Supporting information

Synthesis and Evaluation of Troponoids as a New Class of Antibiotics

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Keywords: Troponoids, antibacterial, Gram-positive, Gram-negative, multi-drug resistance

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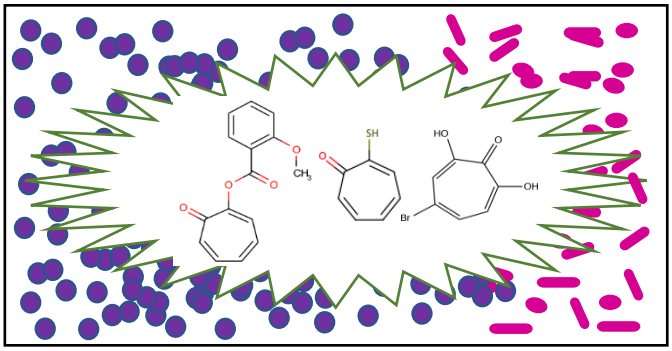


I. Table S1. Troponoids tested for primary and quantitative anti-bacterial screening and CC_{50} results

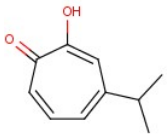
Compound Number	MIC_{80} (μM)				CC_{50} (μM)
	S.aureus (ATCC 29213)	*E.coli* (ATCC35218)	*A.banmannii* (from a patient)	*P. aeruginosa* ATCC 27853	
46	19.8	44.4	100	>71.4	25
47	66.7	44.4	51.2	>71.4	66.4
48	66.7	58.1	100	>71.4	53.4
49	55.6	87.1	>100	>71.4	13.8
50	100	>71.4	>71.4	>71.4	78.6
51	16.5	>100	>71.4	>71.4	>100
52	>100	>71.4	>71.4	>71.4	>100
53	11.7	>100	>71.4	>71.4	>100
54	16.1	66.7	44.4	>71.4	95.6
55	44.4	46.9	>71.4	>71.4	4.2
56	44.4	66.7	>71.4	>71.4	35
57	>100	>71.4	>71.4	>71.4	57
60	>100	>71.4	>71.4	>71.4	
61	>100	>71.4	>71.4	>71.4	30.8
62	19.8	>100	83.4	>71.4	98.9
63	13.2	100	83.4	>100	99.5
106	66.7	>100	>71.4	>71.4	38
107	>100	>71.4	>71.4	>71.4	72.4
108	>100	>71.4	>71.4	>71.4	112.5
109	100	>71.4	>71.4	>71.4	93
110	100	71.4	66.7	>71.4	32
111	66.7	66.7	100	>71.4	35.5
112	66.7	>71.4	>71.4	>71.4	79
113	33.3	>100	71.4	>71.4	47
114	19.8	>71.4	71.4	>71.4	38.3
118	>100	>71.4	>71.4	>71.4	17.2
120	19.8	44.4	100	>71.4	42.4
143	100	>71.4	>71.4	>71.4	>100
144	>100	>71.4	>71.4	>71.4	>100
145	29.6	>71.4	>71.4	>71.4	6.3
146	19.8	>71.4	>71.4	>71.4	28.9
147	44.4	>71.4	>71.4	>71.4	86.8
172	66.7	66.7	>71.4	>100	95
173	44.4	>71.4	>71.4	71.4	76.6
195	>100	>71.4	>71.4	>71.4	92

210	>71.4	>71.4	71.4	>71.4	71
261	17.6	19.8	29.6	>71.4	58
262	19.8	66.7	100	>71.4	57.5
264	100	>71.4	>71.4	>71.4	20.5
265	29.6	44.4	44.4	>71.4	25.5
266	66.7	>71.4	>71.4	>71.4	20
267	44.4	>71.4	>71.4	>71.4	16.9
269	>100	>71.4	>71.4	>71.4	>100
270	44.4	66.7	100	>71.4	12.2
271	>100	>71.4	>71.4	>71.4	36.3
272	44.4	44.4	29.6	>71.4	10.1
273	44.4	>71.4	>71.4	>71.4	>100
274	44.4	>71.4	>71.4	>71.4	>100
280	>100	>71.4	>71.4	>71.4	88.2
281	65.8	>100	>71.4	>71.4	>100
282	18.2	>100	>71.4	>71.4	96.5
283	15.4	>100	>71.4	>71.4	94.6
284	13.2	24.1	55.6	>71.4	91
285	8.8	100	44.4	>71.4	>100
308	44.4	24.7	29.6	>71.4	25.8
309	44.4	>71.4	71.4	>71.4	40
310		29.6	29.6	>71.4	72.6
311	66.7	29.6	44.4	>71.4	31.7
312		71.4	71.4	>71.4	27.8
313	66.7	>71.4	>71.4	>71.4	45.4
314	66.7	>71.4	>71.4	>71.4	20
315	29.6	>71.4	>71.4	>71.4	28.5
316	29.6	>71.4	>71.4	>71.4	>100
318	>100	>71.4	>71.4	>71.4	100
319	>100	>71.4	>71.4	>71.4	50
335	100	>71.4	>71.4	>71.4	78.4
336	44.4	>71.4	100	>71.4	39.5
338	19.8	>71.4	>71.4	>71.4	>100
339	48.2	>100	>71.4	>71.4	>100
340	>100	>71.4	>71.4	>71.4	20
341	44.4	>71.4	>71.4	>71.4	17.1
342	100	>71.4	>71.4	>71.4	35.5
343	>100	>71.4	>71.4	>71.4	0.3
344	29.6	>71.4	>71.4	>71.4	1.6
345	>100	>71.4	>71.4	>71.4	100
346	>100	>71.4	>71.4	>71.4	

347	>100	>71.4	>71.4	>71.4	100
348	8.8	100	44.4	>71.4	100
349	83.4	>71.4	>71.4	>71.4	53.5
350	8.8	100	>71.4	>71.4	46
363	29.6	14.8	>100	>71.4	86
364	13.2	19.8	>71.4	>71.4	66.7
365	>100	>71.4	>71.4	>71.4	
677	100	19.8	>71.4	>71.4	8.6
678	>100	19.8	>71.4	>71.4	33.8
679	>100	>100	>71.4	>71.4	100
680	>100	19.8	>71.4	>71.4	95.7
681	>100	19.8	>71.4	>71.4	22.8
682	>100	66.7	>71.4	>71.4	56.1
684	>100	13.2	>71.4	>71.4	20.8
685	>100	19.8	>71.4	>71.4	9.6
686	>100	>100	>71.4	>71.4	42.6

II. Figure S1. Structures of compounds

Tropolones



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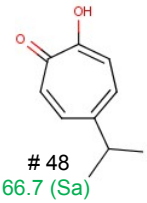
44.4 (Ec)
51.2 (Ab)



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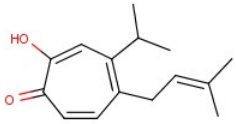

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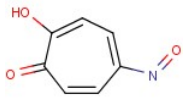

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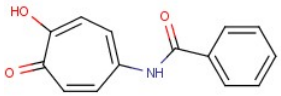

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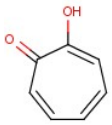

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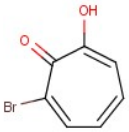

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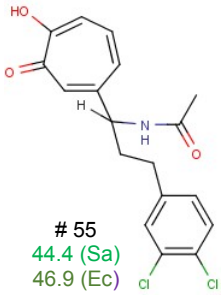

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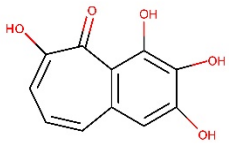

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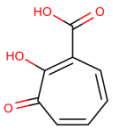

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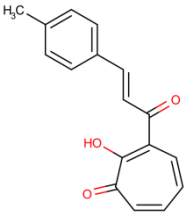

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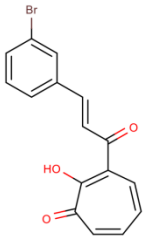

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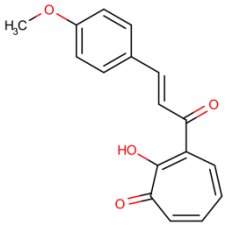

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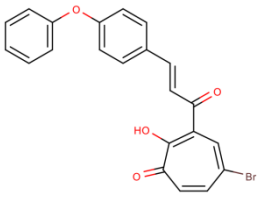

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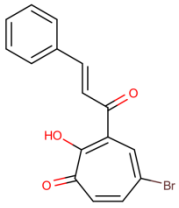

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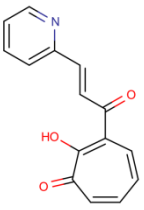

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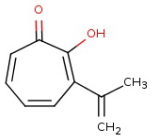

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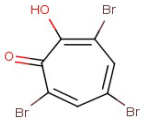

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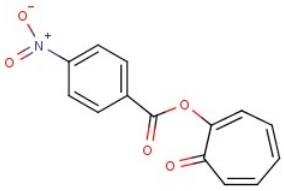
Tropones (1)



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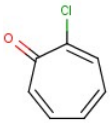

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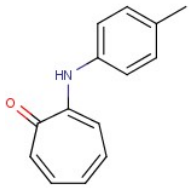

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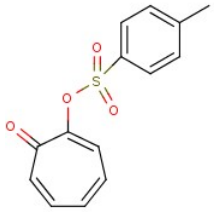

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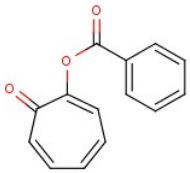

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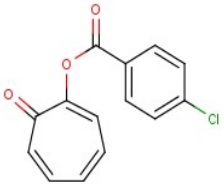

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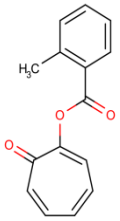

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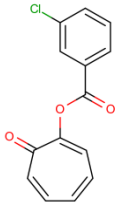

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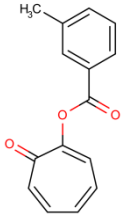

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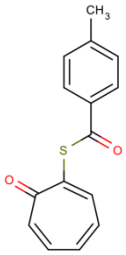

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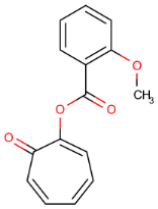

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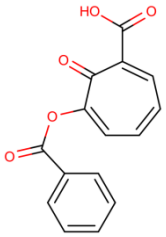

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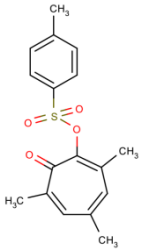

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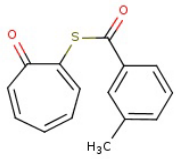

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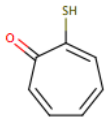

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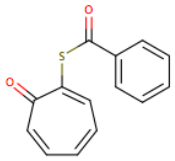

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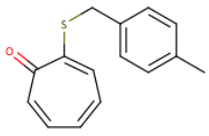

#365
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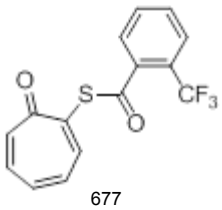
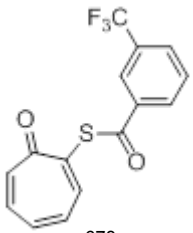
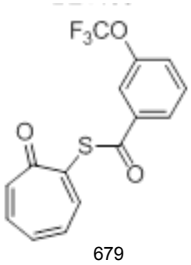


677
19.8 µM (Ec)

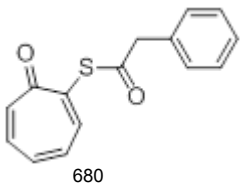


678
19.8 µM (Ec)

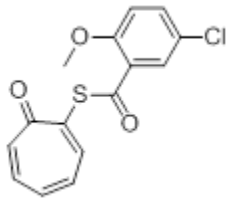


679
>100 µM (Ec)

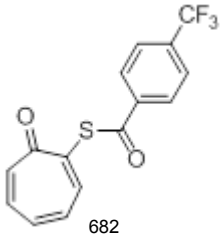
Tropones (2)



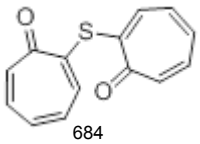
680
19.8 μM (Ec)



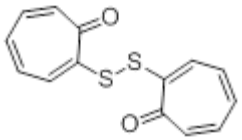
681
19.8 μM (Ec)



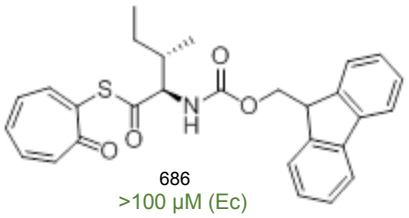
682
66.7 μM (Ec)



684
13.2 μM (Ec)

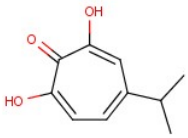


685
19.8 μM (Ec)

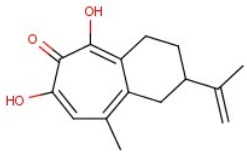


686
>100 μM (Ec)

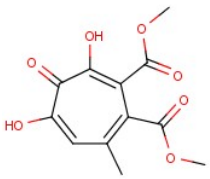
Alpha-hydroxytropolones (1)



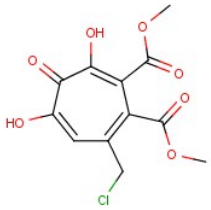
46
19.8 (Sa)
44.4 (Ec)
100 (Ab)



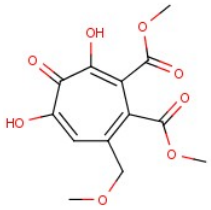
56
44.4 (Sa)
>100 (Ec)



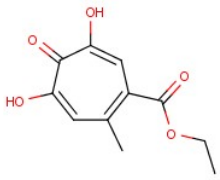
106
66.7 (Sa)
>100 (Ec)



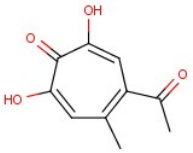
107
>100 (Sa)



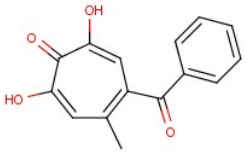
108
>100 (Sa)



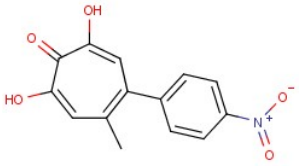
109
100 (Sa)



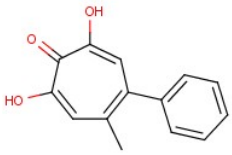
110
100 (Sa)
66.7 (Ab)



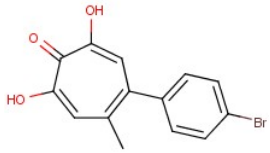
111
66.7 (Sa)
66.7 (Ec)
>100 (Ab)



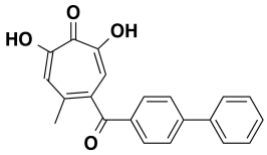
112
66.7 (Sa)
100 (Ec)



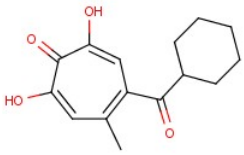
113
33.3 (Sa)
>100 (Ec)



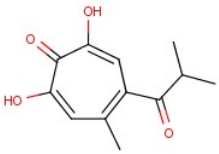
114
19.8 (Sa)



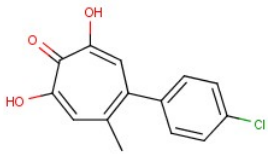
#118
>100 (Sa)



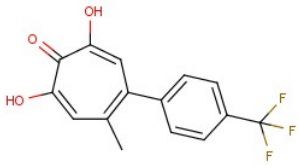
120
19.8 (Sa)
44.4 (Ec)
83.4 (Ab)



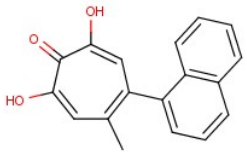
143
100 (Sa)



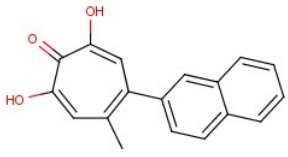
144
>100 (Sa)



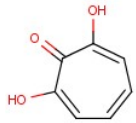
145
29.6 (Sa)



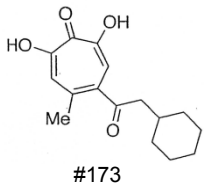
146
19.8 (Sa)



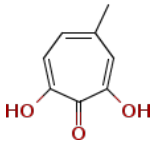
147
44.4 (Sa)



172
66.7 (Sa)
66.7 (Ec)

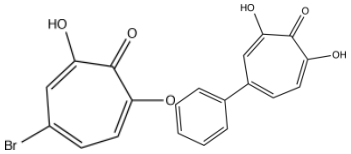


#173
44.4 (Sa)



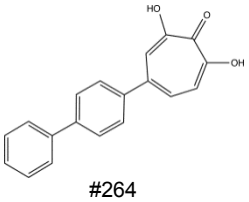
#210
>71.4 (Ss)

Alpha-hydroxytropolones (2)

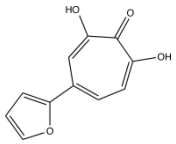


#261
17.6 (Sa)
19.8 (Ec)
29.6 (Ab)

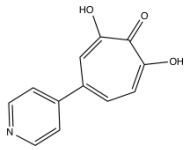
#262
19.8 (Sa)
66.7 (Ec)
100 (Ab)



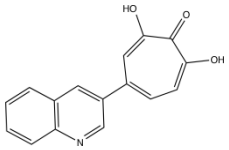
#264
100 (Sa)
44.4 (Ec)



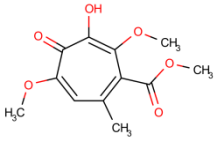
#265
29.6 (Sa)
44.4 (Ec)
44.4 (Ab)



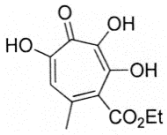
#266
66.7 (Sa)



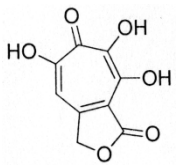
#267
44.4 (Sa)



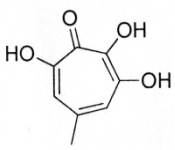
#269
>100 (Sa)



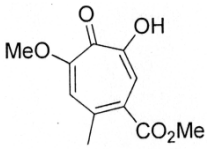
#270
44.4 (Sa)
66.7 (Ec)
100 (Ab)



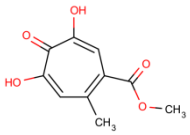
#271
>100 (Sa)



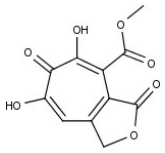
#272
44.4 (Ss)
44.4 (Ec)
29.6 (Ab)



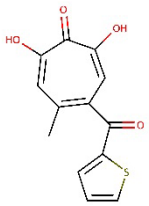
#273
44.4 (Sa)



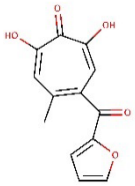
#274
44.4 (Sa)



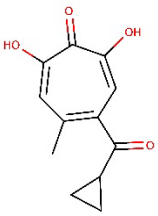
#280
>100 (Sa)
>100 (Ec)



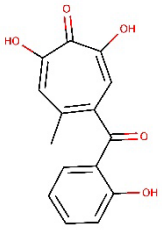
#308
44.4 (Sa)
24.7 (Ec)
32.1 (Ab)



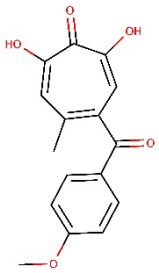
#309
44.4 (Sa)



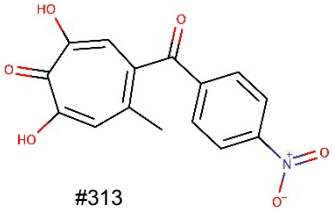
#310
29.6 (Ec)
29.6 (Ab)



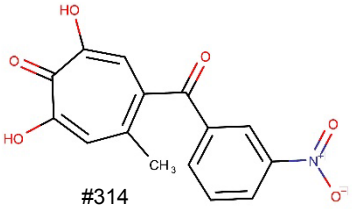
#311
66.7 (Sa)
29.6 (Ec)
44.4 (Ab)



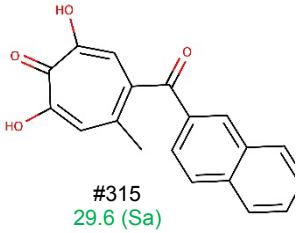
#312



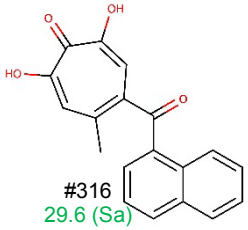
#313
66.7 (Sa)



#314
66.7 (Sa)



#315
29.6 (Sa)



#316
29.6 (Sa)

Alpha-hydroxytropolones (3)

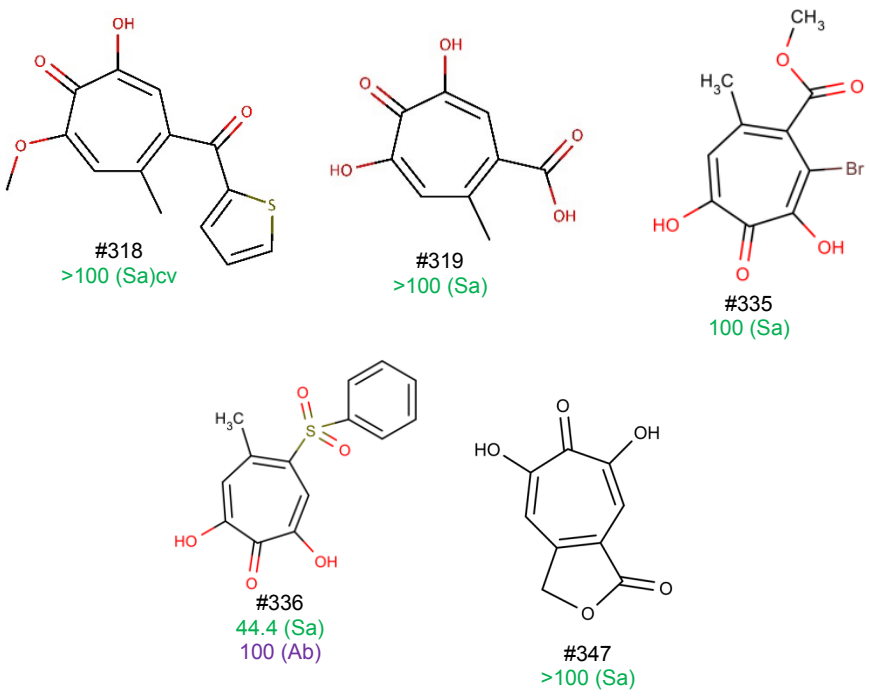
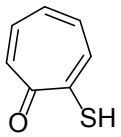


Figure S1. Structures of compounds. Part of the MIC_{80} value is listed on the bottom of each compound and the target organisms are listed in the parenthesis as abbreviations: *S. aureus* (Sa), *E. coli* (Ec) and *A. baumannii* (Ab).

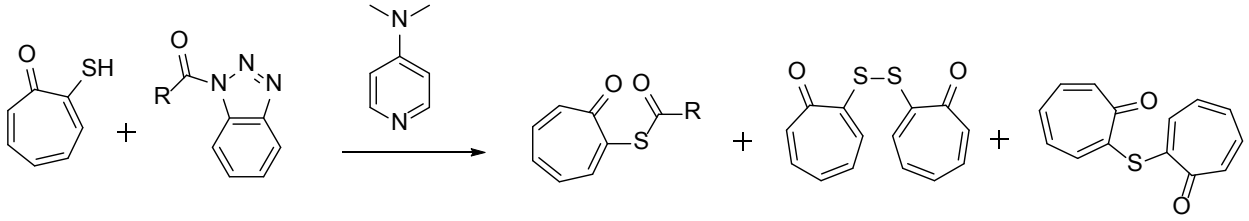
III. Synthesis and Characterization of compound 675-682, 684-686

2-Mercaptocyclohepta-2,4,6-trien-1-one (675)



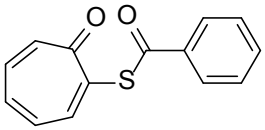
NaSH (4.0 g, 73 mmol) was added portion wise to a solution of **687** (4.0 g, 15 mmol) in ethanol (50 mL). The reaction mixture was stirred for 2 hr, the reaction was monitored by LCMS and TLC till completion. Ethanol was evaporated, and the resulting solid was dissolved in water and acidified with HCl (2 M) then extracted with DCM (3 × 50 mL) to yield the desired product (BE1100) together with its dimer. purification was performed by flash chromatography using mixture of Hexans/EtOAc to obtain the desired compound as an orange crystal (80%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.02 (s, 1H), 8.57 (d, J = 10.4 Hz, 1H), 7.68 – 7.55 (m, 2H), 7.53 – 7.41 (m, 2H).; ^{13}C NMR (100 MHz, $CDCl_3$) δ 183.3, 173.3, 144.6, 139.9, 135.7, 134.9, 121.2; LC/MS m/z: 139 [M+H$^+$].

General procedure for the synthesis of compounds 676-682 and 684-686



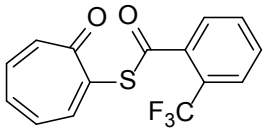
Acyl benzotriazole derivatives (0.5 mmol) was added to a solution of thiotropolone (0.525 mmol) and DMAP (0.5 mmol) in acetonitrile (5 mL). The reaction mixture was stirred for 1 hr, the reaction was monitored by LCMS and TLC till completion. The reaction mixture was concentrated under vacuo. The residue was dissolved in EtOAc (30 mL) and extracted with Na_2CO_3 solution (3 × 20 mL). The organic layer was dried over Na_2SO_4, filtered and the solvent was evaporated in vaccuo. The crude reaction mixture was purified by flash chromatography using Hexans/EtOAc to give the desired products **(676-678, 680-682,** and **686)** in addition to two side products (**684** and **685**) in a pure form.

S-(7-Oxocyclohepta-1,3,5-trien-1-yl) benzothioate (676)



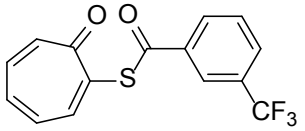
Yellow solid (39%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, J = 7.8 Hz, 2H), 7.86 (d, J = 8.2 Hz, 1H), 7.52 (t, J = 7.4 Hz, 1H), 7.39 (t, J = 7.5 Hz, 2H), 7.16 – 6.91 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 188.2, 183.6, 145.0, 142.6, 139.5, 136.5, 135.5, 135.4, 134.00, 132.4, 128.8, 127.7; LC/MS m/z: 265 [M+Na$^+$].

S-(7-Oxocyclohepta-1,3,5-trien-1-yl) 2-(trifluoromethyl)benzothioate (677)



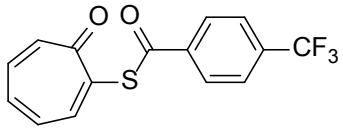
Brownish yellow solid (30%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.00 (d, J = 6.8 Hz, 1H), 7.94 (d, J = 6.1 Hz, 1H), 7.79 (d, J = 6.2 Hz, 1H), 7.73-7.61 (m, 2H), 7.35-7.07 (m, 4H).; ^{13}C NMR (100 MHz, Chloroform-*d*) δ 189.4, 183.2, 144.8, 142.1, 139.7, 137.1, 135.7, 135.7, 132.6, 131.9, 131.6, 129.1, 127.0, 124.6, 121.8; LC/MS m/z: 333 [M + Na$^+$]. HRMS (ESI$^+$): Anal. Calcd for [M + Na$^+$]: $C_{15}H_9F_3O_2SNa^+$ 333.0168; Found 333.0165.

_S_-(7-Oxocyclohepta-1,3,5-trien-1-yl) 3-(trifluoromethyl)benzothioate (678)



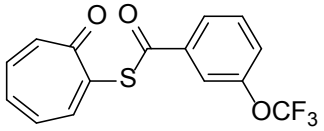
Brownish yellow solid (31%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.29 (s, 1H), 8.24 (d, J = 7.9 Hz, 1H), 7.97 (d, J = 8.9 Hz, 1H), 7.88 (d, J = 7.6 Hz, 1H), 7.65 (t, J = 8.0 Hz, 1H), 7.28 – 7.05 (m, 4H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 187.2, 183.3, 144.3, 143.00, 139.7, 137.1, 135.9, 135.7, 132.4, 131.3, 130.9, 130.3, 129.5, 129.2, 124.6; LC/MS m/z: 333 [M + Na$^+$]. HRMS (ESI$^+$): Anal. Calcd for [M + Na$^+$]: $C_{15}H_9F_3O_2SNa^+$ 333.0168; Found 333.0165.

_S_-(7-Oxocyclohepta-1,3,5-trien-1-yl) 4-(trifluoromethyl)benzothioate (682)



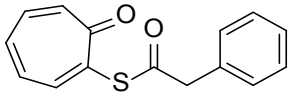
Yellow solid (33%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.17 (d, J = 7.9 Hz, 2H), 7.97 (d, J = 8.6 Hz, 1H), 7.78 (d, J = 7.9 Hz, 2H), 7.30 – 7.04 (m, 4H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 187.5, 183.3, 144.3, 142.8, 139.8, 139.3, 135.8, 135.5, 132.3, 128.1, 125.9; LC/MS m/z: 333 [M + Na$^+$]. HRMS (ESI$^+$): Anal. Calcd for [M + Na$^+$]: $C_{15}H_9F_3O_2SNa^+$ 333.0168; Found 333.0166.

_S_-(7-Oxocyclohepta-1,3,5-trien-1-yl) 3-(trifluoromethoxy) benzothioate (679)



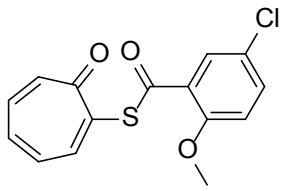
Brownish yellow solid (33%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.07 (d, J = 7.7 Hz, 1H), 7.98 (s, 1H), 7.64 – 7.46 (m, 4H), 7.27 – 7.00 (m, 3H). LC/MS m/z: 349 [M+Na$^+$]. HRMS (ESI$^+$): Anal. Calcd for [M_2 + Na$^+$]: $(C_{15}H_9F_3O_3S)_2Na^+$ 675.0341; Found 675.0337.

_S_-(7-Oxocyclohepta-1,3,5-trien-1-yl) 2-phenylethanethioate (680)



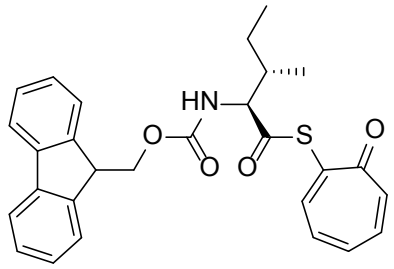
Brown solid (36%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.71 (d, J = 8.6 Hz, 1H), 7.28-7.18 (m, 5H), 7.08 – 6.84 (m, 4H), 3.87 (s, 2H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 193.5, 183.4, 145.2, 141.7, 139.4, 135.6, 135.3, 132.9, 132.5, 129.7, 128.8, 127.7, 50.7; LC/MS m/z: 279 [M + Na$^+$]. HRMS (ESI$^+$): Anal. Calcd for [M + Na$^+$]: $C_{15}H_{12}O_2SNa^+$ 279.0450; Found 279.0449.

S-(7-Oxocyclohepta-1,3,5-trien-1-yl) 5-chloro-2-methoxybenzothioate (681)



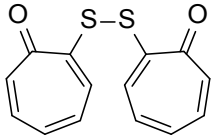
Yellow solid (32%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.89 (t, J = 8.4 Hz, 1H), 7.79 (s, 1H), 7.42 (d, J = 8.7 Hz, 1H), 7.19 – 6.92 (m, 6H), 3.92 (s, 3H). ^{13}C NMR (100 MHz, Chloroform-*d*) δ 186.1, 183.7, 156.9, 145.3, 142.7, 139.7, 135.5, 135.4, 133.9, 132.4, 129.6, 128.6, 125.8, 113.5, 56.2; LC/MS m/z: 328.9 [M + Na^+]. HRMS (ESI^+): Anal. Calcd for [M + Na^+]: $C_{15}H_{11}ClO_3SNa^+$ 329.0009; Found 329.0006.

***S*-(7-Oxocyclohepta-1,3,5-trien-1-yl) (2S,3S)-2-((((9H-fluoren-9-yl)methoxy)carbonyl)-amino)-3-methylpentanethioate (686)**



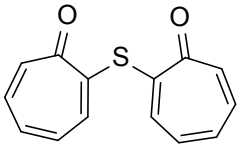
Brown oil (32%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.68 (d, J = 7.7 Hz, 3H), 7.55 (t, J = 6.8 Hz, 2H), 7.31 (t, J = 7.4 Hz, 3H), 7.23 (t, J = 7.6 Hz, 2H), 7.12 – 6.85 (m, 4H), 5.33 (d, J = 9.5 Hz, 1H), 4.47 – 4.28 (m, 3H), 4.18 (t, J = 7.0 Hz, 1H), 2.10 – 1.95 (m, 1H), 1.59 – 1.44 (m, 1H), 1.17 – 1.07 (m, 1H), 0.96 (d, J = 6.8 Hz, 3H), 0.87 (t, J = 7.2 Hz, 3H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 197.2, 183.3, 156.2, 144.7, 143.8, 143.6, 142.8, 141.4, 139.7, 135.6, 132.4, 127.8, 127.1, 125.1, 120.0, 67.3, 65.9, 47.2, 37.8, 24.4, 15.8, 11.7; LC/MS m/z: 496.1 [M + Na^+]. HRMS (ESI^+): Anal. Calcd for [M + Na^+]: $C_{28}H_{27}NO_4SNa^+$ 496.1553; Found 496.1553.

2,2'-Disulfanediylbis(cyclohepta-2,4,6-trien-1-one) (685)



Yellow solid (12-20%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 (d, J = 8.3 Hz, 2H), 7.34 – 7.26 (m, 2H), 7.12 – 6.97 (m, 6H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 184.3, 153.2, 137.4, 136.2, 133.0, 132.5, 129.7. LC/MS m/z: 275 [M + H^+] and 296.9 [M + Na^+]. HRMS (ESI^+): Anal. Calcd for [M + Na^+]: $C_{14}H_{10}O_2S_2Na^+$ 297.0014; Found 297.0013.

2,2'-Thiobis(cyclohepta-2,4,6-trien-1-one) (684)



Canary yellow solid (15-20%). ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.40 (d, J = 8.9 Hz, 2H), 7.20 – 7.13 (m, 2H), 7.12 – 7.02 (m, 2H), 7.01 – 6.85 (m, 4H); ^{13}C NMR (100 MHz, Chloroform-*d*) δ 182.9, 152.2, 138.0, 136.4, 135.7, 133.6, 132.5. LC/MS m/z: 243 [M + H^+] and 265 [M + Na^+]. HRMS (ESI$^+$): Anal. Calcd for [M + Na^+]: $C_{14}H_{10}O_2SNa^+$ 265.0294; Found 265.0292.