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Determination of Elevated Levels of Dysprosium in Serum by Electrothermal Atomic Absorption Spectrometry

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A method has been developed for the determination of elevated levels of dysprosium in serum, from human beings or animals exposed to a dysprosium magnetic resonance imaging (MRI) contrast agent, by electrothermal atomic absorption spectrometry. Several potential chemical modifiers were tested in order to increase the sensitivity and to overcome the memory effect normally experienced with dysprosium. The following elements were tested as potential modifiers: lanthanum, europium, gadolinium, holmium, thulium, ytterbium, lutetium, thorium, tungsten, rhodium and palladium. Among these, lanthanum and gadolinium gave the most obvious positive effects on the analytical performance. By adding 5 μg lanthanum or 10 μg gadolinium to the tube, after the introduction of the sample, increased sensitivity, lowered limit of detection, reduced memory effect and improved precision of the method were obtained. Gadolinium was chosen as the most suitable modifier of the two. The analytical performance of the method was validated by adding dysprosium, as the MRI contrast agent dysprosium diethylenetriaminepentaacetic acid bis(methylamide) (DyDTPA-BMA), to the serum reference material Seronorm. The accuracy for the concentration range 10–400 ng ml^{-1} dysprosium was between 95 and 104% (recovery), the repeatability was typically <2.6% RSD, the reproducibility was between 2.3 and 5.8% RSD and the limit of detection (3s) was 1.1 ng ml^{-1} of Dy (absolute mass 11 pg) in Seronorm. The characteristic mass based on area measurements was 18 pg. The memory effect of the final method was <6%, measured as the % carryover of the previous atomization signal when a blank was atomized. The effect of adding CHF_3 (Freon 23) to the purge gas was also investigated.

Keywords: *Electrothermal atomic absorption spectrometry; dysprosium; serum; modifier; Freon; tantalum foil; tungsten foil*

Electrothermal atomic absorption spectrometry (ETAAS) is a well established technique for the determination of trace elements in biological materials. In the field of occupational and environmental health, about 50 elements have so far been determined by ETAAS in biological materials such as body fluids and tissues, food and related samples.[1] Many of the elements are determined on a routine basis in clinical and biochemical laboratories. However, among the lanthanides, only gadolinium[2] and lanthanum[3] have so far, to our knowledge, been determined in biological samples by ETAAS. This is probably because: the lanthanides have not been of special importance in biological samples; the endogenous levels are very low, *i.e.*, ultratrace levels; and the lanthanides are not among the best suited for ETAAS because of memory effects and low sensitivity.

Owing to the introduction of magnetic resonance imaging (MRI) contrast agents, based on complexes of gadolinium and dysprosium, these elements have become elements of clinical interest[4] and sensitive analytical methods are required in order to investigate their toxicity. An ETAAS method has been developed for the determination of gadolinium in biological samples, suitable for studying the pharmacokinetics and biodistribution of gadolinium in rats by Liang *et al.*[2] who atomized the sample from a tantalum boat after solvent extraction. The characteristic mass and detection limit found were 1000 pg (integrated absorbance) and 2060 pg (2s), respectively.

Inductively coupled plasma atomic emission spectrometry (ICP-AES) has been used to determine elevated levels of dysprosium in monkey serum after the administration of Sprodiamde Injection, an MRI contrast medium based on dysprosiumdiethylenetriaminepentaacetic acid bis(methylamide) (DyDTPA-BMA).[5] In this study the measured dysprosium concentration ranged from 2920 to 4 μg ml^{-1}. It is desirable to be able to measure lower concentrations of dysprosium than could be measured by ICP-AES. Ideally, dysprosium should be measured down to the endogenous concentrations. The purpose of this study was to develop a sensitive and simple ETAAS method for the determination of dysprosium in serum, suitable for studying the biodistribution and kinetics of dysprosium in human or animal bodies exposed to dysprosium MRI contrast agents such as DyDTPA-BMA. Dysprosium has previously been determined by ETAAS in inorganic materials such as rocks, minerals and metals,[6–10] and some fundamental or sensitivity studies have been performed.[11–15] Increased sensitivity and reduced memory effect have been reported for dysprosium atomized from a tantalum lined graphite tube[9,12] or from a tantalum boat or platform inserted into the graphite tube.[7,14] In these atomizers there is no physical contact between the sample and the graphite surface, and the formation of stable dysprosium carbides are prevented. Characteristic masses of 2.8 pg (peak height) and 6.5 pg (integrated absorbance) were obtained when dysprosium was atomized from a tantalum platform.[14] Using pyrolytically coated tubes a characteristic mass as low as 8.8 pg (integrated absorbance) has been reported.[6] Another approach, to prevent physical contact between the sample and the graphite tube, that has been investigated for several elements forming refractory carbides, is to pre-coat the graphite tube to form a metal carbide layer on the graphite surface.[16–22] Another possibility of avoiding carbide formation could be to make the element more volatile by adding a halogenating agent. Welz and Schlemmer[23] have tried this approach for ETAAS by adding 1% CHF_3 (Freon 23) to the purge gas. They experienced a signal depression for titanium when Freon 23 was added during the atomization step and reduced memory effect for molybdenum when Freon was added during the cleaning step.

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For organic samples, physical contact between the analyte and carbon cannot be totally eliminated as the sample itself contains carbon.

In this paper the intention was to find a suitable chemical modifier that could be used to increase the sensitivity and reduce the memory effect for dysprosium. In the development and validation of the method, animal reference serum, Seronorm, spiked with DyDTPA-BMA was used as a dose substitute of real samples.

EXPERIMENTAL

Instruments and Equipment

The ETAAS measurements were made with a Perkin-Elmer (PE) Model 5000 Zeeman spectrometer equipped with an HGA-400 graphite furnace, an AS-40 autosampler and a PE Intensitron dysprosium hollow cathode lamp. Both the absorption signal corrected for background and the background signal were collected and displayed with the PE 3600 data station and an Anadex colour scribe printer. The graphite tubes were pyrolytically coated from Applied Optics AB, Sweden. Argon was used as the purge gas. Some experiments were carried out with Freon 23 added to the purge gas. Argon was of 99.99% purity from AGA, Oslo, Norway and Freon 23 was technical quality from Hydrogas, Oslo, Norway. The HGA-400 graphite furnace programmer is not equipped for an alternate gas, and the introduction of Freon was manually controlled. Argon and Freon supplies were connected *via* a T-coupling, and a digital flowmeter (Bronkhorst High Tec B.V. Model E, Holland) was used for measuring the Freon gas flow.

Tantalum foils (0.25 and 0.025 mm) of purity 99.9 and 99.995%, tungsten foils of purity 99.5% and 0.5 mm wolfram wires were delivered by Johnson Matthey. The dysprosium hollow cathode lamp current was set to 30 mA, and measurements were performed at the 421.2 nm wavelength using a slit-width of 0.2 nm, low.

Reagents and Solutions

DyDTPA-BMA, molecular formula $C_{16}H_{28}DyN_5O_9 \cdot xH_2O$[4] and Seronorm were delivered by Nycomed, Norway. Element solutions of Dy (2.5% HNO_3), Gd (2.5% HCl), La (2.5% HNO_3), Eu (2.5% HNO_3), Ho (2.5% HNO_3), Lu (2.5% HNO_3), Rh (4.9% HCl), Th (2.5% HNO_3), Tm (2.5% HNO_3), Yb (2.5% HCl) and W (5.0% HF) were prepared by diluting 1000 μg ml^{-1} Spectrascan standard solutions from Teknolab AS, Norway. The acid concentrations (m/v) of the final solutions are given in brackets. For higher concentrations of Gd and La, 10 000 μg ml^{-1} standard solutions from Johnson Matthey were used. The Pd solutions were prepared from Spectrascan 1% (m/v) Pd matrix modifier solutions from Teknolab AS, Norway.

Dysprosium standard solutions in the concentration range from 10 to 400 μg ml^{-1}, in 0.1% m/v HNO_3, were prepared by diluting 1000 μg ml^{-1} Spectrascan standard solutions from Teknolab AS, Norway. A 200 μg ml^{-1} dysprosium stock solution was made from solid DyDTPA-BMA. This solution was diluted further and used for spiking of the reconstituted Seronorm.

Seronorm samples with dysprosium concentrations in the range from 10 to 400 ng ml^{-1}, in 0.1% m/v HNO_3 and 0.1% m/v Triton X-100, were prepared. The lyophilized reference serum was first reconstituted by adding 5.00 ml of water. DyDTPA-BMA solutions (100 μl) of different concentrations were then added to 900 μl of reconstituted Seronorm to get serum samples in the concentration range from 10 to 400 ng ml^{-1} dysprosium. Blanks was also prepared, containing all the components except DyDTPA-BMA.

All dilutions were done with de-ionized water. Acids used were of *pro analysis* quality, Merck.

For measuring, the furnace programme 1A or 1B (Table 1) was used.

RESULTS AND DISCUSSION

Comparing the Dysprosium Signal from Standard and Seronorm Solutions

Typical atomization signals for 10 μl of 200 ng ml^{-1} dysprosium, as metal standard solution and as DyDTPA-BMA in Seronorm are shown in Fig. 1. The organic matrix did not have any significant effect on the dysprosium signal, except for a slightly higher memory effect. The explanation may be that carbon from the organic matrix vaporizes at a temperature lower than the temperature demanded for dysprosium carbide formation.

The reduction of the atomization signal after 4 s (Fig. 1) is caused by the introduction of 300 ml min^{-1} of argon after 4 s of gas stop. The use of argon in the last second of atomization and in the clean step reduces the carryover signal, because it more effectively removes dysprosium from the tube and thereby prevents recondensation.

Tube Lining with Tantalum and Tungsten Foils

In the present work, the use of tubes lined with metal foils for the determination of dysprosium was tried out, with no success. Several different ways of lining were tested using 0.25 and 0.025 mm tantalum foil and 0.05 mm tungsten foil. The procedure described by Ma *et al.*[24] of lining the tube with 0.025 mm

Table 1 Temperature–time programmes for dysprosium

Programme 1A

Step	1	2	3	4	5*	6
Temperature/°C	70	100	1500	2700	2700	2700
Ramp/s	5	20	25	0	0	0
Hold/s	10	10	10	4	1	3
Read				read	read	
Internal flow/ml min^{-1}	300	300	300	0	300	300

* Step 5 was used during the method development to see the effect on the signal when 300 ml min^{-1} argon was introduced after gas stop. In routine analyses read can be omitted and hold time extended to 4 s in step 5. Step 6 can then be omitted.

Programme 1B

The same as 1A, except that the temperatures in step 4 and 5 were set to 2800 °C. Programme 1B was used in the first part of the study. To extend the lifetime of the graphite tubes, programme 1A, with reduced temperature, was chosen for further work.

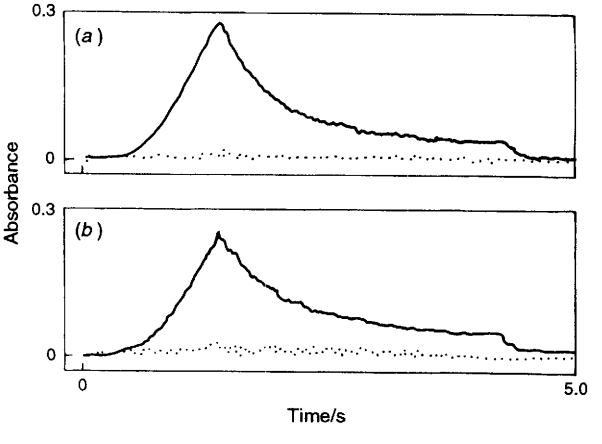

Fig. 1 Absorbance profiles at the 421.2 nm dysprosium wavelength of (*a*) 10 μl of 200 ng ml^{-1} dysprosium standard solution and (*b*) 10 μl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm. The dotted line is the background signal. The furnace programme 1A (Table 1) was used

tantalum foil held in place by a tungsten spiral was also tried. Our experience was that the foils deformed or cracked after only a few atomizations, in addition the sensitivity and the memory effect for dysprosium was not improved. This approach was not further investigated.

Effects of Adding Freon to the Argon Purge Gas

Some experiments, using Freon 23 as an alternative gas, were performed. By adding Freon to the purge gas during the pyrolysis or atomization step the sensitivity of dysprosium was reduced; when 0.2% v/v Freon was added to argon during the entire furnace programme, the dysprosium signal disappeared completely. Volatile dysprosium fluorides are probably formed, but are carried out of the furnace as molecules without being dissociated. However, as is shown in Fig. 2(*b*), when 2% v/v Freon was added to argon in the cleaning step or at the end of the atomization step (step 5 and 6 in furnace programme 1B, Table 1) the memory effect was reduced. Increased concentration of Freon in the cleaning step, see Fig. 2(*c*), resulted in decreased sensitivity of the following atomization signal.

These results are similar to the results obtained, by Welz and Schlemmer.[23]

Investigation of Possible Chemical Modifiers

The following elements were tested as potential modifiers: lanthanum, europium, gadolinium, holmium, thulium, ytterbium, lutetium, thorium, tungsten, rhodium and palladium. The effect of adding 10 μl of 10 100 and 1000 μg ml^{-1} of the carbide forming elements on the absorbance signal of 10 μl of 200 ng ml^{-1} dysprosium standard solution was observed. For rhodium and palladium, 10 μl of 1% m/v was added.

Rhodium was found to give a spectral interference at the 421.172 dysprosium line and was, therefore, unsuitable as a modifier for dysprosium. Europium, thorium, tungsten and palladium had no effect on the dysprosium signal. For holmium, thulium, ytterbium and lutetium, minor enhancement of the signal was observed.

Of all the elements investigated, lanthanum and gadolinium gave the most obvious positive effects on the sensitivity for dysprosium, and these elements were, therefore, further investigated as possible chemical modifiers.

An increased sensitivity for dysprosium in the presence of gadolinium and lanthanum, among several other elements, has previously been reported.[7,13]

Lanthanum Added as Modifier

The effect of adding 10 μl of various concentrations of lanthanum on the sensitivity of 200 ng ml^{-1} dysprosium as pure standard solution and as DyDTPA-BMA added to Seronorm is shown in Fig. 3. The effect of the modifier was the same for the dysprosium standard solutions and the Seronorm solution. The optimal concentration of modifier was in the range 100–1000 μg ml^{-1} of lanthanum. Pyrolysis and atomization curves for dysprosium as DyDTPA-BMA with added 10 μl of 500 μg ml^{-1} lanthanum as modifier are shown in Fig. 4(*b*)

Comparing these curves with the corresponding curves without modifier [Fig. 4(*a*)] shows that adding lanthanum does effect the volatility of dysprosium. Without any modifier dysprosium can be heated to about 2300 °C, which is also the appearance temperature of the element, without any loss of sensitivity. With lanthanum added, dysprosium is lost at about 1900 °C and the appearance temperature is lowered to about 2100 °C. Typical signals for dysprosium without modifier and with lanthanum added as modifier (see Fig. 5) show the earlier appearance and the higher sensitivity when lanthanum is added.

A problem associated with adding large amounts of lanthanum was a residue build-up in the graphite tube that had to be physically removed after about 20 atomizations.

Gadolinium Added as Modifier

The effect of adding 10 μl of various concentrations of gadolinium on the sensitivity of 200 ng ml^{-1} dysprosium as the dysprosium standard solutions and as DyDTPA-BMA added to Seronorm is shown in Fig. 6. The effect on the sensitivity was the same for the dysprosium standard solutions and the Seronorm solution. The optimal concentration of modifier was in the range 500–1000 μg ml^{-1} of gadolinium. Pyrolysis and atomization curves for dysprosium in Seronorm with added 10 μl of 1000 μg ml^{-1} gadolinium as modifier are shown in Fig. 7.

Comparing these curves with the corresponding curves without modifier [Fig. 4(*a*)] shows that adding gadolinium does effect the volatility of dysprosium. With gadolinium added as a modifier, dysprosium is lost at about 1700 °C and the

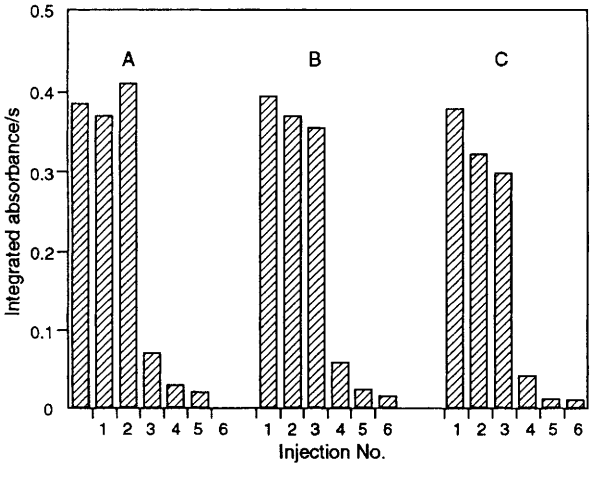

Fig. 2 Memory effect of dysprosium in the graphite tube with A, pure argon as purge gas; B, 2%; and C, 6% Freon 23 in argon as purge gas in the clean steps (step 5 and 6). Three replicate injections of 10 μl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm (1–3) were run, followed by three injections of the blank (4–6). The furnace programme 1B (Table 1) was used

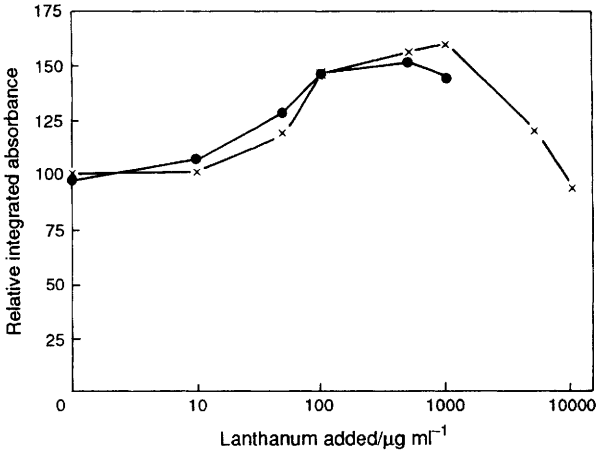

Fig. 3 The relative effect on the integrated dysprosium signal (% peak area) of adding 10 μl lanthanum to 10 μl of 200 ng ml^{-1} dysprosium standard solution (×) and 10 μl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm (●). All the solutions were 5% m/v HNO_3. The furnace programme 1B (Table 1) was used. Three injections were run at each point

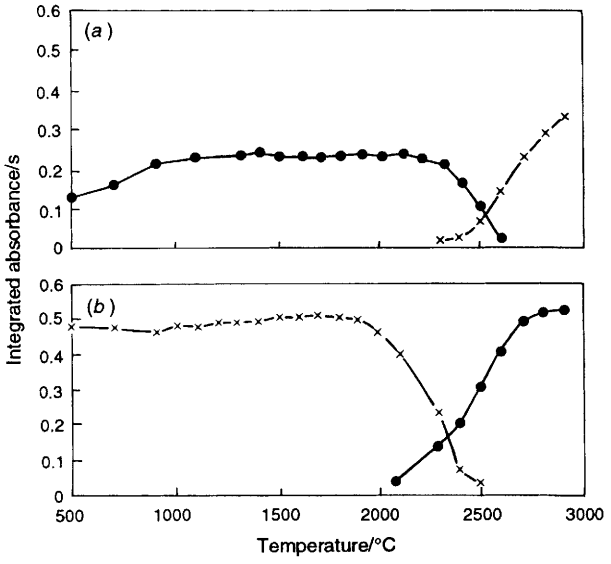

Fig. 4 Pyrolysis and atomization curves for (*a*) 10 µl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm and (*b*) 10 µl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm with 10 µl of 500 µg ml^{-1} lanthanum. The pyrolysis temperature was varied from 500 to 2600 °C with constant a atomization temperature of 2700 °C. The atomization temperature was varied from 2100 to 2900 °C with a constant pyrolysis temperature of 1500 °C. The furnace programme 1A (Table 1) was used. Three injections were run at each temperature

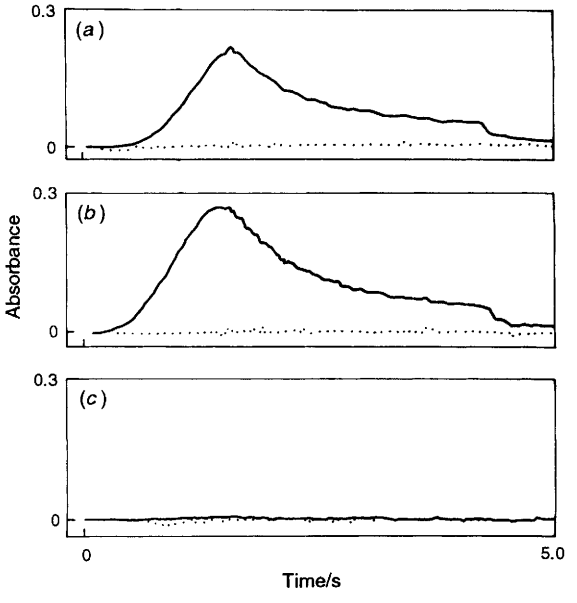

Fig. 5 Absorbance profiles at the 421.2 nm dysprosium wavelength of (*a*) 10 µl of 200 ng ml^{-1} dysprosium standard solution (*b*) 10 µl of 200 ng ml^{-1} dysprosium standard solution with 10 µl of 500 µg ml^{-1} lanthanum and (*c*) 10 µl of 500 µg ml^{-1} lanthanum. The dotted line is the background signal. The furnace programme 1A (Table 1) was used

appearance temperature is lowered to about 2100 °C. Typical signals for dysprosium without modifier and with gadolinium added as modifier, (Fig. 8) show the earlier appearance and the higher sensitivity when gadolinium is added. Contrary to lanthanum, there was no problem with residue build-up of the modifier in the graphite tube. Gadolinium was therefore chosen as the most suitable modifier of the two.

The modifier also had a favourable influence on the memory effect. The % carryover when a blank was atomized after three atomization of 10 µl of 200 ng ml^{-1} dysprosium was reduced from about 15–20% to typically 6% when gadolinium was added and the furnace programme 1B (Table 1) was used. This

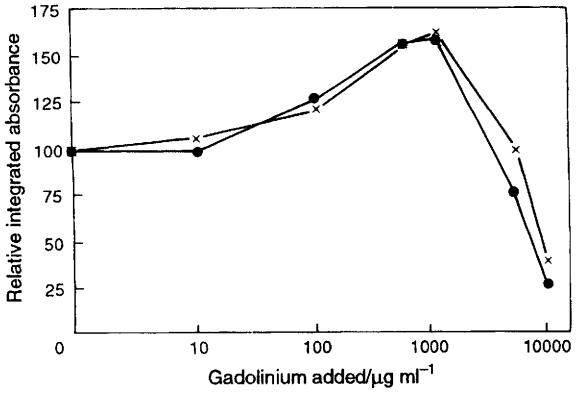

Fig. 6 The relative effect on the integrated dysprosium signal (peak area) of adding 10 µl gadolinium to 10 µl of 200 ng ml^{-1} dysprosium standard solution (×) and 10 µl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm (●). All the solutions were 5% m/v HCl. The furnace programme 1A (Table 1) was used. Three injections were run at each point

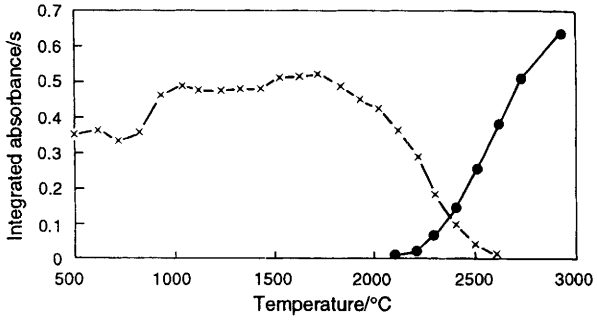

Fig. 7 Pyrolysis and atomization curves for 10 µl of 200 ng ml^{-1} dysprosium (as DyDTPA-BMA) in Seronorm with added 10 µl of 1000 µg ml^{-1} gadolinium. The pyrolysis temperature was varied from 500 to 2600 °C with a constant atomization temperature of 2600 °C. The atomization temperature was varied from 2100 to 2900 °C with a constant pyrolysis temperature of 1500 °C. The furnace programme 1A (Table 1) was used

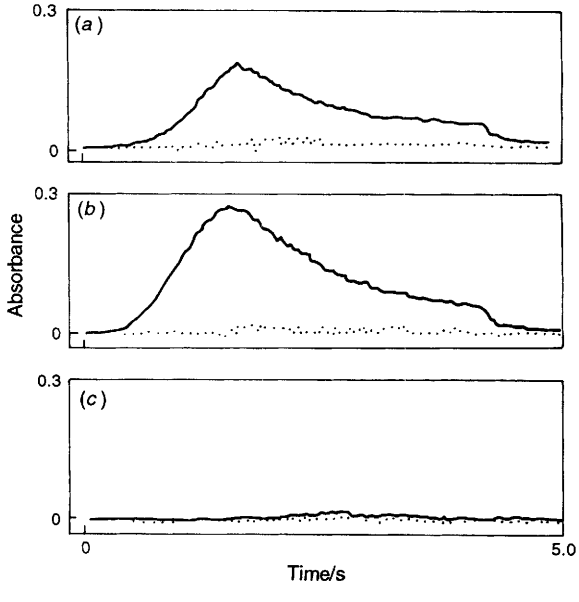

Fig. 8 Absorbance profiles at the 421.2 nm dysprosium line of (*a*) 10 µl of 200 ng ml^{-1} dysprosium standard solution (*b*) 10 µl of 200 ng ml^{-1} dysprosium standard solution with added 10 µl of 1000 µg ml^{-1} gadolinium and (*c*) 10 µl of 500 µg ml^{-1} gadolinium. The dotted line is the background signal. The furnace programme 1A (Table 1) was used

Table 2 Effect of various volumes of gadolinium added, on the integrated absorbance signal for 10 μl of 200 ng ml^{-1} dysprosium standard solution (in 2.5% m/v HCl): average of three injections; furnace programme 1B (Table 1) was used

Volume of Gd added/μl	Concentration of Gd/μg ml^{-1}	Relative Dy signal (% area)	RSD (%)
0	—	100	4
10	1000	154	4
20	500	154	2
50	200	156	5

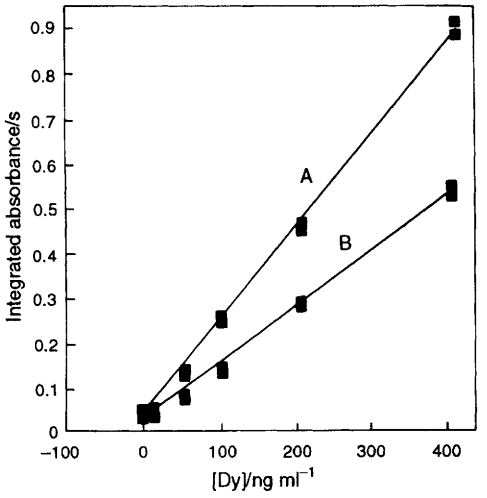

Fig. 9 Linearity of dysprosium: A, with 10 μl of 1000 μg ml^{-1} gadolinium added as modifier ($y = 0.045 + 0.0021x$ and $r = 0.999\,89$); and B, without modifier ($y = 0.033 + 0.0013x$ and $r = 0.999\,75$). The furnace programme 1A (Table 1) was used. Three injections were run at each point

is lower than the result obtained by using Freon 23 in the cleaning step without modifier [Fig. 2(*a*)].

The effect of volume and order of injection of modifier was tested. As shown in Table 2, no significant effect of volume was found. The order of injection of gadolinium and sample was of no importance. Adding modifier after the sample was chosen because it was more practical with the use of an autosampler.

Validation

For comparison, the method was validated with and without the use of modifier. The accuracy and repeatability of the method after three days using gadolinium as modifier, are given in Table 3. There was no significant difference in the accuracy of the method with and without the use of modifier; the recoveries (%) did not differ significantly from 100% at any concentration level. The repeatability and reproducibility improved with the use of gadolinium as a modifier, particularly at the lowest concentration. The reproducibility of the method, given as the overall % RSD of the results obtained after the three days, ranged from 2.3 to 5.8% with the use of modifier and 3.8 to 11.2% without modifier. The method was linear in the concentration range tested. The standard curves and corresponding linear regression equations are given in Fig. 9. With the use of modifier the characteristic mass was reduced from 35 to 18 pg and the limit of detection (3 *s*, $n = 6$) improved from 16 to 11 pg.

With the proposed method, the pyrolytically coated graphite tubes are expected to give good analytical performances for about 150–200 atomizations when no modifier is used, and about 100 atomizations when gadolinium is added as a modifier.

CONCLUSIONS

A convenient analytical method has been developed for the determination of elevated levels of dysprosium in serum in the concentration range 1–400 ng ml^{-1}. For higher concentrations, the samples should be diluted to get within this concentration range. Although ICP-AES[5] and ETAAS can be used, for concentrations lower than about 0.1–1 μg ml^{-1} or when only small amount of sample is available, ETAAS is probably the best method of choice.

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Table 3 % Recovery, repeatability and reproducibility for dysprosium (as DyDTPA-BMA) in Seronorm. % Recovery and repeatability were found after 3 d with three injections of each concentration each day. All the data from the three days are used to calculate the total % recovery and the reproducibility: furnace programme 1B (Table 1) was used; 10 μl of 1000 μg ml^{-1} gadolinium was added as modifier; and injection volumes, 10 μl

	Day 1 ($n = 3$)		Day 2 ($n = 3$)		Day 3 ($n = 3$)		Total ($n = 9$)	
Dysprosium/ng ml^{-1}	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)	Recovery (%)	RSD (%)
10	100	2.5	103	2.6	102	1.8	102	5.8
50	103	2.2	104	1.6	101	2.1	103	2.8
100	98	0.3	95	2.3	100	1.7	98	2.7
200	98	0.7	102	2.1	100	2.2	100	2.3
400	96	1.0	100	1.7	96	2.3	97	3.2