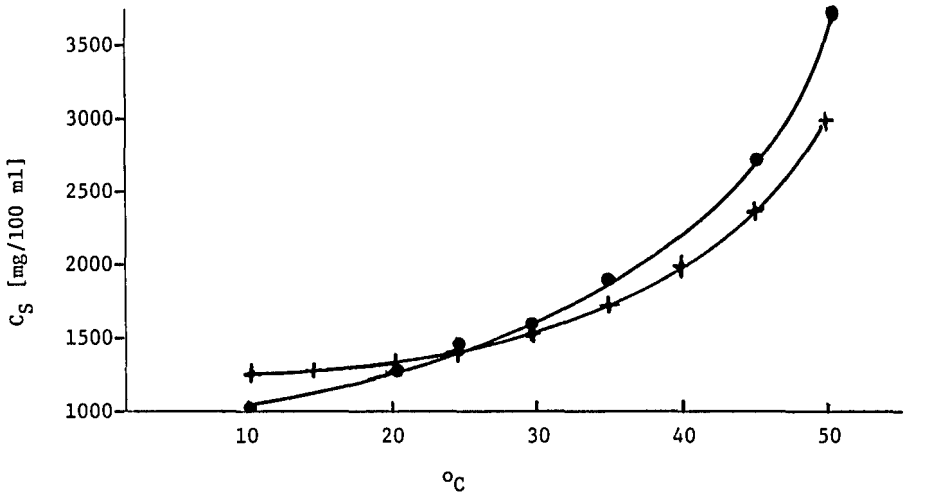
| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzenesulfonamide, 4-amino-N-(amino-thioxomethyl)- , (Badional); $C_7H_9N_3O_2S_2$; [515-49-1]<br>(2) Ethanol; $C_2H_6O$; [64-17-5]<br>(3) Water; $H_2O$; [7732-18-5] | Kuhnert-Brandstätter, M.; Burger, A., *Pharm. Ind.* 1972, *34*, 353-6. |
| VARIABLES:<br>Temperature | PREPARED BY:<br>R. Piekos |

EXPERIMENTAL VALUES:

Saturation solubility, $c_s$, of forms I and II of Badional in 96% ethanol.

Form I (+ + +), form II (● ● ●)




AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of Badional was added to 96% ethanol and stirred until no changes in concn were obsd (2 - 48 h). Aliquots were withdrawn by means of a pipet equipped with a filter, which was preheated to a desired temp, and Badional was assayed spectrophotometrically at 269 nm. The identity of the solid phase was detd by thermomicroscopy and IR spectroscopy.

OURCE AND PURITY OF MATERIALS:

Both forms were obtained by crystn of a compd prep from 96% EtOH;: form I by cooling a hot satd soln to room temp and seeding with crystals of this form, form II by cooling the soln to ∿40°C and scratching the walls.

Purity of the 96% EtOH was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.1°C (authors).

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzenesulfonamide, 4-amino-N-(amino-thioxomethyl)-, (Badional); $C_7H_9N_3O_2S_2$; [515-49-1]<br>(2) Ethanol; $C_2H_6O$; [64-17-5]<br>(3) Water; $H_2O$; [7732-18-5] | Kuhnert-Brandstätter, M.; Burger, A., *Pharm. Ind.* 1972, *34*, 353-6. |
| VARIABLES:<br>Temperature | PREPARED BY:<br>R. Piekos |

EXPERIMENTAL VALUES:

| t/°C | Saturation solubility in 96% ethanol of crystalline form I | | Saturation solubility in 96% ethanol of crystalline form II | |
|---|---|---|---|---|
| | g/100 ml | mol $dm^{-3}$ [a] | g/100 ml | mol $dm^{-3}$ [a] |
| 10.0 | | | 1.00 | 0.0432 |
| 10.1 | 1.06 | 0.0458 | | |
| 14.6 | | | 1.12 | 0.0484 |
| 15.2 | 1.18 | 0.0510 | | |
| 20.0 | 1.37 | 0.0592 | 1.29 | 0.0558 |
| 25.2 | | | 1.54 | 0.0666 |
| 25.4 | 1.57 | 0.0679 | | |
| 30.9 | | | 1.81 | 0.0783 |
| 31.0 | 1.79 | 0.0774 | | |
| 37.0 | | | 2.21 | 0.0955 |
| 37.1 | 2.05 | 0.0886 | | |
| 41.2 | 2.30 | 0.0994 | 2.52 | 0.109 |
| 46.0 | 2.68 | 0.116 | | |
| 46.2 | | | 3.00 | 0.130 |
| 50.8 | 3.06 | 0.132 | | |
| 51.0 | | | 3.50 | 0.151 |

[a] Calculated by compiler.

AUXILIARY INFORMATION

METHOD/APPARATUS/PROCEDURE:

An excess of Badional was added to 96% ethanol and stirred until no changes in concn were obsd (2-48 h). Aliquots were withdrawn by means of a pipet equipped with a filter, which was preheated to a desired temp, and Badional was assayed spectrophotometrically at 269 nm. The identity of the solid phase was detd by thermomicroscopy and IR spectroscopy.

SOURCE AND PURITY OF MATERIALS:

Both forms of Badional were obtained by crystn of a comprep from 96% EtOH: form I, mp 179-181°C, by cooling a hot satd soln to room temp and seeding with crystals of this form; form II, mp 171°C, by cooling the soln to 40°C and scratching the walls. Purity of the 96% ethanol was not specified.

ESTIMATED ERROR:

Soly: not specified.

Temp: ±0.1°C (authors).

REFERENCES:

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzenesulfonamide; 4-amino-N-(amino-thioxomethyl)- (sulfathiourea); $C_7H_9N_3O_2S_2$; [515-49-1]<br>(2) Ethanol; $C_2H_6O$; [64-17-5]<br>(3) 1,2,3-Propanetriol; $C_3H_8O_3$; [56-81-5]<br>(4) Water; $H_2O$; [7732-18-5] | Dolique, R.; Foucault, J.<br>*Trav. soc. pharm. Montpellier* 1952, *12*, 145-53. |
| VARIABLES:<br>One temperature: 26-28°C | PREPARED BY:<br>R. Piekos |

EXPERIMENTAL VALUES:

Solubility of sulfathiourea in a mixture of 1,2,3-propanetriol and 95° ethanol (2:1 by wt) at 26-28°C is 7.72% (0.362 mol $kg^{-1}$ solvent, compiler).

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The sulfathiourea content was detd by diazotization of the amine group in a cold acidified 0.1N $KNO_2$ soln. An excess of $KNO_2$ was detected by using iodinated starch. | Nothing specified. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzenesulfonamide, 4-amino-N-(amino-thioxomethyl)- (sulfathiourea); $C_7H_9N_3O_2S_2$; [515-49-1]<br>(2) Ethanol; $C_2H_6O$; [64-17-5]<br>(3) 1,2,3-Propanetriol; $C_3H_8O_3$; [56-81-5]<br>(4) Urea; $CH_4N_2O$; [57-13-6]<br>(5) Water; $H_2O$; [7732-18-5] | Dolique, R.; Foucault, J.<br>*Trav. soc. pharm. Montpellier* 1952, *12*, 145-53. |
| VARIABLES:<br>One temperature: 26-28°C | PREPARED BY:<br>R. Piekos |

EXPERIMENTAL VALUES:

Solubility of sulfathiourea at 26-28°C in a saturated solution of urea in a mixture of 1,2,3-propanetriol and 95° ethanol (2:1 by wt), containing 54.5 g of urea per 100 g of the mixture, is 9.47% (0.452 mol $kg^{-1}$ solvent, compiler).

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| The sulfathiourea content was detd by diazotization of the amine group in a cold acidified 0.1N $KNO_2$ soln. An excess of $KNO_2$ was detected by using iodinated starch. | Nothing specified. |
| | ESTIMATED ERROR:<br>Nothing specified. |
| | REFERENCES: |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzenesulfonamide, 4-amino-N-[imino-(methylthio)methyl]- (sulfamethylisothiourea); $C_8H_{11}N_3O_2S$; [2651-18-5] | Becher, R.; Leya, S. *Experientia* 1946, *2* 459-60. |
| (2) Water; $H_2O$; [7732-18-5] | |
| VARIABLES: | PREPARED BY: |
| One temperature: 18-19°C. | R. Piekos |

EXPERIMENTAL VALUES:

Solubility of sulfamethylisothiourea in water at room temperature (18-19°C) is 19 mg% ($8.9 \times 10^{-4}$ mol $dm^{-3}$, compiler).

AUXILIARY INFORMATION

| METHOD/APPARATUS/PROCEDURE: | SOURCE AND PURITY OF MATERIALS: |
|---|---|
| After standing for more than two days the soln of sulfamethylisothiourea 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: 1. Druey, J.; Oesterheld, G. *Helv. Chim. Acta* 1942, *25*, 753. |

| COMPONENTS: | ORIGINAL MEASUREMENTS: |
|---|---|
| (1) Benzenesulfonamide, 4-amino-N-[imino-(methylthio)methyl]- (sulfamethylisothiourea); $C_8H_{11}N_3O_2S$; [2651-18-5]<br>(2) Sodium chloride; NaCl; [7647-14-5]<br>(3) Water; $H_2O$; [7732-18-5] | Becher, R.; Leya, S. *Experientia* 1946, *2*, 459-60. |
| **VARIABLES:**<br>One temperature: 18-19°C | **PREPARED BY:**<br>R. Piekos |

**EXPERIMENTAL VALUES:**

Solubility of sulfamethylisothiourea in a 5% NaCl solution at room temperature (18-19°C) is 21 mg% ($9.9 \times 10^{-4}$ mol $dm^{-3}$, compiler).

AUXILIARY INFORMATION

**METHOD/APPARATUS/PROCEDURE:**

After standing for more than two days the soln of sulfamethylisothiourea 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.