Fusion Engineering and Design 107 (2016) 13–24

Contents lists available at ScienceDirect

Fusion Engineering and Design

journal homepage: www.elsevier.com/locate/fusengdes

Round Robin test for the determination of nitrogen concentration in solid Lithium

P. Favuzza [a,*], A. Antonelli [b], T. Furukawa [c], F. Groeschel [d], R. Hedinger [e], T. Higashi [f], Y. Hirakawa [c], M. Iijima [c], Y. Ito [c,1], T. Kanemura [c], J. Knaster [g], H. Kondo [c], G. Miccichè [b], F.S. Nitti [b], S. Ohira [c], M. Severi [h], M. Sugimoto [i], A. Suzuki [f], R. Traversi [h], E. Wakai [c]

[a] ENEA Center, Via Madonna del Piano 10, 50019 Sesto Fiorentino, Italy
[b] ENEA Research Center, Brasimone, 40035, Camugnano, Italy
[c] JAEA Research Center, Tokai-mura, Ibaraki, Japan
[d] KIT Research Center, Hermann-von-Helmholtz-Platz 1,76344 Eggenstein-Leopoldshafen, Germany
[e] F4E Research Center, Boltzmannstraße 2, 85748 Garching, Germany
[f] University of Tokyo, Japan
[g] IFMIF-EVEDA Project Team, Rokkasho, Japan
[h] University of Firenze, Via della Lastruccia 3, 50019 Sesto Fiorentino, Italy
[i] JAEA Research Center, Rokkasho, Japan

HIGHLIGHTS

- Nitrogen contained in solid Lithium is converted into Ammonium ion.
- Ammonium ion is suitably quantified by ionic chromatograph or by Ammonia sensor.
- Good agreement of the partner's results has been achieved.
- Maximum operative reproducibility and blank subtraction are necessary.

ARTICLE INFO

Article history:
Received 2 November 2015
Received in revised form 3 March 2016
Accepted 4 March 2016
Available online 12 April 2016

Keywords:
IFMIF
Lithium
Round Robin
Nitrogen concentration

ABSTRACT

Three different partners, ENEA, JAEA ed University of Tokyo, have been involved during 2014–2015 in the Round Robin experimentation for the assessment of the soundness of the analitycal procedure for the determination of the Nitrogen impurities contained inside a solid Lithium sample. Two different kinds of Lithium samples, differing by about an order of magnitude in Nitrogen concentration (~230 wppm; ~20–30 wppm), have been selected for this cross analysis. The agreement of the achieved results appears very good for what concerns the most concentrated Lithium and indicates each partner's procedure is appropriate and intrinsecally able to lead to meaningful values, characterized by a relative uncertainty of just few %. The smaller agreement in the case of the less concentrated Lithium anyway points out that particular attention must be paid to reduce as much as possible any source of external contamination and highlights the importance of the proper blank subtraction.

1. Introduction

The wide interest of the Fusion Energy community in Lithium arises from the possibility of employing this metal both as a coolant and a tritium breeder. Thanks to its high thermal conductivity, heat capacity and boiling point, it can in fact be considered a suitable first wall and blanket coolant. At the same time, it can be employed in reactors based on the Deuterium-Tritium fuel cycle, being able to react with the fusion generated Neutrons to produce Tritium.

That is not all: Lithium finds application also as a Neutron flux generator, when bombarded by a beam of Deuterons. The International Fusion Material Irradiation Facility (IFMIF) [1,2] exploits this Lithium feature: two 40 MeV Deuteron accelerators focus their beams on a liquid Lithium target, to produce a high energy neutron flux (10^{18} n/m^{2}s, energy peak at 14 MeV) which is directed to an

* Corresponding author.
E-mail address: paolo.favuzza@enea.it (P. Favuzza).
[1] Now moved to GIC Company, Hitachinaka, Ibaraki, Japan.

http://dx.doi.org/10.1016/j.fusengdes.2016.03.026

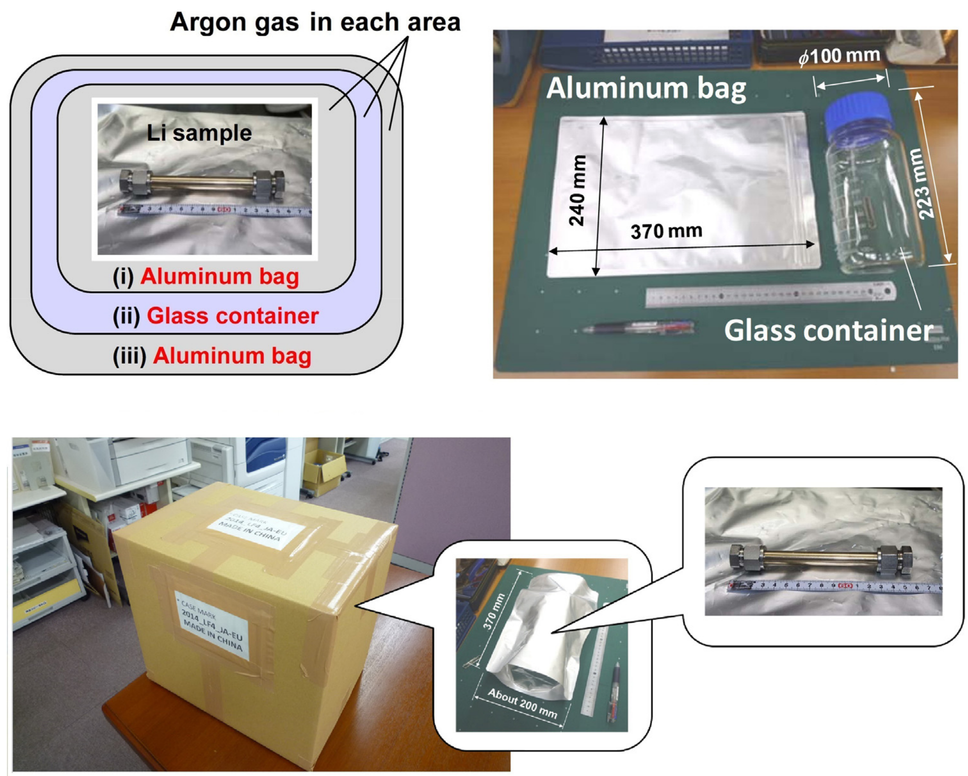

Fig. 1. containers and packaging details for the shipping of 'Japanese' Lithium by JAEA.

array of different materials specimens, to test them as candidates materials for future nuclear fusion applications.

Whatever the application, the compatibility of structural materials with liquid Lithium is one of the main challenges. It is also known that the presence of even small amounts of non metals impurities solved in liquid Lithium, particularly Nitrogen, sure enough enhances the chemical corrosion mechanisms affecting materials like steels [3,4]. Not only Lithium Nitride may erode tubing materials, but the Chromium component in steels may react with Nitrogen and Lithium leading to the steel embrittlement [5].

The reduction and the control of Nitrogen concentration in Lithium is therefore mandatory in many applications and in this regard IFMIF dictates that the weight concentration of Nitrogen does not exceed 10 ppm. No online devices is yet able to real-time measure the Nitrogen concentration in liquid Lithium flowing throughout a plant, anyway it is possible to fill with Lithium a small volume sampler, detach it from the plant after the metal cooling and solidification, and perform a specific batch chemical analysis on it.

This paper deals with the chemical procedure employed to evaluate Nitrogen concentration in a solid Lithium sample. Basically it entails to react the Lithium sample with water and convert all the Nitrogen to Ammonia, which is then quantified through a specific technique. The procedure substantially borrows from a similar method previously adopted for Nitrogen contained in Sodium [6] and has been already proposed and studied during the last years in many variations, mostly differing in the final technique employed to measure the produced Ammonia [7–10]. In none of the previous experiences, anyway, it was possible to dispose of a Lithium standard containing a known and certified value of Nitrogen concentration, so some doubt could remain about the real accuracy of the obtained results.

The work reported in this paper tries to overcome this limit, by implementing a "Round Robin" experimentation. During 2014, three different institutions, ENEA (*Italian National Agency for New Technologies, Energy and Sustainable Development*), JAEA (*Japan Atomic Energy Agency*) and University of Tokyo, which are all partners of the IFMIF project, have therefore performed the analysis of 2 identical Lithium samples, each institute independently, in its own laboratory and with its own specific setup and instrumental solutions. More space is given in the paper to the description of the ENEA procedure, which has never been reported before, while the others procedures are briefly reminded, since based on the same theory and already detailed elsewhere [10–12].

The comparison of the results achieved by the three partners permits to better assess their goodness and to highlight possible general issues in the overall process.

2. Analyzed Lithium

Two different kinds of Lithium have been selected, with the goal of disposing of different values of Nitrogen concentration.

The first one is 'Japanese' Lithium, i.e. a Lithium sampled almost two years before the Round Robin experimentation from a flowing liquid Lithium plant realized in the JAEA O-arai Research Centre in the framework of IFMIF activities. At that time, a fraction of this Lithium had been analyzed, giving an indicative value of Nitrogen concentration around 250 wppm; the rest of the Lithium was conserved inside the original samplers detached from the plant, hermetically closed and stored inside a glovebox filled with Argon, to exclude any possible external contamination.

One of the original samplers has been now reinvestigated by JAEA, by opening and analyzing the Lithium inside; one has been delivered to the University of Tokyo; a third one has been instead delivered to ENEA.

In order to avoid contamination during the long shipping to Italy and also to comply with the strict safety regulations, a multilple packaging was realized, as illustrated in Fig. 1. The original sampler (~25 mL steel tube, closed with Swagelok caps at the ending) was inserted inside a first aluminum bag, this bag inside a glass container, the glass container inside a second larger aluminum bag: