National Institute of Standards & Technology

Certificate

Standard Reference Material 189a

Potassium Tetroxalate

pH Standard

This Standard Reference Material (SRM) is intended for use as a secondary pH standard in preparing solutions for calibrating pH measuring systems. SRM 189a, Potassium Tetroxalate Dihydrate ($KH_3(C_2O_4)_2.2H_2O$), was prepared to ensure high purity and uniformity and to assay close to 100 %. However, this SRM is certified only with respect to pH(S) values, not as a pure substance.

The certified pH(S) values listed below were derived from emf measurements of cells without liquid junction using hydrogen gas and AgCl/Ag electrodes (where the hydrogen gas was at 1 atmosphere) by the method described in reference [1]. The pH(S) value corresponds to log ($1/a_H$) where a_H is the conventional activity of the hydrogen ion referred to the standard state on the molal scale. The uncertainty of the pH(S) is estimated not to exceed ± 0.005 unit for the temperature range 5 to 50 °C.

The liquid-junction potential of the common pH cell displays a considerably greater variability in solutions of pH less than 2.5 than in solutions of pH between 2.5 and 11.5. For this reason, solutions of potassium tetroxalate are not recommended as primary standards of pH. They are useful, however, as secondary standards and for confirmatory purposes, with the expectation that the experimental pH may differ by 0.02 to 0.05 units due to liquid junction potential from the values of pH(S) given below. They are also recommended when standards of pH are needed and a liquid junction is not involved.

The 0.05-molal solution is recommended for the standardization of pH measuring systems. The certified pH(S) values of this solution as a function of temperature are given below. These values apply only to this SRM lot.

°C	pH(S)	°C	pH(S)
5	1.669	30	1.686
10	1.671	35	1.692
15	1.673	40	1.697
20	1.677	45	1.706
25	1.681	50	1.714

The values of pH(S) of SRM 189a are not certified above 50 °C. Refer to reference [2] for more details on pH measurements above 50 °C. The estimated uncertainty of values for the temperature range 55-95 °C is ± 0.01.

The potassium tetroxalate was obtained from Fluka Chemical Corp. It meets the specifications of the American Chemical Society for reagent-grade material, but may not be entirely free of impurities such as traces of occluded water, free acid or alkali, chlorides, sulfur compounds, or heavy metals.

Stability

SRM 189a is stable when stored in its original container, with the cap tightly closed under normal laboratory conditions of temperature and humidity.

Gaithersburg, MD 20899
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(Revision of certificate dated 4-21-86)

William P. Reed, Acting Chief
Standard Reference Materials Program

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The certification measurements were performed by Yung-Chi Wu and W.F. Koch of the Inorganic Analytical Research Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.R. DeVoe, Chief, Inorganic Analytical Research Division.

The technical and support aspects involved in the revision, update, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J.C. Colbert. The original coordination of certification efforts was performed by R.W. Seward.

Directions for Use

Preparation of the 0.05-molal solution:

Add 12.70 g of the SRM to 1000.0 g of distilled water and mix thoroughly. The SRM should not be dried. The distilled water should have an electrolytic conductivity not greater than 2 microsiemens/cm. If volumetric apparatus is to be used, transfer 12.61 g of the SRM to a 1-liter volumetric flask. Add distilled water to dissolve the salt, fill to the mark with distilled water at 25 °C, and mix thoroughly by shaking. (The values given are weights in air.)

The water used in the preparation of this pH buffer solution need not be protected from atmospheric carbon dioxide, and elaborate precautions for the exclusion of air from the solution are not necessary. The solution should, however, be protected against evaporation and contamination by molds. This buffer solution should be replaced, at least every month or whenever mold is detected, if the highest accuracy is required.

References

[1] Wu, Y.C., Koch, W.F. and Marinenko, G., J. Res. Nat'l Bur. Stand. 89 395 (1984).

[2] Wu, Y.C., Koch, W.F. and Durst, R.A., Standard Reference Materials: Standardization of pH Measurements, NBS Spec. Publ. 260-53 (February 1988).