

## Original Research Article

# Comparison of DTPA Extracting Methods for Available Zinc with Multinutrient Extractants in Vertisols and Inceptisols

### ABSTRACT

**Aims:** To develop relationships between some universal soil extractants and legacy methods.

**Place and Duration of Study:** Study was carried out at IISS, Bhopal.

**Methodolog:** Samples were analyzed by different methods being related; e.g. GLOSOLAN/international method or rapid methods/universal extractant and legacy methods. Measured data was obtained under conditions of best achievable laboratory quality control, for maximal transfer function reliability with lowest RMSE or error (Root Mean Sum of Squares). Out of 60 samples in each category representative set 80% samples were used (e.g. by linear regression analysis) for development of transfer function and rest 20% were retained for validation of transfer functions. Residuals (differences) between measured and estimated soil property values were calculated. The correlation and RMSE (Root Mean Square Error) were calculated for the validation data set.

**Results:** For Zinc DTPA and Mehlich-3 had the regression coefficient ( $R^2 = 0.92$ ). DTPA and AB DTPA had the regression coefficient ( $R^2 = 0.72$ ), with extraction in the order of Mehlich3>AB DTPA> DTPA. Hence, Mehlich-3 and AB-DTPA both extractants can replace DTPA extractant for the determination of available micronutrients in soils in Vertisols.

**Conclusion:** In conclusion, different regression equations are developed between the various methods of analysis that must be used in order to compare the results obtained from different extraction methods.

*Keywords: Universal extractants, Zinc, DTPA, Mehlich-3*

## 1. INTRODUCTION

The phrase "universal soil extractant" has been used to describe a reagent that can be used to extract well over one category of elements and/or ions from a soil, the concentration of which can be used to determine the soil's fertility or toxicity levels. The use of universal extractants in soil testing procedures has several benefits, including raising the dependability of the tests, improving their accuracy and precision, and speeding up routine soil analysis. Over the past few years, a number of procedures have been created and released for quick soil tests to detect the amounts of various components in soil. Multi-nutrient extractants present a workable solution to these problems since they allow for the simultaneous extraction of multiple nutrients. The nutrients can be estimated using a multielement analyzer such as the inductively coupled plasma emission spectrometer or atomic absorption spectrophotometer. The invention of extracting solutions containing chelating agents, primarily DTPA and EDTA, represents one of the most significant developments in micronutrient soil testing. In addition to macronutrients, the Mehlich-3 extractant was created as an all-purpose (many nutrients) extractant for routine analyses of micronutrients (Cu, Fe, Mn, Zn, etc.) for a variety of soils over acidic, neutral, and high pH soils. High correlation coefficients between Zn values obtained by TEA-DTPA and AB-DTPA were discovered. Therefore, it is crucial to assist laboratories in selecting the best approach and to encourage the switch to more environmentally friendly ones. In order to do so, the following information is to be taken care of per SOP (standard operating procedures): (i) health risk (related to the use of chemicals and the overall implementation of the procedure by staff); (ii) Environmental risk (related to waste disposal); (iii) Level of technology required to perform the analysis and (iv)

Average duration of the test. Aim of the study was to harmonize methods, data and information to (1) provide reliable and comparable information between countries and projects; (2) allow the generation of new harmonized soil data sets; and (3) support evidence-based decision making for sustainable soil management.

## 2. METHODOLOGY

Extractable micronutrients i.e., zinc, copper were determined using Atomic Absorption Spectrophotometer (AAS) (Lindsay and Norvell, 1978). The multi-nutrient extractant ammonium bicarbonate-DTPA (AB-DTPA) has been shown to be able to extract all of the nutrients—available P, K, and micronutrient simultaneously in alkaline soils. For the chelation of micronutrients, DTPA is employed, whereas bicarbonate is used to remove phosphorus, ammonium for the extraction of potassium,  $\text{HCO}_3$  is for the extraction of P, DTPA for chelation of Ca, Mg, and micronutrients: Zn, and Cu. The solution's pH was adjusted to 7.6 (Soltanpour and Schuwab, 1977). Since ammonium bicarbonate is not stable for longer than 24 hours, fresh solutions were made every day for analysis.

Mehlich (1984); Extracting Solution (0.2 M acetic acid + 0.25 M  $\text{NH}_4\text{NO}_3$  + 0.015 M  $\text{NH}_4\text{F}$  + 0.013 M nitric acid + 0.001 M ethylene di-amine tetra acetic acid) by shaking 2 g of soil with 20 mL of the extractant for 5 min at 1:10 soil/solution ratio soil extracts were filtered through Whatman No. 2 filter paper and the concentration of P was read on. The filtrates were analyzed by ICP at 327.393 nm for Cu, and 206.200 nm for Zn.

Data was interpreted in MS EXCEL using software DATA ANALYSIS tool and OPSTAT. Descriptive statistics and measure of central tendencies were carried out for micronutrients data with different extractants and ratios. Variability in soil parameters (variants) was analyzed using the coefficient of variation (CV). In order to find out whether the mean difference between two sets of observations is zero, the paired t-test statistical approach was used. This approach offers a number of reports for drawing conclusions regarding the variance between two population means from a paired sample. At a significance level of 0.05, 0.01 and 0.001 the null hypothesis is assumed to be zero in this situation and the degree of significance was compared with the p value for the two tail values.

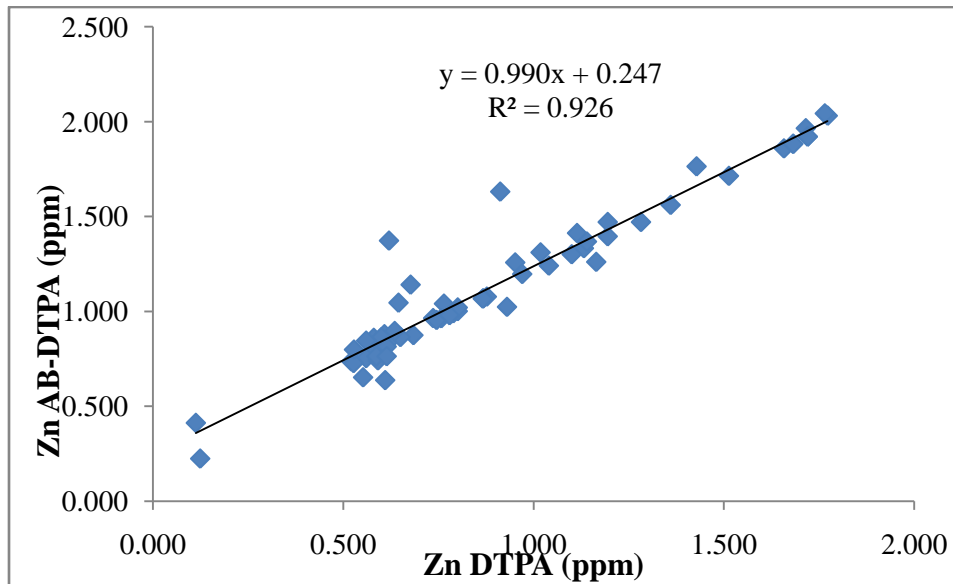
## 3. RESULTS AND DISCUSSION

### 3.1.1 DTPA as soil micronutrient (Zinc) extractant as compared with AB-DTPA and Mehlich-3 in Vertisols

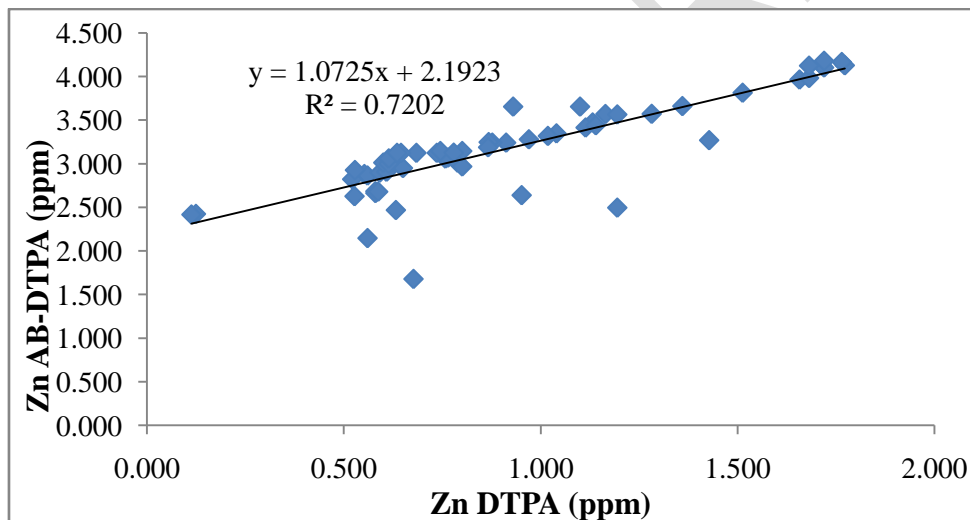
Tables 1, for Vertisols provide a summary of the mean values, errors, and range for zinc with various extractants. According to Table 1 and figure 1 and 2; DTPA extractable Zn had a range of 0.112-1.772 mg/kg with a mean value of 0.922mg/kg and had a correlation ( $r= 0.84^{***}$ ) with Mehlich-3 extractable Zinc in a range of 1.67-4.17 mg/kg with a mean value of 3.18mg/kg and AB-DTPA extractable Zn in a range of 0.22-2.04 mg/kg with a mean value of 1.16 mg/kg and had a correlation ( $r= 0.96^{***}$ ). In comparison to all other extractants for Vertisol, DTPA and Mehlich-3 had the regression coefficient ( $R^2= 0.92$ ) and DTPA and AB-DTPA had the regression coefficient ( $R^2= 0.72$ ), with extraction in the order of Mehlich-3>AB DTPA> DTPA. RMSE and MAE is given in table 2

**Table 1 Range of Concentrations and Means for available Zinc by DTPA, AB-DTPA and Mehlich-3 in Vertisols**

	Extractant	Soil: Solution Ratio	Min	Max	Mean	Median	Std. Error	SD
	DTPA	1:2	0.11	1.77	0.92	0.79	0.05	0.41
<b>Zn</b>	AB DTPA	1:2	0.22	2.04	1.16	1.04	0.05	0.42
	Mehlich-3	1:10	1.67	4.17	3.18	3.12	0.06	0.52



**Fig.1. Relationship between DTPA and AB-DTPA method of available Zn in Vertisols**



**Fig. 2. Relationship between DTPA and AB-DTPA method of available Zn in Vertisols**

### 3.1.2 DTPA as soil micronutrient (Zinc) extractant as compared with AB-DTPA and Mehlich-3 in Inceptisols

Table 2 provides a summary of the mean values, errors, and ranges in Inceptisols soil for Zinc in various extractants. According to Table 2 and figure 3, 4; DTPA extractable Zn had a range of 0.07-1.96 mg/kg with a mean value of 0.92 mg/kg and had a strong correlation ( $r = 0.89^{***}$ ) with Mehlich-3 extractable Zn in a range of 0.13-2.14 mg/kg with a mean value of 1.07 mg/kg. AB-DTPA extractable Zn was in a range of 0.04-1.79 mg/kg with a mean value of 1.07 mg/kg and had a strong correlation ( $r = 0.91^{***}$ ). In comparison to all other extractants for Inceptisols DTPA and Mehlich-3 had the regression coefficient ( $R^2 = 0.82$ ). DTPA and AB-DTPA had the coefficient of determination ( $R^2 = 0.79$ ), with extraction in the order of Mehlich-3 > AB DTPA > DTPA.

### Table 2 Range of Concentrations and Means for available Zn by DTPA, AB-DTPA and Mehlich-3 in Inceptisols

Extractant	Soil to Solution Ratio	Min.	Max.	Mean	Median	Std. Error	Std. Deviation
DTPA	1:2	0.07	1.96	0.92	0.79	0.05	0.42
Zn AB DTPA	1:2	0.04	1.79	0.82	0.77	0.05	0.39
M3	1:10	0.13	2.14	1.07	0.99	0.05	0.41

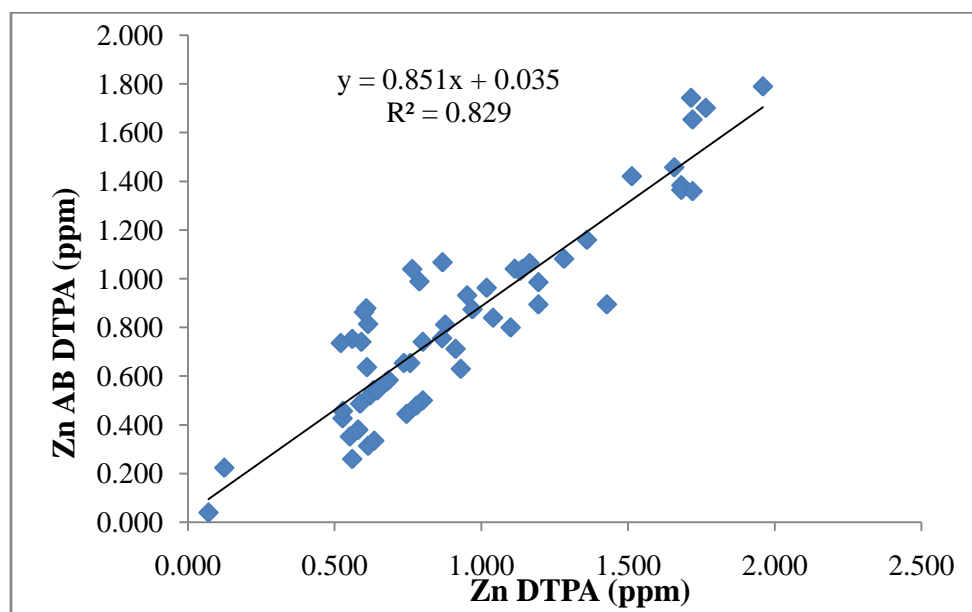


Fig. 3 Relationship between DTPA and AB-DTPA method of available Zn in Inceptisols soil

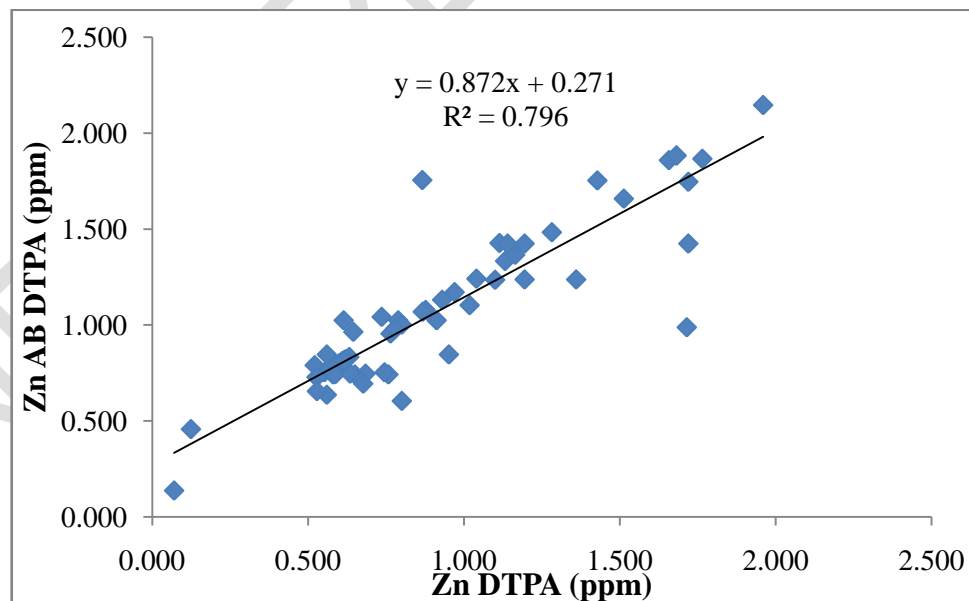


Fig. 4 Relationship between DTPA and AB-DTPA method of available Zn in Inceptisols soil

Table 3 Coefficient of determination ( $R^2$ ), Root Mean Square Error and Mean Absolute Error of DTPA as extractant compared with AB-DTPA and Mehlich-3 method for Zinc in both Vertisols and Inceptisols

Vertisols		R <sup>2</sup>	RMSE	MAE
Zn DTPA	Zn AB-DTPA	0.926	0.176	0.132
	Zn Mehlich-3	0.720	0.152	0.096
<b>Inceptisols</b>				
Zn DTPA	Zn AB-DTPA	0.829	0.136	0.111
	Zn Mehlich-3	0.796	2.190	2.180

**Table 4 Correlation coefficients (r) of micronutrient concentrations (Zinc) extracted by DTPA, Mehlich-3 and AB-DTPA in Vertisol and Inceptisol soils.**

DTPA	Mehlich-3	AB DTPA
Vertisol	0.84***	0.96***
Inceptisol	0.89***	0.91***

#### **DTPA as soil micronutrient (Zinc) extractant as compared with AB-DTPA and Mehlich-3 in Vertisols**

According to Table 1 and figure 1 and 2; DTPA extractable Zn showed highly significant results with a correlation ( $r=0.84^{***}$ ,  $p<0.001$  and  $0.05$ ) with Mehlich-3 which indicates that M3 can extract Zn from soil more than DTPA. AB-DTPA extractable Zn also showed highly significant results with a correlation ( $r=0.96^{***}$ ,  $p<0.001$  and  $0.05$ ). In comparison to all other extractants for Vertisols, DTPA and Mehlich-3 had the regression coefficient ( $R^2=0.92$ ). DTPA and AB-DTPA had the regression coefficient ( $R^2=0.72$ ), with extraction in the order of  $M3>AB\ DTPA>DTPA$ . Hence, M3 and AB-DTPA both extractants can replace DTPA extractant for the determination of available micronutrients in soils of the studied areas.

The presence of acid and EDTA in Mehlich-3 may have contributed to Mehlich-3 higher Zn extraction capability compared to DTPA and AB-DTPA extractants. Moreover, metals such as Fe, Mn, Cu, and Zn are extracted by  $H^+$ , and  $NH_4^+$  ions in M3 extractant. Similar results were reported by authors (Walworth *et al.*, 1992; Volcasek and Friedericks, 1994; Elrashidi *et al.*, 2003; Maftoun *et al.*, 2003; Takrattanasaran *et al.*, 2010) for Vertisols

#### **DTPA as soil micronutrient (Zinc) extractant as compared with AB-DTPA and Mehlich-3 in Inceptisols**

According to Table 3 and 4 and figure 3 and 4 DTPA extractable Zn and Mehlich-3 extractable Zn showed significant results with the regression coefficient ( $R^2=0.79$ ) with correlation ( $r=0.89^*$  at  $P<0.001$  and  $0.05$ ) and DTPA extractable Zn and AB-DTPA extractable Zn showed non-significant results with the regression coefficient ( $R^2=0.82$ ) in Inceptisols soils with extraction in the order of  $M3>AB\ DTPA>DTPA$ . Similar results were reported by authors (Elrashidi *et al.*, 2003; Malathi and Stalin, 2018; Mekala *et al.*, 2019).

#### 4. CONCLUSION

The choice of extraction method for zinc analysis in soil depends on the soil properties, pH conditions, and the specific objectives of the study. DTPA and AB-DTPA are commonly used for acidic to neutral soils, while Mehlich-3 is suitable for a broader pH range. The selection of the appropriate method should consider the specific characteristics of the soil under investigation to obtain accurate and meaningful results for zinc availability assessment. Thus, DTPA and Mehlich-3 cannot be recommended as general test procedures for extraction of the studied micronutrients from natural soil samples having various chemical and physical properties unless field calibrations are collected for these soils.

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