

# Certificate of Analysis

**NRC-CNRC**

Reference Material

## DUWF-1

Durum Wheat Flour Reference Material for Trace Metals and other Constituents

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DUWF-1 is a Reference Material prepared from food grade durum wheat flour. Reference and information quantity values are provided in the following tables for trace metals and other constituents. Reference values are equally weighted means of results from at least two analytical methods and/or laboratories. Uncertainties are expressed as a 95 % confidence interval. All values are expressed on a dry-mass basis.

**Table 1: Reference values for DUWF-1**

Element	Mass fraction, mg/kg
aluminium	$11.7 \pm 4.7$
barium	$2.11 \pm 0.47$
cadmium	$0.11 \pm 0.05$
calcium	$278 \pm 26$
chromium	$0.023 \pm 0.009$
cobalt	$0.008 \pm 0.004$
copper	$4.30 \pm 0.69$
iron	$41.5 \pm 4.0$
lead	$0.023 \pm 0.006$
magnesium	$1070 \pm 80$
manganese	$16.0 \pm 1.0$
molybdenum	$0.70 \pm 0.12$
nickel	$0.17 \pm 0.08$
phosphorus	$2900 \pm 220$
potassium	$3180 \pm 140$
rubidium	$2.0 \pm 0.4$
sodium	$16.0 \pm 6.1$
sulfur	$1930 \pm 280$
strontium	$1.19 \pm 0.09$
vanadium	$0.021 \pm 0.006$
zinc	$22.2 \pm 1.7$



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**Table 2: Information values for trace elements in DUWF-1**

Element	Mass fraction, mg/kg
arsenic	0.03
bromine	6.6
chlorine	680
fluorine	0.1
iodine	0.006
nitrogen	27 000
titanium	5

**Table 2 (continued): Information values for DUWF -1**

Quantity	Value	Unit
ash	1.364	g/(100 g), %
protein	15.48	g/(100 g), %
carbohydrate	80.34	g/(100 g), %
total dietary fibre	4.61	g/(100 g), %
energy content	408.6	kcal/(100 g)
energy content	1709	kJ/(100 g)

The protein concentration was calculated from the nitrogen values using a conversion factor of 6.25; subsequent calculations of carbohydrates and calories were also based on these protein concentrations. The value for energy content is the mean of the individual caloric calculations. The mean proximate values were used for calculation, with caloric equivalents of 9, 4, and 4 for fat, protein, and carbohydrate, respectively.

### Reference values

Reference values are non-certified values for which insufficient data are available to provide a comprehensive estimate of uncertainty to permit their full certification (Table 1).

### Information values

Information values are those for which insufficient data are available to provide any estimate of uncertainty (Table 2).

### History

DUWF-1 (formerly known as RM 8436) was prepared and characterized by the Centre for Land and Biological Resources Research, Agriculture Canada [1-5]. Following the original analyses for elemental value assignment by the laboratories listed in ref [1], the material was distributed by the National Institute of Standards and Technology (NIST) from 1993 until 2009, after which the stock was transferred to the National Research Council Canada (NRC). The NIST Report of Investigation for RM 8436 is available and contains the list of methods used for analysis [1]. In 2014 NRC examined

assigned values in RM 8436. The data were retained if the original reference value was confirmed; otherwise the reference value was relegated to an information value. Nitrogen, chlorine, bromine, iodine, proximates, total dietary fiber and energy content were changed from reference to information values, mercury and selenium were withdrawn and fatty acids removed. The material was renamed DUWF-1 by NRC.

### **Intended use**

This Reference Material (RM) is intended for use in evaluating analytical methods and instruments used for the determination of major, minor, and trace constituent elements in flour and other similar food, agricultural, and biological materials. A minimum sample mass of 500 mg is recommended.

**This material is intended for laboratory use, not for human consumption.**

### **Storage and sampling**

DUWF-1 should be stored at under refrigeration between 2 °C and 8 °C in its original bottle, tightly capped, and not exposed to direct light or ultraviolet radiation. The bottle should warm up to room temperature prior to opening. Prior to each use, contents of the bottle should be well mixed by gentle shaking and rolling of the container. Values reported in this certificate represent total concentrations of elements in this RM.

### **Instructions for drying**

Moisture content should be determined on a separate subsample for conversion of analytical results to a dry-mass basis. The recommended method is drying for 4 hours in an air oven at 85 °C.

### **Preparation of material**

The source of material for DUWF-1 was enriched durum wheat flour, containing reduced iron, ammonium chloride, and potassium bromate, obtained from Ogilvie Mills Ltd., Montreal, Quebec, Canada. All preparatory work following acquisition of the commercial product was performed at the facilities of Agriculture Canada, Ottawa [1-5]. The dry bulk powder was sterilized with <sup>60</sup>Co gamma radiation to 20 kGy by Atomic Energy of Canada Ltd. All subsequent processing was performed using plastic equipment. The material was sieved through nylon monofilament sieve cloths supported in high density white polyethylene holders. Pairs of sieves with openings of approximately 200 µm and 50 µm were used to yield suitable narrow middle cuts constituting the RM. This fraction was blended in a poly-(methylmethacrylate) V-configuration blender and packaged into clean 150 mL brim capacity, colorless glass bottles with triseal polyethylene-lined white polypropylene screw caps. A total of 144 randomly selected units were used for physical and chemical characterization in the original analyses. Units were individually hermetically sealed in aluminium-nylon pouches to enhance long term stability.

### **Stability**

The original values assigned in 1993 were reassessed in 2014 confirming the long term stability of this material.

### **Homogeneity**

Homogeneity testing was performed on randomly selected units for 13 elements by three laboratories [6,7]. In addition, the analytical results obtained from a large number of analysts participating in the interlaboratory characterization campaign were assessed to provide homogeneity estimates for other elements [6,7]. No statistically significant heterogeneity was found for aluminium, barium, bromine,



cadmium, calcium, chlorine, copper, iodine, iron, lead, magnesium, manganese, mercury, molybdenum, nickel, nitrogen, phosphorus, potassium, rubidium, selenium, sodium, strontium, sulfur, vanadium, and zinc in sample sizes required by the analytical technique ranging from 0.1 g to 2 g. Data for all analytes (including the proximates) have been treated as though they are homogeneous, although the homogeneity of other analytes has not been investigated.

### **Uncertainty**

Original uncertainty estimates for RM 8436 were verified at NRC and were retained in this report. As stated in the RM 8436 report [1], uncertainties are imprecision estimates expressed as a 95% confidence interval, based on a sample mass of at least 500 mg. These uncertainties, based on between-method and between-laboratory, between-unit and within-unit estimates of variances, include measures of analytical method and laboratory imprecisions and biases and material inhomogeneity.

### **Metrological traceability**

In 2014 the material was reassessed using SI traceable calibrants and CRMs. As such, DUWF-1 serves as suitable reference material for laboratory quality assurance programs, as outlined in ISO/IEC 17025.

### **Quality System (ISO/IEC 17025, ISO Guide 34)**

This material was produced in compliance with the documented National Research Council of Canada (NRC) Measurement Science and Standards (MSS) Quality System, which conforms with the requirements of ISO/IEC 17025 and ISO Guide 34.

The MSS Quality System supporting NRC calibration and measurement capabilities, as listed in the Bureau international des poids et mesures (BIPM) key comparison database (<http://kcdb.bipm.org/>), has been reviewed and approved under the authority of the Inter-American Metrology System (SIM) and found to be in compliance with the expectations of the Comité international des poids et mesures (CIPM) Mutual Recognition Arrangement. The SIM certificate of approval is available upon request.

### **Updates**

Users should ensure that the certificate they have is current. Our website at [nrc-cnrc.gc.ca/crm](http://nrc-cnrc.gc.ca/crm) will contain any new information.



## References

1. NIST (2008) Reference Material 8436, Durum Wheat Flour. Report of Investigation, National Institute of Standards and Technology, Gaithersburg, MD (revision of 1993 report)
2. M. Ihnat (1988) Preparation of Twelve Candidate Agricultural Reference Materials. *Fresenius' J. Anal. Chem.* 332: 539-545
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4. M. Ihnat, M.S. Wolynetz (1994) An Interlaboratory Characterization (Certification) Campaign to Establish the Elemental Composition of a New Series of Agricultural/food Reference Materials. *Fresenius J. Anal. Chem.* 348: 452-458
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6. M. Ihnat, R.W. Dabeka, M.S. Wolynetz (1994) Preparation and Homogeneity Characterization of Ten Agricultural/Food Reference Materials for Elemental Composition. *Fresenius' J. Anal. Chem.* 348: 445-451
7. M. Ihnat, M. Stoeppler (1990) Preliminary Assessment of Homogeneity of New Candidate Agricultural/Food Reference Materials. *Fresenius J. Anal. Chem.* 338: 455-460

## Authorship

The following staff members of Measurement Science and Standards at NRC contributed to the production of DUWF-1: S. Willie, P. Grinberg, J. Meija, and Z. Mester.

## Acknowledgements

The material was prepared at Agriculture Canada under the direction of M. Ihnat, Centre for Land and Biological Resources Research (CLBRR). Coordination of the technical measurements leading to the value assignment of this RM was performed by M. Ihnat of CLBRR, Agriculture Canada and K.E. Sharpless and S.A. Wise of the NIST Analytical Chemistry Division. Following the original analyses for elemental value assignment, the material was distributed by NIST to Covance Laboratories (Madison, WI), Lancaster Laboratories (Lancaster, PA), Medallion Laboratories (Minneapolis, MN), and Southern Testing and Research Laboratories (Wilson, NC) for the measurement of proximates, fatty acids, calories, and total dietary fibre. Statistical support was provided by M.S. Wolynetz, Statistical Research Section, Research Program Service, Agriculture Canada and L.M. Gill of the NIST Statistical Engineering Division. A complete list of collaborating laboratories and analysts is given in the NIST report [1].



**DUWF-1**

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**Approved by:**



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**This Certificate is only valid if the corresponding product was obtained directly from NRC or one of our qualified vendors.**

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