EXCHANGE OF LYOTROPIC SERIES CATIONS BY MICACEOUS VERMICULITE
AND ITS WEATHERING PRODUCTS DETERMINED BY ELECTRON MICROSCOPY
AND RADIOCHEMICAL ANALYSIS

Final Technical Report

MASTER

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FINAL TECHNICAL REPORT

Marion L. Jackson -- Principal Investigator

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for Period June 1, 1965-October 31, 1978

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ABSTRACT

(Scope and Purpose; 200 words or less)

Micaceous vermiculite was found to be ubiquitous in soils, sediments, and aerosol mineral dusts and to adsorb fission product ions, particularly $^{137}\text{Cs}^+$ and $^{90}\text{Sr}^{2+}$. Crystallographic wedge zones (imaged by ultramicrotomy and electron microscopy) in micaceous vermiculite effected tight fixation of Cs^+ . Lowering of mica layer charge occurred in local areas, electronoscopically imaged by use of blister-swelling cations. Nuclear fission particle tracks of U enhanced translayer diffusion of elements, measured mica layer charge and age. Iron-aluminum hydrous oxide coatings adsorbed divalent cations of the alkaline earth, transition, and heavy metal elements from trace concentrations in the presence of 1 M NaNO_3 . Global deposition of dust by rainfall accounted for the wide distribution of Cs-fixing micaceous vermiculite in soils. Origin of the dust was traced through a method developed for isolation of fine quartz silt (1 to 10 μm diameter). Mass spectrometry of its $^{18}\text{O}/^{16}\text{O}$ isotopic ratios showed distinctly higher ($\delta^{18}\text{O} = 17\text{--}22\%$) and lower ($\delta^{18}\text{O} = 9\text{--}15\%$) ranges in the Northern and Southern Hemispheres, respectively. This difference was traced to the relative proportions of quartz from low-temperature authigenic (chert) vs. igneous-metamorphic origin in the respective latitudes, hinging on trans-equatorial continental drift.

Documentation and Collaboration

This progress report is submitted pursuant to Article B-XX of the Contract, No. DE-AS02-76EV01515 (formerly EY-76-S-02-1515) which has been in operation since June 1, 1965. Under this contract to date, 55 scientific papers have been published (12 sets of reprints are attached) or accepted for publication, and one additional paper has been submitted for publication (all listed near the end of this report). Besides the Ph.D. students and Post-doctoral Project Associates who have participated in the work at Madison, numerous other scientists, on campus and off-campus throughout the world have actively participated in the research, through an International Consortium for Interinstitutional Cooperation in the Advancement of Learning (ICICAL) organized by the Principal Investigator. Major cost-sharing has been achieved because of the interest in cooperation with this project of people who were otherwise supported. Significant research results have been obtained through this collaboration, including co-publication with the collaborators (resulting publications, with cooperating authors, are listed near the end of this report). The DOE/EV/01515 (C00-1515) reporting status is summarized in the last two pages of this report.

I. Mechanism of $^{137}\text{Cs}^+$ adsorption by micaceous vermiculite

(a) Hydrous metal oxide coatings shown by C-replica and scanning electron microscopy. -- Electron microscopic-scale roughness on the surface of micaceous vermiculites was identified (1)* by its solubility in citrate-bicarbonate-dithionite reagent for the removal of oxides of iron (and accessory aluminum and silica) from soils. The aluminum extracted (9) was approximately 10% of the weight of iron (expressed as oxides). This is important because the Al^{3+} is known to provide a positive charge to the ferruginous coatings. The cation-exchange capacity (CEC) increased from 64 to 95 meq per 100 g in a fraction coarser than 1-mm diameter (3), while the Fe^{2+} increased 2-fold, with an equivalent decrease in Fe^{3+} . Treatment with H_2O_2 restored the content of Fe^{3+} but did not affect the CEC. The original increase in CEC on deferration was attributed to removal of positively charged sesquioxide coatings. The valence $\text{Fe}^{2+} \rightleftharpoons \text{Fe}^{3+}$ changes are accompanied by corresponding deprotonation $\rightleftharpoons$ protonation of the structural hydroxyl (9, 32). Addition of interlayer hydroxy aluminum decreased the CEC (2) of vermiculite and such decrease was pH-dependent and exhibited hysteresis. Layer charge is important to cation fixation in wedge-zones (4) at the X-Y interface of mica and vermiculite. Layer charge density is important in the cation-exchange selectivity (CES) for cations such as $^{137}\text{Cs}^+$ (5), in wedges but also on (001) crystal faces (16). A chronosequence of soils in New Zealand (22) showed increasing amounts of sesquioxide coatings of micaceous vermiculite with time of weathering and avid fixation of $^{137}\text{Cs}^+$ by this mineral complex. Vermiculites are held together as mica pseudomorphs by hydrous metal oxide crusts (24).

* Numbers in parenthesis refer to publications (listed near the end of the report) produced under financial support of the project, AT(11-1)-1515, E(11-1)-1515, EY-76-S-02-1515, and DE-AS02-76EV01515.

(b). Layer charge lowering, blister-like swelling by hydrated cations and shrinkage with fixing cations, and thermodynamic metastability of vermiculite weathering from mica. -- Mica layer charge lowering by natural weathering produces the expandable minerals, vermiculite and smectite (29). The weathering products may occur in discrete zones and the resultant complex then gives a series of discrete X-ray diffraction lines (29). However, in the process of mica weathering, the alteration of layer charge was discovered to be initiated in discrete circular zones over the (001) cleavage surface (12, 20), made apparent by the swelling effects of Li^+ , the most hydrated of the monovalent lyotropic series. Non-uniformity of layer charge was thus established by the occurrence of blisters observed by electron microscopy of the swollen areas (20). When the layer charge lowering had progressed sufficiently far, hydrated divalent ions produced swelling of blisters (20) and discrete layers of saponite glided out under Li^+ swelling (20, 29). Fixing cations caused the blisters to shrink back nearly to the original crystal plane (15, 26). An electron microscopic visual model was thus established for the origin of the wedge-shaped X-Y crystallographic boundaries (4) in micaceous vermiculite.

The lowering of mica layer charge was established, not to be caused by the change in iron valence (3, 9, 32), but to arise from authigenic decrease in the amount of tetrahedral substitution in the layers (31). Tetrahedral Fe^{3+} and Al^{3+} were lost and total SiO_2 increased in the weathering product. Higher than theoretical charge in macroflakes of muscovite (10) was attributed to penetration of washing-salt solutions

penetrating into defects and (110) cleavage steps on the surfaces (shown by electron microscopy). Theoretical mica charge was obtained (43) when microscopically planar areas were used for the CEC measurement with uranium salt at pH 2.25 (to preserve mononuclear cations). After neutron activation, fission particle track density from the ^{135}U fissioned was counted. Metastable dioctahedral vermiculite was found (39) in a soil, in metastable equilibrium with mica. Change in $\text{Ca}(\text{HCO}_3)_2$ concentration with seasonal change in CO_2 partial pressure determined the direction of the mica $\rightleftharpoons$ vermiculite reaction in winter as contrasted to summer, with important K^+ deficiency results in the summer growing season. A method for the determination of mononuclear Al^{3+} in solution at a range of less than parts-per-billion was developed (50) to support this type of work.

The high layer charge in the wedge juncture of mica and vermiculite is important in the selective exchange and fixation of K^+ , Rb^+ , and Cs^+ (5, 16). In nature radioactive cesium was stopped within a few meters by micaceous vermiculite under radioactive waste tank leaks into soil on the Hanford reservation (57). Fixation of radioactive cesium on wastes from the Savanna River storage was stopped by mainly kaolinite clay, but the active agent was indicated to be small amounts of micaceous vermiculite in the mainly kaolinite river sediments (34, 54).

(c). Layer sequences by ultramicrotomy and high resolution electron microscopy. -- To provide a visual model of phyllosilicate clay layer configuration, a technique for vacuum casting of Epoxy resin mounts of clay was developed (34). A diamond-knife ultramicrotome was used to section the mounted phyllosilicate specimen perpendicular to the plane of sedimentation of a clay film sandwiched between two Epoxy layers (25). The 7.22 Å fringe spacings of chlorite were imaged (25). Heating of the specimen by the electron microscope beam caused enhancement of a 13.9 Å fringe spacing, with a disappearance of the 7.22 Å fringe spacing (25). This crystallographic effect had previously been noted (by G. W. Brindley) only through heating at 760°C in a furnace.

The significant project results were imaging 10 Å mica layer fringes mixed with relatively pure kaolinite (34). Two types of fringes interspersed were clearly recorded with a microdensitometer (34). The presence of the mica was in accord with the 0.075% K_2O content of the predominately pure kaolinite (34). The potential fixation of radioactive Cs^+ by kaolinitic clays in soils and river sediments could therefore be understood by the well-known ability of mica layers to fix Cs^+ .

The occurrence of a complex array of various clay species was imaged in specimens of micaceous vermiculites (35). The 11 Å (001) the 5.5 Å (002) fringes of Cs^+ saturated vermiculite were clearly resolved from the electron micrograph by microdensitometer tracings. Samples which gave complex X-ray diffraction patterns, without definable diffraction peaks, were shown to be highly crystalline, but consisting of irregularly mixed layers of several clay species (35). This was the first electron

optical visualization of such multicomponent interstratification of phyllosilicate clays. Kaolinite, smectite (montmorillonite), mica, vermiculite, and chlorite spacings were imaged.

Moreover, wedge-zones of the X-Y crystallographic lateral transition from mica to vermiculite gave clearly defined wedge-aligned images of the layers (35). This was the first direct microscopic observation of the wedge phenomenon. This is important in understanding Cs^+ fixation in soil micaceous vermiculites in a form not subject to leaching into the ground water, and hence of health physics significance in reference to sequestering of nuclear fission products.

(d). Fission tracks in mica and vermiculite, effect on cation diffusion, U content, and mica age-dating. -- Fission particle tracks in micaceous vermiculites were made visible by electron microscopy through HF etching (24, 30). Neutron activation in a thermal nuclear reactor increased the track density by fission of ^{135}U naturally present in micas (30). Chemical properties of the micaceous vermiculites were measured before and after neutron activation, but in the absence of HF etching. The rates of release of structural K^+ , Al^{3+} , Fe^{2+} , and $\text{Si}(\text{OH})_4$ were all enhanced by the presence of the increased fission particle track density (microscopically invisible, unetched tracks). The cation-exchange capacity (CEC) of the samples was increased by 40 to 50%. The cation exchange selectivity (CES) of the samples for fixing cations increased from 15 to 100% as a result of the increased number of fission particle tracks. This established that the increased number of edge wedge sites surrounding fission particle tracks provide more adsorption capacity of micaceous vermiculite for nuclear fission products such as $^{137}\text{Cs}^+$ (30).

A replica method was developed for the measurement of fission particle tracks of clay and silt fractions (44). The fine fractions were mounted (as "infinitely thick" layers) on muscovite flakes for neutron activation, and the track counting was then carried out on the flakes after etching. The U content of the fractions was determined by track counting before and after neutron activation. Age-dating of the fine fractions of a soil (residual in metamorphic rock) from Antarctic gave a soil age of 4.1 ± 0.2 million years (44). This age is consistent with the chronology of early Antarctic glaciations. The U content of the mica formed in this soil, as sericite in plagioclase, was 0.5 to 1.6 ppm, which was 20 to 50 times higher than that of the mica of the parent rock (0.03 ppm; 151 MY age). Concentration of U appears to occur during the feldspar-mica transformation under Antarctic conditions (44). This work gives strong evidence of weathering of mica in unfrozen water films in that region. The penetration of CEC-measuring salt solutions through muscovite macroflakes (10) was also further explained by this work.

II. Extent and mechanism of specific adsorption of lyotropic series divalent cations such as $^{45}\text{Ca}^{2+}$, $^{90}\text{Sr}^{2+}$, and $^{65}\text{Zn}^{2+}$ on hydrous metal oxides occurring in soils.

(a). Calcium, strontium, zinc and cadmium adsorption. -- Radioactive strontium was adsorbed by chronosequences of soils containing much micaceous vermiculite in New Zealand (22) and containing kaolinite and hydrous metal oxides in Australia (23). Much of the adsorption capacity of the latter was lost when the hydrous metal oxides (particularly iron oxides) were removed by citrate-bicarbonate-dithionite treatment. Detailed/study of traces of $^{45}\text{Ca}^{2+}$ and $^{90}\text{Sr}^{2+}$ in 1 M NaNO_3 solution by Fe-gels and Al-gels showed a high adsorption capacity, depending on the pH (14). The pH of 50% adsorption, pH_{50} , was lower for Fe-gel (pH 7.1 for both Ca^{2+} and Sr^{2+}) than for Al-gel (pH 8.3 and 9.0), respectively. Because the adsorption of Ca^{2+} and Sr^{2+} occurred from concentrations of 10^{-6} M in 1 M NaNO_3 , it was termed specific adsorption. Interestingly, only one H^+ ion was released to solution for each divalent ion adsorbed.

When a comparison was made to the adsorption of Zn^{2+} and Cd^{2+} by Fe- and Al-gels under similar conditions, pH_{50} values of pH 5.4 and 5.6, respectively, were obtained for Zn^{2+} and pH 5.8 and 6.6 for Cd^{2+} (33, 38, 42). Interestingly, the H^+ released per Zn^{2+} adsorbed was 1.65 and 1.49 for Fe-gel and Al-gel, respectively (42), in contrast to 1.0 for alkaline earth cations (14, 33). The toxic metal, Cd, was a weak competitor for other divalent cations. Aged gels adsorbed the cations more strongly than fresh gels (42). In the environment, most Fe-gel is aged, so its stronger adsorption at trace Cd^{2+} levels may be

significant. The $^{90}\text{Sr}^{2+}$ of tank leaks at Hanford (57) was arrested mainly by CaCO_3 horizon in soil, so the hydrous metal oxides may be important only in holding up the rate of migration of radioactive Sr^{2+} .

(b). Alkaline earths, transition, and heavy metals. -- Eight divalent cations, each at $0.125 \times 10^{-3} \text{ M}$ concentrations were allowed to compete with each other for adsorption sites, in 1 M NaNO_3 of Fe-gel and Al-gel (38). The order of cation selectivity varied between the two gels. The selectivity sequences (the lower the pH, the higher the selectivity; pH_{50} in parentheses) were as follows: Fe gel -- Pb(3.1), Cu(4.4), Zn(5.4), Ni(5.6), Cd(5.8), Co(6.0), Sr(7.4), Mg(7.8); Al gel -- Cu(4.8), Pb(5.2), Zn(5.6), Ni(6.3), Co(6.5), Cd(6.6), Mg(8.1), Sr(9.2). The same sequences were found on duplicate runs, and the pH_{50} values generally agreed within ± 0.1 or $\pm 0.2\text{pH}$. The wide separation of the Mg and Sr curves from the transition metal cation curves indicates that the alkaline earth cations would not be expected to be able to exchange effectively with the transition metal cations.

The pH_{50} values were almost the same with the alkaline earth cations present singly as when present in combination. The selectivity sequence (pH_{50}) for Al gel was Mg(8.5), Ca(9.1), Sr(10.2), Ba(10.5). This corresponds to the reverse of the ionic radii ($\text{\AA}$): Mg^{2+} (0.65), Ca^{2+} (0.99), Sr^{2+} (1.13), Ba^{2+} (1.35). The pH_{50} values were well below hydroxide precipitation values and well below the pH values of 1% hydrolysis (first hydrolysis constant) (38). Therefore, the specific adsorption mechanism (less than equivalent H^+ replaced) very likely represents a degree of anion retention in the diffuse "double layer" (33).

With increasing Ca^{2+} and Zn^{2+} concentrations, the pH_{50} values for adsorption on Fe gels moved to higher pH values (56). The range for Zn^{2+} was from pH 5.0 to 6.3 for 10^{-7} to 10^{-2} M; for Ca^{2+} , from pH 7.2 to 9.6 for 10^{-6} to 10^{-2} M (56). The pH_{50} values were not overlapping for the two ion species. The Langmuir x/m plots against Zn^{2+} concentration were nonlinear, indicating variation in site character for different concentrations (56).

Freshly prepared Fe-gel strongly adsorbed Hg(II) from 2.5×10^{-5} M to 10^{-6} M HgCl_2 solutions (46). There was no significant coadsorption of $^{36}\text{Cl}^-$, indicating that the Hg(II)Cl^+ or Hg(II)Cl(OH) complexes are not adsorbed species. The Hg(II) adsorbed may be coordinated to two surface-OH groups of the Fe-gel. The pH_{50} for Hg(II) is approximately pH 4.4, similar to that of Cu. At pH 5, 90% of the Hg(II) is adsorbed (46). The significance of the work with adsorption of divalent cations on hydrous metal oxides present in soils lies in an indicated "hold up" in the rate of migration of divalent cations of fission products to lower migration velocities than ambient groundwater.

(c). Retention of multielement sequences by instrumental neutron activation and X-ray fluorescence analysis to evaluate potential fission product element retention. -- Standardization of the neutron activation analysis instrumentation was carried out with Certified Reference samples (45). Hydrous oxides of Fe and Mn are ubiquitous weathering products in the soil environment (52). The tendency of these oxides is to retain trace elements by cation substitution, coprecipitation, and adsorption. The retention of the elements Co, Cr, Mn, Sc, Th, U, Zn and the heavy rare earth elements (REE) are significantly associated with Fe oxides during the weathering of diabase (52). In the weathering of granite, the elements As, Co, Cr, Sc, Th, U, Zn and heavy REE are significantly associated with Fe oxides (52). The element Cs^+ is consistently associated with the mica fraction. The association of these potential fission product elements with Fe oxides has important implications in the disposal of radioactive wastes in earthy media.

In the weathering of diabase, the elements Cr, Hf, Ta, and Ti are retained in resistant minerals in the saprolite (55). A method of isolation of the TiO_2 minerals anatase and rutile, based on H_2TiF_6 dissolution of silicates was developed (11). The elements Sc, Th, U and REE are retained in the weathering product as insoluble secondary minerals (55). The concentrations of As, Co, Mn, and Zn as well as the REE's, Yb and Lu, have positive correlations with the Fe concentration. The elements Cs, Rb, and K (the most retained element listed first) are strongly retained in the mica formed during diabase weathering (55). Because of the abundance of radioactive Cs^+ in fission product wastes, its strong retention by soil and rock weathering products is important in preventing the movement of this radionuclide with groundwater.

III. Aerosolic minerals in dusts and sediments which fix radioactive nuclear fission products

(a). Radioactive cesium sequestering by dusts. -- Micaceous vermiculite was found to be ubiquitous in mineral dusts and dust-related soils, in a global distribution of samples (17). The fission product $^{137}\text{Cs}^+$ was found in some of the dusts, in amounts varying from 0 pc/g (pre-nuclear testing Greenland ice core) to 264 pc/g (Franz Joseph Glacier, N.Z.) depending on deposition time by rainfall in relation to time of nuclear testing. All of the dusts fixed Cs^+ from solution and strongly retained it against replacement in 0.01 N CaCl_2 solution. The active Cs^+ fixation site was traced to the wedge zone at the crystallographic X-Y interface between mica and vermiculite. Health physics inferences were drawn to fact that micaceous vermiculite dust can sequester radioactive Cs^+ protectively, since dust is readily washed from the skin and hair of animals. Other dust constituents included free iron and aluminum oxides (dithionite extractable), which absorb $^{90}\text{Sr}^{2+}$. The minerals, quartz, chlorite, feldspars, kaolinite, montmorillonite, amphibole, and talc, in addition to micaceous vermiculite, also occurred in the dusts (17).

Quartz was selected as the 'marker mineral' the stable oxygen isotopic ratios of which were used to trace the origin or provenance of dusts, soils and sediments, in relation to radioactive waste isotopes that they contain or can fix from nuclear fission product materials. A method was first developed (6, 41) for the isolation of fine-grained quartz in monomineralic form. . . . The method was tested for stability of the $^{16}\text{O}/^{18}\text{O}$ ratio with the isolation treatments (37, 47).

(b) Provenance of quartz of aerosol dusts and sediments. -- The extent of global-scale transport of aerosol-size (mainly 1 to 10 μm) dust containing micaceous vermiculite by tropospheric winds was firmly established by oxygen isotopic ratios of the quartz contained therein. Documentation was first published in 1968.¹ The presence of mica and quartz in soils developed on quartz-free basalt in Hawaii (13) and Victoria, Australia (21) was explained by transport of desert dust from land masses. The mean values for quartz oxygen isotopes (Table 1) of $\delta^{18}\text{O} = 17.6 \pm 0.5\%$ on four Islands of Hawaii compared remarkably well with the $\delta^{18}\text{O} = 17.5\% \pm 0.5\%$ in North Pacific pelagic sediments (18) 500 miles or more from land, indicating a common provenance from Northern Hemisphere sources. These aerosol dusts contain quartz having oxygen isotopic ratios ($\delta^{18}\text{O} = 18 \pm 2\%$) intermediate between those of igneous-metamorphic quartz and quartz crystallized at lower temperatures such as chert in sediments (18, 53). A composite mixture of these two sources in silty sediments and soils formed thereon was postulated as the source of atmospheric dusts. To test this, the oxygen isotopic ratios ($\delta^{18}\text{O}$) of whole shale quartz, and the 1-10 μm fraction (long range aerosol size) of shale quartz, were determined for the geologic column in the U.S.A. (40). A range, $\delta^{18}\text{O} = 20 \pm 3\%$, was found for the 1-10 μm quartz fraction of marine shales, which is an intermediate range

¹ R. N. Clayton, R. W. Rex, J. K. Syers, and M. L. Jacson. Oxygen isotopic abundance in quartz from Pacific pelagic sediments. Abstracts, Geol. Soc. Am., Spec. Paper 121, Mexico City, November, 1968, p. 56; M. L. Jackson, Th. W. M. Levelt, J. K. Syers, R. W. Rex, R. N. Clayton, and G. D. Sherman. Geomorphological relationships of tropospherically-derived quartz in soils of the Hawaiian Islands. Agron. Abstracts, New Orleans, November, 1968, p. 153; (7).

Table 1 -- Mean oxygen isotopic ratios of aerosol-sized quartz isolated from dusts, soils and sediments (from Jackson (53)).

Area	Kind of soil or sediment	Quartz size range (μm)	Number of samples (mean of)	Mean $\delta^{18}O^{++}$ (‰)	Reference
Mid-USA	Shales	1-10	30	20.7±1.8	Churchman <u>et al.</u> , 1976
	"	ws*	31	19.1±2.2	" " " "
SW. USA	Soils	1-10	13	19.6±0.3	Sridhar <u>et al.</u> , 1977
		1-10	18	19.0±0.2	" " " "
	Dusts	1-10	21	18.4±0.8	Gillette <u>et al.</u> , 1977
E. Cent. TX, AZ	Soils	1-10	5	17.7±0.6	Sridhar <u>et al.</u> , 1977
Hawaii (4 Islands) [†]	Soils	1-10	5	18.3±0.2	Sridhar <u>et al.</u> , 1977
		ws	25	17.6±0.5	Jackson <u>et al.</u> , 1971
N. Pacific	Pelagic	ws [§]	8	17.5±0.5	Clayton <u>et al.</u> , 1972
S. Pacific	Pelagic	2-5	7	15.1±1.5 [#]	" " " "
Australia, Victoria	Loess in basaltic soils	2-5	10	14.9±0.5	Mokma <u>et al.</u> , 1972
		5-20	11	14.5±0.2	" " " "
SW. USA	Soils	ws	8	14.7±0.9	Sridhar <u>et al.</u> , 1977
New Zealand					
N. Island	In till	5-20	5	13.6±1.0	Mokma <u>et al.</u> , 1972
S. Island	" "	5-20	7	13.2±0.2	" " " "
S. Island	" "	2-5	7	13.3±0.1	" " " "
S. Island	Glacial dust bands	ws	3	13.5±0.4	" " " "
S. Africa	Soils and sediments	1-10	28	12.2±2.1	le Roux <u>et al.</u> , 1980

* Whole quartz from samples.

[†] Including for 1-10 μm two lake sediments from on top of Mauna Kea, and excluding hydrothermal area for ws.

[§] Weighted average calculated from size fractions, excluding Gulf of Alaska.

[#] 9° 13' to 58° 26' S. latitude; regression, $r = -0.73$, 17.7 to 12.8‰ southward.

⁺⁺ $\delta^{18}O = \left[\frac{^{18}O/^{16}O \text{ (sample)}}{^{18}O/^{16}O \text{ (standard)}} - 1 \right] \times 1,000$, relative to Standard Mean Ocean Water (SMOW), expressed as parts per thousand (‰).

like that found for dust. Further mixing of the two sources, as the shales were reworked by fluvial, eolian and glacial processes and subsequent soil formation and wind erosion, appears to be responsible for narrowing the composite oxygen isotopic ratio to the range found for several fine silt quartz systems such as in the North Pacific sediments (13, 18).

Scanning electron microscopic examination of cherts (36) revealed predominant occurrence of very fine crystals (0.5 to 10 μm in diameter). This explains the high $\delta^{18}\text{O}$ values for quartz fine fractions, since the $\delta^{18}\text{O}$ for unmetamorphosed cherts ranges from 24 to 32‰ (8,18,37). Euhedral morphology of quartz in particles and overgrowths on quartz grains were also shown by scanning electron microscopy (36) and therefore quartz particle morphology, to a certain extent, may be used as an indicator of provenance for aerosol dusts and soil silts. Examination of the quartz silt of soils and sediments occasionally revealed many chert aggregates (36), but more frequently blades and chips (1-10 μm in diameter) were observed and these may have formed as overgrowths on quartz in sediments and fractured into the aerosol fraction (37). Biogenic silica (diatomaceous) fragments were found in dusts of several areas and this observation indicated the need for heavy liquid separation as the final step in quartz isolation (37).

The quartz oxygen isotopic ratio varies greatly with quartz grain size (8,13,18,37,47,53). The $\delta^{18}\text{O}$ varied as much as from 16 to 25‰ in going from 20- to 1- μm diameter quartz particles in a given shale (47). This variation is mainly a function of diversity of quartz source

coarser (igneous-metamorphic rock quartz mixed with finer quartz from low-temperature sources such as chert) (13,18,28), rather than oxygen isotopic exchange in an outer skin, as shown by stripping off of the outer 35 to 45% of the quartz with HF (18, 37). Use of the quartz oxygen isotopic ratio as a guide to provenance of aerosol dust would be impaired if the isotopic composition changed much under the environmental conditions in the deposit; however, this research has shown that the change in $\delta^{18}\text{O}$ in quartz silt (1 to 10 μm in diameter) is not a problem under several thousand feet of burial or intensive surficial weathering (47). Quartz oxygen isotopic ratio, therefore, appears to be a reliable indicator of quartz provenance.

(c). Oxygen isotopic tracing of dust origin in major aerosolic dust systems. Tropospheric aerosols from major dust storms arising in cultivated agricultural areas in western Texas and adjacent areas of Arizona, New Mexico, Oklahoma and Colorado were collected with a ground-based airturbine sampler with stack 3-m high and by airplane-borne dust samplers (49). Also, composite soil samples from 13 soil mapping groups strongly affected by wind erosion were collected (48). The quartz in the 1-10 μm fraction from the soil samples gave oxygen isotopic ratios ($\delta^{18}\text{O}$) in the range of $19.6 \pm 0.3\%$. (48). The $\delta^{18}\text{O}$ of this fraction of the Southwestern U.S.A. dusts fell in the interval $18.4 \pm 0.8\%$. (49). The slightly lower value for the fine quartz silt of the dusts was attributed to wind abrasion of coarser particles into the 1-10 μm fraction (49). The oxygen isotopic ratios of the aerosol-sized quartz from the soils and dusts indicate that the southwest U.S.A. (the dustiest area of the continent)

may serve as a reservoir of dusts that move northeast through the mid-continent (27). The micaceous vermiculite of such dusts is able to adsorb and fix radioactive ions from the air (17; as reviewed above), as well as nucleate rain and hail. Some of the dust may enter Easterly winds and rain out into the North Pacific Ocean as pelagic sediments and on surface soils of Hawaiian mountain tops as aerosol size (1 to 10 μm diameter) quartz with $\delta^{18}\text{O} = 18.3 \pm 0.2\%$ in soils and lake sediments of Hawaii (48), just as do the dusts from Asia coming in on the Westerly winds (8,13,18). Eolian deposition of soil horizons over other types of parent material has been noted in North Carolina and the Caribbean (8).

The oxygen isotopic ratios ($\delta^{18}\text{O}$) of whole shale quartz, and of the fine quartz silt fraction (1-10 μm ; long range aerosol size) of shale quartz, were determined ($20.7 \pm 1.8\%$) for 30 marine shale quartz fine silts representative of soil parent material (via glacial, fluvial, and eolian processes) from the Phanerozoic geologic column in the midcontinental U.S.A. (40). The range and standard deviation is wider than for the aerosol size quartz of the Pacific and Texas areas, suggestive of more homogenization of the aerosols by wind mixing (Table 1). The dynamics of water and wind erosion and soil formation which are responsible for narrowing the quartz oxygen isotopic ratio range by compositing from diverse sources also account for the nearly ubiquitous occurrence of $^{137}\text{Cs}^+$ fixing micaceous vermiculite in soils and sediments (17).

The aerosolic dusts containing micaceous vermiculite deposited in Hawaii (13) and in North Pacific pelagic sediments (18) contained quartz

having oxygen isotopic ratios, $\delta^{18}\text{O} = 17.6 \pm 0.5$ and $17.5 \pm 0.5\%$, respectively (53). In the South Pacific area, the quartz both in dusts and pelagic sediments had lower $\delta^{18}\text{O}$ values of 12 to 15‰ (18,19,21). Analyses of 1-10 μm quartz of 28 soils and sediments of southern Africa (51) gave a mean $\delta^{18}\text{O}$ of $12.2 \pm 2.1\%$ for part of this Southern Hemisphere continent in the latitude of the South Pacific area, so the trend may be global. Both sets of values are intermediate between those of igneous-metamorphic (high temperature, low $\delta^{18}\text{O}$) quartz and quartz of chert in sediments (low temperature origin, high $\delta^{18}\text{O}$), but in different proportions in sediments of the two Hemispheres (53). A composite mixture of these two sources in dusts, silty sediments, and soils formed thereon is postulated as the source of atmospheric dusts.

The difference in oxygen isotopic ratios of 1-10 μm quartz between the Northern and Southern Hemispheres is explained (53) as follows: During the several hundred million years of the Phanerozoic Eon, the Northern Hemisphere continents were located in tropical and equatorial latitudes (Fig. 1,B). The soils were subject to the usual intense desilication at these latitudes, with an accompanying discharge of soluble $\text{Si}(\text{OH})_4$ into rivers and oceans where biogenic silica was deposited and from which quartz (chert) gradually formed at low temperature (53). Northern Hemisphere continents were carried northward (Fig. 1,A) across the equator by plate tectonics (28), during and subsequent to the Cretaceous Period, the ocean sediments were uplifted to form great continental plains with their abundance of low-temperature, high $\delta^{18}\text{O}$ quartz. Some land areas in Southern Hemisphere were part of a frigid South Polar supercontinent,

Fig. 1.--Continental drift according to paleomagnetic and paleoclimatic data (after Drewry *et al.*, 1974, *J. Geol.* 82:531-553 used by Jackson (reference 53) to explain the major contrast of low oxygen isotopic ratios ($\delta^{18}\text{O}$) of aerosol size quartz in the majority of late Precambrian to Triassic sediments and Quaternary Pacific pelagic sediments of the Southern Hemisphere, compared to the higher ratios in those of the present Northern Hemisphere continents and North Pacific pelagic sediments (A). The differences are inherited from the continental positions in the earlier interval (B). Evaporites accumulated authigenic fine quartz silt (aerosol size) crystallized at temperate to equatorial temperatures (higher $\delta^{18}\text{O}$) in the mid-latitudes (B), while glacial till derived from igneous rocks of Gondwanaland suture mountains contains mainly quartz formed at igneous and metamorphic temperatures (lower $\delta^{18}\text{O}$) (B).

Gondwanaland, and associated seas in late Paleozoic time. Glacial and fluvial erosion of predominantly igneous and metamorphic rocks, with low $\delta^{18}\text{O}$ values of quartz from the mountains of continental sutures, carried silts onto continental shelves during the late Carboniferous, Permian, and Triassic periods (51). These continental shelves were uplifted into lands as plate motions carried the Southern Hemisphere continents northward into their present positions, along with the low $\delta^{18}\text{O}$ quartz in their silty sediments (53). Of course, large portions of the sediments of these continents were formed before and after the Gondwanaland glacial periods and some areas extended into warm climates (coal beds associated) in time of Gondwanaland's existence. The carbonate and hydrothermally influenced sediments laid down at those times and places contain quartz with oxygen isotopic ratios similar to those of Northern Hemisphere quartz (Fig. 2; 53). The micaceous vermiculites occurring ubiquitously in soils (and important in radionuclide fixation, as in the Oklo natural reactor phenomenon in Africa) arose from the weathering of micas present in the sedimentary rocks (and their igneous and metamorphic precursors) which were spread by tectonic, eolian, fluvial, and glacial processes (53).

Fig. 2. Relations of Northern and Southern Hemisphere quartz oxygen isotopic ratios ($\delta^{18}O$) of long range aerosol size (1-10 μm) particles (Jackson, (53)).

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U.S. ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION WITH THE UNIVERSITY OF WISCONSIN

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3	x	x	Roth et al., ISSS, Aberdeen (1967) Verm sesquioxide coatings	1 Oct 66
(4)	x	na	Jackson, Trip Report, Aberdeen	1 Oct 66
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11	x	na	Science Information Exchange form (1967)	14 Feb 67
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13	x	x	Roth et al., Israel J. Chem. (1968) Fe coatings/CEC	30 June 67
14	x	x	Jackson, ISSS, Adelaide (1968) pH/CEC	1 July 67
15	x	x	Dolcater et al., SSSAProc. (1968) CES/mica-verm layer charge	15 Dec 67
16	x	x	Syers et al., Geo.CA (1968) Quartz Isol/H ₂ SiF ₆	14 Feb 68
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33	x	x	Mokma et al., J. Soil Sci. (1973) ASL dust/isotopes	26 Feb 71
34	x	x	Jackson et al., SSSAProc. (1973) SEM/verm	26 Apr 72
35	x	x	Brown, Jackson, CCMIn. (1973) Ultramicrot./layers	26 Apr 72
36	x	na	Technical Progress Report (1968-71)	26 Feb 71
37	x	x	Sridhar et al., Am. Min. (1972) Blisters on verm/cations	26 Apr 72
38	x	x	Kinniburgh et al., SSSAProc. (1975) Sr, Ca sorp/oxides	26 Apr 72
39	x	na	Science Information Exchange Form (1971)	6 Feb 71
40	x	na	Science Information Exchange Form (1972)	6 Apr 72
41	x	na	Technical Progress Report (1972)	26 Apr 72
42	x	x	Sridhar et al., CCMIn. (1973) Blisters on verm/fixing cations	5 Aug 72
43	na	x	Jackson et al., Geoderma (1972) ASL dusts/isotopes	4 Sept 72
44	x	x	Lee et al., CCMIn. (1975) Mica occlusions kaol/ ¹³⁷ Cs fixation	10 July 73
45	x	x	Lee et al., SSSAProc. (1974) ²³⁸ U Fission tracks/dating	29 Oct 72
46	x	x	Veith, Jackson, CCMIn. (1974) Layer charge change/cation ejection	4 Sept 72
47	x	na	Technical Progress Report (1973)	6 Apr 73
48	x	x	Jackson/Sridhar, SSSAProc. (1974) SEM/verm weathering	2 Nov 73
49	x	x	Jackson et al., Soil Sci. (1973) Global dusts/meteorology	4 May 73
50	x	na	Science Information Exchange Form (1973)	16 Apr 73
51	x	x	Lee et al., SSSAProc. (1975) Mica verm/ultramicrotomy	10 Aug 73
52	x	x	Kinniburgh et al., SSSAProc. Transition metal sorption	15 Mar 75
53	x	x	Churchman et al., J.Geophys.R. Source reservoir eolian materials	30 Apr 74
54	x	x	Jackson ISSS (Moscow) (1974) Eolian additions to soils	1 May 73
55	x	x	Sridhar/Jackson SSSAProc. (1974) Verm layer charge decrease	12 Nov 73
56	x	na	Technical Progress Report (1971-74)	29 Apr 74
57	x	na	Science Information Exchange Form (1974)	29 Apr 74

* Numbers refer to published papers, Technical Progress Reports, and Science Information Exchange Forms in the COO-1515-x series. Numbers in () incorrectly used for other than Technical Progress Reports.

x = Submitted to U.S. AEC/ERDA with earlier reports. na = Not applicable.

Key to C00-1515 Report Series (Cont.)

Sent			Author, Journal, Content	Manuscript
C00-1515	Ms.	Re-print		
58*	x	x	Henderson et al., CCM (1976), Verm CaCO ₃ /fixing cations	11 April 75
59	x	x	Sridhar et al., SSSAJ (1975), Isotopic ratio/source	13 March 75
60			Jackson, Lee, CCM, Chlor-mica ultramicro/elect. mic.	In preparation
61	x	xx	Lee, Jackson, SSSAJ, Fission particle/layer charge	1 May 76
62	x	xx	le Roux, Clayton, Jackson, GCA, Oxygen isotopic ratio	1 May 77
63	na	xx	Koons, Helmke, Neutron activation analysis	25 Oct 77
64	na	xx	Kinniburgh, Jackson, SSSAJ, Hg(II) ³⁶ Cl sorp./Fe	17 Jan. 77
65	x	x	Jackson, Sayin, Clayton, SSSAJ, H ₂ SiF ₆ reagent/loess	28 Jan. 76
66	x	xx	Clayton, Jackson, Sridhar, GCA, Quartz isotopic resist.	24 Mar. 76
67	x	na	Technical Progress Report (1975)	27 April 75
68	x	na	Science Information Exchange Form (1975)	27 April 75
69	na	xx	May, Helmke, Jackson, Chem. Geol., Al determ at 0.1 ppb	21 July 77
70	x	x	Sayin/Jackson, CCM, SEM cherts	12 Feb. 75
71	x	x	Jackson, Lee, Helmke, Soil Sci., U cont. fission tra	4 Mar. 76
72	x	xx	Kinniburgh, Sridhar, Jackson, Battelle Conf., Zn, Cd sorp.	1 Sept. 75
73	x	na	Technical Progress Report (1976)	26 Apr. 76
74	x	na	Science Information Exchange Form (1976)	26 Apr. 76
75	na	xx	Sridhar et al., SSSAJ, Oxy. Iso. Ratio/dust TX	10 Jan. 77
76			Koons, Helmke, Jackson, GCA, Cs retention during mica wea.	30 Sept. 78
77	na	xx	Gillette, Jackson et al., J. Appl. Meteor., Oxy. Isotopic Ratio dust/TX	10 June 77
78	x		Jackson, GSA Memoir, Oxygen Isotopes/dusts	15 June 77
79	x	na	Technical Progress Report (1974-1977)	26 July 77
80	x	na	Science Information Exchange Form (1977)	26 July 77
81	xx		Kinniburgh, Jackson, SSSAJ, Radioactive Ca, Zn	20 June 80
82	x	xx	Koons, Helmke, Jackson, SSSAJ, Cs retention in wea. zone	3 Aug. 78
83	x	xx	Lim, Jackson et al., CCM, Cs retention in clays	10 July 78
84	xx	na	Jackson, Final Technical Report	31 Oct. 78

*Numbers refer to published papers, Technical Progress Reports, and Science Information Exchange Forms..

x = Submitted to U.S. AEC/ERDA with earlier reports.

xx = Sent with Technical Progress Report (C00-1515-84).

na = Not applicable.

EFFORT LEVEL OF PERMANENT PERSONNEL:

Principal Investigator, Marion L. Jackson, 0.25 man-year equivalent, for 12 months (25% of time in year to end of contract)

Co-Investigator, Philip A. Helmke, 0.15 man-year equivalent, for 12 months (15% of time in year to end of contract)