One-Pot Synthesis and Characterization of Some New Types of 5,5′-Disubstituted Bis(imidazolidine-2,4-diones)

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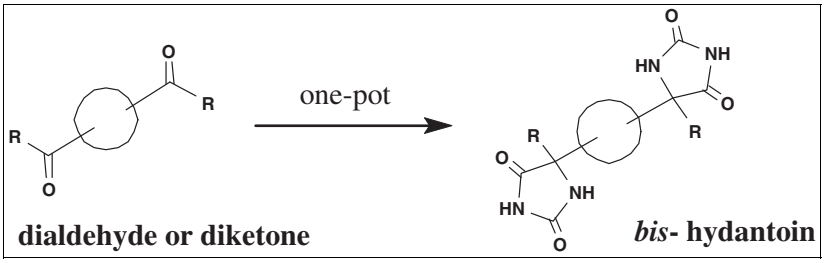

The synthesis and structural elucidation of some novel 5,5′-disubstituted spiro and nonspiro-bis-hydantoins are reported. The Bucherer Burge's method has been modified for the preparation of some 5,5′-substituted bis(imidazolidine-2,4-dione) derivatives starting with diketones (**1–5**) and dialdehydes (**6**, **7**). In some cases, diastereoisomeric mixtures of compounds were obtained. The resulting bis-hydantoins (**8–11**, **13**, **14**) have not to our knowledge been previously reported in the literature.

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INTRODUCTION

Drug discovery continues to be an important area of investigation for synthetic chemist. The bis-hydantoin compounds, as potential bis-drugs, are expected to be capable of double therapeutic behavior. Some pharmacological and therapeutic properties of bis-hydantoins have been reported [1–9]. For example, recently there has been a growing concern about how to reduce or eliminate infections completely, especially those caused by antibiotic-resistant bacterial strains. Some *N*-halomine derivatives of bis-hydantoins possessing excellent biocidal activity against a wide range of microorganisms including fungi, bacteria, viruses, and yeasts have been designed [4]. Other application of bis-hydantoin derivatives include their use as flame retardants for a variety of polymeric systems, as intermediates for further transformations [10, 11] and as precursors to diaminodicarboxylic acid derivatives [15]. Some bis-imidazolidineiminothiones exhibit cytotoxic activity against various tumor cell lines as well as antiviral, antimicrobial, and antifungal properties [2]. Also the 1,3,10,13-tetraza-dispiro[4.2.4.2] tetradecane-2,4,10,12-tetraone **12** (a bis-hydantoin) has been found to function as a bis-intercalator in recognizing specific DNA sequences [7].

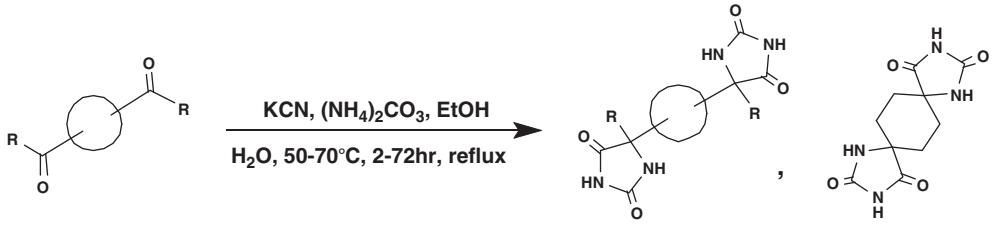
Since a wide range of bis-heterocyclic compounds have been reported to exhibit superior antibacterial activities when compared to their mono-heterocyclic counterparts [2], in this study we decided to prepare some novel bis-hydantoins via one-pot experimental protocol, developed from that used in our previous works [12, 13]. Structures of all synthesized compounds in this study were elucidated by the use of TLC, melting point, IR, and ^{1}H-NMR. Bis-hydantoins were also analyzed by ^{13}C-NMR and mass spectromertry.

RESULTS AND DISCUSSION

A numbers of new and older synthetic methods have been reported for the preparation of mono-hydantoins [1, 12–14, 16–19]. One such general method is the Bucherer–Bergs synthesis of 5,5-disubstituted hydantoins from either aldehydes or ketones through the action of potassium cyanide and ammonium carbonate. Our work the Bucherer–Bergs procedure was modified to permit its use for the one-pot synthesis of some novel 5,5′-disubstituted bis(imidazolidine-2,4-dione) derivatives, as depicted Schemes 1 and 2.

Diketones **1–5** and dialdehydes **6, 7** (Fig. 1) upon reaction with $(NH_4)_2CO_3$ and KCN in a mixture of 50% (v/v) EtOH/H_2O solvent, led to the formation of several new bis(imidazolidine-2,4-diones) (Fig. 2 and Table 1). As we reported in our previous work [13], α-diketones when subjected to similar reaction conditions fragmented to mono-hydantoins. Therefore, to overcome this challenge to prepare bis-analogues, we replaced α-diketones with several different diketones containing alkyl, *o*-alkyl, indole-alkyl and aryl linkages. The IR, ^{1}H-NMR, and ^{13}C-NMR spectral data of the products confirmed the structures of the proposed bis-hydantoins. Spectral data for bis-hydantoin products showed typical symmetry

Scheme 1. Synthesis of bis-hydantoins.



1-7 **8-11,13,14** **12**

of structure in most cases. For example, the ^{1}H-NMR spectrum of **13** was consistent with a perfecty symmetrical structure (e.g. the spectrum contained four signals corresponding to 10 protons). However, in the remaining cases additional peaks in the spectra may be due to a subtle conformational phenomenon. For example, the spectrum of compound **12** exhibited the presence of a conformer. The NMR spectra of the bis-hydantoins **8–11**, with two asymmetric carbon atoms in their structures, showed a mixture of the *dl* and *meso* isomers (approximate ratio 85:15).

CONCLUSION

By modification of the Bucherer–Bergs reaction, several novel bis-hydantoins were successfully designed and synthesized in one-pot fashion. Diastereoisomeric mixtures of products were obtained in some cases. The bis-hydantoins (**8**–**11**, **13**, **14**) prepared have not been previously described.

EXPERIMENTAL

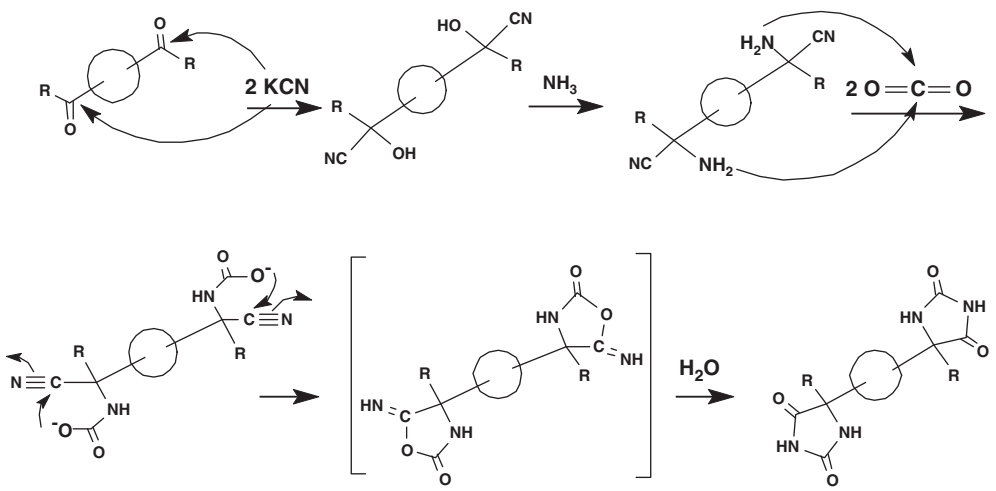
General remarks. High-resolution ^{1}H-NMR (500 MHz) and ^{13}C-NMR (125 MHz) spectra were obtained with a Bruker 500 DRX-Avance NMR spectrometer. Compounds were dissolved in deuterated DMSO as NMR solvent. IR data were obtained with a Shimadzu 470 spectrometer. Melting points of crystalline compounds were measured with an electrothermal melting point apparatus and have not been corrected. Mass spectra were obtained using a GC-MS Agilent Technologies QP-5973N MSD instrument. Purification of crystalline compounds was performed by recrystallization and in some cases by preparative thin-layer chromatography with silica gel 60 GF_{254}. All chemicals were purchased from Aldrich Chemical Company, Merck or Fluka.

Typical reaction procedures and spectroscopic data for bis-hidantoins and their precursor are described below.

Typical experimental procedure for synthesis of diketones 1, 2. 1-(4-hydroxyphenyl)ethanone (4-hydroxyacetophenone) (5.12 g, 37.6 mmol) was dissolved in ethanol (50 mL) and KOH (3.2 g, 80 mmol) was added. The mixture was equipped with a reflux condenser and refluxed at 50–60°C for 15 min. Then 1,4-dibromobutane (2.12 mL, 18 mmol) or 1,6-dibromohexane (2.73 mL, 18 mmol) was added to the reaction mixture (for preparing **1** and **2,** respectively) and the mixture refluxed for 18 h. The reaction was cooled to room temperature and resulting precipitate was filtered and recrystallized from ethanol.

1,1′-((Butane-1,4-diylbis(oxy))bis(4,1-phenylene)) diethanone (1). White powder crystals. m.p. (recrystallized from ethanol) 140°C. yield 50%, 2.96 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.90 (p, 2 H, 2 × CH_aH_b), 2.49 (s, 3 H, 2 × CH_3), 2.50 (DMSO), 3.31 (H_2O), 4.13 (t, 2 H, 2 × CH_aH_bO), 7.03 (d, J = 8.81 Hz, 2 H, 4 × CH, Ar), 7.91 (d, J = 8.81 Hz, 2 H, 4 × CH, Ar) ppm. IR (KBr,): ν_{max} = 1250

Scheme 2. Schematic reaction mechanism.



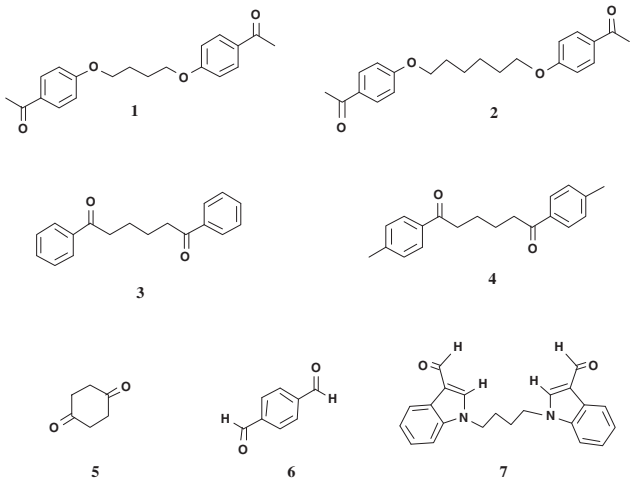


Figure 1. Structure of diketones (**1–5**) and dialdehydes (**6,7**).

(C—O), 1600 (C═C), 1675 (C═O), 2850–2950 (Ali H), 3050 (Ar H) cm^{-1}.

1,1′-((Hexane-1,6-diylbis(oxy))bis(4,1-phenylene))diethanone (2). White powder crystals. m.p. (recrystallized from ethanol) 141°C. yield 99%, 6.36 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.47 (P, J = 6.94 Hz, 2 H, 2 × CH_aH_b), 1.74 (p, J = 6.38, 6.35 Hz, 2 H, 2 × CH_aH_b), 2.49 (DMSO), 2.50 (s, 3 H, 2 × CH_3), 3.32 (H_2O), 4.05 (t, J = 6.46 Hz, 2 H, 2 × CH_aH_bO), 7.00 (d, J = 8.85 Hz, 2 H, 4 × CH, Ar), 7.90 (d, J = 8.84 Hz, 2 H, 4 × CH, Ar) ppm. IR (KBr): ν_{max} = 1250 (C—O), 1600 (C═C), 1667 (C═O), 2850–2930 (Ali H), 3050 (Ar H) cm^{-1}.

Typical experimental procedure for synthesis of diketones 3, 4. A mixture of anhydrous Aluminum chloride (1.67 g, 12.5 mmol) and benzene (used in excess as reactant and solvent, 6 mL) was placed in a three-necked flask equipped with a stirrer, a reflux condenser, and a dropping funnel. With rapid stirring, adipoyl chloride (0.73 mL, 5 mmol) was added through the dropping funnel at an even rate during 15 min and then the mixture refluxed under 50–65°C for 2 h. The solution was poured slowly, with constant stirring, into a mixture of cracked ice. The residual liquid was filtered using a Buchner funnel and the solid washed with cool diethylether (20 mL) and recrystallized from ethanol. Toluene (6 mL) instead of benzene was used for the synthesis of **4**.

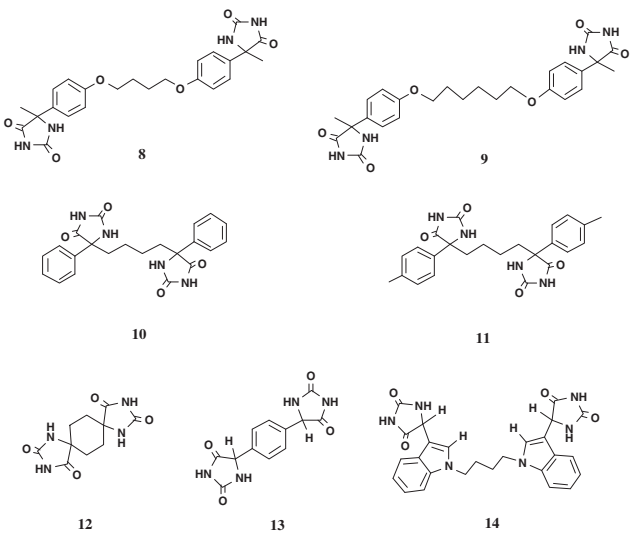


Figure 2. Structure of synthesized bis-hydantoin derivatives.

Table 1

Reaction time and yields of produced *bis*-hydantoins.

Entry	*Bis*-hydantoin product	Time (h)	Yield (%)	M.P. (°C)
1	**8**	24	40	$200_{dec.}$
2	**9**	72	35	$220_{dec.}$
3	**10**	48	77	$180_{dec.}$
4	**11**	72	41	136
5	**12**	3	63	$>290_{dec.}$
6	**13**	24	86	$295_{dec.}$
7	**14**	72	20	120

1,6-Diphenylhexane-1,6-dione (3). White powder crystals. m.p. (recrystallized from ethanol) 106°C. yield 50%, 0.66 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.68 (P, J = 3.53, 2.87, 3.82 Hz, 2 H, 2 × CH_aH_b), 2.49 (DMSO), 3.07 (t, J = 6.5 Hz, 2 H, 2 × CHaH_bCO), 3.32 (H_2O), 7.51 (t, J = 7.75 Hz, 2 H, 4 × CH, Ar), 7.62 (t, J = 7.39 Hz, 1 H, 2 × CH, Ar), 7.97 (dd, J = 8.15, 1.2 Hz, 2 H, 4 × CH, Ar) ppm. IR (KBr): ν_{max} = 1590 (C═C), 1680 (C═O), 2860-2930 (Ali H), 3050 (Ar H) cm^{-1}.

1,6-Di-p-tolylhexane-1,6-dione (4). White powder crystals. m.p. (recrystallized from ethanol) 132°C. yield 60%, 0.88 g. ^{1}H-NMR (500 MHz, DMSO-d_6): δ = 1.66 (P, J = 2.79, 2.60, 3.73 Hz, 2 H, 2 × CH_aH_b), 2.36 (s, 3 H, 2 ×CH_3), 2.49 (DMSO), 3.02 (t, 2 H, 2 × CH_aH_bCO), 3.31 (H_2O), 7.31 (d, J = 7.95 Hz, 2 H, 4 × CH, Ar), 7.86 (d, J = 8.07 Hz, 2 H, 4 × CH, Ar) ppm. IR (KBr): ν_{max} = 1600 (C═C), 1670 (C═O), 2850–2930 (Ali H), 3050 (Ar H) cm^{-1}.

Experimental procedure for synthesis of dialdehyde 7. This compound was prepared in our laboratory according to the literature procedure [20].

Typical experimental procedure for synthesis of bis-hydantoins 8–14. CAUTION: HCN may be liberated; wear gloves and work in hood. Dialdehyde or diketone (**1–7**) (8 mmol) was dissolved in 50 mL of a 1:1 (v/v) mixture of ethanol and water in a 100-mL round-bottom flask equipped with a stirrer and reflux condenser, and potassium cyanide (2.16 g, 33 mmol) and ammonium carbonate (6.38 g, 66 mmol) were added . The mixture was allowed to reflux at 50–70°C with constant stirring for 2–72 h. The solution was allowed to cool to room temperature, acidified to pH = 2 by drop-wise addition of concentrated HCl with constant stirring and the resulting precipitate after isolation by filtration was recrystallized from ethanol. All products were solids.

5,5′-((butane-1,4-diylbis(oxy))bis(4,1-phenylene))bis(5-methylimidazolidine-2,4-dione) (8). White powder crystals. m.p. (recrystallized from ethanol) 200°$C_{dec.}$. yield 40%, 1.5 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.60 (s, 3 H, 2 ×CH_3), 1.87 (p, J = 3.18, 3.01 Hz, 2 H, 2 × CH_aH_b), 2.49 (DMSO), 3.31 (H_2O), 4.03 (t, J = 5.89 Hz, 1 H, 2 × CH_aO), 4.12 (t, J = 5.8, 3.7 Hz, 1 H, 2 × CH_bO), 6.94 (d, J = 8.87 Hz, 1 H, 2 × CH, Ar), 7.03 (d, J = 6.89 Hz, 1 H, 2 × CH, Ar), 7.35 (d, J = 8.83 Hz, 1 H,

2 × C*H*, Ar), 7.91 (d, *J* = 8.83 Hz, 1 H, 2 × C*H*, Ar), 8.50 (w s, N_1H *meso*), 8.52 (s, 0.5 H, 2 × N_1H *dl*), 10.67 (br s, 2 × N_3H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 21.99 (2 × CH_3), 24.24 (2 × CH_2), 40.23 (DMSO), 65.52 (2 × CH_2O), 75.30 (2 × C_5, ring), 120.12 (4 × *C*H, Ar), 129.19 (4 × *C*H, Ar), 136.33 (2 × *C*, Ar), 155.74 (2 × *C*, Ar), 157.79 (2 × *C*═O, urea), 173.33 (2 × *C*═O) ppm. IR (KBr): ν_{max} = 1250 (C—O), 1600 (C═C), 1718 (asym C═O), 1760 (sym C═O), 2850–2950 (Ali H), 3050 (Ar H), 3150 (w N_3H), 3250 (N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{24}H_{26}N_4O_6$, 466.1852; found 466.1856.

5,5′-((Hexane-1,6-diylbis(oxy))bis(4,1-phenylene))bis(5-methylimidazolidine-2,4-dione) (9). White powder crystals. m.p. (recrystallized from ethanol) 220°C$_{dec.}$. yield 35%, 1.4 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.46 (p, *J* = 3.97, 3.79 Hz, 2 H, 2 × CH_aH_b), 1.59 (s, 3 H, CH_3), 1.64 (s, 3 H, CH_3), 1.73 (p, *J* = 6.63, 7.19, 6.77 Hz, 2 H, 2 × CH_aH_b), 2.49 (DMSO), 3.34 (H_2O), 3.95 (t, *J* = 6.49 Hz, 1 H, 2 × CH_aO), 4.06 (t, *J* = 6.48 Hz, 1 H, 2 × CH_bO), 6.92 (d, *J* = 8.81 Hz, 1 H, 2 × C*H*, Ar), 7.01 (d, *J* = 8.81 Hz, 1 H, 2 × C*H*, Ar), 7.28 (d, *J* = 5.38 Hz, 1 H, 2 × C*H*, Ar), 7.91 (d, *J* = 8.78 Hz, 1 H, 2 × C*H*, Ar), [8.48 (w s, 2 × N_1H *meso*), 8.49 (s, 2 × N_1H *dl*), 1 H], 8.90 (w s, 2 × N_3H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 25.19 (2 × CH_3), 25.81 (2 × CH_2), 29.21 (2 × CH_2), 40.31 (DMSO), 67.30 (2 × CH_2O), 70.11 (2 × C_5, ring), 115.33 (4 × *C*H, Ar), 124.41 (4 × *C*H, Ar), 134.09 (2 × *C*, Ar), 156.16 (2 × *C*, Ar), 159.49 (2 × *C*═O, urea), 179.81 (2 × *C*═O) ppm. IR (KBr): ν_{max} = 1250 (C—O), 1600 (C═C), 1718(asym C═O), 1760 (sym C═O), 2850-2930 (Ali H), 3040 (Ar H), 3150 (w N_3H), 3250 (N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{26}H_{30}N_4O_6$, 494.2165; found 494.2167.

5,5′-(Butane-1,4-diyl)bis(5-phenylimidazolidine-2,4-dione) (10). White powder crystals. m.p. (recrystallized from ethanol) 180° C$_{dec.}$. yield 77%, 2.50 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.19 (m, 2 H, CH_aH_b), 1.68 (m, 2 H, CH_aH_b), 1.84 (m, 1 H, CH_a), 2.01 (m, 1 H, CH_b), 2.49 (DMSO), 3.07 (m, 2 H, CH_aH_b), 3.33 (H_2O),7.30 (t, *J* = 6.93 Hz, 1 H, 1 × C*H*, Ar), 7.37 (t, *J* = 7.38 Hz, 2 H, 2 × C*H*, Ar), 7.47-7.53 (m, *J* = 8.22, 2.45, 8.36, 7.48 Hz, 4 H, 4 × C*H*, Ar), 7.62 (t, *J* = 7.07 Hz, 1 H, 1 × C*H*, Ar), 7.96 (t, *J* = 10.17 Hz, 2 H, 2 × C*H*, Ar), 8.60 (s, 1 H, 2 × N_1H *dl*), 8.64 (w s, 2 × N_1H *meso*), 10.74 (s, 1 H, 2 × N_3H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 24.24 (2 × CH_2), 38.63 (2 × CH_2), 40.40 (DMSO), 68.30 (2 × C_5, ring), 126.20 (1 × *C*H, Ar), 128.61 (1 × *C*H, Ar), 128.75 (2 × *C*H, Ar), 129.28 (2 × *C*H, Ar), 129.55 (2 × *C*H, Ar), 133.89 (2 × *C*H, Ar), 137.59 (1 × *C*, Ar), 140.05 (1 × *C*, Ar), 157.39 (2 × *C*═O, urea), 177.04 (2 × *C*═O), 200.87(solvent) ppm. IR (KBr): ν_{max} = 1590 (C═C), 1715(asym C═O), 1765 (sym C═O), 2850-2920 (Ali H), 3040 (Ar H), 3150 (sh N_3H), 3235 (N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{22}H_{22}N_4O_4$, 406.1641; found 406.1638.

5,5′-(Butane-1,4-diyl)bis(5-(p-tolyl)imidazolidine-2,4-dione) (11). Cream powder crystals. m.p. (recrystallized from ethanol) 136°C. yield 41%, 1.44 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.85 (m, 1 H, CH_a), 1.98 (m, 1 H, CH_b), 2.17 (t, *J* = 7.19 Hz, 2 H, CH_aH_b), 2.23 (t, *J* = 7.21 Hz, 2 H, CH_aH_b), 2.27 (s, 3 H, CH_3), 2.36 (s, 3 H, CH_3), 2.49 (DMSO), 2.98 (t, *J* = 7.21 Hz, 2 H, CH_aH_b), 3.32 (H_2O), 7.18 (d, *J* = 7.98 Hz, 2 H, 2 × C*H*, Ar), 7.31 (d, *J* = 7.91 Hz, 2 H, 2 × C*H*, Ar), 7.36 (d, *J* = 8.09 Hz, 2 H, 2 × C*H*, Ar), 7.86 (d, *J* = 8.01 Hz, 2 H, 2 × C*H*, Ar), 8.54 (w s, 2 × N_1H *meso*), 8.58 (s, 1 H, 2 × N_1H *dl*), 10.70 (br s, 1 H, 2 × N_3H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 21.40 (CH_3), 21.98 (CH_3), 24.31 (CH_2), 25.06 (CH_2), 34.54 (CH_2), 38.36 (CH_2), 40.39 (DMSO), 68.15 (2 × C_5, ring), 126.10 (4 × *C*H, Ar), 128.87 (4 × *C*H, Ar), 129.82 (2 × *C*H, Ar), 130.09 (2 × *C*, Ar), 135.11 (2 × *C*═O, urea), 157.38 (2 × *C*═O) ppm. IR (KBr): ν_{max} = 1600 (C═C), 1700(asym C═O), 1715 (sym C═O), 2850-2950 (Ali H), 3050 (Ar H), 3150 (w N_3H), 3250 (N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{24}H_{26}N_4O_4$, 434.1954; found 434.1957.

Spirobis-hydantoin (12). Cream powder crystals. m.p. (recrystallized from ethanol) >290°C$_{dec.}$. yield 63%, 1.26 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.59 (d, *J* = 9.02 Hz, 2 H, 4 × CH_a), 1.63–1.67 (m, CH_a minor stereoisomer), 1.95 (d, *J* = 9.34 Hz, 2 H, 4 × CH_b), 2.05-2.09 (m, CH_b minor stereoisomer), 2.49 (DMSO), 3.33 (H_2O), 8.17 (w s, 2 × N_1H minor stereoisomer), 8.48 (s, 1 H, 2 × N_1H), 10.56 (br s, 1 H, 2 × N_3H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 27.81 (4 × CH_2), 40.36 (DMSO), 60.15 (2 × C_5, ring), 155.23 (2 × *C*═O, urea), 176.22 (2 × *C*═O) ppm. IR (KBr): ν_{max} = 1575 (C═C), 1730 (asym C═O), 1770 (sym C═O), 2750–3050 (Ali H), 3170 (sh N_3H), 3260 (N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{10}H_{12}N_4O_4$, 252.0859; found 252.0856.

5,5′-(1,4-Phenylene)bis(imidazolidine-2,4-dione) (13). Cream powder crystals; m.p. (recrystallized from ethanol) 295°C$_{dec.}$. yield 86%, 1.88 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 2.49 (DMSO), 3.34 (H_2O), 5.17 (s, 1 H, 2 × CH_5CON), 7.35 (s, 2 H, 4 × C*H*, Ar), 8.40 (s, 1 H, 2 × N_1H), 10.79 (s, 1 H, 2 × N_3H); ^{13}C NMR (125 MHz, DMSO-d_6): δ = 40.36 (DMSO), 61.80 (2 × C_5, ring), 127.90 (4 × *C*H, Ar), 136.95 (2 × *C*, Ar), 158.35 (2 × *C*═O, urea), 174.93 (2 × *C*═O) ppm. IR (KBr): ν_{max} = 1515 (C═C), 1700 (asym C═O), 1780 (sym C═O), 2755 (CH_5CON), 3000 (Ar H), 3200 (N_3H), 3300 (N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{12}H_{10}N_4O_4$, 274.0702; found 274.0706.

5,5′-(1,1′-(Butane-1,4-diyl)bis(1H-indole-3,1-diyl))bis(imidazolidine-2,4-dione) (14). Beige powder crystals. m.p. (recrystallized from ethanol) 120°C. Yield 20%, 0.75 g. ^{1}H NMR (500 MHz, DMSO-d_6): δ = 1.82 (m, 2 H, 2 × CH_aH_b), 2.49 (DMSO), 3.31 (H_2O), 4.31 (m, 2 H, 2 × CH_aH_bN), 5.37 (s, 1 H, 2 × CH_5CON), 7.23–7.30 (m, *J* = 7.22, 7.48, 7.73, 7.93 Hz, 2 H, 4 × C*H*, Ar), 7.61 (d, *J* = 8.04 Hz, 1 H, 2 × C*H*, Ar), 7.80 (s, 1 H, C*H*, olefin), 8.10 (d, *J* = 7.54 Hz, 1 H, 2 × C*H*, Ar), 8.29 (s, 1 H, 2 × N_1H), 9.88 (s, 1 H, 2 × N_3H) ppm. ^{13}C NMR (125 MHz, DMSO-d_6): δ = 26.36 (2 × CH_2), 40.39 (DMSO), 53.20 (2 × CH_2N), 61.90 (2 × C_5H, ring), 107.88 (2 × *C*H, Ar), 111.78 (2 × *C*, pyrrolic), 115.11 (2 × *C*H, Ar), 118.23 (2 × *C*H, Ar), 120.08 (2 × *C*H, Ar), 124.52 (2 × *C*, Ar), 128.08 (2 × *C*H, pyrrolic), 136.82 (2 × *C*, Ar), 155.15 (2 × *C*═O, urea), 174.83 (2 × *C*═O) ppm. IR (KBr): ν_{max} = 1605 (C═C), 1715(asym C═O), 1770 (sym C═O), 2850-2930 (Ali H), 3050 (Ar H), 3100 (Olepine H), 3200 (sh N_3H), 3300 (sh N_1H) cm^{-1}. Exact mass: (M^+): calcd. for $C_{26}H_{24}N_6O_4$, 484.1859; found 484.1858.

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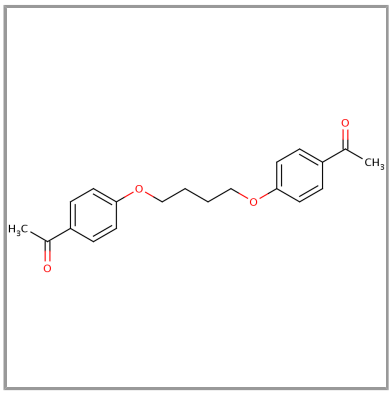

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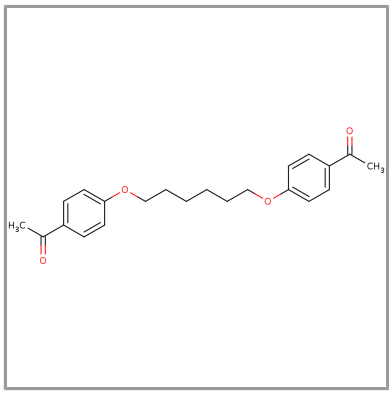

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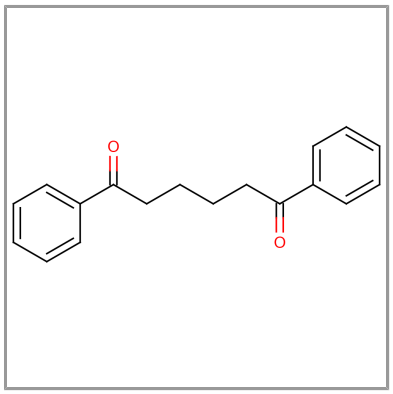

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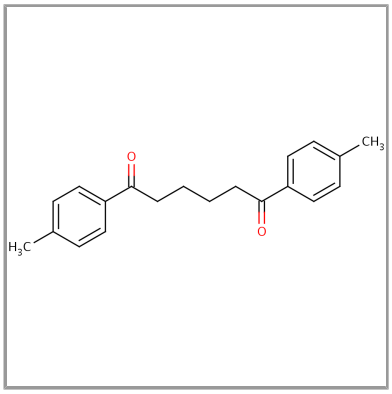

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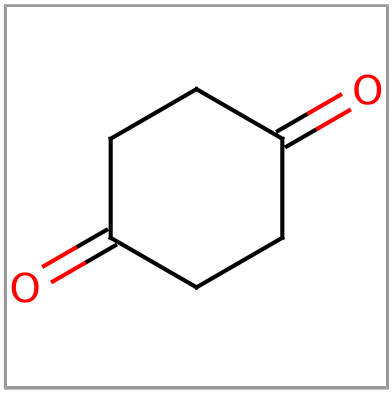

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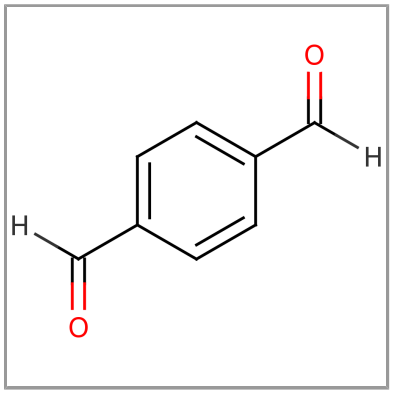

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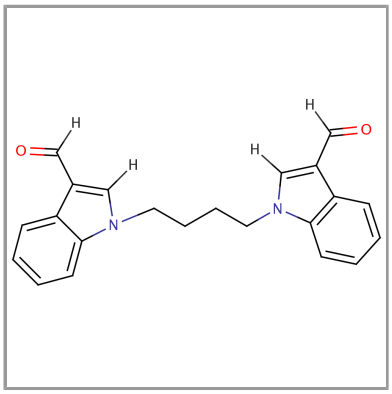

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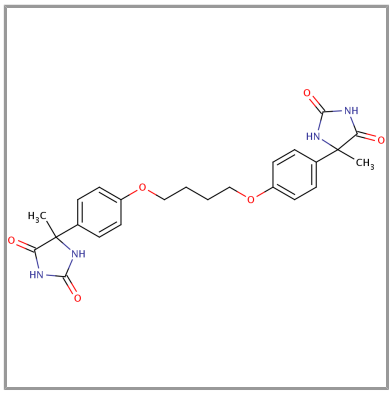

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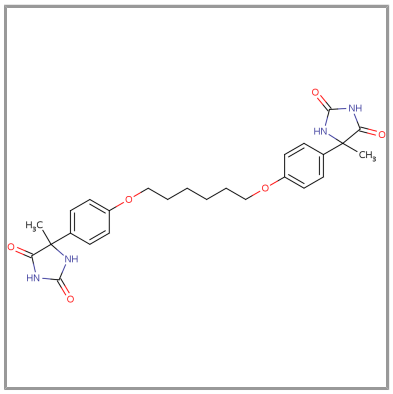

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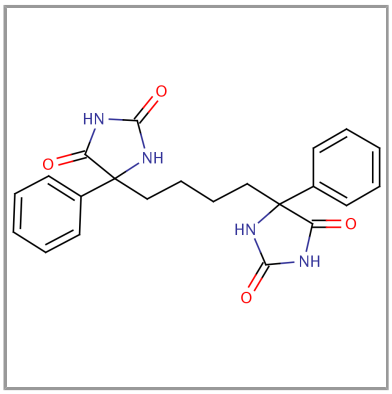

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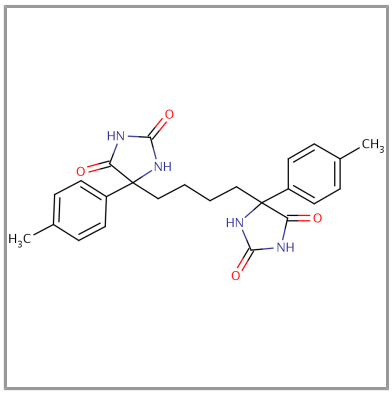

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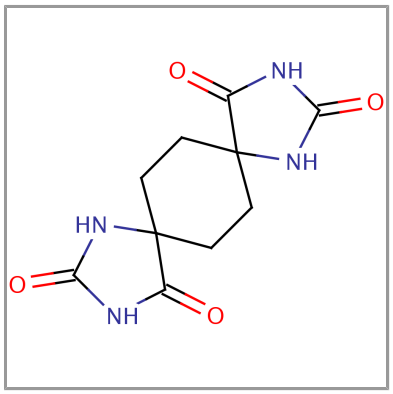

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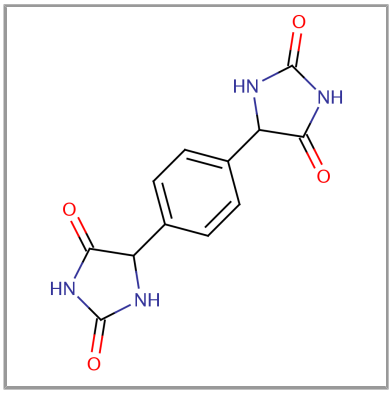

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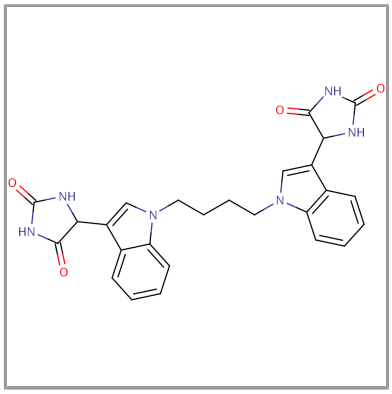

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