



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3072

Diquat Dibromide Monohydrate in Water

Standard Reference Material (SRM) 3072 is a solution of diquat dibromide monohydrate (Chemical Abstracts Registry Number 85-00-07) in water intended primarily for use in the calibration of chromatographic instrumentation used for the determination of diquat dibromide monohydrate. This SRM can also be used to fortify aqueous samples with known amounts of diquat dibromide monohydrate. A unit of SRM 3072 consists of five 2 mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Diquat Dibromide: The certified concentration value [1,2], given below, is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using liquid chromatography (LC). A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Diquat Dibromide Monohydrate 39.7 mg/kg \pm 0.8 mg/kg

The results are expressed as the certified value \pm an expanded uncertainty. The certified value is the unweighted average of the concentration determined by gravimetric and chromatographic methods. The expanded uncertainty, at a 95 % level of confidence, is calculated as $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO Guide [3] and $k = 2$ is the coverage factor. The value of u_c includes both a correction for estimated purity and an allowance for differences between the concentrations determined by gravimetric preparation and chromatographic measurements. The concentration in mg/L is the same as the certified concentration based on the density of the solution at 22 °C (1.0 g/mL).

Expiration of Certification: The certification of this SRM lot is valid until **31 July 2011**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see *Instructions for Use*). However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Group by B.S. MacDonald.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 06 November 2002

John Rumble, Jr., Chief
Standard Reference Materials Program

Partial support for the preparation and certification of this SRM was provided by the U.S. Environmental Protection Agency Office of Water, Office of Enforcement and Compliance Assurance, and Office of Research and Development.

Coordination of the technical measurements leading to the certification was under the direction of L.C. Sander and M.M. Schantz of the NIST Analytical Chemistry Division.

Preparation of the SRM was performed by M.P. Cronise of the NIST Standard Reference Materials Group and L.C. Sander of the NIST Analytical Chemistry Division.

Analytical measurements were performed by and J. Brown Thomas and L.C. Sander of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

INSTRUCTIONS FOR USE

Handling: This material should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

Opening of Ampoule: Open ampoules carefully to prevent contamination and injury. The ampoules are pre-scored and should **NOT** be opened using a file. Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified value to be valid within the stated uncertainty. Certified values are not applicable to material stored in ampoules that have been opened for more than five minutes, even if they are resealed.

PREPARATION AND ANALYSIS¹

SRM Preparation: The diquat dibromide monohydrate used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the diquat dibromide monohydrate into the water. The weighed diquat dibromide monohydrate was added to the water and mixed until completely dissolved and homogenized. The total mass of this solution was measured and the concentration calculated from this gravimetric procedure. These gravimetric concentrations were adjusted for the consensus purity estimation of the diquat dibromide monohydrate, which was determined using LC with ultraviolet (UV) detection at 310 nm. Aliquots (1.2 mL) of the bulk solution were dispensed into 2 mL amber glass ampoules, which were then flame sealed.

SRM Analysis: Aliquots from nine ampoules, selected according to a modified, random number generator scheme, were analyzed in duplicate by using LC with UV detection at 310 nm employing an aminosilane column (4.6 mm x 250 mm, 5 µm particle diameter Zorbax[®] column; Mac-Mod Analytical, Inc., Chadds Ford, PA, USA). Isocratic elution (75 % acetonitrile: 15 % water: 10 % methanol adjusted to pH 3) at a flow rate of 1.5 mL/min was used for the determination of diquat dibromide monohydrate in the solution [4]. The concentration of diquat dibromide monohydrate in the SRM was determined by using the external standard method based on response factors determined from analyses of four calibration solutions of the compound.

¹Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assessment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000).
- [2] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, 1995 Ed. (1995).
- [3] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, 1993; see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office; Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.
- [4] Nagayama T.; Maki, T.; Iida, M.; Nishima, T.; *Reverse-Phase Liquid Chromatographic Determination of Paraquat and Diquat in Agricultural Products*; J. Assoc. Off. Anal. Chem., Vol. 70, pp. 1008-1010 (1987).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Group at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.