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A major oceanic redox condition change correlated to the rebound of marine animal diversity during the Late Ordovician

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Analytical Methods:

TOC and Pyrite: Each sample was crushed and milled to a uniform powder in an automated agate mortar device. For organic carbon analysis, about 2 g of powdered sample were treated with 6 N HCl for 24 h to remove carbonate minerals, followed by washing and filtering of the residue. The residue was then dried and weighed. Organic carbon contents were determined using a NC Instruments NC 2500TM elemental analyzer. Pyrite was quantitatively extracted by the chromium reduction method of Canfield et al. (1986), and the sulfide liberated was precipitated as Ag₂S. The precipitate was weighed to determine the concentration of pyrite sulfur, and the amount of Fe bound in pyrite was calculated with a molar Fe:S ratio of 1:2.

V and Ni: Trace elements of V and Ni were analyzed in this study. Because organic matter cannot be digested completely by microwave oven, we applied a more effective digestion procedure as follows: Firstly, ~1.0 g sample powder was weighted into a porcelain crucible, and was combusted in a muffle oven at 800 °C for 2 h, and weighted again after the crucible cooled. The mass lost on ignition was recorded and used for latter correction. Then, ~100 mg of the ashed sample was weighted into a PTFE microwave digest vessel, followed by adding of 4 ml HNO₃, 1ml HF and 1 ml HClO₄. The mixture was allowed to react for a couple of hours and then subjected to Microwave-assisted heating. After cooling down, the resulting solution was transferred quantitatively into a 100 ml PTFE beaker, and the solution was heated on a hotplate until dry. Subsequently, another 1 ml HClO₄ was added and was heated again till dry to drive out HF. Finally, the material was re-dissolved in 20% HNO₃ and diluted to 50 ml with double distilled water. The obtained sample solution was diluted further 5 folds for ICP-MS analysis (Varian Single Collector Quadropole 820). During analysis, 80 ppb in solution was added in and worked as an internal standard. A commercially available multi-element standard was used to calibrate the instrument externally, and a certified reference material SDO-1 was used to validate this measurement and used for quality control purposes. Results were corrected to the weight of bulk rock, and analytical precision for trace element concentration is usually better than 5%.

Table DR1: TOC, Pyrite contents, and V/(V+Ni) ratios for the Late Ordovician sediments from Poland

Sample	Depth (m)	Stage (I)	TOC (%)	Py (%)	$\delta^{34}\text{S}$		V (ppm)	Ni (ppm)	V/(V+Ni)
					(‰)	V (ppm)			
W63	603.7	K	0.07	0.55					
W62	604.8	K	0.08	0.57					
W61	605.8	K	0.11	0.08	6.4				
W60	606.8	K	0.08	0.08					
W59	607.6	K	0.04	0.20					
W58	608.5	K	0.01	0.24	14.9	48	42.3	0.53	
W57	610	K	0.06	0.18					
W56	611	K	0.02	0.57	21.1				
W55	612	K	0.04	0.30	19.4				
W54	613.2	K	0.03	0.67					
W53	614.2	K	0.03	0.21	25.2	115	79.9	0.59	
W52	615	K	0.08	0.47	15.9				
W51	616	K	0.09	0.03	-2.4				
W50	617.2	K	0.12	0.02					
W49	618.5	K	0.10	0.02	-0.4	116	72.9	0.61	
W48	619.4	K	0.08	0.20	31.8				
W47	620.5	K	0.06	0.66	29.1				
W46	621.5	K	0.08	0.21					
W45	623	K	0.07	0.45	21.0	98	66.5	0.60	
W44	624	K	0.08	0.04					
W43	625.5	K	0.08	0.47	28.1				
W42	626.7	K	0.06	0.31	29.8				
W70	627.6	K	0.05	0.40	9.3	119	91.8	0.56	
W41	628.5	K	0.09	0.01					
W40	630	K	0.09	0.03					
W39	631.5	K	0.07	0.07	4.3	121	86.1	0.58	
W38	633.2	K	0.06	0.08					
W37	634	K	0.06	0.24	32.5				
W36	635.4	K	0.08	0.01					
W35	637.3	K	0.09	0.02	16.7	95	92.9	0.51	
W34	638.4	K	0.10	0.00					
W33	639.2	K	0.15	0.02	14.4				
W32	640.5	K	0.10	0.00					
W31	641.5	K	0.09	0.30	37.4				
W30	642	K	0.06	0.08	21.8	110	82.8	0.57	

W29	643.5	K	0.08	0.01				
W28	645	K	0.07	0.07				
W27	646.2	K	0.15	0.06	-7.3	129	116	0.53
W26	647.6	K	0.07	0.00				
W25	648.6	K	0.10	0.02				
W24	650	K	0.12	0.09	-8.2	135	124	0.52
W69	651	K	0.05	0.20				
W23	652	K	0.11	0.07	18.5			
W22	653.5	K	0.09	0.43	30.1	156	120	0.56
W21	655.5	K	0.09	0.29	23.2			
W20	656.5	K	0.11	0.04	13.4	132	147	0.47
W19	657.5	K	0.12	0.06				
W18	658.5	K	0.08	0.22	21.3			
W17	659.5	K	0.09	0.06	8.7	136	124	0.52
W16	660.4	K	0.15	0.06				
W15	662.3	K	0.13	0.14	-4.9	136	115	0.54
W14	663.7	K	0.10	0.09	17.1			
W13	665.5	K	0.12	0.28	-3.2			
W68	666.6	K	0.14	0.17				
W12	667.2	K	0.10	1.21	25.0			
W11	668.5	K	0.18	0.41	6.6	160	108	0.60
W10	669.5	K	0.15	1.23	9.9			
W9	670	K	0.29	0.71				
W67	671.5	K	0.10	0.09				
W66	673	K	0.13	0.11	-3.9	147	110.2	0.57
W65	674.5	K	0.08	0.02	22.0			
D115	207	K	0.94	1.25				
D140	208	K	1.03	1.47	12.9			
D114	209	K	1.08	3.72				
D113	210	K	1.34	1.26				
D112	211	K	0.69	0.91	4.5			
D111	212	K	1.45	0.72				
D110	213	K	1.28	1.47				
D109	214	K	0.66	1.18	7.0	141	43.8	0.76
D108	215	K	0.81	1.17				
D107	216	K	0.69	1.24				
D106	217	K	0.66	1.72	6.6	139	58.3	0.70
D105	218.5	K	0.63	1.45				
D104	219.3	K	0.72	1.10	2.7			
D103	220.4	K	0.73	0.95				

D102	221	K	0.73	1.34	6.1	120	48.3	0.71
D101	222	K	0.67	0.95				
D100	223	K	0.62	1.11				
D139	224	K	0.67	1.06	4.9			
D99	225	K	0.66	1.06				
D98	226	K	0.56	1.91	6.8	126	39.7	0.76
D97	227.3	K	0.61	1.16	2.8			
D96	228.3	K	0.69	2.85				
D95	230	K	0.69	1.15	3.8			
D94	231	K	0.70	0.89				
D93	232.4	K	0.73	1.44	6.7	114	50.3	0.69
D138	233.3	K	0.76	1.50				
D92	234.2	K	0.14	1.03				
D91	235	S	0.15	0.95	15.9	170	45.8	0.79
D90	236.3	S	0.06	1.13				
D89	237.4	S	0.08	1.00	24.4	39	70.8	0.35
D88	238	S	0.11	1.93				
D87	239.5	S	0.16	0.74				
D86	240	S	0.20	1.89	14.5	106	93.0	0.53
D85	241	S	0.31	1.48				
D84	242	S	0.53	1.22				
D137	243.5	S	0.61	1.69	1.3	126	63.2	0.67
D136	245	S	0.27	0.81				
D83	246.3	S	0.28	1.44	9.8	102	51.6	0.66
D135	247.2	S	0.37	2.12				
D82	248	S	0.25	0.60	11.9			
D81	249	S	0.26	0.99				
D80	250	S	0.28	1.04	14.5	89	65.6	0.57
D79	251	S	0.30	0.95				
D78	252	S	0.28	1.28				
D77	253.8	S	0.34	0.77	5.2			
D76	254	S	0.26	1.10				
D75	255	S	0.33	1.12	1.7			
D74	256	S	0.40	2.50	10.5	100	59.2	0.63
D73	257.8	S	0.31	1.24				
D72	258	S	0.26	0.96	-1.7	61	51.5	0.54
D71	259	S	0.37	1.21				
D70	260	S	0.25	0.96	6.2			
D69	261	S	0.28	1.03				
D68	262	S	0.35	1.81				
D67	263	S	0.36	2.24	2.3	96	71.0	0.58

D134	264	S	0.46	1.17				
D66	265	S	0.43	1.32	2.0			
D65	266	S	0.39	2.47				
D64	267.2	S	0.45	2.73	3.4	88	45.5	0.66
D63	268	S	0.52	2.16				
D62	269.5	S	0.45	1.92	3.1			
D61	270	S	0.48	1.73				
D60	271.7	S	0.41	2.03	5.6	83	57.6	0.59
D59	272.4	S	0.60	1.71				
D58	273	S	0.62	1.56				
D133	274	S	0.41	2.26	1.4	98	59.3	0.62
D57	275	S	0.47	2.06				
D56	276	S	0.42	1.37				
D55	277.5	S	0.52	2.31	4.4	86	49.5	0.64
D54	278	S	0.51	1.25				
D132	279.2	S	0.46	3.07	0.2	93	45.1	0.67
D53	280.4	S	0.17	0.39				
D52	281	S	0.66	2.15				
D51	282	S	0.57	3.39	-2.3	105	49.9	0.68
D50	283	S	0.38	2.39				
D131	284	S	0.43	1.68				
D49	285	S	0.52	2.08	-0.1			
D48	286	S	0.49	2.28	-0.9			
D47	287.2	S	0.51	2.73				
D46	288.1	S	0.58	3.67	-1.9	98	51.4	0.66
D45	289.7	S	0.45	6.52	-3.3	76	64.1	0.54
D44	290	S	0.57	2.39				
D43	291	S	0.32	2.14				
D42	292.4	S	0.58	1.99				
D41	293.3	S	0.56	1.87	5.1	63	31.6	0.66
D40	294	S	0.33	1.76				
D130	295	S	0.38	1.59	3.2			
D39	296	S	0.56	2.48				
D38	297.7	S	0.37	1.19				
D37	298	S	1.04	5.95	4.7	73	52.3	0.58
D36	299.7	S	0.81	2.59				
D35	300	S	0.66	2.55				
D34	301	S	1.19	3.40	10.0	71	50.2	0.59
D33	302	S	0.59	2.45				
D32	303.3	S	0.54	2.93				
D31	304.3	S	0.48	3.25	11.8	84	47.4	0.64

D30	305.4	S	0.56	2.84				
D29	306.4	S	0.83	2.51				
D28	307.4	S	0.61	3.04	13.1	89	50.5	0.64
D27	308.4	S	0.80	1.40				
D129	309.5	S	0.56	4.22	11.1	67	45.4	0.60
D26	310.5	S	0.59	3.07				
D25	311.5	S	0.59	3.15	13.2	87	48.6	0.64
D24	312.5	S	0.71	1.70				
D23	313.4	S	0.55	2.26	10.6	87	51.9	0.63
D22	314.5	S	0.60	1.83				
D21	315.5	S	0.62	3.74	4.1	105	72.2	0.59
D20	316.4	S	0.75	1.28				
D128	317.3	S	0.69	2.35	11.3	78	45.6	0.63
D19	318.1	S	0.55	1.88				
D18	319	S	0.65	1.33				
D17	320.3	S	0.48	1.00	-7.8	207	203	0.50
D16	321.6	S	0.38	1.62				
D15	322.5	S	0.55	2.67	18.1	103	67.9	0.60
D14	323.2	S	0.47	0.60				
D13	324	S	0.51	1.49				
D12	325.5	S	0.44	0.51	-10.1	170	92.2	0.65
D127	326.4	S	0.50	1.49	-9.7	211	176.5	0.54
D11	327.3	S	0.48	0.17				
D10	328.6	S	0.52	1.93				
D9	329.6	S	0.38	1.97	9.3	139	80.2	0.63
D126	330.4	LD	0.48	3.59	0.2	118	74.3	0.61
D8	331.2	LD	0.44	0.48				
D7	332.5	LD	0.48	2.08	9.5	97	46.9	0.67
D6	333.6	LD	0.68	1.24				
D5	334.7	LD	0.55	0.47				
D4	335.5	LD	0.55	1.96	3.4	112	63.0	0.64
D3	336.4	LD	0.58	1.08	-1.2			
D2	338.5	LD	0.18	0.59				
D1	339.6	LD	0.45	0.29	2.9			

Stage (I) – Stage (International); K-Katian; S-Sandbian; LD-Latest Darriwilian

Figure DR1

