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Phosphate Movement from an Agricultural Watershed During Two Rainfall Periods

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The distribution of available phosphate between dissolved and adsorbed forms carried by stream water and sediments was measured at two sampling stations on a stream draining an agricultural watershed during high water flows. During an intense summer storm at the watershed the total available phosphate carried by the stream was 0.762 g per ha. In a less intense spring storm the loss was 0.028 g/ha. Changes in phosphate concentration during the storms were interpreted in terms of the adsorption isotherms of the sediments, which show that material derived from subsoils and stream banks has a large adsorption capacity. Concentrations of about 200 ppb (P) in water from fertile topsoil were reduced to less than 15 ppb as it moved downstream. Detailed prediction of phosphate concentrations in moving streams depends on adequate knowledge of the quantity, chemical characteristics, and origin of the sediment carried by the stream.

The rate of flow of streams draining water from the land shows wide variation with time, depending upon seasonal and local precipitation, soil characteristics, and other factors. The amount of nutrients carried by stream water must therefore show similar variation, depending both on the concentration of nutrients and on the rate of water discharge. During a 4-year study of the amount of nutrients carried by streams draining farmland in central Ohio, Taylor *et al.* (1971) found that essentially all the phosphate removal occurred during the periods of highest water flow, usually in the late winter and early spring. Sediment production is also a sporadic process, dependent upon the energy of moving water which is, in turn, related to stream flow. The rate of removal of phosphate either in solution or in adsorbed forms carried by sediments must therefore be estimated on the basis of measurements during periods of high stream flow. Since the conditions in streams at high flow stages may differ considerably from those under low flow conditions, and high flow conditions only occur for relatively short periods of time, estimates of long-term "average" rates of phosphate loss should be calculated only over long periods of time, preferably several years.

This paper reports a study of the amounts and forms of phosphate carried by two streams within the Mahantango watershed in east-central Pennsylvania during two periods of high flow. The watershed was chosen because of the availability of hydrologic data in a predominantly agricultural area having a limited input of urban sewage and industrial wastes. About 60% of the total land area is under cultivation and the remainder is made up of mountains, forests, meadows, and small towns. Corn is the major crop, with smaller acreages of tomatoes, barley, oats, and rye. Chickens for egg production, dairy cattle, and beef cattle are raised in scattered parts of this watershed. Taylor and Kunishi (1971) presented a detailed description of the Mahantango watershed.

EXPERIMENTAL

Samples were taken at two locations within the watershed during two separate storms. The first, occurring in July 1970, was a short duration high-intensity thunderstorm. The second, in May 1971, was a late spring rainfall of lower intensity but longer duration. The two sampling stations are designated Pillow and Malta after the towns near which they are located. Runoff passes the Pillow station from the 26-square-kilometer (10-square-mile) watershed of Little Deep Creek before entering the main channel. The Malta station is a U.S. Geological Survey gauge 6 miles downstream from Pillow, measuring all the water which drains from the 420-square-kilometer (162-square-mile) Mahantango watershed, including the Little Deep Creek region.

Stream gauge heights were recorded every 15 min at the Pillow station and at 60-min intervals at the Malta station. Suspended sediment concentration samples were taken at the same time. In addition, at four times during each event, approximately 19 l. of sample were taken from each location for determination of soluble phosphate on a clarified extract and for determination of available phosphate on the suspended material. Soluble phosphate in stream water was determined using the stannous chloride reduced molybdophosphoric blue color method (Jackson, 1958). The procedure [Taylor and Kunishi (1971)] which includes incineration at 500°C for 4 hr of a residue obtained by evaporating to dryness a clarified stream water extract, measures both organic and inorganic phosphates. All phosphate concentrations in water are expressed in ppb of P.

Suspended material was concentrated using a continuous

Table I. Water Volume, Soluble Phosphate Concentration, Suspended Material, and Available Phosphate on Suspended Material Obtained on July 9 and 10, 1970, and May 12 to 15, 1971, During Hydrologic Events in the Mahantango Watershed

Location and time	Water, l. × 10^6	Soluble phosphate as P		Suspended material		Available phosphate as P on suspended material	
		Concentration, ppb	Amount, g	Concentration (air-dried), ppm	Amount, kg	Concentration, ppm	Amount, g
July 9 and 10, 1970							
Pillow							
1300–1700	5	25.0	150	270	1,600	97	145
1700–2400	48	130.0	6200	1180[a]	55,600	72	4,070
0–0900	41	100.0	4100	270	11,100	64	717
0900–1400	13	40.0	520	80	1,040	85	91
Totals	108		10,970		70,340		5,023
		av = 102					
Malta							
1100–1900	104	2.0	210	100	10,400	52	540
1900–0900	710	16.0	11,400	600[b]	426,000	33	14,060
0900–1300	298	9.0	2,680	520	155,000	9	1,390
1300–1500	134	9.0	1,210	360	48,300	12	580
Totals	1246		15,500		639,700		16,570
		av = 12.4					
May 12–15, 1971							
Pillow							
1815–1500	1.8	6.4	11.5	22	38	125	4.8
1500–2100	1.8	41.1	74.0	191	344	104	35.48
2100–1000	2.9	32.0	92.8	59	169	115	19.4
1000–2100	1.7	6.8	11.6	19	33	118	3.9
Totals	8.2		189.9		584		63.9
		av = 23.1					
Malta							
1900–0100[c]	54	0.5	27	4	220	147	32.3
0100–1430	81	5.2	421	180	14,600	8	116.8
1430–1000	86	3.6	310	91	7,800	13	101.4
1000–2100	46	2.8	129	30	1,370	35	47.9
Totals	266		887		23,990		298.4
		av = 3.3					

[a] 2200 ppm maximum at runoff peak.   870 ppm maximum at runoff peak.   [c] 30-hr/time period.

flow centrifugation process on a Sorvall centrifuge. Available phosphate on suspended material was estimated by resin extraction following the method of Hislop and Cooke (1968). One- and-five-tenths gram of a strongly basic (trimethyl benzyl ammonium) anion-exchange resin, Dowex 21K, was equilibrated with 0.5 g of suspended material in 10 ml of water. The samples were gently stirred with magnetic stirring bars 1 cm long for 6 hr. The resin was removed from the mixture by trapping on a 60-mesh sieve. The phosphate sorbed on the resin was desorbed with 50 ml of Na_2SO_4 solution (35 g/l.). The desorbed phosphate was measured by the same procedure as that for soluble phosphate in stream water, except that the solution was acidified with H_2SO_4 instead of HCl. A correction was made for sulfate interference with the phosphate determination. In both acid systems the relative concentration error for phosphate analysis is estimated at 2%. The phosphate content of all suspended materials is expressed in ppm (dry weight) of P.

Phosphate potential curves were obtained for selected suspended material samples using the method of Taylor and Kunishi (1971). The samples used to determine these phosphate potential curves were taken at the time of maximum water flow in each event when the suspended material load was greatest. After transport to the laboratory the samples were centrifuged to concentrate the suspended material, and the phosphate concentration was measured in the supernatant. A subsample of the concentrated suspended material was air dried to measure solid content. The bulk of the sample was stored as a slurry without drying. The phosphate potential curves were then determined by diluting the concentrated slurry back to the original solid:solution ratio with solutions containing four concentrations of phosphate chosen to bracket the original concentration in the stream water (P_s in Table I). A fifth, phosphate-free solution was also used. All added solutions were 0.01 M in $CaCl_2$. Five drops of chloroform were added to each sample to retard microbial activity. After equilibration for 6 hr at room temperature under continuous agitation with a magnetic stirrer, the suspensions were centrifuged and the final concentrations of phosphate measured in the equilibrated solution.

RESULTS

Phosphate Budget. Data obtained during the first runoff event are presented in Table I. This event was characterized by moderately heavy rainfall over a short time period of about one-half day. The values for the amounts of suspended material are hourly or quarter-hourly integrations of water flow volume and concentration of suspended material. At the Pillow weir, the water flow and suspended material peaks coincided near 2000 hr on 7-9-70. At Malta, the peaks coincided at 0700 hour on 7-10-70, about 11 hr later.

The 420-square-kilometer (162-square-mile) watershed released about 32 kg (70.5 lb) of available phosphate during the 1-day period of sampling (6.3 cm total precipitation). This corresponds to 0.762 g per ha or 0.290 g per acre. At the
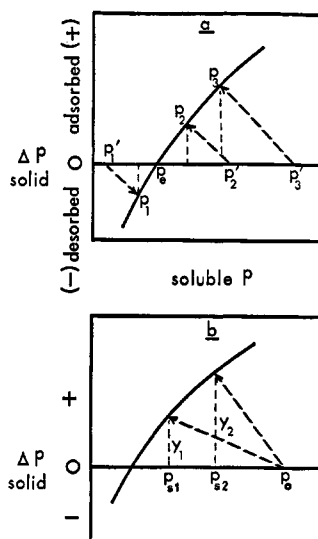
Figure 1. a. The determination of the phosphate potential curve by equilibrating a sediment sample with solutions having initial phosphate concentrations of P_1', P_2', and P_3'. P_1, P_2, and P_3 are the phosphate concentrations after equilibration. P_e is the phosphate concentration where neither adsorption nor desorption occurs. b. An estimate of the amounts of phosphate sorbed by the sediment (Y_1 and Y_2), and the soluble phosphate concentration (P_0) before sediment and solutions were mixed, from the phosphate potenital curve and P_s (measured phosphate concentrations following equilibration) values
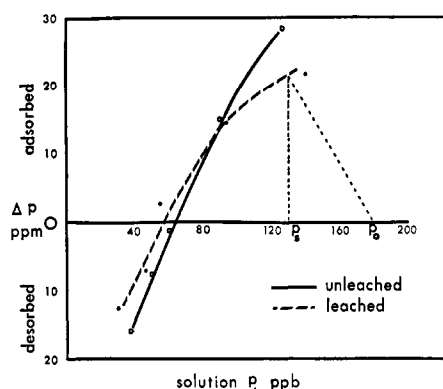

Figure 2. Phosphate potential curve for suspended material obtained at Pillow during peak runoff period (2200 ppm concentration of sediment), July 1970 storm

watershed outflow this was distributed about equally between available sorbed phosphate and phosphate in true solution.

Even though the average concentration of suspended material was higher at Pillow (0.065%) than at the main gauge (0.051%), the relative amounts of resin-extractable phosphate carried on the suspended material were only 31% of total available phosphate loss at Pillow, as compared with over 50% at Malta. Possible explanations for these results will be presented after phosphate potential curves have been examined.

Data obtained during the second event are presented in Table I. This event was characterized by light to moderate rainfall over a much longer time of 3 days (3.4 cm total precipitation). The runoff and suspended material peaks coincided at the Pillow weir at 1700 on 5–13–71. As in the first event, the peaks at Malta occurred 11 hr later. The entire Mahantango Watershed released about 1.18 kg (2.6 lb) of available phosphate during the 75-hr period of measurement.

This amount represents only 28 mg per ha or 10.5 mg per acre, an amount 30 times less than in the first event, even though the amounts of water passing by the gauge for the different times of measurement was only five times less.

The rate of phosphate loss is far less during this second event than during the first, even after allowing for differences in total amounts of water carried downstream. Not only was soil and stream bank erosion less during the second event, but even the soluble phosphate concentrations were consistently lower; this was an unexpected observation. Owing to the much lower concentration level of suspended material, the movement of phosphate past the Malta gauge during this second event was three times greater in solution than on suspended material.

The agricultural Mahantango watershed is thus seen to release stream water of very low soluble phosphate concentration to the Susquehanna River even during periods of moderate to heavy rainfall. The concentration is well below that required for optimum growth of aquatic plants.

Use of Phosphate Potential Curves to Interpret Suspended Material Stream Water Systems. Phosphate potential curves are ideally suited to help answer questions concerning the changes of distribution of phosphate between suspended material and stream water. Along with soluble phosphate concentrations, these curves yield information that can provide a basis for understanding reactions which take place when unequilibrated suspended material and stream water containing different levels of soluble phosphate are brought together.

Use of the phosphate potential diagram to obtain estimates of the amounts of phosphate sorbed and the initial soluble phosphate levels before sorption occurred requires that three inputs be manipulated in correct order: the potential curve itself; the soluble phosphate concentration at steady state; and the concentration of the suspended material. The interrelation among these factors may be seen in Figures 1a and 1b. In the determination of the phosphate potential curve, a known weight of soil or sediment is equilibrated with a set of solutions of known phosphate concentrations, P_1', P_2', P_3'. As the solid adsorbs or releases phosphate, these are changed to P_1, P_2, P_3. The amounts of phosphate adsorbed or released are then calculated from the changes in solution composition, and the potential curve is constructed as in Figure 1a. In such a plot the abscissa is most conveniently drawn at $\Delta P = 0$, so that the intersection defines the solution concentration at which no change would take place. This is defined as the P_e value. It may be assumed that during the equilibration process the relation between the amounts of phosphate adsorbed and the solution concentration will follow the vectors $P_1' \to P_1$, $P_2' \to P_2$, $P_3' \to P_3$. The slopes of these vectors are defined solely by the solid/solution ratio. Since the slope increases as this ratio decreases, high solid/solution ratios give much more satisfactory measurements of the shape of the adsorption isotherm, particularly where it is steep. At the sediment loads found in stream waters, artificial concentration of the sediment by centrifugation may be essential to obtain satisfactory results.

Once the potential curve has been measured the capacity of the sediment for adsorbing or releasing phosphate can be calculated at any desired sediment concentration. Figure 1b illustrates the difference expected when two suspensions of the same solid material but different solid/solution ratios are equilibrated with the same initial phosphate concentration, P_0. The more concentrated suspension will reduce the phosphate concentration to P_{s1}, adsorbing Y_1 ppm of its own weight

of phosphate, whereas the more dilute suspension will only reduce the concentration to P_{s2}, but take up Y_2 ppm. Where the intersection of the potential curve and the vector is known, this calculation may be reversed to estimate the initial but unknown phosphate concentration. Examples of the latter calculation are given below.

These calculations depend upon two assumptions. The first is that the curve has not been grossly altered by the process of sorption or desorption of phosphate. The second is that at the time of clarification of the stream water from the suspended material, the system was at steady state. This second assumption appears reasonable in that there has been a 3- to 5-day time lapse between sampling and completion of the separation of suspended material and water. A later study on the time rate at which equilibrium is approached supports this view.

The first assumption was tested experimentally with suspended material from the Pillow station obtained during the July 1970 storm. This sample, because of its lower capacity to sorb phosphate, was expected to exhibit larger changes in the phsophate potential curve when leached of phosphate than those at Malta. The original potential curve presented in Figure 2 was calculated as described by Taylor and Kunishi (1971). In the calculation no correction was necessary for the amount of phosphate remaining in the solution contained in the concentrated slurry; the large amount of dilution needed to restore the original suspended material/solution ratio reduced this concentration to a negligible level. After about 18 ppm of P was removed by equilibrating for 6 hr with distilled water, the curve was again determined. This 18 ppm of P represents about the amount of phosphate sorbed by the material under the conditions prevailing in the stream. The results in Figure 2 show a small shift of the unleached sample curve to lower solution P concentration with leaching such that the equilibrium phosphate concentration (P_e), *i.e.*, the soluble concentration at which there is neither gain nor loss of phosphate, is decreased from about 66 ppb of P to 56 ppb of P. The overall change is small, and the unleached suspended material yields a phosphate potential curve which provides an approximation of the phosphate potential curve of the original suspended material before any changes took place.

Phosphate Concentrations and Potential Curves. The data in Table I show a marked difference in the concentration of dissolved phosphate between the two sampling stations. In the July event, the average phosphate concentration (defined as total P/total water) was 102 ppb at Pillow and 12.4 at Malta, decreasing by a factor of 8.2. Although the amount of water at Malta is greater by a factor of 11.6, this difference in phosphate concentration cannot all be due to dilution of water from Pillow merging with the main stream and moving to Malta, since runoff derived from the rest of the Mahantango watershed will also contribute dissolved phosphate. Furthermore, when the Pillow sediment (Figure 3) mixes with the main stream sediment the resultant mixture yields the phosphate potential curve of the Malta sediment (Figure 2). This combined sediment, showing greater sorption of soluble phosphate than the Pillow sediment, will adsorb considerable phosphate from solution when the Pillow discharge mixes with the main stream. Measurements of the rate at which the phosphate is adsorbed from solution made during the study of the potential curve in Figure 2 show that the approach to a steady state is 90% complete within 1 hr. This, with earlier observations (Taylor and Kunishi, 1971), suggests that the Little Deep Creek and the main stream discharge were nearly equilibrated by the time the composite
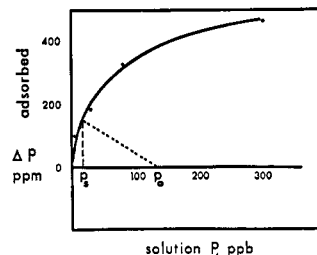

Figure 3. Phosphate potential curve for suspended material obtained at Malta during peak runoff period (870 ppm concentration of sediment), July 1970 storm
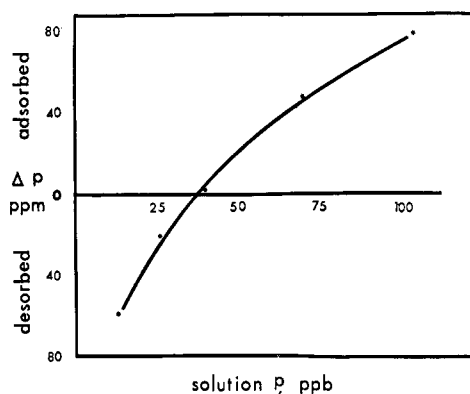

Figure 4. Phosphate potential curve for suspended material obtained at Pillow during peak runoff period (190 ppm concentration of suspended material), May 1971 storm

discharge arrived at Malta, 6 mi downstream. Equilibrium was certainly reached at the time the samples were centrifuged. Similar considerations apply to the May event.

The most important feature of the potential curves obtained from the Pillow station during the July 1970 storm (Figure 2) is that they compare closely with those previously found for 1:1 or 1:2 mixtures of fertilized topsoil and phosphate-deficient subsoil from the watershed of the same stream (Taylor and Kunishi, 1971). The phosphate sorption behavior of the suspended material sample is thus consistent with the movement and mixing of material from topsoil and stream bank erosion to be expected during an intense storm.

The companion data for the suspended material obtained at the Pillow weir during the event of May 1971 are presented in Figure 4. Here the P_e for the suspended material is lower than the July sample and very close to the concentration of dissolved phosphate in the original sample (P_s). The phosphate buffering capacity of the suspended material is also greater and the system more stable in time. All these features would be characteristic of suspended material with a larger proportion of phosphate-deficient subsoil material.

The data for the samples at the Malta station are presented in Figures 3 and 5. The P_e of both are very low, less than 5 ppb of P, which is consistent with the concentrations found in the original samples, and the potential adsorption capacities of the suspended material are very high. The sample in the May 1971 storm represents extremely phosphate-deficient material with a very large phosphate "fixing capacity."

Redistribution of Phosphate During Streamflow. In analyzing the changes in the distribution taking place in the stream, it is useful to remember that the data from the Pillow and Malta stations represent two different sets of conditions. The results obtained at the Pillow weir represent runoff and
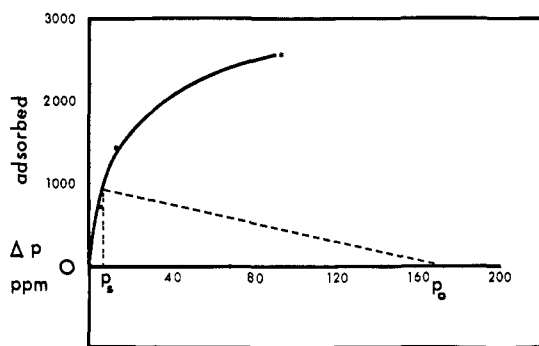
Figure 5. Phosphate potential curve for suspended material obtained at Malta during peak runoff period (180 ppm suspended material concentration), May 1971 storm

sediment in a stream fed directly from agricultural land. The Malta station is located in a small river draining the whole agricultural watershed; before reaching this point most of the water will have traveled a further 6 mi in the stream channel.

The magnitude of the phosphate sink provided by the suspended material at the two locations depends on the character of the material as well as the solid/solution ratio and the initial phosphate concentration. In the July storm at the Pillow weir the highest phosphate concentration was 130 ppb of P when the concentration of suspended solids was 2200 ppm; this latter is higher than the values presented in Table I, which are averages taken over the stated time intervals. Extrapolation backward along the appropriate vector from the point on the potential curve corresponding to 130 ppb (Figure 2) indicates that the initial phosphate concentration was close to 180 ppb of P, and that the sediment had adsorbed close to 22 ppm of P at the time of sampling. The estimate of 180 ppb of P for the initial concentration of phosphate in the water entering the stream is a maximum figure; if a lower soil/solution ratio is used, the value will be less. The figure does, however, correspond closely to that measured in solutions equilibrated with fertile topsoil from the Little Deep Creek area (Taylor and Kunishi, 1971).

The equilibrium concentration of phosphate for the Malta sample in the July storm is less than 5 ppb, but the phosphate concentration observed during the event was again higher, reaching a maximum of 16 ppb with an average of 12.4 ppb. A similar calculation, using 870 ppm as the sediment concentration, indicates that the latter had adsorbed about 140 ppm of P (Figure 3). This corresponds to a total of about 90 kg of P. The total resin-extractable phosphate was 16.6 kg of P, indicating that only one-fifth of the amount adsorbed remained available. The capacity of the suspended material for further adsorption of phosphate is very large.

The extremely high fixing capacity of the sediment carried by the main stream is even more clearly shown in the data from the May event. Assuming that the potential curve of Figure 5 is representative of the entire event, the average phosphate concentration of 3 ppb indicates that 600 ppm of P, or a total of about 15 kg, had been adsorbed by these sediments. The phosphate dissolved in the water represents only 6% of that which had been absorbed by the solids. Resin extraction indicates that only one-twentieth (0.3 kg) of the adsorbed remained available.

The difference in character of the suspended materials in the two events implies that materials of different origins are mixing in different proportions. Of the two events, the

amount and concentration of sediment at the Pillow weir were much higher in the July storm, suggesting that on this date the suspended material at the Malta weir contained a much higher proportion of topsoil, giving a composited sample of lower fixing capacity than in May. The very high fixing capacity of the May storm may represent mainly material eroded from stream banks weakened during the spring thaw conditions, or material containing a higher proportion of iron-rich oxides draining a mine waste region. The physical characteristics of the suspended material have been observed to change sharply at the Malta station when material derived from the farthest reaches of the watershed, where a coal mine waste region is located, arrives at the station at and near the peak runoff period. Suspended material that was previously brown becomes nearly black. On these dark samples from Malta, isotopic dilution measurements of available phosphate by ^{32}P tracer methods gave abnormally high values when compared with the resin extraction values presented in Table I. Amer *et al.* (1969) have observed high rates of ^{32}P fixation with samples enriched with precipitated iron oxides. In such samples the tracer method grossly overestimates "available" phosphate.

The source of the suspended materials cannot be identified on the basis of the data available. The most important point remains the observation that the solid material discharged by the main watershed stream had a large capacity for the adsorption of phosphate from solutions containing more than 10 to 15 ppb of P.

Estimates of Phosphate Concentrations in Initial Runoff. The calculations presented above suggest that the initial phosphate concentration in the runoff above the Pillow weir in the July storm was about 180 ppb of P. In the May event the suspended solids were more nearly in equilibrium and the initial concentration was probably closer to 43 ppb of P (not diagrammed in Figure 4).

Extrapolation of the data from the Malta samples is less reliable than those from Pillow, owing to the high phosphate sorptive capacity of the Malta sediment and the uncertainties in the change in sediment load during the event. If, however, the maximum phosphate concentrations and sediment loads presented in Table I are used with the potential curves in Figures 3 and 5, the data suggest that the initial runoff concentrations were about 130 and 170 ppb of P.

In the low intensity storm of May 1971, the initial phosphate concentration is lower at Pillow, 43 ppb of P, than at Malta, 170 ppb of P. A look at the amount of runoff at Pillow compared to that at Malta during these two storms indicates that there was less than one half as much runoff, proportionally, at the sub-watershed draining through Pillow during the low intensity storm than during the high intensity one (8.6 *vs.* 3.1%), so that there was relatively less hydrologic activity in the Pillow sub-watershed during this second event. This is reflected in the fact that the 43 ppb of P estimated as being the initial level of soluble phosphate at Pillow is a level close to that found in this stream during base flow, when suspended material is negligible (Taylor and Kunishi, 1971).

Although the uncertainty is rather large, it is clear that the entire body of data is consistent with the suggestion that runoff from agricultural land can carry phosphate concentrations ranging from 50 to 200 ppb or more, but as these solutions are exposed to phosphate-deficient sediments derived from subsoils in stream banks or other sources, these concentrations are reduced to 10–15 ppb. These adsorption reactions take place as the materials are mixed and transported in the moving water. There is also clear evidence that only a fraction of the

phosphate that is adsorbed remains available; the rest is fixed by the stream sediments in forms that cannot be easily extracted.

SUMMARY

Amounts of soluble phosphate, suspended material and the available phosphate carried by it, and water flow were determined at two stations located on the Mahantango watershed during two storms in July 1970 and May 1971. The lower station measured total discharge from the watershed; the upper provided a measure of the contribution of purely agricultural land.

During the 1-day sampling period in July 1970, the 420-km^2 watershed released 32 kg of available phosphate (as P). At the discharge point 15.5 kg were in solution, with an average concentration of 12 ppb of P, and 16.5 kg were carried as available phosphate on the suspended material. In the May storm 1.17 kg of available P was lost over a 3-day period; 75% of this was in solution at a concentration of 3 ppb of P, with the remainder carried by the suspended material.

The phosphate potential curves of the suspended material in the Little Deep Creek region (upper station at Pillow) are characteristic of a mixture of eroded topsoil and stream bank material. The soluble phosphate concentration was in the range of 40–100 ppb of P at steady state, but was changing rapidly when eroded material and runoff were brought together.

The suspended material samples from the foot of the stream at Malta (lower station) have a high phosphate sorption capacity and are characteristic of stream bank and subsoil materials. The soluble phosphate concentration in equilibrium with these materials is between 3 and 13 ppb of P.

Comparison of the distribution of phosphate between water and suspended material at the two sampling locations demonstrates that as the suspended material is moved and mixed downstream, phosphate is sorbed from solution. The amount of available phosphate per unit weight of suspended material carried by the stream is much lower than that on topsoil.

The whole system is highly dynamic. While the changes in these two events are consistent with the known character of the soils, subsoils, and the stream bank materials of the area, detailed predictions of the changes that will take place depend greatly on a better knowledge of the origins of the actual suspended material carried by the stream in any particular event. The most confident general prediction that can be made is that where stream water carries suspended material of high sorptive capacity, the concentration in water will be reduced to levels of 10–15 ppb of P.

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