COMP(DNENTS: Benzenesulfonamide, 4-amino-	EVALUATOR: Anthony N. Paruta
(sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1] (2) Water		Department of Pharmaceutics University of Rhode Island Kingston, Rhode Island, USA and
	Water	
(3) Aqueous sodium salicylate		Ryszard Piekos Faculty of Pharmacy, University of Gdansk Gdansk, Poland 1986

CRITICAL EVALUATION:

The solubilities of sulfanilamide, the parent compound of the derivatives found in these volumes, in water at various temperatures are given in Tables I-III on the mol $\rm dm^{-3}$ concentration scale. The originating author's values are always stated in the units reported. All recommended values are given as the simple average of the referenced magnitudes on the mol $\rm dm^{-3}$ scale.

Table I: Solubility of Sulfanilamide in water, 293K

Reference	10^2 mol dm ⁻³ (*indicates mol kg ⁻¹)
5	3.1*
6	2.3*
7	3.5 (291-292K)
10	3.1
11	3.07*
12	3.1
14	3.07*
17	3.07
18	3.34
20	3.662 (polymorph)
21	3.1
22	3.07 (294-298K)
23	3.06*
24	3.447
27	3.539* (beta polymorph)

The solubility given by Weinstein and McDonald (6) is about two-thirds of the other values and was therefore rejected. The results of Becher and Leya (7), and of Gusyakov et al. (22) were also rejected since they refer to a temperature range of two to four degrees. Further, Becher and Leya (7), give a value about 15% higher than the apparent average. In 1944, Sapozhnikova and Postovskii (5), and Knazko (18) in 1966, used equilibrium times of one hour and three to four hours, respectively. These equilibrium times are considered inadequate for a saturation to be reached, and even though the values appear to be valid, they were not considered further. In 1971, Rohdewald (24) used a two hour equilibrium time which is considered as a pre-saturation time, yet Rohdewald (24) reported a value that was about 15% too high with respect to the apparent average. Ito and Sekiguchi (20), and Sekiguchi et al. (27) in 1967 and 1975, respectively, used polymorphic forms of sulfanilamide which may not be identifiable with the solute under consideration. Since the reports (20,27) do not give melting points for the polymorphs, they cannot be used in this evaluation. It is interesting to note that the beta form used by Sekiguchi et al. (27) is the stable form and should be quite close to the sulfanilamide value. However, the value provided is calculated rather than experimental, and would not be appropriate to mix with experimental results. The remaining values are sufficiently close in magnitude to evaluate further. The values of Gusyakov and Sukmans'ka in 1960 and 1961 (10-12), and Shkadova (23) in 1969, indicate that equilibrium had been reached, and may be assumed reasonably accurate. However, in two cases (10,12), the solubility technique of adding solute to a solution of the solute until saturation has been reached, is considered invalid, and were not used although the values given were "good" (concurring with the apparent average) numbers. The final pool of values (11,14,17,21), using at least an 8 hour equilibration time and appropriate analytical methods, allow for an average to be calculated and the recommended value for sulfanilamide in water at 293K is given as 3.08×10^{-2} mol dm⁻³. It is interesting to note that even if the values from other workers (10,12,23) were used in the calculation, there would be no difference in the final result in the recommended value due obviously to their concurrence with the average value.

Aqueous solubility of sulfanilamide was studied at room temperature, 298K by eight workers (3,8,9,13,15,16,20,27), and the reported numerical data are summarized in Table II. There are three reports by Sekiguchi et al. (16,20,27) dealing with the polymorphic forms of sulfanilamide. While the value of 4.711 x 10⁻² mol dm⁻³ in reference 20 is quite close to other values, as was the value in reference 27, none of the polymorphic forms were considered further. The value given by Khawan et al. (13) for 297K, appears to be too low by about 15% and was not placed in the final pool of acceptable values. The remaining four values by Clark et al. (3), Dolique and Foucault (8), Matsuura and Sekiguchi (9) and Paruta et al. (15) using equilibrium times longer than 9 hours,

up to 72 hours, were determined by appropriate analytical methods. The initial average value of the four acceptable values allow for a tentative recommeded value of 4.72 x 10^{-2} mol dm^{-3} at 298K.

Table II: Solubility of Sulfanilamide in water, 298K

Reference	10^2 mol dm ⁻³ (*indicates mol kg ⁻¹)
3	4.86*
8	4.7* (299K)
9	4.55
13	4.16 (297K)
15	4.8 (296.7K)
16	4.1925 (beta polymorph)
20	4.711 (unspecified polymorph)
27	4.677* (beta polymorph)
27	5.451* (alpha polymorph)

The tentative designation results from the slight differences in temperature reported in these papers (8,15), which were 299K and 297.6K, as given by Dolique and Foucault (8) and Paruta et al. (15). The authors give values within 1% of the average value using Clark et al. (3) and Matsuura and Sekiguchi (9) values alone leading to an average value of 4.70×10^{-2} mol dm⁻³.

The solubility of sulfanilamide at body temperature, 310K in water as reported by various workers are given in Table III.

Table III: Solubility of Sulfanilamide in water, 310K

Reference	10^2 mol dm ⁻³ (*indicates mol kg ⁻¹)
1	8.59
2	11*
3	8.48*
4	8.64*
5	8.4*
26	8.19
28	8.65
29	9.93

The value given by Trefoull (2) is obviously too high. That of Goto et al. (29) is also higher than the apparent average by about 15% and neither is considered in the final pool of acceptable values. Sapozhnikova and Postovskii (5), and Kitao et al. (26) provide reasonable values, though the former used an equilibrium time of one hour only and the latter give no specification relative to saturation. Clark et al. (3), Kienle and Sayward (4) and Kaneniwa et al. (28) used adequate equilibration times and accurate analytical methods. Durel and Allinne (1) used 24 hours for saturation, however, no analytical method was specified. Thus, the saturation solubility for sulfanilamide at 310K can be given as 8.59×10^{-2} mol dm⁻³. There were two additional solubility determinations in water. Yamazaki et al. (19) reported a value of 6.1 x 10^{-2} mol dm⁻³ at 303K, which is about the correct magnitude. Burger (25) gave solubilities of polymorphic Form II with a melting point of 429K over a temperature range of 313K-358K.

Solubility was also determined in an aqueous hydrotropic 1 mol dm $^{-3}$ sodium salicylate (30-32) at 293K and a tentative value of 8.10 x 10^{-2} mol dm $^{-3}$ can be suggested. The 1 mol ${\rm dm}^{-3}$ solution of sodium salicylate causes a 2.63 fold increase in the solubility of sulfanilamide.

- Durel, M.P.; Allinne, M. Bull. Soc. Med. Hop. Paris III 1941, Tréfouël, M. Bull. Acad. Med. Paris 1941, 124, 546-54. (1) 251-9.
- (2)
- Clark, W.G.; Strakosch, E.A.; Levitan, N.I. J. Lab. C. Kienle, R.H.; Sayward, J.M. J. Am. Chem. Soc. 1942, Clin. Med. 1942, (3) 28, 188-9.
- 64, $246\overline{4-8}$. (4)
- Sapozhnikova, N.V.; Postovskii, I.Ya. Zh. Prikl. Khim. (5) 1944, 17, 427-34.
- Weinstein, L.; McDonald, A. Science 1945, Becher, R.; Leya, S. Experientia 1946, 2, (6) 101, 44-5.
- 459-60. (7)
- Dolique, R.; Foucault, J. Trav. soc. pharm. Montpellier 1952, (8) 12, 145-53.
- Matsuura, H.; Sekiguchi, K. Yakuzaigaku 1960, 20, 213-Gusyakov, V.P.; Sukmans ka, I.V. Farm. Zh. (Kiev) 1960, 20, 213-18.(9)
- 15(1), (10) 20-23.
- Gusyakov, V.P.; Likhol'ot, N.M. Farm. Zh. (Kiev) 1960, 21-4. (11)15(3),
- 16, (12) Gusyakov, V.P.; Sukmans'ka, I.V. Farm. Zhur. (Kiev) 1961, 25-8.
- Khawan, M.N.; Tawashi, R.; Czetsch-Lindenwald, H.V. Sci. Pharm. 1964, 32, (13)271-9.
- Likhol'ot, N.M.; Gusyakov, V.P. Farm. Zh. (Kiev) 1964, 19(1), (14)

- (14) Likhol'ot, N.M.; Gusyakov, V.P. Farm. Zh. (Kiev) <u>1964</u>, 19(1), 52-5.
- (15)
- (16)
- (17)
- (18)
- (19)
- (20)
- (21)
- (22)
- (23)
- (24)
- (25)
- Likhol'ot, N.M.; Gusyakov, V.P. Farm. Zh. (Kiev) 1964, 19(1), 52-5.

 Paruta, A.N. J. Pharm. Sci. 1964, 53(10), 1252-4.

 Sekiguchi, K.; Ito, K. Chem. Pharm. Bull. 1965, 13(4), 405-13.

 Likhol'ot, N.M. Farm. Zh. (Kiev) 1965, 20(5), 44-6.

 Knazko, L. Farm. Obsor 1966, 35, 298-311.

 Yamazaki, M.; Aoki, M.; Kamada, A.; Yata, N. Yakuzaigaku 1967, 27(1), 37-40.

 Ito, K.; Sekiguchi, K. Chem. Pharm. Bull. 1967, 15(4), 420-6.

 Gusyakov, V.P.; Likhol'ot, N.M.; Kutna, I.M. Farm. Zh. (Kiev) 1967, 22(3), 34-9.

 Gusyakov, V.P.; Likhol'ot, N.M.; Kutna, I.M. Farm. Zh. (Kiev) 1968, 23(6), 56-61.

 Shkadova, A.I. Farm. Zh. (Kiev) 1969, 24(3), 39-41.

 Rohdewald, P. Pharm. Ztg. 1971, No. 38, 1342-4.

 Burger, A. Pharm. Ind. 1973, 35, 626-33.

 Kitao, K.; Kubo, K.; Morishita, T.; Yata, N.; Kamada, A. Chem. Pharm. Bull.

 1973, 21, 2417-26. (26)
- (27)
- 23, 1353-62. 26(9), 2603-14. (28)
- 1973, 21, 2417-26.
 Sekiguchi, T.; Tsuda, Y.; Kanke, M. Chem. Pharm. Bull. 1975, 23, 13
 Kaneniwa, N.; Watari, N.; Iijima, H. Chem. Pharm. Bull. 1978, 26(9),
 Goto, S.; Komatsu, M.; Tagawa, K.; Kawata, M. Chem. Pharm. Bull. 1983, 31(1). (29) 256-61.

- (30) Knazko, L. Farm. Obzor 1966, 35, 298-311.
 (31) Gusyakov, V.P.; Sukmans'ka, I.V. Farm. Zh. (Kiev) 1960, 15(1),
 (32) Gusyakov, V.P.; Sukmans'ka, I.V. Farm. Zhur. (Kiev) 1961, 16,

16 COMPONENTS: (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) Water; H₂0; [7732-18-5] VARIABLES: Temperature EXPERIMENTAL VALUES: 10 9 SOLUBILITY in WATER per cent w/v 8 7

6

5

4

3 2

1

p-aminobenzenesulphonamide.

AUXILIARY INFORMATION

50

60

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Temperature, Centigrade Fig. 1. - The variation with temperature of the solubility in water of

30

20

METHOD/APPARATUS/PROCEDURE:

Nothing specified.

SOURCE AND PURITY OF MATERIALS:

ORIGINAL MEASUREMENTS:

Quart. J. Pharm. Pharmacol.

1936,

Allport, N. L.

9, 360-6.

PREPARED BY:

R. Piekos

The sulfanilamide was prepd in the author's laboratory on a semitechnical scale and melted quite sharply at 166.0°C. Specimens recrystd from dil alcohol melted at 166.5°C. No other details were given. Purity of the water was not specified.

ESTIMATED ERROR:

Temp: not specified. Soly: not specified.

COMPONENTS: (1) Benzenesulfonamide, 4-amino- (sulfa-	ORIGINAL MEASUREMENTS: Durel, M.P.; Allinne. M.
nilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Bull. Soc. Med. Hop. Paris III
(2) Water; H ₂ O; [7732-18-5]	1941, 251-9.
_	<u> </u>
VARIABLES:	DDSD 4DSD DV
One temperature: 37°C	PREPARED BY: R. Piekos
·	
EXPERIMENTAL VALUES:	,
Solubility of sulfanilamide in water at $37^{\circ}\mathrm{C}$ compiler)	is 14.8 g/liter (8.59 x 10 ⁻² mol dm ⁻³ ,
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
A mixt of sulfanilamide and water was	Source and purity of sulfanilamide was
agitated for 24 hours at 37°C.	not specified.
	Distilled water was used.
	ESTIMATED ERROR:
	Nothing specified.
	moening openitions
	REFERENCES:
	REAL ENGINEERS;
	1

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino- (sulfa-	Tréfouël, M.
nilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Bull. Acad. Med. Paris 1941, 124,
(2) Water; H ₂ O; [7732-18-5]	546-54.
,	
VARIABLES: One temperature: 37°C	PREPARED BY:
One temperature: 3/ C	R. Piekos
EXPERIMENTAL VALUES:	
Solubility of sulfanilamide in water at 37°C	is 1.8 parts per 100 parts water
(0.11 mol kg ⁻¹ water, compiler).	
, (0.11)	
	1
ĺ	
AUXILIARY	INFORMATION
ACTIVIDA /ARDA DATIVICA (PROCEDURA)	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS;
Sulfanilamide was diazotized, coupled with	Nothing specified.
N-naphthyl-1-N,N-diethyl-3-propylenediamine	
and assayed colorimetrically.	
	i
<u>{</u>	
	ESTIMATED ERROR:
}	Nothing angelfied
	Nothing specified.
į	
	REFERENCES:
	}
{	
	İ

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{c}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Clark. W.G.; Strakosch, E.A.; Levitan, N.I. *J. Lab. Clin. Med.* 1942, 28, 188-9.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/°c	Solubility	
£/ C	g/100 g water	10 ² mol kg ⁻¹ water ^a
25	0.836	4.86
37	1.460	8.48

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small tinted glass container contg excess sulfanilamide in water was shaken in a water bath thermostat for 24 h. The satd soln was then filtered by aspiration through a washed and dried asbestos filter stick into a weighed weighing bottle. The entire app was kept at the temp at which the compd was dissolved. The amt dissolved was then detd by the method of Bratton and Marshall (1), using a photoelectric colorimeter.

SOURCE AND PURITY OF MATERIALS:

Neither source nor purity of sulfanilamide was specified.

CO2-free distd water was used.

ESTIMATED ERROR:

Soly: not specified. Temp: $\pm 0.1^{\circ}$ C (authors).

REFERENCES:

Bratton, A.C.; Marshall, E.K., Jr.
 J. Biol. Chem. <u>1939</u>,128, 537.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: Temperature ORIGINAL MEASUREMENTS: Kienle, R. H.; Sayward, J. M. J. Am. Chem. Soc. 1942, 64, 2464-8. PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Solubility

	· -•		
t/°C	g/100 g soln (20)	mol kg ^{-1^a}	
23.0	0.64; 0.64	0.0374	
24.0	0.69	0.0403	
26.0	0.77	0.0451	
27.0	0.77 0.82; 0.82 ⁵ , b 0.82; 0.83 ±0.012	0.0480 $\frac{1}{2} \pm 0.00070$	
27.0	0.82; 0.83	0.0480; 0.0486	
28.0	0.87; 0.87	0.0510	
30.5	1 01	0.0592	
31.7	1.08; 1.08 ^b	0.0634	
33.0	1 10	0.0699	
34.0	1.26 1.27 ^b ; 1.27 +0.015	0.07417	
34.0	1.27 ; 1.27 5	0.0747 +0.000871	
35.5	1.37	0.0807	
37.0	1.47; 1.47)	0.0866)	
37.0	$ \begin{array}{cccc} 1.47; & 1.47 \\ 1.47^{b} & 1.47^{b} \\ & 1.46^{c} \end{array} $ ± 0.010	0.0866 \(\frac{+}{0.000581}	
37.0	1.46°)	0.0860)	
39.0	1.61; 1.61	0.0950	
42.0	1.84	0.1088	
46.0	2.21_	0.1312	
50.0	2.68 ^c	0.1599	

a calculated by compiler; bequilibrium approached from below;

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was rotated with water usually overnight. Equilibrium was approached usually from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol dm⁻³ NaNO₂ soln to first blue on starch - iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrystd from alcohol and from hot water; mp 165.9°C. Titrn with nitrite indicated that the compd was 100±0.3% pure. Elemental analysis and mixed mp detns confirmed this value. Purity of the water was not specified.

ESTIMATED ERROR: Soly: ±0.01 g/100 g soln or ±0.012 x 10⁻³ in mole fraction (authors). Temp: ±0.02°C (authors).

duration less than twelve hours.

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sapozhnikova, N.V.; Postovskii, I. Ya. Zh. Prikl. Khim. 1944, 17, 427-34.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/ ^o c	Solubility	
	Weight %	mol kg ⁻¹ water ^a
20	0.53	0.031
37	1.42	0.084
50	2.63	0.157
75	7.58	0.476
99	19.2	1.380

acalculated by compiler.

AUXILIARY INFORMATION

METHOD /APPARATUS / PROCEDURE:

Sulfanilamide was dissolved in water to form a satd soln which was occasionally agitated in a glass vessel immersed in a thermostat. The equilibrium was usually attained after 1 h. Five to 100-cm³ samples of the satd soln were placed in Pt crucibles or dishes and evapd to dryness at temps lower than 110-115°C. The residue was dried to const wt at 105-110°C and weighed.

SOURCE AND PURITY OF MATERIALS:

Pure, recrystd sulfanilamide was used. Its mp conformed to that reported in the literature.

Purity of the water was not specified.

ESTIMATED ERROR: Soly: quite reliable results were obtained over the temp range 20-75°C. At higher temps the accuracy was poor due to evapn of water during sampling (authors). Temp: ±0.05°C (authors).

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino- (sulfa-	Weinstein, L.; McDonald, A.
nilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Science 1945, 101, 44-5.
(2) Water; H ₂ O; [7732-18-5]	
WART ADI DC.	DDEDARD OV.
VARIABLES:	PREPARED BY:
One temperature: 20°C	R. Piekos
EXPERIMENTAL VALUES:	
	3
Solubility of sulfanilamide in water at 20° mol kg ⁻¹ , compiler).	C is 400 mg/100 cm water (2.3 x 10
morkg , compiler).	
	ļ
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Nothing specified.	Nothing specified.
	ESTIMATED ERROR:
	Nothing specified.
	REFERENCES:

23 COMPONENTS: (1) Benzenesulfonamide, 4-amino- (sulfa-ORIGINAL MEASUREMENTS: nilamide); $C_6H_8N_2O_2S$; [63-74-1] Becher, R.; Leya, S. (2) Water: H₂O [7732-18-5] Experientia 1946 2, 459-60 VARIABLES: PREPARED BY: One temperature: 18-19°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at room temperature (18-19 $^{\circ}$ C) is 610 mg% $(3.5 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler}).$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

After standing for more than two days the soln of sulfanilamide in water was filtered and sulfanilamide was assayed in the filtrate colorimetrically by the method of Druey and Oesterheld (1).

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Druey, J.; Oesterheld, G. Helv. Chim. Acta 1942, 25, 753.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino- (sulfa-	Dolique, R; Foucault, J.
nilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Trav. soc. pharm. Montpellier 1952,
(2) Water; H ₂ O; [7732-18-5]	<i>12</i> , 145-53.
,,,	12, 143 331
VARIABLES:	PREPARED BY:
One temperature: 26°C	
one temperature: 20 C	R. Piekos
EXPERIMENTAL VALUES:	
Solubility of sulfanilamide in water at 26°	C is 0.8 g/100 g water
$(4.7 \times 10^{-2} \text{ mol kg}^{-1} \text{ water - compiler}).$	
}	
{	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
j	}
The satd soln of sulfanilamide was shaken	Nothing specified.
in a test tube for 12 h at 26°C and	
filtered. The filtrate was evapd at	
100-110°C and the residue was weighed.	
1	
	ESTIMATED ERROR:
	Nothing specified.
	nothing specificati
	REFERENCES:
	1

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Matsuura, H.; Sekiguchi, K. Yakuzaigaku 1960, 20, 213-18.

VARIABLES:

One temperature: 25°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in water at 25° C is 0.782 g/100 ml (4.55 x 10^{-2} mol L⁻¹).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was agitated in water at 25°C for more than 9 h. Aliquots of the satd soln were withdrawn with a pipet fitted with a filter and sulfanilamide was detd spectrophotometrically using the Tsuda reagent for producing color.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was of the Japanese Pharmacopeia grade and was recrystd. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the error was 1.5% (authors).

Temp: $\pm 0.05^{\circ}$ C (authors).

COMPONENTS: ORIGINAL MEASUREMENTS: Gusyakov, V. P.; Sukmans'ka, I.V. (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] Farm. Zh. (Kiev) 1960, 15(1), 20-23. (2) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at 20°C is 0.53 g/100 ml water $(3.1 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler}).$ AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: To 50-ml tightly stoppered test tubes Sulfanilamide conformed to the requirecontg 25 ml water, placed in a thermostat, ments of the State Pharmacopeia VIII. accurately weighed 0.02-0.002-g Distilled water was used. quantities of sulfanilamide were added under agitation until satn was attained. ESTIMATED ERROR: Temp: $\pm 0.1^{\circ}$ C (authors). Soly: the accuracy of the detn of the concn was similar to that attained by volumetric method (authors). REFERENCES:

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3), 21-4.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in water at 20° C is 0.528 g/100 g water (3.07 x 10^{-2} mol kg⁻¹, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 h in a 50-ml test tube with 20 ml of water. Aliquots were taken with a pipet fitted with a filter. Sulfanilamide was detd at 285 nm using a SF-4 spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was crystd three times from hot water. Its purity conformed to the requirements of the State Pharmacopeia VIII.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the accuracy corresponded to that of colorimetric detns (authors). Temp: not specified.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: One temperature: 20°C EXPERIMENTAL VALUES: ORIGINAL MEASUREMENTS: Gusyakov, V. P.; Sukmans'ka, I. V. Farm. Zh. (Kiev) 1961, 16, 25-8.

Solubility of sulfanilamide in water at 20° C is 0.53 g/100 ml water (3.1 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions (0.02 - 0.002 g) to a known volume of water, held on a water bath, until satn was attained. Moreover, the concn of sulfanilamide was detd by means of a FEK-M photoelectrocolorimeter.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was a pharmacopeial product. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: results of the colorimetric and gravimetric runs differed by 1-3% (authors). Temp: $\pm 0.1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}S$; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawan, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. V. Sci. Pharm. 1964, 32, 271-9.

VARIABLES:

PREPARED BY:

Temperature

R. Piekos

EXPERIMENTAL VALUES:

t ^o C	Solubility	
EU	g/1	10 ² mo1 dm ^{-3^a}
13	3.58	2.08
15	3.79	2.20
16	3.85	2.24
24	7.17	4.16
34	12.71	7.38
44	20.36	11.82

acalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in excess to water and the mixture was stirred for 30 min. The soln was then kept in a drying cabinet for 24 h and occasionally shaken. After filtration the solution was assayed in the filtrate by the USP XVI method based on diazotization. The end point was detected by means of a starch paste as an indicator.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: Measurements were made in

duplicate (authors).

Temp: ±1°C (authors).

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: One temperature: 20°C ORIGINAL MEASUREMENTS: Likhol'ot, N. M.; Gusyakov, V. P. Farm. Zh. (Kiev) 1964, 19(1), 52-5. PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in water at 20° C is 0.528 g/100 g water (3.07 x 10^{-2} mol kg⁻¹ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was employed whereby a small excess of sulfanilamide was equilibrated with 20 ml of water in a 50-ml test tube for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified)
was recrystd from water.
Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3), 21.
- 2. The Extra Pharmacopeia (Martindale) 1955, 2(23), 353, 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Paruta, A. N.

J. Pharm. Sci. 1964, 53(10) 1252-4.

VARIABLES:

One temperature: 24.6°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in water at 24.6°C is 8.3 mg/ml (4.8 x 10^{-2} mol dm⁻³ - compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soly was detd in 15-ml vials attached to a submerged rotating plastic disk. The plastic disk unit was attached to a dispersator motor fitted with an Al shaft and submerged with attached samples in an 8-gal water bath. The temp of the bath was maintained at 24.6+0.2°C by a Sargent thermomonitor unit. After equilibration for about 72 h, samples were withdrawn through a fine glass-wool plug fitted to a pipet, placed in a volumetric flask and dild for subsequent analysis. The concn of the solute was detd by a Beckman DK-2 spectrophotometer from sample absorbance and a previously detd Beer-Lambert law plot. The absorption max for sulfanilamide was 259 mu.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd from an EtOH - water mixt and dried to const wt at 40° C. Its source was not specified. Dist water was used.

ESTIMATED ERROR:

Soly: not specified.
Tem: ±0.2°C (author).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sekiguchi, K.: Ito, K.

Chem. Pharm. Bull. 1965, 13(4), 405-13.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/°C	Solubili	
	10 mol dm solution	g dm ^{-3^a}
15	23.111	3.9797
25	41.925	7.2195
35	77.928	13.419

aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

In a 200-ml egg-plant type flask, immersed in a thermostat, an excess of sulfanilamide was placed with 100 ml of redistd water (pH 5.7 ~ 5.9) which was previously kept at appropriate temp. Immediately after addn of water, the mixt was vigorously agitated with an elec stirrer. Aliquots were withdrawn at certain time intervals with a pipet equipped with a filter, and the concn of solute was detd spectrophotometrically at 258 mµ.

SOURCE AND PURITY OF MATERIALS: Sulfanilamide was a comm product of J.P.

grade. The most stable polymorphic modification was used.

Redistd water was used.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.05°C (authors.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: One temperature: 20°C ORIGINAL MEASUREMENTS: Likhol'ot, N. M. Farm. Zh. (Kiev) 1965, 20(5), 44-6. PREPARED BY: R. Piekos

EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at 20° C is 0.528 g/100 ml (3.07 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier described method was employed (1) whereby a small excess of sulfanilamide was equilibrated with 20 ml of water for 8 h is a 50-ml test tube. Aliquots were removed through a filter and sulfanilamide was assayed bromatometrically.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.1°C (authors).

REFERENCES:

 Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(8), 21.

COMPONENTS: ORIGINAL MEASUREMENTS: Kňažko, L. Farm. Obzor 1966, 35, (1) Benzenesulfonamide, 4-amino-298-311. (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) Water; H₂0; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at $20\,^{\circ}\text{C}$ is 0.575 g/100 ml water $(3.34 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler}).$ AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: Sulfanilamide conformed to the require-Sulfanilamide was added in small portions ments of the Czechoslovak Pharmacopeia 2 to a known volume of water until reaching (1954), Suppl. 1959. Purity of the satn. The equilibration time was 3-4 h under stirring. The temp was held const water was not specified. by means of the Hoppler ultrathermostat. ESTIMATED ERROR: Nothing specified. REFERENCES:

35 COMPONENTS: ORIGINAL MEASUREMENTS: Yamazaki, M.; Aoki, M.; Kamada, A.; (1) Benzenesulfonamide, 4-amino-Yata, N. Yakuzaigaku 1967, 27(1), (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) Water; H₂0; [7732-18-5] 37-40. VARIABLES: PREPARED BY: One temperature: 30°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at 30°C is 61.0 mmol/L (10.50 g dm⁻³, compiler). AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: Sulfanilamide (0.5 g) was placed in an Nothing specified. L-shaped tube together with 20 ml of water. The mixt was shaken in a thermostat until equilibrium was attained. The sulfanilamide was then assayed in the supernatant spectrophotometrically at 545 nm on a Beckman DU spectrophotometer. The results were ESTIMATED ERROR: taken from a calibration graph. Soly: not specified. Temp: +1°C (authors). REFERENCES:

COMPONENTS: (1) Benzensulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: Temperature ORIGINAL MEASUREMENTS: Ito, K.; Sekiguchi, K. Chem. Pharm. Bull. 1967, 15(4), 420-6.

EXPERIMENTAL VALUES:

t/°c	Solubility	
ι, σ	10 ³ mol dm ⁻³ solution	g dm ^{-3^a}
20	3.622	0.6237
25	4.711	0.8112
30	5.988	1.0311
35	7.703	1.3265

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The earlier described method (1) was used; in a 200-ml egg-plant type flask, immersed in a thermostat, an excess of sulfanilamide was placed with 100 ml of distd water which was previously kept at appropriate temp. Immediately after addn of water the mixt was vigorously agitated by an elec stirrer. Aliquots were withdrawn at certain time intervals with a pipet equipped with a filter, and the concn of solute was detd spectrophotometrically at 258 mµ.

SOURCE AND PURITY OF MATERIALS:

Polymorphic modifications of sulfanilamide (source not specified) were prepd by the method of Watanabe (2). Distd water was used.

ESTIMATED ERROR:

Nothing specified.

- 1. Sekiguchi, K.; Ito, K. Chem. Pharm. Bull. 1965, 13(4), 405.
- Watanabe, A.; Kamio, H. Yakugaku Zasshi 1942, 62 501.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: One temperature: 20°C EXPERIMENTAL VALUES: ORIGINAL MEASUREMENTS: Gusyakov, V.P.; Likhol'ot, N.M.; Kutna, I.M. Farm. Zh. (Kiev) 1967, 22(3) 34-9. PREPARED BY: R. Piekos

Solubility of sulfanilamide in water at 20° C is 0.53 g/100 ml (3.1 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide in water was equilibrated for 24 h in an ampul immersed in a water thermostat. Aliquots of the satd soln were withdrawn through a filter and the sulfanilamide content was assayed in the filtrate photometrically.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of the State Pharmacopeia IX.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified. Temp: +0.1°C (authors).

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino-	Gusyakov, V.P.; Likhol'ot, N.M.;
(sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Kutna, I.M. Farm. Zh. (Kiev) 1968, 23(6)
(2) Water; H ₂ O; [7732-18-5]	56-61.
VARIABLES:	PREPARED BY:
One temperature: 21-25°C	R. Piekos
one temperature: 21-25 C	r. Flexos
EXPERIMENTAL VALUES:	
Solubility of sulfanilamide in water at	room temperature (21-25°C)
is 0.528 g/100 ml (3.07 x 10 ⁻² mol dm ⁻³ ,	compiler).
18 0.320 g/100 ml (3.07 x 10 mol dm ,	Complication.
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AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Small quantities (2-4 mg) of sulfanila-	Sulfanilamide: neither source nor
mide were added to a known quantity of	purity was specified. Purity of the
	1
water under stirring until satn was	water was not specified.
achieved.	
	ESTIMATED ERROR:
	Nothing specified.
	REFERENCES:

COMPONENTS: ORIGINAL MEASUREMENTS: (1) Benzenesulfonamide, 4-amino-Shkadova, A.I. (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] Farm. Zh. (Kiev) 1969, 24(3), 39-41. (2) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at 20° C is 3.06×10^{-2} mol/kg (0.53 g/100 g, compiler). AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: A satd aqueous solution of sulfanilamide Purity of sulfanilamide conformed to was equilibrated in a water thermostat the requirements of the State Pharmaat 20±0.1°C. The concn of sulfanilamide copeia IX. was detd bromatometrically. Distd water was used. ESTIMATED ERROR: Soly: not specified. Temp: $\pm 0.1^{\circ}$ C (author). REFERENCES:

COMPONENTS: ORIGINAL MEASUREMENTS: Rohdewald, P. Pharm. Ztg. 1971, No. 38 (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] 1342-4. (2) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in water at 20° C is 0.296_{7} g/50 ml (3.447 x 10^{-2} mol dm⁻³, compiler). AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: The source and purity of the materials The soln was equilibrated by agitation for 2 h at 20°C and the sulfanilamide was was not specified. assayed by differential gravimetric analysis. No details were given. ESTIMATED ERROR: Soly: mean std deviation 68.3% of results deviating by S g), S = 0.028; relative std deviation 9.37%; no of detns 131 (author). Temp: ±0.05°C (author). REFERENCES:

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Water; H₂O; [7732-18-5] VARIABLES: Temperature ORIGINAL MEASUREMENTS: Burger, A. Pharm. Ind. 1973, 35, 626-33.

EXPERIMENTAL VALUES:

t/ ^o C	Saturation solubility, C	a, of crystalline form II
	mg/100 ml solution	mol dm ^{-3^b}
40.0	1440	0.084
45.0	1870	0.109
50.0	2390	0.140
55.0	3080	0.180
60.0	3950	0.230
65.0	5100	0.296
70.0	6600	0.383
75.0	8900	0.517

 $^{^{}a}C_{s} = [HA] + [A]$, where [HA] is the molar concentration of the dissolved, undissociated molecules of sulfanilamide and [A] is the concentration of the dissolved anion of sulfanilamide.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was assayed spectrophotometrically at 258.5 nm using a Zeiss PMQ II spectrophotometer and a 1/15 M phosphate buffer of pH 7.00 ($E_{1~\rm cm}^{1\%}$ = 945).

SOURCE AND PURITY OF MATERIALS:

Form II of sulfanilamide, mp 156°C, was obtained by the known procedure (1). Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.1°C (author).

REFERENCES:

 Burger, A. Sci. Pharm. 1973, 41, 290 and 303.

bCalculated by compiler.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino-	Kitao, K.; Kubo, K.; Morishita, T.;
(sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Yata, N.; Kamada, A. Chem. Pharm. Bull.
(2) Water; H ₂ O; [7732-18-5]	<u>1973</u> , <i>21</i> , 2417-26.
2	
VARIABLES:	PREPARED BY:
One temperature: 37°C	R. Piekos
EXPERIMENTAL VALUES:	
Solubility of sulfanilamide in water at	37°C is 1.41 g/100 cm ⁻³ solution.
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	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Sulfanilamide was assayed by diazotiza-	Comm available sulfanilamide was
tion. No details were given.	used as supplied.
	Deionized water was used.
	ESTIMATED ERROR:
	Soly: not specified.
	Temp: $\pm 1^{\circ}$ C (authors).
	REFERENCES:

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sekiguchi, K.; Tsuda, Y.; Kanke, M. Chem. Pharm. Bull. 1975, 23, 1353-62.

VARIABLES:

PREPARED BY:

Temperature

R. Piekos

EXPERIMENTAL VALUES:

t/°C	Solubility			
t/ C	α - form		β-form	
	g/100 g soln	10 ³ mol kg ⁻¹ water ^a	g/100 g soln	10 ³ mol kg ⁻¹ water ^a
20	0.68	0.3976	0.615 ^b	0.3593
25	0.93	0.5451	0.799 ^b	0.4677
30	1.15	0.6756	1.024 ^b	0.6008
35	1.49	0.8783	1.299	0.7643
40	1.90	1.1247	1.639	0.9676
45	2.17	1.2881	2.069	1.2269
50	3.33	2.0004	2.562	1.5269

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A sufficient amt of sample powder was placed in a 50 g of distd water in a dissoln measurement cell and stirred at 600 rpm. At appropriate time intervals samples were taken by glass syringes until the concn attained equilibrium. The sample solns were immediately filtered through a 0.45- μ membrane filter. The filtrate was weighed and dild for spectrophotometric assay at 225 nm on a Hitachi-139 UV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

The α -form of sulfanilamide was obtained by crystn of a comm product of the JP VIII grade from isoamyl, n-amyl or n-butyl alcohol, by holding the warm soln at room temp or cooling it immediately. The β -form was crystd from EtOH by gradual cooling of its warm soln to room temp and maintaining for 2-3 days. The forms were characterized by instrumental methods.

Distilled water was used.

ESTIMATED ERROR:

Nothing specified.

^bCalculated from the equation $C_s' = C_s \times \frac{dE'/dt}{dE/dt}$, where C_s and dE/dt, and C_s' and dE'/dt are the solubility and dissolution rate of the stable (β) and metastable (α) crystalline forms of sulfanilamide, respectively.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kaneniwa, N.; Watari, N.; Iijima, H. Chem. Pharm. Bull. 1978, 26(9), 2603-14.

VARIABLES:

One temperature: 37°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in water at 37° C is 14.9 mg/ml solution (8.65 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was placed in a flask contg 25 ml of water. The flask was shaken (2 strokes/s at the amplitude of 3 cm) in a thermostatically controlled water bath at 37° C. One-ml sample was withdrawn every 6 h (total equilibration period was 3-5 days) using a warmed Millipore filter syringe with a filter pore size of 0.45 μ (Millipore HAWP 01300) and the filtrate was dild with water and assayed spectrophotometrically (1).

SOURCE AND PURITY OF MATERIALS:

Commercial sulfanilamide of the Japanese Pharmacopeia grade and distd water were used.

ESTIMATED ERROR:

Soly: not specified.

Temp: $\pm 0.05^{\circ}$ C (authors).

REFERENCES:

Kaneniwa, N.; Watari, N.
 Chem. Pharm. Bull. 1974, 22, 1699.

COMPONENTS: (1) Benzenesulfonamide, 4-amino- (sulf-anilamide); C₆H₈N₂O₂S; [63-74-1]

H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Goto, S.; Komatsu, M.;
Tagawa, K.; Kawata, M.
Chem. Pharm. Bull. 1983, 31(1), 256-61.

VARIABLES:

(2) Water;

PREPARED BY:

Temperature

R. Piekos

EXPERIMENTAL VALUES:

t/°C	Solubility		
	g/1	$10^2 \text{ mol} \text{ dm}^{-3} \text{ a}$	
37	17.10	9.930	
55	39.86	23.150	

^aCalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A 3 g sample of sulfanilamide powder was accurately weighed into a 20-ml ampule and 10 ml of water was added. the ampule was sealed, placed in a const temp (37° or 55°C) bath and allowed to stand for several days. The equilibrium concn of the solute was measured spectrophotometrically at 540 nm after diazotization with the 0.1% Tsuda reagent (1).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide had mp 163-6°C.

The purity of water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

Tsuda, K.; Matsunaga, S.
 Yakugaku Zasshi 1942, 62, 362.

COMPONENTS: (1) Benzenesulfonamide, 4-amino-(sulfanilimide); $C_6H_8N_2O_2S$; [63-74-1] (2) Hydrochloric acid; HC1; [7647-01-0]

Paal, T.; Regosz, P.

ORIGINAL MEASUREMENTS:

Gyogyszereszet 1973, 17, 59-63.

VARIABLES:

Concentration of HCl

(3) Water; H₂O; [7732-18-5]

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Concentration of the most concentrated real
of HCl	solution of sulfanilamide at 26°C
N	mol dm ⁻³ solvent
5	0.3
1	1.15
0.1	0.13

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns were prepd by addn of increasing amts of aq HCl to weighed quantities of sulfanilamide. After the dissoln was completed, the soln was stirred with a magnetic stirrer and allowed to stand for 24 h. The soln was considered stable, if it remained clear during a 24-h period.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was a com product (source not specified), doubly crystd from 98% EtOH. Its purity was > 99.5% as detd by diazotization. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: accuracy +10% (authors).

Temp: $\pm 3^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Perchloric acid; HClO₄; [7601-90-3]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Paal, T.; Regösz, P. Gyögyszerészet 1973, 17,59-63.

VARIABLES:

Concentration of $HC10_4$

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Concentration of the most concentrated real
of HC10 ₄	solution of sulfanilamide at 26°C
N .	mol dm ⁻³ solvent
5	0.4 (1.75) ^a
1	1.15
0.1	0.13

^aConcentration of the most concentrated metastable solution that could be prepared without precipitation of the solute.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns were prepd by addn of increasing amts of aq HClO₄ to weighed quantities of sulfanilamide. After the dissoln was completed, the soln was stirred with a magnetic stirrer and allowed to stand for 24 h. The soln was considered stable, if it remained clear during a 24-h period. If the solute fell out from the clear soln, the soln was considered metastable.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was a comm product (source not specified), double crystd from 98% EtOH. Its purity was > 99.5% as detd by diazotization. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: accuracy $\pm 10\%$ (authors).

Temp: $\pm 3^{\circ}$ C (authors).

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Nitric acid HNO₃; [7697-37-2] (3) Water; H₂O; [7732-18-5] VARIABLES: Concentration of HNO₃ ORIGINAL MEASUREMENTS: Paal, T.; Regösz, P. Gyögyszereszet 1973, 17, 59-63 PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Concentration of HNO ₃	Concentration of the most concentrated real solution of sulfanilamide at 26°C mol dm ⁻³ solvent
5	0.15
1	1.15
0.1	9×10^{-2}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns were prepd by addn of increasing amts of aq HNO₃ to weighed quantities of sulfanilamide. After the dissolution was completed, the soln was stirred with a magnetic stirrer and allowed to stand for 24 h. The soln was considered stable, if it remained clear during a 24-h period.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was a comm product (source not specified), doubly crystd from 98% EtOH. Its purity was > 99.5% as detd by diazotization. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: accuracy $\pm 10\%$ (authors). Temp: $\pm 3^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanil-amide); C₆H₈N₂O₂S; [63-74-1]
- (2) Lithium chloride; LiC1; [7447-41-8]
- (3) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likholet, M. A.; Gusyakov, V. P. Med. Prom. SSSR <u>1963</u>, 17(5), 28-31.

VARIABLES:

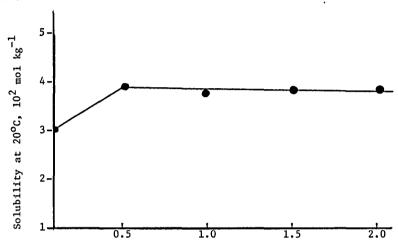
Concentration of LiC1

T/K = 293

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of LiC1
mol kg-1

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a LiCl soln was placed and a small excess of sulfanilamide. the mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, diluted, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. LiCl was purified by a recommeded procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.10C (authors).

REFERENCES:

 Karysakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

COMPONENTS: ORIGINAL MEASUREMENTS: Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (1) Becher, R.; Leya, S. Experientia 1946, 2, 459-60. (2) Sodium chloride; NaCl; [7647-14-5] (3) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 18-19°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in a 5% NaCl solution at room temperature $(18-19^{\circ}C)$ is 610 mg% $(3.5 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler})$. AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: After standing for more than two days the Nothing specified. soln of sulfanilamide was filtered and sulfanilamide was assayed in the filtrate colorimetrically by the method of Druey and Oesterheld (1). ESTIMATED ERROR: Nothing specified. REFERENCES: 1. Druey, J.; Oesterheld, G. Helv. Chim. Acta 1942,25, 753.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Sodium chloride; NaCl; [7647-14-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Langecker, H.

Arch. Exptl. Fath. Pharmakol. 1948, 205, 291-301.

VARIABLES:

One temperature: 37°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in physiological saline (0.9% w/w NaCl solution) at 37° C is 1098 mg% (6.376 x $10^{-2} \text{ mol dm}^{-3}$, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was added to physiological saline and boiled for 1 h in a sealed ampul followed by keeping the ampul at 37°C. Sulfanilamide was assayed colorimetrically by the method of Bratton and Marshall (1) using a Havemann colorimeter (2), and by microanal detn of the solid residue.

SOURCE AND PURITY OF MATERIALS:

Source and purity of sulfanilamide was not specified.

The water was free of oxidants.

ESTIMATED ERROR:

Nothing specified.

- Bratton, A.G.; Marshall, E.K. J. Biol. Chem. 1939, 128, 537.
- 2. Havemann, R. Klin. Wochenschr. 1940, p. 503.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Sodium chloride; NaCl; [7647-14-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Matsuura, H.; Sekiguchi, K. Yakuzaigaku 1960, 20, 213-18

VARIABLES:

Concentration of NaCl

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility of sulfanilamide at 25°C		
of NaCl Formula weight/L	g/100 ml	10 ² mo1/L	
0	0.782	4.55	
0.5	0.720	4.18	
1.0	0.623	3.77	
1.5	0.580	3.37	
2.0	0.470	2.94	
2.5	0.387	2.25	
3.0	0.244	1.58	

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added to NaCl solns in such amts as to obtain satn. The mixts were agitated for more than 9 h. Aliquots were withdrawn with a pipet fitted with a filter and sulfanilamide was detd spectrophotometrically using the Tsuda reagent for producing color.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was of the Japanese Pharmacopeia grade and was recrystd. NaCl was an extra pure reagent conforming to the first degree of the Japanese Industrial Standard.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the error was 1.5% (authors). Temp: $\pm 0.05^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino- (sulfanil-amide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Sodium chloride; NaCl; [7647-14-5]
- (3) Water; H₂0; [7732-18-5]

VARIABLES:

Concentration of NaCl

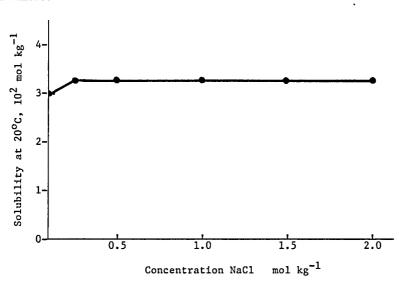
ORIGINAL MEASUREMENTS:

Likholet, M. N.; Gusyakov, V. P. *Med. Prom. SSSR* 1963, 17(5), 28-31.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a NaCl soln was placed and as small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, diluted and assayed spectrophotometrically at 285 nm on SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. NaCl was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C (authors).

REFERENCES:

 Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sodium bromide; NaBr; [7647-15-6]
- (3) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Matsuura, H.; Sekiguchi, K. Yakuzaigaku 1960, 20, 213-18.

VARIABLES:

Concentration of NaBr

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility of sulf	anilamide at 25°C
of NaBr Formula weight/L	g/100 ml	10 ² mo1/L
0	0.782	4.55
0.448	0.786	4.57
0.673	0.778	4.52
0.345	0.784	4.58
2.018	0.786	4.56
2.690	0.788	4.58
3.363	0.767	4.45
3.766	0.750	4.36
5.380	0.692	4.02

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added to NaBr solns in such amts as to obtain satn. The mixts were agitated for more than 9 h. Aliquots were withdrawn with a pipet fitted with a filter and sulfanilamide was detd spectrophotometrically using the Tsuda reagent for producing color.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was of the Japanese Pharmacopeia grade and was recrystd. NaBr was an extra pure reagent conforming to the first degree of the Japanese Industrial Standard.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the error was 1.5% (authors).

Temp: $\pm 0.05^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sodium iodide; NaI; [7681-82-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Matsuura, H.; Sekiguchi, K. Yakuzaigaku 1960, 20, 213-18.

VARIABLES:

Concentration of NaI

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of NaI g/100 ml Formula wt/L		Solubility of sulfanilamide at 25 g/100 ml 10 ² mol/L		
0	0	0.782	4.55	
0.683	0.456	0.858	4.98	
4.10	0.941	0.895	5.20	
18.07	1.205	0.963	5.59	
23.13	1.543	1.044	6.06	
33.35	2.225	1.219	7.08	
43.25	2.885	1.343	7.97	

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added to NaI solns in such amts as to obtain satn. The mixts were agitated for more than 9 h. Aliquots were withdrawn with a pipet fitted with a filter and sulfanilamide was detd spectrophotometrically using the Tsuda reagent for producing color.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was of the Japanese Pharmacopeia grade and was recrystd. NaI was an extra pure reagent conforming to the first degree of the Japanese Industrial Standard.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the error was 1.5% (authors).

Temp: +0.05°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Carbonic acid, monosodium salt; NaHCO₃; [144-55-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Takubo, T.; Matsumaru, H.; Tsuchiya, S.; Hiura, M. Chem. Pharm. Bull. 1973, 21(7), 1440-5.

VARIABLES:

One temperature: 37°C; one pH: 8.4

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a NaHCO $_3$ solution (1.680 g NaHCO $_3$ /100 ml water) of pH 8.4 at 37°C is 13.82 mg/ml solution^a (8.026 x 10^{-2} mol dm⁻³ solution, compiler).

^aNumerical value to the graphical one was given by one of the authors (S.T.) in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Aliquots of the NaHCO₃ soln were placed in glass-stoppered flasks with excess of sulfanilamide. The flasks were allowed to stand at 37±1°C and shaken vigorously for 4 h until equilibrium was established. One ml of the supernatant was removed by means of a filter pipet and the sulfanilamide was assayed by the previously reported method (1).

SOURCE AND PURITY OF MATERIALS:

The sulfanilamide was of pharmaceutical grade. Source and purity of ${\tt NaHCO}_3$ was not specified.

Distd water was used.

ESTIMATED ERROR:

Soly and pH: not specified. Temp: $\pm 1^{\circ}$ C (authors).

REFERENCES:

 Takubo, T.; Tsuchiya, S.; Hiura, M. Yakuzaigaku 1971, 31, 298.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $c_6H_8N_2O_2S$; [63-74-1]
- (2) Carbonic acid, disodium salt; Na₂CO₃; [497-19-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Takubo, T.; Matsumaru, H.; Tsuchiya, S.; Hiura, M. Chem. Pharm. Bull. 1973, 21(7), 1440-5.

VARIABLES:

One temperature: 37°C; one pH: 11.3

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a $\rm Na_2CO_3$ solution (2.120 g $\rm Na_2CO_3/100$ ml water) of pH 11.3 at 37°C is 15.39 mg/ml solution^a (8.937 x $\rm 10^{-2}$ mol dm⁻³ solution, compiler).

^aNumerical value to the graphical one was given by one of the authors (S.T.) in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Aliquots of the Na₂CO₃ soln were placed in glass-stoppered flasks with excess of sulfanilamide. The flasks were allowed to stand at 37±1°C and shaken vigorously for 4 h until equilibrium was established. One ml of the supernatant was removed by means of a filter and the sulfanilamide was assayed by the previously reported method (1).

SOURCE AND PURITY OF MATERIALS:

The sulfanilamide was of pharmaceutical grade. Source and purity of $\mathrm{Na_2CO_3}$ was not specified.

Distd water was used.

ESTIMATED ERROR:

Soly and pH: not specified. Temp: $\pm 1^{\circ}$ C (authors).

REFERENCES:

 Takubo, T.; Tsuchiya, S.; Hiura, M. Yakuzaigaku 1971, 31, 298.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Phosphoric acid, disodium salt; Na₂HPO₄; [7558-94-4]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

Arch. Dermatol. Syphilis <u>1942</u>, 183, 90-116.

VARIABLES:

One temperature: ca 20°C; one pH: 8.74

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 0.705 M (10%) Na_2HPO_4 solution of pH 8.74, at room temperature (about $20^{\circ}C$), is 0.57 g% (3.3 x 10^{-2} mol dm⁻³ solution, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide (0.5 g) was dissolved in 10 cm³ of a 0.705M (10%) Na₂HPO₄ soln, shaken for 2 h, and filtered. A 1-cm³ aliquot of the filtrate was then withdrawn, cooled, acidified with 2N HCl, and the sulfanilamide content was detd colorimetrically by the method of Marshall modified by Kimmig (1) using an Autenrieth colorimeter. The pH was detd on an ultraionograph using a glass electrode.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was the product manufd by Schering AG. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: precision +5% (author)

Temp: not specified

pH: +0.05 pH unit (author)

REFERENCES:

Kimmig, J. Arch. Dermatol. 1938, 176,
 722; Erg. Hyg. 1941, 24, 398.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}{}_{8}{}^{N}{}_{2}{}^{O}{}_{2}{}^{S}$; [63-74-1]
- (2) Potassium chloride; KC1; [7747-40-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V.P.; Likhol'ot, N.M.

Farm. Zh. (Kiev) 1960, 15(3), 21-4.

VARIABLES:

PREPARED BY:

Concentration of KCl

R. Piekos

EXPERIMENTAL VALUES:

Concentration of KCl	Solubility at 20°C		
Weight %	g/100 g water	10 ² mol kg ^{-1^a}	
0.74	0.656	3.81	
1.82	0.662	3.84	
3.59	0.700	4.07	
6.93	0.763	4.43	
12.97	0.771	4.48	
15.70	0.818	4.75	

acalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 h in a 50-ml test tube with 20 ml of aqueous KCl soln. Aliquots were taken with a pipet fitted with a filter. Sulfanilamide was detd in the filtrate at 285 nm using a SF-4 spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was crystd three times from hot water. Its purity conformed to the requirements of the State Pharmacopeia VIII. KCl was doubly crystd. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the accuracy corresponded to that of colorimetric detns (authors).

Temp: not specified.

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Potassium chloride; KCl; [7447-40-7]
- (3) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likholet, N. M.; Gusyakov, V. P. Med. Prom. SSSR 1963, 17(5), 28-31.

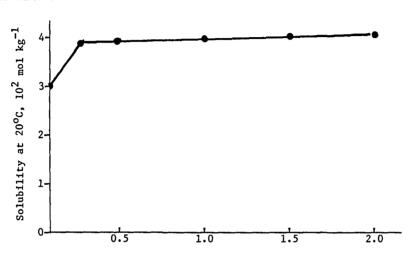
VARIABLES:

Concentration of KC1

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of KC1 mol kg-1

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-m1 tightly closed ampuls in which 20~m1of a KCl was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. KCl was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several

times (authors). Temp: $\pm 0.1^{\circ}$ C (authors).

REFERENCES:

1. Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S};$ [63-74-1]
- (2) Potassium bromide; KBr; [7759-02-3]
- (3) Water; H₂O [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V.P.; Likhol'ot, N.M.

Farm. Zh. (Kiev) 1960, 15(3) 21-4

VARIABLES:

Concentration of KBr

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

oncentration of KBr	Solubility at 20°C		
Weight %	g/100 g water	10 ² mol kg ⁻¹ ^a	
1.17	0.738	4.29	
2.88	0.739	4.29	
5.61	0.798	4.63	
10.63	0.844	4.90	
19.22	0.993	5.77	

acalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 h in a 50-ml test tube with 20 ml of aqueous KBr soln. Aliquots were taken with a pipet fitted with a filter. Sulfanilamide was detd in the filtrate at 285 nm using a SF-4 spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was crystd three times from hot water. Its purity conformed to the requirements of the State Pharmacopeia VIII. KBr was doubly crystd. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the accuracy corresponded to that of colorimetric detns (authors).

Temp: not specified.

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Potassium bromide; KBr; [7758-02-3]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likholet, N. M.; Gusyakov, V. P. Med. Prom. SSSR <u>1963</u>, 17(5), 28-31.

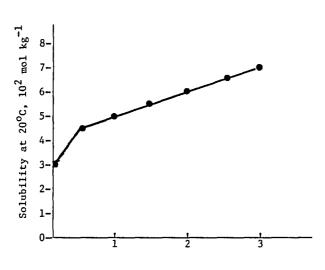
VARIABLES:

Concentration of KBr

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of KBr mol kg⁻¹

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a KBr soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. KBr was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C (authors).

REFERENCES:

 Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Potassium iodide; KI; [7681-11-0]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V.P.; Likhol'ot, N.M.

Farm. Zh. (Kiev) 1960, 15(3) 21-4

VARIABLES:

Concentration of KI

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of KI	Solubility	at 20°C
Weight %	g/100 g water	10 ² mol kg ^{-1^a}
1.63	0.724	4.20
3.98	0.762	4.43
7.66	0.833	4.84
14.23	1.023	5.94

acalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 h in a 50-ml test tube with 20 ml of aqueous KI soln. Aliquots were taken with a pipet fitted with a filter. Sulfanilamide was detd in the filtrate at 285 nm using a SF-4 spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was crystd three times from hot water. Its purity conformed to the requirements of the State Pharmacopeia VIII. KI was doubly crystd. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the accuracy corresponded to that of colorimetric detns (authors).

Temp: not specified.

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Potassium iodide; KI; [7681-11-0]
- (3) Water; H₂0; [7732-18-5]

VARIABLES:

Concentration of KI

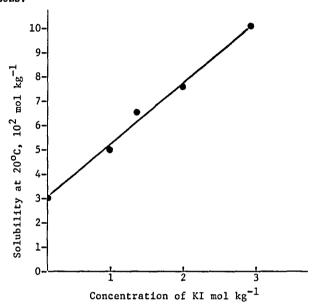
ORIGINAL MEASUREMENTS:

Likholet, M. N.; Gusyakov, V. P. Med. Prom. SSSR 1963, 17(5), 28-31.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a KI soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. KI was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C (authors).

REFERENCES:

 Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Potassium thiocyanate; KSCN; [333-20-0]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V.P.; Likhol'ot, N.M. Farm. Zh. (Kiev) 1960, 15(3), 21-4.

VARIABLES:

Concentration of KSCN

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of KSCN	Solubility at 20°C		
Weight %	g/100 g water	10 ² mol kg ^{-1a}	
0.96	0.757	4.40	
2.37	0.784	4.55	
4.63	0.851	4.94	
8.85	1.101	6.39	

acalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 h in a 50-ml test tube with 20 ml of aqueous KSCN soln. Aliquots were taken with a pipet fitted with a filter. Sulfanilamide was detd in the filtrate at 285 nm using a SF-4 spectro-photometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was crystd three times from hot water. Its purity conformed to the requirements of the State Pharmacopeia VIII. KSCN was doubly crystd. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: the accuracy corresponded to that of colorimetric detns (authors).

Temp: not specified.

- (1) Benzenesulfonamide, 4-amino- (sulfanil-amide); C₆H₈N₂O₂S; [63-74-1]
- (2) Thiocyanic acid, potassium salt; KCNS; [333-20-0]
- (3) Water; H₂0; [7732-18-5]

VARIABLES:

Concentration of KCNS

ORIGINAL MEASUREMENTS:

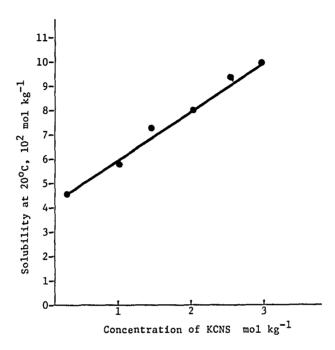
Likholet, M. N.; Gusyakov, V. P.

Med. Prom. SSSR <u>1963</u>, 17(5), 28-31.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a KCNS soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. KCNS was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C

REFERENCES:

 Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Phosphoric acid, monopotassium salt; KH₂PO₄; [7778-77-0]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kruger-Thiemer, E.

Arch. Dermatol. Syphilis 1942, 183, 90-116.

VARIABLES:

PREPARED BY:

One temperature: ca 20°C; one pH: 4.37 R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 0.735 M (10%) $\mathrm{KH_2PO_4}$ solution of pH 4.37, at room temperature (about 20° C) is 0.572 g% (3.32 x 10^{-2} mol dm⁻³ solution, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide (0.5 g) was dissolved in 10 cm^3 of a 0.735 M(10%) KH_2PO_L soln, shaken for 2 h, and filtered. A 1-cm aliquot of the filtrate was then withdrawn, cooled, acidified with 2 N HCl, and the sulfanilamide content was detd colorimetrically the method of Marshall modified by Kimmig (1) using an Authenreith colorimeter. The pH was detd on an ultraionograph using a glass electrode.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was the product manufd by Schering AG. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: precision ±5% (author)

Temp: not specified

pH: +0.05 pH unit (author)

REFERENCES:

1. Kimmig, J. Arch. Dermatol. 1938, 176, 722; Erg. Hyg. 1941, 24, 398.

- (1) Benzenesulfonamide, 4-amino- (sulfanil-amide); C₆H₈N₂O₂S; [63-74-1]
- (2) Ammonium chloride; NH₄C1; [12125-02-9]
- (3) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likholet, M. N.; Gusyakov, V. P.

Med. Prom. SSSR 1963, 17(5), 28-31.

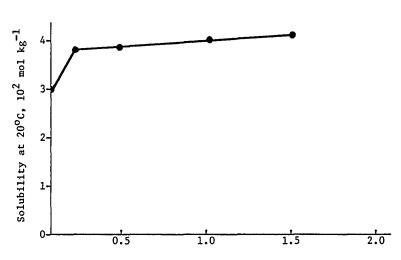
VARIABLES:

Concentration of NHAC1

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of NH₄Cl mol kg⁻¹

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a NH₄Cl soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. NH₄Cl was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C (authors).

REFERENCES:

Karyakin, Ya. V.; Angelov, I. I.
 Chistye khimicheskye reaktivy,
 Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Magnesium chloride; MgCl₂; [7786-30-3]
- (4) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likholet, M. N.; Gusyakov, V. P. 1963, 17(5), 28-31. Med. Prom. SSSR

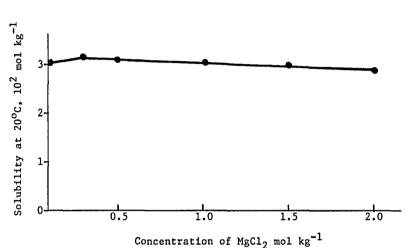
VARIABLES:

Concentration of MgCl₂

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a MgCl₂ soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. MgCl₂ was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C (authors).

REFERENCES:

1. Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino- (sulfanil-amide); C₆H₈N₂O₂S; [63-74-1]
- (2) Calcium chloride; CaCl₂; [10043-52-4]
- (3) Water; H₂O; [7732-18-5]

VARIABLES:

Concentration of $CaCl_2$

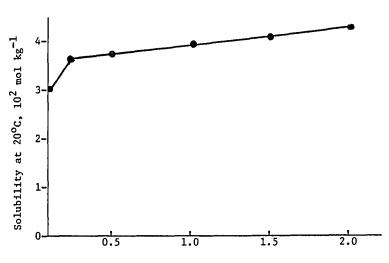
ORIGINAL MEASUREMENTS:

Likholet, M. N.; Gusyakov, V. P. Med. Prom. SSSR 1963, 17(5), 28-31.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of $CaCl_2 mol kg^{-1}$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were prepd in 50-ml tightly closed ampuls in which 20 ml of a CaCl₂ soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pippeted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. CaCl_2 was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1 (authors).

REFERENCES:

 Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy, Moscow, 1955.

VARIABLES:

- (1) Benzenesulfonamide, 4-amino- (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Barium chloride; BaCl₂; [10361-37-2]

Concentration of BaCl₂

(3) Water; H₂0; [7732-18-5]

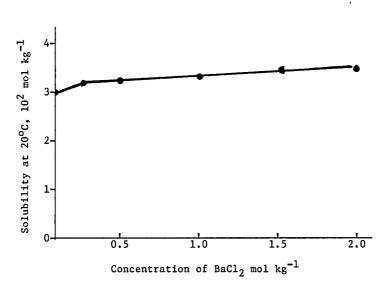
ORIGINAL MEASUREMENTS:

Likholet, M. N.; Gusyakov, V. P. Med. Prom. SSSR <u>1963</u>, *17(5)*, 28-31.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide was prepd in 50-ml tightly closed ampuls in which 20 ml of a BaCl2 soln was placed and a small excess of sulfanilamide. The mixts were equilibrated for 18 h at 20°C. Aliquots were pipetted out through a filter, dild, and assayed spectrophotometrically at 285 nm on a SF-IV spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd. Its purity was 99.22%. $BaCl_2$ was purified by a recommended procedure (1).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated several times (authors).

Temp: ±0.1°C (authors).

REFERENCES:

1. Karyakin, Ya. V.; Angelov, I. I. Chistye khimicheskye reaktivy Moscow, 1955.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- Phosphoric acid, disodium salt; Na₂HPO₄; [7558-94-4]
- (3) Phosphoric acid, monopotassium salt; KH2PO4; [7778-77-0]
- (4) Water; H₂O; [7732-18-5]

VARIABLES:

Temperature, pH

ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

Arch. Dermatol. Suphilis 1942, 183, 90-116.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Composition	οf	1/15M	phosphate
buffer	ജവി	lutions	

Solubility pН room temp (ca 20°C) 37⁰C 10^2 mol dm^{-3} 10^2 mol dm^{-3} g% g% solution^a solutiona Na2HPO4 KH2PO4 %Content 99.0 1.0 0.91 4.944 0.694 4.03 10.0 90.0 0.91 5.906 0.704 4.09 1.220 7.08 61.1 38.9 0.93 7.005 0.698 4.05 1.260 7.32 0.733^b 9.5 0.5 7.51 0.573 3.33 94.7 5.3 0.95 8.018 0.694 4.03

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfamilamide (0.5 g) was dissolved in 10 cm³ of a buffer soln, shaken for 2 h at 20° C (or left for 48 h at 37° C), and filtered at respective temp. A 1-cm3 aliquot of the filtrate was then withdrawn, cooled (dild for expts at 37°C), acidified with 1 $\rm cm^3$ of 2N HCl, and the sulfanilamide content was detd colorimetrically by the method of Marshall modified by Kimmig (1, 2) using an Autenrieth colorimeter. The pH was detd on an ultraionograph using a glass electrode.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was the product manufd by Schering AG. The source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: precision $\pm 5\%$ (author)

Temp: not specified

pH: ± 0.05 pH unit (author)

- 1. Kimmig, J. Arch. Dermatol. 1938, 176, 722.
- 2. Kimmig, J. Erg. Hyg. 1941, 24, 398.

^aCalculated by compiler

bMolar content; 10% buffer solution

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Phosphoric acid, disodium salt; Na₂HPO₄; [7778-94-4]
- (3) Phosphoric acid, monopotassium salt; KH₂PO₄; [7778-77-0]
- (4) Water; H₂O; [7732-18-5]

VARIABLES:

One temperature: 37°C; one pH: 6.9

ORIGINAL MEASUREMENTS:

Kienle, R.H.; Sayward, J.M.

J. Am. Chem. Soc. 1942, 64, 2464-8.

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a buffer solution prepared by mixing together 55.2 cm³ of 1/15 M Na₂HPO₄ with 44.8 cm³ of 1/15 M KH₂PO₄ (pH 6.9, ionic strength calculated from dissociation constants 0.03^a) at 37.0° C is 1.44 g/100 cm³ solution (8.36 x 10^{-2} mol dm⁻³, compiler).

a Not specified for which reactions were the dissociation constants calculated - compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was rotated with the buffer soln usually overnight. Equilibrium was approached from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol dm⁻³ NaNO₂ soln to first blue on a starch – iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrystd from alcohol and from hot water; mp 165.9°C. Titrn with nitrite indicated that the compd was 100.0±0.3% pure. Elemental analysis confirmed this value.

Source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: ± 0.01 g/100 g soln or ± 0.012 x 10^{-3} in mole fraction (authors).

Temp: ±0.02°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide) $C_6H_8N_2O_2S$; [63-74-1] (2) Phosphoric acid, disodium salt;
- Na₂HPO₄; [7558-94-4]
- (3) Phosphoric acid, monopotassium salt; KH_2HPO_4 ; [7778-77-0]
- (4) Water; H₂O; [7732-18-5]

PREPARED BY:

ORIGINAL MEASUREMENTS:

Arch. Exptl. Path. Pharmakol. 1948,

Langecker, H.

205, 291-301.

R. Piekos

VARIABLES:

рΗ

EXPERIMENTAL VALUES:

pH of the 1/15M	Solubility at 37°C		
phosphate buffer	mg%	10 ² mol dm ^{-3^a}	
4.9	1064	6.179	
5.9	1050	6.097	
6.9	1023	5.941	
7.5	1167	6.777	

^aCalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was added to buffer solution and boiled for 1 h in a sealed ampul at 37°C. Sulfanilamide was assayed colorimetrically by the method of Bratton and Marshall (1) using a Havemann colorimeter (2), and by microanal detn of the solid residue.

SOURCE AND PURITY OF MATERIALS:

Source and purity of the materials was not specified.

ESTIMATED ERROR:

Nothing specified.

- Bratton, A.G.; Marshall, E.K. J. Biol. Chem. 1939, 128, 537.
- 2. Havemann, R. Klin. Wochenschr. 1940, p. 503.

	75
COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino-	Yamazaki, M.; Aoki, M.; Kamada, A; Yata, N.
(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]	Yakuzaigaku <u>1967</u> , 27(1), 37-40.
(2) Phosphoric acid, disodium salt; Na ₂ HPO ₄ ; [7558-94-4]	
(3) Phosphoric acid, monopotassium salt:	
кн ₂ Ро ₄ ; [7778-77-0]	
(4) Water; H ₂ 0; [7732-18-5]	
VARIABLES: One temperature: 30°C; one pH: 7.4	PREPARED BY: R. Piekos
EXPERIMENTAL VALUES:	•
Solubility of sulfanilamide in a phospha	
at 30° C is 57.0 mmo1/L (9.815 g dm ⁻³ , co	ompiler).
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE: Sulfanilamide (0.5 g) was placed in an L-	SOURCE AND PURITY OF MATERIALS:
shaped tube together with 20 ml of the buffer	Nothing specified.
soln. The mixt was shaken in a thermostat	
until equilibrium was attained. The sulfa-	
nilamide was then assayed in the supernatant	
spectrophotometrically at 545 nm on a	
Beckmann DU spectrophotometer. The results	1
were taken from a calibration graph.	
and the same and the same same same	
	ESTIMATED ERROR:
}	Soly and pH: not specified.
	Temp: ±1°C (authors).
	REFERENCES:

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Carbonic acid, disodium salt; Na₂CO₃; [497-19-8]
- (3) Carbonic acid, monosodium salt; NaHCO₃; [144-55-8] (4) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Takubo, T.; Matsumaru, H.; Tsuchiya, S.; Hiura, M. Chem. Pharm. Bull. 1973, 21(7) 1440-5.

VARIABLES:

pН

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Na ₂ CO ₃	NaHCO ₃		Solubil:	ity at 37 ⁰ C
g/100 ml water	g/100 ml water	pН	mg/ml soln ^a	10 ² mol dm ⁻³ soln ^b
0.212	1.512	9.1	13.99	8.124
0.848	1.008	9.8	14.95	8.682
1.908	0.168	10.7	14.26	8.281

^aNumerical values to the graphical data were given by one of the authors (S.T.) in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Aliquots of the buffer solns were placed in glass-stoppered flasks with excess of sulfanilamide. The flasks were allowed to stand at 37±1°C and shaken vigorously for 4 h until equilibrium was established. One ml of the supernatant was removed by means of a filter pipet and the sulfanilamide was assayed by the previously reported method (1).

SOURCE AND PURITY OF MATERIALS:

The sulfanilamide was of pharmaceutical grade. The source and purity of Na2CO2 and NaHCO3 was not specified. Distd water was used.

ESTIMATED ERROR:

Soly and pH: not specified. Temp: $\pm 1^{\circ}$ C (authors).

REFERENCES:

1. Takubo, T.; Tsuchiya, S.; Hiura, M. Yakuzaigaku 1971, 31, 298.

^bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Hydrochloric acid; HCl; [7647-14-5]
- (3) Sodium chloride; NaC1; [7647-14-5]
- (4) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Avico, U.; Cavazutti, G.; di Francesco, R.; Signoretti Ciranni, E.; Zuccaro, P.

Farmaco, Ed. Pratica 1975, 30(1), 40-6.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of amorphous sulfanilamide in equimolal ${\rm t/}^{\rm O}{\rm C}$ NaCl solutions containing a small excess of HCl

	g/100 g water	10 ² mol kg ⁻¹ water ^a
25	4.72	2.74
35	6.50	3.78
40	7.40	4.30

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A soln of sulfanilamide-HCl was added to an NaOH soln contg stoichiometric quantity of the base to neutralize the HCl salt. A small excess of HCl was used to dissolve the sulfanilamide. The neutralization was carried out in a thermostat and the pH of the mixt was maintained close to that of a satd aq sulfanilamide soln. The procedure was repeated using various initial concns of the reagents to find the max concn of sulfanilamide at which no pptn occurred.

SOURCE AND PURITY OF MATERIALS:

Source and purity of sulfanilamide was not specified. The mp of crystalline sulfanilamide was 164.5-6.5°C.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
 (2) Hydrochloric acid; HCl; [7647-01-0]
- (3) Potassium chloride; KC1; [7447-40-7]
- (4) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kienle, R. H.; Sayward, J. M.

J. Am. Chem. Soc. 1942, 64, 2464-8.

VARIABLES:

pH; ionic strength

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

pH of HC1-KC1		Solubility at 37.0°C		
	Ionic strength ^a	g/100 cm ³	mol dm ^{-3^b}	
1.2	0.12	4.07	0.236	
2.2	0.06	1.57	0.091	

^acalculated from dissociation constants.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was rotated with a buffer soln usually overnight. Equilibrium was approached usually from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol dm⁻³ NaNO₂ soln to first blue on starch - iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrystd from alcohol and from hot water; mp 165.9°C. Titrn with nitrite indicated that the compd was 100+0.3% pure. Elemental analysis and mixed mp detns confirmed this value. Purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: ± 0.01 g/100 g soln or ± 0.012 x 10^{-3} in mole fraction (authors).

Temp: $\pm 0.02^{\circ}$ C (authors).

bcalculated by compiler

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Boric acid, H₃BO₃; [10043-35-3] (3) Potassium chloride; KC1; [7447-40-7]

- (4) Sodium hydroxide; NaOH; [1310-73-2]
- (5) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kienle, R. H.; Sayward, J. M.

J. Am. Chem. Soc. 1942, 64, 2464-8.

VARIABLES:

pH; ionic strength

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

pН		Solubility at 37.0°C		
of borate buffer	Ionic strength ^a	g/100 cm ³ solution	10 ² mol dm ^{-3^b}	
9.4 ^c	0.08	1.55	9.00	
9.7 ^d	0.09	1.60	9.29	

^aCalculated from dissociation constants (reactions not specified).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was rotated with the buffer soln usually overnight. Equilibrium was approached from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol dm⁻³ NaNO₂ soln to first blue on a starch - iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrystd from alcohol and from hot water; mp 165.9°C. Titrn with nitrite indicated that the compd was 100.0+0.3% pure. Elemental analysis confirmed this value.

Source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: ± 0.01 g/100 g soln or ± 0.012 x 10^{-3} in mole fraction (authors).

Temp: $\pm 0.02^{\circ}$ C (authors).

bCalculated by compiler.

 $^{^{\}rm c}$ Obtained by mixing together 50 cm $^{\rm 3}$ of a 0.1 M solution in both ${\rm H_3BO_3}$ and KC1 with 32.1 cm^3 of 0.1 M NaOH and diluting with water up to 100 cm^3 .

 $^{^{} exttt{C}}$ Obtained by mixing together 50 cm 3 of a 0.1 M solution in both $^{ exttt{H}}_3 exttt{BO}_3$ and KC1 with 38.75 $\rm cm^3$ of a 0.1 M NaOH and diluting with water up to 100 $\rm cm^3$.

VARIABLES:

- (1) Benzenesulfonamide, 4-amino (sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Phosphoric acid, disodium salt; Na₂HPO₄; [7558-94-4]
- (3) 1,2,3-Propanetricarboxylic acid, 2-hydroxy- (citric acid); C₆H₈O₇;[77-92-9]
- (4) Water; H₂O; [7732-18-5]

. [7732_18_5]

One temperature: 37°C; one pH: 4.2

PREPARED BY:

ORIGINAL MEASUREMENTS:

Kienle, R. H.; Sayward, J. M. J. Am. Chem. Soc. 1942, 64, 2464-8.

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a solution prepared by mixing together 41.4 cm 3 of 0.2 M Na $_2$ HPO $_4$ with 58.6 cm 3 of 0.1M citric acid (pH 4.2, ionic strength calculated from dissociation constants 0.84 4) at 37.0°C is 1.40 g/100 cm 3 solution (8.13 x 10 $^{-2}$ mol dm $^{-3}$, compiler).

^aNot specified for which reactions were the dissociation constants calculated - compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was rotated with the buffer soln usually overnight. Equilibrium was approached from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol dm⁻³ NaNO₂ soln to first blue on a starch – iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrysted from alcohol and from hot water; mp 165.9°C.

Titrn with nitrite indicated that the compd was 100.0±0.3% pure. Elemental analysis confirmed this value. Source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: ± 0.01 g/100 g soln or $\pm 0.012 \times 10^{-3}$ in mole fraction (authors). Temp: ± 0.02 °C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Phosphoric acid, disodium salt; Na₂HPO₄; [7558-94-4]
- (3) 1,2,3-Propanetricarboxylic acid, 2hydroxy- (citric acid); C₆H₈O₇; [77-92-9]

(4) Water; H₂O; [7732-18-5]

Likhol'ot, N. M. Farm. Zh. (Kiev)

ORIGINAL MEASUREMENTS:

1965, 20(5), 44-6.

VARIABLES:

pН

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

pН	Solubility at 20°C		
of McIlvaine's buffer solution	g/100 ml	10 ² mol dm ^{-3a}	
4.1	0.525	3.05	
5.1	0.504	2.93	
5.9	0.488	2.83	
6.5	0.475	2.76	
6.9	0.467	2.71	
7.5	0.458	2.66	

acalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier described method was employed (1) whereby a small excess of sulfanilamide was equilibrated with the McIlvaine's buffer soln for 8 h in a 50-ml test tube. Aliquots were removed through a filter and sulfanilamide was assayed bromatometrically.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide; not specified.

McIlvaine's buffer solns were prepd from a 0.2M Na₂HPO, and a 0.1M citric acid solns. Source and purity of the buffer components were not specified.

ESTIMATED ERROR:

Soly: not specified. Temp: $\pm 0.1^{\circ}$ C (authors). pH: not specified.

REFERENCES:

1. Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(8), 21.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) Phosphoric acid, disodium salt;
- Na₂HPO₄; [7558-94-4]
- (3) 1,2,3-Propanetricarboxylic acid, 2hydroxy-(citric acid); $C_6H_8O_7$:[77-92-9]
- (4) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Takubo, T.; Matsumaru, H.; Tsuchiya, S.; Hiura, M. Chem. Pharm. Bull. 1973, 21(7) 1440-5.

VARIABLES:

pН

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Citric acid	tric acid Na ₂ HPO ₄ pH		Solubil:	ity at 37°C
g/100 ml water	g/100 ml water		mg/ml soln ^a	10 ² mol dm ⁻³ soln ^b
1.680	0.572	3.1	16.03	9.309
1.260	1.144	4.2	14.40	8.362
0.840	1.716	5.8	13.00	7.549
0.420	2.228	6.8	12.60	7.317

^aNumerical values to the graphical data were given by one of the authors (S.T.) in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Aliquots of the buffer solns were placed in glass-stoppered flasks with excess of sulfanilamide. The flasks were allowed to stand at 37 ± 1 °C and shaken vigorously for 4 h until equilibrium was established. One ml of the supernatant was removed by means of a filter pipet and the sulfanilamide was assayed by the previously reported method (1).

SOURCE AND PURITY OF MATERIALS:

The sulfanilamide was of pharmaceutical grade. The source and purity of citric acid and Na_2HPO_{Λ} was not specified. Distd water was used.

ESTIMATED ERROR:

Soly and pH: not specified. Temp: +1°C (authors).

REFERENCES:

1. Takubo, T.; Tsuchiya, S.; Hiura, M. Yakuzaigaku 1971, 31, 298.

bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-
- (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) 1,2-Benzenedicarboxylic acid, monopotassium salt; $C_8H_5KO_4$; [877-24-7]
- (3) Hydrochloric acid; HC1; [7647-01-0]
- (4) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kienle, R. H.; Sayward, J. M.

J. Am. Chem. Soc. 1942, 64, 2464-8.

VARIABLES:

One temperature: 37.0°C; one pH: 2.2

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a buffer solution prepared by mixing together 50 cm³ of 0.1M monopotassium 1,2-benzenedicarboxylate with 49.5 cm³ of 0.1M ${\tt HC1}$ and diluting up to 100 ${\tt cm}^3$ with water (pH 2.2, ionic strength calculated from dissociation constants 0.06^a) at 37.0°C is 1.79 g/100 cm³ solution $(0.104 \text{ mol dm}^{-3}, \text{ compiler}).$

ANot specified for which reactions were the dissociation constants calculated - compiler.

AUXILIARY INFORMATION

METHOD /APPARATUS / PROCEDURE:

An excess of sulfanilamide was rotated with the buffer soln usually overnight. Equilibrium was approached from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol $\mathrm{dm}^{-3}~\mathrm{NaNo}_2$ soln to first blue on a starch - iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrystd from alcohol and from hot water; mp 165.9°C. Titrn with nitrite indicated that the compd was 100+0.3% pure. Elemental analysis confirmed this value.

Purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: $\pm 0.01 \text{ g/}100 \text{ g soln or } \pm 0.012 \text{ x } 10^{-3}$ in mole fraction (authors);

Temp: $\pm 0.02^{\circ}$ C (authors).

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino-	Meyer, E. J. E., Pharm. Weekblad 1939,
(sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1] (2) Ethanamine, N-ethyl-, (diethylamine);	<i>76</i> , 977-9.
(2) Ethanamine, N-ethyl-, (diethylamine); C ₄ H ₁₁ N; [109-89-7]	
(3) Sodium chloride; NaCl; [7647-14-5]	
(4) Water; H ₂ O; [7732-18-5]	
(,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,, ,,	
VARIABLES:	PREPARED BY:
One temperature: 20°C	R. Piekos
EXPERIMENTAL VALUES:	
EA DATIBITIES VILLOUS.	
Solubility of sulfanilamide in a 6.5% di	ethylamine solution in physiological
saline (0.9% aqueous NaCl) solution at 2	20° C is 6% (0.35 mol kg ⁻¹ solution,
compiler).	
	-1
[diethylamine] = 0.89 mol kg	, compiler
$[NaC1] = 0.15 \text{ mol kg}^{-1}, \text{ comprision}$	ıter
}	
]	
AUXILIARY INFORMATION	
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
	1
Nothing specified.	Nothing specified.
	1
	ESTIMATED ERROR:
	Nothing specified.
	REFERENCES:
	1

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide) $C_6H_8N_2O_2S$; [63-74-1]
- (2) Formic acid, sodium salt; CHNaO₂; [141-53-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P.

Farm. Zh. (Kiev) 1964, 19(1), 52-5.

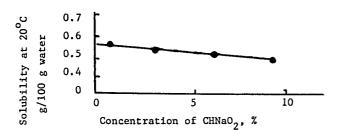
VARIABLES:

Concentration of CHNaO,

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



The solubility in a 1 molal (mol kg $^{-1}$ water, compiler) CHNaO $_2$ solution at 20 $^{\circ}$ C is 0.497 g/100 g water (2.89 x 10 $^{-2}$ mol kg $^{-1}$ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was employed whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a CHNaO₂ soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed colorimetrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) was recrystd from water.

 ${\rm CHNaO}_2$ (source not specified) was also recrystd from water.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M. Farm. 2h. (Kiev) 1960, 15(3), 21.
- Predtechenskii, B. E.; Borovskaya, V.M.; Margolina, L. T., Laboratornye metody issledovanya, Medgiz, Moscow, 1950, p. 371.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Acetic acid, sodium salt (Na acetate); ${}^{\rm C}_{2}{}^{\rm H}_{3}{}^{\rm NaO}_{2};$ [127-09-3]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P.

Farm. Zh. (Kiev) 1964, 19(1), 52-5.

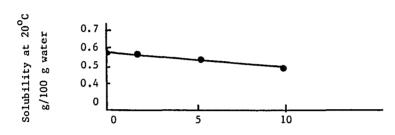
VARIABLES:

Concentration of Na acetate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of Na acetate, %

The solubility in a 1 molal (mol kg $^{-1}$ water, compiler) Na acetate solution at 20 $^{\circ}$ C is 0.511 g/100 g water (2.97 x 10 $^{-2}$ mol kg $^{-1}$ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was employed whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a Na acetate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) was recrystd from water. Na acetate (source not specified) was also recrystd from water.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3), 21.
- 2. The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Propanoic acid, sodium salt (Na propionate); C₃H₅NaO₂; [137-40-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P. Farm. Zh. (Kiev) 1964, 19(1) 52-5.

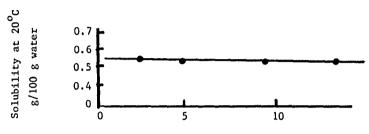
VARIABLES:

Concentration of Na propionate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of Na propionate, %

The solubility in a 1 molal (mol kg $^{-1}$ water, compiler) Na propionate solution at 20 $^{\circ}$ C is 0.541 g/100 g water (3.14 x 10^{-2} mol kg $^{-1}$ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was used whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a Na propionate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd from water. Its source was not specified. Na propionate was prepd by neutralization of propionic acid (source not specified) with Na₂CO₃ or NaOH.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3) 21.
- The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389

COMPONENTS: (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) Pentanoic acid, sodium salt (Na valerate); C₅H₉NaO₂;[6106-41-8]

ORIGINAL MEASUREMENTS:

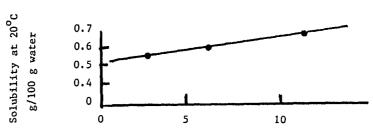
Likhol'ot, N. M.; Gusyakov, V. P. Farm. Zh. (Kiev) 1964, 19(1) 52-5.

Water; H₂0; [7732-18-5]

VARIABLES:
Concentration of Na valerate

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:



Concentration of Na valerate, %

The solubility in a 1 molal (mol kg^{-1} water, compiler) Na valerate solution at $20^{\circ}C$ is 0.678 g/100 g water $(3.94 \times 10^{-2} \text{ mol kg}^{-1} \text{ water, compiler})$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was used whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a Na valerate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd from water. Na valerate was prepd by neutralization of valeric acid with $\mathrm{Na_2CO_3}$ or $\mathrm{NaOH.}$ The source and purity of the materials was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3), 21. The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benzenesulfonamide, 4-amino-sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Hexanoic acid, sodium salt (Na caproate); $C_6H_{11}NaO_2$; [10051-44-2]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P. Farm. Zh. (Kiev) 1964, 19(1) 52-5.

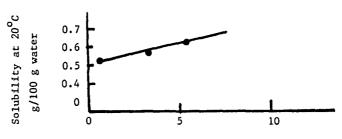
VARIABLES:

Concentration of Na caproate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of Na caproate, %

The solubility in a 0.4 molal (mol kg $^{-1}$ water, compiler) Na caproate solution at 20 $^{\circ}$ C is 0.651 g/100 g water (3.78 x 10 $^{-2}$ mol/kg water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was used whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a Na caproate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd from water. Na caproate was prepd by neutralization of caproic acid with Na₂CO₃ or NaOH. The source and purity of the materials was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3), 21.
- 2. The Extra Pharmacopeia (Martindale) 1955, 2(23) 353 and 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Hexanoic acid, sodium salt, (Na caproate); C₆H₁₁NaO₂; [10051-44-2]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P., Farm. Zh. (Kiev) 1964, 19(1), 52-5.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 0.4 mol kg⁻¹ (molal) sodium caproate solution at 20° C is 0.651 g/100 g water (3.78 x 10^{-2} mol kg⁻¹ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 hr in a 50-ml open test tube with 20 ml of Na caproate soln. Aliquots were removed through a filter, and sulfanilamide was assayed bromatometrically (1).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) was recrystd from water (purity not specified). Na caproate was prepd by neutralization of caproic acid (source and purity not specified) with Na carbonate or hydroxide (source and purity not specified).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Aminoacetic acid (glycine); $^{\text{C}}_{2}^{\text{H}}_{5}^{\text{NO}}_{2}$; [56-40-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kienle, R. H.; Sayward, J. M.

J. Am. Chem. Soc. 1942, 64, 2464-8.

VARIABLES:

One temperature: 37.0°C; one pH: 11.8

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in an aqueous solution of glycine (pH 11.8, ionic strength calculated from dissociation constants 0.11^a) at 37.0° C is 1.93 g/100 cm³ solution (0.112 mol dm⁻³, compiler).

^aNot specified for which reactions were the dissociation constants calculated - compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was rotated with the glycine soln usually overnight. Equilibrium was approached from above. Sampling was accomplished by forcing the soln through a filter into a pycnometer. From the pycnometer the contents were flushed into a volumetric flask. Duplicate aliquots were acidified, iced below 15°C and titrated with a 0.04 mol dm⁻³ NaNO₂ soln to first blue on a starch - iodide paper.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (U.S.P.) from plant production was recrystd from alcohol and from hot water; mp 165.9°C. Titrn with nitrite indicated that the compd was 100.0 ±0.3% pure. Elemental analysis confirmed this value. Source and purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: ± 0.01 g/100 g soln or ± 0.012 x 10^{-3} in mole fraction (authors). Temp: ± 0.02 °C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Pentanoic acid, sodium salt, (Na valerate); C₅H₀NaO₂; [6106-41-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P., Farm. Zh. (Kiev) 1964, 19(1), 52-5.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 mol kg^{-1} (molal) sodium valerate solution at 20° C is 0.678 g/100 g water (3.94 x 10^{-2} mol kg^{-1} water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was equilibrated for 8 hr in a 50-ml test tube with 20 ml of Na valerate soln. Aliquots were taken through a filter and sulfanilamide was assayed bromatometrically (1).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) was recrystd from water (purity not specified). Na valerate was prepd by neutralization of valeric acid (source and purity not specified) with Na carbonate or hydroxide (source and purity not specified).

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

COMPONENTS: Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]

- (2) Propanoic acid, 2-hydroxy-, monosodium salt (Na hydroxypropionate) C₃H₅NaO₃; [72-17-3]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P.

Farm. 2h. (Kiev) 1964, 19(1), 52-5.

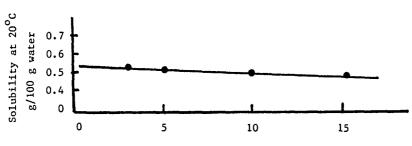
VARIABLES:

Concentration of Na hydroxypropionate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of Na hydroxypropionate, %

The solubility in a 1 molal (mol kg $^{-1}$ water, compiler) Na hydroxypropionate solution at 20 $^{\circ}$ C is 0.493 g/100 g water (2.86 x 10 $^{-2}$ mol kg $^{-1}$ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was used whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a Na hydroxypropionate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd from water.

Its source was not specified. The soln of Na 2-hydroxypropionate was of the Czechoslovak origin (purity not specified).

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3) 21.
- 2. The Extra Pharmacopoia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benezenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Propanoic acid, 2-amino-, monosodium salt (Na aminopropionate); C₃H₆NNaO₂; [23388-69-4]
- (3) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P. Farm. Zh. (Kiev) 1964, 19(1) 52-5.

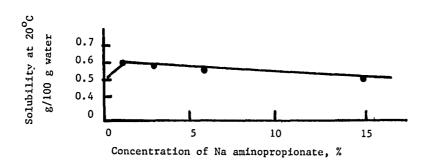
VARIABLES:

Concentration of Na aminopropionate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



The solubility in a 1 molal (mol kg⁻¹ water, compiler) Na aminopropionate solution at 20° C is 0.533 g/100 g water (3.10 x 10^{-2} mol kg⁻¹ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method was used (1) whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a Na aminopropionate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was recrystd from water. Na aminopropionate was prepd by neutralization of 2-aminopropionic acid with ${\rm Na_2^{CO}}_3$ or NaOH. The source and purity of the materials was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3) 21.
 The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Butanedioic acid, 2,3-dihydroxydisodium salt (di-Na tartrate);
 C₄H₄Na₂O₆; [868-18-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P.

Farm. Zh. (Kiev) 1964, 19(1), 52-5.

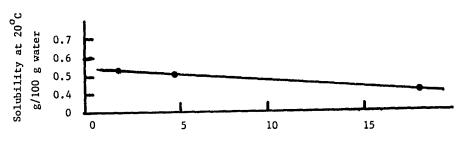
VARIABLES:

Concentration of di-Na tartrate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of di-Na tartrate, %

The solubility in a 1 molal (mol kg $^{-1}$ water, compiler) di-Na tartrate solution at 20 $^{\circ}$ C is 0.426 g/100 g water (2.47 x 10 $^{-2}$ mol kg $^{-1}$ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was used whereby a small excess of sulfanilimide was equilibrated in a 50-ml test tube with 20 ml of a di-Na tartrate soln for 8 h. Aliquots of the satd soln were removed through a filter and sulfanilamide was assayed bromatometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide and di-Na tartrate (source not specified) were purified by recrystn from water.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960, 15(3), 21.
 The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) 1,2,3-Propanetricarboxylic acid, 2-hydroxy- (citric acid); C₆H₈O₇; [77-92-9]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Takubo, T.; Matsumaru, H.; Tsuchiya, S.; Hiura, M. *Chem. Pharm. Bull.* 1973, 21(7), 1440-5.

VARIABLES:

One temperature: 37°C; one pH: 2.1

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a citric acid solution (2.100 g citric acid per 100 ml water) of pH 2.1 at 37° C is 19.24 mg/ml solution^a (0.1117 mol dm⁻³ solution, compiler).

^aNumerical value to the graphical one was given by one of the authors (S.T.) in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Aliquots of the citric acid soln were placed in glass-stoppered flasks with excess of sulfanilamide. The flasks were allowed to stand at $37\pm1^{\circ}\text{C}$ and shaken vigorously for 4 h until equilibrium was established. One ml of the supernatant was removed by means of a filter pipet and the sulfanilamide was assayed by the previously reported method (1).

SOURCE AND PURITY OF MATERIALS:

The sulfanilamide was of pharmaceutical grade. Source and purity of the citric acid was not specified.

Distd water was used.

ESTIMATED ERROR:

Soly: not specified.

pH: not specified.

Temp: +1°C (authors).

REFERENCES:

 Takubo, T.; Tsuchiya, S.; Hiura, M. Yakuzaigaku 1971, 31, 298.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- **(2)** 1,2,3-Propane tricarboxylic acid, 2-hydroxy-, trisodium salt: (tri-Na citrate); C₆H₅Na₃O₇; [68-04-2]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Likhol'ot, N. M.; Gusyakov, V. P. Farm. Zh. (Kiev) 1964, 19(1), 52-5.

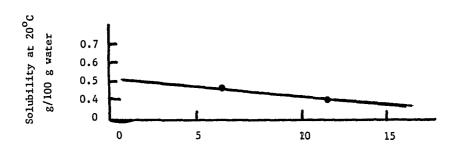
VARIABLES:

Concentration of tri-Na citrate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of tri-Na citrate, %

The solubility in a 1 molal (mol kg - 1 water, compiler) tri-Na citrate solution at 20° C is 0.281 g/100 g water (1.63 x 10^{-2} mol kg⁻¹ water, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously described method (1) was used whereby a small excess of sulfanilamide was equilibrated in a 50-ml test tube with 20 ml of a tri-Na citrate soln for 8 h. Aliquots of the satd soln were removed through a filter and assayed bromatrometrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide and tri-Na citrate (source not specified) were purified by crystn from water.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- 1. Gusyakov, V. P.; Likhol'ot, N. M. Farm. Zh. (Kiev) 1960. 15(3), 21.
- 2. The Extra Pharmacopeia (Martindale) 1955, 2(23), 353 and 389.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Benzoic acid, sodium salt; $C_7^H_5NaO_2$; [532-32-1]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Sukmans'ka, I. V. Farm. Zh. (Kiev) 1960, 15(1), 20-23.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 mol kg^{-1} water sodium benzoate solution at 20°C is 0.94 g/100 ml solution (5.5 x 10^{-2} mol dm^{-3} , compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

To 50-ml tightly stoppered test tubes contg 25 ml of a 1 mol kg⁻¹ water Na benzoate soln, placed in a thermostat, accurately weighed 0.02-0.002-g portions of sulfanilamde were added under agitation until satn was attained.

SOURCE AND PURITY OF MATERIALS:

Both sulfanilamide and Na benzoate conformed to the requirements of the State Pharmacopeia VIII.

Distilled water was used.

ESTIMATED ERROR:

Temp: +0.1°C (authors).

Soly: the accuracy of the detn of the concn was similar to that attained by volumetric method (authors).

99 ORIGINAL MEASUREMENTS: COMPONENTS: (1) Benzenesulfonamide, 4-amino-Khažko, L. Farm. Obzor 1966, 35, 298-311. (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] (2) Benzoic acid, sodium salt; C₇H₅NaO₂; [532-32-1] (3) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in a 0.5 mol dm⁻³ sodium benzoate solution at 20° C is 0.81 g/100 ml solution (4.7 x 10^{-2} mol dm⁻³, compiler) or

0.79/100 g solution.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions to a known volume of Na benzoate soln until reaching satn. The Pharmacopeia 2, Suppl. 1959. equilibration time was 3-4 h under stirring. The temp was held const by means of the Höppler ultrathermostat.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide and Na benzoate conformed to the requirements of the Czechoslovak Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Benzoic acid, 2-hydroxy-, monosodium salt (Na salicylate); C7H5NaO3; [54-21-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kňažko, L. Farm. Obzor 1966, 35, 298-311.

VARIABLES:

Concentration of Na salicylate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of	Solub	ility at 20 ⁰	C
Na salicylate (mol dm ⁻³)	g/100 ml	g/100 g	10 ² mol dm ^{-3^a}
0.5	1.05	1.02	6.10
1	1.50	1.415	8.71
1.5	1.83	1.674	10.63
2	2.79	2.48	16.20
2.5	3.78	3.27	21.95
3	4.238	3.57	24.61

^aof Na salicylate solution, calculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions to a known volume of Na salicylate solns until reaching satn. The equilibration time was 3-4 h under stirring. The temp was held const by means of the Höppler ultrathermostat.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide and Na salicylate conformed to the requirements of the Czechoslovak Pharmacopeia 2, Suppl. 1959.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Benzoic acid, 2-hydroxy-, monosodium salt (Na salicylate); C₇H₅NaO₃; [54-21-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Sukmans'ka, I. V. Farm. Zh. (Kiev) 1960, 15(1), 20-23.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 mol kg $^{-1}$ water Na salicylate solution at 20 $^{\circ}$ C is 1.29 g/100 ml solution (7.49 x 10 $^{-2}$ mol dm $^{-3}$, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

To 50-ml tightly stoppered test tubes contg 25 ml of a 1 mol kg⁻¹ water Na salicylate soln, placed in a thermostat, accurately weighed 0.02-0.002-g portions of sulfanilamide were added under agitation until satn was attained.

SOURCE AND PURITY OF MATERIALS:

Both sulfanilamide and Na salicylate conformed to the requirements of the State Pharmacopeia VIII.

Distilled water was used.

ESTIMATED ERROR:

Temp: $\pm 0.1^{\circ}$ C (authors).

Soly: the accuracy of the detn of the concn was similar to that attained by volumetric method (authors).

ORIGINAL MEASUREMENTS: COMPONENTS: (1) Benzenesulfonamide, 4-amino-Gusyakov, V. P.; Sukmans'ka, I. V. (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] Farm. Zhur. (Kiev) 1961, 16, 25-8. (2) Benzoic acid, 2-hydroxy-, monosodium salt; $C_7^{\rm H_5NaO}_3$; [54-21-7] (3) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 molal monosodium 2-hydroxybenzoate solution at 20°C is 1.29 g/100 ml monosodium 2-hydroxybenzoate solution $(7.49 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler}).$

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions (0.02 - 0.002 g) to a known volume of 1 molal monosodium 2hydroxybenzoate soln, held on a water bath, until satn was attained. Moreover, the concn of sulfanilamide was assessed by means of a FEK-M photoelectrocolorimeter.

SOURCE AND PURITY OF MATERIALS: Sulfanilamide and Na 2-hydroxybenzoate were pharmacopeial products.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: results of the colorimetric and gravimetric runs differed by 1-3% (authors). Temp: $\pm 0.1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Benzoic acid, 4-hyroxy-, monosodium salt; C₇H₅NaO₃; [114-63-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Sukmans'ka, I. V.

Farm. Zh. (Kiev) 1961, 16, 25-8.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 molal monosodium 4-hydroxybenzoate solution at 20° C is 1.04 g/100 ml monosodium 4-hydroxybenzoate solution (6.04 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions (0.02 - 0.002 g) to a known volume of 1 molal monosodium 4-hydroxybenzoate soln, held on a water bath, until satn was attained. Moreover, the concn of sulfanilamide was detd by means of a FEK-M photoelectrocolorimeter.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was a pharmacopeial product. Na 4-hydroxybenzoate was obtained by the authors by neutralization of 4-hydroxybenzoic acid which was 99.7% pure.

ESTIMATED ERROR:

Soly: results of colorimetric and gravimetric runs differed by 1-3% (authors).

Temp: +0.1°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Benzoic acid, 4-amino-2-hydroxy-, monosodium salt; C₇H₆NNaO₃; [133-10-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Sukmans'ka, I. V. Farm. Zh. (Kiev) 1960, 15(1) 20-23.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 mol kg⁻¹ water Na 4-amino-2-hydroxybenzoate solution at 20° C is 1.42 g/100 ml solution (8.25 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

To 50-ml tightly stoppered test tubes contg 25 ml of a 1 mol kg⁻¹ water Na 4-amino-2-hydroxybenzoate soln, placed in a thermostat, accurately weighed 0.02-0.002-g quantities of sulfanilamide were added under agitation until satn was attained.

SOURCE AND PURITY OF MATERIALS:

Both sulfanilamide and Na 4-amino-2hydroxybenzoate conformed to the requirements of the State Pharmacopeia VIII.

Distilled water was used.

ESTIMATED ERROR:

Temp. ±0.1°C (authors).

Soly: the accuracy of the detn of the concn was similar to that attained by volumetric method (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}{}_{8}{}^{N}{}_{2}{}^{O}{}_{2}{}^{S}$; [63-74-1]
- (2) Benzenesulfonic acid, 4-methyl-, sodium salt (Na 4-toluenesulfonate) $C_7^H_7^{NaO}_3^S$; [657-84-1]
- (3) Water; H₂O; [7732-18-4]

ORIGINAL MEASUREMENTS:

Kňažko, L. Farm. Obzor 1966, 35, 298-311.

VARIABLES:

Concentration of Na 4-toluenesulfonate

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of	Solubility at 20°C		
Na 4-toluenesulfonate (mol dm ⁻³)	g/100 ml	g/100 g	10 ² mol dm ^{-3^a}
0.5	0.95	0.92	5.52
1	1.25	1.165	7.26
1.5	1.56	1.405	9.06

a of Na 4-toluenesulfonate solution, calculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions to a known volume of Na 4-toluenesulfonate solns until reaching satn. The equilibration time was 3-4 h under stirring. The temp was held const by means of the Höppler ultrathermostat.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide and Na 4-tolenesulfonate conformed to the requirements of the Czechoslovak Pharmacopeia 2, Suppl. 1959.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Benzoic acid, 3-hydroxy-, monosodium salt; C₇H₅NaO₃; [7720-19-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Sukmans'ka, I. V. Farm. Zh. (Kiev) 1961, 16, 25-8.

VARIABLES:

One temperature: 20°C

PREPARED BY:

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 1 molal monosodium 3-hydroxybenzoate solution at 20° C is 1.09 g/100 ml monosodium 3-hydroxybenzoate solution (6.33 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in small portions (0.02 - 0.002 g) to a known volume of 1 molal monosodium 3-hydroxybenzoate soln, held on a water bath, until satn was attained. Moreover, the concn of sulfanilamide was detd by means of a FEK-M photoelectrocolorimeter.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide was a pharmacopeial product. Na 3-hydroxybenzoate was obtained by neutralization of a comm 3-hydroxybenzoic acid with Na₂CO₃. The purity of the product was 97.7% (authors).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: results of the colorimetric and gravimetric runs differed by 1-3% (authors).

Temp: +0.1°C (authors).

- (1) Benzenesulfonamide, 4-amino-, (sulfanilamide); ${}^{C}_{6}{}^{H}{}_{8}{}^{N}{}_{2}{}^{O}{}_{2}{}^{S}$; [63-74-1]
- (2) Pectin; $(C_{13}H_{18}O_{12})_n$; [9000-69-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Becher, R.; Leya, S., Experientia 1946, 2, 459-60.

VARIABLES:

One temperature: 18-19°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 2.5% pectin solution ([pectin] = 6.8×10^{-2} mol kg⁻¹, compiler), of pH about 2.6, at room temperature ($18 - 19^{\circ}$ C) is 866 mg% (5.00×10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soln was allowed to stand for more than 2 days at room temp. The soln was then filtered, and sulfanilamide assayed in the filtrate colorimetrically by the method of Druey and Oesterheld (1).

SOURCE AND PURITY OF MATERIALS:

A high quality apple pectin was used: the rel viscosity of a 0.5% soln was 6.2, and for neutralization of 1 g of the pectin, 1.67 cm³ of 1 mol dm⁻³ NaOH was used. The source and purity of sulfanilamide and water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Druey, J.; Oesterheld, G., Helv. Chim. Acta 1942, 25, 753.

COMPONENTS: (1) Benzenesulfonamide, 4-amino-, (sulfanilamide); C₆H₆N₂O₂S; [63-74-1] (2) Pectinic acid, sodium salt; (C₁₃H₁₇NaO₁₂)_n; [9049-37-0] (3) Water; H₂O; [7732-18-5] VARIABLES: One temperature: 18-19°C EXPERIMENTAL VALUES: ORIGINAL MEASUREMENTS: Becher, R., Leya, S., Experientia 1946, 2, 459-60.

Solubility of sulfonamide in a 2.6% neutral sodium pectinate solution at room temperature $(18-19^{\circ}C)$ is 757 mg% $(4.40 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler})$.

[Na pectinate] = $6.7 \times 10^{-2} \text{ mol kg}^{-1}$, compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soln was allowed to stand for two days at room temp. The soln was then filtered, and sulfanilamide assayed in the filtrate colorimetrically by the method of Druey and Oesterheld (1).

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Druey, J.; Oesterheld, G., Helv. Chim. Acta 1942, 25, 753.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Ethanol; C₂H₆O; [64-17-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Sapozhnikova, N. V.; Postovskii, I. Ya. Zh. Prikl. Khim. 1944, 17, 527-34.

VARIABLES:

Concentration of ethanol

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 37 ⁰ C		
of ethanol Weight%	Weight%	mol kg ⁻¹ solvent ^a	
0	1.4	0.84	
38.3	2.4	0.14	
57.6	3.8	0.23	
67.2	4.9	0.30	
76.4	7.0	0.44	
86	4.8	0.29	
96	3.9	0.24	

acalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was dissolved in EtOH-water mixts to form satd solns which were occasionally agitated in glass vessels immersed in a thermostat. The equilibrium was usually attained after 1 h. Fiveto 100-cm³ samples of the satd soln were placed in Pt crucibles or dishes and evapd to dryness at temps lower than 110-115°C. The residue was dried to const wt at 105-110°C and weighed.

SOURCE AND PURITY OF MATERIALS:

Pure, recrystd. sulfanilamide was used. Its mp conformed to that reported in the literature.

The purity of ethanol and water was not specified.

ESTIMATED ERROR:

Soly: quite reliable results were obtained (authors).

Temp: ±0.05°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Ethanol; C₂H₆O; [64-17-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Shkadova, A. I.

Farm. Zh. (Kiev) 1969, 24(3), 39-41.

VARIABLES:

Concentration of ethanol

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Concentration of	ethanol_	Solubility at	: 20°C
mole %	weight %	10^2 mol kg $^{-1}$	g/100 g ^e
0	0	3.06	0.527
10	22.14	7.54	1.298
20	39.01	17.15	2.953
30	52.31	21.67	3.732
40	63.04	25.25	4.348
50	71.90	26.89	4.630
60	79.33	25.09	4.320
70	85.65	23.01	3.962
80	91.10	20.77	3.577
90	95.83	14.50	2.496
			

acalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide were equilibrated with the solvent in a water thermostat at 20±0.1°C. Sulfanilamide was detd bromatometrically.

SOURCE AND PURITY OF MATERIALS:

Purity of sulfanilamide conformed to the requirements of the State Pharmacopoeia IX. The EtOH-water mixtures were prepd from abs EtOH (purity and source not specified) and distd water.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.1°C (author).

сомроненть:

(1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]

(2) Ethanol; C₂H₆O; [64-17-5]

(3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Burger, A.

Pharm. Ind. 1973, 35, 626-33.

VARIABLES:

Concentration of ethanol

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Concentration of ethanol	Saturation solubility, of sulf	C, a of crystalline form II anilamide at 20.0 C
Volume %	mg/100 ml soln	mol dm ^{-3b}
96	2680	0.1556
90	3279	0.1904
80	3735	0.2169
70	3931	0.2283
65	4024	0.2337
60	3843	0.2332
55	3409	0.1980
47.5	2615	0.1519
45	2475	0.1437
40	2285	0.1327

^aC = [HA] + [A], where [HA] is the molar concentration of the dissolved, undissociated molecules of sulfanilamide and [A] is the concentration of the dissolved anion of the sulfanilamide.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was assayed spectrophotometrically at 258.5 nm using a Zeiss PMQ II spectrophotometer, in 1/15 M phosphate buffer of pH 7.00 ($E_{1~cm}^{1\%}$ = 945).

SOURCE AND PURITY OF MATERIALS:

Form II of sulfanilamide was obtained by the common method (1). Its purity was not specified.

Purity of the water and EtOH was not specified.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.1°C (author)

REFERENCES:

 Burger, A. Sci. Pharm. 1973, 41, 290 and 303.

bCalculated by compiler.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) 1,2-Ethanediol; C₂H₆O₂; [107-21-1] (3) Water; H₂O; [7732-18-5] VARIABLES: Concentration of 1,2-ethanediol ORIGINAL MEASUREMENTS: Mingoia, Q. Ann. Chim. Farm. (Suppl. to Farm. Ital.) Apr., 1939, 48-58. PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

		Solubilit	y of sulfanilamide at 20°C
% Water % 1,2-Ethanedic	% 1,2-Ethanediol	wt.%	10 mol kg ⁻¹ solution ^a
90	10	0.81	0.47
75	25	1.40	0.81
50	50	3.22	1.87
40	60	6.49	3.77
25	75	9.07	5.27

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Five cm³ of a sulfanilamide soln in aq 1,2-ethanediol was evapd to const wt on a boiling water bath. The residue was dissolved in distd water and its volume was adjusted to 50 cm³ with the water. In 25 cm³ of this soln sulfanilamide was assayed by known methods (probably colorimetric).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide: source and purity not specified.

1,2-Ethanediol was from Merck (purity not specified.

Distilled water was used.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) 1,2-Propanedio1; C₃H₈O₂; [57-55-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Mingoia, Q.

Ann. Chim. Farm. (Suppl. to Farm. Ital.)
Apr., 1939, 48-58.

VARIABLES:

Concentration of 1,2-propanediol

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

_		Solubility of sulfanilamide at	
% Water	% 1,2-Propanediol	wt%	10 mol kg ⁻¹ solution
90	10	1.05	0.61
75	25	1.88	1.09
50	50	3.85	2.24
40	60	5.58	3.24
25	75	7.90	4.59

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Five cm³ of a sulfanilamide soln in aq 1,2-propanediol was evapd to const wt on a boiling water bath. The residue was dissolved in distd water and its volume was adjusted to 50 cm³ with the water. In 25 cm³ of this soln sulfanilamide was assayed by known methods (probably colorimetric).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide: source and purity not specified.

1,2-Propanediol was from Merck (purity not specified).

Distilled water was used.

ESTIMATED ERROR:

Nothing specified.

114 COMPONENTS: ORIGINAL MEASUREMENTS: (1) Benzenesulfonamide, 4-amino-Dolique, R.; Foucault, J. (sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] Trav. soc. pharm. Montpellier 1952, 12, (2) Ethanol; C₂H₆O; [64-17-5] 145-53. (3) 1,2,3-Propanetriol; $C_3H_8O_3$; [56-81-5] (4) Water; H₂O; [7732-18-5] VARIABLES: PREPARED BY: One temperature: 26-28°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in a mixture of 1,2,3-propanetriol and 95° ethanol (2:1 by wt) at 26-28°C is 6.23% (0.386 mol kg⁻¹ solvent, compiler). AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: The sulfanilamide content was detd by Nothing specified. diazotization of the amine group in a cold acidified 0.1N KNO_2 soln. An excess of KNO₂ was detected by using iodinated starch. ESTIMATED ERROR: Nothing specified. REFERENCES:

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Ethanol, 2,2'-oxybis- (diethylene glycol); C₄H₁₀O₃; [111-46-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Mingoia, Q.

Ann. Chim. Farm. (Suppl. to Farm. Ital.)
Apr., 1939, 48-58.

VARIABLES:

Concentration of diethylene glycol

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

	_	Solubility of sulfanilamide at 20°C	
% Water	% Diethylene glycol	wt%	10 mol kg ⁻¹ solution
90	10	1.38	0.801
75	25	2.98	1.73
50	50	6.48	3.76
40	60	12.60	7.317
25	75	26.75	15.53

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Five cm³ of a sulfanilamide soln in aq diethylene glycol was evapd to const wt on a boiling water bath. The residue was dissolved in distd water and its volume was adjusted to 50 cm³ with the water. In 25 cm³ of this soln sulfanilamide was assayed by known methods (probably colorimetric).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide: source and purity not specified.

Diethylene glycol was from Carbide and Carbon Co. (purity was not specified). Distilled water was used.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Urea; CH₄N₂O; [57-13-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Dolique, R.; Foucault, J.

Trav. soc. pharm. Montpellier 1952, 12 145-53.

VARIABLES:

Concentration of urea

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

Concentration of urea	Solubility of sulfanilamide at 2	
g/100 g water	g/100 g water	mol kg ⁻¹ water ^a
0	0.8	.05
20	1.93	.11
40	3.3 ₅	.20
80	5.9	.34
120	9.9	.58

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The satd soln was agitated for 12 h at 26°C and filtered. The filtrate was evapd at $100\text{--}110^{\circ}\text{C}$ and the residue was weighed. Measurements were carried out in test tubes containing 25 cm³ of water.

SOURCE AND PURITY OF MATERIALS: Nothing specified.

ESTIMATED ERROR:

Nothing specified.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Urea; CH₄N₂O; [57-13-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of urea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C	
of urea mol/1 ^a	g/100 ml	10 ² mo1 dm ^{-3^b}
0.100	0.572	3.32
0.200	0.610	3.54
0.300	0.628	3.65
0.489	0.604	3.51
0.700	0.660	3.83
0.957	0.676	3.93
1.551	0.814	4.73

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm³) were placed in 100-cm³ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:
Sulfanilamide (source not specified)
conformed to the requirements of the
DAB 7-BRD. Urea (Schuchardt) was
recrystd from aq MeOH. Purity of the
water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.05°C (author).

REFERENCES:

 Schulte, K. E.; Rohdewald, R.; Weinhold, P. Pharmazie 1968, 23(5), 252.

bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- 3-Pyridinecarboxamide; C6H6N2O; [98-92-0]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P. Pharm. Ztg. 1971, No. 38, 1342-4.

VARIABLES:

Concentration of 3-pyridinecarboxamide

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

$$-k_s = log \frac{L_{H_2O}}{L_s c_s} = 0.66 \text{ 1/mol}$$

 $-k_{_{\rm S}} = \log \frac{L_{\rm H_2O}}{L_{_{\rm S}} c_{_{\rm S}}} = 0.66 \; \rm 1/mol,$ where $L_{\rm H_2O}$ (0.318 $_{\rm 8}$ g/50 ml = 2.975 $_{\rm 8}$ x 10 $^{-2}$ mol dm $^{-3}$, compiler) and $L_{_{\rm S}}$ are solubilities of sulfamilamide in water and in aqueous 3-pyridinecarboxamide solution, respectively, and $\boldsymbol{c}_{\boldsymbol{g}}$ is the concentration of 3-pyridinecarboxamide. $\mathbf{L}_{_{\mathbf{S}}}$ values were supplied by the author in personal communication and are shown

Concentration of

L at 20°C

3-pyridinecarboxamide		
mo1/1	g/100 ml	$10^2 \text{ mol dm}^{-3}^{\mathbf{a}}$
0.030	0.542	3.15
0.082	0.612	3.55
0.164	0.686	3.98
0.328	0.854	4.96
0.500	1.090	6.33

^aCalculated by compiler

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solns were equilibrated by agitation for 2 h at 20°C and the sulfanilamide was assayed by differential gravimetric analysis. No details were given.

SOURCE AND PURITY OF MATERIALS:

The source and purity of sulfanilamide and water was not specified. Anal reagent grade 3-pyridinecarboxamide (source not specified) dried over mol sieve was used.

ESTIMATED ERROR:

Soly: not specified. Temp: $\pm 0.05^{\circ}$ C (author).

- Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (1)
- 3-Pyridinecarboxamide, N,N-diethyl-(nicetamide); C₁₀H₁₄N₂O; [59-26-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P. Pharm. Ztg. 1971, No. 38 1342-4.

VARIABLES:

Concentration of nicetamide

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

$$-k_x = log \frac{L_{H_2O}}{L_c c_c} = 0.64 l/mo1$$

 $\frac{L_{\rm H_2O}}{-k_{_{\rm X}}=\log\frac{L_{_{\rm H_2O}}}{L_{_{\rm S}}\,c_{_{\rm S}}}=0.64~\rm 1/mol,}$ where $L_{\rm H_2O}$ (0.318₈ g/40 ml = 2.975₈ x 10^{-2} mol dm⁻³, compiler) and $L_{_{\rm S}}$ are solubilities of sulfanilamide in water and in aqueous nicetamide solutions, respectively, and c_s is the concentration of nicetamide.

 $\mathbf{L}_{\mathbf{c}}$ values were supplied by the author in personal communication and are shown below.

Concentration	L _s at 20°C	
of nicetamide mol/1	g/100 ml	10 ² mol dm ^{-3^a}
0.100	0.674	3.91
0.200	0.696	4.04
0.300	0.932	5.41
0.500	1.246	7.24

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The solns were equilibrated by agitation for 2 h at 20°C and the sulfanilamide was assayed by differential gravimetric analysis. No details were given.

SOURCE AND PURITY OF MATERIALS:

The source and purity of sulfanilamide and water was not specified. Anal reagent grade nicetamide (source not specified) dried over mol sieve was used.

ESTIMATED ERROR:

Soly: not specified. Temp: ±0.05°C (author).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1]
- (2) Urea, methyl-; C₂H₆N₂O; [598-50-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7) 460-3.

VARIABLES:

Concentration of methylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of methylurea mol/1	Solubility at 20°C	
	g/100 ml	10 ² mol cm ^{-3^b}
0.050	0.576	3.34
0.100	0.586	3.40
0.200	0.602	3.50
0.400	0.614	3.57
0.600	0.676	3.93
0.700	0.732	4.25
0.800	0.740	4.30
1.000	0.790	4.59

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm³) were placed in 100-cm³ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) conformed to the requirements of the DAB 7-BRD. Methylurea (Schuchardt) was recrystd from aq MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: +0.05°C (author).

REFERENCES:

 Schulte, K. E.; Rohdewald, P.; Weinhold, P. Pharmazie 1968, 23(5), 252.

^bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Urea, ethyl-; C₃H₈N₂O; [625-52-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of ethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of ethylurea	Solubili	ty at 20°C
mol/1 ^a	g/100 ml	10 ² mol dm ^{-3^b}
0.200	0.620	3.60
0.300	0.664	3.86
0.500	0.738	4.29
0.700	0.826	4.80

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm³) were placed in 100-cm³ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) conformed to the requirements of the DAB 7-BRD. Ethylurea (Schuchardt) was recrystd from aq MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.05°C (author).

REFERENCES:

bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Urea, N,N'-dimethyl-; C₃H₈N₂O; [96-31-1]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7). 460-3.

VARIABLES:

Concentration of N,N'-dimethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of N,N'-dimethylurea mol/1ª	Solubility at 20°C			
	g/100 ml	10 ² mo1 dm ^{-3^b}		
0.200	0.624	3.62		
0.300	0.660	3.83		
0.400	0.678	3.94		
0.500	0.726	4.22		
0.600	0.750	4.35		
0.700	0.792	4.60		
0.800	0.842	4.89		

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm³) were placed in 100-cm³ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) conformed to the requirements of the DAB 7-BRD. N,N'-dimethylurea (Schuchardt) was recrystd from aq MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: $\pm 0.05^{\circ}$ C (author).

REFERENCES:

 $^{^{\}mathrm{b}}\mathrm{Calculated}$ by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Urea, N,N-dimethy1-; C₃H₈N₂O; [598-94-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7) 460-3.

VARIABLES:

Concentration of N,N-dimethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration of N,N-dimethylurea	Solubility at 20°C			
mol/1 ^a	g/100 ml	10 ² mol dm ^{-3^b}		
0.200	0.656	3.81		
0.300	0.678	3.94		
0.500	0.708	4.11		
0.700	0.838	4.87		
0.800	0.870	5.05		
0.927	1.016	5.90		

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 $\rm cm^3$) were placed in 100- $\rm cm^3$ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) conformed to the requirements of DAB 7-BRD. N,N-dimethylurea (Schuchardt) was recrystd from aq MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.05°C (author).

REFERENCES:

bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Urea, tetramethyl-; $C_5H_{12}N_2O$; [632-22-4]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmazie 1975, 30(7), 460-3.

VARIABLES:

Concentration of tetramethylurea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of tetramethylurea mol/1ª	g/100 ml	10 ² mo1 dm ^{-3^b}		
0.200	0.850	4.936		
0.400	1.004	5.830		
0.600	1.196	6.945		
0.800	1.412	8.200		
1.000	1.668	9.686		

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used wereby the solns (50 cm³) were placed in 100-cm³ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 30°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:
Sulfanilamide (source not specified)
conformed to the requirements of the
DAB 7-BRD. Tetramethylurea (Schuchardt)
was recrystd from aq MeOH. Purity of
the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.05°C (author).

REFERENCES:

^bCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Thiourea; CH_AN₂S; [62-56-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Rohdewald, P.

Pharmasie 1975, 30(7), 460-3.

VARIABLES:

Concentration of thiourea

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Concentration	Solubility at 20°C			
of thiourea_ mol/la	g/100 ml	10 ² mol dm ^{-3b}		
0.300	0.646	3.75		
0.400	0.664	3.86		
0.600	0.706	4.10		
0.800	0.784	4.55		
1.000	0.880	5.11		
1.200	0.952	5.53		

^aNumerical values given by the author in personal communication.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The previously employed method (1) was used whereby the solns (50 cm³) were placed in 100-cm³ flasks with glass stoppers and rotated in a thermostated bath for 2 h. They were then filtered through a G3 glass filter and the undissolved sulfanilamide was dried at 90°C to const wt and weighed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (source not specified) conformed to the requirements of the DAB 7-BRD. Thiourea (Schuchardt) was recrystd from aq MeOH. Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: +0.05°C (author).

REFERENCES:

bCalculated by compiler.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino-, (sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Weinstein, L.; McDonald, A., Science <u>1945</u> , <i>101</i> , 44-5.
(2) Carbamic acid, ethyl ester (urethane); C ₃ H ₇ NO ₂ ; [51-79-6]	<u> </u>
(3) Water; H ₂ O; [7732-18-5]	
VARIABLES:	PREPARED BY:
One temperature: 20°C	R. Piekos
EXPERIMENTAL VALUES:	
Solubility of sulfanilamide in a 10% aqui 1000 mg/100 cm ³ urethane solution (6 x 1	neous urethane solution at 20°C .0 ⁻² mol dm ⁻³ , compiler).
·-	
AUXILIARY	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Nothing specified.	Nothing specified.
	ESTIMATED ERROR: Nothing specified.
	REFERENCES:

		12,
COMPO	NENTS:	ORIGINAL MEASUREMENTS:
(1)	Benzenesulfonamide, 4-amino-	Dolique, R.; Foucault, J.
(0)	(sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Trav. soc. pharm. Montpellier 1952, 12,
	Ethanol; C ₂ H ₆ O; [64-17-5]	145-53.
	1,2,3-Propanetriol; C ₃ H ₈ O ₃ ; [56-81-5]	
	Urea; CH ₄ N ₂ O; [57-13-6]	
(5)	Water; H ₂ O; [7732-18-5]	Para car
AKIA		PREPARED BY:
	One temperature: 26-28°C	R. Piekos
XPER	IMENTAL VALUES:	
	Solubility of sulfanilamide at 26-28°C:	in a saturated solution of urea in a
	mixture of 1,2,3-propanetriol and 95° es	
	of urea per 100 g of the mixture, is 8.2	
	or area per 100 g or the mineure, 10 or	are (0.22 mor 16 gordent) comprise.
_		
		INFORMATION
	DD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
T	he sulfanilamide content was detd by	Nothing specified.
d:	iazotization of the amine group in a	
C	old acidified 0.1N KNO, soln. An excess	
	f KNO ₂ was detected by using iodinated	
	tarch.	
_		
		ECTIMATED EDDOR.
		ESTIMATED ERROR:
		Nothing specified.
		REFERENCES:
		1
		1

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Poly (oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 200); (C₂H₄O)_nH₂O; [25322-68-3] 200
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 151-61.

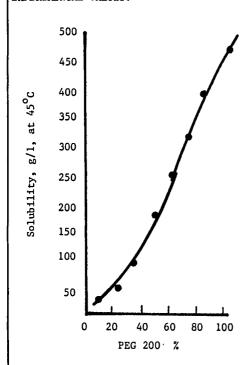
VARIABLES:

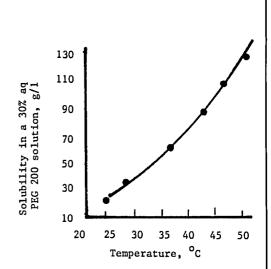
Concentration of PEG 200; temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:





AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier developed method was employed (1) whereby a 100-ml conical flask contg a PEG 200 soln was placed in a drying cabinet at a given temp and an excess of sulfanilamide was added under stirring for 1 h. After 12 h the soln was filtered or decanted and the solute was assayed in the filtrate spectrophotometrically at 260 mm using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed with the requirements of BP 1953 or USP XIV. It was recrystd, dried, powdered, and again dried at 105° C to const wt. PEG 200 was a product of Farbwerke Hoechst. It was kept over concd $\mathrm{H_2SO_4}$ in a desiccator.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 90.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Poly(oxy-1,2-ethanediy1), α -hydroω-hydroxy- (PEG 400); ($C_2H_4O)_nH_2O$; [25322-68-3] 400
- (3) Water; H₂0; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1964, 32, 271-9.

VARIABLES:

One temperature: 45°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 10% aqueous solution of PEG 400 at 45°C is 5.57 g/100 g PEG 400 solution (0.323 mol kg^{-1} , compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in excess to an aq PEG 400 soln and the mixt was stirred for 30 min. The soln was then placed in a drying cabinet for 24 h and occasionally shaken. After filtration the sulfanilamide was assayed in the filtrate by the USP XVI method based on diazotization. The end point was detected by means of a starch paste as an indicator. Corrections were made for consumption of the 0.1 N NaNO2 soln by PEG 400.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: measurements were made in duplicate (authors).

Temp: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Poly(oxy-1,2-ethanedly1), α-hydroω-hydroxy- (PEG 400); (C₂H₄0)_nH₂0; [25322-68-3] 400
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 151-61.

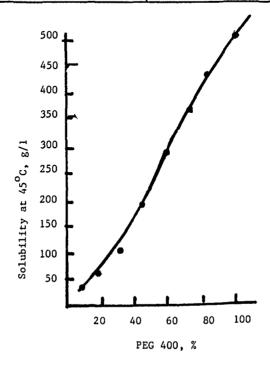
VARIABLES:

Concentration of PEG 400

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier developed method was employed (1) whereby a 100-ml conical flask contg a PEG 400 soln was placed in a drying cabinet at 45°C and an excess of sulfanilamide was added under stirring for 1 h. After 12 h the soln was filtered or decanted and the solute was assayed in the filtrate spectrophotometrically at 260 mµ using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed with the requirements of BP 1953 or USP XIV. It was recrystd, dried, powdered, and again dried at 105° C to const wt. PEG 400 was a product of Farbwerke Hoechst. It was kept over concd ${\rm H_2SO_4}$ in a desiccator. Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Soi. Pharm. 1965, 33, 90.

COMPONENTS:	ORIGINAL MEASUREMENTS:
(1) Benzenesulfonamide, 4-amino- (sulfanil-	Gusyakov, V. P.; Likhol'ot, N. M.;
amide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Kutna, I. M. Farm. 2h. (Kiev) <u>1968,</u>
(2) Poly(oxy-1,2-ethanediy1), α -hydro- ω -	23(6), 56-61.
hydroxy- (PEG 400); (C ₂ H ₄ 0) _n H ₂ 0;	
[25322-68-3] 400	
VARIABLES:	PREPARED BY:
One temperature: 21-25°C	R. Piekos
one temperature. 21-25 C	r. riekos
DUDD TAGUMAT VALUEO.	
EXPERIMENTAL VALUES:	
	İ
Solubility of sulfanilamide in α -hydro-	- ω -hydroxypoly(oxy-1,2-ethanediy1) 400
at room temperature (21-25°C) is 87.	5% by weight (40.7 mol kg ⁻¹ PEG 400 ,
compiler).	
AUUTT T ADV	TUPO DA MACAN
	INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Small quantities (2-4 mg) of sulfanilamide	Sulfanilamide: neither source nor purity
were added to a known quantity of PEG 400	were specified. PEG 400: source not spe-
under stirring until sath was attained.	cified; sp gr 1.127 g cm ⁻³ ; temp of
	solidification approx 6°C; refractive
	index 1.466 (temp not indicated).
	1
	ESTIMATED ERROR:
	Nothing specified.
	and oppositions
	REFERENCES:
1	}

132 COMPONENTS: ORIGINAL MEASUREMENTS: (1) Benzenesulfonamide, 4-amino-Khawan, M. N.; Tawashi, R.; Czetsch-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] Lindenwald, H. v. Sci. Pharm. 1964, 32, (2) Poly(oxy-1,2-ethanediy1), α-hydro-ω-hydroxy- (PEG 400); (C₂H₄O)₁H₂O; [25322-68-3] 400 271-9. VARIABLES: PREPARED BY: One temperature: 45°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in PEG 400 at 45°C is 15.25 g/100 g PEG 400 (0.8856 mol kg⁻¹, compiler). AUXILIARY INFORMATION SOURCE AND PURITY OF MATERIALS: METHOD/APPARATUS/PROCEDURE: Sulfanilamide conformed to the require-Small weighed samples of sulfanilamide were added to PEG 400 under stirring ments of USP XVI. until dissoln occurred. Source and purity of PEG 400 was not specified.

ESTIMATED ERROR:

Soly: measurements were made in

duplicate (authors).

Temp: ±1°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 600); (C₂H₄O)_nH₂O; [25322-68-3] 600
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 153-61.

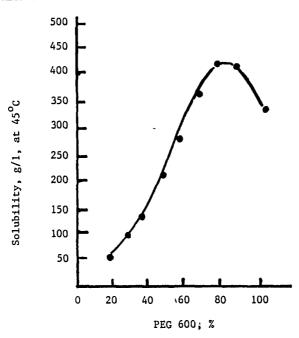
VARIABLES:

Concentration of PEG 600

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier developed method was employed
(1) whereby a 100-ml conical flask contg
a PEG 600 soln was placed in a drying
cabinet at a given temp and an excess of
sulfanilamide was added under stirring for
1 h. After 12 h the soln was filtered or
decanted and the solute was assayed in the
filtrate spectrophotometrically at 260 mµ
using a Unicam SP 500 spectrophotometer
and 1-ml quartz cuvets. Results were taken
from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of BP 1953 or USP XIV. It was recrystd, dried, powdered, and again dried at 105° C to const wt. PEG 600 was a product of Farbwerke Hoechst. It was kept over concd $\mathrm{H}_2\mathrm{SO}_{\Delta}$ in a desiccator.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 90.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 600); (C₂H₄O)_nH₂O; [25322-68-3] 600
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Likhol'ot, N. M.; Kutna, I. M. Farm. Zh. (Kiev) 1968, 23(6), 56-61.

VARIABLES:

One temperature: 21-25°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 5% (by weight) aqueous α -hydro- ω -hydroxypoly(oxy-1,2-ethanediy1) 600 at room temperature (21-25°C) is 0.702 g/100 ml (4.08 x 10⁻² mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was added to a 5% (by wt) aq PEG 600 soln, the mixt was sealed in an ampul and agitated for 24 h (1). The concn of sulfanilamide was detd colorimetrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide: neither source nor purity was specified. PEG 600 was of the Austrian or West German origin; purity not specified.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M.; Kutna, I. M. Farm. Zh. (Kiev) 1967, 22(3), 34.
- Predchetenskii, B. E.; Borovskaya,
 V. M.; Morgolina, L. T. Laboratornye metody issledovaniya, Medgiz, Moscow 1950, p. 371

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- Poly(oxy-1,2-ethanediy1), α -hydroω-hydroxy- (PEG 1500); (C₂H₄O)_nH₂O; [25322-68-3] 1500
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 151-61.

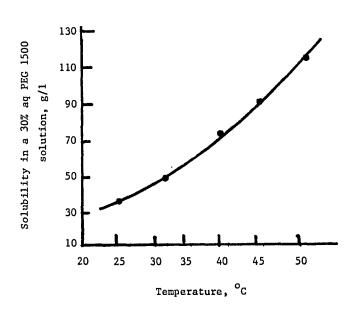
VARIABLES:

PREPARED BY:

R. Piekos

Temperature

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier developed method was employed (1) whereby a 100-ml conical flask contg a PEG 1500 soln was placed in a drying cabinet at a given temp and an excess of sulfanilamide was added under stirring for 1 h. After 12 h the soln was filtered or decanted and the solute was assayed in the filtrate spectrophotometrically at 260 mu using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed with the requirements of BP 1953 and USP XIV. It was recrystd, dried, powdered, and again dried at 105°C to const wt. PEG 1500 was a product of Farbwerke Hoechst. It was kept over concd H2SO4 in a desiccator.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

1. Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 90.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 4000); (C₂H₄O)_nH₂O; [25322-68-3] 4000
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Soi. Pharm. 1964, 32, 271-9.

VARIABLES:

One temperature: 45°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 10% aqueous solution of PEG 4000 at 45° C is 20.5 g/100 g PEG 4000 solution (1.19 mol kg⁻¹, compiler).

AUXILIARY INFORMATION

METHOD /APPARATUS / PROCEDURE:

Sulfanilamide was added in excess to an aq PEG 4000 soln and the mixt was stirred for 30 min. The soln was then placed in a drying cabinet for 24 h and occasionally shaken. After filtration the sulfonamide was assayed in the filtrate by the USP XVI method based on diazotization. The end point was detected by means of a starch paste as an indicator. Corrections were made for consumption of the 0.1 N NaNO₂ soln by PEG 4000.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: measurements were made in duplicate (authors).

Temp: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 4000); (C₂H₄O)_nH₂O; [25322-68-3] 4000
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Likhol'ot, N. M.; Kutna, I. M. Farm. Zh. (Kiev) 1968, 23(6), 56-61.

VARIABLES:

One temperature: 21-25°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 5% (by weight) aqueous α -hydro- ω -hydroxypoly(oxy-1,2-ethanediy1) 4000 at room temperature (21-25°C) is 0.766 g/100 ml (4.45 x 10^{-2} mol dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A small excess of sulfanilamide was added to a 5% (by wt) aq PEG 4000 soln, the mixture was sealed in an ampul and agitated for 24 h (1). The concn of sulfanilamide was detd colorimetrically (2).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide: neither source nor purity was specified. PEG 4000 was of the Austrian or West German origin. Its purity was not specified.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

- Gusyakov, V. P.; Likhol'ot, N. M.; Kutna, I. M. Farm. Zh. (Kiev) 1967, 22(3), 34.
- Predchetenskii, B. E.; Borovskaya,
 V. M.; Morgolina, L. T. Laboratormye metody issledovaniya, Medgiz, Moscow 1950, p. 371.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 6000); (C₂H₄O)₁H₂O; [25322-68-3] 6000 (3) Water; H₂O; [7732-18-5] VARIABLES: One temperature: 45°C ORIGINAL MEASUREMENTS: Khawam, M. N.; Tawashi, R.; CzetschLindenwald, H. v. Soi. Pharm. 1964, 32, 271-9.

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in a 10% aqueous PEG 6000 solution at 45° C is 21.8 g/100 g PEG 6000 solution (1.27 mol kg⁻¹, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in excess to an aq PEG 6000 soln and the mixt was stirred for 30 min. The soln was then placed in a drying cabinet for 24 h and occasionally shaken. After filtration the sulfonamide was assayed in the filtrate by the USP XVI method based on diazotization. The end point was detected by means of a starch paste as an indicator. Corrections were made for consumption of the 0.1 N NaNO₂ soln by PEG 6000.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Purity of the remaining materials was not specified.

ESTIMATED ERROR:

Soly: measurements were made in duplicate (authors).

Temp: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (PEG 6000); (C2H40) nH20; [25322-68-3] 6000
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Kawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 153-61.

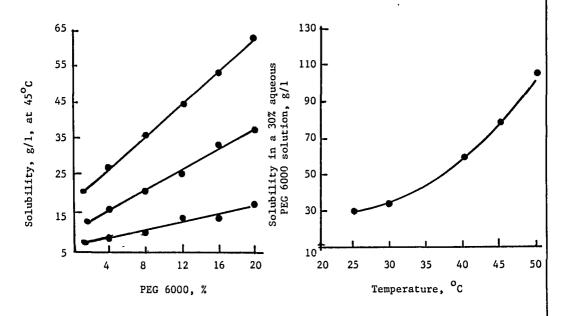
VARIABLES:

Concentration of PEG 6000; temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An earlier developed method was employed (1) whereby a 100-ml conical flask contg a PEG 6000 soln was placed in a drying cabinet at a given temp and an excess of sulfanilamide was added under stirring for 1 h. After 12 h the soln was filtered or decanted and the solute was assayed in the filtrate spectrophotometrically at 260 mµ using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed with the requirements of BP 1953 or USP XIV. It was recrystd, dried, powdered, and again dried at 105°C to const wt. PEG 6000 was a product of Farbwerke Hoechst. It was kept over concd H_2SO^4 in a desiccator.

Purity of the water was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 90.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monolaurate, polyoxyethylene derivatives (Tween 20); [9005-64-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1964, 32, 271-9.

VARIABLES:

Temperature; concentration of Tween 20

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/°C Solubility of sulfanilamide in g/1 (10² mol dm^{-3^a)} at various Tween 20 concentrations

	0.1%	0.5%	1%	2%	3%	4% (w/v)
16	4.43	4.71	4.91	5.62	6.10	6.75
	(2.57)	(2.73)	(2.85)	(3.26)	(3.54)	(3.92)
24	6.98	7.6	8.20	9.18	9.81	10.33
	(4.05)	(4.41)	(4.76)	(5.33)	(5.70)	(6.00)
34	12.85	13.47	13.9	15.54	16.14	16.88
	(7.46)	(7.82)	(8.07)	(9.02)	(9.37)	(9.80)
44	21.22	21.98	21.22	23.81	25.05	27.72
	(12.32)	(12.76)	(12.32)	(13.83)	(14.55)	(16.10)

aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in excess to an aq Tween 20 soln and the mixture was stirred for 30 min. The soln was then placed in a drying cabinet for 24 h and occasionally shaken. After filtration the sulfanilamide was assayed in the filtrate by the USP XIV method based on diazotization . The end point was detected by means of a starch paste as an indicator. Corrections were made for consumption of the 0.1 N NaNO₂ soln by Tween 20.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of the USP XVI.

Tween 20 was a commercially available reagent with a HLB value of 16.7, manufd by Atlas Goldschmidt A. G., Essen, West Germany.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: Measurements were made in duplicate (authors).

Tem;: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monolaurate, polyoxyethylene derivatives (Tween 20); [9005-64-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

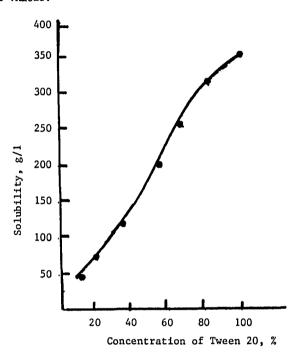
Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 90-101.

VARIABLES:

Concentration of Tween 20

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A 100-ml conical flask contg a Tween 20 soln was placed in a drying cabinet at 25°C and sulfanilamide was added in excess under stirring for 1 h. After 12 h the soln was filtered or decanted and sulfanilamide was assayed in the filtrate spectrophotometrically at 260 mµ using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (BP 1953 and USP XIV) was recrystd, dried, powdered, and dried again at 105°C to const wt. Tween 20 was an Atlas-Goldschmidt product with HLB = 16.7 and dielec const 9.89.

Distd water was used.

ESTIMATED ERROR:

Soly: not specified. Temp: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monolaurate, polyoxyethylene derivatives (Tween 20); [9005-64-5]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Likhol'ot, N. M.; Kutna, I. M. Farm. Zh. (Kiev) 1967, 22(3), 34-9.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

$$S/S_0 = 1.23 \text{ at } 20^{\circ}C$$

where S is the solubility of sulfanilamide in a 2% by weight aqueous Tween 20 solution, and

 S_{o} is the solubility of sulfanilamide in water (0.53 g/100 ml).

Hence S = 0.65 g/100 ml (3.8 x 10^{-2} mol dm⁻³) - compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide in a 2% by wt aq Tween 20 soln was equilibrated for 24 h in an ampul immersed in a water thermostat. Aliquots of the satd soln were withdrawn through a filter and the sulfanilamide content was assayed in the filtrate photometrically.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of the State Pharmacopeia IX.

Tween 20 was a product of Gee Lawson, England.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: $\pm 0.1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monopalmitate, polyoxyethylene derivatives (Tween 40); [9005-66-7]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Likhol'ot, N. M.;
Kutna, I. M. Farm. 2h. (Kiev.) 1967, 22(3)

Kutna, I. M. Farm. Zh. (Kiev) 1967, 22(3)

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

 $S/S_0 = 1.1 \text{ at } 20^{\circ}C$

where S is the solubility of sulfanilamide in a 2% by weight aqueous Tween 40 solution, and

 S_{α} is the solubility of sulfanilamide in water (0.53 g/100 ml).

Hence S = 0.58 g/100 ml (3.4 x 10^{-2} mol dm⁻³) - compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide in a 2% by wt aq Tween 40 soln was equilibrated for 24 h in an ampul immersed in a water thermostat. Aliquots of the satd soln were withdrawn through a filter and the sulfanilamide content was assayed in the filtrate photometrically.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of the State Pharmacopeia IX.

Tween 40 was a product of Gee Lawson, England.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: +0.1°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monostearate, polyoxyethylene derivatives (Tween 60); [9005-67-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. *Sci. Pharm.* 1964, 32, 271-9.

VARIABLES:

Temperature; concentration of Tween 60

PREPARED BY: R. Piekos

EXPERIMENTAL VALUES:

t/°C Solubility of sulfanilamide in g/1 (10² mol dm^{-3^a}) at various Tween 60 concentrations

				actons		
	0.1%	0.5%	1%	2%	3%	4% (w/v)
15	4.04	4.32	4.59	5.22	5.70	6.31
	(2.35)	(2.51)	(2.66)	(3.03)	(3.31)	(3.66)
24	7.25	7.67	8.67	9.23	9.92	10.7
	(4.21)	(4.45)	(5.03)	(5.36)	(5.76)	(6.21)
34	12.71	13.50	14.49	15.28	16.51	17.05
	(7.38)	(7.84)	(8.41)	(8.87)	(9.59)	(9.90)
44	20.79	21.35	21.63	23.20	24.65	26.26
	(12.07)	(12.40)	(12.56)	(13.47)	(14.31	(15.25)

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in excess to an aq Tween 60 soln and the mixture was stirred for 30 min. The soln was then placed in a drying cabinet for 24 h and occasionally shaken. After filtration the sulfanilamide was assayed in the filtrate by the USP XVI method based on diazotization. The end point was detected by means of a starch paste as an indicator. Corrections were made for consumption of the 0.1N NaNO₂ soln by Tween 60.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Tween 60 was a commercially available reagent (source and purity not specified).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: Measurements were made in duplicate (authors).

Temp: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monolaurate, polyoxyethylene derivatives (Tween 60); [9005-67-8]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1965, 33, 90-101.

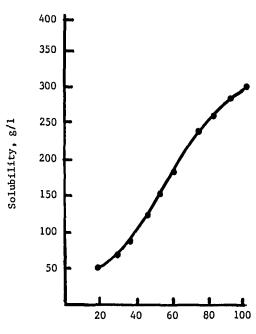
VARIABLES:

Concentration of Tween 60

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



Concentration of Tween 60, %

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A 100-ml conical flask contg Tween 60 soln was placed in a drying cabinet at 25°C and sulfanilamide was added in excess under stirring for 1 h. After 12 h the soln was filtered or decanted and sulfanilamide was assayed in the filtrate spectrophotometrically at 260 mµ using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (BP 1953 and USP XIV) was recrystd, dried, powdered, and dried again at 105°C to const wt. Tween 20 was an Atlas-Goldschmidt product with HLB = 14.9 and dielec const 8.27.

Distd water was used.

ESTIMATED ERROR:

Soly: not specified.

Temp: $\pm 1^{\circ}C$ (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monooleate, polyoxyethylene derivatives (Tween 80); [9005-65-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1964, 32, 271-9.

VARIABLES:

Temperature; concentration of Tween 60

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/°C Solubility of sulfanilamide in g/l (10² mol dm^{-3^a}) at various
Tween 80 concentrations

		_				
	0.1%	0.5%	1%	2%	3%	4% (w/v)
13	3.62	3.86	4.06	4.68	5.30	5.88
	(2.10)	(2.24)	(2.36)	(2.72)	(3.08)	(3.41)
24	7.40	7.76	8.21	8.90	10.37	10.85
	(4.30)	(4.51)	(4.77)	(5.17)	(6.02)	(6.30)
34	12.47	13.47	13.50	13.7	15.2	17.69
	(7.24)	(7.82)	(7.84)	(7.96)	(8.83)	(10.27)
44	21.05	22.02	21.42	24.15	25.79	26.71
	(12.22)	(12.79)	(12.44)	(14.02)	(14.98)	(15.51)

aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was added in excess to an aq Tween 80 soln and the mixt was stirred for 30 min. The soln was then placed in a drying cabinet for 24 h and occasionally shaken. After filtration the sulfanilamide was assayed in the filtrate by the USP XVI method based on diazotization. The end point was detected by means of a starch paste as an indicator. Corrections were made for consumption of the 0.1N NaNO₂ soln by Tween 80.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Tween 80 was a commercially available reagent (source and purity not specified).

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: Measurements were made in duplicate (authors).

Temp: +1°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monolaurate, polyoxyethylene derivatives (Tween 80); [9005-65-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. *Sci. Pharm.* 1965, 33, 90-101.

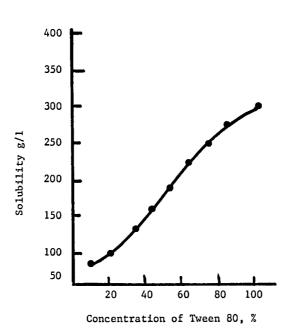
VARIABLES:

Concentration of Tween 80

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:



AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A 100-ml conical flask contg a Tween 80 soln was placed in a drying cabinet at 25°C and sulfanilamide was added in excess under stirring for 1 h. After 12 h the soln was filtered or decanted and sulfanilamide was assayed in the filtrate spectrophotometrically at 260 mµ using a Unicam SP 500 spectrophotometer and 1-ml quartz cuvets. Results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (BP 1953 and USP XIV) was recrystd, dried, powdered, and dried again at 105° C to const wt. Tween 80 was an Atlas-Goldschmidt product with HLB = 15.0 and dielec const 8.75.

Distd water was used.

ESTIMATED ERROR:

Soly: not specified. Temp: +1°C (authors).

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monooleate, polyoxyethylene derivatives (Tween 80); [9005-65-6]
- (3) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Gusyakov, V. P.; Likhol'ot, N. M.;
Kutna, I. M. Farm. Zh. (Kiev) 1967, 22(3),
34-9.

VARIABLES:

One temperature: 20°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

$$S/S_0 = 1.19 \text{ at } 20^{\circ}C$$

where S is the solubility of sulfanilamide in a 2% by weight aqueous Tween 80 solution, and

 S_0 is the solubility of sulfanilamide in water (0.53 g/100 ml).

Hence S = $0.63 \text{ g/100 m1} (3.7 \times 10^{-2} \text{ mol dm}^{-3}) - \text{compiler}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide in a 2% by wt aq Tween 80 soln was equilibrated for 24 h in an ampul immersed in a water thermostat. Aliquots of the satd soln were withdrawn through a filter and the sulfanilamide content was assayed in the filtrate photometrically.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of the State Pharmacopeia IX.

Tween 80 was a product of Gee Lawson, England.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.
Temp: ±0.1°C (authors).

ORIGINAL MEASUREMENTS: COMPONENTS: (1) Benzenesulfonamide, 4-amino-(sulfanilamide); ${}^{C}_{6}{}^{H}_{8}{}^{N}_{2}{}^{O}_{2}{}^{S}$; [63-74-1] Becher, R.; Leya, S. Experientia 1946, 2, 459-60. (2) D-Glucose; C₆H₁₂O₆; [50-99-7] (3) Water; H₂O; [7732-18-5] **VARIABLES:** PREPARED BY: One temperature: 18-19°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in a 10% D-glucose solution at room temperature $(18-19^{\circ}C)$ is 654 mg% $(3.80 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler})$. AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: After standing for more than two days the Nothing specified. soln of sulfanilamide was filtered and the sulfonamide was assayed in the filtrate colorimetrically by the method of Druey and Oesterheld (1). ESTIMATED ERROR: Nothing specified. REFERENCES: 1. Druey, J.; Oesterheld, G. Helv. Chim. Acta 1942, 25, 753.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) 2-Propanol; C₃H₈O; [67-63-0] VARIABLES: ORIGINAL MEASUREMENTS: Burlage, H. M. J. Am. Pharm. Assoc., Sci. Ed. 1948, 37 345.

EXPERIMENTAL VALUES:

One temperature: 25°C

Solubility of sulfanilamide in 2-propanol at 25° C is 0.7970 g/100 cm³ solution $(4.628 \times 10^{-2} \text{ mol dm}^{-3}, \text{ compiler})$.

R. Piekos

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Satd solns of sulfanilamide in 2-propanol were prepd at 25°C and definite vols of the solns were measured into tared dishes by means of standard pipets. The alcohol was allowed to evap at room temp and the residue was dried at 105°C. In the case of losses due to apparent decompn, the residue was dried in a dessicator (1).

SOURCE AND PURITY OF MATERIALS:

The sulfonamide was manufd by Gane and Ingram and was of the U.S.P. purity. The source and purity of 2-propanol was not specified.

ESTIMATED ERROR:

Nothing specified.

REFERENCES:

 Burlage, H. M. J. Am. Pharm. Assoc., Sci. Ed. 1947, 36(1), 16.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- (2) 1-Butanol; C₄H₁₀O; [71-36-3]

ORIGINAL MEASUREMENTS:

Burger, A.

Sci. Pharm. 1973, 4, 303-14.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/ ^o C	Saturation solubility, C _s , of crystalline form II		
	mg/100 ml soln	10^2 mol dm $^{-3a}$	
10.4	192	1.12	
15.3	226	1.31	
20.0	272	1.58	
26.0	336	1.95	
30.5	392	2.28	
35.5	462	2.68	
40.9	552	3.21	
45.8	648	3.76	

t/ ^o C	Saturation solu crystalline for	m II
	mg/100 ml soln	10 ² mol dm ^{-3a}
50.5	780	4.53
55.8	937	5.44
60.5	1096	6.36
66.0	1325	7.69
70.5	1596	9.27
75.5	1963	11.4
80.3	2261	13.1

The following equation was derived based on the above data ($c_{\rm g}$ is in mg/100 ml solution): $C_s = 156.79 + 4643.79 \text{ T}^{-1} + 25.80 \text{ lnT}$, where T is absolute temperature.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

method (1). Sulfanilamide was assayed photometrically at 263 nm ($E_{1 \text{ cm}}^{1\%} \approx 1130$) on a Zeiss - PMQ II spectrophotometer.

SOURCE AND PURITY OF MATERIALS:

Satn soly was detd by the earlier developed Cryst form II of sulfanilamide mp 156°C, was obtained by crystn from 96% EtOH (2). Source and purity of the BuOH was not specified.

ESTIMATED ERROR:

Soly: not specified. Temp: $\pm 0.1^{\circ}$ C (author).

- 1. Kuhnert-Brandstätter, M.; Burger, A. Pharm. Ind. 1972, 34, 187.
- 2. Burger, A. Sci. Pharm. 1973, 41, 290.

aCalculated by compiler.

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) 1-Pentanol, (amyl alcohol); C₅H₁₂O; [71-41-0]

ORIGINAL MEASUREMENTS:

Lin, O. H.; Ph.D. Dissertation; The University of Iowa; Iowa City, IA; 1971; p. 76.

VARIABLES:

Four temperatures; four crystalline forms.

PREPARED BY:

J. K. Guillory

EXPERIMENTAL VALUES:

Solubilities of Sulfanilamide Polymorphic Forms:

t/°C		mol dm ⁻³	•	
τ, σ	α	β	Υ	δ
30.0	1.36 x 10 ⁻²	1.26 x 10 ⁻²	1.35 x 10 ⁻²	1.49 x 10 ⁻²
37.0	1.74×10^{-2}	1.73×10^{-2}	1.72×10^{-2}	1.93×10^{-2}
45.0	2.32×10^{-2}	2.34×10^{-2}	2.33×10^{-2}	2.50×10^{-2}
50.0	2.90×10^{-2}	2.90×10^{-2}	2.86×10^{-2}	3.04×10^{-2}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of sulfanilamide was placed in 20-ml screw-capped vials with 20 ml of pentanol. Vials were rotated end-over-end in a bath whose temperature was controlled to ±0.1°C. Equilibrium was attained after 36 h or less. Supernatant was filtered through sintered glass, diluted 1:24 with 95% ethanol, and analyzed spectrophotometrically at 262 nm. Measurements were performed in duplicate. Residual crystals were analyzed by differential thermal analysis to detect change in crystal form. None was observed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (Mallinckrodt) was recrystd from 95% ethanol, methanol, n-pentanol, or n-butanol. Crystal forms were identified using densities, refractive indexes, x-ray diffraction measurements (1) and by infrared spectrophotometry and differential thermal analysis (2).

1-Pentanol (Fisher Certified).

ESTIMATED ERROR:

Uncertainty of temperature (±0.1°C). Uncertainty of solubility measurements probably (1-2%) based on agreement of duplicate absorbance measurements.

- Lin, H. O.; Baenziger, N. C.; and Guillory, J. K., J. Pharm. Soi. 1974, 63, 145-6.
- Lin, H. O.; Guillory, J. K., J. Pharm. Sci. 1970, 59, 972-5.

153 COMPONENTS: ORIGINAL MEASUREMENTS: (1) Benzenesulfonamide, 4-amino-Mingoia, Q. (sulfanilamide); C₆H₈N₂O₂S; [63-74-1] Ann. Chim. Farm. (Suppl. to Farm. Ital.) (2) 1,2-Ethanediol; C₂H₆O₂; [107-21-1] Apr., 1939, 48-58. VARIABLES: PREPARED BY: One temperature: 20°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in 1,2-ethanediol at $20^{\circ}\mathrm{C}$ is 9.80 wt.% (0.569 mol kg⁻¹ solution, compiler). AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Five cm³ of a sulfanilamide soln in 1,2-ethanediol were diluted with distilled water to 50 cm³ and sulfanilamide was assayed in this solution by known methods (probably colorimetric).

SOURCE AND PURITY OF MATERIALS:

Source and purity of sulfanilamide were not specified.

1,2-Ethanediol was from Merck (purity not specified).

ESTIMATED ERROR:

Nothing specified.

COMPONE	ENTS .	ORIGINAL MEASUREMENTS:
COMFONE		
(1)	Benzenesulfonamide, 4-amino-	Mingoia, Q.
(2)	(sulfanilamide); $C_{6}H_{8}N_{2}O_{2}S$; [63-74-1]	Ann. Chim. Farm. (Suppl. to Farm. Ital.)
(2)	1,2-Propanedio1; C ₃ H ₈ O ₂ ; [57-55-6]	<i>Apr.</i> , <u>1939</u> , 48-58.
ļ		
VARIABI	LES:	PREPARED BY:
	One temperature: 20°C	R. Piekos
EXPERI	MENTAL VALUES:	
ĺ	Solubility of sulfanilamide in 1,2-pro	panediol at 20°C is 11.46 wt%
]	(0.6655 mol kg^{-1} solution, compiler).	
ļ		
]		
l		
j		
1		
1		
1	AUXILIARY	INFORMATION
METHOD	/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
	cm ³ of a sulfanilamide soln in	Source and purity of sulfanilamide were
	propanediol were diluted with dis-	not specified.
	ed water to 50 cm ³ and sulfanilamide	
	assayed in this solution by known	1,2-Propanediol was from Merck (purity not
1	assayed in this solution by known and colorimetric).	specified).
mecn	lods (probably colorimetric).	
1		
ļ		
1		ESTIMATED ERROR:
t		Nothing specified.
1		
		REFERENCES:
		}
}		
1		}

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
 (2) Poly(oxy-1,2-ethanediyl), α-hydro-
- (2) Poly(oxy-1,2-ethaned1y1), α-hydroω-hydroxy- (PEG 400); (C₂H₄O)_nH₂O; [25322-68-3] 400

ORIGINAL MEASUREMENTS:

Wahlgren, S., Svensk farm. tidskr. 1962, 66, 585-91.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

O	Solubility	in PEG 4000
t/ [°] C	weight%	mol kg ^{-1a}
20	21	1.5
60	37	3.4

^aCalculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soly detns were made in 100-cm³ Erlenmeyer flasks immersed in a consttemp bath. The suspension was stirred with an electrically driven propeller stirrer for at least 4 h.

SOURCE AND PURITY OF MATERIALS:

The source and purity of sulfanilamide was not specified. PEG 400: pH 4.7 (1.00 g in 20.0 g of water), ash content 0.030%, free acid: 0.30 cm³ of 0.1 N NaOH soln was required to neutralize free acids in 5.0 g of PEG 400 dissolved in 20 cm³ of EtOH; average mol wt 400; water content 0.2%.

ESTIMATED ERROR:

Temp: $\pm 0.5^{\circ}$ C (author).

Soly: duplicate tests were made of concns on both sides of the borderline value (author).

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Poly(oxy-1,2-ethanediy1), α-hydroω-hydroxy- (poly(ethylene glyco1) (PEG 3000); (C₂H₄O)_nH₂O; [25322-68-3] 3000 VARIABLES: One temperature: 60°C ORIGINAL MEASUREMENTS: Wahlgren, S., Svensk farm. tidskr. 1962, 66, 585-91.

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in poly(ethylene glycol) 3000 at 60° C is 17% by weight (0.99 mol kg⁻¹, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

The soly detns were made in 100-cm³ Erlenmeyer flasks immersed in a consttemp bath. The suspension was stirred with an electrically driven propeller stirrer for at least 4 h.

SOURCE AND PURITY OF MATERIALS:

The source and purity of sulfanilamide was not specified. PEG 3000: mp 56°C, pH 6.4 (1.00 g in 20.0 g of water); ash content 0.025%; free acid: 0.05 cm³ of 0.1 N NaOH was required to neutralize free acids in 5.0 g of PEG dissolved in 20 cm³ of EtOH against phenolphthalein; average mol wt 3000; water content 0.4%.

ESTIMATED ERROR:

Temp: $\pm 0.5^{\circ}$ C (author).

Soly: duplicate tests were made of concns on both sides of the borderline value (author).

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Ethanol, 2,2'-oxybis- (diethylene glycol; C₄H₁₀O₃; [111-46-6] VARIABLES: One temperature: 20°C EXPERIMENTAL VALUES: Solubility of sulfanilamide in diethylene glycol at 20°C is 39.66 wt.% (2.303 mol kz⁻¹ solution, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Five cm³ of a sulfanilamide soln in diethylene glycol were diluted with distilled water to 50 cm³ and sulfanilamide was assayed in this solution by known methods (probably colorimetric).

SOURCE AND PURITY OF MATERIALS:

Source and purity of sulfanilamide were not specified.

Diethylene glycol was from Carbide and Carbon Co. (purity not specified).

ESTIMATED ERROR:

Nothing specified.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Benzene; C₆H₆; [71-43-2] ORIGINAL MEASUREMENTS: Lin, H. O.; Ph.D. Dissertation; The University of Iowa; Iowa City, IA; 1971; p. 77.

VARIABLES:

Four temperatures; four crystalline forms

PREPARED BY:

J. K. Guillory

EXPERIMENTAL VALUES:

Solubilities of Sulfanilamide Polymorphic Forms:

t/°C	mole dm ⁻³				
	α	β	Υ	δ	
25.0	2.70 x 10 ⁻⁴	2.60×10^{-4}	2.54 x 10 ⁻⁴	2.68 x 10 ⁻⁴	
30.0	3.12×10^{-4}	2.90×10^{-4}	2.88×10^{-4}	2.93×10^{-4}	
33.0	3.54×10^{-4}	3.52×10^{-4}	3.43×10^{-4}	3.65×10^{-4}	
37.0	3.97×10^{-4}	3.96×10^{-4}	3.97×10^{-4}	4.25×10^{-4}	

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of crystalline sulfanilamide was placed in 20-ml screw-capped vials with 20 ml of benzene. Vials were rotated endover-end in a bath whose temperature was controlled to ±0.1°C. Equilibrium was attained after 36 h or less. Supernatant was filtered through sintered glass, diluted 1:9 with 95% ethanol, and analyzed spectrophotometrically at 272 nm. Measurements were performed in duplicate, triplicate or quadruplicate. Residual crystals were analyzed by differential thermal analysis to detect change in crystal form. None was observed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (Mallinckrodt) was recrystd from 95% ethanol, methanol, n-pentanol, or n-butanol. Crystal forms were identified using densities, refractive indexes, x-ray diffraction measurements (1) and by infrared spectrophotometry and differential thermal analysis (2).

Benzene (Mallinckrodt).

ESTIMATED ERROR:

Uncertainty of temperature (±0.1°C). Uncertainty of solubility measurements probably (±1-2%) based on agreement of replicate absorbance measurements.

- Lin, H. O.; Baenziger, N. C.; and Guillory, J. K., J. Pharm. Soi. 1974, 63, 145-6.
- Lin, H. O.; Guillory, J. K., J. Pharm. Sci. 1970, 59, 972-5.

COMPONENTS: (1) Benzenesulfonamide, 4-amino(sulfanilamide); C₆H₈N₂O₂S; [63-74-1] (2) Methane, trichloro- (chloroform); CHCl₃; [67-66-3] VARIABLES: One temperature: 30°C ORIGINAL MEASUREMENTS: Yamazaki, M.; Aoki, M.; Kamada, A.; Yata, N. Yakuzaigaku 1967, 27(1), 37-40.

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in chloroform at 30°C is 1.32 mmol/L (0.227 g dm⁻³, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide (0.5 g) was placed in an L-shaped tube together with 20 ml of chloroform. The mixt was shaken in a thermostat until equilibrium was attained. The sulfanilamide was then assayed in the supernatant spectrophotometrically at 545 nm on a Beckmann DU spectrophotometer. The results were taken from a calibration graph.

SOURCE AND PURITY OF MATERIALS:

Nothing specified.

ESTIMATED ERROR:

Soly: not specified. Temp: $\pm 1^{\circ}$ C (authors).

ORIGINAL MEASUREMENTS: COMPONENTS: Kitao, K.; Kubo, K.; Morishita, T.; (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] Yata, N.; Kamada, A. Chem. Pharm. Bull. (2) Methane, trichloro-; CHCl₃; [67-66-3] 1973, 21, 2417-26. VARIABLES: PREPARED BY: One temperature: 37°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in CHCl $_3$ at 37 $^{\rm o}$ C is 1.40 mmol dm $^{-3}$ solution. AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: Comm available sulfanilamide was used One ml of the $CHCl_3$ soln of sulfanilamide at equilibrium was taken into a test tube. as supplied. After evapn of the solvent, the residue Purity of the $CHCl_3$ was not specified. was dissolved in 1N NaOH, the soln was properly dild with deionized water, and the concn of sulfanilamide was detd by diazotization. ESTIMATED ERROR: Soly: not specified. Temp: $\pm 1^{\circ}$ C (authors). REFERENCES:

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1]
- 2-Propanone (acetone); C3H60; [67-64-1]

ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

Anales fis. quim. (Madrid) 1945, 41, 537-60.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

t/°C	G ^a	Ep	x _g /1 ^c	mo1/1 acetone	mmol/mol acetone	1:X _g d	1 + X _{cc} e
0	25.620	20.395	208.701	1.212	86	3.90	4.79
5	27.299	21.445	220.997	1.283	92	3.66	4.53
10	27.597	21.628	222.104	1.289	93	3.62	4.52
15	28.318	22.069	225.723	1.311	95	3.53	4.43
20	29.965	23.056	237.083	1.377	101	3.34	4.22
25	31.132	23.741	244.445	1.419	105	3.21	4.09
30	32.516	24.537	253.397	1.471	110	3.08	3.95
35	34.509	25.656	266.858	1.549	116	2.90	3.75
40	37.067	27.043	284.452	1.652	125	2.70	3.52
45	39.914	28.578	303.905	1.765	135	2.51	3.29
50	45.110	31.087	340.806	1.979	152	2.22	2.93

 $^{^{}a}_{G}=\frac{p}{P}\frac{100}{-p},$ where p and P are the weights of solute and solution, resp. $^{b}_{E}=\frac{G}{G}\frac{100}{+100}$.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

A special all-glass app was constructed enabling the prepn of satd solns, agitation by bubbling a stream of Me, CO-satd N, filtration, and distn off the solvent without the contact with air. Two exchangeable dissoln vessels of 15 and 8 $\,\mathrm{cm}^3$ working capacity were used depending on the soly of the solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm³ and the equilibration time was 2-2.5 h. The satd solns were filtered, weighed, the solvent was distd off, the residues were dried at 105°C, weighed, and examd for the presence of solvated Me,CO.

SOURCE AND PURITY OF MATERIALS:

The source of the materials was not specified. Pure, anhyd Me, CO was used. The absence of impurities and water was confirmed by procedures of the German Pharmacopeia VI and Spanish Pharmacopoeia VIII.

The purity of sulfanilamide was not specified.

ESTIMATED ERROR:

Soly: measurements were repeated until obtaining 2 values not differing in the second decimal (author).

Temp: ±0.1°C (author).

cg/l acetone.

dg of acetone required to dissolve 1 g of solute.

evolume (cm³) of acetone required to dissolve 1 g of solute.

COMPONENTS:		ORIGINAL MEASUREMENTS:		
(1) Benzenesulfonamide, 4-amino-		Barber, H. J.; Wilkinson, J. H.		
	(sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	Quart. J. Pharm. Pharmacol. 1946, 19,		
(2)	Methylcyclohexanone; C ₇ H ₁₂ O; [1331-22-2]	248-55.		
	[1331-22-2]			
VARI	ABLES:	PREPARED BY:		
	One temperature: 25°C	R. Piekos		
EXPE	RIMENTAL VALUES:			
	Approximate solubility of sulfanilamide	in methylcyclohexanone at 25°C		
	is 4.7 per cent w/v (0.27 mol dm ⁻³ solut	ion, compiler).		
		,,		
		Ĭ		
		}		
	AUXILIARY	INFORMATION		
METH	OD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:		
	ob/in : indizos/ t Noobbolds.	TOURIST AND TORTH OF MATERIALS,		
No	thing specified.	Nothing specified.		
		ļ		
		ESTIMATED ERROR:		
		Nothing specified.		
		REFERENCES:		

COMPONENTS: (1) Benzenesulfonamide, 4-amino- (sulfanilamide); C ₆ H ₈ N ₂ O ₂ S; [63-74-1]	
(2) Methylcyclohexanone; C ₇ H ₁₂ O; [1331-22-	-2) -2)
VARIABLES:	PREPARED BY:
One temperature: 25°C	R. Piekos
EXPERIMENTAL VALUES:	
Approximate solubility of sulfanilamic 4.7 per cent w/v (0.27 mol dm ⁻³ solution)	ie in methylcyclohexanone at 25°C is ion, compiler).
AUVITTAL	RY INFORMATION
METHOD/APPARATUS/PROCEDURE:	SOURCE AND PURITY OF MATERIALS:
Nothing specified.	Nothing specified.
	ESTIMATED ERROR:
	Nothing specified.
	REFERENCES:

ORIGINAL MEASUREMENTS: COMPONENTS: (1) Benzenesulfonamide, 4-amino-Khawam, M. N.; Tawashi, R.; Czetsch-(sulfanilamide); $C_6H_8N_2O_2S$; [63-74-1] Lindenwald, H. v. Soi. Pharm. 1964, (2) Sorbitan monolaurate (Span 20); ${}^{\rm C}_{18}{}^{\rm H}_{34}{}^{\rm O}_{6}$; [1338-39-2] 32, 271-9. VARIABLES: PREPARED BY: One temperature: 45°C R. Piekos EXPERIMENTAL VALUES: Solubility of sulfanilamide in Span 20 at 45°C is 0 g/100 g Span 20. AUXILIARY INFORMATION METHOD/APPARATUS/PROCEDURE: SOURCE AND PURITY OF MATERIALS: Sulfanilamide conformed to the require-Small weighed samples of sulfanilamide ments of USP XVI. were added to Span 20 under stirring until dissoln occurred. Source and purity of Span 20 was not specified. ESTIMATED ERROR: Soly: measurements were made in duplicate (authors). Temp: $\pm 1^{\circ}$ C (authors). REFERENCES:

- (1) Benzenesulfonamide, 4-amino-(sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Sorbitan monooleate, polyoxyethylene derivatives (Tween 80); [9005-65-6]

ORIGINAL MEASUREMENTS:

Khawam, M. N.; Tawashi, R.; Czetsch-Lindenwald, H. v. Sci. Pharm. 1964, 32, 271-9.

VARIABLES:

One temperature: 45°C

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

Solubility of sulfanilamide in Tween 80 at 45° C is 30 g/100 g Tween 80 (1.7 mol kg⁻¹, compiler).

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Small weighed samples of sulfanilamide were added to Tween 80 under stirring until dissoln occurred.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide conformed to the requirements of USP XVI.

Tween 80 was a commercially available reagent with a HLB value of 15.

ESTIMATED ERROR:

Soly: measurements were made in duplicate

(authors).

Temp: $\pm 1^{\circ}$ C (authors).

- (1) Benzenesulfonamide, 4-amino-, (sulfanilamide); C₆H₈N₂O₂S; [63-74-1]
- (2) Butane, 1,1' oxybis-, (butyl ether); $C_8H_{18}O$; [142-96-1]

ORIGINAL MEASUREMENTS:

Lin, H. O.; Ph.D. Dissertation; The University of Iowa; Iowa City, IA; 1971; p. 76.

VARIABLES:

Four temperatures; four crystalline forms

PREPARED BY:

J. K. Guillory

EXPERIMENTAL VALUES:

t/ ^o C	Solubilities of Sulfanilamide Polymorphic Forms: mol dm ⁻³			
	a	β	Υ	δ
30.0	2.55 x 10 ⁻⁴	2.60 x 10 ⁻⁴	2.92×10^{-4}	2.83×10^{-4}
33.0	2.76×10^{-4}	2.80×10^{-4}	3.05×10^{-4}	3.06×10^{-4}
37.0	2.95×10^{-4}	2.98×10^{-4}	3.26×10^{-4}	3.37×10^{-4}
45.0	3.56×10^{-4}	3.60×10^{-4}	3.84 x 10 ⁻⁴	3.88×10^{-4}

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of crystalline sulfanilamide was placed in 20-ml screw-capped vials with 20 ml of butyl ether. Vials were rotated end-overend in a bath whose temperature was controlled to ±0.1°C. Equilibrium was attained after 36 h or less. Supernatant was filtered through sintered glass, diluted 2:23 with 95% ethanol, and analyzed spectrophotometrically at 262 nm. Measurements were performed in triplicate. Residual crystals were analyzed by differential thermal analysis to detect change in crystal form. None was observed.

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide (Mallinckrodt) was recrystd from 95% ethanol, methanol, n-pentanol, or n-butanol. Crystal forms were identified using densities, refractive indexes, x-ray diffraction measurements (1) and by infrared spectrophotometry and differential thermal analysis (2).

Butyl ether (Matheson Coleman & Bell).

ESTIMATED ERROR:

Uncertainty of temperature (±0.1°C). Uncertainty of solubility measurements probably (±1-2%) based on agreement of triplicate absorbance measurements.

- Lin, H. O.; Baenziger, N. C.; and Guillory, J. K., J. Pharm. Sci. 1974, 63, 145-6.
- Lin, H. O.; Guillory, J. K., J. Pharm. Sci. 1970, 59, 972-5.

- (1) Benzenesulfonamide, 4-amino-, monohydrate, (sulfanilamide monohydrate); $C_6^{H_8}N_2^{O_2}S \cdot H_2^{O_3}$ [20203-81-0]
- (2) Water; H₂O; [7732-18-5]

ORIGINAL MEASUREMENTS:

Burger, A.

Pharm. Ind. 1973, 35, 626-33.

VARIABLES:

PREPARED BY:

Temperature

R. Piekos

EXPERIMENTAL VALUES:

t/ ^o c	Saturation solubility, C a			
-, -	mg/100 ml solution	10 ² mol dm ^{-3b}		
4.4	182	0.957		
10.2	251	1.32		
15.0	337	1.77		
20.0	465	2.44		
25.0	600	3.15		
30.0	820	4.31		
35.0	1100	5.78		
37.0	1270	6.68		

^aC_s = [HA] + [A], where [HA] is the molar concentration of the dissolved, undissociated molecules of sulfanilamide and [A] is the concentration of the dissolved anion of sulfanilamide.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

Sulfanilamide was assayed spectrophotometrically at 258.5 nm using a Zeiss PMQ II spectrophotometer and a 1/15 M phosphate buffer of pH 7.00 ($E_{1~\rm cm}^{1\%}=945$).

SOURCE AND PURITY OF MATERIALS:

Sulfanilamide monohydrate was obtained by vacuum evapn of a sulfanilamide (source and purity not specified) soln at 40°C and cooling the crystals to 20°C at normal pressure.

Purity of the water was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.1°C (author).

bCalculated by compiler.