National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2695

Fluoride in Vegetation

(In Cooperation with the Aluminum Association, Inc.)

This Standard Reference Material (SRM) is intended for use as an analytical control material for the determination of fluoride in vegetation. SRM 2695 consists of two 25 g bottles of powdered timothy grass, one each at the low and high fluoride levels. Both levels are elevated above the fluoride concentrations representative of baseline in uncontaminated plant materials.

The certified values given below are based on the determination of the fluoride content of samples randomly selected from the lot by one NIST and six cooperating laboratories. NIST determinations were made by ion-selective electrode (ISE) measurement after oxygen bomb decomposition of the dried sample. Most cooperating laboratories employed a semi-automated method (AOAC and Intersociety methods) based on colorimetric alizarin measurement following fusion and microdistillation from sulfuric acid [1,2]. One laboratory determined fluoride by titration with thorium nitrate following fusion and distillation.

Material	Fluoride, $\mu g/g$
Low level	64.0 ± 8.4
High level	277 ± 27

The uncertainties of the certified values are the 95%, 95% tolerance intervals, which include both the material variability and the measurement error. The interval formed by the certified value minus and plus the uncertainty will cover the true concentration of fluoride in 95% of the samples with 95% confidence [3].

The overall coordination of the Aluminum Association, Inc. activities in identifying material and making measurements leading to certification was performed by J.H. Goldman.

The overall coordination of the NIST technical measurements leading to certification was performed by W. F. Koch of the Inorganic Analytical Research Division. The statistical consultation and analyses were provided by S.B. Schiller of the Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J. S. Kane.

Source and Preparation of Material: The material for this SRM, timothy grass at two fluoride concentration levels, was provided to NIST by Intalco Aluminum, Ferndale, WA. The material was cryogenically ground to -40 mesh and radiation sterilized. Preliminary testing showed considerable particle-size variation and inhomogeneous fluoride distribution as a function of particle size. Only the -100 +150 mesh fraction of material at each concentration was used for the SRM.

Homogeneity testing: Measurements at NIST by the ISE method following oxygen bomb decomposition (Z. Chun, W. F. Koch) showed material heterogeneity of 8% and 6.5% (relative standard deviation) for the low and high level samples, respectively. Similar testing at the Boyce Thompson Institute supported these findings.

Gaithersburg, MD 20899
August 2, 1991

William P. Reed, Chief
Standard Reference Materials Program

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Certification analyses were performed in cooperation with the Aluminum Association, Inc. by the following laboratories:

NIST, Inorganic Analytical Research Division, Z. Chun, L.A. Holland, and W.F. Koch.

Boyce Thompson Institute, Cornell University, Ithaca, NY, J.S. Jacobson and L. Weinstein.

Environmental Strategies, Ithaca, NY, R.H. Mandl and J. Heath.

National-Southwire Aluminum Company, Howesville, KY, A.G. Self.

Columbia Falls Aluminum Company, Columbia Falls, MO, D.F. Ryan.

Alcan Smelters and Chemicals, Ltd., Jonquire, Quebec, Canada, G. Desbiens.

Aluminum Company of America, Alcoa Center, PA, N.J. Hornung.

REFERENCES

[1.] Association of Official Analytical Chemists 1990. Official Methods of Analysis, 15th Edition, Vol. 1. Fluoride in Plants Semi-automated Method 978.03, pp. 52-55.

[2.] Intersociety Committee (1989). Methods of Air Sampling and Analysis, 3rd edition. J. P. Lodge, Jr., Editor, Method 204, pp.332-356.

[3.] M. Natrella, Experimental Statistics, NBS Handbook 91, pp. 2-13 to 2-15.