

Certificate of Analysis

Standard Reference Materials

Potassium Dihydrogen Phosphate (2186—I)

Disodium Hydrogen Phosphate (2186—II)

Purity

These lots of potassium dihydrogen phosphate (KH_2PO_4) and disodium hydrogen phosphate (Na_2HPO_4) were prepared to ensure high purity and uniformity. They meet the specifications of the American Chemical Society for reagent-grade materials but should not be considered as entirely free from impurities such as traces of water, free acid or alkali, carbon dioxide, chlorides, sulfur compounds and heavy metals.

 $pD(S)$ Values

The $pD(S)$ values listed below correspond to $\log(1/a_D)$, where a_D is the *conventional* activity of the deuterium ion referred to the standard state on the scale of molality. The values were derived from the emf of cells without liquid junction by a method of calculation analogous to that described for the assignment of $pH(S)$ values [Journal of Research of the National Bureau of Standards, 66A, 179 (1962)]. The uncertainty of the assigned values for $pD(S)$ is estimated not to exceed 0.01 unit. The values listed below apply only to the lots here certified. Minor variations of $pD(S)$ (of the order of a few thousandths of a unit) may be expected to occur between different lots.

The solution 0.025 molal with respect to both KD_2PO_4 and Na_2DPO_4 is recommended for the calibration of pH meters to be used for the measurement of pD in deuterium oxide. These compounds are prepared *in situ* by hydrogen-deuterium exchange between the protium salts, KH_2PO_4 and Na_2HPO_4 and the deuterium oxide solvent. The $pD(S)$ of this solution as a function of temperature is given below:

| $t, ^\circ\text{C}$ | $pD(S)$ | $t, ^\circ\text{C}$ | $pD(S)$ | $t, ^\circ\text{C}$ | $pD(S)$ |
|---------------------|---------|---------------------|---------|---------------------|---------|
| 5 | 7.539 | 25 | 7.428 | 40 | 7.387 |
| 10 | 7.504 | 30 | 7.411 | 45 | 7.381 |
| 15 | 7.475 | 35 | 7.397 | 50 | 7.377 |
| 20 | 7.449 | | | | |

DIRECTIONS FOR USE

The preparation of the 0.025 molal solution should be carried out by the addition of weighed quantities of the salts to weighed quantities of deuterium oxide in the following proportions (weights in vacuo): 0.003402g KH_2PO_4 and 0.003549g Na_2HPO_4 per g of deuterium oxide. The deuterium oxide should have an isotopic composition of at least 99.5 mole percent D_2O . It should not contain dissolved carbon dioxide or other gases and should have a conductivity no greater than $2 \times 10^{-6} \Omega^{-1} \text{cm}^{-1}$. The salts should be dried for 2 hr at 100 to 130 $^\circ\text{C}$ before use. Although elaborate precautions to prevent contamination of the buffer solution with atmospheric carbon dioxide are usually unnecessary, the container should be kept tightly stoppered at all times when a sample is not actually being removed.

The development of the pD scale and the experimental work leading to the certification of these materials were performed by Maya Paabo and Roger G. Bates.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of Roger G. Bates.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.