ^{13}C-NMR spectroscopic methods. The most important evidence that the reaction took place is the proton and carbon data of the 1,2,3 triazole ring. C-H proton signal belonging to 1,2,3-triazole ring was seen at 8.51-9.88 ppm as a singlet in the ^{1}H NMR spectra of these compounds. Carbon peaks belonging to 1,2,3 triazole ring were observed at 115.70-138.72 ppm in the ^{13}C- NMR spectra of these compounds. Other proton and carbon data belonging to the compounds also appeared in the desired regions.

3.2. Antioxidant activity

The antioxidant properties of the analyzed compounds were assessed using the DPPH radical scavenging assay and the FRAP (Ferric Reducing Antioxidant Power) assay. The obtained results highlight significant variability in antioxidant activities among the tested compounds.

Table 1. DPPH and FRAP activities of compounds 3, 6, 7, 9, 10 and 12

Compound	DPPH (IC50: mg/mL)*	FRAP (μM Trolox Equivalent/mg compound)*
3	2,452±0,029^d	79,51±0,42^c
6	1,44±0,031^b	35,34±0,82^c
7	0,022±0,002^a	6204,82±75,87^a
9	2,125±0,022^c	25,97±0,19^c
10	0,009±0,000^a	4933,99±35,68^b
12	3,475±0,003^e	16,38±0,65^c
Trolox	0,0074±0,000^a	Not tested

*Same letters in each column were not significantly different at $P < 0.05$ (Tukey's range test). The means of three replicates were given with ± standard deviations.

The DPPH assay was employed to evaluate the free radical scavenging potential of the compounds, where a lower IC_{50} value corresponds to a higher antioxidant capacity. Among the tested compounds, compound **10** exhibited the strongest radical scavenging activity (IC_{50} = 0.009 mg/mL), followed by compound **7** (IC_{50} = 0.022 mg/mL). Both demonstrated a considerably higher efficiency in scavenging free radicals than the reference antioxidant Trolox (IC_{50} = 0.121 mg/mL).

Compound **6** (IC_{50} = 1.44 mg/mL) displayed relatively high scavenging activity, while compound **9** (IC_{50} = 2.12 mg/mL) and compound **3** (IC_{50} = 2.45 mg/mL) were in the moderate range. Compound **12** (IC_{50} = 3.47 mg/mL) was the least effective among the tested compounds in terms of radical scavenging.

The FRAP assay, which measures the capacity of compounds to reduce Fe^{3+} to Fe^{2+}, revealed that compound **7** (6204.82 μM Trolox equivalent/mg compound) had the highest reducing power, followed by compound **10** (4933.99 μM Trolox equivalent/mg compound). These results indicate that these two compounds possess strong electron-donating abilities. Compound **3** exhibited moderate reducing power (79.51 μM Trolox equivalent/mg compound), whereas compound **6** (35.34 μM), compound **9** (25.97 μM), and compound **12** (16.38 μM) demonstrated relatively weak activity.

Overall, compounds **7** and **10** emerged as the most potent antioxidant candidates, displaying the strongest effects in both assays. Compounds 6, 9, and 3 exhibited moderate antioxidant activity, while compound **12** had the weakest performance. The significantly higher antioxidant potential of compounds **7** and **10** compared to Trolox suggests that they may serve as promising antioxidant agents.

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