

<b>COMPONENTS:</b> (1) Acetamide, N-[4-[(3,4-dimethyl-5-isoxazolyl)amino]sulfonyl]phenyl]- (acetyl sulfisoxazole); $C_{13}H_{15}N_3O_4S$ ; [4206-74-0] (2) Phosphoric acid, disodium salt; $Na_2HPO_4$ ; [7558-94-4] (3) Phosphoric acid, monopotassium salt; $KH_2PO_4$ ; [7778-77-0] (4) Water; $H_2O$ ; [7732-18-5]	<b>ORIGINAL MEASUREMENTS:</b> Bandelin, F. J. ; Malesh, W. <i>J. Am. Pharm. Assoc. Sci. Ed.</i> <u>1959</u> , 48, 177-81.																											
<b>VARIABLES:</b> pH	<b>PREPARED BY:</b> R. Piekos																											
<b>EXPERIMENTAL VALUES:</b> <p>Solubility of acetyl sulfisoxazole in buffers of varying mixtures of <math>Na_2HPO_4 \cdot 7H_2O</math> (71.6 g/l distilled water; <math>0.27 \text{ mol dm}^{-3}</math>, compiler) and <math>KH_2PO_4</math> (36.3 g/l distilled water; <math>0.27 \text{ mol dm}^{-3}</math>, compiler) at <math>37^\circ\text{C}</math></p> <table border="1" data-bbox="336 665 1034 1058"> <thead> <tr> <th colspan="3" style="text-align: center;"><u>Solubility (based on sulfisoxazole)</u></th> </tr> <tr> <th style="text-align: center;">Equilibrium pH</th> <th style="text-align: center;">mg/100 ml</th> <th style="text-align: center;"><math>10^2 \text{ mol dm}^{-3} \text{ }^a</math></th> </tr> </thead> <tbody> <tr><td style="text-align: center;">4.5</td><td style="text-align: center;">8</td><td style="text-align: center;">0.030</td></tr> <tr><td style="text-align: center;">5.0</td><td style="text-align: center;">12</td><td style="text-align: center;">0.045</td></tr> <tr><td style="text-align: center;">5.5</td><td style="text-align: center;">38</td><td style="text-align: center;">0.140</td></tr> <tr><td style="text-align: center;">6.0</td><td style="text-align: center;">105</td><td style="text-align: center;">0.393</td></tr> <tr><td style="text-align: center;">6.4</td><td style="text-align: center;">190</td><td style="text-align: center;">0.711</td></tr> <tr><td style="text-align: center;">6.8</td><td style="text-align: center;">375</td><td style="text-align: center;">1.400</td></tr> <tr><td style="text-align: center;">7.2</td><td style="text-align: center;">1040</td><td style="text-align: center;">3.891</td></tr> </tbody> </table> <p style="text-align: center;"><sup>a</sup> calculated by compiler</p>		<u>Solubility (based on sulfisoxazole)</u>			Equilibrium pH	mg/100 ml	$10^2 \text{ mol dm}^{-3} \text{ }^a$	4.5	8	0.030	5.0	12	0.045	5.5	38	0.140	6.0	105	0.393	6.4	190	0.711	6.8	375	1.400	7.2	1040	3.891
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<b>AUXILIARY INFORMATION</b>																												
<b>METHOD/APPARATUS/PROCEDURE:</b> <p>Solns were prepd by adding an excess of acetyl sulfisoxazole to a 10 ml of buffer soln at each pH level in 18 x 150-mm test tubes, stoppering the tubes, and placing them in water bath at <math>37^\circ\text{C}</math> with gentle agitation for 24 h. The solute was then hydrolyzed with 5% <math>H_2SO_4</math> for 1 h to liberate the free sulfonamide. One-ml aliquot of the hydrolyzate was accurately pipetted into a volumetric flask for diln and analysis. The sulfonamide was assayed colorimetrically by the method of Bratton and Marshall as described in detail by Biamonte and Schneller (1). A standard curve was prepd using accurately prepd standard solutions.</p>	<b>SOURCE AND PURITY OF MATERIALS:</b> <p>Neither source nor purity of the reagents were specified. Distilled water was used.</p> <b>ESTIMATED ERROR:</b> <p>Soly: av values of duplicate runs are reported (authors).            Temp and pH: not specified</p> <b>REFERENCES:</b> <p>1. Biamonte, A. R.; Schneller, G. E.  <i>J. Am. Pharm. Assoc., Sci. Ed.</i>  <u>1952</u>, 41, 341.</p>																											

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(1) Acetamide, N-[4-[(3,4-dimethyl-5-isoxazolyl)amino]sulfonyl]phenyl]- (N <sup>4</sup> -acetylsulfafurazole)* C <sub>13</sub> H <sub>15</sub> N <sub>3</sub> O <sub>4</sub> S; [4206-74-0] (2) Phosphoric acid, disodium salt; Na <sub>2</sub> HPO <sub>4</sub> ; [7558-94-4] (3) Phosphoric acid, monopotassium salt; KH <sub>2</sub> PO <sub>4</sub> ; [7778-77-0] (4) Water; H <sub>2</sub> O; [7732-18-5]	Hekster, Ch. A.; Vree, T. B. <i>Antibiotics Chemother.</i> <u>1982</u> , <u>31</u> , 22-118.											
VARIABLES: <p style="text-align: center;">pH</p>	PREPARED BY: <p style="text-align: center;">R. Piekos</p>											
EXPERIMENTAL VALUES: <table border="1" style="margin: 20px auto; width: 60%;"> <thead> <tr> <th rowspan="2">pH</th> <th colspan="2">Solubility at 25°C</th> </tr> <tr> <th>mg/l</th> <th>mol dm<sup>-3</sup> a</th> </tr> </thead> <tbody> <tr> <td>5.5</td> <td>250</td> <td>8.08 x 10<sup>-4</sup></td> </tr> <tr> <td>7.5<sup>b</sup></td> <td>6,893</td> <td>2.228 x 10<sup>-2</sup></td> </tr> </tbody> </table> <p style="margin-left: 40px;"><sup>a</sup>Calculated by compiler</p> <p style="margin-left: 40px;"><sup>b</sup>Erroneous pH value of 7.0 is given in the article</p> <p style="margin-left: 40px;">*Another common trivial name is acetyl sulfisoxazole.</p>		pH	Solubility at 25°C		mg/l	mol dm <sup>-3</sup> a	5.5	250	8.08 x 10 <sup>-4</sup>	7.5 <sup>b</sup>	6,893	2.228 x 10 <sup>-2</sup>
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METHOD/APPARATUS/PROCEDURE: The earlier developed method (1) was used (personal communication). Satd solns of N <sup>4</sup> -acetylsulfafurazole* were prepd in phosphate buffers of pH 5.5 and 7.5 at 25°C. The concn of the solute was measured by means of a Spectra Physics 3500B high-performance liquid chromatograph equipped with a Model 748 column oven and a Pye-Unicam LC-UV spectrophotometric detector.	SOURCE AND PURITY OF MATERIALS: Neither source nor the purity of the materials was specified. <table border="1" style="margin-top: 20px;"> <thead> <tr> <th data-bbox="674 1598 1229 1631">ESTIMATED ERROR:</th> </tr> </thead> <tbody> <tr> <td data-bbox="674 1639 1229 1721">               Soly: the detection limit of the solute by HPLC was 0.5 mg/l (authors). The errors in temp and pH were not specified.             </td> </tr> <tr> <th data-bbox="674 1729 1229 1753">REFERENCES:</th> </tr> <tr> <td data-bbox="674 1761 1229 1917">               1. Hekster, Y.A. Vree, T. B.; Damsma, J. E.; Friesen, W. T. <i>J. Antimicrob. Chemother.</i> <u>1981</u>, <u>8</u>, 133.             </td> </tr> </tbody> </table>	ESTIMATED ERROR:	Soly: the detection limit of the solute by HPLC was 0.5 mg/l (authors). The errors in temp and pH were not specified.	REFERENCES:	1. Hekster, Y.A. Vree, T. B.; Damsma, J. E.; Friesen, W. T. <i>J. Antimicrob. Chemother.</i> <u>1981</u> , <u>8</u> , 133.							
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<p><b>EXPERIMENTAL VALUES:</b></p> <p>Solubility of acetyl sulfafurazole* in McIlvaine's disodium phosphate-citric acid buffer solution at 37°C</p> <table border="1" data-bbox="353 620 1108 975"> <thead> <tr> <th rowspan="2">Initial pH of buffer</th> <th colspan="2">Solubility</th> <th rowspan="2">Final pH</th> </tr> <tr> <th>mg/100 ml solution</th> <th>10<sup>3</sup> mol dm<sup>-3</sup> a</th> </tr> </thead> <tbody> <tr> <td>4.5</td> <td>6.0</td> <td>0.19</td> <td>4.5</td> </tr> <tr> <td>5.0</td> <td>17.3</td> <td>0.56</td> <td>5.0</td> </tr> <tr> <td>6.0</td> <td>126.1</td> <td>4.08</td> <td>6.0</td> </tr> <tr> <td>7.0</td> <td>757.9</td> <td>24.50</td> <td>6.7</td> </tr> </tbody> </table> <p><sup>a</sup> Calculated by compiler</p> <p>*Another common trivial name is acetyl sulfisoxazole.</p>		Initial pH of buffer	Solubility		Final pH	mg/100 ml solution	10 <sup>3</sup> mol dm <sup>-3</sup> a	4.5	6.0	0.19	4.5	5.0	17.3	0.56	5.0	6.0	126.1	4.08	6.0	7.0	757.9	24.50	6.7
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<p><b>METHOD/APPARATUS/PROCEDURE:</b></p> <p>Acetyl sulfafurazole* was equilibrated with 50 ml of the buffer soln for 18 h at 37°C with agitation. The suspension was immediately filtered through a Whatman No. 1 paper. The compd was assayed in the filtrate after boiling for 15-20 min with 30% NaOH soln as sulfafurazole* by the method of Bratton and Marshall (1) using a Beckman DU spectrophotometer, at 545 nm.</p>	<p><b>SOURCE AND PURITY OF MATERIALS:</b></p> <p>Acetyl sulfafurazole*, mp 214.8-15.9°C, was supplied by the American Cyanamid Co, Calco Chem Div, Bound Brook, N.J. The source and purity of the remaining materials was not specified.</p> <p><b>ESTIMATED ERROR:</b>pH and temp: not specified. Accuracy of the anal method was illustrated by the following values: expected 2.003, 3.004, 4.006, 5.007 mg/100 ml; found 2.08; 3.06, 4.12, 5.10 resp.</p> <p><b>REFERENCES:</b></p> <p>1. Bratton, A. C.; Marshall, E. K. Jr. <i>J. Biol. Chem.</i> <u>1939</u>, <i>128</i>, 537.</p>																						

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<b>VARIABLES:</b> pH at 37° C	<b>PREPARED BY:</b> R. Piekos																							
<b>EXPERIMENTAL VALUES:</b> Solubility of acetyl sulfisoxazole in a solution containing $CaCl_2$ 0.143, $MgCl_2$ 0.121, $NH_4H_2PO_4$ 0.300, $KCl$ 1.660, $NaCl$ 2.950 and urea 20 g/dm <sup>3</sup> (synthetic urine, Mosher Vehicle) at 37°C <table border="1" data-bbox="237 662 1012 1024"> <thead> <tr> <th rowspan="2">Equilibrium pH</th> <th colspan="2">Solubility</th> </tr> <tr> <th>mg/100 ml as sulfisoxazole</th> <th>10<sup>2</sup> mol dm<sup>-3</sup> a</th> </tr> </thead> <tbody> <tr> <td>4.5</td> <td>30</td> <td>0.097</td> </tr> <tr> <td>5.0</td> <td>44</td> <td>0.140</td> </tr> <tr> <td>5.5</td> <td>70</td> <td>0.230</td> </tr> <tr> <td>6.0</td> <td>160</td> <td>0.520</td> </tr> <tr> <td>6.5</td> <td>560</td> <td>1.810</td> </tr> <tr> <td>7.0</td> <td>1230</td> <td>3.980</td> </tr> </tbody> </table> <p data-bbox="262 1064 554 1095">a calculated by compiler</p>		Equilibrium pH	Solubility		mg/100 ml as sulfisoxazole	10 <sup>2</sup> mol dm <sup>-3</sup> a	4.5	30	0.097	5.0	44	0.140	5.5	70	0.230	6.0	160	0.520	6.5	560	1.810	7.0	1230	3.980
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<b>METHOD/APPARATUS/PROCEDURE:</b> Excess acetyl sulfisoxazole was added to aliquots of synthetic urine solns and 1% $H_3PO_4$ or 1% $NaOH$ solns were used to adjust the pH to the required value. The solns were agitated for 24 h with addn of acid or base to keep them at the desired pH level until equilibrium was attained. Then the solns were filtered and in aliquots the acetyl sulfonamide was assayed spectrophotometrically by the method described by Biamonte and Schneller (1). Before detn the soln was refluxed with 5% $H_2SO_4$ for 1 h to liberate the free amino compound.	<b>SOURCE AND PURITY OF MATERIALS:</b> Nothing specified <b>ESTIMATED ERROR:</b> Soly: average values of 2 detns were given. Temp: not specified. pH : not specified. <b>REFERENCES:</b> 1. Biamonte, A. R.; Schneller, G. E., <i>J. Am. Pharm. Assoc., Sci. Ed.</i> <u>1952</u> , 41, 341.																							

