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Antimicrobial activity of novel isothiuronium salts with 7-chloro-4,6-dinitrobenzofuroxan-5-olate anion

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Experimental Section

All chemicals purchased from Sigma-Aldrich were reagent grade and used without purification. IR spectra (400-3700 cm^{-1}) were recorded on the PerkinElmer UATR Two FT-IR Spectrophotometer (Spectrum Two). The elemental analysis was carried out on a CHNS analyzer EuroEA3028-HT-OM (Eurovector SpA, Italy). The purity and thermal stability of crystal compounds were determined by simultaneous TG/DSC analysis on a NETZSCH STA 449C instrument (temperature range 50-450 °C, heating rate 10 deg min^{-1}, argon atmosphere).

General procedure for the synthesis of alkykisothiouronium of 7-chloro-4,6-dinitrobenzofuroxan-5-olates 2a-e. To a magnetically stirred solution of alkylisothiouronium bromide **1a-e** in anhydrous ethanol (25 ml), an equimolar amount of 5,7-dichloro-4,6-dinitrobenzofuroxan in the same solvent (25 ml) was added. The mixture was heated to reflux under nitrogen atmosphere for 2 h. The mixture was kept for 24 h at room temperature, cooled, and stored in refrigerator at -6 °C until yellow flakes consisting of single crystals appeared. The solvent was removed, and fine yellow crystals were left for aging for 48 h or more. The crystals were filtered off on a Schott filter and washed with ethanol and diethyl ether to remove unreacted initial compounds. Table S1 shows some characteristics of compounds **2a-e**.

Table S1 Physical data for the compounds **2a-e**.

Comp.	Mw ($g\ mol^{-1}$)	Yield (%)	Empirical formula	Found (calc.) (%)				mp/dec. (°C)
				C	H	N	S	
2a	492.93	83	$C_{17}H_{25}ClN_6O_7S$	41.42 (41.33)	5.11 (5.05)	17.07 (16.97)	6.50 (6.37)	141
2b	520.99	79	$C_{19}H_{29}ClN_6O_7S$	43.80 (43.32)	5.61 (5.27)	16.13 (16.32)	6.15 (5.88)	142
2c	549.04	85	$C_{21}H_{33}ClN_6O_7S$	45.94 (45.78)	6.06 (6.19)	15.31 (15.39)	5.84 (5.59)	143
2d	577.09	79	$C_{23}H_{37}ClN_6O_7S$	47.87 (47.77)	6.46 (6.39)	14.56 (14.32)	5.56 (5.52)	144
2e	605.15	87	$C_{25}H_{41}ClN_6O_7S$	49.62 (49.37)	6.83 (6.69)	13.89 (13.72)	5.30 (5.32)	146

2-Decylisothiouronium 7-chloro-4,6-dinitro-1-oxido-2,1,3-benzoxadiazol-5-olate ***2a.***

Yellow crystals, mp 141°C, yield: 83%. IR (ν/cm^{-1}): 3400-3000 (NH), 2920 - 2852 (CH), 1651 (C=N), 1606 (C=N-O), 1547 (NO_2), 1299 (NO_2), 1480 (C-S). Elemental analysis: Found, %: C 41.42, H 5.11, N 17.07, S 6.50. $C_{17}H_{25}ClN_6O_7S$. Calculated, %: C 41.33, H 5.05, N 16.97, S 6.37.

2-Dodecylisothiouronium 7-chloro-4,6-dinitro-1-oxido-2,1,3-benzoxadiazol-5-olate ***2b.***

Yellow crystals, mp 142°C, yield: 79%. IR (ν/cm^{-1}): 3400-3000 (NH), 2918-2849 (CH), 1653 (C=N), 1607 (C=N-O), 1547 (NO_2), 1299 (NO_2), 1482 (C-S). Elemental analysis: Found, %: C 43.80, H 5.61, N 16.13, S 6.15. $C_{19}H_{29}ClN_6O_7S$. Calculated, %: C 43.32, H 5.27, N 16.32, S 5.88.

2-Tetradecylisothiouronium 7-chloro-4,6-dinitro-1-oxido-2,1,3-benzoxadiazol-5-olate ***2c.***

Yellow crystals, mp 143°C, yield: 85%. IR (ν/cm^{-1}): 3400-3009 (NH), 2919-2850 (CH), 1630 (C=N), 1610 (C=N-O), 1544 (NO_2), 1299 (NO_2), 1436 (C-S). Elemental analysis: Found, %: C 45.94, H 6.06, N 15.31, S 5.84. $C_{21}H_{33}ClN_6O_7S$. Calculated, %: C 45.78, H 6.19, N 15.39, S 5.59.

2-Hexadecylisothiouronium 7-chloro-4,6-dinitro-1-oxido-2,1,3-benzoxadiazol-5-olate ***2d.***

Yellow crystals, mp 144°C, yield: 79%. IR 3450-3050 (NH), 2919-2849 (CH), 1654 (C=N), 1607 (C=N-O), 1547 (NO_2), 1229 (NO_2), 1482 (C-S). Elemental analysis: Found, %: C 47.87, H 6.46, N 14.56, S 5.56. $C_{23}H_{37}ClN_6O_7S$. Calculated, %: C 47.77, H 6.39, N 14.32, S 5.52.

2-Octadecylisothiouronium 7-chloro-4,6-dinitro-1-oxido-2,1,3-benzoxadiazol-5-olate ***2e.***

Yellow crystals, mp 146°C, yield: 87%. IR (ν/cm^{-1}): 3320-3050 (NH), 2917-2849 (CH), 1614 (C=N), 1595 (C=N-O), 1542 (NO_2), 1282 (NO_2), 1487 (C-S). Elemental analysis: Found, %: C 49.62, H 6.83, N 13.89, S 5.30. $C_{25}H_{41}ClN_6O_7S$. Calculated, %: C 49.37, H 6.69, N 13.72, S 5.32.

X-Ray Crystallography.

‡*Crystal Data for* **2d**. $C_{23}H_{37}ClN_6O_7S$ (M = 577.09), triclinic, space group $P\bar{1}$ at 100.00(10) K: a = 5.24790(10), b = 11.1856(2) and c = 24.7646(4) Å, α = 93.506(2)°, β = 94.7520(10)°, γ = 103.048(2)°, V = 1406.52(4) Å^3, Z = 2, d_{calc} = 1.363 g cm^{-3}, μ(CuKα) = 2.342 mm^{-1}, F(000)=612.0. Total of 16150 reflections were collected (5666 independent reflections, R_{int} = 0.0470) and used in the refinement, which converged to wR_2 = 0.1292, GOOF = 1.038 for all independent reflections [R_1 = 0.0457 was calculated for 5666 reflections with $I > 2\sigma(I)$].

Data set for single crystals **2d** was collected on a Rigaku XtaLab Synergy S instrument with a HyPix detector and a PhotonJet microfocus X-ray tube using Cu Kα (1.54184 Å) radiation at 100 K. Images were indexed and integrated using the CrysAlisPro data reduction package. Data were corrected for systematic errors and absorption using the ABSPACK module. The GRAL module was used for analysis of systematic absences and space group determination. Using Olex2 [S1], structure was solved by direct methods with SHELXT [S2] and refined by the full-matrix least-

squares on F^2 using SHELXL [3]. Non-hydrogen atoms were refined anisotropically. The figures were generated using Mercury 4.1 [S4] program.

CCDC 1990614 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/structures.

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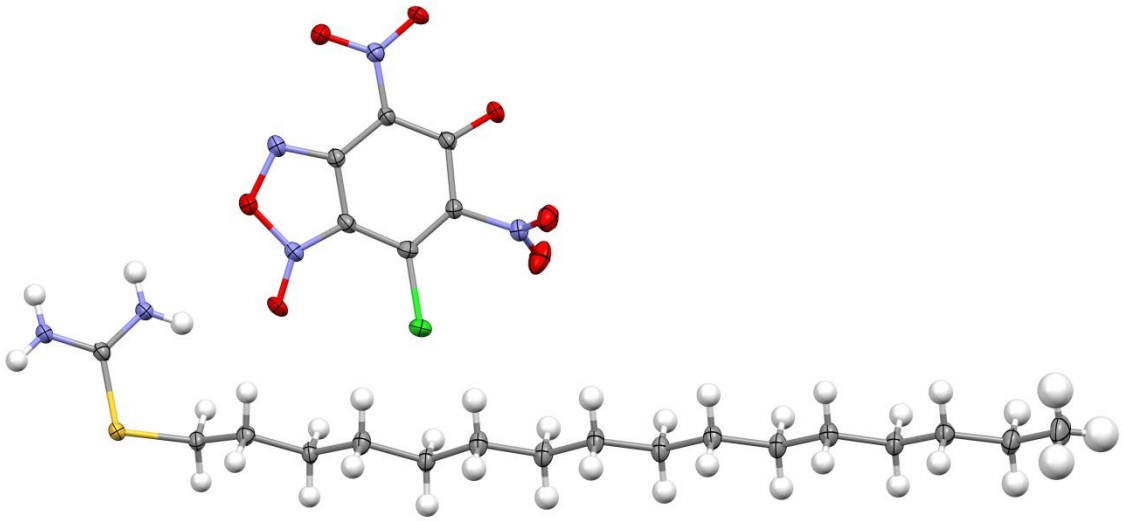

Figure S1 Crystal structure of compound **2d** according to the X-ray diffraction data.

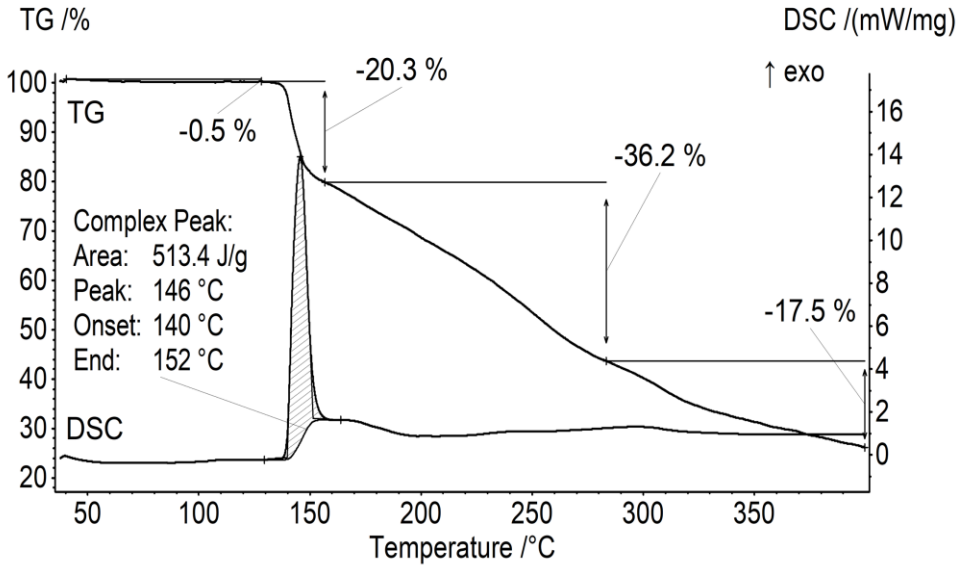

Figure S2 Results of TG/DSC analysis of compound **2e** in the dynamic argon atmosphere 75 ml min^{-1} in the temperature range 40-400 °C. Heating rate is 10 K min^{-1}.

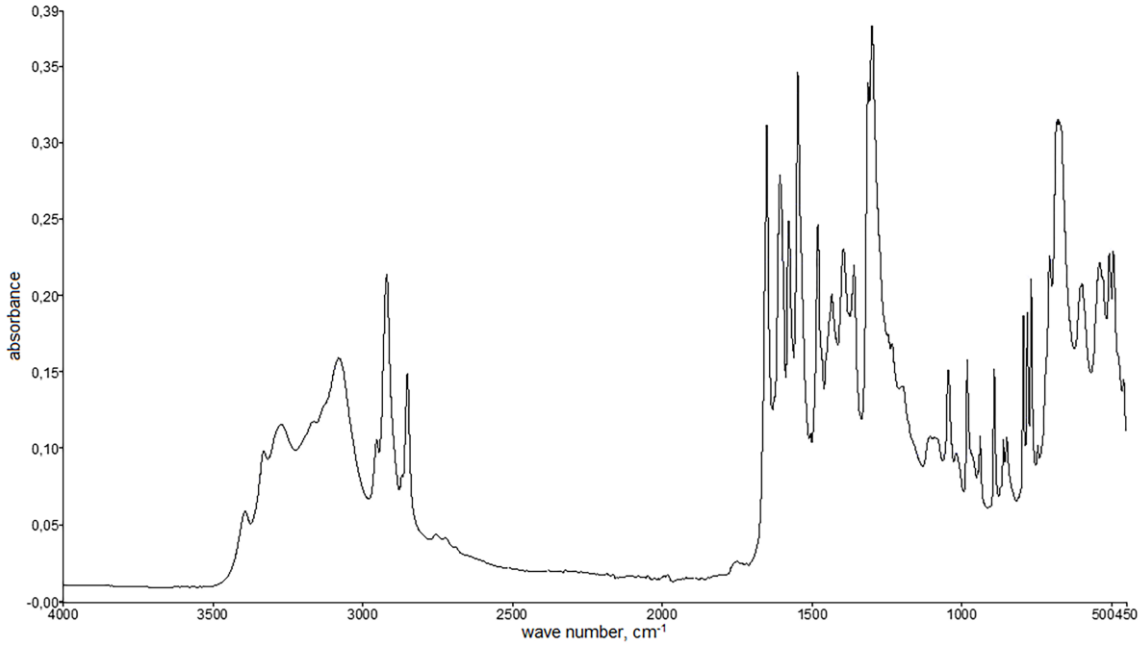

Figure S3 IR spectrum of compound **2a**.