COMPONENTS: (1) Benzenesulfonamide, 4-amino-N-4- [(aminosulfony1)pheny1]-; (disulfan)  C12H13N3O4S2; [547-52-4]	ORIGINAL MEASUREMENTS:  Becher, R.; Leya, S. Experientia  1946, 2, 459-60.
(2) Water; H <sub>2</sub> 0; [7732-18-5]	
VARIABLES:	PREPARED BY:
One temperature: 18-19°C	R. Piekos
Solubility of 4-amino-N-4-[(aminosu water at room temperature (18-19°C) compiler).	lfonyl)phenyl]benzenesulfonamide in is 30 mg% ( 9.2 x 10 <sup>-4</sup> mol dm <sup>-3</sup> ,
water at room temperature (18-19°C)	
water at room temperature (18-19°C) compiler).	

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

Nothing specified.

## ESTIMATED ERROR:

Nothing specified.

## REFERENCES:

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

# COMPONENTS: (1) Benzenesulfonamide, 4-amino-N-4[(aminosulfonyl)phenyl]-;(disulfan) C12H13N304S2; [547-52-4] (2) Sodium chloride; NaC1; [7647-14-5] (3) Water; H20; [7732-18-5] VARIABLES: One temperature: 18-19°C ORIGINAL MEASUREMENTS: Becher, R.; Leya, S. Experientia 1946, 2, 459-60.

## EXPERIMENTAL VALUES:

Solubility of 4-amino-N-4-[(aminosulfonyl)phenyl]benzenesulfonamide in a 5% NaCl solution at room temperature (  $18-19^{\circ}C$  ) is 28 mg% (  $8.6 \times 10^{-4}$  mol dm<sup>-3</sup>, compiler ).

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

After standing for more than two days the soln of the sulfonamide was filtered and the sulfonamide 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:

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

- (1) Benzenesulfonamide, 4-amino-N-4-[(aminosulfonyl)phenyl]- (disulfan); C<sub>12</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>S<sub>2</sub>; [547-52-4]
- (2) Pectin; (C<sub>13</sub>H<sub>18</sub>O<sub>12</sub>)<sub>n</sub>; [9000-69-5]
- (3) Water; H<sub>2</sub>0; [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 disulfan in a 2.5% pectin solution ( [pectin] =  $6.8 \times 10^{-2}$  mol kg<sup>-1</sup>, compiler ), of pH about 2.6 at room temperature (  $18-19^{\circ}C$  ) is 41 mg% (  $1.3 \times 10^{-3} \text{ mol dm}^{-3}$ , compiler ).

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

The soln was allowed to stand at room temp for more than 2 days. The soln was then filtered, and disulfan 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 cm<sup>3</sup> of a 1 mol dm<sup>-3</sup> NaOH soln was used. The source and purity of disulfan and water were not specified.

## ESTIMATED ERROR:

Nothing specified.

## REFERENCES:

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

- (1) Benzenesulfonamide, 4-amino-N-4-[(aminosulfonyl)phenyl]-, (disulfan);  $C_{12}H_{13}N_3O_4S_2$ ; [547-52-4]  $C_{12}H_{13}N_3O_4S_2;$
- (2) Pectinic acid, sodium salt;  $(C_{13}H_{17}NaO_{12})_n;$  [9049-37-0]
- (3) Water; H<sub>2</sub>O; [7732-18-5]

## ORIGINAL MEASUREMENTS:

Becher, R.; Leya, S. Experientia <u>1946</u>, 2, 459-60.

#### VARIABLES:

One temperature: 18-19°C

## PREPARED BY:

R. Piekos

## EXPERIMENTAL VALUES:

Solubility of disulfan in a 2.6% neutral sodium pectinate solution ([sodium pectinate] =  $6.7 \times 10^{-2} \text{ mol kg}^{-1}$  (n = 1) - compiler) at room temperature (  $18-19^{\circ}$ C ) is 41 mg% (  $1.3 \times 10^{-3}$  mol dm<sup>-3</sup> 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 sulfonamide 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.

- (1) Benzenesulfonamide, 4-amino-N-4-[(aminosulfonyl)phenyl]-; (disulfan)  $C_{12}H_{13}N_3O_4S_2$ ; [547-52-4]
- (2) D-Glucose; C<sub>6</sub>H<sub>12</sub>O<sub>6</sub>; [50-99-7]
- (3) Water; H<sub>2</sub>0; [7732-18-5]

## ORIGINAL MEASUREMENTS:

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

VARIABLES:

PREPARED BY:

One temperature: 18-19°C

R. Piekos

## EXPERIMENTAL VALUES:

Solubility of 4-amino-N-4-[(aminosulfonyl)phenyl]benzenesulfonamide in a 10% D-glucose solution at room temperature (  $18-19^{\circ}C$  ) is 30 mg%  $(9.2 \times 10^{-4} \text{ mol dm}^{-3}, \text{ compiler}).$ 

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

After standing for more than two days the soln of 4-amino-N-4-[(aminosulfonyl)phenyl]benzenesulfonamide was filtered and the sulfonamide 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.

- (1) Benzenesulfonamide, 4-amino-N-4[(aminosulfonyl)phenyl]- (disulfan);

  C<sub>12</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>S<sub>2</sub>; [547-52-4]
- (2) 2-Propanone (acetone); C<sub>3</sub>H<sub>6</sub>0; [67-64-1]

## ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

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

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIM	ENTAL VALI	JES:					
t/°C	$G^{\mathbf{a}}$	Ep	$x_g/1^c$	$mo1/1^{d}$ acetone	mmo1/mo1	1:X <sub>g</sub>	$1+X_{cc}^{f}$
					acetone		
0	49.983	33.325	407.161	1243	88.7	2.00	2.46
5	50.500	33.554	408.444	1248	89.6	1.98	2.45
10	50.998	33.772	409.514	1251	90.5	1.96	2.44
15	52.271	34.327	416.652	1272	92.7	1.91	2.40
20	53.312	34.735	421.805	1288	94.6	1.88	2.37
25	54.024	35.076	424.196	1295	95.8	1.85	2.36
30	55.934	35.742	435.894	1331	99.2	1.81	2.29
35	57.800	36.629	446.967	1365	102.5	1.73	2.24
40	60.198	37.577	461.959	1411	106.8	1.66	2.16
45	64.817	39.326	493.517	1507	114.9	1.54	2.03
50	69.244	40.914	523.139	1597	122.8	1.47	1.91

 $a_{G} = \frac{p\ 100}{P\ -\ p}$  , where p and P are the weights of solute and solution, resp.

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

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

# SOURCE AND PURITY OF MATERIALS:

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

The purity of disulfan was not specified.

## ESTIMATED ERROR:

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

Temp: ±0.1°C (authors).

 $<sup>^{</sup>b}E = \frac{G\ 100}{G+100}$ ;  $^{c}g/1$  acetone;  $^{d}$ should be mmol/1 acetone (compiler);

 $<sup>^{\</sup>rm e}$ g of acetone required to dissolve 1 g of solute;  $^{\rm f}$ volume (cm $^{\rm 3}$ ) of acetone required to dissolve 1 g of solute.

- (1) Benzenesulfonamide, 4-amino-N-4-[(aminosulfonyl)phenyl]-, monohydrochloride (disulfan-HC1); C<sub>12</sub>H<sub>13</sub>N<sub>3</sub>O<sub>4</sub>S<sub>2</sub>·HC1; [77400-69-2]
- (2) 2-Propanone (acetone); C<sub>3</sub>H<sub>6</sub>0;

## ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

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

[67-64-1]

# VARIABLES:

Temperature

## PREPARED BY:

R. Piekos

## EXPERIMENTAL VALUES:

t/°C	G <sup>a</sup>	Ep	X <sub>g</sub> /1 <sup>c</sup>	mol/1 <sup>d</sup> acetone	mmol/mol acetone	1:X <sub>g</sub>	1 + X <sup>f</sup> <sub>cc</sub>
15	0.406	0.404	3.236	8.89	0.65	246.31	309.02
20	0.420	0.418	3.323	9.13	0.67	238.09	300.93
25	0.433	0.431	3.400	9.34	0.69	230.95	294.12

 $a_{G} = \frac{p + 100}{P - p}$ , where p and P are the weights of solute and solution, resp.

 $^{b}E = \frac{G \ 100}{G + 100}$ ;  $^{c}g/1$  acetone;  $^{d}$ should be mmol/1 acetone (compiler);

<sup>e</sup>g of acetone required to dissolve 1 g of solute; <sup>f</sup>volume (cm<sup>3</sup>) of acetone required to dissolve 1 g of solute.

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

A special all-glass app was constructed enabling the prepn of satd solns, agitation by bubbling a stream of acetone-satd N, filtration, and distn off the solvent without contact with air. Two exchangeable dissoln vessels of 15 and 8 cm<sup>3</sup> working capacity were used depending on the soly of solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm<sup>3</sup>, 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  $105^{\circ}\mathrm{C}$ , weighed, and examd for the presence of solvated acetone.

## SOURCE AND PURITY OF MATERIALS:

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

The purity of disulfan-HCl was not specified

## ESTIMATED ERROR:

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

Temp: ±0.1°C (author).

- (1) Benzenesulfonamide, 4-amino-N-4-[(aminosulfonyl)phenyl]-, monosodium salt (Na disulfan); C<sub>12</sub>H<sub>12</sub>N<sub>3</sub>NaO<sub>4</sub>S<sub>2</sub>; [77400-68-1]
- (2) 2-Propanone (acetone); C<sub>3</sub>H<sub>6</sub>0; [67-64-1]

## ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

Anales fis. quim. (Madrid)  $\underline{1945}$ , 41, 537-60.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

#### EXPERIMENTAL VALUES:

t/°C	G <sup>a</sup>	Ep	x <sub>g</sub> /1 <sup>c</sup>	mol/l <sup>d</sup> acetone	mmol/mol acetone	1:X <sup>e</sup> <sub>g</sub>	1 + x <sup>f</sup> <sub>cc</sub>
0	0.144	0.144	1.173	3.3	0.24	694.44	852.52
10	0.161	0.161	1.293	3.7	0.26	621.12	773.39
20	0.174	0.174	1.377	3.9	0.29	574.71	726.21
30	0.191	0.190	1.488	4.2	0.32	523.56	672.04
40	0.206	0.205	1.581	4.5	0.34	485.43	632.51
50	0.220	0.219	1.655	4.7	0.36	454.54	604.24

 $a_{G} = \frac{p + 100}{P - p}$ , where p and P are the weights of solute and solution, resp.

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

A special all-glass app was contructed enabling the prepn of satd solns, agitation by bubbling a stream of acetone-satd N, filtration, and distn off the solvent without contact with air. Two exchangeable dissoln vessels of 15 and 8 cm<sup>3</sup> working capacity were used depending on the soly of solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm<sup>3</sup>, 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 acetone.

## SOURCE AND PURITY OF MATERIALS:

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

The purity of Na disulfan was not specified.

## ESTIMATED ERROR:

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

Temp: ±0.1°C (authors).

 $<sup>^{</sup>b}E = \frac{G - 100}{G + 100}$ ;  $^{c}$  g/1 acetone;  $^{d}$  should be mmol/1 acetone (compiler);

e g of acetone required to dissolve 1 g of solute; f volume (cm<sup>3</sup>) of acetone required to dissolve 1 g of solute.

- (1) Benzenesulfonamide, 4-amino-N-[4-(aminosulfonyl)phenyl]-,monosodium salt, monohydrate (Na disulfan monohydrate); C<sub>12</sub>H<sub>12</sub>N<sub>3</sub>NaO<sub>4</sub>S<sub>2</sub>·H<sub>2</sub>O; [81815-35-2]
- (2) Water; H<sub>2</sub>0; [7732-18-5]

## ORIGINAL MEASUREMENTS:

Crossley, M. L.; Northey, E. H.; Hultquist, M. E.

J. Am. Chem. Soc. 1938, 60, 2222-4.

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

EXPERIMENTAL VALUES:

## Solubility

t/ <sup>o</sup> C		
2, 0	$g/100 \text{ cm}^3$	mol dm <sup>-3</sup> a
10	9.6	0.26
37	20.0	0.54

<sup>a</sup>Calculated by compiler

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Nothing specified.

## SOURCE AND PURITY OF MATERIALS:

The Na disulfan monohydrate was prepd by the authors and purified by recrystn from concd aq soln with use of activated charcoal. Analysis - calcd 6.28% Na, found 6.35% Na. Assay by nitrite 100.2% Purity of the water was not specified.

## ESTIMATED ERROR:

Nothing specified.

(1) Acetamide, [4-[[[4-(aminosulfonyl)phenyl]-amino]sulfonyl]phenyl]- (acetyl disulf-anilamide); C<sub>14</sub>H<sub>14</sub>N<sub>3</sub>O<sub>5</sub>S<sub>2</sub>; [56444-82-7]

(2) 2-Propanone (acetone); C<sub>3</sub>H<sub>6</sub>0; [67-64-1]

## ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

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

VARIABLES:

PREPARED BY:

R. Piekos

Temperature

EXPERIMEN	TAL VALUE	ES:					
t/°C	G <sup>a</sup>	Ep	x <sub>g</sub> /1 <sup>c</sup>	mol/l <sup>d</sup> acetone	mmol/mol acetone	1:X <sub>g</sub>	1 + X <sup>f</sup> <sub>cc</sub>
0	1.271	1.255	10.354	28.0	2.0	78.52	96.58
5	1.402	1.382	11.339	30.7	2.2	71.32	88.19
10	1.608	1.563	12.912	34.9	2.5	62.19	77.45
15	1.687	1.659	13.457	36.2	2.7	59.28	74.32
20	1.749	1.719	13.838	37.5	2.8	55.75	72.26
25	1.984	1.945	15.578	42.2	3.1	50.40	64.19
30	2.234	2.185	17.410	47.1	3.5	44.76	57.44
35	2.515	2.453	19.448	52.6	3.9	39.76	51.36
40	2.854	2.775	21.902	59.3	4.5	35.03	45.65
45	3.250	3.138	24.746	66.9	5.1	30.77	40.41
50	3.679	3.548	27.795	75.2	5.8	25.55	39.13

 $a_G = \frac{p}{P-p}$ ; where p and P are the weights of solute and solution, resp.

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

A special all-glass app was constructed enabling the prepn of satd solns, agitation by bubbling a stream of acetone-satd N, filtration, and distn off the solvent without contact with air. Two exchangeable dissoln vessels of 15 and 8 cm<sup>3</sup> working capacity were used depending on the soly of solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm<sup>3</sup>, 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 acetone.

## SOURCE AND PURITY OF MATERIALS:

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

The purity of the solute was not specified.

## ESTIMATED ERROR:

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

Temp: ±0.1°C (author).

 $b_E = \frac{G}{G + 100}$ ;  $c_g/1$  acetone;  $d_{should}$  be mmol/1 (compiler).

 $<sup>^{\</sup>rm e}$ g of acetone required to dissolve 1 g of solute;  $^{\rm f}$ volume (cm $^{\rm 3}$ ) of acetone required to dissolve 1 g of solute.

- (1) Benzenesulfonamide, 4-amino-N-[4-[(methylamino)sulfonyl]phenyl]- (Neouliron); C<sub>13</sub>H<sub>15</sub>N<sub>3</sub>O<sub>4</sub>S<sub>2</sub>; [547-53-5]
- (2) Phosphoric acid, monopotassium salt; KH<sub>2</sub>PO<sub>4</sub>; [7778-77-0]
- (3) Water; H<sub>2</sub>0; [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: 4.37

## PREPARED BY:

R. Piekos

## EXPERIMENTAL VALUES:

Solubility of Neo-uliron in a 0.735M (10%)  $\rm KH_2PO_4$  solution of pH 4.37, at room temperature (about  $20^{\circ}\rm C$ ), is 0.0157 g% (4.60 x  $10^{-4}$  mol dm<sup>-3</sup> solution, compiler).

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Neo-uliron (0.5 g) was dissolved in 10 cm<sup>3</sup> of the 0.735N (10%) KH<sub>2</sub>PO<sub>4</sub> soln, shaken for 2 h, and filtered. A 1-cm<sup>3</sup> aliquot of the filtrate was then withdrawn, cooled, acidified with 2N HCl, and the sulfonamide content was detd colorimetrically by the method of Marshall modified by Kimmig (1) using an Authenrieth colorimeter. The pH was measured on an ultraionograph using a glass electrode.

## SOURCE AND PURITY OF MATERIALS:

Neo-uliron was the product manufd by "Bayer". The source and purity of the remaining materials were not specified.

#### ESTIMATED ERROR:

Soly: precision ±5% (author).

Temp: not specified.

pH : ±0.05 pH unit (author).

## REFERENCES:

Kimmig, J. Arch. Dermatol. <u>1938</u>,
 722; Erg. Hyg. <u>1941</u>, 24, 398.

- (1) Benzenesulfonamide, 4-amino-[4-[(methylamino)sulfonyl]phenyl]- (Neouliron);  $C_{13}H_{15}N_3O_4S_2$ ; [547-53-5]
- (2) Phosphoric acid, disodium salt; Na<sub>2</sub>HPO<sub>4</sub>; [7558-94-4]
- H<sub>2</sub>O; [7732-18-5] (3) Water;

## ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

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

VARIABLES:

#### PREPARED BY:

R. Piekos

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

## EXPERIMENTAL VALUES:

Solubility of Neo-uliron in a 0.705M (10%)  $Na_2HPO_4$  solution of pH 8.74, at room temperature (about  $20^{\circ}$ C) is 0.130 g% (  $3.81 \times 10^{-3}$  mol dm<sup>-3</sup> solution, compiler ).

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Neo-uliron (0.5 g) was dissolved in  $10 cm^3$ of the 0.705M (10%) Na<sub>2</sub>HPO<sub>4</sub> soln, shaken for 2 h, and filtered. A 1-cm<sup>3</sup> aliquot of the filtrate was then withdrawn, cooled, acidified with 2N HCl, and the sulfonamide content was detd colorimetrically by the method of Marshall modified by Kimmig (1) using an Authenrieth colorimeter. The pH was measured on an ultraionograph using a glass elec- ESTIMATED ERROR: trode.

## SOURCE AND PURITY OF MATERIALS:

Neo-uliron was the product manufd by "Bayer". The source and purity of the remaining materials were not specified.

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-N-[4-[(methylamino)sulfonyl]phenyl]- (Neouliron);  $C_{13}H_{15}N_3O_4S_2$ ; [547-53-5]
- (2) Phosphoric acid, disodium salt; Na<sub>2</sub>HPO<sub>4</sub>; [7558-94-4]
- (3) Phosphoric acid, monopotassium salt;

[7778-77-0] H<sub>2</sub>0; [7732-18-5] KH<sub>2</sub>PO<sub>4</sub>; Water:

PREPARED BY:

90-116.

ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

R. Piekos

Arch. Dermatol. Syphilis 1942, 183,

## VARIABLES:

Temperature, pH

EXPERIMENTAL VALUES:

Composition	on of 1/15M	phosphate			Solubility					
bu:	ffer soluti	ons	pН	Room	temp (ca 20°C)		37°C			
Na <sub>2</sub> HPO <sub>4</sub>	KH <sub>2</sub> PO <sub>4</sub>	%Content		g%	10 <sup>4</sup> mol dm <sup>-3</sup> solution <sup>a</sup>	g%	10 <sup>4</sup> mol dm <sup>-3</sup> solution <sup>a</sup>			
1.0	99.0	0.91	4.944	0.028	8.20	-	-			
10.0	90.0	0.91	5.906	0.025	7.32	0.046	13.47			
61.1	38.9	0.93	7.005	0.027	7.91	0.058	16.99			
9.5	0.5	0.733 <sup>b</sup>	7.51	0.032	9.37	-	-			
94.7	5.3	0.95	8.018	0.078	22.85	-	-			

<sup>&</sup>lt;sup>a</sup>Calculated by compiler

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Neo-uliron (0.5 g ) was dissolved in  $10 \text{ cm}^3$ of a buffer soln, shaken for 2 h at 20°C (or left for 48 h at  $37^{\circ}\text{C}$ ), 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 cm<sup>3</sup> of 2N HCl and the sulfonamide content was detd colorimetrically by the method of Marshall modified by Kimmig (1) using an Authenrieth colorimeter. The pH was detd on an ultraionograph using a glass electrode.

## SOURCE AND PURITY OF MATERIALS:

Neo-uliron was the product manufd by "Bayer". The source and purity of the remaining materials were 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.

bMolar content; 10% buffer solution

(1) Benzenesulfonamide, 4-amino-N-[4-[(methylamino)sulfony1]pheny1]-; C<sub>13</sub>H<sub>15</sub>N<sub>3</sub>O<sub>4</sub>S<sub>2</sub>; [547-53-5]

(2) 2-Propanone (acetone); C<sub>3</sub>H<sub>6</sub>0; [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/ <sup>o</sup> C	G <sup>a</sup>	Ep	x <sub>g</sub> /1 <sup>c</sup>	mol/1 <sup>d</sup> acetone	mmol/mol acetone	1:X <sub>g</sub>	1 + X <sup>f</sup> <sub>cc</sub>
0	17.565	14.940	143,084	419.1	29.80	5.79	6.98
5	18.507	15.617	149,685	438.4	31.41	5.40	6.67
10	19.309	16.185	154.530	452.6	32.84	5.17	6.47
15	20.062	16.709	159.914	468.4	34.12	4.98	6.25
20	22.107	18.104	174.911	512.3	37.60	4.52	5.72
25	23.202	18.881	182.182	533.6	39.46	4.32	5.49
30	25.474	20.302	198.507	580.5	43.33	3.92	5.04
35	28.002	21.876	216.539	634.3	47.63	3.55	4.52
40	30.735	23.509	235.887	690.9	52.28	3.25	4.24
45	37.109	27.065	282.631	827.8	63.12	2.69	3.54
50	47.626	32.261	359.814	1053.9	82.71	2.10	2.78

 $<sup>{}^{</sup>a}G = \frac{p \cdot 100}{P - p}$ , where p and P are the weights of solute and solution, resp.

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

A special all-glass app was contructed enabling the prepn of satd solns, agitation by bubbling a stream of acetone-satd N, fitration, and distn off the solvent without contact with air. Two exhangeable dissoln vessels of 15 and 8 cm<sup>3</sup> working capacity were used depending on the soly of solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm<sup>3</sup>, 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 acetone.

## SOURCE AND PURITY OF MATERIALS:

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

The purity of the solute was not specified.

## ESTIMATED ERROR:

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

Temp: ±0.1°C (author).

 $b_E = \frac{G\ 100}{G+100}$ ;  $c_{g/1}$  acetone;  $d_{should}$  be mmol/1 (compiler).

<sup>&</sup>lt;sup>e</sup>g of acetone required to dissolve 1 g of solute; <sup>f</sup>volume (cm<sup>3</sup>) of acetone required to dissolve 1 g of solute.

- (1) Acetamide, N-[4-[[[4-[(methylamino)-sulfony1]pheny1]animo]sulfony1]pheny1]-(acety1 Neo-uliron); C<sub>15</sub>H<sub>17</sub>N<sub>3</sub>O<sub>5</sub>S<sub>2</sub>; [71119-14-7]
- (2) Phosphoric acid, disodium salt; Na<sub>2</sub>HPO<sub>4</sub>; [7558-94-4]
- (3) Water; H<sub>2</sub>0; ]7732-18-5]

## ORIGINAL MEASUREMENTS:

Kriger-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 acetyl Neo-uliron in a 0.705 M (10%)  $Na_2HPO_4$  solution of pH 8.74 at room temperature (about  $20^{\circ}C$ ) is 0.021 g% (5.5 x  $10^{-4}$  mol dm<sup>-3</sup> solution, compiler).

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Acetyl Neo-uliron (0.5 g) was dissolved in 10 cm<sup>3</sup> of the 0.705 M (10%) Na<sub>2</sub>HPO<sub>4</sub> soln of pH 8.74, shaken for 2 h at room temp (about 20°C), and filtered. The filtrate was treated with equal vol of 2N HCl, and refluxed for 15 min. After proper diln, a 1-cm<sup>3</sup> aliquot was withdrawn, acidified, cooled, and the sulfonamide content was detd colorimetrically (as Neo-uliron) by the Marshall method modified by Kimmig (1) using an Authenrieth colorimeter. The pH was detd on an ultraionograph using a glass electrode.

## SOURCE AND PURITY OF MATERIALS:

Acetyl Neo-uliron (source not specified) gave no coloration upon diazotization of its satd soln, thus showing absence of Neo-uliron.

The source and purity of the remaining materials were not specified.

## ESTIMATED ERROR:

Soly: precision ±5% (author)

Temp: not specified

pH : ±0.05 pH unit (author)

## REFERENCES:

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

- (1) Acetamide, N-[4-[[[4-[(methylamino)sulfonyl]phenyl]amino]sulfonyl]phenyl]-(acetyl Neo-uliron); C<sub>15</sub>H<sub>17</sub>N<sub>3</sub>O<sub>5</sub>S<sub>2</sub>; [71119-14-7]
- (2) Phosphoric acid, monopotassium salt; KH<sub>2</sub>PO<sub>4</sub>; [7778-77-0]
- (3) Water; H<sub>2</sub>0; [7732-18-5]

# ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

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

## VARIABLES:

One temperature: ca 20°; one pH: 4.37

## PREPARED BY:

R. Piekos

## EXPERIMENTAL VALUES:

Solubility of acetyl Neo-uliron in a 0.735M (10%)  $\mathrm{KH_2PO_4}$  solution of pH 4.37 at room temperature (about  $20^{\circ}$ C) is 0.0020 g% ( 5.2 x  $10^{-5}$  mol dm<sup>-3</sup> solution, compiler ).

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Acetyl Neo-uliron (0.5 g) was dissolved in 10 cm<sup>3</sup> of the 0.735M (10%)  $KH_2PO_A$  soln of pH 4.37, shaken for 2 h at room temp (about 20°C), and filtered. The filtrate was treated with equal vol of 2N HCl, and refluxed for 15 min. After proper diln, a 1-cm3 aliquot was withdrawn, acidified, cooled, and the sulfonamide content was detd colorimetrically (as Neo-uliron) by the Marshall method modified by Kimmig (1) using an Authenrieth colorimeter. The pH was detd on an ultraionograph using a glass electrode.

## SOURCE AND PURITY OF MATERIALS:

Acetyl Neo-uliron (source not specified) gave no coloration upon diazotization of its sat soln, thus showing absence of Neo-uliron.

The source and purity of the remaining materials were 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) Acetamide, N-[4-[[4-[(methylamino)sulfony1[pheny1]amino]sulfony1]pheny1]-(acetyl Neo-uliron); C<sub>15</sub>H<sub>17</sub>N<sub>3</sub>O<sub>5</sub>S<sub>2</sub>; [71119-14-7]
- (2) Phosphoric acid, disodium salt; Na<sub>2</sub>HPO<sub>4</sub>; [7558-94-4]
- Phosphoric acid, monopotassium salt; КH<sub>2</sub>PO<sub>4</sub>; [7778-77-0]
- Water; H<sub>2</sub>0; [7732-18-5]

VARIABLES:

94.7

Temperature, pH

#### ORIGINAL MEASUREMENTS:

Krüger-Thiemer, E.

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

#### PREPARED BY:

R. Piekos

## EXPERIMENTAL VALUES:

Composition of 1/15M phosphate				Solubility				
buffer solutions				Room t	emp (ca 20°C)	3	37°C	
Na <sub>2</sub> HPO <sub>4</sub>	кн <sub>2</sub> РО <sub>4</sub>	%Content	рН	g%	10 <sup>4</sup> mol dm <sup>-3</sup> solution	g%	10 <sup>4</sup> mol dm <sup>-3</sup> solution	
1.0	99.0	0.91	4.944	0.0019	0.495	_	-	
10.0	90.0	0.91	5.906	0.0022	0.573	0.0024	0.625	
61.1	38.9	0.93	7.005	0.0038	0.991	0.0051	1.33	
9.5	0.5	0.733 <sup>b</sup>	7.51	0.0040	1.043	-	-	

0.0128

8.018

5.3

0.95

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

Acetyl Neo-uliron (0.5 g) was dissolved in 10 cm<sup>3</sup> of a buffer soln, shaken for 2 h at 20°C (or left for 48 h at 37°C), filtered at respective temp. The filtrate was treated with equal vol of 2N HCl and refluxed for 15 min. After proper diln, a 1 cm<sup>3</sup> aliquot was withdrawn, cooled, and the sulfonamide content was detd colorimetrically (as Neo-uliron) by the Marshall method modi- ESTIMATED ERROR: fied by Kimmig (1) using Authenrieth colorimeter. the pH was detd on an ultraionograph using a glass electrode.

## SOURCE AND PURITY OF MATERIALS:

3.338

Acetyl Neo-uliron (source not specified) gave no coloration upon diazotization of its satd soln, thus showing absence of Neo-uliron.

The source and purity of the remaining materials were not specified.

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.

aCalculated by compiler.

bMolar content; 10% buffer solution.

- (1) Benzenesulfonamide, 4-amino-N-[4-[(dimethylamino)sulfonyl]phenyl]-;  $c_{14}H_{17}N_3O_4S_2$ ; [515-67-3]
- (2) 2-Propanone (acetone); C<sub>3</sub>H<sub>6</sub>O; [67-64-1]

## ORIGINAL MEASUREMENTS:

Gutierrez, F. H.

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

VARIABLES:

Temperature

PREPARED BY:

R. Piekos

xPERIN t/°C	ÆNTAL VA G <sup>a</sup>	LUES: Eb	X <sub>g</sub> /1 <sup>c</sup>	mol/1 <sup>d</sup> acetone	mmo1/mo1 acetone	1:X <sub>g</sub>	1 + X <sup>f</sup> <sub>cc</sub>
0	26.007	20.639	211.853	509	42.5	3.84	4.72
5	27.025	21.275	218.575	615	44.1	3.70	4.57
10	28.036	21.897	225.129	633	45.8	3.56	4.44
15	29.064	22.519	231.669	654	47.5	3.44	4.31
20	30.092	23.130	238.072	672	49.0	3.32	4.20
25	31.120	23.733	244.354	689	50.9	3.21	4.09
30	31.898	24.214	248.581	701	52.1	3.13	4.92
35	33.500	25.093	259.055	731	54.7	2.98	3.86
40	35.705	26.311	274.000	745	58.3	2.80	3.65
45	39.169	28.144	298.242	841	64.0	2.55	3.35
50	44.509	30.800	336.265	949	72.7	2.25	2.98

 $<sup>{}^{</sup>a}G = \frac{p}{P} \frac{100}{P}$ , where p and P are the weights of solute and solution, resp.

## AUXILIARY INFORMATION

## METHOD/APPARATUS/PROCEDURE:

A special all-glass app was contructed enabling the prepn of satd solns, agitation by bubbling a stream of acetone-satd N, filtration, distn off the solvent without contact with air. Two exchangeable dissoln vessels of 15 and 8 cm<sup>3</sup> working capacity were used depending on the soly of solute. The app was immersed in a thermostat. The vols of acetone used were 15 or 5 cm<sup>3</sup>, and the equi-ESTIMATED ERROR: libration 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 acetone.

## SOURCE AND PURITY OF MATERIALS:

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

The purity of the solute was not specified.

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

Temp: ±0.1°C (author).

 $<sup>^{</sup>b}E = \frac{G - 100}{G + 100}$ ;  $^{c}g/1$  acetone;  $^{d}$ should be mmol/1 (compiler);

 $<sup>^{\</sup>rm e}$ g of acetone required to dissolve 1 g of solute;  $^{\rm f}$ volume (cm $^{\rm 3}$ ) of acetone required to dissolve 1 g of solute.