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Autor: West, Fanny B. / Ottaway, John M. / Rowston, William B.

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The Effect of Tantalum Coated Tubes in Graphite Furnace Atomic Absorption Spectrophotometry: A Short Communication

Fanny B. West, John M. Ottaway and William B. Rowston

Matrix effects have hampered significantly the determination of trace metals by CFAAS in biological and environmental samples. As a result many authors have examined ways to minimise the effects of interferents present in the matrix, most of which involve the use of matrix modification.

More recently, there has been a growing interest in the development of a pretreatment process of the carbon tube. Already, since the inception of the technique, pyrolytically coated furnaces have been advocated. These, however, have had a major disadvantage in that the coating is progressively removed from the surface during use. Several workers have altered the characteristic of the graphite tubes by pretreatment with carbide forming salts, such as lanthanum, zirconium, molybdenum. A coating of tantalum carbide for graphite atomisers has also been proposed by several authors in an attempt to increase the sensitivity of many elements forming low volatility carbides. We have recently investigated the use of tantalum coated tubes as a means of alleviating matrix interferences.

The lining of the furnace with a suitable carbide forming element can improve the detection limits and reduce the interference effects of some matrix components. The coating prevents physical contact between the carbon of the furnace and subsequent samples, precluding further carbide formation by elements in the sample. Tantalum carbide was chosen because of its reputed outstanding characteristics. It has a high melting point ($3400^{\circ}\text{C} \pm 50^{\circ}\text{C}$ in the presence of graphite) and low chemical reactivity (of all the refractory carbides, tantalum carbide is the most stable toward acids). As a result these tubes have exhibited increased sensitivity, greater lifetimes, improved reproducibility of the response, and a reduction in chloride interferences. Here is a brief look at the

lifetimes and reproducibility of these tubes. These tubes have been found to have much longer life times. Zatka (1) has claimed that the tubes survived 350–400 firings with continued high precision on replicate analysis even at high temperatures of 2700°C . My tubes also averaged a lifetime of about 300 firings. One colleague has had tubes lasting over 1000 injections compared with around 500 for the pyrolytically coated tubes. Work done with Philips tantalum coated tubes have also survived a great many firings even at high temperatures.

The reproducibility of replicate atomisation signals was also looked at. For this, the tubes considered were a) new uncoated and coated tubes, and b) old uncoated and coated tubes (having had more than 150 firings). The results are shown on the following table (table 1). This table shows that reproducibility

Tab. 1. Reproducibility of the Lead Response with a Coated and Uncoated Furnace

Determination Number	Peak height in number of Chart Divisions			
	Uncoated tube		Ta-coated tube	
	Old	New	Old	New
1	51	28	66	38
2	55	29.5	64	38
3	56	29.5	67	37
4	53	29	67	37
5	54	29.5	66	38
6	50	29	65	38
7	49	32	66	39
8	55	27	66	38
9	57	31	65	39
10	58	31	67	39
11	55	30	67	39
12	51	31	66	38
13	54	30	66	40
14	58	34.5	65	38
15	60	31	65	40
\bar{x}	54.4	30.13	65.87	38.4
Std. deviation	3.18	1.77	0.92	0.91
RSD	9.9%	5.9%	1.4%	2.4%

ty of the lead signal is the same in both a new and old uncoated furnace. The reproducibility in the treated furnace is better than in the untreated furnace and the results are somewhat better in an older furnace. It was observed throughout the work that

newly coated tubes needed a few firings before the signal became stable and the firing could be reproducible.

Interferences: The interference of chloride and other anions on the determination of lead is a widespread analytical problem and

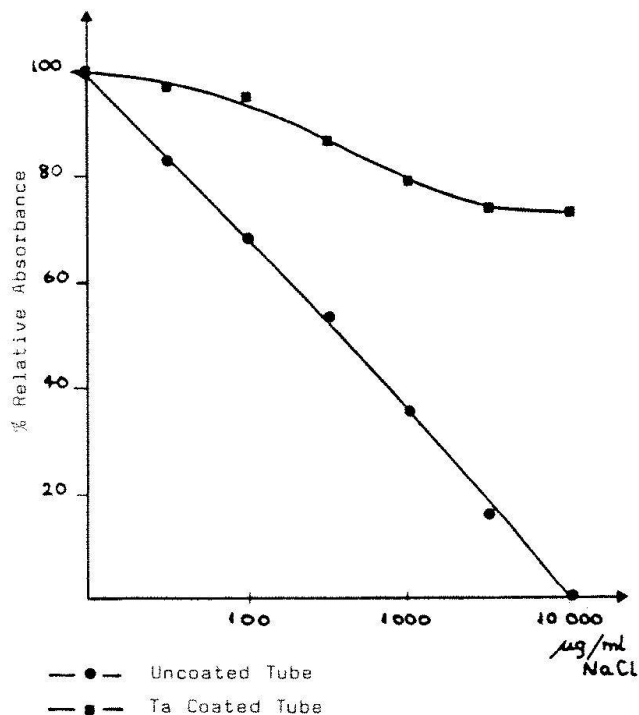


Fig. 1. Effect of NaCl on 0.5 µg/ml Pb as PbCl₂ in 0.1M HCl.

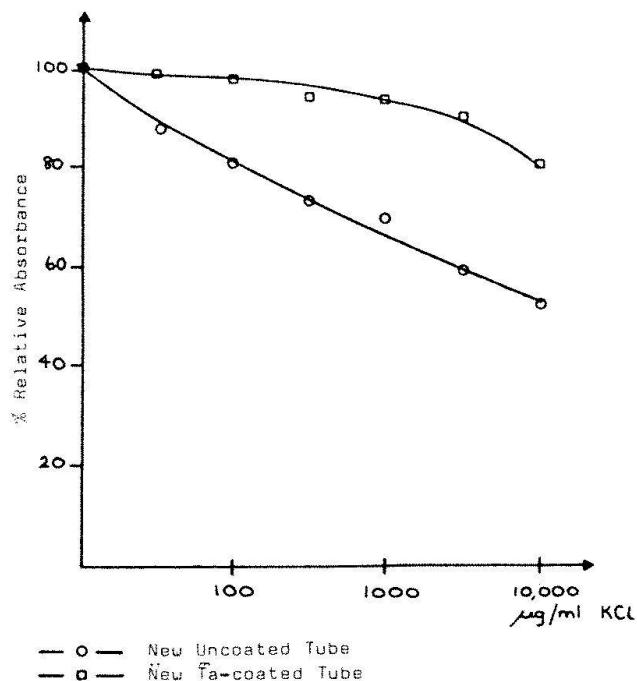


Fig. 3. Effect of KCl on 0.5 µg/ml Pb as PbCl₂ in 0.1M HCl.

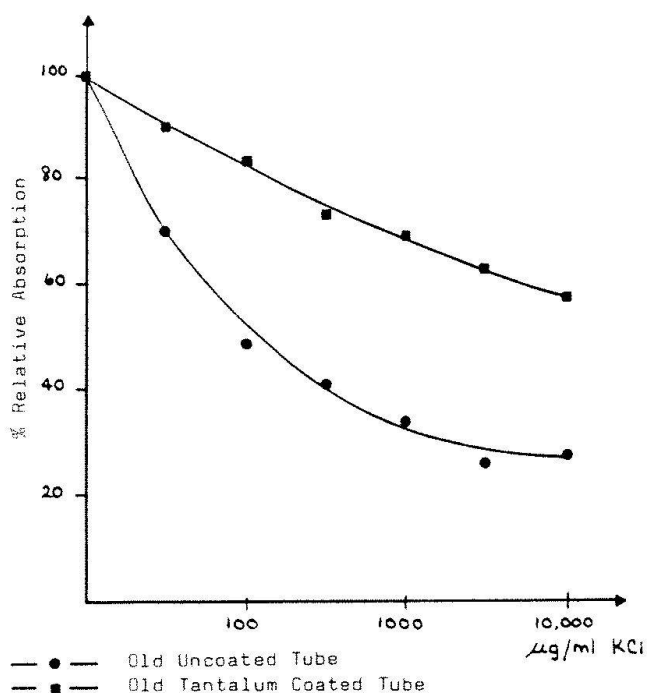


Fig. 2. Effect of KCl on 0.5 µg/ml Pb as PbCl₂ in 0.1M HCl.

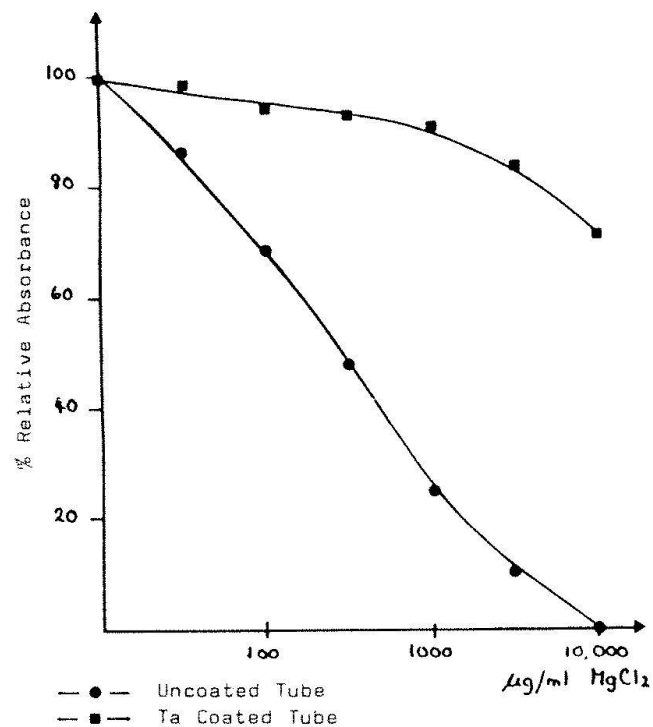


Fig. 4. Effect of MgCl₂ on 0.5 µg/ml Pb as PbCl₂ in 0.1M HCl.

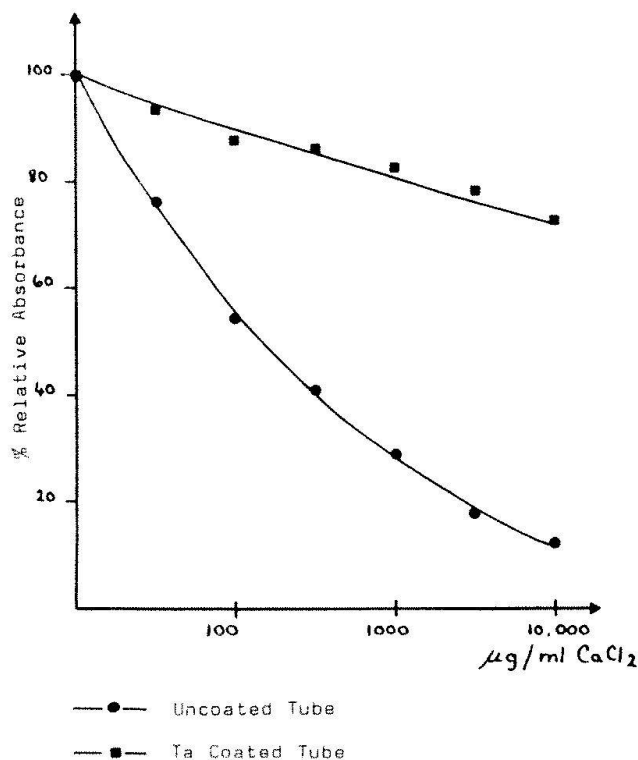


Fig. 5. Effect of CaCl_2 on $0.5 \mu\text{g/ml Pb}$ as PbCl_2 in 0.1M HCl .

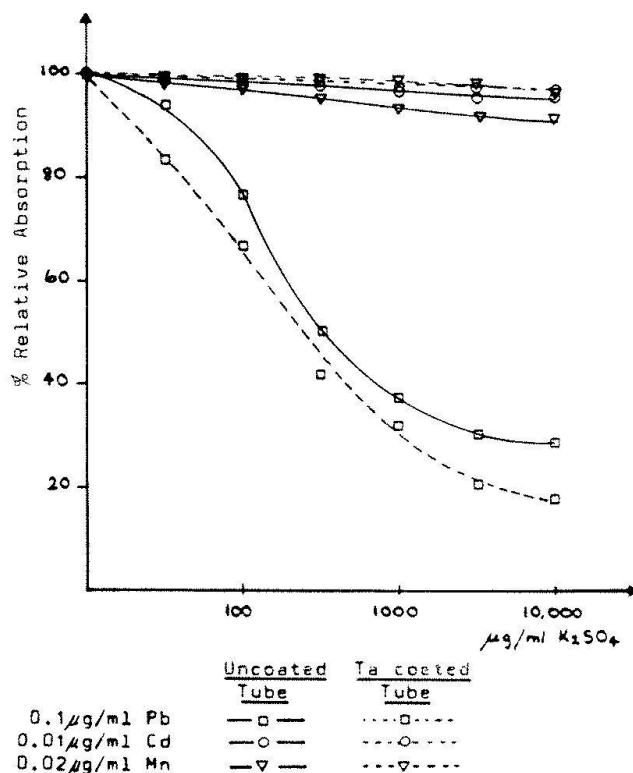


Fig. 6. Effect of K_2SO_4 on the Absorption Signal of Lead, Cadmium, Manganese.

has been the subject of many papers. The suppression of the Pb signal by NaCl has been attributed mainly to the large background molecular absorption signal of sodium which cannot be compensated for by the background correction system actually in use. The suppressive effect of MgCl_2 has been attributed to a chemical effect (vapour phase interference).

We studied the effect of increasing the concentration of chlorides in the sample matrix on the lead absorption peak. On Figures 1 and 2 we see the effect of various amounts of sodium and potassium chloride on the determination of lead in the 2 types of tube. A severe depression of the absorption signal for lead can be seen in a normal uncoated tube while in a tantalum coated tube the interference effect is reduced if not completely eliminated. These determinations were done using tubes having had more than 50 firings. The analysis with KCl as the interferent was repeated but this time using 2 new graphite tubes and the results can be seen in the Figure 3. The absorption signal for Pb is now increased in both tubes, although in the tantalum coated tube, the

improvement is more pronounced. The effect of Mg and CaCl_2 is shown in Figures 4 and 5. Once again the improvement in the absorption signal for lead is more pronounced when the determination is done using a Ta-coated tube.

These are the results of a systematic study of the problem. When it comes to real sample matrix, the interference problems are often more complex. One of the difficult determination is that of lead in sea water, particularly due to the presence of large amounts of salts in the matrix. As a result the lead must first be preconcentrated and separated from the interfering components. M.C. Halliday et al. (2) has combined the use of a matrix modifier and a tantalum tube in the direct determination of lead in polluted sea water. Both NaCl and MgCl_2 suppressed strongly the signal in pyrolytically coated tube. The addition of ammonium nitrate (15%) was found to relieve completely the suppressive effect of MgCl_2 , even enhancing the signal at certain concentrations. In Ta-coated tubes the interference effects of MgCl_2 was minimal at normal sea water concentrations, even in the absence of ammonium nitrate.

Other anions, also reported problematic in the literature were also investigated. We looked at the effect of nitrates, phosphates and sulphates in both types of tubes. The results are less promising than with chlorides. We found that there was a small decrease in sensitivity of the signal in the Ta-coated tubes in the presence of nitrates. The interferences were minimal when lead was determined in the presence of Na_2HPO_4 in either the coated or uncoated tubes.

The most pronounced effect on the lead absorption signal in a Ta-coated tube came from sulphate as the interferent anion as can be seen in Figure 6. We see here that the problem is particular to lead, the depressive effect of potassium sulphate being minimal on the other elements, namely cadmium and manganese.

As we have seen briefly, tantalum coated tubes are not the ultimate answers to the analyst's problems. But they do possess advantages which makes them useful especially in matrices where chloride is a predominant interferent. It is also significant that the life time of the tubes is increased 5 fold when using these tubes.

References

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Address of the authors:

Fanny B. West
John M. Ottaway
William B. Rowston
Department of Pure and Applied Chemistry
University of Strathclyde
Glasgow (Scotland)
Great Britain

William B. Rowston
Glasgow College of Technology
Glasgow (Scotland)
Great Britain