

is thought to be very important (8).

The wood replaced by tridymite varies in color from white to yellow-brown, to brownish-gray, to dark brown; it is very soft, dull, and fibrous (asbestiform), resembling decayed wood. The fibers are easily pulled apart, but they lack the tensile strength of asbestos. An x-ray study of several fibers showed the tridymite to be present as exceedingly fine, randomly oriented grains unrelated to the fiber axis. The wood fragments range from very small to several centimeters in length. The material is very abundant in the host rock, an impure, gray, medium-grained arkose.

The arkose, which is composed of gray quartz and kaolinized feldspar, is also host to small pyrite concretionary masses; gray clods of a mixture of kaolinite, white mica, and quartz; large quartz pebbles; and charcoal-like fragments (amorphous to x-rays) often having pyrite inclusions. Casts of large tree limbs ranging up to 2 m in length are also in the rock. Although I made no attempt to identify plant materials, other studies (9) of the Patuxent formation, based primarily upon leaf impressions in clay, have shown a flora composed of numerous species. A study of weathering products and secondary minerals associated with the arkose, and an underlying montmorillonite bed at the exposure (10), has shown cobaltian pickeringite, alunogen, melanterite, and jarosite.

The exposure, a low, overhanging cliff about 50-m long, is on Hazel Run within the city of Fredericksburg, about half-way between Lafayette Boulevard and Jefferson Davis Highway.

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Manganese and Related Elements in the Interstitial Water of Marine Sediments

Abstract. Analyses for manganese, nickel, iron, cobalt, sodium, and lithium in the interstitial water of cores from the southern California borderland and six deep-sea cores in the area of the East Pacific Rise show great variation in concentration of trace elements. Oxidizing near-shore sediments showed a 50-fold enrichment in manganese in contrast to sulfide-rich reducing sediments, which showed no enrichment. Deep-sea sediments were variable in their concentration of the trace metals. All but one core showed a high enrichment in dissolved manganese, with a maximum of 6.6 parts per million. Two cores showed a 100-fold enrichment in nickel and cobalt. The manganese appears to be in solution either as Mn^{2+} or as a complex. The results appear to support manganese nodule formation in deep-sea sediments through a diffusion of manganese from depth to the surface.

Concentrations of manganese in deep-sea sediments are high compared with terrestrial igneous and sedimentary rocks, and many workers (1-5) have speculated about the origin of the manganese. Earlier work has been confined mainly to analysis of the solid phase, owing to lack of sensitive methods for the analysis of interstitial waters.

A sensitive analytical technique has recently been developed in our laboratory for the analysis of dissolved trace metals in the parts-per-billion range by a combination of solvent extraction and atomic absorption spectrophotometry (6). This method has been applied to the analysis of several constituents in interstitial water from sediments of the California continental margin (7) and in several deep-sea cores collected by the Scripps Institution of Oceanography in the East Pacific area.

The results reported in this study are from the two areas described above and shown in Fig. 1. The California Borderland sediments were collected in a (25- by 40-cm) polystyrene-coated stainless-steel box corer, and deep-sea cores were taken in a polythene-lined pipe 5 cm in diameter. The interstitial water was removed via a Teflon-lined, gas-operated squeezer (8). Eh and pH measurements were made on the water immediately after removal, minimizing changes occurring through gas exchange. The water was then filtered through a 0.45- μ millipore filter and acidified to pH 3. Manganese, iron, nickel, and cobalt were complexed by adding ammonium pyrrolidine dithiocarbamate to the samples and to standard controls, and extracting the complexes into a small volume of methylisobutyl ketone. Incomplete extraction is corrected for by comparing the samples with standards extracted in the same way (6).

The extracts were analyzed within 30 minutes of phase separation. In all but two cores (Nos. 2 and 8) manganese concentration was sufficiently high for direct analysis of the interstitial water. Sodium and lithium were determined directly by flame emission analysis, with diluted seawater used as a standard for both elements (7). The solid sediment was analyzed by atomic absorption spectrophotometry after decomposition of the sediment with aqua regia, evaporation, and resolution of the metal ions in dilute hydrochloric acid.

The analytical results for the waters are given in Table 1. Sodium was analyzed in all samples as a reference in order to indicate if salinities were similar to average seawater. It can be seen that the California Borderland samples were on the average 1 to 2 percent higher, whereas the deep-sea brines were about 4 to 5 percent higher than values for coastal seawater.

In the waters from the two coastal sediments, iron and nickel showed higher concentrations in the reducing, sulfide-rich Santa Barbara Basin (core No. 2) than in the oxidizing Santa Cruz Basin (core No. 1). Cobalt showed little concentration in relation to seawater, although the highly reducing interstitial water appeared to be somewhat more enriched, especially at the surface of the core.

In view of the published thermodynamic data on manganese (2, 9, 10), it was expected that manganese should follow iron and would be most soluble in the Santa Barbara Basin at a low Eh. The results of the analysis show, however, that in the Santa Barbara Basin, manganese was slightly depleted with respect to its normal concentration in seawater, whereas in the oxidizing Santa Cruz Basin sediments it was concentrated by a factor of 30 to 50.

This unexpected result led to the analysis of several gravity cores (Nos. 3 to 6, and 8) and a piston core (No. 7) from the East Pacific Rise in the general area of the East Pacific Rise (Fig. 1). The sediment was fine-grained, containing abundant coccoliths but relatively low quantities of forams. Eh values (Table 1) showed core No. 7 to be the most oxidizing, whereas No. 8 was relatively the most reducing of this series of cores.

In the interstitial water of the East Pacific Rise sediments, iron (with the exception of one sample in No. 4) showed no marked increase over that in oxidizing near-shore basins such as Santa Cruz, whereas cobalt showed a significant increase over its concentration in seawater. Nickel concentrations were variable and, in all but one core (No. 8), nickel concentration was, by as much as two orders of magnitude, higher than in near-shore basin sediments. Two important correlations are apparent here. First, the nickel concentration does not appear to follow that of iron as it does in near-shore sediments. Second, cobalt appears to correlate with nickel, as in cores Nos. 3 and 5.

Manganese again showed the greatest variation in concentration. With the exception of core No. 8, its concentration showed an increase from 50- to 3,000-fold over its value in seawater. The distribution was quite random and did not appear to follow any pattern with depth down the column. The longest relatively oxidizing core showed the least changes, being consistently enriched in manganese. The most-reducing cores (Nos. 2 and 8) showed little enrichment of dissolved manganese or any of the other trace metals measured.

Thermodynamic calculations indicate (2, 9) that above Eh +400 at pH 7.8, the stability field of insoluble MnO_2 is entered and the concentration of Mn^{2+} should drop below 500 parts per billion. Colorimetric studies by J. J. Morgan, using crystal violet in acetate buffer at pH 4.0 on several cores, indicated that manganese was present only in the reduced form. It therefore exists either as ions such as Mn^{2+} , $MnHCO_3^+$, the ion pair $MnSO_4$, or as an organic complex (11). Such ion pairs may stabilize Mn^{2+} somewhat, preventing its rapid oxidation to MnO_2 (9).

This anomalous behavior of manganese in relation to redox potential has also been observed in the Dead Sea,

where concentrations of this element reach 120 parts per million in the interstitial water of shallow near-shore oxidizing sediments and drop to 10 parts per million in the water of the deeper reducing sediments (12). Here, however, the pH is in the range of 5.7 to 6.2.

Two questions may now be asked: What is the reason for the variability in the manganese (and nickel) content and does the high manganese concentration in interstitial water bear any relationship to the formation of manganese nodules on the ocean floor?

The data show that the high redox potential of oxidizing sediments is apparently not the sole cause for removal of manganese from solution and there is a surprisingly great difference in concentration in cores coming from the

same province. The assumption that such differences are due to *in situ* leaching processes of pelagic sediment by trapped seawater is illogical and unsupported by any data. Recent analytical results from studies on Red Sea brines and interstitial water (13) show similar great differences in trace metal concentration between adjacent basins, and it is obvious that the source of the metals is at depth and not concentration from the overlying water (14). In both the Dead Sea and Red Sea, brines and interstitial waters are highly enriched in lithium, relative to sodium and potassium. Although these environments are different from the East Pacific area in terms of overall salinity, measurement of lithium concentration was undertaken in the oceanic cores (Table 1) for comparison and to determine if an

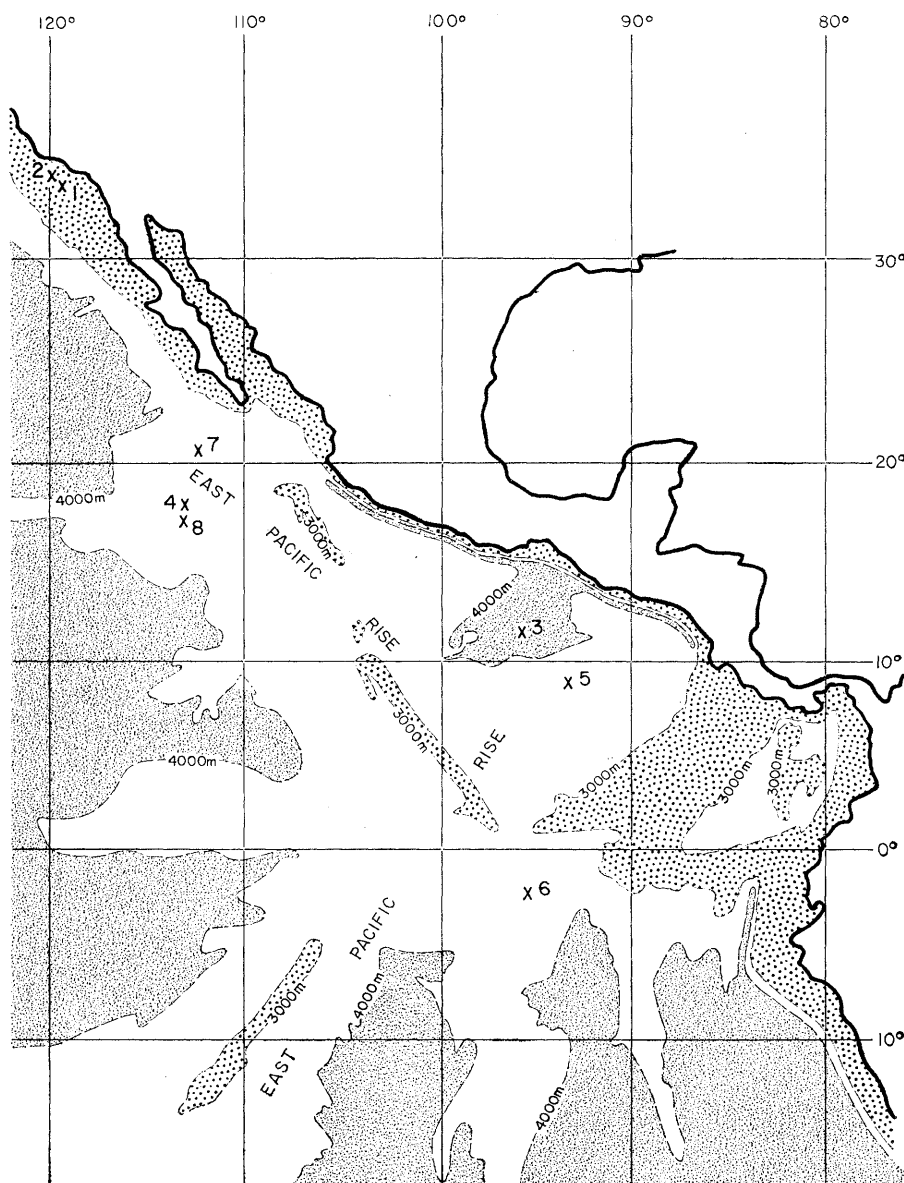


Fig. 1. Map of East Pacific Rise, showing sampling points.

anomaly exists. Our results for lithium showed that whereas the near-shore cores were close to the Na/Li ratio of seawater (6×10^4), the deep-sea cores showed more variable results, and in the two cores where nickel was most abundant the ratio dropped to 5×10^4 . The lithium enrichment was not as noticeable, however, as in the Red Sea brines, where the ratio decreased to approximately 10^4 (13). Only in one sample analyzed in this study did the lithium appear anomalously high, giving a value of 492 parts per billion. No ready explanation is available for this value (15). The high lithium in the hypersaline brines mentioned above is

due in part to leaching of salt deposits and sediments and in part to contribution of hydrothermal exhalations, whereas the source material in the Pacific for the high manganese and nickel is probably only slightly enriched in lithium.

The source of the high manganese is probably variable and depends on the locality. In normal continental shelf environments it is probably due to the leaching of detrital terrigenous sediments, precipitation out of solution as a colloid, or as a colloid adsorbed to clay particles. Since rivers carry on the average about 70 parts of manganese per billion and it is present in coastal

waters in about the same abundance as open ocean water (1 to 3 parts per billion), it must be removed rapidly after introduction into the sea. In the deep-sea sediments, especially those close to mid-oceanic rises or in areas of active submarine vulcanism, two additional sources must be added: (i) contribution of volcanic products which are leached *in situ*, and (ii) injection from depth of metal-rich "juvenile" water. Both of these sources can be very localized (16). A third source, important in areas of high productivity, is biological concentration of metals from the sea and their subsequent introduction into the sediment. This may be the

Table 1. Trace elements in the interstitial water of marine sediments. Concentrations are in percent for sodium and parts per billion for other elements.

Core No.	Position of core	Description	Depth (cm)	Mean Eh (mv)	Mean pH	Na (%)	Co (ppb)	Fe (ppb)	Ni (ppb)	Mn (ppb)	Li (ppb)
1	33°40'N, 119°29'W, at 1900 m	Grey-green mud with no detectable sulfide odor	0-5	+400	8.2	1.07	<0.3	15	3.5	74	176
			5-10			1.09	<0.3	18	4.1	74	191
			10-15			1.07	<0.3	4.0	6.8	94	152
			15-20			1.06	<0.3	4.5	3.3	87	155
			20-25			1.07	<0.3	4.0	2.5	72	186
			25-30			1.09	<0.3	5.5	2.8	95	170
			35-40			1.09	<0.3	4.5	1.5	60	186
2	34°06'N, 119°59'W, at 567 m	Black mud with strong sulfide odor	0-5	-90	7.8	1.06	2.7	51	6.5	1.5	159
			5-10			1.05	2.4	48	6.5	1.0	165
			15-20			1.09	2.5	34	9.0	3.0	188
			20-25			1.05	<0.3	13	4.2	<1.0	147
			25-30			1.06	<0.3	18	2.8	1.5	100
			30-35			1.06	<0.3	31	3.0	1.0	132
			35-40			1.06	<0.3	23	3.7	1.6	111
40-45	1.06	1.7	49	5.8	2.0	114					
3	11°40'N, 95°23'W, at 4141 m	Green-grey clay, 1-cm layer brown mud at top	0-40	+380	8.0	1.12	5.6	7.2	340	1480	223
			40-80			1.12	20	8.2	210	4000	217
			80-125			1.12	18	8.7	400	5930	223
4	17°30'N, 113°00'W, at 3696 m	Brown clay with light striations	0-10	+320	7.7	1.05				2100	492
			10-70			1.07	3.6	30	16	450	226
			70-130			1.11	4.2	7.5	19	307	196
			130-175			1.11	6.2	6.6	19	385	193
5	8°53'N, 93°15'W, at 3493 m	Green-grey clay streaked with black	0-40	+370	7.9	1.11	12	7.1	240	525	226
			20-66			1.11	12	7.8	256	1850	226
6	2°00'S, 96°02'W, at 2506 m	Calcareous ooze, 5 cm red clay at top	0-50	+410	7.7	1.11	2.5	3.4	28	168	217
			50-90			1.11	2.2	4.4	20	92	199
			90-130			1.11	4.2	3.9	23	267	208
7	20°15'N, 112°42'W, at 3527 m	Red clay	0-40	+420	7.7	1.11	4.0	1.9	12	5000	214
			40-80			1.08	3.9	1.9	12	6600	209
			80-120			1.08	3.1	2.4	12	6400	205
			120-150			1.09	2.6	4.7	19	5720	268
			150-180			1.09	4.5	3.4	13	4700	202
			180-210			1.10	3.9	4.8	11	4560	196
			210-240			1.11	4.2	3.0	13	5700	196
			240-270			1.11	4.5	4.4	21	5750	196
			270-330			1.11	5.0	3.8	15	5580	187
			330-390			1.11	4.0	4.4	14	5250	192
			390-450			1.11	2.9	4.4	13	5500	196
			450-500			1.11	4.0	4.4	14	5970	187
			500-560			1.11	5.0	4.8	14	5770	176
			560-600			1.11	6.5	7.6	15	5870	179
			8			16°46'N, 113°10'W, at 3410 m	Brown clay with light mottling	0-40	+290	7.7	1.09
40-80	1.09	1.5		2.6	1.4			10			
80-120	1.09	1.5		2.6	1.4			10			
120-160	1.09	2.3		4.4	2.6			10			
Sea water off coast of southern California (7)				+450		1.05	0.1	2	2	2	170

cause for enrichment in highly calcareous or siliceous oozes.

Having established the fact that manganese is enriched in the interstitial waters of deep-sea cores, it may now be possible to consider the mechanism of the formation of manganese nodules from the dissolved manganese. Several authors, in particular Arrhenius and his co-workers (3-5), have stressed the variability of the composition and occurrence of the manganese nodules. Whereas the abyssal floor may be littered with nodules in one area, an adjacent area may be barren. These authors have further pointed to the possibility of diffusion of soluble manganese as being an important mechanism in accretion of this element at the surface. By assuming a concentration of approximately 1 part per million and a diffusion coefficient of 2×10^{-5} cm²/sec, Lynn and Bonatti (5) calculated that diffusion of manganese can account for one to two orders of magnitude more than is necessary for accretion of this element in the nodules. The finding of over 6 parts per million in some sections certainly enhances the possibility of such an accretion mechanism.

Wide variations in the composition of the manganese nodules are evident in the Pacific occurrences (1, 3). In general, the Mn/Fe ratio is between 1 and 10, although the bulk composition of the sediment may show a Fe/Mn ratio of 5 to 20 (17), with variable amounts of nickel, cobalt, and copper. The variation in dissolved nickel and cobalt is evident from our data (Table 1), but semiquantitative analysis of copper and zinc showed no anomalous enrichment in the interstitial water over seawater.

Analyses of the bulk sediments in Table 2 show differences in concentration among the different cores of the present study but reasonably good internal constancy in any single core. The Continental Borderland samples were low in manganese, nickel and cobalt. The calcareous core (No. 6) was also low in these elements, with the exception of manganese in the surface sample. A high manganese anomaly did appear at the top of the core in four out of the six cores, supporting Lynn and Bonatti's (5) arguments for upward diffusion. The differences in concentration of the trace elements in the bulk material from different cores further points to localized sources of supply (16).

Table 2. Concentrations (percent or parts per million) of ferrides in the solid phase of marine sediments.

Core No.	Depth (cm)	Fe ₂ O ₃ (%)	MnO (%)	NiO (ppm)	CoO (ppm)
1	0-5	3.83	0.030	61	14
	40-45	4.91	.032	65	14
2	0-5	4.05	.027	59	15
	40-45	4.96	.041	64	15
3	0-20	6.00	.206	249	32
	60-80	5.56	.161	354	52
	100-125	3.14	.155	204	22
4	0-10	6.50	3.100	407	93
	50-70	7.21	0.819	224	87
	150-175	6.93	.561	181	71
5	0-20	4.50	.155	314	34
	20-40	5.14	.193	314	34
	40-66	4.14	.206	294	30
6	0-10	0.86	.278	59	25
	50-70	1.14	.045	53	20
	110-130	0.43	.039	47	20
7	0-20	6.50	1.200	264	89
	300-310	5.71	0.786	154	71
	580-600	5.85	1.030	198	71
8	0-20	6.35	0.945	152	76
	60-80	6.56	1.188	191	73
	140-160	6.06	1.035	179	69

To summarize, it may be stated that the relatively high concentrations of manganese and nickel in deep-sea cores of the East Pacific have a different origin from the dissolved metals of continental shelf sediments. The latter are probably derived from terrigenous weathering, whereas the former have, in addition, contributions from shallow vulcanism (in the form of pyroclastic ejecta) or deep-seated introduction of volatiles into the sediment. A volcanic layer may produce localized concentrations at any one depth, whereas injection from depth may produce more uniform vertical distribution of the trace metals. Our measured values show that over 1000 parts of manganese per billion can be maintained in solution at an Eh of +400 mv at a pH of 7.8, whereas iron appears to be effectively removed (probably as ferric oxide). Low redox potentials are often associated with dissolved hydrogen sulfide and metabolic bicarbonate, which may remove manganese from solution. Diffusion of manganese from depth to the surface, either along a concentration gradient or due to compaction of the sediment, will cause this element to reach the surface of the sediment. Here the Mn²⁺ may become oxidized to MnO₂ due to the presence of dissolved molecular oxygen. Colloidal manganese oxide will accrete with iron oxide and catalyze the precipitation of nickel and cobalt. Other trace elements, both in the

seawater and dissolved in the interstitial water will be adsorbed at the surface of the ferro-manganese oxide particles and will ultimately be incorporated into minerals of larger nodules (18).

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Stanfieldite: A New Phosphate Mineral from Stony-Iron Meteorites

Abstract. A new mineral, stanfieldite, $\text{Ca}_4(\text{Mg,Fe})_5(\text{PO}_4)_6$, has been found in the Estherville mesosiderite and several pallasites: Santa Rosalia, Albin, Finmarken, Imilac, Mount Vernon, and Newport. The atom ratio Mg:Fe of this mineral varies from 1.5 in Estherville to a constant ratio of about 15 in the pallasites. X-ray, optical, and chemical data for the mineral resemble those for the only intermediate compound in the system $\text{Mg}_3(\text{PO}_4)_2$ - $\text{Ca}_3(\text{PO}_4)_2$.

Stanfieldite (I), $\text{Ca}_4(\text{Mg,Fe})_5(\text{PO}_4)_6$, a mineral unknown in terrestrial rocks, has been discovered in trace amounts (less than 1 percent) as irregular-to-subhedral grains, up to 1 mm in diameter, concentrated along the walls of fracture cracks in two specimens examined of the Estherville mesosiderite. Merrill (2) described this meteorite, a fall, noting the presence of an unidentified calcium phosphate mineral. Since stanfieldite is here associated with whitlockite [ideal formula, $\text{Ca}_3(\text{PO}_4)_2$], it is not known which was observed by him because both minerals were then unidentified. Stanfieldite also occurs in six pallasites, all of which are finds.

The mineral assemblage in Estherville indicates that nonequilibrium conditions existed during the history of this meteorite. Thus tridymite is present within 0.1 mm of olivine; and the iron and magnesium contents of the orthopyroxene show large variations from grain to grain, although no zoning of these elements was observed within grains that were removed for electron-microprobe analysis. The mole percentage of the ferrosilite molecule (FeSiO_3) varies from 14 to 32 in orthopyroxene; that of the fayalite molecule (Fe_2SiO_4) varies from 26 to 34 in olivine. Other minerals present are taenite, kamacite, troilite, schreibersite, ilmenite, chromite, and calcium plagioclase (An_{90}). Lamellae observed in the chromite have been shown to be rutile (3).

Three phosphate minerals were identified in the pallasites studied: stanfieldite, whitlockite, and farringtonite

[ideally $\text{Mg}_3(\text{PO}_4)_2$]. Stanfieldite was the only phosphate found in Albin, Newport, Finmarken, and Imilac; it occurs with whitlockite in Santa Rosalia and Mount Vernon; only farringtonite was found in Krasnojarsk, Phillips County, and Springwater—the latter was the “type” meteorite in which farringtonite was discovered (4). All identifications were confirmed by x-ray powder photographs. No phosphates were found in Admire, Glorieta Mountain, or Brenham (5).

The phosphate minerals in the pallasites occur as thin veinlets, a fraction of 1 mm in width, penetrating cracks in olivine, or as pockets up to several millimeters in diameter in the olivine matrix. One inclusion of stanfieldite in Newport was practically enclosed by troilite. One should mention that the observed occurrences are limited by the size and nature of the particular specimens available; more suitable samples may reveal findings other than those I report for these particular samples.

However, a special effort was made to find stanfieldite or whitlockite, or both, in meteorites containing farringtonite, with negative results. Wahl (6) has reported farringtonite from Newport and three other pallasites not available to me. If his identifications are correct (his evidence is not reported), Newport contains both stanfieldite and farringtonite. The association of these two minerals is important for fixing of the range of the calcium:magnesium ratio in the phosphatic material prior to crystallization.

Other minerals in the pallasites are

given by Mason (7) as kamacite, taenite, troilite, schreibersite, olivine, chromite, and lawrencite; they do not include plagioclase, orthopyroxene, or tridymite. I have found copper metal in Newport and Mount Vernon. Mason reports that the olivine is of uniform composition within each pallasite; thus, unlike the Estherville occurrence, stanfieldite in the pallasites probably crystallized in an equilibrium environment.

Under the microscope, stanfieldite is generally clear and transparent but has a reddish-to-amber tint when viewed in a specimen. Pockets of the mineral in Finmarken and Mount Vernon are bluish-white because of weathering (8). No discernible cleavage was noted for any of the occurrences. The optical properties for the mineral in Estherville follow: biaxial positive; refractive indices for sodium light are $\gamma = 1.631$, $\beta = 1.622$, $\alpha = 1.619$ (all ± 0.002); $2V$, $50^\circ \pm 2^\circ$. The indices for the mineral in Santa Rosalia are lower: $\gamma = 1.604$ and $\alpha = 1.594$ —probably because of its lower FeO content. The mineral in Albin had the largest $2V$, 55° to 60° ; otherwise the indices equal those in Santa Rosalia.

Stanfieldite is monoclinic; x-ray powder data appear in Table 1. Cell constants, determined from Weissenberg photographs of the Estherville material, are: a , 17.16 ± 0.03 Å; b ,

Table 1. X-ray powder diffraction data for stanfieldite from the Estherville meteorite. Norelco powder camera (diameter, 11.45 cm); $\text{CoK}_{\alpha 1, \alpha 2}$ Fe filter. Diamond powder used as an internal standard. There were 49 additional lines of intensity 2 or less below 1.847 Å.

Relative intensity	dÅ observed	Relative intensity	dÅ observed
5	8.31	10	2.817
5	6.01	2	2.734
½	5.412	3	2.695
3	5.006	½	2.651
½	4.810	½	2.603
½	4.603	8	2.505
1	4.340	1	2.414
½	4.209	½	2.348
½	4.110	2	2.294
½	3.941	1	2.264
6	3.845	1	2.210
8	3.747	2	2.170
½	3.596	2	2.130
3	3.256	1	2.080
½	3.196	2	2.014
1	3.098	1	1.961
3	3.048	½	1.917
1	2.959	½	1.888
		4	1.870
		1	1.847