oxides of titanium (*10*). The intense absorption spectra of these oxides can be explained by the model of narrow d-bands formed by d-orbital overlap proposed by Goodenough (*11*). The t_{2g} ground state orbitals overlap directly across shared faces in the case of Ti_2O_3 and through the intermediary of the oxygen *p*-orbitals in the case of TiO_2. The narrow bands thus formed lie within the forbidden energy gap of the structures.

The spectra of transparent kyanite crystals show no features below 40,000 cm^{-1}, so the Ti^{+++} absorption lies well within the forbidden gap. The aluminum-oxygen octahedra in kyanite are arranged with shared edges forming chains parallel to the *c*-axis (*4*). The closest aluminum-aluminum distances along the main chain are 2.79 Å, which may be compared with the titanium-titanium edge-sharing distance in Ti_2O_3 of 2.79 Å (*12*). This distance is less than the critical separation required for one lobe of the t_{2g} ground state orbital to overlap an adjacent orbital to form a narrow band. Since the concentration of Ti^{+++} in kyanite is very low, this model further requires that the Ti^{+++} ions be clustered to permit a sufficient number of Ti^{+++} cation neighbors for collective electron behavior.

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Sedimentary Phosphate Method for Estimating Paleosalinities

Abstract. *The widespread occurrence of sedimentary phosphate in argillaceous sediments provides the basis for a new method of paleosalinity estimation. Sedimentary phosphate contains iron- and calcium-phosphate fractions, the relative proportions of which are sensitive to salinity of the water at sites of deposition. The sedimentary phosphate method provides direct estimates of paleosalinity throughout the freshwater to marine range.*

Recently great efforts have been made to develop geochemical techniques to estimate environmental parameters such as temperature and salinity. While the oxygen isotope method for paleotemperatures (*1*) has been employed extensively, efforts to refine methods for paleosalinities have been less successful. Most paleosalinity methods are based on Goldschmidt's (*2*) classical observations on the occurrence and distribution of trace elements in different sedimentary environments. For example, Degens *et al.* (*3*) differentiated marine from freshwater shales on the basis of spectrochemical analysis for boron, gallium, and rubidium. Similarly, Potter *et al.* (*4*) demonstrated that each of the elements boron, chromium, copper, nickel, and vanadium is more abundant in marine than in freshwater argillaceous sediments. The utility of trace element paleosalinity indicators as employed by such authors is limited, because the absolute abundance of an element in a sediment sample depends upon the trace element content of the detrital minerals, grain size, mineral composition, organic content, sedimentation rate, and so forth, as well as upon salinity of the water at the site of deposition. Therefore, the abundance data for a particular environment cluster around a mean value, but there is sufficient spread that significant distinctions cannot be made between environments that differ by small increments in salinity.

Most recent research has concentrated on developing the single element boron method for paleosalinity determination (*5*). Several studies have tested the conditions required for the boron method to be a useful indicator (*6*). The results to date (*7*) indicate that when the whole-rock boron is below 50 parts per million the sample probably is freshwater in origin, while a boron content above 50 parts per million suggests marine conditions. It appears that only a general distinction between freshwater and marine sediments can be made. The several factors that control the abundance of boron mask the sensitivity of the method to all but the extreme environmental salinity variations. The absolute abundance depends on the boron content of the detrital sediment, as well as upon conditions in the depositional basin. Furthermore, the conditions leading to boron adsorption in the depositional environment are imperfectly known (*8*). It appears that a sensitive and generally applicable method of paleosalinity estimation will have to be based on a new principle. It is the purpose of this paper to propose such an alternative.

The new method is based on the discovery that both recent and ancient argillaceous sediments contain small quantities of sedimentary phosphate that are distributed widely in sediments from different environments. The sedimentary phosphate can be extracted selectively from the sediment and differentiated into fractions whose relative proportions are sensitive to the salinity of the water at the site of deposition. The phosphate fractions are interdependent variables affected by salinity. The ratio between them is independent of their absolute abundance in the sediment and many of the difficulties inherent in the methods discussed above are avoided. For convenience, the new method of estimating paleosalinities is called the Sedimentary Phosphate Method.

Compared to the small concentrations found in natural waters, relatively large amounts of phosphorus occur in sediments. Sediments act as a phosphorus reservoir in natural systems. Mortimer's (*9*) classic study of lakes demonstrated that phosphorus is associated with iron in lake sediments and that under certain conditions it is released to the lake water. Similarly, there is exchange between water and the sediment reservoir in estuarine and marine environments (*10*). The mineral or chemical composition of the phosphorous compounds in estuarine and marine sediments has not been determined, although it is well known that marine phosphorite is essentially $Ca_{10}(PO_4)_5(CO_3)(F,OH)_2$. The objective of the present study was to characterize the types of sediment phosphorus that exist over a range of salinities and to determine the extent of any interaction between the sediment and its environment.

The sediment phosphorus was characterized by means of a fractionation scheme proposed by Chang and Jackson (*11*) for soil phosphorus. In soils the predominant phosphorous compounds are variscite, $AlPO_4.2H_2O$, strengite, $FePO_4.2H_2O$, and hydroxyapatite, $Ca_{10}(PO_4)_6(OH)_2$, each of which ordinarily is very insoluble. However, each compound may be extracted selectively by reacting in sequence with $0.5N$ NH_4F (for variscite), $0.1N$ NaOH (for strengite), and $0.5N$ H_2SO_4 (for hydroxyapatite), and by carefully controlling the conditions one can determine the relative amounts consistently. Table 1 shows the results obtained from a group of sediment samples extracted following this scheme.

The majority of samples are from the Rappahannock River estuary, a tributary of Chesapeake Bay. Sediments derived from the soils associated with crystalline rocks in the Virginia Piedmont enter the head of the estuary and as they are transported through it are exposed to progressively higher salinities. In Table 1 the designations R-58 and so on identify stations located 58, 50 and so on nautical miles from the mouth of the estuary. The maximum bottom water salinities to be expected at each station are given in the third column. These have been determined by standard hydrographic work extending over nearly 20 years; typical horizontal and vertical salinity profiles and seasonal averages are summarized in Stroup and Lynn (*12*). The salinity values listed correspond with those of the sediment pore waters at each station, in general, within 1 per mille. Sediment samples with similar provenance but exposed to salinities higher than those encountered in the Rappahannock were obtained from the lower York River and Chesapeake Bay (*13*).

The total sedimentary phosphate probably is related both to the phosphate content of the source sediment and to biogeochemical accumulation processes in the depositional basin. Each of the values for the phosphate fractions listed in Table 1 represents an average of four or five samples a few centimeters apart in cores of the upper 50 cm of sediment. Iron and calcium phosphate are the dominant fractions found in these estuarine sediments. Their relative proportions vary systematically along the estuary as shown by the final column of the table. The ratio in this column employs the molar concentrations of calcium and iron phosphate found in the sediment and is referred to subsequently as the calcium-phosphate fraction ($F_{Ca\text{-}P}$). The calcium-phosphate fraction, as thus defined, is independent of the absolute abundance of calcium phosphate or of iron phosphate.

Over the observed salinity range the calcium-phosphate fraction is a very sensitive salinity indicator. Consider the values obtained from sediment collected at different depths at one cross section, R-12 for example. Because the water column is stratified with less-saline water above and more-saline below, the sediment in shallow water is exposed to lower salinities than sediment in deeper water. The sediments from deeper water give higher calcium-phosphate fractions than those from shallower water in the same section. These variations are explained by the higher average salinity of the deeper waters. Even where the seaward increase in salinity between sections is small, for example between R-7 and R-2 or R-21 and R-12, the calcium-phosphate fraction is affected. These variations indicate that the calcium-phosphate fraction is sensitive to changes in salinity of 1 per mille or less.

The Rappahannock and lower Chesapeake Bay data were used to construct the accompanying figure, along with similar data listed in Table 1 from several samples that fill out the extremes of the salinity range (*13*). The line of best fit can be described by the following equation: calcium-phosphate fraction = .09 + .026 salinity. The correlation coefficient is .97, which indicates that considerable confidence can be placed in the general trend of the calcium-phosphate/salinity relationship that has been determined. The observed points have a standard deviation of 2.0 per mille salinity from the line. The dashed lines on the figure represent 95 percent confidence limits, that is, ± 4 per mille. The separation of these two lines is a measure of the uncertainty of the calcium-phosphate/salinity relationship from present data. Much of the uncertainty is caused by scatter due to the inconstant position of isohalines in the Rappahannock estuary where seasonal variations in freshwater inflow cause a fluctuation in salinity at any cross section. Clearly, the calcium-phosphate fraction depends on the ratio between the chemical activities of iron and calcium in the sediment pore water (*14*). The chemical activity of iron decreases in the transition from freshwater to marine muds, while the chemical activity of calcium increases, following the increase of salinity. The relatively high calcium-phosphate fraction for those samples nearest the head of the Rappahannock estuary probably reflects the still appreciable calcium activity of the influent fresh river water, where pCa = 4. In sediments from rivers and lakes where the water is soft (poor in alkaline earths) the sedimentary phosphate should be iron-phosphate predominantly. Marine sediments

Table 1. Sedimentary phosphate fractions in some recent sediments.

Section No.*	Depth (m)	Salinity (per mille)	Al-PO_4†	Fe-PO_4†	CaPO_4†	Fe + Ca	$\frac{Ca}{Fe + Ca}$
R-58	8	1.5	0.97	15.19	3.79	18.98	0.20
R-50	5	4	.58	8.86	3.27	12.13	.26
R-39	5	10	1.28	11.66	7.01	18.67	.33
R-26	3	14	1.34	8.10	6.46	14.56	.44
R-26	6		1.01	6.66	4.69	11.10	.42
R-21	8	17	0.96	5.40	4.88	10.43	.45
R-21	9		1.82	3.88	4.17	8.05	.52
R-21	14	18	1.61	4.46	6.33	10.90	.53
R-12	11	20	0.54	2.78	4.76	7.54	.63
R-12	15		.29	2.58	4.91	7.49	.69
R-12	20	21	1.51	2.58	5.66	8.24	.71
R-7	14	21	0.24	2.91	5.30	8.21	.64
R-7	21	22	.37	2.06	5.25	7.31	.72
R-2	8	23	.79	2.16	7.91	10.07	.77
R-2	12	23	.66	1.83	6.99	8.82	.79
YCB-4	14	23	1.80	3.14	6.33	9.37	.68
YCB-3	12	25	1.70	2.46	7.45	9.91	.75
YCB-2	11	27	1.88	2.72	9.74	12.46	.78
YCB-1	10	29	0.95	2.08	9.51	11.59	.82
CT-1	1362	36	1.29		11.30	11.30	1.00
BP-154		35	0.19	0.24	13.76	14.00	0.98
LMSp	7	0	.32	4.55	0.23	4.78	.05
SCSR	446–5075	35	.53	0.40	9.36	9.76	.96

*R, Rappahannock River samples; YCB, lower York River and Chesapeake Bay samples; CT, Cariaco Trench sample; BP, Bermuda Platform sample; LMSp, Lake Murray, South Carolina, sample; SCSR, 16 South Carolina continental slope and rise samples. †Sedimentary phosphate was determined on 1-g, air-dried samples and is expressed in micromoles of phosphate per gram.

contain calcium phosphate almost exclusively, the final line of Table 1 being the average of 16 typical continental slope and rise samples.

In order to determine if sedimentary phosphates occur in ancient sediments a group of Paleozoic shales and clays were analyzed by the modified Chang and Jackson procedure, with the results shown in Table 2. The clays and shales contain sedimentary phosphate varying in amount from 1 to 32 μmole/g. This variability in total phosphate probably reflects both grain size variations and environmental differences. The shales are not as fine grained or as homogeneous as the recent muds. Also, the clays and shales from the Pennsylvanian coal measures were deposited under deltaic conditions whereas the Devonian and Mississippian shales are more typically marine sequences. Table 2 demonstrates that it is useful to calculate the calcium-phosphate fraction in order to compare samples with variable amounts of total sedimentary phosphate. When this fraction is used to estimate the salinities by employing Fig. 1, the values in the final column of Table 2 are obtained.

The results are consistent with previous stratigraphic and fossil evidence. The Bedford shale, which in places contains a few brachiopods, appears to be a normal marine formation. In view of the controversial theories on the origin of red beds, however, it is interesting that the red Bedford appears to be normal marine. Samples 4 to 6 from the lowest Bedford may be transitional to the underlying Devonian shales. As a group the Devonian shales are mostly marine, but certain samples of the Cleveland and Vermilion black shale facies and the Chagrin gray clay shale seem to be of brackish water origin. If these inferences about the environments of deposition can be substantiated, they will add a new dimension to our comprehension of the paleoecology of the shales from Ohio.

The results for the Pennsylvanian sediments suggest that it should be possible to make detailed paleoecologic interpretations for the different rock units that are part of the cyclical sequences between marine, brackish, and freshwater conditions so typical of the coal measures. The results in Table 2 are highly consistent with the interpretations based on fossil evidence heretofore available.

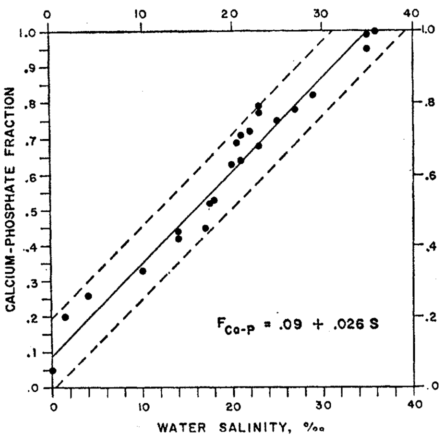

Fig. 1. Relation between calcium-phosphate fraction and bottom water salinity (per mille) for some recent sediments.

The preceding observations show that there exist in both recent and ancient argillaceous sediments detectable fractions of sedimentary phosphate. In recent sediments the relative proportions of iron phosphate and calcium phosphate are sensitive to changes in the salinity of the water in which the sediments are deposited, and the mole fraction of calcium phosphate increases continuously over the normal salinity range. If the iron- and calcium-phosphate fractions in ancient sediments formed in a manner similar to those in the recent and if the ratio between them is preserved, then the calcium-phosphate mole fraction can be used to estimate paleosalinities.

The paleosalinity of a sample whose iron- and calcium-phosphate fractions have been determined by the controlled extraction procedure employed here may be estimated from a diagram such as Fig. 1. For example, a calcium-phosphate fraction of 0.40 estimates a paleosalinity of 12 per mille ± 4 per mille; one of 0.90, a paleosalinity of 31 per mille ± 4 per mille. The variability of 4 per mille represents the imprecision of present data. It can be expected that future work will increase the precision of the paleosalinity estimate.

The advantages of the Sedimentary Phosphate Paleosalinity Method over existing techniques are as follows: (i) A definite estimate of any extreme or intermediate salinity over the freshwater to marine range is provided. (ii) The estimate depends on the ratio between calcium phosphate and total phosphate, so neither the ratio nor the precision of the estimate is affected by variations in the absolute abundance of phosphate, calcium, and so forth. (iii) The estimate depends on the composition of a chemical precipitate resulting from the interaction between sediment

Table 2. Sedimentary phosphate in paleozoic shales and clays.

Formation	Al-PO_4	Fe-Po_4	CaPO_4	Fe + Ca	$\frac{\text{Ca}}{\text{Fe + Ca}}$	Salinity* (per mille)
			Mississippian, Northern Ohio			
1. Red Bedford	0.15	0.75	6.32	7.07	0.89	31
2. Gray Bedford	.14	.81	16.80	17.61	.95	33
3. Gray Bedford	.03	.35	6.67	7.02	.95	33
4. Gray Bedford		.54	10.99	11.53	.95	33
5. Gray Bedford	.20	.08	2.69	2.77	.97	34
6. Gray Bedford	.10	.86	3.36	4.22	.80	27
			Devonian, Northern Ohio			
7. Cleveland Facies	.50	3.14	3.23	6.37	.51	16
8. Cleveland Facies	.32	2.90	6.67	9.57	.70	24
9. Vermilion Facies	.13	0.99	7.34	8.33	.88	30
10. Vermilion Facies	1.06	2.07	2.26	4.33	.51	16
11. Huron Facies	0.10	1.75	5.59	7.34	.76	26
12. Huron Facies	.29	0.51	8.09	8.60	.94	33
13. Olmsted Facies		.10	7.34	7.44	.99	35
14. Olmsted Facies	.06	2.31	20.48	22.81	.90	31
15. Chagrin	.20	1.53	3.98	5.51	.72	24
16. Chagrin	.15	0.38	1.67	2.05	.82	28
17. Silty Chagrin	.01	.75	3.04	3.79	.80	27
18. Silty Chagrin		.24	4.87	5.11	.95	33
			Pennsylvanian, Western Pennsylvania			
19. Lower Kittanning, marine zone	.12	.13	31.76	31.89	.99	35
20. Lower Kittanning	.17	.62	14.80	15.42	.96	34
21. Lower Kittanning	.16	1.05	2.77	3.82	.72	24
22. Lower Kittanning	.69	0.27	0.81	1.08	.75	25
23. Lower Kittanning underclay	.23	.64	.78	1.42	.55	18
24. Mercer Black underclay	.22	.91	.59	1.50	.39	12
25. Mercer Gray underclay	.02	.43		0.43		0
26. Flint clay	.19	.56		.56		0
27. Plastic clay	.44	1.99	.24	2.23	.11	1

*Salinities estimated from Fig. 1.

and calcium, a major chemical constituent of seawater. Great sensitivity is possible, because of the large increase in abundance of calcium in passing from freshwater to marine salinities. With trace element indicators the range of abundances is small, so the sensitivity is low, and a complex chemistry controls their incorporation into the sediment. (iv) The chemical technique for extracting the phosphate and determining the different fractions is simple and inexpensive.

It seems likely that the iron- and calcium-phosphate fractions formed in ancient sediments in much the way that they do today, if the composition of the ancient seas was similar to those of the present. Once the mixed calcium-iron-phosphate crystallized, the insolubility of the various components in ordinary pore solutions should prevent any alteration in composition during lithification. Of course, the extent to which sedimentary phosphate may be used to estimate paleosalinities in sediments that undergo progressive metamorphism needs to be investigated. Also, once the applicability of the method to a variety of sedimentary rocks is established, the possibility exists that it may be used to study the paleosalinities of ancient seas where the composition, compared to recent oceans, is open to question.

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15. The results were obtained while I was supported by FWPCA research grant WP-00651-04.

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Gastropod Urosalpinx: pH of Accessory Boring Organ while Boring

Abstract. *Recent development of a microelectrode has enabled the first continuous recording of the* pH *of the secretion of the normally functioning accessory boring organ of the shell-boring predatory snail* Urosalpinx. *The recording was made in an incomplete borehole in a glass-shell model. The minimum* pH *recorded was 3.8; hitherto the secretion had been considered neutral.*

Penetration of hard, densely mineralized, calcareous substrates by lower plants and invertebrates is common (*1–3*), but the mechanisms by which this is effected are little understood. Those hypothesized are mechanical, chemomechanical, and chemical.

Carriker *et al.* determined that the boring of holes in the shell of bivalve prey by predatory muricid and naticid gastropods, to obtain food, consists of two alternating phases: (i) chemical, in which an accessory boring organ (ABO) or demineralization gland, secretes an uncharacterized substance that etches and weakens the shell at the site of penetration, and (ii) mechanical, during which the radula rasps off and swallows some of the weakened shell as minute flakes (*1–5*). The boring process is not visible from outside the snail and prey.

Recent determinations of the concentration of hydrogen ions in the surface fluid of forcefully extruded and of excised ABO's, and of ABO homogenates, of muricid snails with minute strips of short-range *p*H papers, the color being verified under the microscope against standardized buffer solutions checked with a *p*H meter, consistently gave reactions close to neutrality (*3*). These results confirmed earlier observations (*6*) of the ABO's of naticid snails, with the exception of Schiemenz's (*7*), who obtained a red color when blue litmus paper was applied to the intact exposed organ of a dying *Natica*. On this basis he suggested that the secretion is acid, but could not say how acid. Development by Charlton (*8*) of a special glass microelectrode has enabled for the first time continuous recording of the *p*H of the secretion of the intact, normally functioning, muricid ABO in an incomplete borehole in prey shell. The microelectrode discloses that, during this phase of boring, the *p*H of the secretion of the ABO drops to a minimum of 3.8. We now report the results of these studies.

We used large muricid snails, *Urosalpinx cinerea follyensis* Baker from Wachapreague, Virginia, ranging in height from 33 to 40 mm; they were maintained in the laboratory in running sea water (salinity, 32 per mille; *p*H 8.2; temperature, about 18°C). In this medium they bored holes (about 1 mm in diameter) in the valves of and fed actively on local Cape Cod oysters, *Crassostrea virginica* (Gmelin).

A recently improved glass-shell oyster model permitted observation of the boring process, as well as access to the extended ABO through a pore (1 mm in diameter) in the glass plate covering an artificial, incomplete borehole (approximately 1.2 mm in diameter and 1 to 2 mm in depth) in the shell at the glass-shell juncture (Figs. 1–3). Models were filled with live, actively metabolizing, de-shelled young oysters and placed among snails in running sea water. In time, snails located the incomplete boreholes, apparently attracted by oyster metabolites diffusing from the boreholes through the glass-shell interface, and bored in them.

The ventral surface of the foot was tightly appressed to the glass and shell