AAS
APPLICATION NOTES

The determination of essential metals in vitamin tablets

GBC

Introduction

Levels of essential metals and impurities must be closely monitored in all pharmaceutical preparations. It is therefore necessary to apply an effective dissolution technique to the samples, followed by a quick and simple method of analysis. The following technique provided accurate results. No interferences were found, so aqueous standards were used throughout this work.

The essential metals, manganese, zinc, copper, magnesium and iron, were determined in a sample of premix vitamin powder. The essential ingredients of the vitamin powder are shown in Table 1.

Each kg contains:

Ingredient	Unit	Level
Vitamin A	kiu	2500
Vitamin D3	kiu	300
Vitamin E	mg	10000
Vitamin K3	mg	1000
Vitamin B2	mg	1500
Vitamin B12	mg	10
Niacin	mg	15000
Pantothenic acid	mg	5000
D-Biotin	mg	5
Choline	g	160
Methionine	g	90
Manganese*	g	25
Zinc*	g	50
Copper*	g	50
Magnesium*	g	15
Iron*	g	75
Organic iodide	mg	1200
Thyroxine	mg	200
L-calcium lactate	mg	40000

Table 1: Composition of premix powder (*determined essential elements)

Experimental

Sample preparation

1 g of powder was added to 20 mL concentrated hydrochloric acid and heated for 10 min., avoiding violent boiling. Once cooled the mixture was passed through a Whatman No. 1 (qualitative) filter paper to remove residual solids. The filtrate was then transferred to a 1 litre volumetric flask and diluted to the mark. The concentrations of the essential metals in this solution are shown in Table 2.

Manganese	25 mg/L
Zinc	50 mg/L
Copper	50 mg/L
Magnesium	15 mg/L
Iron	75 mg/L

Table 2: Metal content of stock solution

By appropriate choice of wavelength, manganese, copper and iron were able to be determined without further dilution. However zinc required further dilution by 200 and magnesium by 50. The optimum working ranges for the wavelengths used are shown in Table 3.

Element	Wavelength (nm)	Optimum Working Range (mg/L)	Characteristic Concentration (mg/L)
Manganese	403.1	7 – 27	0.15
Zinc	213.9	0.4 – 1.5	0.008
Copper	249.2	180 – 730	4.0
Magnesium	202.6	5 – 20	1
Iron	385.9	36 – 145	0.8

Table 3: Optimum wavelengths for the determination of manganese, zinc, copper, magnesium and iron

Aqueous calibration standards were prepared from 1000 ppm analytical standards. All were prepared in 2% HCL. Their concentrations are shown in Table 4.

Element	Concentration (mg/L)
Manganese	10, 20, 30
Zinc	0.25, 0.75
Copper	40, 80
Magnesium	0.5, 1
Iron	50, 100

Table 4: Calibration standards

Instrumentation

The GBC atomic absorption spectrophotometer was used for all measurements. This instrument utilizes direct concentration output after internal interpolation from a calibration curve. All analyses were performed with an air-acetylene flame. The operating conditions are shown in Table 5.

Element	Bandpass (nm)	Lamp Current (mA)	Background Correction
Manganese	0.2	5	Off
Zinc	0.5	4.5	On
Copper	0.5	5	On
Magnesium	1.0	10	On
Iron	0.2	6	Off

Table 5: Operating Conditions for the determination of manganese, zinc, copper, magnesium and iron

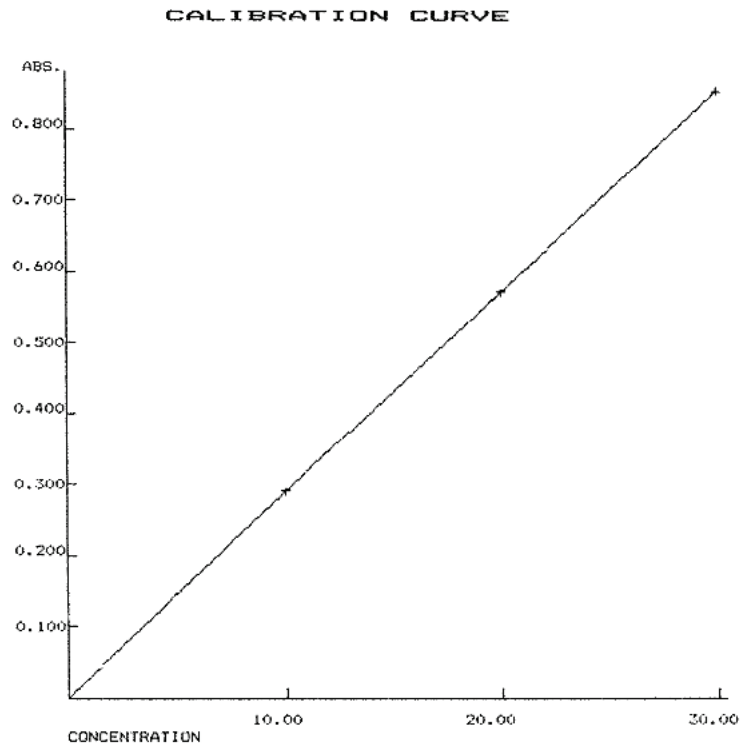
Results

To confirm the absences of interferences, the result obtained from the normal calibration was compared with the result of a standard additions measurement. Figures 1 and 2 show complete outputs of the results of the two methods of measurement for manganese. The measured concentrations of 24.04 from standard additions and 23.76 from the normal calibration are in excellent agreement and confirm that there are no interferences.

Element	Mn
Wavelength	403.1 nm
Slit Width	0.2 nm
Atomization	Air-Acetylene
Lamp Current	5.0 mA
EHT (gain)	– 380 V
Scale Expansion	1.000
Integration Time	3.0 s

Calibration					
Standard/Blank	**Reading 1**	**Reading 2**	**Reading 3**	**Mean**	**RSD (%)**
Blank 1	–0.000	–0.000	0.000	–0.000	–
Standard 1	0.291	0.288	0.290	0.290	0.55
Blank 2	0.000	–0.000	–0.001	–0.000	–
Standard 2	0.568	0.574	0.570	0.571	0.56
Blank 3	–0.001	–0.001	–0.000	–0.001	–
Standard 3	0.852	0.851	0.857	0.853	0.40

Calibration Table		
Standard	**Absorbance**	**Concentration**
1	0.290	10.00
2	0.571	20.00
3	0.854	30.00



Concentration – Sample Number 3	
Readings	**Concentration**
1	23.84
2	23.63
3	23.81
4	23.76
5	23.78
Mean: 23.76 RSD: 0.33%	

Figure 1: Output of manganese results using a normal calibration

Element	Mn
Wavelength	403.1 nm
Slit Width	0.2 nm
Atomization	Air Acetylene
Lamp Current	5.0 mA
EHT (gain)	- 376 V
Scale Expansion	1.000
Integration Time	3.0 s

Standard Additions	
Total Volume (µL)	100.0
Sample Volume (µL)	10.00
No. of Additions	2
Standard Volume (µL)	0.100
Standard Concentration (µL/L)	1000

Calibration					
Standard/Blank	**Reading 1**	**Reading 2**	**Reading 3**	**Mean**	**RSD (%)**
Blank 1	-0.003	-0.002	-0.003	-0.003	-
Sample	0.081	0.081	0.081	0.081	0.48
Blank 2	-0.003	-0.004	-0.005	-0.004	-
Addition 1	0.115	0.114	0.118	0.116	1.60
Blank 3	-0.004	-0.003	-0.003	-0.003	-
Addition 2	0.151	0.151	0.151	0.151	0.17
Sample Concentration: 24.04 Largest Deviation: 0.2%					

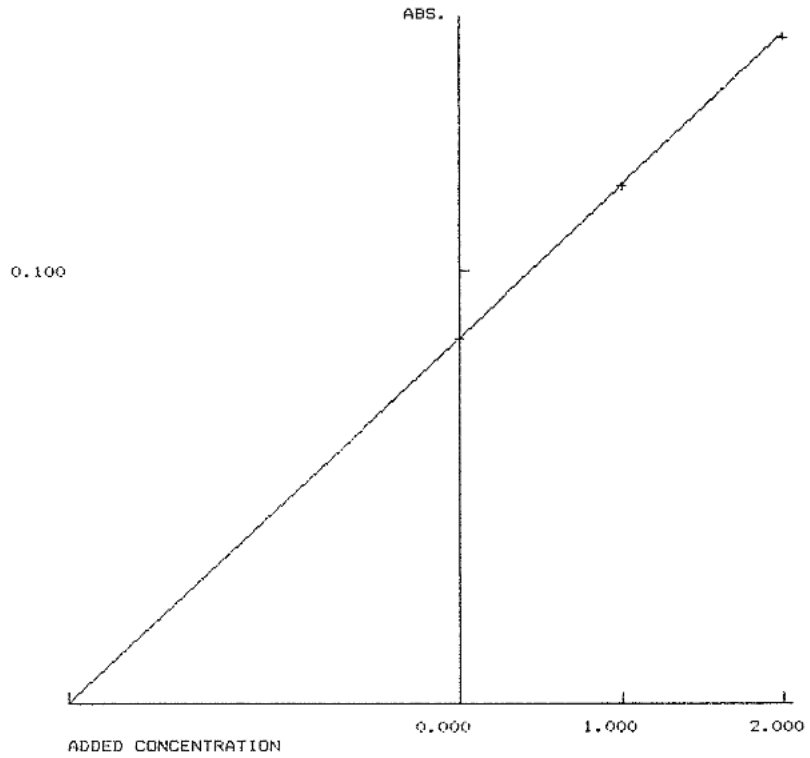


Figure 2: Output of manganese results using the method of standard additions

Table 6 shows the measured concentration for each element compared with the certified value.

Element	Certified Value (mg/L)	Obtained Value (mg/L)	Recovery
Manganese	25	24	96%
Zinc	50	51	102%
Copper	50	50	100%
Magnesium	15	14	95%
Iron	75	74	99%

Table 6: Results for the determination of manganese, zinc, copper, magnesium and iron

Conclusion

The excellent recoveries obtained verified the dissolution method and operating conditions. It should be noted that further dilution for some elements would eliminate the need for the use of alternate lines.

References

1. USEPA Standard Methods of Examination of Water and Wastewater, 1983, Methods 206.2 and 207.2

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