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AN EVALUATION OF A MECHANICAL VACUUM EXTRACTOR FOR THE DETERMINATION OF CATION EXCHANGE CAPACITY AND EXTRACTABLE CATIONS IN CALCAREOUS SOILS

Y.P. Kalra and D.G. Maynard
Northern Forestry Centre, Forestry Canada, Edmonton, Alberta, Canada

One of the most widely used methods for the determination of cation exchange capacity (CEC) and extractable cations involves saturation of the exchange sites with NH_4 followed by the displacement and determination of the NH_4 that is adsorbed by the soil. The present study was undertaken to compare a mechanical vacuum extractor (Holmgren et al. 1977) against the manual Büchner funnel method for leaching (Atkinson et al. 1958).

Eight organic and mineral soils (4 calcareous, 4 noncalcareous) were analyzed. The pH (in 0.01 M $CaCl_2$) of the soils ranged from 3.4 to 9.6. The $CaCO_3$ equivalent in the four calcareous soils ranged from 11.5 to 31.5% (Sample 4, 6, 7 and 8). These soils had been used earlier for three inter-laboratory check sample studies carried out by different organizations.

Soils were leached, using vacuum, with 1.0 M NH_4OAc, pH 7.0 by a mechanical extractor and the traditional manual Büchner funnel method. For the manual method 10 g organic or 25 g mineral soil was leached with 250 ml solution; for the mechanical extractor 0.5 g organic or 2.5 g mineral soil was leached with 50 mL NH_4OAc solution over a 16-h extraction time.

Ammonium-N in the soil was determined by an automated distillation - titration method using the Kjeltec Auto 1030 Analyzer (Tecator). Distilled NH_4 was collected into 1% H_3BO_3 solution containing a mixed indicator (methyl red and bromo cresol green). The titration was performed with standard HCl. Calcium, Mg, K, and Na in the NH_4OAc leachate were determined by inductively coupled plasma-atomic emission spectroscopy (ICP-AES).

Table 1 compares results from the manual and the mechanical extraction. There were no differences between the manual Büchner funnel (BF) and the mechanical vacuum extractor (MVE) methods for CEC and exchangeable K and Na (not shown). Both leaching techniques gave similar results for Ca and Mg on noncalcareous soils. For calcareous soils, however, the mechanical extractor gave significantly higher results for Ca and to a lesser degree Mg than the manual technique.

In calcareous soils, NH_4OAc overestimates exchangeable Ca as substantial amounts of Ca are extracted by this solution from carbonates. This problem is accentuated when MVE is used. In the BF technique the leaching takes 1 to 6 hours while in MVE it is carried out in approximately 16 hours. Longer leaching time, plus the fact that fresh NH_4OAc is added drop-by-drop, resulted in higher solubilization of Ca from the carbonates. The amount of exchangeable Ca in calcareous soils can be estimated by the following: Exchangeable Ca = CEC - (Mg + K + Na).

For the determination of CEC, inconsistency in the NH_4^+ saturating and rinsing steps is the greatest source of error (Cappo et al. 1987). One of the problems with the BF procedure is that the time the soil and leaching solution are in contact is never the same, particularly when soils of varying textures are leached together. When the MVE is used, the time of contact is the same for all samples.

In conclusion, the MVE is suitable for routine measurement of CEC and extractable Ca, Mg, K and Na of noncalcareous soils. Significantly higher results were obtained, however, for Ca and to a lesser degree Mg on calcareous soils.

The advantages of the MVE are that it requires less sample and reagents and provides more uniform leaching. The system is compact and therefore requires much less space than the BF method. The instrument does not require operator attention during the extraction cycle. Presently studies are underway in the Forestry Canada laboratory comparing several commonly used extracting solutions for CEC and exchangeable cations by the MVE technique.

Table 1. Comparison of results obtained by the manual Büchner funnel (BF) and mechanical vacuum extractor (MVE) leaching techniques

Sample no.	CEC		Extractable cations			
			Ca		Mg	
	BF	MVE	BF	MVE	BF	MVE
	cmol (+) kg^{-1}					
1	84.5	85.2	6.22	6.17	2.17	2.01
2	85.0	89.2	6.70	6.84	2.28	2.25
3	32.2	35.3	0.08	0.10	0.03	0.02
4*	3.0	3.5	14.9**	33.1**	0.43	0.53
5	21.4	21.2	2.9	2.71	11.5	10.1
6*	9.1	8.6	14.6**	48.4**	2.17	5.92
7*	2.0	1.8	15.4**	29.0**	3.06	6.59
8*	16.1	16.3	19.8**	41.1**	2.87	3.72

*Calcareous soils.

**These are apparent exchangeable Ca values as they include the exchangeable Ca plus Ca dissolved by the extracting solution from calcareous material (and $CaSO_4.2H_2O$, if present).

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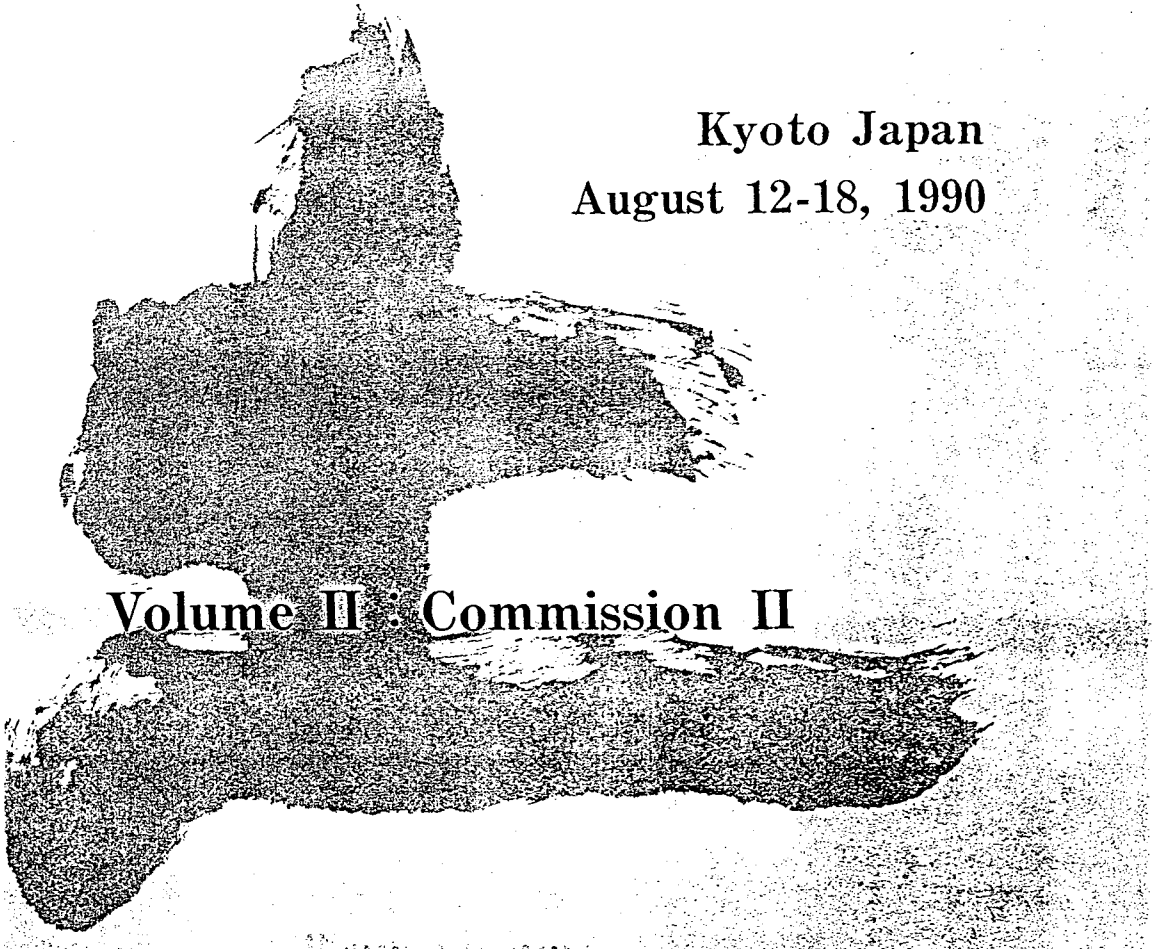

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