

TABLE I<sup>a</sup>  
 N,N'-BIS-(ARYLSULFONYL)-PIPERAZINES AND N,N'-PIPERAZINIUM BIS-(ARYLSULFONATES)

R	M. p., °C. (cor.)	Yield, %	Formula	Analyses, %			
				Nitrogen		Sulfur	
				Calcd.	Found	Calcd.	Found
N,N'-Bis-(R-sulfonyl)-piperazine							
Benzene	291.3-291.7	83	C <sub>16</sub> H <sub>18</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>	7.65	7.56		
<i>p</i> -Toluene	298.4-298.6	35	C <sub>15</sub> H <sub>22</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>	7.11	7.01		
<i>o</i> -Toluene	209.0-209.4	23	C <sub>15</sub> H <sub>22</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>	7.11	7.15	16.26	16.25
<i>p</i> -Bromobenzene	Not below 300	25	C <sub>16</sub> H <sub>14</sub> Br <sub>2</sub> N <sub>2</sub> O <sub>4</sub> S <sub>2</sub>	5.35	5.55	12.23	12.47
2-Nitrotoluene	278.0-278.3	51	C <sub>15</sub> H <sub>20</sub> N <sub>2</sub> O <sub>5</sub> S <sub>2</sub>	11.57	11.52	13.24	12.95
N,N'-Piperazinium Bis-(R-sulfonate)							
Benzene	Not below 300	57	C <sub>16</sub> H <sub>22</sub> N <sub>2</sub> O <sub>6</sub> S <sub>2</sub>	6.96	6.84	15.94	15.88
<i>p</i> -Toluene	Not below 300	80	C <sub>15</sub> H <sub>26</sub> N <sub>2</sub> O <sub>6</sub> S <sub>2</sub>	6.51	6.53	14.67	14.45
4-Nitrochlorobenzene	Not below 300	68	C <sub>16</sub> H <sub>13</sub> Cl <sub>2</sub> N <sub>4</sub> O <sub>10</sub> S <sub>2</sub>	9.98	9.98	11.43	11.27
2,5-Dichlorobenzene	Not below 300	62	C <sub>16</sub> H <sub>13</sub> Cl <sub>4</sub> N <sub>2</sub> O <sub>6</sub> S <sub>2</sub>	5.19	5.04	11.87	11.68

<sup>a</sup> Previous preparations of symmetrical sulfonyl derivatives have been carried out by Marckwald and von Droste-Huelshoff, *Ber.* 31, 3261 (1898); Peacock and Dutta, *J. Chem. Soc.*, 1303 (1934); Kermack and Tebrich, *ibid.*, 202 (1940); Gough and King, *J. Chem. Soc.*, 2426 (1928), prepared a piperazinium sulfonate.

g. of anhydrous potassium carbonate for seven hours. In all cases the resulting amorphous white material was washed several times with hot water and ethanol before being recrystallized twice from hot nitrobenzene. The crystals were then filtered out and washed with hot water, hot ethanol, and ether to remove the last traces of the nitrobenzene. These compounds were insoluble in water, ethanol, and ether, but could be recrystallized from glacial acetic acid, acetamide or pyridine.

**N,N'-Piperazinium Bis-(arylsulfonates).**—A solution of 0.05 mole (9.7 g.) of piperazine hexahydrate in 50 ml. of water was added to 0.1 mole of the appropriate sulfonic acid dissolved in a minimum quantity of water. If no precipitate formed at once, the solution was concentrated on the steam-bath. The resulting precipitate was filtered off from the solution and was recrystallized twice from the appropriate hot solvent. N,N'-Piperazinium bis-(2,5-dichlorobenzenesulfonate) was recrystallized from water, N,N'-piperazinium bis-(4-nitro-chlorobenzene-2-sulfonate) from 50% ethanol, and the other two compounds from 95% ethanol. In general these piperazinium salts were insoluble in ether, slightly soluble in alcohol, moderately soluble in cold water, and quite soluble in hot water.

A summary of these compounds is shown in Table I.

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#### Pentahydrate of 2-(*p*-Aminobenzenesulfonamido)-thiazole Sodium Salt

Lott and Bergeim<sup>1</sup> report the preparation of 2-(*p*-aminobenzenesulfonamido)-thiazole sodium salt by a method stated to be a modification of that used by Marshall<sup>2</sup> for sodium sulfapyridine.

The necessity for preparation of sulfathiazole sodium salt prompted an investigation of this compound. Using a modification of the Marshall method, we obtained white

plates which formed in clusters. These crystals melted at 55°, and on further heating with loss of water at 100°, recrystallized and melted again at 264.5° with decomposition. The second melting point is in good agreement with that reported by Lott for his sodium sulfathiazole. *Anal.* Calcd. for C<sub>9</sub>H<sub>8</sub>N<sub>2</sub>O<sub>2</sub>Na·5H<sub>2</sub>O: H<sub>2</sub>O, 24.52. Found: H<sub>2</sub>O, 24.55. This pentahydrate (by analysis) was readily soluble in cold water, alcohol and acetone. When an aqueous solution of the salt was added to a copper sulfate solution the characteristic purple precipitate of copper sulfathiazole as described by Lott was formed.

The pentahydrate loses moisture slowly on standing in dry air and yellows in the presence of light. Electro-metric pH determinations showed aqueous solutions to have the same basicity as solutions of equal molarity prepared from stoichiometric equivalents of standard sodium hydroxide solution and pure sulfathiazole, m. p. 202-202.5° with decomposition.

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#### α-Naphthylcyclopentanol-1

Fifty grams of α-bromonaphthalene was converted to α-naphthylmagnesium bromide in the usual way.<sup>1</sup> After adding 100 ml. of benzene, the solution was cooled in ice-water and 22 g. of cyclopentanone dissolved in 50 ml. of dry ether was added dropwise. The product was then hydrolyzed and isolated in the customary manner. The carbinol was recrystallized three times from ligroin. It formed clusters of white needles which melted at 74-76°, yield 70%.

*Anal.* Calcd. for C<sub>15</sub>H<sub>16</sub>O: C, 84.8; H, 7.55. Found: C, 84.72; H, 7.68.

(1) Gilman, *et al.*, "Organic Syntheses," Vol. XI, p. 80.

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(1) Lott and Bergeim, *This Journal*, 61, 3593 (1939).

(2) Marshall, *Science*, 88, 597 (1938).