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Determination of Magnesium in Needle Biopsy Samples of Muscle Tissue by Means of Neutron Activation Analysis

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Summary

Magnesium has been determined by means of neutron-activation analysis in needle biopsy samples of the order of magnitude 1 mg dry weight. The procedure applied was to extract the Mg^{27} activity from irradiated muscle tissue with concentrated hydrochloric acid followed by a fast hydroxide precipitation and gamma-spectrometric measurements.

The Mg^{27} activity was recovered in the muscle tissue samples to (97 ± 2) per cent.

The sensitivity for the magnesium determination is estimated as 0.3 µg.

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1.0 Introduction

The estimation of intracellular electrolytes in man is known to be of great importance in many diseased states, though in clinical practice they must for the most part be estimated indirectly from serum values. As regards magnesium, even serum values are difficult to obtain, owing to the lack of suitable analytical methods. However, in recent years flame photometry has proved to be the most suitable method for the analysis of magnesium in research, though it has not yet been incorporated in clinical routine analysis. Magnesium has been estimated by a few researchers in surgical macrobiopsies performed in human material (1, 2, 3), and once in needle biopsies with flame photometry (4). With the method of flame photometry, the sample is thereafter consumed for one magnesium determination.

However, the method of neutron activation analysis has the advantage of greater sensitivity, and it affords, moreover, the possibility of determining more elements of special clinical importance such as chlorine, potassium and sodium in the same sample. These elements have earlier been determined in the same needle-biopsy sample from human muscle (5).

Magnesium has previously been determined in biological material e.g. blood and blood-fractions with neutron activation analysis (6, 7). However, little information is available concerning the precision of these determinations. In the present investigation the Mg^{27} activity was extracted with concentrated HCl from dried, neutron-irradiated muscle samples. In this process the Szilard-Chalmers effect was found to play an important role (8).

2.0 Experimental

2.1 Samples

Muscle biopsy samples of 0.5 - 4 mg dry fat-free weight were irradiated in a thermal neutron flux of 2×10^{13} n/cm^2 sec. for periods of 10 minutes. After the irradiation the samples were crushed. The small biopsy samples had been weighed on a continuously recording electronic "Cahn" balance for three minutes and the wet weight was

then extrapolated to the moment for the performance of the biopsy. The fat-free dry weight of the sample was determined on the same balance after drying in 100 °C.

2.2 Extraction and precipitation

After the irradiation the homogenized samples were extracted three times with concentrated HCl in portions of 4 ml at a temperature of 100 °C. The concentrated HCl solution contained ~ 1 mg magnesium as carrier.

The solution was passed through a G4 filter, and 10 mg magnesium was then added before precipitation in strong alkaline solution (pH > 12). The solution was warmed to about 80 °C and allowed to stand for a few minutes to make the hydroxide precipitate settle.

The solution was then passed through a Hyflogel filter of 5 mm height, which was washed with ~ 30 ml NaOH solution of pH > 12. The filter had earlier been washed with the same solution.

3.0 Measurements

The gamma-spectrometric measurements were performed with a 256-channel pulse height analyser attached to a 3" x 3" NaI (Tl) crystal. The quantitative determinations were made with the 1.02 MeV full energy peak (fig. 1). Small remanent disturbing activities of Na^{24} and Cl^{38} together with the Mn^{56} activity that was also precipitated can easily be stripped off.

4.0 Results

The Mg^{27} activity was recovered in muscle tissue specimens to (97 ± 2) per cent. The value expresses the mean value of yield of 5 determinations with standard deviation of a single value.

The precipitation of magnesium as hydroxide including the filtration on a Hyflogel filter was found to give losses of less than 1 per cent.

Triple magnesium determinations performed on muscle tissue from the same rat of 1 to 3 mg dry fat-free weight gave as result 1.24 ± 0.12 µg/mg sample. With flame photometry a mean value of 1.22 ± 0.08 µg/mg sample was obtained. In the last case somewhat larger samples were analysed.

Magnesium in needle-biopsy specimens from living human quadriceps muscle of dry weight of 0.5 mg to 3.5 mg were also determined by this activation analytical method. In a series of 5 samples from different subjects a magnesium content of 1.16 ± 0.17 µg/mg sample was obtained. Values slightly less than 1 has previously been reported (2, 4).

5.0 Discussion

In the present study magnesium was determined in biopsy samples as small as 0.5 mg dry weight. This may be advantageous in clinical studies, as such small needle biopsies can be taken from human quadriceps muscle without discomfort for the patient. At the same time as the magnesium is determined, chlorine, potassium and sodium can easily be determined in the same sample as previously described by Bergström (5) using beta and gamma measurements or as follows, by means of an extraction procedure and gamma-ray spectrometry.

1. The Cl^{38} activity is determined by performing measurements directly on the sample.

2. The Mg^{27} activity is then extracted from the sample, as described in the present paper.

3. The Na^{24} activity is determined by measuring the extract solution after the decay of Cl^{38}. K^{42} is simultaneously determined when stripping off the Na^{24} contribution in the gamma-spectrum. (The activities belonging to the alkali metals have earlier been found to be nearly quantitatively extractable in concentrated HCl (8).)

   For the determination of phosphorous, which is also of special interest in this connection, longer irradiation periods have to be used (5).

In the Mg^{27} determination, the following two interfering reactions have been considered:

$Al^{27}(n,p)\ Mg^{27}$ [cross section for fission spectrum neutrons: 4.3 mb (9)]

$Si^{30}(n,\alpha)\ Mg^{27}$ [cross section for fission spectrum neutrons: 1 mb (9)]

Assuming the Al and Si content in muscle tissue to be 1 ppm and 20 ppm (10, 11), and the fast fission flux to be less than 1/10 of the thermal flux in the irradiation position used (12), the contribution of Mg^{27} from these reactions is less than one tenth of a per cent, and may therefore be neglected.

The sensitivity for magnesium with this method is estimated as 0.3 µg when irradiating biological samples at thermal neutron fluxes of 2×10^{13} n/cm^2 sec.

6.0 Acknowledgement

We are greatly indebted to Mr L. Åström for skillful technical assistance.

7.0 Note

After the preparation of this paper, an activation analytical method for magnesium yielding a recovery of 75 per cent has been published: Bowen, H.J.M., Cawse, P.A., and Daglish, M.: The Determination of Calcium and Magnesium in Biological Material by Radioactivation Analysis. The Analyst, Vol. 89, No. 1057, 266 April 1964.

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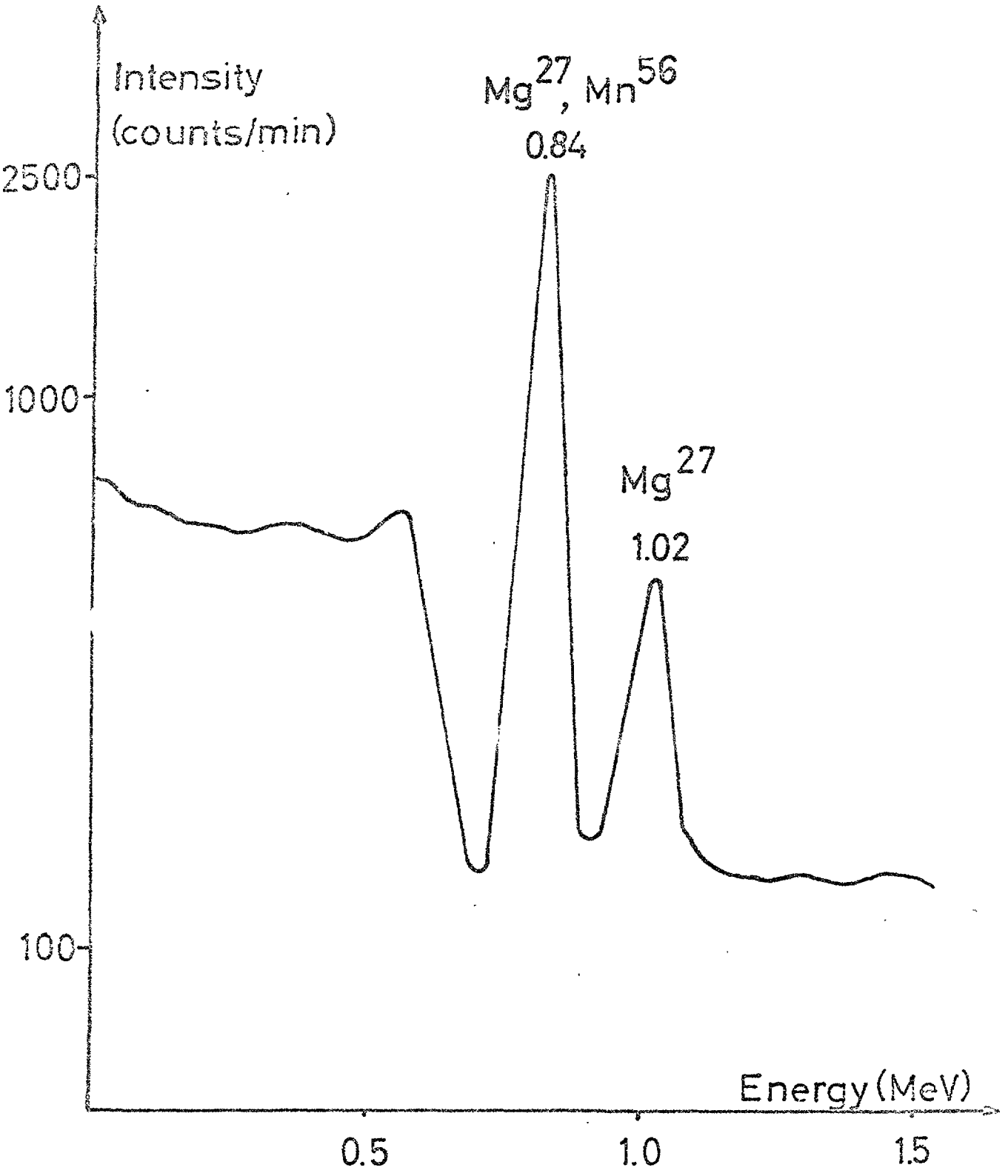
FIGURE I



Spectrum of the magnesium hydroxide precipitate.

Stripping not performed.

Mg^{27} γ : 0.84 MeV (~70%)
1.02 MeV (~30%)

Mn^{56} γ : 0.84 MeV (~100%)
1.81 MeV (~30%)
2.13 MeV (~20%)

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