

# Relationship of Ammonium Nitrogen Distribution to Mineralogy in a Hapludalf Soil<sup>1</sup>

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## ABSTRACT

Distribution of fixed and exchangeable ammonium were examined in three soil profiles of the Shrouts series from the Knobs region of eastern Kentucky. Shrouts soils (fine, mixed, mesic, Typic Hapludalfs) have high base status; exhibit strong, prismatic structure; and are derived from calcareous, high magnesium, soft (weakly cemented) clay shale. Soil pH ranges from 6.3 in the Ap horizon to 7.9 in the C. Analyses, using the Silva and Bremner procedure, showed considerable fixed  $\text{NH}_4^+\text{-N}$  in all horizons of each profile. Mean fixed  $\text{NH}_4^+\text{-N}$  in the profiles was 365 ppm for the Ap, 463 ppm for the B2t, 433 ppm for the B22t, 469 ppm for the C, and 543 ppm for the Cr horizons. Exchangeable  $\text{NH}_4^+\text{-N}$  also increased with depth, with values being about 1.2 to 1.5% of the fixed  $\text{NH}_4^+\text{-N}$  values. X-ray diffraction analyses of the clays and silts revealed predominantly mica in the 2–0.2 and <0.2  $\mu\text{m}$  fractions, with some vermiculite present, and large quantities of mica in the silt fractions. High correlation ( $r = 0.92$ ) was found between fixed  $\text{NH}_4^+\text{-N}$  and exchangeable  $\text{NH}_4^+\text{-N}$ . A correlation exists between fixed  $\text{NH}_4^+\text{-N}$  and mica + vermiculite content with an  $r$  value of 0.68. The fixed  $\text{NH}_4^+\text{-N}$  in the lower horizons is presumed to be indigenous, since no  $\text{NH}_4^+\text{-N}$  has been added recently to the soil.

**Additional Index Words:** silt mineralogy, clay mineralogy, exchangeable ammonium.

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THE AMOUNT OF indigenous fixed  $\text{NH}_4^+$  present in a soil depends to a large extent on the kind and amount of layer silicate minerals present. The soil minerals that are chiefly responsible for  $\text{NH}_4^+$  fixation are vermiculite, soil mica, and montmorillonite (Allison et al., 1951; Bremner, 1959; Stevenson and Dhariwal, 1958). Dyal and Hendricks (1952) found that vermiculites have the greatest capacity to fix  $\text{NH}_4^+$ , whereas illite may or may not fix  $\text{NH}_4^+$ , depending on the degree of weathering and the  $\text{K}^+$  saturation on exchange sites. They also found that montmorillonite does not fix  $\text{NH}_4^+$  under moist conditions.

It has been known for some time that many soils have the ability to fix and hold substantial amounts of added  $\text{NH}_4^+\text{-N}$ , and that some soils contain large quantities of indigenous fixed  $\text{NH}_4^+\text{-N}$ . Bremner and Keeney (1966) note that most workers have defined fixed  $\text{NH}_4^+$  as that which is not extractable by 1M KCl at laboratory temperatures. Barshad (1951) proposed that fixed  $\text{NH}_4^+$  should be defined as  $\text{NH}_4^+$  that is not replaceable after long exposure to  $\text{K}^+$ -salt solutions added to the soil.

The type of parent material from which a soil has formed also has a significant effect on the amount of

fixed  $\text{NH}_4^+$  present in a soil profile. Adams and Stevenson (1964), in a detailed study of  $\text{NH}_4^+$  sorption and release from rocks and minerals, noted that  $\text{NH}_4^+$  was found along with nonexchangeable  $\text{K}^+$  in primary silicates. They concluded that the micas were primarily responsible for  $\text{NH}_4^+$  fixation by igneous rocks. Stevenson (1959) conducted a study of fixed  $\text{NH}_4^+$  in several specimens of shale and granite, using a modified Dhariwal and Stevenson (1958) method. He found that all specimens contained fixed  $\text{NH}_4^+\text{-N}$ . The content of fixed  $\text{NH}_4^+$  in the shales varied between 330 and 420  $\mu\text{g N/g}$ , whereas the contents in granite rocks varied between 5 and 27  $\mu\text{g N/g}$ . Stevenson concluded that rocks are able to accumulate N through retention of  $\text{NH}_4^+$  from rain water. He discovered that up to two-thirds of the total N in some Paleozoic shales occurs as fixed  $\text{NH}_4^+$  and speculated that native fixed  $\text{NH}_4^+$  may have been constructed into the crystal structure at the time of synthesis of the minerals. Walsh and Murdock (1960) found that two shale-derived Wisconsin soils contained fairly large amounts of indigenous fixed  $\text{NH}_4^+$ . The amounts of fixed  $\text{NH}_4^+$  found were related to the parent material from which the soil formed. Rich (1960) assumed that the fixed  $\text{NH}_4^+$  in muscovite parent material was substituted isomorphously for  $\text{K}^+$ . He further concluded that, for any soil, there was a perpetual ratio between total  $\text{K}^+$  and fixed  $\text{NH}_4^+$  in the parent material.

A large number of workers have studied the distribution of fixed  $\text{NH}_4^+\text{-N}$  through various soil profiles (Allison et al., 1953; Rodrigues, 1954; Hanway and Scott, 1956; Bremner, 1959; Stevenson and Dhariwal, 1958; Rich, 1960; Walsh and Murdock, 1960; Hinman, 1964). Meints and Peterson (1972) found that fixed  $\text{NH}_4^+\text{-N}$  constituted 90–100% of the total inorganic N in certain Nebraska soils. Hinman (1964), working with some Canadian soils, found that the amount of fixed  $\text{NH}_4^+$  increased with increasing soil depth. Total amounts of fixed  $\text{NH}_4^+$  found through five horizons of a depth of 122 cm ranged from 2,600 lbs/acre in the loam surface horizons, to 4641 lbs/acre in the more clayey subhorizons.

This study was initiated for the purpose of acquiring basic physical, chemical, and mineralogical characteristics of the Shrouts soil series. This paper examines the relationship between fixed  $\text{NH}_4^+\text{-N}$  distribution and the mineralogy of the Shrouts series (Typic Hapludalf).

## METHODS AND MATERIALS

These studies were conducted for three soil profiles of the Shrouts series. The profiles were sampled from Bath, Fleming, and Lewis Counties in the Knobs Region of eastern Kentucky. The Shrouts series is currently classified as a Typic Hapludalf. The soils are fine-textured, usually well-drained, and most commonly occur on narrow ridges and side slopes of the upland. Slopes range from 6 to 30%. The surface layer is a thin, dark grayish-brown silty clay loam. The subsoil is an olive-gray clay and is characterized by strong, prismatic structure. The parent material is a calcareous, high in exchangeable Mg, soft weakly cemented clay shale from the upper part of the Crab Orchard formation of Silurian geologic age. Descriptions and

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bulk horizon samples of each profile were taken from specially dug pits at each site. The bulk soil samples were air dried, ground to pass a 2-mm sieve, and thoroughly mixed before further analysis.

Particle size measurements were determined by the pipette method (SCS-USDA, 1967; Kilmer and Alexander, 1949). Additional samples were fractionated using 50 g of soil dispersed with 1N Na<sub>2</sub>CO<sub>3</sub> following organic matter removal with H<sub>2</sub>O<sub>2</sub>. Sands (200–50 μm) were removed by wet sieving. Silt was separated into 50–20, 20–10, and 5–2 μm fractions by elutriation with water adjusted to pH 10 with Na<sub>2</sub>CO<sub>3</sub> (Beavers and Jones, 1966). Clays were separated into 2.0–2 μm and <0.2 μm fractions by supercentrifugation (Jackson, 1956).

Cation exchange capacity of each horizon was determined using the 1N NH<sub>4</sub>OAc method of Peech et al. (1947). Exchangeable NH<sub>4</sub><sup>+</sup> was determined with the procedure of Bremner and Kenney (1966). Fixed ammonium was determined with the method of Silva and Bremner (1966). Total N was determined using the procedure described by Bremner (1965).

Sufficient quantities of each of the clay and silt fractions separated by elutriation process were placed on ceramic tiles for X-ray diffraction measurements (Rich, 1975). The suction apparatus used to coat the tiles with clay was as proposed by Rich (1969). These fractions were either K-saturated by leaching 40 ml of 1N KCl through the clay or silt specimens followed by deionized water, or Mg-saturated by a similar manner using 40 ml of 1N MgCl<sub>2</sub> followed with 10 ml of deionized H<sub>2</sub>O and 10 ml of 20% glycerol in water. The tiles were X-rayed using a Cu X-ray tube and Ni filter. The K-saturated tiles were X-rayed after heat treatment at 25, 110, 300, and 550°C. The Mg-saturated, glycerol-treated tiles were X-rayed at 25 and 110°C. The percentage of the various minerals was estimated using peak heights and *d*-spacings from the X-ray patterns. Values reported were rounded off to the nearest 5% although the accuracy for any given value may be ±10% of the stated value. For example, a value reported as 15% is considered to be in the range of 5 to 25%.

## RESULTS AND DISCUSSION

The particle size distribution data (Table 1) show that the sand percentages were extremely low and silt percentages high in all horizons of the three profiles. Clay contents were consistently highest in the upper B horizons with greater than 30% clay present in all horizons of the profiles.

Soil pH (H<sub>2</sub>O), Table 1, generally increased with increasing depth, becoming mildly to moderately alkaline in the Cr soil horizons. The high pH in the partially weathered Cr may be due to the high content

of free bases, particularly Mg<sup>2+</sup>. The lower pH (5.0) in the B21t horizon of the Lewis County profile may have resulted from more intense weathering of this profile. This hypothesis is supported by the higher percentage of vermiculite found in this profile than in the other two profiles (Table 2).

The high CEC (Table 1) in the Ap horizons of the profiles can be ascribed dually to the high organic matter content and to the relatively high quantities of clay (>40%) in these horizons. The high clay content is primarily responsible for high CEC in the B horizons of the profiles. In all cases, the CEC generally decreased with increasing depth as organic matter content decreased.

Organic carbon content (Table 1) was high (3.4 to 4.4%) in the Ap horizons of the profiles and decreased with depth. The C/N ratio also decreased with soil depth with the organic C content decreasing more rapidly than total N.

### Clay Mineralogy

Mica was the predominant clay mineral (Table 2) in both the 2-0.2 and <0.2 μm fractions of the Bath County profile. Kaolinite was found in both fractions of all horizons, with a lesser amount of vermiculite occurring primarily in the 2-0.2 μm fraction.

Mica contents found in the Fleming County profile were similar to the Bath profile, however, larger amounts of vermiculite were present. Mica in the <0.2 μm fraction tended to increase with depth.

The 2-0.2 μm and the <0.2 μm fractions of the Lewis County profile showed smaller amounts of mica than present in the other profiles and greater contents of vermiculite were present in the Ap, B21t, and Cr horizons. Vermiculite was present only in the Ap horizon of the <0.2 μm fraction and quantities of mica generally increased with increasing soil depth.

### Silt Mineralogy

For the Bath County profile (Table 2), quartz predominated in the 50-20 μm and 20-10 μm fractions.

Table 1—Physical and chemical properties of three Shrouts soil profiles.

Horizon	Depth cm	Sand	Silt	Clay	pH	CEC (meq/100 g)	Organic	C/N	Total N	Fixed NH <sub>4</sub> <sup>+</sup> -N ppm	Exch. NH <sub>4</sub> <sup>+</sup> -N
		(2–0.05 mm)	(50–2 μm)	(<2 μm)	(1:1 H <sub>2</sub> O)		C				
<b>Bath County</b>											
Ap	0–10	5.4	54.6	40.0	7.0	20.5	4.41	7.9	5,570	301	4.5
B21t	10–23	0.7	44.0	55.3	7.4	17.4	1.04	4.9	2,120	470	6.6
B22t	23–46	1.6	56.1	42.3	7.8	12.2	0.52	3.8	1,370	470	6.6
C	46–76	1.3	60.3	38.4	7.8	10.4	0.29	2.8	1,050	484	6.7
Cr	76–102	1.3	65.9	32.8	7.8	9.9	0.17	2.0	830	538	7.1
<b>Fleming County</b>											
Ap	0–10	4.8	47.7	47.5	6.0	23.2	4.00	7.5	5,350	455	6.5
B21t	10–28	1.4	53.4	45.2	6.7	19.1	1.22	6.0	2,010	509	7.1
B22t	28–46	2.9	49.0	48.1	7.9	14.3	0.52	3.9	1,330	420	6.0
C	46–69	2.2	48.4	49.4	8.0	12.4	0.35	3.1	1,110	492	6.9
Cr	69–102	1.3	59.7	39.0	8.0	9.3	0.17	2.0	850	550	7.2
<b>Lewis County</b>											
Ap	0–15	6.2	52.0	41.8	6.0	25.5	3.43	7.2	4,790	340	4.8
B21t	15–25	0.8	36.2	63.0	5.0	28.5	0.58	4.5	1,300	412	5.0
B22t	25–51	0.8	35.9	63.3	6.7	29.4	0.52	3.7	1,420	424	5.2
B3	51–76	1.5	48.6	49.9	7.5	20.8	0.29	2.7	1,070	408	5.0
C	76–97	2.2	58.3	39.5	7.9	15.2	0.17	1.9	920	430	5.4
Cr	97–127	1.3	60.0	38.7	7.6	15.3	0.12	–	–	541	6.9

Table 2—Estimated† mineralogy from x-ray diffraction of silt and clay fractions of the three Shrouts soil profiles.

		Mineralogy of soil fractions																								
		50-20 (μm)				20-10 (μm)				10-5 (μm)				5-2 (μm)				2-0.2 (μm)				<0.2 (μm)				
Horizon	Depth	QZ	MI	KK	FD	QZ	MI	KK	FD	QZ	MI	KK	FD	QZ	MI	KK	FD	QZ	MI	KK	VR	QZ	MI	KK	VR	
cm		%																								
<b>Bath County</b>																										
Ap	0-10	100				60	30		10	50	30	20		30	45	25		5	65	30				75	25	
B21t	10-23	65	20	15		55	20	15		50	35	15		30	45	25		5	60	25	10			85	15	
B22t	23-46	55	25	20		65	25	10		40	40	20		30	40	15	15	5	60	25	10			90	10	
C	46-76	45	55			25	40	20	15	30	50	20		15	50	20	15		70	20	10			85	15	
Cr	76-102	55	45			30	45	25		25	55	20		15	65	20		5	70	20	5	10		80	10	
<b>Fleming County</b>																										
Ap	0-10	25	40	20	15	45	30	25		55	25	10	10	30	45				65	20	15			85	15	
B21t	10-28	55	25	20		50	30	20		30	45	25		20	55	25			60	25	15			85	15	
B22t	28-46	30	30	20	20	40	40	20		45	35	20		25	45	25	5		65	25	10			85	15	
C	46-69	50	30	20		40	25	15	15	50	30	25	5	25	50	25		5	65	20	10			90	10	
Cr	69-102	35	20	20	25	45	35	20		35	40	25		25	50	25		5	65	20	10			90	10	
<b>Lewis County</b>																										
Ap	0-15	70	20		10	50	20	15	15	55	20	20	10	40	30	30		5	40	35	20			65	25	10
B21t	15-25	60	20	20		40	25	20	15	55	25	10	10	40	35	25		5	45	25	25			75	25	
B22t	25-51	75	25			45	20	15	20	50	30	20		35	35	20	10	5	45	30	15			70	30	
B3	51-76	70	10	20		40	25	20	15	45	35	20		35	40	25		5	55	30	5			75	25	
C	76-97	25	25	30	20	40	40	20		40	40	20		25	45	30		5	55	25	15			75	25	
Cr	97-127	35	35		30	40	30	20	10	35	45	20		25	50	25		5	55	25	15			75	20	5

† Estimates should be considered to within  $\pm 10\%$  of the value listed. QZ—quartz; MI—mica; KK—kaolinite; FD—feldspar; VR—vermiculite.

The content of mica was high, however, even in the coarsest fraction. In all but the 50-20  $\mu\text{m}$  fraction, the Cr horizon contained the greatest percentage of mica. Considerable amounts of mica were present, particularly in the 10-5 and 5-2  $\mu\text{m}$  fractions of the Fleming County profile. The amounts of mica were fairly uniform in all fractions through all horizons. The mica contents of the Lewis County profile were somewhat lower, particularly in the finer fractions, than for the other profiles. A considerable amount of mica existed in all fractions however, with more being found in the C and Cr horizons than the A and B horizons.

#### Fixed and Exchangeable Ammonium in Relation to Mineralogy

Large amounts of fixed  $\text{NH}_4^+\text{-N}$  (Table 1) were found in all horizons of the Bath County profile with 301 ppm  $\text{NH}_4^+\text{-N}$  found in the Ap horizon and increasing to 538 ppm in the Cr horizon. This conforms to the trend found by Rodrigues (1954) and Hinman (1963). The fixed  $\text{NH}_4^+\text{-N}$  increased with depth which coincided with the high percentage of clay and mica in the lower horizons and the influence of the shale parent material. Stevenson (1959) has shown that certain shales can contain 300-420 ppm indigenous fixed  $\text{NH}_4^+\text{-N}$ . The silt fractions also (Table 2) contained large amounts of mica, which can contribute significantly to the amount of fixed  $\text{NH}_4^+\text{-N}$ . The amount of fixed  $\text{NH}_4^+\text{-N}$  in respect to total soil N showed a high of 64.8% in the Cr horizon of the Bath County site and a low of 5.4% in the Ap horizon at this site.

The Fleming County profile (Table 1), generally contained more fixed  $\text{NH}_4^+\text{-N}$  than the Bath County profile. This is attributed to the higher clay contents, particularly in the Ap horizon. The clay fractions of this profile also contained more vermiculite than the Bath County profile, so more fixed  $\text{NH}_4^+\text{-N}$  would be

expected. The large amount of fixed  $\text{NH}_4^+\text{-N}$  in the lower horizons of the profile can be ascribed to the presence of a larger percentage of mica and to the lack of weathering of the shale parent material. The exchangeable  $\text{NH}_4^+\text{-N}$  increased as fixed  $\text{NH}_4^+\text{-N}$  increased (Table 1), as in the Bath profile, being greatest in the Cr horizon.

The amount of fixed  $\text{NH}_4^+\text{-N}$  in the Lewis County profile (Table 1) was similar to the other two profiles. In all profiles, the amount of exchangeable  $\text{NH}_4^+\text{-N}$  (Table 1) increased with increasing amounts of fixed  $\text{NH}_4^+\text{-N}$ , being the highest in the Cr and lowest in the Ap horizon. However, the ratio of exchangeable  $\text{NH}_4^+\text{-N}$  to fixed  $\text{NH}_4^+\text{-N}$  decreased with soil depth, which may be related to soil weathering.

The influence of mineralogy on fixed  $\text{NH}_4^+\text{-N}$  is reflected in Fig. 1. A correlation exists between fixed  $\text{NH}_4^+\text{-N}$  and mica + vermiculite content with an  $r$

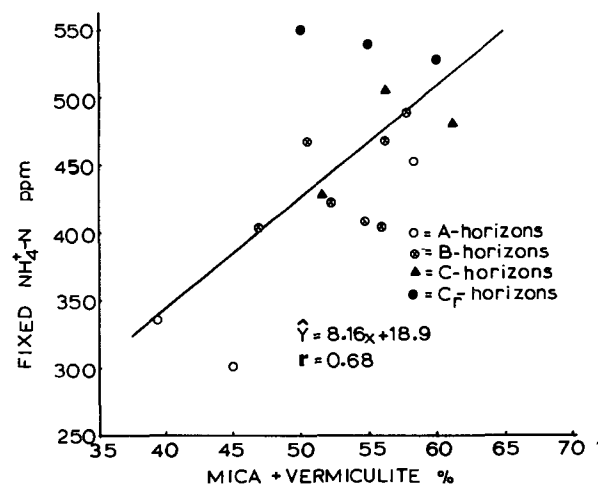


Fig. 1—Regression analysis of fixed ammonium and percent mica + vermiculite expressed on a whole soil basis for three Shrouts profiles.

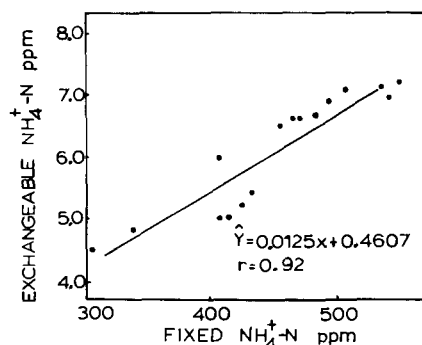


Fig. 2—Regression analysis of fixed and exchangeable ammonium for three Shrouts profiles.

value of 0.68. The relatively unweathered Cr horizons contained the highest levels of fixed  $\text{NH}_4^+\text{-N}$  for all three sites and this nitrogen is considered to be indigenous (Table 1). These data suggest that a relationship exists in Shrouts soils between the amount of minerals present that are considered to be capable of fixing  $\text{NH}_4^+$  to the actual amount of fixed  $\text{NH}_4^+\text{-N}$  measured.

A good correlation exists between fixed and exchangeable  $\text{NH}_4^+\text{-N}$  with an  $r$  value of 0.92. Some of the fixed  $\text{NH}_4^+\text{-N}$  is slowly released by weathering of the micas and expansion of the clay structure releasing  $\text{NH}_4^+\text{-N}$  to the exchangeable form. The difference in exchangeable  $\text{NH}_4^+\text{-N}$  content with depth suggests an equilibrium between the two forms of  $\text{NH}_4^+\text{-N}$ . The close correlation between fixed and exchangeable  $\text{NH}_4^+\text{-N}$  supports this hypothesis. Where there is more fixed  $\text{NH}_4^+\text{-N}$ , there would be expected to be more exchangeable  $\text{NH}_4^+\text{-N}$  released (e.g., in the lower horizons). The exchangeable  $\text{NH}_4^+\text{-N}$  as a percent of fixed  $\text{NH}_4^+\text{-N}$  tends toward slightly less availability with depth (Table 1), with the values all in the 1.2 to 1.5% range.

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