3. MATERIALS AND METHODS

3.1 Selection of Sites

A limited number of sites were chosen for the study so that a more detailed assessment of the soil conditions on each site could be obtained. The selection of the sites was determined by the following factors:

1. The dominant soil series,

2. The procurement of agreements with the property owners or city government,

3. The need to obtain a variety of land use and soil conditions, and

4. The availability of rainfall and runoff data.

Two residential sites were selected in Raleigh, NC: Meredith Townes Subdivision and Mine Valley Subdivision. The Meredith Townes site is a condominium project that was developed in the mid-1960's. It provided a good example of moderate density suburban residential development; how-ever, no runoff data were available. The zoning density at the Meredith Townes Subdivision is R-10[1]. The construction of the Mine Valley Subdivision did not begin until the research program was underway. This allowed for a pre-suburban mapping and evaluation of the property. The development of the first phase of the subdivision during the research provided for an on-going examination of the morphology of the land use conversion. No runoff data were available for the Mine Valley site. The zoning density of the Mine Valley Subdivision is R-4[2].

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[1]R-10 refers to a zoning density of 10 residential units per acre.

[2]R-4 refers to a zoning density of 4 residential units per acre.

The Briar Creek tributary 6 at Sudbury Road watershed in Charlotte, NC was selected. The U.S. Geological Survey had collected rainfall-runoff data for the drainage basin from 1966 through 1970. The Briar Creek at Sudbury Road watershed (henceforth referred to as the Sudbury Suburban Watershed) was developed in the 1960's into primarily 0.10 to 0.15 ha residential lots, or approximately a zoning density of R-4. Along with these three suburban sites, the Carl Alwin Schenck Forest watershed and the adjacent pasture watershed monitored by the North Carolina State University School of Forest Resources were used. The watersheds have adequate runoff records, but rainfall records were taken from the nearby North Carolina State University Research Farm, official N. C. State weather station.

3.2 <u>Suburban Soils Mapping</u>

The three suburban sites provided the opportunity to experiment with suburban soils mapping. Mapping was conducted at a scale of 1:2400 for the Sudbury Suburban Watershed and the Meredith Townes site, which at a scale of 1:1200 for the Mine Valley site.

During the process of soils mapping locations of representative examples of different soils were selected. These locations served as type locations for soil profile descriptions and characterizations. Since a significant amount of the soil surfaces had been disturbed dur-ing urbanization, certain operational definitions for the classification and mapping of the disturbed or man-made soils had to be employed. Several general definitions were selected to taxonomically separate man-made soil profiles within the Entisol Order:

Entisols that--

1. Have fragments of diagnostic horizons and/or pedorelicts that occur more or less without discernible order in the soil below 25 cm, but within a depth of 1 m, and <u>are</u> <u>not</u> permanently saturated with water and do not have characteristics associated with wetness...................................Arents
2. Have fragments of diagnostic horizons and/or pedorelicts that occur more or less without discernible order in the soil below 25 cm, but within a depth of 1 m, and <u>are</u> permanently saturated with water and do have characteristics associated with wetness ....Aquents
3. Have had a portion of the original profile removed, <u>do</u> <u>not</u> have mixing or filling of earthy material or fragments of diagnostic horizons below 25 cm and 1 m in depth, and do not have a subsurface or surface diagnostic horizon ....Orthents

Fourth, those man-made soil profiles that did not conform to these three definitions were classified as usual according to Soil Taxonomy. Within each of these four major groups more precise separations were made as mapping unit criteria. Mapping unit criteria included:

1. Thickness and texture of surface horizon,

2. Bulk density of subsurface horizons,

3. Depth and texture of mixed or filled soil material,

4. Moisture regime of mixed or filled soil material,

5. Compaction and high bulk density of surface horizon, and

6. Thickness of the argillic horizon (used to differentiate the depth of shallow cuts from nearly intact soil profiles).

The mapping units were constructed so as to correspond in an interpretative sense to areas of differing infiltration capacity. The map was later used to select specific sites for infiltration tests.

3.3 <u>Profile Characterization</u>

3.3.1 <u>Field Descriptions</u>

Type location soil profiles were selected for different soils or soil conditions on the various sites. The Sudbury Suburban Watershed provided good examples of both Arents and Orthents. One or more type

locations were selected for each site depending upon the number of important soils present.

Soil profile pits were dug to a depth of 1 to 1.5 m, sampled and described morphologically generally according to the Soil Survey Manual (Soil Survey Staff, 1951) and Soil Taxonomy (Soil Survey Staff, 1975). A 7.5-cm open bucket auger was used to describe the soils at greater depths. Bulk samples from 2 to 5 kg depending upon the relative amount of coarse fragments were taken from each horizon.

3.3.2 Particle Size Analysis

The bulk samples were air-dried, clods were carefully crushed with mortar and pestle to not break gravel and sands, and sieved through a series of sieves with 76 to 2-mm diameter openings. Oven-dried weights were obtained for the percent retained in each fraction: 0 to 2.0 mm, 2.0 to 4.8 mm, 4.8 to 9.5 mm, 9.5 to 19.1 mm, 19.1 to 38.1 mm, and 38.1 to 76.0 mm. The fraction <2 mm is termed the fine-earth fraction, while that 76 to 2-mm range is the coarse-earth fraction.

Particle size analysis of the fine-earth fraction was conducted using the pipette method (Day, 1965). The organic matter was destroyed with 30% hydrogen peroxide. Duplicate 20-g samples were dispersed with a 10% solution of hexa-metaphosphate and a 3-minute treatment with an ultrasonic probe. The samples were sieved through a 50-μm screen and the solution retained in sedimentation cylinders. Pipette samples were taken to achieve fractions of 20 to 50 μm, 5 to 20 μm, 2 to 5 μm and <2 μm. The percent of each fraction was determined from 24-hour 110°C oven-dried weights. The sands were dry-sieved into the five particle size fractions (2 to 1 mm, 1 to 0.5 mm, 0.5 to 0.25 mm, 0.25 to 0.1 mm and 0.1 mm to 0.05 mm), oven-dried and weighed.

3.3.3 Organic Carbon

Organic carbon was determined by acid dichromate digestion and iron sulfate titration (Soil Survey Investigations, 1972).

3.4 Infiltration Measurement

3.4.1 Selection of Infiltration Test Locations

The soil maps were used as a basis for selecting locations for infiltration tests. Tests were conducted at all type locations (except for one, where the volume of coarse fragments was prohibitive). Other locations were selected that appeared to provide typical examples of various suburban soil conditions. Locations were selected in mapping units of significant cuts and fills as well as locations that had no or only slight indications of soil profile alteration and/or compaction. A sufficient number of sampling locations for each type of mapping unit were tested to evaluate the general magnitude of differences. Soil infiltration conditions are heterogeneous centimeter by centimeter across a single mapping unit, therefore the degree of microheterogeneity in a single meter^2 portion of a hectare may be much the same as the degree of macroheterogeneity in the hectare. For these reasons, fewer sampling locations were established so that the infiltration tests could be clustered in triplicate in a 4-m^2 area. Locations selected for infiltration tests had to have no more than a 2% slope of the land surface. The locations were chosen such that they appeared to be typical of the mapping unit. Care was taken so that when a location could not be established in the center portion of a yard or lot, the more peripheral location was representative.

3.4.2 <u>Operation of Infiltrometers</u>

Double ring flooding infiltrometers were used for the study. The rings were made of 0.32cm thick steel with a reinforced driving edge. A 30-cm diameter inner ring was driven into the soil to a depth of 20 to 30 cm, until the ring extended into the argillic horizon or until extreme soil resistance prohibited continuation. A 60-cm diameter outer ring was driven to a depth of about 12 to 15 cm. A 2-cm constant head of water was maintained in the inner ring using a 1 or 2.5-liter plastic graduated cylinder device. The graduated tube was clamped to the inner ring in a vertical and central position.

Three infiltrometers were placed about 1m apart in a triangular pattern at each location. The flooding infiltrometer was operated by: (1) flooding between the inner and outer rings to a depth of about 2 cm and manually maintaining the depth, (2) filling the graduated cylinder, (3) flooding in the inner ring around the graduated cylinder to a depth of 2 cm, (4) opening the water port(s) on the cylinder, (5) recording the water level in the graduated tube at regular time intervals and (6) refilling the graduated tube when necessary. The design of the graduated cylinder required that the water port(s) on the tube be closed to refill the chamber. This provided for somewhat erratic values when measuring extremely fast infiltration rates, <u>e</u>.<u>g</u>., forest conditions. The initial filling of the inner ring could not be achieved with consistent accuracy, therefore, the first infiltration rate value at 1 minute is the least accurate.

Examination of rainfall-runoff records for the Sudbury Suburban Watershed indicated that the watershed runoff was extremely sensitive to the 1-day antecedent rainfall. Based upon this analysis and supporting

soil physical conditions, initial infiltration tests were performed under moist antecedent soil moisture conditions. Twenty-four hours later in the same location a second infiltration test was performed under wet antecedent soil moisture conditions. The initial moist conditions were selected by initiating the tests 3 to 5 days following the last rainfall event and when the subsoil was judged to be in a moist state. The wet antecedent condition was taken at 24 hours after the initial 60-minute infiltration test. Thus the amount of water applied to achieve wet antecedent condition was proportional to the moist antecedent mass infiltration. The infiltration rings were normally removed after the initial test and replaced for the wet antecedent test due to the safety hazard and liability that could be incurred by leaving the rings unattended in a suburban yard or lot.

The infiltration rate was measured for a period of 60 minutes. This length of infiltration period was selected since the critical rainstorm duration for small suburban watersheds is generally 30 to 60 minutes or less. The infiltration curves normally would achieve a rather constant rate of decline within the 60-minute period for the wet antecedent test.

3.5 Undisturbed Soil Core Samples

3.5.1 Soil Core Sampling

Pseudo-undisturbed soil cores were taken with a modified Uhland sampler. The modified sampler was specifically designed for compacted urban soil conditions. It was constructed with replaceable heat-treated cutting tips, a thicker metal hammer shaft and a more sturdy basic framework. Vertical cores were cut by the force of the impact hammer. The

soil was retained in 7.62-cm inside diameter aluminum rings 7.62 cm in height. The samples were carefully cut to the correct length in the field. Care was taken to minimize the disturbance to the soil pores as the cores were cut to the proper length. The top end of surface soil cores was left with the natural conditions and thus they were not cut as were the bottoms. Soil cores containing coarse sand and gravel created problems in cutting the cores to length without leaving an irregular boundary. The boundary was smoothed after the gravel along the contact was removed, even though this damaged the pore network continuity. Each sample remaining in the metal ring was placed in a plastic bag and cardboard carton and was transported and stored in a refrigerator until use in the laboratory.

During the early portion of the study 12 surface cores and six subsurface cores were taken at various infiltration test locations. The samples were taken from the soil inside the inner infiltration ring 1 to 2 days after the wet antecedent infiltration test. Later it was decided to reverse the number of samples and take 12 samples from the subsoil and six from the surface. When core samples were taken at a location where there was no infiltration test, the samples were collected randomly over a 4-m^2 area.

3.5.2 Soil-Water Measurements

Soil-water characteristic data were measured by desorbing initially saturated undisturbed soil cores by applying positive pressure in selected increments. A soil-water characteristic tension table apparatus with 600-ml Pyrex funnels fitted with fine filter plates was used to hold the core samples during desorption. The positive pressure was applied to

the cores for 24 hours at each increment. The volume of water released was measured at pressures of -3.8, -10, -20, -30, -40, -50, -60, -100 and -345 cm water. The first increment, -3.8 cm water, is one-half the height of the cores or the average pressure created by gravitation forces.

The samples were again saturated for measurement of saturated hydraulic conductivity after Klute (1965). A constant head hydraulic gradient was maintained. Hydraulic conductivity at 25°C was calculated from flow data using the Darcy equation. The soil cores were oven-dried at 110°C for 24 hours and weighed. Bulk density, total porosity, macro-porosity (taken to be equivalent to the water volume released between 0 and -60 cm water tension), microporosity, differential water capacity and pore size distribution were calculated from the soil-water data. Pore size distribution data were calculated from soil-water characteristic desorption data using the formula for the rise of water in capillary tubes, thus assuming that only smooth non-tortuous vertical pores exist.

3.6 Thin Sections

Undisturbed soil clod samples were air-dried for approximately 48 hours and impregnated under vacuum in an apparatus described by Buol and Fadness (1961). A similar procedure was used to impregnate selected undisturbed soil cores after soil-water measurements. The undisturbed cores were oven-dried at 110°C for 24 hours prior to impregnation. A mixture of Castolite resin (600 ml), methyl methacrylate (400 ml) and benzoyl peroxide (3g) was used for impregnation. After the resin had been allowed to enter the soil pores under vacuum the samples were placed in an oven at a maximum of 35°C for about 3 weeks

and then allowed to further harden at room temperature for 2 to 5 weeks. The impregnation soil core containers normally had to be refilled with the resin mixture once during the 3-week period in the oven due to the volatile nature of the mixture. If the oven temperature exceeded 35°C, the resin mixture would harden too quickly and crack. The large resin blocks containing the soil cores had to be dried considerably longer than the small samples before the blocks could be satisfactorily sliced.

The impregnated soil clods were cut on a diamond blade saw, ground and polished, mounted on glass slides and ground to approximately 30 μm thickness. The terminology in Brewer (1976) was used to describe the soil pores and fabric of the 30-μm thin sections. A Wild M21 polarizing microscope was used to examine the thin sections. Panatomic-X black and white and Kodachrome II color slide films were used to record microscopic images.

The impregnated soil cores were cut with a wide diamond blade parallel to the original ground surface, ground and polished on one side and mounted on 10 by 10-cm double strength glass slides. The specimens were ground and polished to approximately 100 μm thickness, thus creating a macro thin section. Portions of the terminology in Brewer (1976) and Soil Taxonomy (Soil Survey Staff, 1975) concerning soil pores were used to qualitatively describe the soil pore network. Low power enlargement of the macro thin sections was obtained with a Wild M5 stereomicroscope. Panatomic-X black and white film was used with transmitted light to record pore networks. Ehodamine red dye in a 1:10 water mixture was used to delineate "active" pores.

3.7 <u>Mineralogical Methods</u>

3.7.1 <u>Clay Fraction Separation</u>

Subsoil samples from the nine type locations were fractionated to collect total clay (<2 μm) samples. The procedure involved the oxidation of the organic matter with hydrogen peroxide, washing with 1 <u>N</u> NaCl solution, washing (twice) with 60% methanol, dispersion with a Branson Sonifier Cell Disruptor (ultrasonic probe) for 3 minutes, sedimentation to remove most of the sand fraction, sieving through a 50-μm screen, sedimentation in a dilute Na_2CO_3 suspension for 12 hours, repeated (five times) centrifugation for 4 minutes at 750 rpm collecting the supernatant after each centrifugation and redispersal of sediment. The supernatant samples were flocculated, concentrated, washed (twice) with 60% methanol, redispersed in water, frozen in a dry ice trichloroethylene slurry and dried in a Virtis lyophilizer.

3.7.2 <u>X-Ray Diffraction</u>

Paste smear samples of the total clay fraction samples were prepared. About 1 ml of paste containing approximately 100 mg of clay was smeared on 25 by 75-mm glass microscope slides and allowed to dry. The paste had been saturated and/or slide treated in such a fashion to aid in the identification of clay minerals (Jackson, 1974). The sample treatments were: (1) Mg-saturation at room temperature, (2) Mg-saturation plus solvation with 10% glycerol solution at room temperature, (3) K-saturation at room temperature, and (4) K-saturation and heating at 350° and 550°C.

A Diano XRD 700 diffractometer with Ni-filtered CuK_α radiation was used to analyze the total clay samples. The diffractograms were used to identify the type and relative amounts of clay minerals.

3.7.3 <u>Differential Thermal Analysis</u>

Total clay samples were quantitatively analyzed for kaolinite and gibbsite by differential thermal analysis. Approximately 20-mg samples of Na-saturated freeze-dried clay was packed into platinum sample pans and were assessed in a high temperature cell of a DuPont 900 Differential Thermal Analyzer operating at a heating rate of 15°C per minute in an air atmosphere. The area under the gibbsite and kaolinite endotherms were planimetered and compared to standard curves (Dixon, 1965). Kaolinite #29 of the United Sierra Division, Aiken, SC and Ward's gibbsite were used for standards.

3.7.4 <u>Surface Area of Clays</u>

The total clay samples were used to determine the total surface, internal surface and external surface areas. A Quantasorb Surface Area Analyzer was used to determine values of nitrogen gas adsorption for 100 to 200-mg freeze-dried samples redried at 110°C. The weight of the adsorbed gas was calculated using a three-point B.E.T. equation (Brunauer <u>et</u> <u>al</u>., 1938). The calculation was based on the amount of gas desorbed at three partial pressures of nitrogen gas in a helium carrier. The volume of desorbed gas at each partial pressure was determined based upon calibration with a signal from a known volume of pure N_2 gas. The volumes of desorbed gas at the three partial pressures were correlated to the B.E.T. equation. The slope and intercept were calculated. The total external surface area was calculated from the relationship:

$$St = \frac{Xm \ (6.02 \times 10^{23}) \ Acs}{Ma}$$

where:

St = total external surface area of the sample;

Xm = weight of N_2 in the monolayer, Xm = 1/slope + intercept;

Acs = cross sectional area of an N_2 molecule in the monolayer (16.2 x 10^{-20} m^2) and

Ma = molecular weight of N_2 (28.01).

The specific surface area was calculated by dividing the total surface area by the sample weight. The value is termed the external surface area per 100g clay.

The total surface area (external plus internal) was determined for triplicate samples of about 200 to 300-mg freeze-dried clay samples redried at 110°C. The samples were saturated with ethylene glycol monoethyl ether and placed in a calcium chloride vacuum desiccator until the samples equilibrated (Carter et al., 1965; Heilman et al., 1965). The surface area was calculated from the weight gain assuming a uniform closely packed monolayer on both internal and external surfaces of the clay. The weight gain was assumed to be proportional to the weight gain from theoretically pure smectite clay minerals (Carter et al., 1965). The internal surface area is taken as the difference between the ethyl-ene glycol monoethyl ether surface area and the N_2, surface area. The values are reported as area per 100 g clay.

3.8 Chemical Methods

3.8.1 Cation Exchange Capacity

Cation exchange capacity of the clay samples were determined by a modification of the method used by Chapman and Jackson (1969). Triplicate total clay freeze-dried and redried at 110°C samples weighing about 200 mg were placed in 50-ml plastic centrifuge tubes and saturated with 1 N

NH_4OAc at pH 7. The samples were twice dispersed in 20 ml 1 $\underline{N}$ NH_4OAc at pH 7 by use of a Branson Sonifier Cell Disrupter with a micro-tip (ultrasonic probe) for 1 minute. The samples were centrifuged until the supernatant was clear. The samples were saturated with 1 $\underline{N}$ NaCl in a similar fashion and then washed three times with 0.01 $\underline{N}$ NaCl, each time dispersing with the ultrasonic probe. Care was taken not to loose any of the clay sample. The wet weight was recorded to calculate the volume of the entrained solution. The samples were next saturated three times with 20 ml of 0.1 $\underline{N}$ $Ca(NO_3)_2$, dispersed and centrifuged in the previous manner. The supernatant was collected and diluted. Sodium was determined using a Perkin-Elmer 403 Atomic Absorption Spectrophotometer. The cation exchange capacity calculated from these values is termed the CEC at pH 7 per 100 g clay. The cation exchange capacity of the total soil was calculated from the data and was termed the CEC at pH 7 per 100 g soil.

3.8.2 <u>Base Saturation</u>

The percent base saturation was determined by the sum of bases procedure (Soil Survey Investigations, 1972) for the purpose of classification of the soils. Exchangeable Ca, Mg, K and Na were extracted from 25 g fine earth fraction samples using 1 $\underline{N}$ NH_4OAc at pH 7. Ca and Mg were determined using a Perkin-Elmer 403 Atomic Absorption Spectrophotometer. K and Na were determined using flame photometry. Exchangeable acidity (Al + H) was extracted with $BaCl_2$-trietholamine at pH 8.2 from 5 g fine earth fraction samples. Ba was determined by flame photometry. Base saturation was calculated by dividing the sum of NH_4OAc extracted bases by the sum of NH_4OAc extracted bases and $BaCl_2$ extracted

acidity (sum of bases divided by sum of cations). Cation exchange capacity was calculated from these determinations. The cation exchange capacity from these values is termed the CEC at pH 8.2 per 100 g soil.

3.8.3 Fertility Analysis

Soil samples were randomly collected in the area immediately surrounding the location of infiltration tests for the three suburban sites. Samples were collected using an Oakfield probe for 0 to 2.5 cm, 2.5 to 7.5 cm and 7.5 to 15 cm depths. Duplicate samples were sent to the Soil Testing Section, Agronomic Division, North Carolina Department of Agriculture Laboratory for testing. The standard soil fertility analysis using the New Mehlich extractant was conducted. The analysis included determinations for pH, buffer acidity, organic matter, phosphorus, potassium, calcium, magnesium, manganese and zinc. Standard formula for fescue lawns were used to calculate lime, phosphorus and potassium requirements.

3.9 Rainfall-Runoff Analysis

Digital rainfall-runoff data collected by the U.S. Geological Survey for the Sudbury Suburban Watershed (Briar Creek tributary 6 at Sudbury Road, Charlotte, NC) was obtained. A flood stage and rainfall recorder obtained five-minute increment data.

Copies of selected 24-hour rainfall charts were obtained from the National Climatic Center for the N.C. State Station. The Schenck Forest and Pasture Watersheds are 3.7 and 3.2 km northwest of the weather station. Data were taken from the charts for 15-minute increment periods. Hydrograph charts from the Carl Alwin Schenck Forest and adjacent pasture watersheds were obtained from the NCSU School of Forest Resources.

Weekly charts were maintained for the two 90° V-notch sharp crested weirs. Discharge rates were calculated from stage data for 15-increment periods of selected hydrographs. Rainfall intensity—duration—frequency curves (U.S. Weather Bureau, 1955) which were derived from weather data from 1903 to 1951 for Charlotte and Raleigh, NC were used. The rainfall intensity—duration-frequency curves were compared to infiltration curves to generate estimated runoff coefficients for typical short duration high intensity summer rainstorms.

Standard methods were used to analyze the hydrograph and hyetograph data (Chow, 1964). Hydrograph separation was made for the selected rainfall-runoff events to produce a direct-runoff hydrograph. Data which were organized for the selected rainfall-runoff events included: date and time of peak rainfall intensity, duration of event (min), maximum rainfall intensity (cm), rainfall of event (cm), 1-day antecedent rainfall (cm), 2-day antecedent rainfall (cm), 5-day antecedent rainfall (cm), 10-day antecedent rainfall (cm), peak watershed discharge rate (cms), watershed lag time (min), time of concentration (min), total volume of watershed discharge (ha-m), calculated volume of direct-runoff (ha-m), average watershed discharge (cm), average watershed storage (cm) and percent watershed runoff.

4. RESULTS AND DISCUSSION

4.1 Survey of Research Sites

The three suburban research sites were selected to provide a variety of residential land use and soil conditions. Prior to urban development the dominant soil on each of the three sites was the Cecil series.

In 1938 the Sudbury Suburban Watershed was located in the rural lands surrounding Charlotte, NC (Figure 4.1.1). The land use pattern though dominantly of cultivated fields had a significant amount of pine and hardwood forest. After World War II and by 1951, the eastern portion of the basin along Sharon-Amity Road had been subdivided into residential lots (Figure 4.1.2). The residential development did not take place as a large speculative subdivision, typical of later development, but was constructed by a number of builders who worked on individual lots. This initial housing was located along the broad flat to gently sloping drainage divide, therefore the extent of grading and alteration to the soil was limited. Elsewhere on the watershed a portion of the previously cultivated fields had been reforested.

By 1956 additional residential lots had been developed in the eastern portion of the basin (Figure 4.1.3). A residential street was extended into the watershed from Central Avenue along the southern portion. The 1968 aerial photograph shows the extensive suburban development that occurred in the early 1960's (Figure 4.1.4). The watershed had been almost completely developed by 1968. The Windsor Park Elementary School was constructed at the northern edge of the basin. Extensive alteration of soil, by grading and filling was necessary to completely develop the watershed.