PHOSPHATE EVALUATION

Surface Area of Natural and Processed Phosphates

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Reliable information was needed on the ranges of surface area likely to be encountered in bone products, dicalcium phosphate, basic calcium phosphates, and phosphate rock. Basic calcium phosphate and steamed bone showed surfaces (4.5 to 142 square meters per gram) of the same order as clays. Dicalcium phosphate and phosphate rock ranged from 0.6 to 37 square meters per gram. The influence of particle size (sieve range) on measured surface was practically negligible for steamed bone. The surface of some varieties of phosphate rock increased very little with finer grinding, whereas that of others increased markedly. The elementary grain size of phosphate rocks estimated from surface measurements agrees in order of magnitude with figures obtained by other methods (10^{-2} to >1 micron). The results provide a basis for studies on the surface chemistry of water-insoluble phosphates. Elementary grain size appears to be a reliable index of chemical reactivity and agronomic merit of phosphate rock varieties (usual grinds) that carry the normal complement of fluorine.

THE SHARE OF WATER-INSOLUBLE PHOSPHATES in phosphate fertilizer consumption has increased notably during the past two decades. The shift from soluble toward slightly soluble phosphate compounds has resulted mainly from the growing use of ammonia and other neutralizing agents in the preparation of mixed fertilizers and from the expanding use of phosphate rock for direct application to the soil. The prominence of slightly soluble phosphates in present-day fertilizers emphasizes the importance of surface-chemical effects in their technology and use. Nevertheless, interest in surface area measurements of phosphates has centered mainly on bone and tooth substances, thus reflecting intensive biochemical research on the formation and constitution of bone, whereas little attention has been given other major economic classes of water-insoluble phosphates. This paper is a gesture toward supplying the lacking data. The primary objective is to indicate the ranges of surface areas likely to be encountered in the most important classes of the slightly soluble phosphates. Surface quality, apart from extent, is not discussed.

Materials and Method

The test materials comprised ox bone, phosphate rock, apatite, thermal phosphates, dicalcium phosphate, and basic calcium phosphate. The steamed bone was prepared by heating commercial degreased bone in an autoclave for 1 month under a steam pressure of about 40 pounds per square inch. The phosphate rocks, apatite, and thermal phosphates were in most instances commercial materials, whereas the phosphate compounds were mainly experimental preparations. The fineness of the sample is specified in two general ways. In one, the sample was ground in a plate mill just to pass a chosen sieve with some care to avoid overgrinding; in the other, material crushed or ground to pass a chosen sieve was separated into mechanical fractions. Materials with a fineness in the subsieve range were obtained by regrinding a −200-mesh (or finer) composite sample in a mechanical mortar and separating the fines with the use of a Roller analyzer. Pertinent characteristics of the individual samples are given in the tables of results.

Surface area was determined by gas adsorption (3, 5) on weighed samples of about 5 grams with the use of nitrogen gas at the temperature of liquid nitrogen. Determinations were made on a reimbursable basis by H. T. Freeman, Bone Char Research, National Bureau of Standards. The sample was outgassed for 18 hours, the temperature being 100° C. for bone, phosphate rock, apatite, and thermal phosphates, and 25° to 30° C. for dicalcium and basic calcium phosphates. The results were reproducible within 10% for surface areas of less than 5 square meters per gram and within 5% for surface areas of 5 square meters per gram or greater.

Surface Area of Natural Phosphates and Derivatives

Teeth and Bone. Surface measurements on different sieve fractions of enamel, dentine, and two preparations of bone, and on bone char and bone ash are given in Table I. The surfaces of enamel, dentine, and commercial chipped bone range up to 11 square meters per gram; that of steamed bone runs 64 to 67 square meters per gram. The large increase in measurable surface of bone as a consequence of steam treatment is attributed to the removal of organic substance from the pores of the inorganic skeletal material. Furthermore, since the surface area of the steamed bone is nearly independent of the mesh size of the sample, free communication of the pore spaces with the outside is strongly indicated. On the other hand, the increases in surface area of enamel and dentine with reduction in mesh size point to the presence of blocked pore spaces within the granules. Other results (16) indicate that the total surface area of bone is at least 100 square meters per gram of bone salt, and on the basis of this figure the estimated surface of the bone crystals in the skeleton of a 155-pound person exceeds 100 acres.

New bone char for use in sugar refining has a surface of about 120 square meters per gram. That the surface is mainly on the phosphate rather than on carbon is indicated by the high surface of bone ash prepared from new char. In use the bone-char surface gradually decreases

Table I. Surface Area of Tooth and Bone Materials

Item No.	Material	Fineness, Mesh	Surface Area, Sq. M./G.
1	Enamel, human teeth (15) Composite sample 40.0% P_2O_5, 50.9% CaO	60–80	0.94
		100–120	1.2
		200–230	1.5
		–325	9.7
2	Dentine, human teeth (15) Composite sample 29.6% P_2O_5, 37.3% CaO	60–70	3.2
		100–120	3.9
		200–230	4.0
		–325	11.2
3	Commercial bone, chipped, degreased (6)	25–30	3.1
		50–60	3.1
		80–100	3.7
		170–200	3.0
		270–325	4.5
4	Steamed bone[a] Composite sample 38.5% P_2O_5, 51.0% CaO	4–8	63.7
		16–30	66.2
		100–200	66.2
		–200	67.0
5	Bone char, new (2)	Bulk vol. 1.52 cc./g.	123
6	Bone char, new (6)	Bulk vol. 1.25 cc./g.	120
7	Bone char, item 6 after use[b] in sugar refining (6)	....	25
8	Bone char substitute (2)	Bulk vol. 1.51 cc./g.	90
9	Bone char, item 6 heated to 950° C., inert atmos. (6)	....	25
10	Bone ash, item 6 heated in flowing air (6)	....	100
11	Bone ash, item 7 heated in flowing air (6)	....	16
12	Bone ash, item 3 heated with KOH and glycerol 0.5 hr. at 180° C. (6)	25–30	92
13	Bone ash, item 3 heated with KOH and glycerol 4 hr. at 250° C. (6)	25–30	113

[a] Results given in part by Hendricks and Hill (9).
[b] Through 300 cycles (estimated) of use and reactivation.

show somewhat lower surfaces in substantially the same order. Interestingly enough, the surface of the Tunis rock exceeds by a considerable margin that of pseudowavellite, a finely divided clay-like material.

The set of mechanical fractions (Table II), having been prepared for use in an agronomic experiment, were also characterized with respect to fluorine content, solubility in neutral ammonium citrate solution, particle weight, and particle density. The results are given in Table III. Eight of the ten materials fall in the same order with respect to both citrate solubility and surface area. The South Carolina and Curaçao rocks show high solubilities in comparison with their measured surfaces. The solubility of the latter rock is, however, in line with the relatively high figures commonly found for low-fluorine rocks. The average-particle density ranged from 2.75 for South Carolina rock to 3.19 for Virginia apatite without any apparent consistent relationship to the measured area. Reasonably uniform particle size distributions among the materials are indicated by the narrow range of equivalent spheres (last column). Thus, the sphere diameters range from 122 to 138 microns and average 129 microns in comparison with a mean of 127 microns for the openings of the classifying sieves.

A matter of special interest to people concerned with the preparation and use of ground phosphate rock is the increase in surface area that can be realized by reducing the size of the coarser particles. In order to meet this interest, surface measurements were made on −100-, −200-, and −325-mesh preparations (composite) of seven rocks. The results are depicted in Figure 1. Also shown in the figure are surfaces for −10-micron

with successive reactivation, so that after some 300 cycles it drops to about 25 square meters per gram. Ash prepared with the use of potassium hydroxide and glycerol is influenced noticeably by the ashing procedure.

Phosphate Rock and Apatite. Results for surface area of typical phosphate rocks from several world deposits are given in Table II. The −100-mesh preparations range from 1.1 square meters per gram for Virginia apatite to 37.2 square meters per gram for Tunisian rock. The 100- to 150-mesh fractions

Table II. Surface Area of Commercial Phosphate Rock and Apatite

Lot No.	Material Variety or Source	−100-Mesh Composite Sample		100–150-Mesh Fraction[a]	
		Total P_2O_5, %	Surface area, sq. m./g.	Total P_2O_5, %	Surface area, sq. m./g.
1551	Tunis, Gafsa	29.0	37.2	29.9	19.4
2240	Morocco	...	...	32.1	14.5
910	Florida land pebble[b]	31.7	9.3	30.9	10.3
1446	Florida land pebble[c]	33.2	...	32.8	8.6
985	Curaçao Island	37.6	6.4	37.9	3.6
907	Tennessee brown rock	34.6	7.8	35.0	6.8
1484	S. Carolina land rock	27.6	4.7	27.1	6.5
1253	Idaho, Conda	31.4	5.4	33.0	3.0
1252	Montana, Garrison	36.4	2.9	37.1	1.6
1295	Virginia apatite	40.7	1.1	40.3	0.6
1145[d]	Francolite (Podolite from U.S.S.R.)	...	...	35.5[e]	3.2[f]
2437	Pseudowavellite from Florida[g]	24.5	20.2	...	...

[a] Not from same grind as −100-mesh composite sample.
[b] Washer rock.
[c] Concentrate prepared by fatty acid flotation.
[d] Results for this mineral specimen reported by Hendricks and Hill (9).
[e] Sample (100–200 mesh) contained 49.5% CaO and 4.4% CO_2.
[f] Result for 100- to 200-mesh fraction. Results for other fractions: 20–35 mesh, 1.6; −10-micron, 7.3 sq. meters per gram.
[g] Pembroke clay concentrate (10).

Table III. Some Measured Properties of 100- to 150-Mesh Phosphate Rocks

Lot No.	Variety or Source of Phosphate	F:P₂O₅ Weight Ratio	Total P₂O₅, %	Citrate-Soluble P₂O₅, % of Total	Surface Area, Sq. M./G.	Average Particle Weight[a], γ	Av. Apparent Particle Density[b], G./Cc.	Diameter of Equivalent Sphere[c], μ
1551	Tunis, Gafsa	0.121	29.9	16.4	19.4	2.6	2.76	122
2240	Morocco	0.131	32.1	13.6	14.5	3.0	2.89	126
910	Florida land pebble	0.114	30.9	10.4	10.3	2.8	2.90	123
1446	Florida land pebble	0.107	32.8	7.6	8.6	3.0	2.93	125
907	Tennessee brown rock	0.105	35.0	5.1	6.8	3.4	2.90	131
1253	Idaho, Conda	0.102	33.0	4.5	3.0	4.0	2.93	138
1252	Montana, Garrison	0.106	37.1	3.0	1.6	3.8	3.10	133
1295	Virginia apatite	0.072	40.3	1.5	0.6	4.2	3.19	136
1484	So. Carolina land rock	0.136	27.1	10.0	6.5	3.1	2.75	129
985	Curaçao Island	0.020	37.9	11.9	3.6	3.2	2.96	127

[a] Determined by counting and weighing (microbalance) ten 300-microgram portions of sample.
[b] Determined by displacement of Decalin.
[c] Sphere having volume equal to average-particle volume.

material. The latter, being mechanical fractions instead of composite samples, are not strictly comparable with the others. The domestic phosphates, other than Florida land pebble, showed only slight increases in surface within the practical sieve range as a consequence of grinding finer than 100-mesh. On the other hand, the surfaces of the Florida land pebble and the Morocco rock were markedly increased by finer grinding, and the results for the sieve range lie fairly well in line with that for −10-micron material. The minute contribution of the boundary surface of the particles to the surface area is indicated by the curve (dotted) for geometric surface of uniform spheres having a density of 3.2.

Measurement of surface areas provides a rigorous check on the consistency of the grinding technique used in preparing a fineness series of a material, since the points for successive finenesses of composite samples must fall along a smooth curve (Figure 1). It also affords an exacting test of the reproducibility of grinds, in which respect phosphate rocks apparently differ widely. For example, very limited results obtained in this study indicate that duplication of grinds is much more difficult with the South Carolina and Idaho rocks than with the other test rocks.

Thermal Phosphates. Surface measurements on three fertilizer phosphates prepared from phosphate rock by thermal processes are given in Table IV. These materials present relatively small surfaces in comparison with phosphate rock.

Surface Area of Phosphate Compounds

Dicalcium Phosphate. Results for surface area of commercial and experimental preparations of dicalcium phosphate and water-insoluble phosphates obtained from fertilizers are given in Table V. The surfaces range from less than 1 square meter per gram for a food-grade salt to 26 square meters per gram for the water-insoluble fraction of

an ammoniated triple superphosphate. Cleansing the surface by washing with dilute acid had only a slight effect on the measurement.

Samples R-39 and R-42 are typical of phosphorus-32–labeled dicalcium phosphate prepared for agronomic experi-

mentation in 1953. The next four listed materials were selected from a series of tests intended to point the way to closer control of the surface of radioactive preparations and to a technique for producing material with larger surface area. The outcome of these tests is

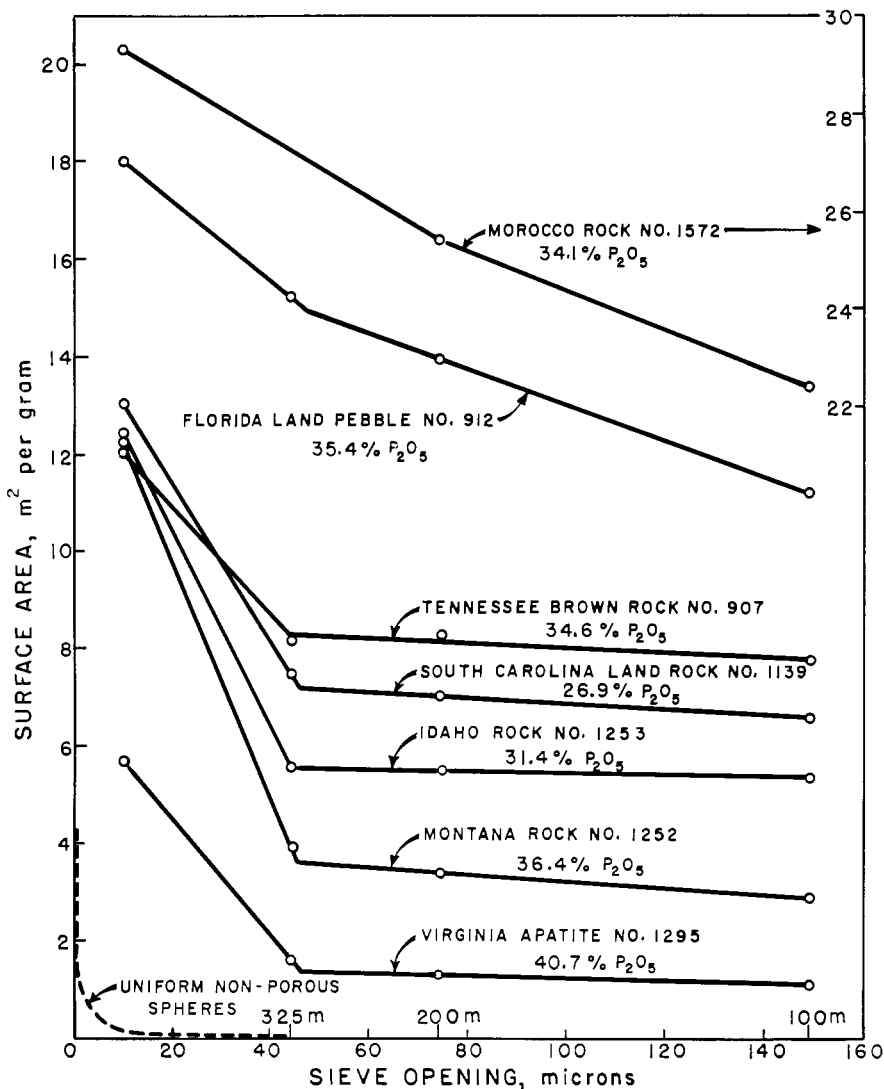## Figure 1. Influence of fine grinding on the surface area of phosphate rock

MOROCCO ROCK NO. 1572
34.1 % P₂O₅

FLORIDA LAND PEBBLE NO. 912
35.4 % P₂O₅

TENNESSEE BROWN ROCK NO. 907
34.6 % P₂O₅

SOUTH CAROLINA LAND ROCK NO. 1139
26.9 % P₂O₅

IDAHO ROCK NO. 1253
31.4 % P₂O₅

MONTANA ROCK NO. 1252
36.4 % P₂O₅

VIRGINIA APATITE NO. 1295
40.7 % P₂O₅

UNIFORM NON-POROUS SPHERES   325m   200m   100m

SURFACE AREA, m² per gram

SIEVE OPENING, microns

Table IV. Surface Area of Phosphates Prepared by Thermal Processes

| | Material | | | |
Lot No.	Variety	Total P₂O₅, %	Fineness, Mesh	Surface Area, Sq. M./G.
2497-a	Phosphate rock–magnesium silicate glass[a]	20.2	−100	0.2
			−200	0.3
			−300	0.9
			−10 μ	2.4
2986	Rhenania phosphate, imported[b]	26.1	−100 (75%)	0.6
2987	Rhenania phosphate, domestic[c]	24.4	−100	1.1

$Total\ P_2O_5$

[a] Material used by Hill *et al.* (*11*); product no longer produced domestically.
[b] Material used by Schmehl and Brenes (*18*).
[c] Experimental material simulating Rhenania phosphate.

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illustrated by materials 3165-a and -c. Control of the reaction temperature within a narrow range near room temperature yielded a reproducible material with a surface just under 2 square meters per gram. Protracted digestion of this product with water at simmering temperature gave products ranging around 13 square meters per gram. The gain in surface was accompanied by alteration of the phosphorus-calcium ratio in the direction of basic phosphate.

Basic Calcium Phosphate. Surface measurements on experimental and commercial preparations of basic calcium phosphate are given in Table VI. The results for the first six listed materials, selected from the work of Gee (*7*), mark out the range of observed surface areas (4.5 to 142 square meters per gram) for this type of material. The other materials range from 10 square meters per gram for a material formed at low pH to 100 square meters per gram for a com-

mercial adsorbent grade of basic phosphate. A low pH and an elevated temperature favor the formation of material with a low surface area.

Phosphates Compared with Other Materials

Observed ranges in surface area for phosphates and several other materials are collected in Table VII. Comparison of the figures reveals the rank of phosphates among high-surface materials. Basic phosphate and steamed bone range with clays, phosphate rock parallels diatomaceous earth, and dicalcium phosphate falls somewhat lower on the scale.

Elementary Particles of Natural Phosphates

The large measured surface area of coarse specimens of bone and phosphate

rock is attributable to pore space within the particles. Such materials are commonly viewed as aggregates of fine crystals whose size range is several orders of magnitude smaller than the smallest practical sieve dimension. The average dimension of the elementary grains can be deduced from x-ray diffraction measurements, and such determinations have been made on a few natural phosphates. A figure can also be calculated from surface area measurements on suitably fine samples. Results obtained on certain varieties (different samples) of phosphate by these two independent methods are collected in Table VIII. The agreement between the two sets of figures for elementary grain size is within one order of magnitude, which is surprisingly close for results that, having been obtained on different samples, include the influence of expected variation in varietal characteristics.

Surprisingly enough, the grain size of

Table V. Surface Area of Dicalcium Phosphate

| | | | Material | | Surface |
Lot No.	P₂O₅, %	P:Ca, Mole Ratio	Method of Preparation	Fineness, Mesh	Area, Sq. M./G.
2450	50.7	0.988	Commercial food-grade material	−100, +325[a]	0.7
			Same, acid washed[b]	−100, +325[a]	0.8
2961-a	51.4	1.002	Commercial food-grade material	−325	1.5
				−10 μ	3.7
			Same, acid washed[b]	−325	1.6
3101	51.5	1.018	H₃PO₄ + CaCO₃, 90–100° C.	−100 (80%)	1.4
			Same, acid washed[b]	−100 (80%)	1.5
R-39[c]	51.1	1.013	H₃PO₄ + Ca(OH)₂, 30–100° C.	−100 (83%)	5.2[d]
R-42[c]	51.1	0.973	H₃PO₄ + Ca(OH)₂, 30–100° C.	−100 (65%)	1.6
3167	50.5	0.981	H₃PO₄ + Ca(OH)₂, 30–65° C., digested 100° C. 7 hr.	−100 (92%)	7.2
3165-a	50.7	0.987	H₃PO₄ + Ca(OH)₂, 30–35° C.	−100 (70%)	1.7[d]
3165-c	49.1	0.903	No. 3165-a digested with water at 100° C. 18 hr.	−100 (65%)	13.5
3207-a	49.0	0.909	H₃PO₄ + Ca(OH)₂, 30–35°, digested 100° C. 20 hr.	−60	9.2[d]
				−325	12.5[d]
2560	43.5	0.875	Water-insoluble material from fertilizer[e]	. . .	15.7
2216-b	40.1	0.876	Water-insoluble material from fertilizer[f]	. . .	26.0

[a] Material was mainly aggregates.
[b] 0.001*M* hydrochloric acid.
[c] Radioactive preparation.
[d] Determined with use of P³² exchange procedure by J. H. Caro.
[e] A nitric phosphate, commonly regarded as being essentially CaHPO₄ and NH₄NO₃.
[f] Ammoniated triple superphosphate, 5% NH₃, commonly regarded as being essentially CaHPO₄ and NH₄H₂PO₄.

Tunis and Morocco rocks is of the same order as that of dentine. From the viewpoint of phosphate rock technology and use, however, a greater interest attaches to a comparison of these rocks with Florida land pebble. The indicated grain size of the latter phosphate is two- to tenfold that of the North African rock. Paralleling this difference in size of the elementary particles is the recognized (especially in European circles) agronomic superiority of North African rock over Florida land pebble for direct application to the soil (*13*). These observations suggest elementary-grain size as one of the properties that determine the intrinsic agronomic merit of a phosphate rock. A coarse-grained rock probably cannot, by practical reduction in fineness, be made equivalent to the moderately fine grind of fine-grained rock.

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Table VI. Surface Area of Air-Dried Basic Calcium Phosphates

Lot No.	P:Ca, Mole Ratio	Method of Preparation	Surface Area, Sq. M./G.
20[a]	0.601	Precipitated from dilute solution, 100° C.	4.5
11[a]	0.585	Same with excess calcium in solution, 100° C.	20
28[a]	0.632	Same with excess phosphate in solution, 100° C.	20
22[a]	0.703	Precipitated from $Ca(HCO_3)_2$ solution, 25° C.	68
25[a]	0.644	Precipitated from dilute solution, 25° C.	128
27[a]	0.665	Precipitated from dilute solution, 2° C.	142
2186-b	0.631	Hydrolysis of $CaHPO_4$ at 100° C. with NH_3 neutralization, pH <5.5	10
12	...	Hydrolysis of $CaHPO_4$ at 100° C. with NaOH neutralization, pH <5.5	27
1540-a[b]	0.629	Product, $Ca:PO_4 = 1.596$, heated 16 hr. with ammonium citrate solution at 100° C.	36
2187[c]	0.603	$0.5M$ $Ca(NO_3)_2$ + $0.5M$ $(NH_4)_3PO_4$, product washed by repeated autoclaving	43
2610	0.608	Commercial product, conditioner grade, received in 1949	55
2453[b]	0.629	Commercial product, conditioner grade, received in 1946	58
d	0.567	Commercial product, for use as adsorbent	100

[a] Prepared and studied by Gee (*7*). Lime water (0.02*M*) and 0.008 to 0.011*M* Ca-$(H_2PO_4)_2$ solution were flowed at same rate into 6 liters of water with continuous gentle stirring.
[b] Material used by Olsen (*17*) in surface phosphate measurements.
[c] Material used by Whittaker et al. (*19*) in crop experiments.
[d] Results supplied by producer.

Table VII. Surface Areas of Phosphates and Some Other Substances

Material	Particle Size	Surface Area, Sq. M./G.	Source of Results
Coconut charcoal	....	1700	(*4*)
Silica gel	....	414–866	(*4*)
Clays	−0.3 μ	15.5–97.1	(*4*)
Basic calcium phosphate	....	4.5–142	Table VI
Steamed bone	4–8 and −200 mesh	64–67	Table I
Diatomaceous earth	−325 mesh, 88–100%	12–40	(*12*)
Phosphate rock, commercial	−100 mesh	2.9–37	Table II
Dicalcium phosphate	....	0.6–26	Table V
Limestone, agricultural	25–30-mesh	0.24–11.5	(*14*)
Quartz	−400 mesh	0.36	(*4*)
Potassium chloride	−200 mesh	0.24	(*4*)

Table VIII. Size of Elementary Grains of Some Natural Phosphates

Phosphate	Average Grain Size, μ, from Measured specific surface[a]	Average Grain Size, μ, from Broadening of x-ray bands[b]
Bone salt	0.028[c] (−200 mesh)	0.024[d]
Tunis rock	0.05 (−100 mesh)	Ca. 10^{-2}
Morocco rock	0.065 (−10 μ)	Ca. 10^{-2}
Florida land pebble	0.10 (−10 μ)	Ca. 10^{-1}
Tennessee brown rock, So. Carolina land rock, Idaho rock, Montana rock	0.15 (−10 μ)	...
Apatite	0.33[e] (−10 μ)	>1.0[f]

[a] Fineness of sample given in parentheses.
[b] Results other than for bone salt given by Geiger (*8*).
[c] Result for steamed bone.
[d] Result for dentine given by Bale et al. (*1*); their figure for enamel is 0.27.
[e] Apatite from Virginia, U.S.A.
[f] Apatite from Kola Peninsula, U.S.S.R.