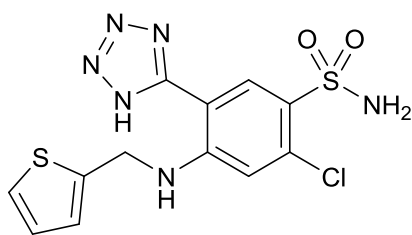


1 **Azosemide**

2 アゾセמיד



3

4 $C_{12}H_{11}ClN_6O_2S_2$: 370.845 2-Chloro-5-(1*H*-tetrazol-5-yl)-4-[(thien-2-ylmethyl)amino]

6 benzenesulfonamide

7 [27589-33-9]

8

9 Azosemide, when dried, contains not less than 99.0% and
10 not more than 101.0% of azosemide ($C_{12}H_{11}ClN_6O_2S_2$).

11 **Description** Azosemide occurs as a white to yellow-white crys-
12 talline powder.

13 It is freely soluble in *N,N*-dimethylformamide, slightly soluble
14 in methanol and in ethanol (99.5), and practically insoluble in wa-
15 ter.

16 It dissolves in dilute sodium hydroxide TS.

17 It is gradually colored to yellow by light.

18 Melting point: about 226°C (with decomposition).

19 **Identification** (1) Determine the absorption spectrum of a so-
20 lution of Azosemide in dilute sodium hydroxide TS (3 in 500,000)
21 as directed under Ultraviolet-visible Spectrophotometry <2.24>,
22 and compare the spectrum with the Reference Spectrum: both
23 spectra exhibit similar intensities of absorption at the same wave-
24 lengths.

25 (2) Determine the infrared absorption spectrum of Azosemide,
26 previously dried, as directed in the potassium bromide disk
27 method under Infrared Spectrophotometry <2.25>, and compare
28 the spectrum with the Reference Spectrum: both spectra exhibit
29 similar intensities of absorption at the same wave numbers.

30 **Purity** (1) Chloride <1.03>—To 1.0 g of Azosemide add 60
31 mL of dilute sodium hydroxide TS, dissolve by warming. After
32 cooling, add 0.5 mL of nitric acid and filter. To 30 mL of the fil-
33 trate add 6 mL of dilute nitric acid and water to make 50 mL. Per-
34 form the test using this solution as the test solution. Prepare the
35 control solution with 0.45 mL of 0.01 mol/L hydrochloric acid VS
36 (not more than 0.032%).

37 (2) Heavy metal <1.07>—Proceed with 1.0 g of Azosemide
38 according to Method 2, and perform the test. Prepare the control
39 solution with 2.0 mL of Standard Lead Solution (not more than 20
40 ppm).

41 (3) Primary aromatic amines—Dissolve 20 mg of Azosemide
42 in 5 mL of *N,N*-dimethylformamide, add 12 mL of water, 1.0 mL
43 of a solution of sodium nitrite (1 in 200) and 2.0 mL of diluted

44 hydrochloric acid (1 in 10) under ice-cooling, shake, and allow to
45 stand for 3 minutes. Add 1.0 mL of ammonium amidosulfate TS,
46 shake thoroughly, allow to stand for 3 minutes, and add 1.0 mL of
47 a solution of *N*-1-naphthylethylenediamine dihydrochloride (1 in
48 200). Shake this solution, and add *N,N*-dimethylformamide to
49 make exactly 50 mL. Determine the absorbance of this solution at
50 540 nm as directed under Ultraviolet-visible Spectrophotometry
51 <2.24>, using a solution prepared in the same manner with 5 mL
52 of *N,N*-dimethylformamide, as the blank: the absorbance is not
53 more than 0.15.

54 **Loss on drying** <2.41> Not more than 0.5% (1 g, 105°C, 3 hours).

55 **Residue on ignition** <2.44> Not more than 0.1% (1 g).

56 **Assay** Weigh accurately about 0.6 g of Azosemide, previously
57 dried, dissolve in 50 mL of *N,N*-dimethylformamide, and titrate
58 <2.50> with 0.1 mol/L potassium hydroxide-ethanol VS until the
59 color of the solution changes from yellow to yellow-green (indi-
60 cator: 10 drops of thymol blue-dimethylformamide TS). Perform
61 a blank determination with a solution prepared by adding 15 mL
62 of ethanol (95) to 50 mL of *N,N*-dimethylformamide, and make
63 any necessary correction.

64 Each mL of 0.1 mol/L potassium hydroxide-ethanol VS
65 = 37.08 mg of $C_{12}H_{11}ClN_6O_2S_2$

66 **Containers and storage** Containers—Tight containers.

67 Storage—Light-resistant.