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AUTOMATIC SAMPLING TECHNIQUE TO DETERMINE EXTENT OF POLLUTION IN RUNOFF FROM AGRICULTURAL WATERSHEDS¹

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Concern over the quality of water in streams, rivers, and lakes is leading to a critical evaluation of how farming practices affect the quality of water coming from agricultural lands. The movement of agricultural chemicals from fields into waterways is of special interest.

Agricultural chemicals vary widely in the way they react with the soil and move across and within it. At one extreme are those that move freely with the soil water. These are readily leached into the ground water and the streams that drain the area. At the other extreme are those chemicals that are bound tenaciously to the soil colloids. Very little leaching of these compounds occurs, but they may still be removed from the area by erosion and be transported downstream with the eroded soil material.

In the spring of 1966, an experiment was undertaken on two instrumented runoff watersheds at Coshocton. Ohio, to study the movement of dieldrin, nitrates, and phosphates downward through the soil profile and laterally across the sloping surface of the hill, and to measure the losses resulting from erosion and runoff. The Coshocton station is located in an area of high-intensity summer rainstorms. These cause soil splash and severe sheet erosion. Single storms of 4 inches per hour for 15 to 60 minutes' duration have caused as much as 10 tons of soil loss from 2- to 3-acre watersheds. The soil in these watersheds is a well-drained Muskingum or slowly permeable Keene silt loam developed from residual shale or sandstone on 6- to 15-percent slopes.

In this report are described the sampling requirements and the equipment and techniques that are developed to obtain samples of runoff from the watersheds and preserve them prior to laboratory analysis.

SAMPLING REQUIREMENTS

Since the runoff rate and concentration of dissolved and suspended materials vary during any given runoff period, samples are required periodically throughout the runoff period. Because the greatest changes in rate of runoff and transported materials occur during the rising stages of the hydrograph, samples must be collected more frequently during this period than on the recession side (fig. 1). Thus the sampling equipment must be capable of collecting individual samples at variable predetermined time intervals throughout the runoff hydrograph.

The sampling equipment must operate unattended. It must remain on a standby basis, ready to operate at all times, detect initial runoff from the test watershed, and initiate and execute the programed sampling sequence. Concurrently, the runoff rate and time must be monitored and recorded in order that the relationship of each sample to the total can be evaluated.

The samples must be refrigerated immediately after they are collected and kept in this condition to minimize biological, chemical, and physical changes prior to laboratory analysis. Solids separated from the sample about 16 hours after collection are frozen and retained in this condition until analyzed.

 $^{^1}$ Contribution from North Appalachian Experimental Watershed, Corn Belt Branch, Coshocton, Ohio, and U_oS. Soils Laboratory, Beltsville, Md_o, Soil and Water Conservation Research Division, Agricultural Research Service, in cooperation with Ohio Agricultural Research and Development Center, Wooster, Ohio.

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Figure 1.--Simplified runoff hydrograph and sediment load from small agricultural watersheds.

SAMPLING EQUIPMENT

The sampling mechanism (fig. 2) consists primarily of the following standard products:³

(A) Liquid sampler (fig. 3) (series 46, Nappe Corp., Pelham, N.Y.), with the following main features:

(1) Self-priming pump (I) delivering 5 gallons per minute.

(2) Intake (\underline{L}) and exhaust (\underline{K}) hoses. They should be of Teflon tubing to minimize possible

loss or contamination of pesticide in runoff sample.

(3) Adjustable timer (<u>F</u>), with interval between samples of 0 to 60 minutes.

(4) Timer (\underline{G}), with sample pumping duration of 0 to 30 seconds.

(5) Heater and thermostat control (\underline{B}) to prevent formation of ice in pump system.

(B) Liquid-level switch (fig. 4) (Kenco Pump Div., Lorain, Ohio, model 108, ON-OFF range limit of 1 inch of water) adjusted to actuate or stop pump motor at specified water depth.

(C) Sampling tube in rate measuring flume (fig. 5) made in shop. Sampling tube ($\frac{1}{2}$ inch ID stainless steel) is pivoted at anchor mounted in floor near upstream end of flume. Float

³ Mention of commercial products and company names in this report does not imply endorsement by the U_{*}S_• Department of Agriculture over similar products and companies not named.



Figure 2,--Schematic of automatic hydraulic system designed to obtain 12 samples of runoff.



Figure 3,--Liquid sampler and control mechanism: A. On-off switch; B. heater thermostat; C. test switch; D. fuse; E. fuse; F. timer (interval between samples); G. timer (sampler pumping duration); H. motor; I. self-priming pump; J. solenoid valve; K. exhaust hose; L. intake hose; M. heating coil.



Figure 4,--Liquid level switch.



Figure 5.---Sample intake system in water.

attached to downstream end of this tube rises as water depth increases. Intake ports (3/16 inch diameter) are drilled in bottom side of tube and covered with copper window screen wire in shape of small bubble (fig. 2). One port is at upper end of tube and other two are one-third and two-thirds down length of tube.

(D) Solenoid valves (fig. 6) (Skinner valve, normally closed special purpose $\frac{1}{2}$ -inch full port A2 DE 4017, 115 volts, 60 cycles, or special purpose 3/8-inch V52LA 3012, 115 volts, 60 cycles). They deliver water sample from pump system to collector jugs in 12cubic-foot refrigerator (fig. 7). Single solenoid valve (Magnatrol Valve Corp., 67 Fifth Ave., Hawthorne, N.J., cat. No. 18 NR 22 V, $\frac{1}{2}$ -inch pipe (F), 3/8-inch valve port, normally open) serves as main valve (fig. 2) in delivery system. When closed, it diverts flow to sample valve, which is opened at same instant.

(E) Stepping switch (figs. 8 and 9) (Automatic Switch Electric Co., Dayton, Ohio, type 45, with terminal block, OFF-NORMAL switch, and stepping switch) to transmit timing signals to solenoid valves.

The liquid sampler has timing control features that are set to flush out the system, deliver to a 1-gallon glass jug a sample of predetermined amount, and reflush and resample at set intervals. The stepping switch provides a means of lengthening the interval between samples by multiples of the initial interval.

The selection of soft glass jugs for use as sample containers was based on a test conducted with various containers. Solutions containing one part per billion of dieldrin in water were stored for 11 days in the various containers in a refrigerator. Dieldrin remaining in the water was then determined. The results were as follows:

	Percent recovery
Container material	of dieldrin
Polyethylene	12
Polypropylene	76
Pyrex glass	92
Soft glass	
Teflon	93
Tin can	83

Some dieldrin was adsorbed by all materials, but highest recoveries were obtained from solutions stored in Pyrex, Teflon, and soft



Figure 6,--Solenoid valve system designed to obtain 12 samples of runoff,



Figure 7.--Sampling 1-gallon jugs (<u>A</u>) and Solenoid valve system (<u>B</u>) with exhaust (<u>C</u>) and supply (<u>D</u>) hoses in refrigerator (<u>E</u>) at sampling site





Figure 8.--Automatic switch consisting of terminal block, OFF-NORMAL switch, and stepping switch.



glass containers. The soft glass containers were selected because they were the least expensive.

OPERATION OF SYSTEM

The Coshocton station sampling program is operated as follows:

(A) The liquid-level switch (fig. 2) is set to actuate the pump motor when the flow reaches a depth sufficient to cover the intake ports and prevent air suction--an inch or more. To set this swtich to operate at the desired flow depth, it is necessary to insert dams in the flume inlet and outlet. The exact depths at which the switch is activated and deactivated are determined by in-place calibration. This calibration is important because it enables one to determine the water depth and flow rate at the time of each sampling. The hydrograph, or water-stage recorder graph, for each storm runoff period is scanned, and the time of the beginning depth is marked so that depths can be noted on the chart at the scheduled subsequent sampling intervals.

(B) The basic sampling timing sequence, started after actuation of the liquid level switch, is as follows: The pump starts immediately; with the main solenoid valve open and the sampling solenoid valves closed, the system is purged for 15 seconds. Then the main solenoid valve closes and the first of the sampling valves opens, permitting the first sample jar to be filled in about 20 seconds. Then the next cycle starts, so that the time interval between the initiation of taking successive samples is 3 minutes.

Without a manual modification to the timer setting (fig. 3) prior to the runoff period, the time delay of 2 minutes and 45 seconds for the initial sample is too large. This can be reduced to 15 seconds by manually advancing the timer prior to runoff. This causes the pump to start immediately upon closing the liquid-level switch.

A constant interval between successive samples of 3 minutes throughout the entire flow period is unnecessary. The interval can be increased during the latter part of the flow period, when flow rates and concentrations are more constant. This is accomplished by skipping certain positions on the stepping switch that selects the solenoid values to be opened in sequence. By connecting the 12 values to terminals 1, 2, 3, 4, 5, 7, 9, 11, 13, 17, 21, and 25, the time interval between samples becomes 3, 3, 3, 3, 6, 6, 6, 6, 12, 12, and 12 minutes, respectively. It is important that the timing between samples be checked carefully, because this time is used in conjunction with the record of the water stage recorders to determine the discharge rate at the time of sampling.

Flow rates during the winter and early spring are lower and more uniform than those during the summer. Since they also persist longer, the sampling time sequence should be changed accordingly. This can be done very easily on the existing equipment by changing the setting on the timer dial from a 3-minute interval to the desired time interval. For example, if a 10-minute interval is selected, the sampling time sequence then becomes 10, 10, 10, 10, 20, 20, 20, 20, 40, 40, and 40 minutes.

A test switch shorting out the water level control and an advancing switch to pulse the selector switch (fig. 9) enable field checking of the equipment and preparing it for automatic operation. The advancing switch is operated the number of times necessary to move the stepping switch to the starting point. The purge duration pointer on the timer interval control is run down to 15 seconds by closing the test shorting switch until the pointer is at the proper position, which is determined by trial.

The OFF-NORMAL switch (fig. 9) normally closed (N.C.) is set to stop the entire system after the last jug (No. 12) is filled. No more samples can be obtained even though the water level switch is still calling for samples. The system will have to be serviced by replacing the filled jugs with empty ones and by recycling the stepping switch.

(C) <u>Storm checks</u> on operation of the system are desirable. Periodic hand samples are collected by holding a gallon jug in the stream flow at the flume outlet at the same time the automatic system is delivering a sample to the jug. The water stage recorder graph is marked at this same time to note the flow depth and time. Both the hand and the automatic sample jugs are labeled so as to compare the results of laboratory analyses.

(D) <u>Servicing the system</u> after a storm runoff period is accomplished as follows:

(1) Remove all jugs containing samples and make complete notes of jug number and sample position number of each, as No. 1, 2, etc.

(2) Measure depth of sediment on floor of flume and its paved approach area and compute volume. Place samples of this material in Teflon bags and freeze preparatory to delivery for laboratory analysis.

(3) Disconnect intake line between intake pipe and pump.

(4) Force clean water back through line and intake pipe to flush out foreign material.

(5) Place suction end of this separation into can of clean water and pump through system and into spare jug placed at position No. 1.

(6) Open test shorting switch and close advancing switch to set stepping switch at initial point.

(7) Remove spare jug at position No. 1 and insert clean jugs at all positions.

SAMPLING INSTALLATION

A complete layout of a sampling installation appears in figure 10. The runoff measuring flume (D) with shelter (C) for float stilling well and water stage recorder has been in operation since June 1939 on this 2.68-acre cropped watershed. The average land slope is 13.6 percent. The precalibrated sheet metal flume is H-type, 3 feet deep, and has a flow rate capacity of 30 c.f.s. Maximum flow depth through this flume was 1.63 feet (7.51 c.f.s.) in June 1940.

Total cost of this installation in 1966 was approximately \$3,000, including equipment, materials, and labor. This does not include the cost of the flume and water stage recorder.

EVALUATION OF SAMPLING EQUIPMENT

The sampling equipment described in this report was installed on two watersheds at



Figure 10,--Runoff measuring and sampling installation, looking downhill from experimental watershed: <u>A</u>, switchbox for electric power; <u>B</u>, shelter for refrigerator, sampler pump, and controls; <u>C</u>, shelter for water-stage recorder, and stilling well for float and liquid-level switch; <u>D</u>, runoff rate measuring flume; E, intake tube for samples; F, purge discharge line from pump.



Figure 11.---Flow rate and percent solids in runoff on 7.59-acre watershed, Sept. 3, 1966.

Coshocton in the spring of 1966. Both watersheds are located on Muskingum silt loam. Watershed 128, containing 2.68 acres with an average slope of 13.6 percent, was treated with 200 pounds per acre each of nitrogen, phosphorus, and potassium incorporated into the plow depth and 5 pounds per acre of dieldrin incorporated into the surface 3 inches of soil. Watershed 192, containing 7.59 acres with an average slope of 15.8 percent, was used as a control. Corn was grown on both watersheds.

Although rainfall during the growing season was adequate for crop growth, it did not follow the normal distribution pattern. No storm occurred that caused runoff from the treated plot. Two sets of runoff samples were obtained from the untreated watershed, which was more susceptible to erosion than the smaller watershed. These samples were collected with the automatic equipment. Hand samples were also collected simultaneously several times during the storm. The percent solids was determined for those samples obtained from the automatic equipment and plotted as a function of time in figure 11. Only a few hand samples were taken-all at concentrations less than 0.6 percent. The composition of almost all the automatic samples agreed with that of the hand samples within 0.05-percent concentration.

Distribution of flow rates with time was typical of runoff from intense summer storms (fig. 11). Distribution of solids in the runoff, as determined from five samples at 3-minute intervals followed by two at 6-minute intervals, was about as expected--high concentrations during the first part of the runoff and diminishing during the period of lessening flow rates. The reason for the lower concentration value of the second sample is not apparent. This happened before. Additional observations and study may explain this unexpected variation.

SUMMAR Y

Great care must be exercised in collecting, preparing, and storing runoff samples to be used in studying the loss of pesticides and other chemicals from agricultural lands. Several individual samples must be collected at variable predetermined time intervals throughout the runoff hydrograph of each storm. Each sample must be related to runoff rate at the time of sampling.

A detailed description is presented of the construction and operation of automatic sampling equipment installed on two experimental watersheds at Coshocton, Ohio. The equipment is designed to operate unattended. It remains on a standby basis and initiates a predetermined sampling sequence at the onset of runoff from the watershed. Samples are collected in l-gallon glass jugs, which are stored in a refrigerator. The sampling mechanism turns itself off after the required number of samples is collected or if runoff stops prior to this.

The composition of samples collected by the automatic equipment compares very favorably with that of samples collected by hand from below the flume. ÷ 4

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