DETERMINATION OF METALS IN ARTISTIC PIGMENTS USING THE OPTIMISED GFAAS METHOD AND RAMAN SPECTROSCOPY

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APPENDIX 1

Fig.1. Sampling locations of paint layer from the painting “Coronation of the Virgin” (1836), by unknown artist, Church of the Shroud of the Holy Virgin in Barič

Fig.2. Painting “Coronation of the Virgin” after conservation and restoration

Experimental conditions, temperature programs and calibration curves

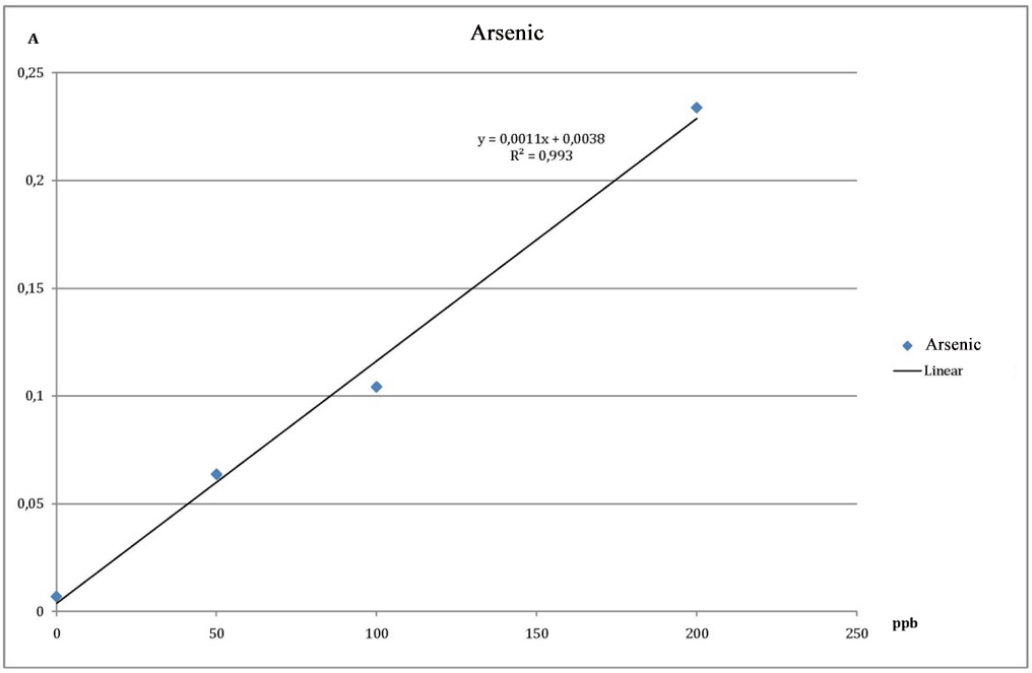

Fig.3. Calibration graph for arsenic

Table 1. Experimental conditions for arsenic

Wavelength (nm)	197.3
Slit (nm)	2.0
Current (mA)	15
Time of reading (s)	5 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	50-200
Modifier	0.2% $NaNO_3$
Volume of the modifier (µl)	5

Table 2. Temperature program for arsenic

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	110	1	10	
3	130	15	30	
4	1500	10	20	
5	2100	1	4	stop flow, read
6	2650	1	3	
7	20	20	20	

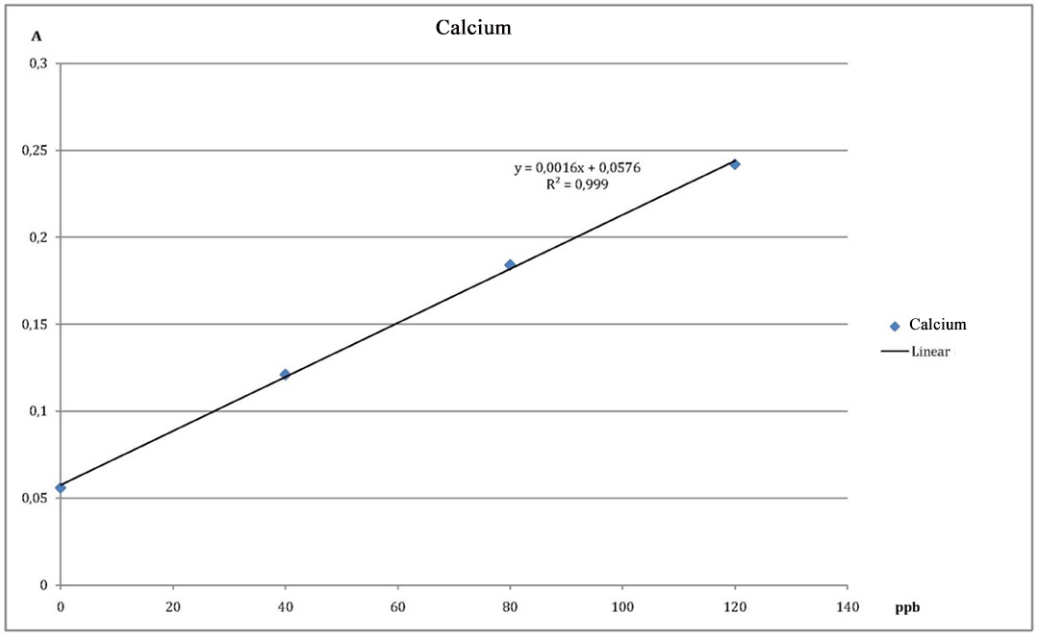

Fig.4. Calibration graph for calcium

Table 3. Experimental conditions for calcium

Wavelength (nm)	422.7
Slit (nm)	0.7
Current (mA)	8
Time of reading (s)	2.5 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	40-120
Modifier	/
Volume of the modifier (µl)	/

Table 4. Temperature program for calcium

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1100	5	10	
4	2600	1	3	min flow, read
5	2650	1	3	
6	20	20	20	

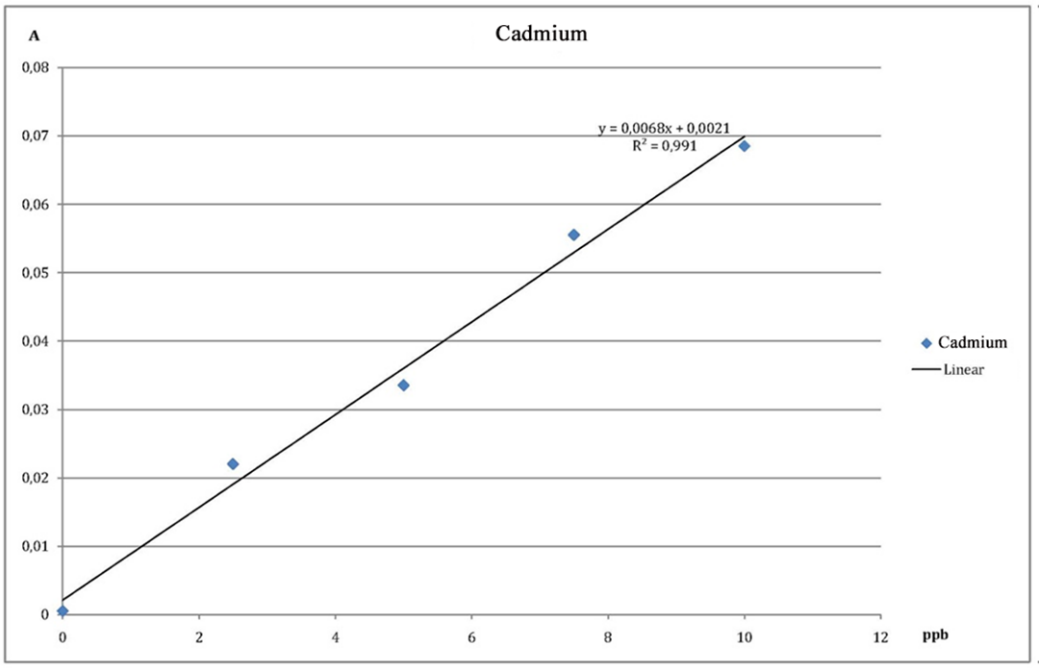

Fig.5. Calibration graph for cadmium

Table 5. Experimental conditions for cadmium

Wavelength (nm)	228.8
Slit (nm)	0.7
Current (mA)	3
Time of reading (s)	3 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	2.5-10.0
Modifier	/
Volume of the modifier (µl)	/

Table 6. Temperature program for cadmium

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	700	5	10	
4	1600	1	3	min flow, read
5	2200	1	3	
6	20	20	20	

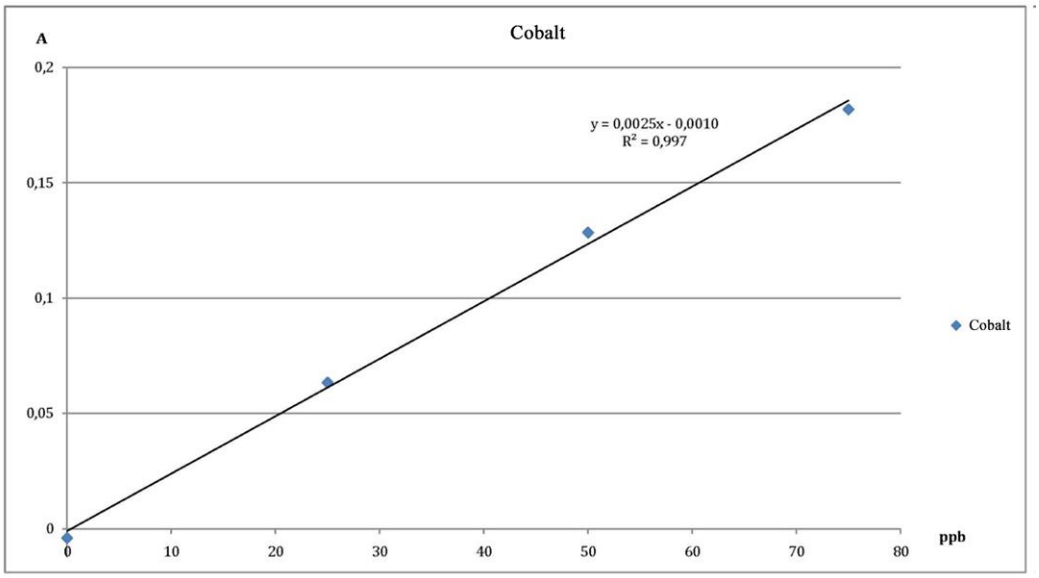

Fig.6. Calibration graph for cobalt

Table 7. Experimental conditions for cobalt

Wavelength (nm)	240.7
Slit (nm)	0.2
Current (mA)	30
Time of reading (s)	2 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	25-75
Modifier	/
Volume of the modifier (µl)	/

Table 8. Temperature program for cobalt

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1400	5	10	
4	2500	1	3	min flow, read
5	2650	1	3	
6	20	20	20	

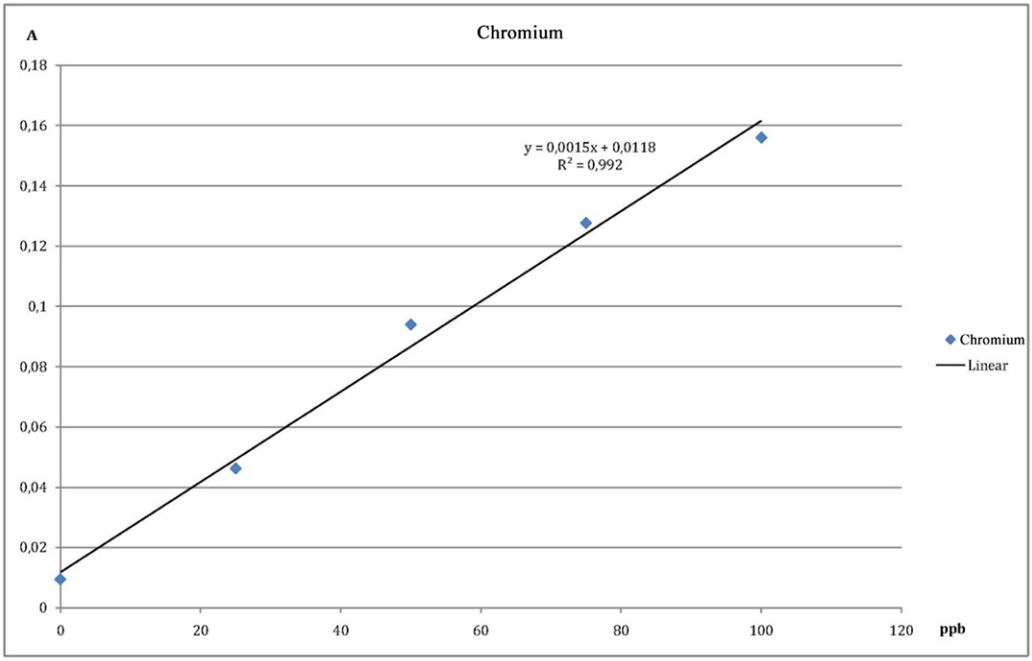

Fig.7. Calibration graph for chromium

Table 9. Experimental conditions

Wavelength (nm)	357.9
Slit (nm)	0.7
Current (mA)	6
Time of reading (s)	2 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	25-100
Modifier	/
Volume of the modifier (µl)	/

Table 10. Temperature program

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1650	5	10	
4	2500	1	3	min flow, read
5	2650	1	3	
6	20	20	20	

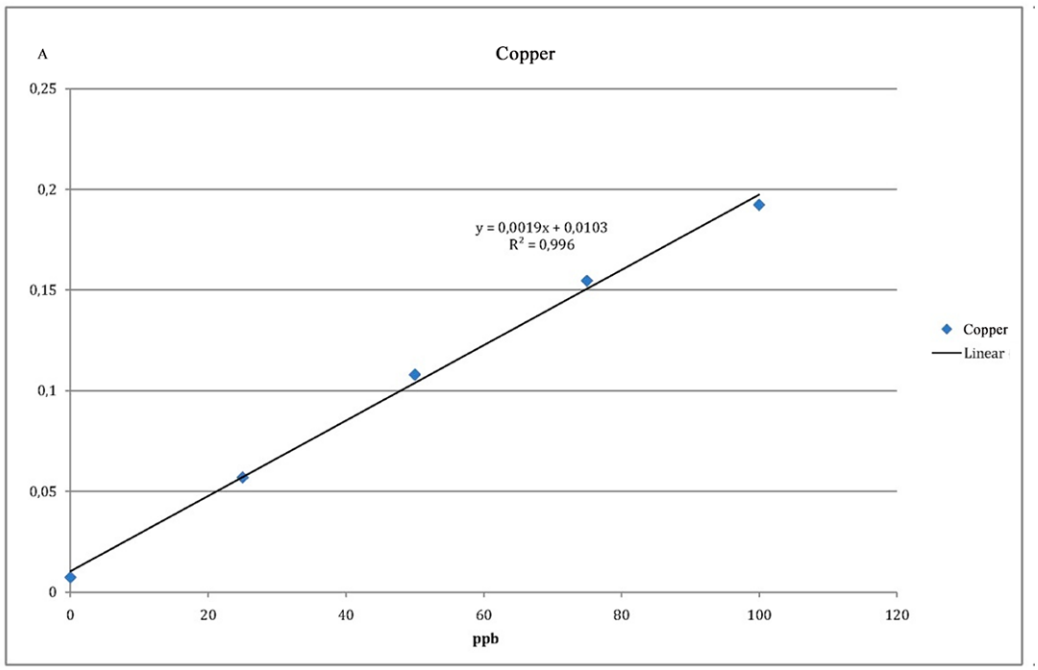

Fig.8. Calibration graph for copper

Table 11. Experimental conditions for copper

Wavelength (nm)	324.8
Slit (nm)	0.7
Current (mA)	15
Time of reading (s)	2 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	25-100
Modifier	/
Volume of the modifier (µl)	/

Table 12. Temperature program for copper

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1200	5	10	
4	2300	1	3	min flow, read
5	2600	1	3	
6	20	20	20	

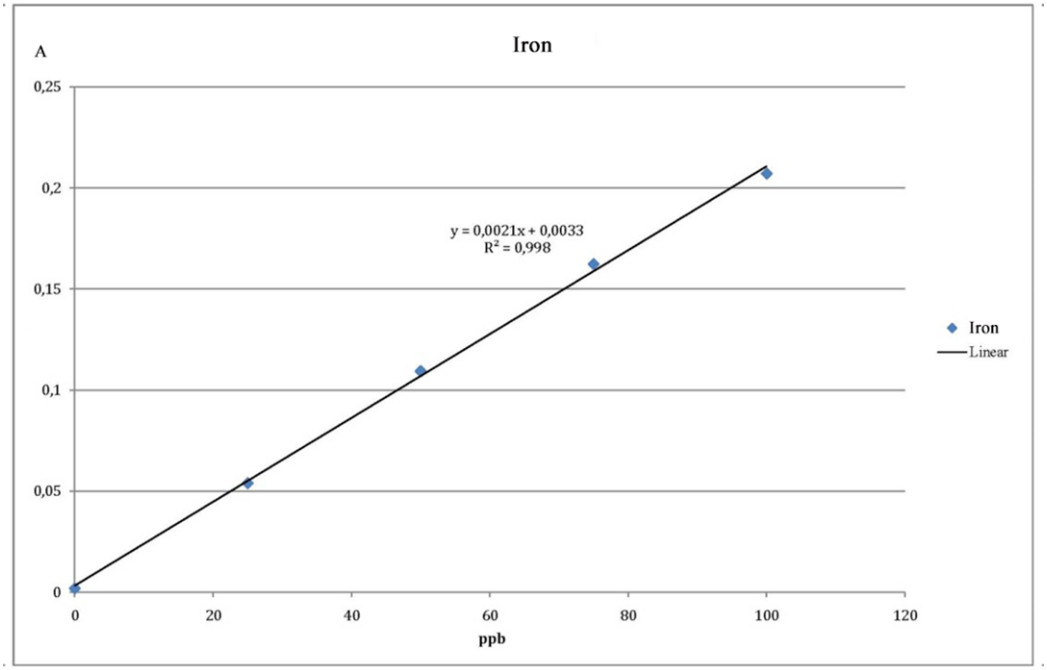

Fig.9. Calibration graph for iron

Table 13. Experimental conditions for iron

Wavelength (nm)	248.3
Slit (nm)	0.2
Current (mA)	15
Time of reading (s)	2 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	25-100
Modifier	/
Volume of the modifier (µl)	/

Table 14. Temperature program for iron

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1400	5	10	
4	2400	1	3	min flow, read
5	2600	1	3	
6	20	20	20	

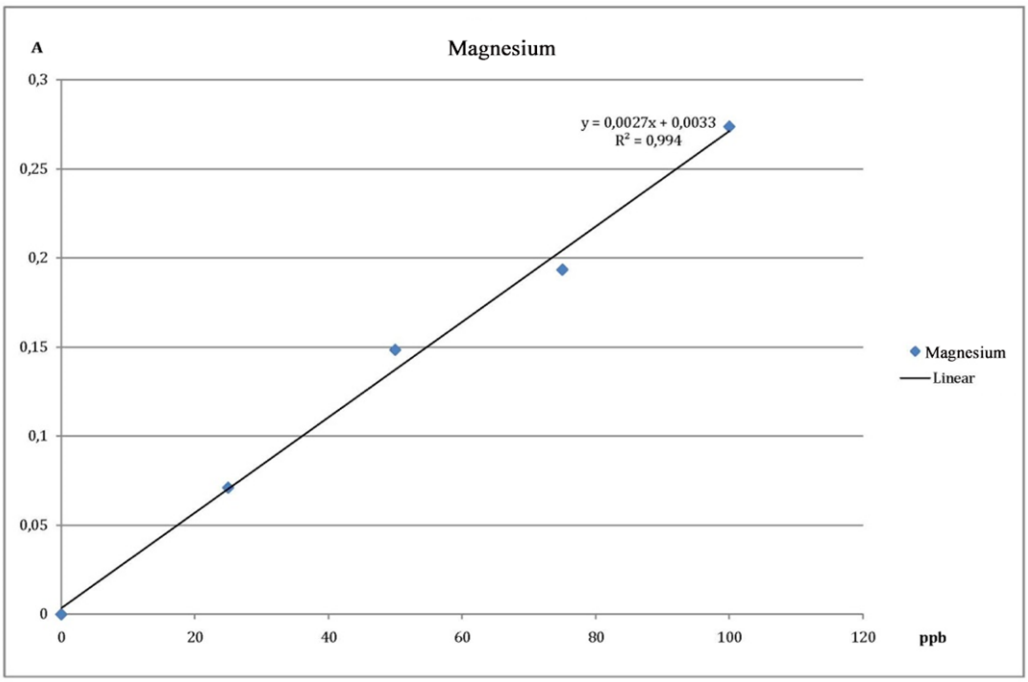

Fig.10. Calibration graph for magnesium

Table 15. Experimental conditions for magnesium

Wavelength (nm)	285.2
Slit (nm)	0.7
Current (mA)	4
Time of reading (s)	3 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	25-100
Modifier	/
Volume of the modifier (µl)	/

Table 16. Temperature program for magnesium

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	900	5	10	
4	1700	1	3	min flow, read
5	2100	1	3	
6	20	20	20	

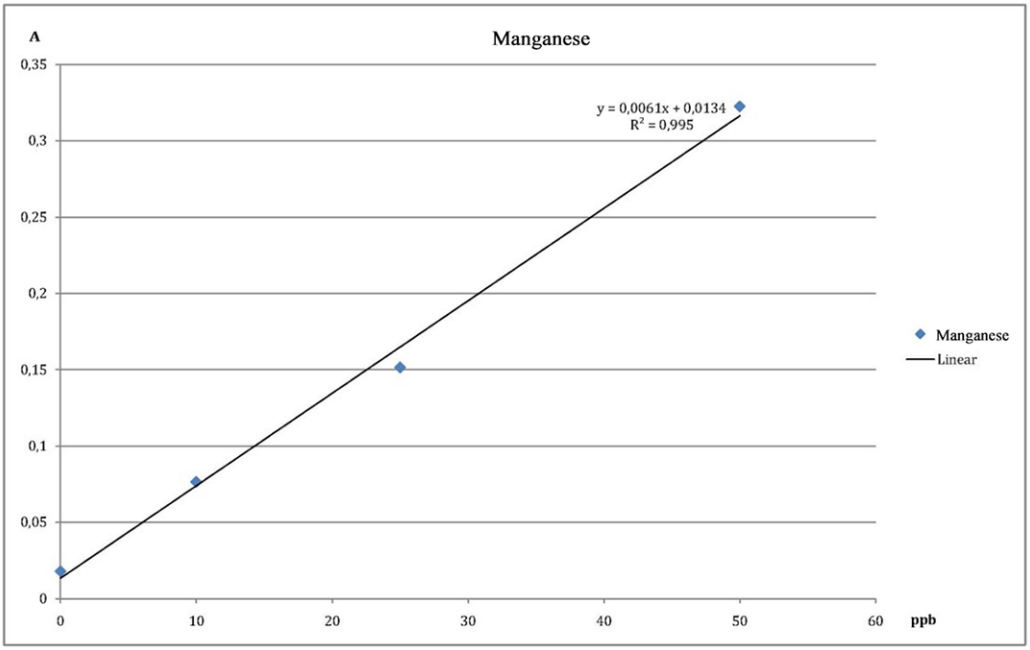

Fig.11. Calibration graph for manganese

Table 17. Experimental conditions for manganese

Wavelength (nm)	279.5
Slit (nm)	0.2
Current (mA)	12
Time of reading (s)	3 s, peak area
Background correction	da
Sample volume (µl)	20
Linear range (ppb)	10-50
Modifier	1g/L $Mg(NO_3)_2$
Volume of the modifier (µl)	5

Table 18. Temperature program for manganese

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1400	5	10	
4	2200	1	3	min flow, read
5	2400	1	3	
6	20	20	20	

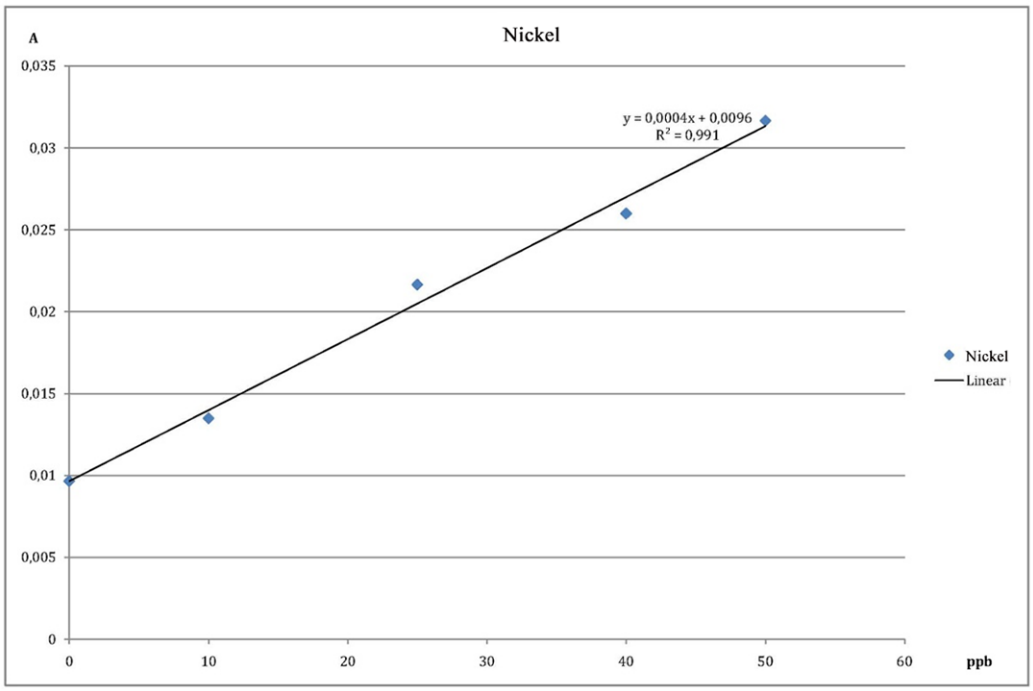

Fig.12. Calibration graph for nickel

Table 19. Experimental conditions for nickel

Wavelength (nm)	232.0
Slit (nm)	0.2
Current (mA)	4
Time of reading (s)	4 s, peak area
Background correction	yes
Sample volume (µl)	10
Linear range (ppb)	10-50
Modifier	/
Volume of the modifier (µl)	/

Table 20. Temperature program for nickel

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	1400	5	10	
4	2500	1	3	stop flow, read
5	2650	1	3	
6	20	20	20	

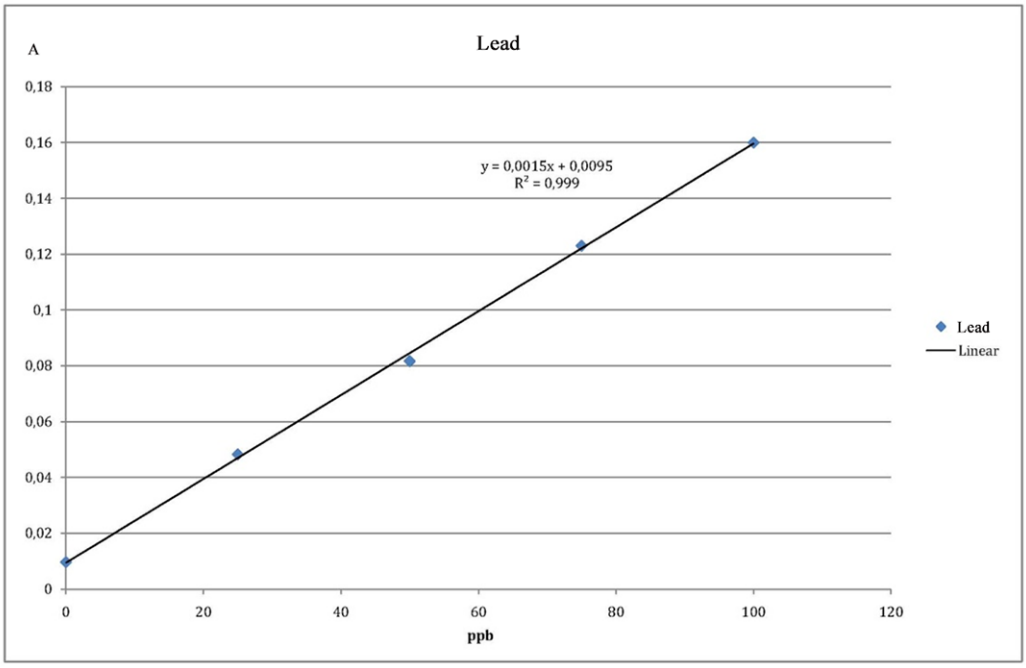

Fig.13. Calibration graph for lead

Table 21. Experimental conditions for lead

Wavelength (nm)	217.0
Slit (nm)	0.7
Current (mA)	11
Time of reading (s)	2 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	25-100
Modifier	/
Volume of the modifier (µl)	/

Table 22. Temperature program for lead

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	5	10	
2	250	10	20	
3	850	5	10	
4	1800	1	3	min flow, read
5	2100	1	3	
6	20	20	20	

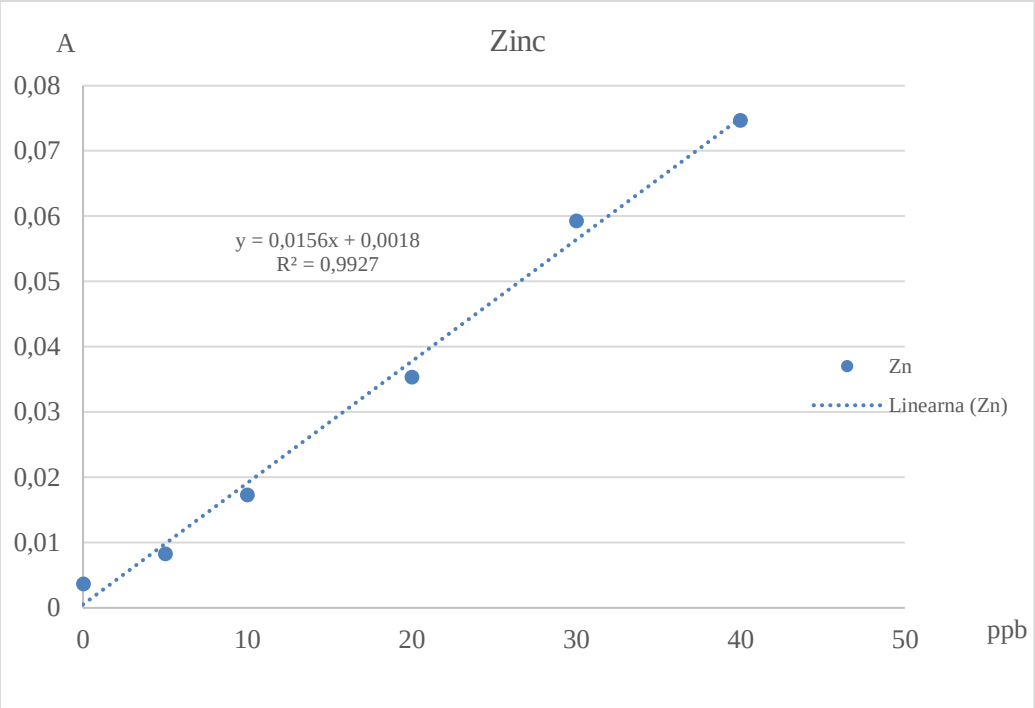

Fig.13. Calibration graph for zinc

Table 21. Experimental conditions for zinc

Wavelength (nm)	213.9
Slit (nm)	0.7
Current (mA)	7
Time of reading (s)	2 s, peak area
Background correction	yes
Sample volume (µl)	20
Linear range (ppb)	5-40
Modifier	/
Volume of the modifier (µl)	/

Table 22. Temperature program for zinc

Step	Temperature (^{o}C)	Ramp time (s)	Hold time (s)	
1	90	10	20	
2	120	10	10	
3	400	5	15	
4	1800	1	3	min flow, read
5	2000	1	3	
6	20	20	20	