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Determination of Oxygen in Aluminum by Means of 14 Me V Neutrons with an Account of Flux Attenuation in the Sample

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SUMMARY

This study concerns the determination of oxygen present at low levels in aluminum using the 14 MeV neutron activation technique.

The sensitivity obtained amounted to 0.2 mg oxygen.

Various nuclear methods for the oxygen determination have been briefly reviewed.

The attenuation of fast neutrons inside the aluminum samples has been calculated.

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INTRODUCTION

14 MeV neutrons produced in neutron generators give rise as is well-known, to the following nuclear reaction in oxygen

$$O^{16}\ (n, p)\ N^{16}$$

This reaction has been successfully utilized for the determination of oxygen in metals by several investigators, e.g. Guinn [1], Strain [2], Mott and Orange [3], Fujii et al. [4] and Aude and Laverlochere [5]. The oxygen analysis of organic materials such as mineral oils has also been performed with this technique [6].

The 14 MeV neutron activation analysis is used for the determination of, besides oxygen, several other elements, e.g. nitrogen, phosphorus and silicon. The sensitivity for the various elements determined with this activation type has previously been treated by Gillespie and Hill [7]. A valuable compilation of the cross-section values for the 14 MeV neutron reactions has, moreover, been published by Neuert and Pollehn [8].

The fast neutron activation analysis is at present characterized by a high degree of automation. Samples to be irradiated are transferred in pneumatic tubes from target to detector within a few seconds and the irradiation, transfer and counting sequence may be automatically controlled. In the routine analysis of oxygen in metals the application of the dual sample irradiation technique implying the simultaneous irradiation of the standard and the sample in a rotating device in front of the target gives a high degree of precision in the analysis. Thus at higher oxygen levels a standard deviation of less than 1 % has been obtained with this technique [3]. Generally, this figure amounts to 1-5 % [1, 4]. At lower concentrations the precision is considerably reduced. At an oxygen level of 10 ppm in steel samples of about 10 g a standard deviation of 30 % has been reported [4]. However, this amount of oxygen is within the range of the practical detection limit, 0.1-0.2 mg oxygen [1, 2], with reference to a fast neutron facility of about 10^9 n/cm^2 sec.

Fujii et al. [4] compared the results obtained for the oxy-analysis in steel by the fast neutron activation method and the vacuum fusion technique. They obtained results that were in good agreement in the concentration range investigated, 0.2--0.001 %. Neutron activation analysis has in this case proved superior to the vacuum fusion method with regard to rapidity.

DETERMINATION OF OXYGEN BY VARIOUS OTHER NUCLEAR METHODS

A compilation of various nuclear methods for oxygen determination has previously been given by Bate [9]. Accordingly, only a few of these methods will be briefly mentioned in the following and recent works emphasized.

Aumann and Born [10] determined the O^{18}-content of water by exposing the aqueous samples to fast neutrons in a reactor and measuring the F^{18} activity induced by a recoil proton mechanism in hydrogen containing materials

$$O^{18}(p,n)F^{18}$$

This method is reported to give a sensitivity of about 10^{-4} g O^{18} in a 1 g aqueous sample irradiated for 2 hours in a fast flux of 6.7×10^{11} n/cm^2 sec. The F^{18} activity may also serve as indicator for another nuclear reaction.

Thus the reaction chain

$$Li^{6}(n,\alpha)H^{3} \quad \text{and} \quad O^{16}(t,n)F^{18}$$

can be utilized in the determination of lithium. The sensitivity of this method is, however, limited by several factors [10].

The triton activation technique has also been studied in detail by Goeij and Houtman [11]. Oxygen has, moreover, been determined in surfaces of solid samples by means of triton activation [12].

Amiel and Nir [13] have described a method for O^{18} analysis based on the reaction

$$O^{18}(\alpha,n)Ne^{21}$$

These authors bombarded O^{18}-labelled compounds with α-particles from a thoron source and detected the O^{18}-quantity by measuring the neutrons produced in this reaction. In the analysis of this amount in paper chromatograms they obtained a sensitivity of a few micrograms of O^{18} per cm^2 paper area when irradiating the samples in a flux of about 10^7 α/sec.

Using charged particle activation technique, accomplished with protons, deutrons, alpha- and helium- 3 particles accelerated to an appropriate energy by means of a linear accelerator, a cyclotron or a Van de Graaff machine, a higher degree of sensitivity can be obtained in the oxygen determination.

Thus Lemberg et al. [14] determined the content of O^{18} also by means of the reaction

$$O^{18}(\alpha, n)Ne^{21}$$

However, they used α-particles accelerated to 4.6 MeV in a cyclotron for the activation process, and measured the yield of the γ-quanta emitted from the excited Ne^{21} nuclei. In their work sensitivities of 2×10^{-9} g for O^{18} and 10^{-6} g for naturally occurring oxygen were obtained.

Using protons of energies up to 20 MeV and α-particles of 44 MeV in the activation process Engelmann [15] reported sensitivities of 10^{-2} ppm and 10^{-3} ppm respectively in the oxygen analysis. The following reactions are utilized in this context

$$O^{18}(p, n)F^{18},$$
$$O^{16}(p, \alpha)N^{13},$$
$$O^{16}(\alpha, pn)F^{18},$$
$$O^{16}(\alpha, d)F^{18},$$
$$O^{16}(\alpha, 2n)Ne^{18} \xrightarrow{\beta+} F^{18}$$

Lacroix and Tousset [16] studied the excitation function of the reaction

$$O^{16}(d, n)F^{17}$$

and they have also discussed the application of this reaction in the oxygen determination.

The sensitivity can be further improved if the oxygen determination is performed by means of He^3-particle activation technique, reported by Marcowitz and Mahony [17], which has also attracted great interest. Sensitivities at the ppb level have thus been reached. The following two reactions are used in the determination

$$O^{16}\ (He^3,\ p)\ F^{18} \quad \text{and} \quad O^{16}\ (He^3,\ n)\ Ne^{18}\ \beta^+\ F^{18}$$

The simultaneous determination of O^{16} and O^{18} has been accomplished with this technique [18] when the O^{18}-content is measured via the reaction

$$O^{18}\ (He^3,\ p)\ F^{20}$$

In a recent work by Ricci and Hahn [19] the sensitivities of 15 light elements for activation with 18 MeV He^3-particles have been treated.

The He^3-particle activation technique in the oxygen analysis has, moreover, been studied by Holm et al. [20]. In their work the channeling effect of He^3-ions in single crystals of germanium and its application to activation analysis has been given a preliminary study.

Oxygen can also be determined by photon irradiation.

With the "bremsstrahlung" of a 28 MeV linear accelator the oxygen analysis has been carried out with a sensitivity of 0.05 μg using the reaction [21]

$$O^{16}\ (\gamma,\ n)\ O^{15}$$

THE FLUX ATTENUATION INSIDE THE SAMPLE

The neutron source, i.e. the tritium target, is a thin slab and may be considered as a plane neutron source. If the sample is much smaller than the target and the distance between them is also small relative to the target diameter, then the flux attenuation between the source and the sample will be negligible. However, as in this case, the sample diameter and thickness are usually of the same order of magnitude as the diameter of the target. Hence, it is necessary to take into account the pure geo-

metric attenuation of the flux through the sample. The variation of the flux with the distance from a plane target has previously been investigated for one typical target size by Wilkniss and Wynne [22]. Applying their results to the geometry in this study, it is found that the average flux in an 0.5 cm thick sample is approximately 70 % of the flux at the target surface. However, the introduction of this geometric flux depression correction is avoided by using a standard having the same geometry as the sample.

The neutron flux is also attenuated in the sample for two other reasons:

1) the neutrons are scattered out of the sample,

2) the neutrons slow down below the threshold energy for the (n, p)-reaction in O^{16} through elastic and inelastic scattering in the sample.

The calculation of this "neutron transport" attenuation effect simultaneously with the geometric effect is rather complicated. This problem is avoided by using a standard of the same material and density as the sample.

However, carrying out such an experiment with a standard of the same material but with different density or of a different material, e.g. plexiglass, a flux correction factor has to be calculated. A reasonable estimate can be made if the geometric effect is assumed to be separable from the "transport" effect. Let us consider an experiment where the sample is of aluminum (density 2.7 g/cm^3) and the standard of plexiglass (density 1.2 g/cm^3) and both are 0.5 cm thick. It is in this connection reasonable to adopt the following model for the (n, p)-cross section in O^{16}: below the incident energy (E_o = 14 MeV) $\sigma_{n,p}$ is constant down to the effective threshold energy (E_t = 11 MeV) and below E_t $\sigma_{n,p}$ is zero. Further, it is reasonable to assume that the inelastic scattering at 14 MeV in aluminum, carbon and oxygen transfers the neutrons below E_t = 11 MeV [23] and that the same applies to the elastic scattering in hydrogen. If the neutrons are followed from collision to collision we derive the following approximate expression for the average flux depression factor (F) in the sample and standard due to scattering out of the sample and slowing down:

$$F = P_{es}\left[1 + P_c\frac{\Sigma_{el}}{\Sigma_t} + \left(P_c\frac{\Sigma_{el}}{\Sigma_t}\right)^2 + \ldots\ldots\ldots + \left(P_c\frac{\Sigma_{el}}{\Sigma_t}\right)^n\right] \quad (1)$$

where P_{es} is the escape probability for neutrons at the energy E_o, and Σ_{el} and Σ_t are the elastic scattering and total macroscopic cross section, respectively, averaged over the energy region $E_o - E_t$, and P_c is the corresponding collision probability. P_c is a function of $(a\,\Sigma_t)$ where a is the thickness of the sample, n is the average number of elastic collisions between E_o and E_t, i.e. $n = \frac{1}{\xi}\ln\frac{E_o}{E_t}$, where ξ is the average logarithmic energy loss.

For aluminum $\xi = 0.022$ [24], which means that $n \approx 10$, and for plexiglass $\xi = 0.054$ giving $n = 4$. In both cases P_c is so small and n so large that F could be calculated according to

$$F = P_{es}\left(1 - P_c\frac{\Sigma_{el}}{\Sigma_t}\right)^{-1}$$

Note that F is the ratio of the perturbed and unperturbed flux. From BNL-325 [23] the following averaged cross sections are taken;

Nuclide	σ_T (in barns)	σ_{el} (in barns)
Al^{27}	1.7	0.8
C^{12}	1.4	0.9
O^{16}	1.5	0.8
H^1	0.8	0.75 (this value is not included in Σ_{el})

For an 0.5 cm thick aluminum slab (2.7 g/cm^3) $P_c = 0.098$ and $P_{es} = 0.90$ [25] giving $\underline{F = 0.95}$. Similarly, for an 0.5 cm thick plexiglass slab $P_c = 0.107$ and $P_{es} = 0.89$ [25] giving $\underline{F = 0.93}$. Hence, with such a choice of materials for sample and standard a correction of 2 % has to be introduced for this particular thickness; for thicker samples the correction will be larger.

In this investigation encapsulated aluminum powder containing aluminum oxide (density 1.5 g/cm^3) was used as standard. For such a slab $\underline{F = 0.97}$, which also means a correction of 2 %.

EXPERIMENTAL

Neutron generator

The generator belonging to the Research Institute of National Defence situated at Ursvik outside Stockholm was used as neutron facility. This machine, which operates at 180 kV and about 300 μA has previously been described by During et al. [26].

Flux measurements

The fast neutron flux produced according to the reaction

$$H^3 (d, n) He^4$$

was measured by means of aluminum foils (diameter: 20 mm thickness: 1 mm). The Mg^{27} activity induced through the reaction

$$Al^{27} (n, p) Mg^{27}$$

was standardized by means of a Mn^{54} standard. With a fresh 5 Ci tritium target (diameter: 25 mm) a fast flux of 3.0×10^9 n/cm^2 sec. was measured close to the target area. The target was cooled by means of a film of streaming water.

Samples and pneumatic system

The aluminum samples had the form of disks or cylinders of various size and were polished before being introduced into the pneumatic system.

Two pneumatic systems were applied in this investigation.

For smaller samples (150 mg to about 1 g) polyethylene tubes of about 15 m length and various diameters were used. The system was operated with nitrogen gas, and the transport times were less than 5 sec.

The larger samples of about 5 g (diameter 20 mm and height 5 mm) were transferred within 2 sec. from target position to detector position in a pneumatic tube of aluminum with a length of 10 m and diameter of 32 mm. During the transport the sample

was held in an aluminum container functioning as a stabilizer in order to ensure reliable transfer. However, before being measured the sample was disconnected from this holder.

Measurements

The N^{16}-activity was measured by a 3" x 3" Na I (Tl) scintillation detector. The pulses between 4 and 8 MeV being registered. The samples were irradiated for 20 sec. and counted for 12 sec. Half-life measurements of the N^{16}-activity being carried out with a 400 channel pulse height analyzer, TMC.

Standards

The standards constituted cylinders with dimensions equal to those of the samples, and consisted of pure aluminum powder mixed with aluminum oxide. The standards were encapsulated in aluminum.

RESULTS AND DISCUSSION

In this study the detection limit for oxygen amounted to 0.2 mg. At this level the oxygen determination is carried out with a standard deviation of the single value of about 20 %, whereas this figure amounts to less than 5 % at oxygen levels exceeding 2 mg. The detection limit is based on the registration of a pulse number originating from N^{16} which is equal to the sum of the instrumental background and the background induced in the samples themselves.

The sensitivity of the oxygen analysis is influenced by factors such as the fast flux of the generator, the transfer system, the crystal dimensions etc., and it is also limited by the flourine contents of the sample. Thus, fluorine gives rise to N^{16} activity according to the following well-known reaction with 14 MeV neutrons

$$F^{19}\ (n,\ \alpha)\ N^{16}$$

Fluorine produces less than half the amount of N^{16} activity through this reaction that the same amount of oxygen produces through the corresponding (n, p)-reaction [27].

This interference does not, however, seem to be very common, and the extent of this "extra" N^{16} activity may be estimated by determining the fluorine content of the sample by means of another reaction, e.g. by F^{19} (n, p) O^{19} (5) or by F^{19} (n, 2n) F^{18}. The F^{18} activity is identified by means of the 0.51 MeV annihilation peak. However, the induced sodium activities in the aluminum samples also give rise to this peak. In this study direct spectrometric measurements of the fluorine content down to 1 mg in 1 g aluminum samples were performed. At lower fluorine concentrations the F^{18} activity may be measured after the separation from sodium with distillation technique [28].

According to the Texas Convention [29] the following reaction has been recommended for the determination of the fast flux

$$Cu^{63} (n, 2n) Cu^{62}$$

The applicability of this reaction in accurate fast flux measurements has previously been treated by Ricci [30] in his detailed study of the fast flux disturbances in the target assembly.

In the present investigation the flux has been determined with the reaction

$$Al^{27} (n, p) Mg^{27}$$

on account of the simplicity of the evaluation of the disintegration rate of Mg^{27}.

The unperturbed fast neutron flux in the monitor position used (close to the target) was calculated to be 95 % ($P_c = 0.05$). Consequently, the correction which has to be performed in the measurement of the average fast flux using this reaction amounts only to a few per cent. The activity contribution from neutrons of reduced energy [31] has been accounted for. The average energy of the fast neutrons in the monitor position was estimated to (14.5 ± 0.5) MeV taking into account the neutron angular distribution as well as the deutron energy [32].

With an oxygen standard consisting of plexiglass or aluminum powder containing aluminum oxide in the form of a slab with dimensions as given above, corrections of 2 % have to be made for the difference in neutron attenuation in sample and standard. However, when oxygen is determined at very low levels, i.e. less than 100 ppm, this correction is comparatively unimportant.

Considering the correction for differences in gamma-ray attenuation in sample and standard, this effect may be neglected.

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