

UC Davis

**The Proceedings of the International Plant Nutrition
Colloquium XVI**

Title

Iron interference in Mo determination in Mehlich-1 and Mehlich-3 soil extracts

Permalink

<https://escholarship.org/uc/item/31w6t5pn>

Authors

Fontes, Renildes L. F.
Coelho, Herica A
mello, Jaime W. V.

Publication Date

2009-05-20

Peer reviewed

Introduction

Molybdenum has the lowest concentration in soils and plants. A routine procedure for the determination of its soil availability is not yet established. Many soil extractors and dosage procedures have been tested but no method has been used in a routine basis for soil analysis in Brazilian laboratories. The dosage of Mo in soil extracts by using the KI + reaction was proposed by Yatsimirskii (1964), modified by Fuge (1970) and Eivazi et al. (1982) and used by Fontes et al. (2000). This method suffers from interference of Fe (Eivazi and Sims, 1997) which may be removed by NH_4F (Quin and Woods, 1979). In tropical soils, the high Fe contents may render the determination of Mo availability difficult.

The extractor Mehlich-1 (M1) has been used for determination of P, K, Fe, Mn, Cu and Zn availability in Brazilian soils. It is used for micronutrients, primarily, based on the operational convenience of having the same extractor for various elements. Mehlich-3 (M3) is reported as a promising multi-element extractor since it allows simultaneous extraction of P, K, Ca, Mg, Mn, Cu, Zn, Mo and B (Jones Jr, 1990).

This work aimed to evaluate the interference of Fe on the determination of Mo in M1 and M3 soil extracts and in Mo standard solutions.

Material and methods

To evaluate Fe interference, 0.00; 0.01; 0.03; 0.05; 0.07; 0.10; 0.12 and 0.15 mg L^{-1} Mo (in HCl 0.125 mol L^{-1}) solutions were placed in glass tubes. Each tube received FeCl_3 solution to get 0.000; 0.001; 0.010; 0.100 and 1.000 mg L^{-1} Fe. All tubes received 1.0 mL H_2O_2 0.65 mL L^{-1} and 1.0 mL KI 2.5 g L^{-1} . After 10 minutes, the absorbances in the extracts were read (in 1.0 cm length quartz cube, at 350 nm wavelength). The determinations were replicated three times.

Soil samples (5.0 cm^3) from four Brazilian soils (0-20 cm surface layer) were extracted with 50 mL of M1 and M3 solutions plus 0.5 g activated charcoal. After 5 minutes shaking, 16 hours standing, and filtration, Mo was dosed in the extracts. Each extract (3 mL) was mixed with 1.0 mL of 0.65 mL L^{-1} H_2O_2 and 1.0 mL 2.5 g L^{-1} KI in glass tubes. To each tube, Fe solution was added to get 0.000, 0.001, 0.100, 0.100 and 1.000 mg L^{-1} Fe concentrations. After 10 minutes, the absorbance was read as described for the Mo standard solutions.

Regression equations were adjusted for absorbance readings as a function of Mo concentrations in the standard solutions at the different Fe levels; and for soil extract Mo adjusted as a function of the Fe levels in the M1 and M3 extracts. Determination coefficients were calculated.

Results and discussion

The absorbance readings in the standard solutions increased as the Fe concentration in the solutions increased (Table 1). The slope of the absorbance versus Mo concentration equation increased from 11.403 to 11.777, from 11.77 to 12.051, then decreased from 12.051 to 11.989, and increased again from 11.989 to 12.626, as the Fe level increased, sequentially, from 0.000 to 1.000 mg L^{-1} (Table 1). It seems there is a pattern of variation with increase in the slope as the Fe level in the solution was raised. The increase in absorbance seemed to be related to the Fe level and not to the Mo concentration in solution. It was reported that the reaction $\text{KI} + \text{H}_2\text{O}_2$ is catalyzed by compounds of Fe (III) (Quin and Woods, 1979) which may interfere in the Mo determination. The solutions

originally did not contain other relevant interfering agents, therefore, the increasing Fe levels were responsible for the increase in absorbance in the solutions. In soils with high Fe contents, the use of the KI + H₂O₂ method may result in overestimation of the soil Mo. In this case, in most Brazilian soils, the Mo determination may be subjected to Fe interference.

Table 1: Absorbance readings as a function of Mo concentrations in standard solutions at increasing Fe concentrations and regression equations for the absorbance readings as a function of Mo concentrations at increasing Fe concentrations.

Fe (mg L ⁻¹)	Mo (mg L ⁻¹)							
	0.00	0.01	0.03	0.05	0.07	0.10	0.12	0.15
	-----Absorbance-----							
0.000	0.00	0.154	0.442	0.717	0.956	1.227	1.474	1.721
0.001	0.00	0.163	0.403	0.724	0.972	1.268	1.491	1.748
0.010	0.00	0.187	0.441	0.763	1.070	1.346	1.563	1.776
0.100	0.00	0.199	0.431	0.765	1.031	1.327	1.539	1.789
1.000	0.00	0.213	0.492	0.776	1.058	1.392	1.623	1.897
Fe concentration (mg L ⁻¹)	Regression Equations [absorbance (\hat{Y}) and Mo (X)]							R ²
0.000	$\hat{Y} = 0.089 + 11.403X$							0.9896*
0.001	$\hat{Y} = 0.065 + 11.777X$							0.9913*
0.010	$\hat{Y} = 0.095 + 12.051X$							0.9831*
0.100	$\hat{Y} = 0.091 + 11.989X$							0.9879*
1.000	$\hat{Y} = 0.089 + 12.626X$							0.9918*

Significant at the 5 % probability level.

The mean of the soil Mo was 3.4 mg L⁻¹ in M1 extracts and 0.9 mg L⁻¹ in M3 extracts. Additionally, the equation slopes for the M1 extracts were about 2 to 5 times greater than those for the M3 extracts (Table 2). It has to be noted that the M3 solution is supposed to be more efficient (due to the presence of NH₄F and EDTA) than M1 for the extraction of soil Mo. The NH₄F present in the M3 solution removing the Fe interference seems to be the explanation for this.

Table 2: Concentrations of Mo in M1 and M3 soil extracts at different Fe concentrations (NH₄F was to added only to 0.0 mg L⁻¹ Fe extracts) and regression equations for the Mo conc. as a function of Fe conc. in the extracts without NH₄F.

Fe	Mo concentration								
	Mehlich-1				Mehlich-3				
	CXbd	PVAe	LVd	LVAdh	CXbd	PVAe	LVd	LVAdh	
mg L ⁻¹	-----				-----				
0.000	2.50	2.73	3.12	3.52	0.45	1.10	0.79	1.31	
0.001	2.56	2.90	3.14	3.92	0.46	1.18	0.83	1.32	
0.010	2.78	3.00	3.17	4.37	0.59	1.21	0.85	1.22	
0.100	2.95	3.29	4.46	3.52	0.61	1.19	0.85	1.31	
1.000	3.22	4.06	4.43	4.89	0.69	1.25	0.75	1.37	
Soil ^a	Mehlich-1				Soil ^a	Mehlich-3			
	Equations: Mo (\hat{Y}) Fe (X)			R ²		Equations: Mo (\hat{Y}) Fe (X)			R ²
CXbd	$\hat{Y} = 0.5665 + 2.6761X$			0.7060*	CXbd	$\hat{Y} = 0.1771 + 0.5206X$			0.5648*
PVAe	$\hat{Y} = 1.1414 + 2.9424X$			0.9051*	PVAe	$\hat{Y} = 0.0847 + 1.1672X$			0.4516*
LVd	$\hat{Y} = 1.1008 + 3.4194X$			0.4545*	LVd	$\hat{Y} = 0.0786 + 0.8315X$			0.6277*
LVAdh	$\hat{Y} = 1.0439 + 3.812X$			0.5999*	LVAdh	$\hat{Y} = 0.0833 + 1.2875X$			0.4515*

*Significant at the 5 % probability level; *CXbd = Cambissol; PVAe = Eutrophic Red-Yellow Argissol; LVd = Dystrophic Red Latossol; LVAdh = Humic Dystrophic Red-Yellow Latossol.

Conclusions

The dosage of soil Mo by using the KI + H₂O₂ reaction overestimates the soil Mo concentrations in Mehlich-1 extracts as compared to M3 extracts. This difference is due to the presence of NH₄F in the Mehlich-3 solution.

References

- Eivazi, F., Sims J.L. 1997. Analytical techniques of molybdenum determination in plant and soils. In: Molybdenum in agriculture. Ed. U.C. Gupta. pp. 92-110. Cambridge University, Cambridge.
- Eivazi, F. et al. 1982. Determination of molybdenum in plant material using a rapid, automated method. *Soil Sci. Plant Anal.*, v. 13, p. 135-150, 1982.
- Fontes, R. L. F. et al. Determination of molybdenum in soil test extracts with potassium iodide plus hydrogen peroxide reaction. *Commun Soil Sci. Plant Anal.*, v. 31, n. 15/16, p. 2671-2683, 2000.
- Fuge, R. An automated method for the determination of molybdenum in geological and biological samples. *The Analyst*, v. 95, p. 171-176, 1970.
- Jones Jr., J. B. Universal soil extractants, their composition and use. *Commun Soil Sci. Plant Anal.* 1990, 21, 1091-1101.
- Quin, B. F., Woods, P. H. Automated catalytic method for the routine determination of molybdenum in plant materials. *Analyst*, v. 104, p. 552-559, 1979.
- Yatsimirskii, K.B. The use of catalytic reactions involving hydrogen peroxide in the study of the formation of complexes and in the development of very sensitive analytical methods. In: A. A. Balandin et al. *Catalysis and Chemical Kinetics*. New York : Academic Press, 1964. p. 201-206.

