

## Full Paper

# Application of Cerimetric Methods for Determining the Metallic Aluminum Content in Ultrafine Aluminum Powders

Tatyana D. Fedotova\*

Novosibirsk State University, 630090 Novosibirsk (Russia)

Oleg G. Glotov, Vladimir E. Zarko

Institute of Chemical Kinetics and Combustion, Russian Academy of Sciences, 630090 Novosibirsk (Russia)

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## Abstract

Ultrafine (nano-sized) aluminum is a promising component for solid propellants, gel propellants, explosives, etc. The work is focused upon a problem of determining active (metallic) aluminum content in nanoAl powders as one of the characteristics of their reactivity. It is shown that the high reactivity, the presence of either the gases adsorbed or coating matter on the particle surface restricts the traditionally used permanganometric and volumetric analytic methods. A new technique determining the active aluminum content is presented. This is the adaptation of known cerimetric method based on the analytical reaction  $Ce^{4+} + e^- = Ce^{3+}$ . The method can be applied for analysis of metallic aluminum in the probes of nanoAl (including ones with organic coating), as well as micron sized aluminum, and their condensed combustion products.

**Keywords:** Nanoaluminum, Metal Aluminum Content, Cerimetric Method, Combustion

## 1 Introduction

It is known [1] that a decrease in aluminum particle size leads to an increase in specific surface area of powder and to the enhancement of its chemical activity. The development of techniques for producing ultrafine aluminum (below refer to as nanoAl) with particle size less than 200 nm, such as vaporization/condensation (Gen-Miller method) [2–4], plasma recondensation [5–6], and electroexplosion of wire [7–10] has initiated a number of works studying the combustion of various propellant compositions containing nanoAl.

The possible areas of using nanoAl in chemical propulsion systems, i. e. in classical and hybrid rocket motors, are:

- solid propellant formulations to change the burning rate and vary its dependence on pressure,
- solid propellant formulations to reduce agglomeration,
- mixtures with liquid hydrocarbon fuels for jet engines,

- mixtures with gelled water as a promising non-toxic propellant for space applications [11–12].

Brief reviews of the basic publications are presented in [1, 13–15]. In all cases, the use of nanoAl requires objective characterization of its properties. One of the most important characteristics is the content of active (metal) aluminum. It is worth noting that the corresponding analysis methods are still under development, since conventional methods used for characterization of micron sized aluminum powders are either often unsuitable for the analysis of nanoAl or require substantial modification. In the present work, the problem of the determination of metal aluminum in nanoAl particles by means of analytical chemistry is considered.

## 2 The Difficulties of the Chemical Analysis of NanoAl

A decrease in particle size causes changes in the specific surface of the powder, its structure, phase composition, and relative thickness of the oxide shell. These parameters are also affected by the method of powder production. As a result, the reactivity of nanopowders changes substantially, and the oxidation and combustion of nanoAl proceed in a manner differing from that for the micrometric aluminum powders [1, 13–15]. A very indicative example is the interaction of nanoAl in the stoichiometric mixture with gelled water in the regime of layer-by-layer self-sustaining combustion [16]. Moreover, nanoAl reacts with water even at room temperature [17]. The interaction between fine aluminum particles and water is one of the factors adding complexity in the chemical analysis.

Whatever the method of production, the nanoAl powders are subjected to passivation to ensure their stability. The passivation involves the treatment of surface with either gaseous or liquid reagents [9–10, 18–19] that form a protective layer. In the simplest case, this can be an oxide layer. In more complicated cases, a layer of special-purpose

\* Corresponding author; e-mail: ftor@academ.org

substance is created on the surface in addition to the oxide layer. For example, some organic compounds form coatings that prevent particles from conglutination [18]. Others render hydrophobic properties and stability against water [19]. The latter is important for the nanoAl/gelled H<sub>2</sub>O propellants [11–12]. From the viewpoint of chemical analysis, the presence of coatings other than oxide can be a complicating factor to be taken into account.

A gaseous “shell” is known to be formed on the surface of particles when nanoAl is produced by the electroexplosion of wire. The content of adsorbed gases can reach 7% [15]. Here and below, mass percent values are used. It was shown [17] that the presence of adsorbed gases is a factor, which may lead to overestimated aluminum content determined by means of gas volumetric analysis in the powder obtained by electroexplosion method.

In order to eliminate a systematic error of the gas volumetric method, some specific preparation procedures can be used for removing the particles’ gaseous shell in the tested sample through vacuumization or thermodesorption. However, one cannot be quite sure that this would not lead to the partial oxidation of the surface, the changes in the chemical composition of the sample under analysis and the distortion of the results of analysis. Therefore, the problem of the chemical analysis of nanoAl requires the application of independent analysis methods followed by the comparison of the results obtained.

The permanganatometric method [21] has been successfully used to analyze both the virgin aluminum powders and the condensed combustion products (in general, consisting of Al<sub>2</sub>O<sub>3</sub> and unburned Al). Actually, in our practice this method, being more convenient and accurate, has completely replaced the volumetric method. However, when it was initially used to analyze a number of nanoAl powders, first of all those with coatings, unsatisfactory results were obtained. The powders exhibited high chemical activity, which resulted in a competitive reaction accompanied with hydrogen evolution and, as a consequence, caused poor reproducibility and gave a negative systematic error. This finding was confirmed by the results of chelatometric determination of the total aluminum content in these powders. The oxygen content, calculated as a difference, allowed estimating the content of aluminum oxide as the main compound on the surface of metal particles. With respect to the mass balance, the obtained values indicated that the metal aluminum content determined by permanganatometric procedure was underestimated, even taking into account that the nanoAl powders contain the admixtures of other metals and gases adsorbed on the surface.

Actually, although the passivating coatings protect partially particle surface against oxidation by the air oxygen, they are easily permeable under the action of oxidizing reagents in solution and do not hamper the manifestation of the high chemical activity of nanoAl during analytical reactions.

In addition, an important feature of nanoAl (including the particles with coatings) is their ageing, i. e. the degradation of properties during storage [19].

The importance of controlling the nanoAl chemical composition dictates some additional requirements to chemical analytical methods: they should be reproducible, convenient for multiple analyses, etc.

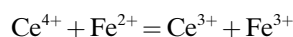
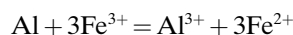
### 3 Cerimetry as a Procedure to Solve the Problem

The use of redox reactions for analytical purposes implies a definite difference in the oxidative-reductive potential (ORP) values for the corresponding semi-reactions which provide their completeness. However, when calculating the corresponding equilibrium constants for nanoAl powders one cannot use the standard ORP values, because for the nanopowders produced by electroexplosion method the recorded ORP values are substantially lower than those for the bulk metal [15]. This is because the fine aluminum behaves as stronger reducing agent than the coarse particles or the bulk samples and readily interacts with the protons of acids and water. The finely divided state of the powder results in accelerating the process macrokinetics. Thus, the hydrogen evolving at a high rate has not enough time to reduce Fe<sup>3+</sup> ions in solution in course of permanganatometric analysis.

Based on these prerequisites, we decided to use a stronger oxidizing agent than Fe<sup>3+</sup> ion (whose standard ORP is 0.77 V) to oxidize aluminum, in order to suppress the participation of protons in this process and to avoid hydrogen evolution from the reaction mixture. This idea is realized in cerimetric method, which is widely known [21–22] but has never been used for systems under study.

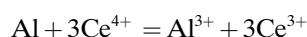
The cerimetric procedure is based on the analytical reaction  $Ce^{4+} + e^- = Ce^{3+}$ , which proceeds in the strong acid environment. The value of its potential depends on the nature and concentration of an acid. In a 1 M–4 M solution of sulphuric acid it comprises 1.44–1.42 V. The large ORP value, the absence of side reactions, the stability of the working solution of the titrant and the simplicity of fixing the equivalence point are the advantages of this method.

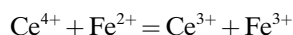
A direct titration by a cerium(IV) solution can be used in principle to determine the content of Fe<sup>2+</sup> ions formed in the solution of ferric ammonium sulfate during the oxidation of metal aluminum, instead of potassium permanganate or dichromate according to the following reactions:



This was tested for the commercial grade Russian aluminum powders PA-4 and ASD-6 by both methods and similar results were obtained.

In order to analyze metal aluminum in aluminum powder, including nanoAl with coatings, a special procedure of back titration was developed according to the following reactions:





The powder dissolution is carried out in a solution of cerium(IV) sulphate; its excess is titrated with a solution of Mohr's salt (ferrous ammonium sulphate). The dissolution of nanoAl and ASD-6 powder proceeded quietly, without noticeable gas evolution under room temperature for 10–15 minutes; in the case of PA-4 powder the slight heating was necessary in order to speed up the process.

The reagents used are:

1. Cerium(IV) sulphate, 0.20 N–0.25 N solution in 1 M sulphuric acid.
2. Mohr's salt, 0.20 N–0.25 N solution in 1 M sulphuric acid.
3. Phenylanthranilic acid or ferroin (0.1% aqueous solution of an indicator).

The precisely measured volume of the solution of cerium sulphate taken in excess (25.00–40.00 mL) is poured into a flask for titration. The aluminum powder sample (30–40 mg) is weighed in a small glass cup and placed into the flask, so that the powder is immersed into the solution together with the cup. When the sample is completely dissolved at the room temperature for nanoAl or under slight heating for coarse powders (and subsequent cooling back to room temperature), 1–2 drops of the indicator are added to the solution; the titration with Mohr's salt is carried out until pink colour appears.

The metal aluminum content (mass %) is calculated from the equation:

$$\% \text{Al} = (V_{\text{Ce}} * N_{\text{Ce}} - V_{\text{Fe}} * N_{\text{Fe}}) * M_E(\text{Al}) * 100\% / m,$$

where  $V$  is the volume of solution in litre;  $N$  is the concentration (equiv/L) of solution;  $M_E(\text{Al}) = 8.993$  g/equiv is the equivalent mass of aluminum;  $m$  is the sample mass in gram.

One of the two procedures is used to standardize the working solutions:

1. The given volume of the solution of cerium(IV) sulphate is titrated with the solution of Mohr's salt, preliminarily standardized by means of either permanganatometric or dichromatometric procedures.

2. An excess of cerium(IV) sulphate is added to the precisely weighed portion of sodium oxalate. The mixture is heated up to 50–60 °C, kept at this temperature for 5–10 minutes, and cooled. The indicator is added, and excess of cerium is titrated with the solution of Mohr's salt.

The preparation of solutions and the methods of their standardization are described in the textbooks on analytical chemistry [22–23]. The cerium solution titre does not vary with time. The Mohr's salt solution titre, which decreases during storage, must be checked just before analysis by using the cerium solution titre.

## 4 Results and Discussion

A brief description of 9 different aluminum powder samples given by their producers is presented below and the results of analyses are summarized in Table 1. The powders 1–6 were used in [17] as the test objects. The “working powders” 7–9 represent nanoAl powders demonstrating “good combustion behaviour” in mixtures with gelled water [11, 12]. This means that the nanoAl/H<sub>2</sub>O mixtures were easily ignited and burned in self-sustaining mode at atmospheric pressure. For powders 7–9 we used the original designations given by the producers.

It is worth to note that the test powders 1–6 have been produced in 2001 and stored for 3 years (column “age” in Table 1) before analysis. This should be kept in mind when comparing our data with those provided by the powder producers [18, 19]. The working powders 7–9 were produced in 2004 (approximately 1 year age).

Aluminum powder samples:

- 1: PA-4 is the commercial aluminum powder. Teardrop-shaped particles with mean sizes  $D_{10} = 8.6$  μm,  $D_{30} = 14.6$  μm, and  $D_{43} = 41$  μm (data obtained by using automated granulometer “Malvern 3600E”).

- 2: ASD-6 is the commercial aluminum powder produced by liquid metal pulverization. Spherical particles with mean sizes  $D_{10} = 3.3$  μm,  $D_{30} = 3.8$  μm, and  $D_{43} = 5.4$  μm (“Malvern” data).

**Table 1.** Results of chemical analyses.

No.	Sample	Total Al, % mass our data	Metal Al, % mass our data	Metal Al, % mass. Volumetric method, producer's data	Age of powder, years
Test powders					
1	PA-4	97.9 ± 0.4	95.5 ± 0.6	N/A	>3
2	ASD-6	99.0 ± 0.1	97.3 ± 0.9	N/A	>3
3	L-Alex(2001)	87.6 ± 0.2	78.5 ± 0.5	86.1 [19]	3
4	NanoAl0	96.0 ± 0.1	89.1 ± 0.4	86.7 ± 0.5 [18]	3
5	NanoAl1	95.9 ± 0.2	87.7 ± 0.3	85.5 ± 0.7 [18]	3
6	NanoAl2	95.6 ± 0.3	90.5 ± 0.6	90 ± 0.6 [18]	3
Working powders					
7	Al1-Ox	92.5 ± 0.2	76.6 ± 0.8	N/A	1
8	L-Alex(2004)	90.7 ± 0.4	82.0 ± 0.7	N/A	1
9	Al(N <sub>2</sub> /N <sub>2</sub> )	88.6 ± 0.4	76.1 ± 0.5	75.4 [19, 18],	1

**3:** L-Alex4(2001) is the nanoAl powder produced by the wire electroexplosion method. Spherical particles of mean size 100–200 nm (BET), exposed to the vapor of palmitic acid with the formation of the layer of aluminium palmitate whose salt is chemically bound to the metal [19].

**4:** NanoAl0 is produced by Gen-Miller's method. Spherical particles of 150 nm mean size (BET), stabilized by means of soft oxidation in the air. The thickness of hydrophilic oxide film is 2–3 nm [18].

**5:** NanoAl1 is produced by Gen-Miller's method. Spherical particles of mean size 150 nm (BET). The surface is modified by the vapour of propionic acid and stabilized by soft oxidation in the air. The coating is in the form of sandwich. The surface is hydrophobized by a quasi-polymer film [18].

**6:** NanoAl2 is produced by Gen-Miller's method. Spherical particles of mean size 150 nm. The surface is modified by the vapour of hexamethyldisilazane and stabilized by means of soft oxidation in the air. The coating is in the form of sandwich. The surface is hydrophobized by a monolayer of hydrophobic film [18].

**7:** Al1-Ox is the nanoAl powder produced by Gen-Miller's method. The powder consists of two fractions of particles with mean size 18 nm (8%) and 256 nm (92%). The surface is treated by dry gaseous oxygen in flow. Powder is further stabilized by means of soft oxidation in the air.

**8:** L-Alex(2004) is the nanoAl powder similar to L-Alex(2001), produced by wire electroexplosion method. The surface is modified by the vapour of palmitic acid [19].

**9:** Al(N<sub>2</sub>/N<sub>2</sub>) is the nanoAl powder produced by wire electroexplosion method. The designation (N<sub>2</sub>/N<sub>2</sub>) means that the powder was produced in nitrogen and passivated in nitrogen [20].

As follows from the methods of nanoAl powder production and storage, all the samples under analysis had a protective coating (organic or oxide) on the particle surface to prevent their oxidation. The powders were stored under normal conditions in the air for a long time, which allowed us to consider their state as quite established one.

The PA-4 and ASD-6 powders were analyzed using three techniques: the permanganatometric method [21], the cerimetric method with direct titration involving alum, and the cerimetric method with back titration. For each of these powders the results obtained by different methods coincided within the random error value. The percentage of metal aluminum shown in Table 1 for these samples is the averaged result of measurements performed by the aforementioned three methods.

The nanoAl powders were analyzed using the proposed cerimetric method with back titration. A relative standard deviation ( $s_r = s/x_{mean}$ ) of measured value does not exceed 0.8% that indicates the good results' reproducibility.

In addition, the nanoAl powders were analyzed to determine the total aluminum content (both active and oxidized) by means of the back chelatometric titration [21]. The measured total aluminum content turned out in a good agreement with the results of the cerimetric method.

When analyzing the data presented in Table 1, we can state the following:

1. The metal aluminum contents in the NanoAl0, NanoAl1, NanoAl2 powders after 3 year storage, determined by using the cerimetric procedure, are close to each other and to the contents reported in [18] determined by the gas volumetric procedure after 30 day storage. This allows making the conclusion that the different methods of powder treatment [18] (the gas-phase deposition of hydrophobic coating, or the passivation by air with the formation of hydrophilic coating) result in approximately equal stabilization degree of powders and prevent them from further oxidation in the air.

2. The Al1-Ox powder without organic coating exhibits a low content of metallic aluminum as compared with powders NanoAl0, NanoAl1, and NanoAl2 despite its relatively small age. This indicates that the organic coatings improve the conservation of powders at their contact with air.

3. The L-Alex(2001) and L-Alex(2004) powders differ appreciably in the content of both metallic aluminum and total aluminum. At the same time, the ratios of these values are equal ( $\approx 0.90$ ) for both powders. This indicates that the powders differ, probably, in the mass fraction of the coating material.

4. The content of metal aluminum in the L-Alex(2001) powder, determined by the cerimetric procedure, is noticeably lower than that determined in [19] by using the gas volumetric procedure. This difference can be attributed to the systematic error of volumetric procedure due to the presence of adsorbed gases, and/or to the degradation of the properties of powder with time.

5. All powders **7–9** (Al1-Ox, L-Alex(2004), Al(N<sub>2</sub>/N<sub>2</sub>)) demonstrating good combustion behaviour are characterized by moderate content of metallic aluminum (in the range 76.1%–82.0%). It indicates that for nanoAl/water compositions other characteristics of the powder reactivity should be taken into account along with the metal aluminum content.

## 5 Conclusions

An extremely high reactivity of the nanoAl powders hampers the determination of the metal aluminum content by means of permanganatometric method, which was developed and successfully used for the analyses of micron sized aluminum powders and for their condensed combustion products. The application of volumetric method to nanoAl powders produced by means of wire electroexplosion can lead to systematic error because of the presence of adsorbed gases.

For the analysis of nanoAl powders, including those with various passivating coatings, the method of cerimetric determination of metal aluminum has been developed. The method is free from the aforementioned shortcomings.

The method proposed is suitable not only for the analysis of nanoAl powders but also for the micron sized aluminum

powders and their condensed combustion products. The method is rather simple, accurate, and reliable (no systematic error, good results reproducibility). It is recommended for multiple analyses.

Although the new method can replace, in some cases, the traditional permanganatometric and volumetric methods, the different independent techniques should be used followed by comparison of the results to obtain complete and reliable information.

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