マイクロフォーカスX線CTの紹介と海底資源研究への応用
Introduction of microfocus X-ray CT scanner and application of marine resources

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マイクロフォーカスX線CT（Zeiss社製）が、高知大学海洋コア総合研究センターに新たに導入された。マイクロフォーカスX線CTは、対象物の内部構造をマイクロ~ナノレベルで非破壊観察が可能であり、地質試料にも応用されている。本発表では、海底資源であるマンガンクラストやマンガンノジュールの内部構造の観察例や他の分析機器と組み合わせた、今後の応用展開についても紹介する。

キーワード：マイクロフォーカスX線CT、海底資源、マンガンノジュール
Keywords: microfocus X-ray CT, marine resources, manganese nodule

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Comparative analysis of microbial communities on hydrogenetic ferromanganese crusts in the northwest Pacific

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Ferromanganese crusts are commonly found on outcrops of slopes of seamounts in the Pacific, and contain economically valuable elements, such as Co, Ni, Cu, Pt and REE, therefore the crusts are of great interest in deep-sea mining. Microorganisms are thought to contribute to formation of the crusts and play a significant role in accumulation of the elements. Actually, the presence of abundant and diverse microorganisms on a ferromanganese crust collected at 3000 m water depth in the Takuyo-Daigo Seamount has been reported (Nitahara et al., 2011). However, our knowledge of commonality and difference in the abundance, diversity and distribution of microorganisms of the ferromanganese crusts is still limited. To assess the commonality and difference, in the present study, we collected ferromanganese crusts from three regions (Takuyo-Daigo Seamount, Ryusei Seamount, and Daito Ridge) at several water depths (1200 m to 2200 m) during the cruises NT09-02, KY11-02 and NT12-25, and analyzed microbial communities of the crusts. In addition, we collected and analyzed surrounding sediments and bottom seawater as references to assess the uniqueness of the crust communities. Cell densities estimated by quantitative PCR were significantly higher in the crusts than the seawater, but comparable to or lower than the sediments. Both of bacterial and archaeal 16S rRNA genes were detected in all samples. Phylogenetic diversities were higher in the crusts than the seawater, but comparable to or slightly lower than the sediments. Comparative analyses of the community compositions showed 1) the presence of unique microorganisms to the crusts, which were not detected in the sediments and seawater, and 2) the presence of common microorganisms among the crusts at every region and almost every depth, which are likely key members for ecosystem functioning on the crusts. Based on the results, microbial contribution to the formation of the crusts will be discussed.

Keywords: Co-rich ferromanganese crust, Microbial community, Northwest Pacific
Manganese nodules ubiquitously occur in the abyssal plain, with varying concentrations of metals, including manganese, iron, nickel, cobalt, and copper. Typical manganese nodules observed in pelagic sediments are in centimeter to sub-millimeter size of spherical crusts and often concreted to the pavement over the seafloor. During the Integrated Ocean Drilling Program (IODP) Expedition 329 in 2010, we drilled the entire sedimentary sequence at 6 sites in the ultra-oligotrophic region of the South Pacific Gyre (SPG), where dissolved O$_2$ and aerobic microbial communities are present from the seafloor to the sediment-basement interface [1]. Massive manganese nodules often occur at the seafloor of these drilling sites, and the subseafloor sediments generally consist of zeolitic metalliferous clay. Using a newly developed sediment observation technique [2], we observed numerous micrometer-scale particles of (ferro-)manganese minerals in the oxic zeolitic clay from present-day to several tens of millions of years. Using a synchrotron-based X-ray microtomography and FIB-SEM-EDS, high-resolution three-dimensional micro-textures of manganese microparticles, as well as elemental compositions, were visualized, suggesting that those particles were deposited from the water column, and well preserved in the widespread area of deep-sea oxic sediments over geologic time.


Distributions of Rare Earth Elements between Seawater and Fe-Mn Crust Surface at the Tokyo Daigo Seamount, Northwestern Pacific

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Since the Late Paleocene, ferromanganese crusts (Fe-Mn crusts) have been deposited extensively on the surface of seamounts or plateaus at 400 to 6000 meters below sea level (mbsl) with a thickness range of 1 to 20 cm. Owing to the exceedingly slow growth rates (1 to 10 mm/Myr) and the nm-scale constituents of Fe and Mn oxides/oxyhydroxides, rare earth elements (REEs) are highly concentrated in Fe-Mn crusts from ambient seawater. Over the past two decades, the speciation across the solid-water interface has been intensively studied for many REEs. In terms of the distributions of REEs between seawater and Fe-Mn crusts, apparent distribution coefficients of REEs have been estimated between reference seawater and Fe-Mn crusts with variable locations, depths and ages. Although this compilation provides fundamental insights into the selective enrichment pattern of REEs, it might be deviated from the intrinsic values by taking the heterogeneity of vertical profiles of REE concentrations in seawater adjacent to Fe-Mn crusts and the progressive transformation from metastable Fe and Mn oxides/oxyhydroxides to their stable states in Fe-Mn crusts. For accurate estimation of the apparent distribution coefficients, it is necessary to clarify equilibrium solute and sorbate concentrations in seawater and Fe-Mn crusts, because in situ physicochemical conditions represented by high hydraulic pressure, partial pressures of CO₂ and O₂, and constant fluxes of low solute concentrations are difficult to be reproduced experimentally. In addition, the complex nm-scale mineral assemblages of Fe-Mn crusts are impossible to be synthesized. As for sorbate concentrations in Fe-Mn crusts, it is critical to sample from the surface layer in Fe-Mn crusts, which is equilibrated with ambient seawater. To collect ambient seawater adjacent to the outcrop of Fe-Mn crusts, seawater sampling by a submersible is of technical advance in comparison to the deployment of a water sampler such as the rosette from a research vessel. Furthermore, seawater sampling needs to be optimized by developing a sampling device to minimize the levels of contamination and loss of trace elements. In this study, it is aimed to obtain the apparent distribution coefficients of REEs between seawater and Fe-Mn crusts by using vertical profiles of REE concentrations of the surface layer of Fe-Mn crusts and the corresponding ambient seawater from one of the largest deposits of Fe-Mn crusts in the northwestern Pacific. As for Ce, anomalously low concentrations in ambient seawater and high apparent coefficients were evident at depths. We will discuss the potential causes of the Ce anomaly as well as the distribution patterns of the other REEs.
Pair of Zirconium (Zr) and Hafnium (Hf) is called “Geochemical twins” due to the consistencies of their ionic valences and radii. As a consequence, this elemental pair shows same behaviour and uniform Zr/Hf ratio, which is theoretically same as that of chondrite meteorite, in the environment. However, the significant fractionation between these elements in natural samples has been found, and it has been proposed as enigma of Zr-Hf fractionation (Niu et al. 2012). The large fractionation of these elemental couple has also been found in the ferromanganese crust (FMC) (Bau 1996). In this study, we attempted to clarify the accumulation mechanism of Zr and Hf in FMCs with some methods including X-ray absorption fine structure (XAFS) technique for synthesised and natural samples.

Six FMC samples were collected from the Takuyo-Daigo and Ryusei seamounts, from 950 m to 3000 m water depth, with hyper-dolphin (remotely operated vehicle) equipped with live video camera and manipulators. Near surface layers (less than 1 mm) of all FMCs were analysed with XRD, and measured elemental concentrations by ICP-MS after the total decomposition of samples. Sequential extraction was conducted by following Koschinsky et al. (1995) to confirm the existence phase of Zr and Hf in FMC. XAFS analysis was also conducted to confirm the mineral composition and speciation of Zr together with chemical reagents, Zr minerals and rock samples as standard materials. Furthermore, distribution coefficients (Kd) and chemical states were determined through the adsorption experiments of Zr and Hf DFO complexes with ferrihydrite and d-MnO$_2$.

The major mineral composition of Fe and Mn had no significant variation with the water depth of these seamounts. The concentrations of Zr and Hf were increased with depth, and their ratios were varied without showing any trends. However, these ratios were totally fractionated from those of the seawater (Firdaus et al., 2011), and Hf was more enriched in all FMCs than Zr. The observed Zr chemical states in FMCs were 1) coprecipitation with ferrihydrite, 2) coprecipitation with d-MnO$_2$, and 3) basalt-like composition, although the results of sequential extraction showed that Zr and Hf dominantly exist in Fe fraction. From the results of adsorption experiments of Zr and Hf with ferrihydrite and d-MnO$_2$, it was found that the Hf-DFO was more adsorbed compared with Zr-DFO. In this case, the observed bond length of Hf-O was significantly shorter than that of Zr-O in the synthesised minerals.
東北日本弧新第三系に産する層状マンガン鉱床についての最近の研究
A Review: Recent Studies of Neogene Strata-bound Manganese Oxide Deposits From the Northeastern Japan Arc

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In 1950s and 1960s, Neogene strata-bound manganese oxide deposits distributed in the Northeastern Japan Arc had studied vigorously in the viewpoint of the mineral exploration (e.g. Yoshimura. 1952, 1967). Based on the mineralogy, the major elements chemistry and the geological setting, it has been concluded that these deposits formed by submarine hydrothermal activities. Miura et al. (1992) carried out the geochemical studies including trace elements and rare earth elements (REE) compositions for the Neogene strata-bound manganese oxide deposits from southwestern Hokkaido and also concluded them to having hydrothermal origin. On the other hand, they also pointed out that these deposits showed the positive Ce anomalies, which are different characters from the hydrothermal manganese deposits occurred around the modern spreading centers. Recently, Sakai et al. (in prep.) showed radiolarian stratigraphy in the Fukaura district, one of the main occurrences of the Neogene strata-bound manganese oxide deposit in the Northeastern Japan Arc, and found the long term (ca. 6 to 7 m.y.) hiatus just above the manganese deposit horizon. The long-term exposure of the manganese oxide deposit to oxic seawater may be the cause of the REE pattern showing the positive Ce anomaly.

キーワード: マンガン鉱床、東北日本弧、放散虫
Keywords: Manganese deposits, Northeastern Japan Arc, Radiolarians
AUV「うらしま」で得られた拓洋第5海山南西尾根の微地形

Microtopographies around southwestern ridge of Takuyo-daigo seamount obtained by AUV "Urashima"

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It has been aware of that existence of ferromanganese crusts are closely concerned to microtopographies of seamounts. However, there are insurmountable geographical "scale gap" between bathymetry obtained by ship's multi-beam echo sounder (MBES, ~100m grid data) and observation records of Remotely Operated Vehicle (ROV, description of few meter-scale outcrop). A research cruise YK15-15, Strategic Innovation Program (SIP), New-generation Technology for Ocean Resources Survey (ZIPANG in ocean), "Research on formation process and mechanism of cobalt-rich ferromanganese crusts covering over seamounts in the northwestern Pacific - precise bathymetric survey and seawater sampling by AUV -", was carried out in 2015, to obtain detailed seafloor topography and side scan sonar (SSS) images using Autonomous Underwater Vehicle (AUV) "Urashima", for realizing a relationship between distribution of the crusts and microtopographies around southwestern ridge of Takuyo-daigo seamount, where we researched as a typical field of ferromanganese crusts since 2009. Here we report some of the results about detailed topographies and SSS images obtained by AUV "Urashima", comparing with dive logs and photographs obtained by past ROV dives. This project has been implemented as part of "Scientific Research on Genesis of Marine Resources" for “Next-generation Technology for Ocean Resources Exploration,” an initiative that is part of the “Cross-ministerial Strategic Innovation Promotion Program (SIP)” by the Japanese government.

キーワード：鉄マンガンクラスト、拓洋第5海山、マルチビーム、AUV、うらしま

Keywords: ferromanganese crust, Takuyo-daigo seamount, MBES, AUV, Urashima
北西太平洋から得られた鉄マンガンクラストの超微細磁気層序から推定した成長速度
Sub-millimeter scale magnetostratigraphy of ferromanganese crust from north western Pacific: High fidelity estimate of growth rate

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鉄マンガンクラストには数千万年にわたる海洋環境の記録が残されており、正確な形成年代を決定することで過去の地球環境変動の復元が可能になるのではないかと期待されている。近年注目されている年代決定の手法として、古地磁気学的手法を用いたものがあり、いくつかの先行研究がなされている。そのなかでも最も新しい研究であるOda et al. (2011)では超伝導量子干渉素子(SQUID)を利用した走査型SQUID顕微鏡(Scanning SQUID Microscope : SSM)を用い、85 μmという高分解能での磁気測定の結果を得て、その結果を古地磁気極性年代表とうまく対比することに成功した。対応に基づき推定された成長速度は5.1±0.2 ㎜/Maであり、これは18Be/16Be法で推定された6.0±0.2 ㎜/Maと整合的であった。

本研究では、南鳥島南西方約15Kmに位置する拓洋第5海山(22°41.04’N, 153°14.63’E, 2239 m )より採取された鉄マンガンクラストを対象とし、SSMを用いた磁気測定による形成年代および、成長速度の推定を目的とした。

具体的な方法については、採取された試料から成長方向に垂直に切り出し作成した一辺19 mm、厚さ30-50 μ mの薄片を測定試料とし、自然残留磁化の段階交流消磁(0-80 mT)および、SSMによる磁気測定を行った。測定された磁気データには外部磁場などのノイズが含まれていたため、補正を行い試料の磁気データのみを取り出した。補正後の磁気データからイメージ図を作成し、薄片試料写真と重ねることで磁気イメージ図の最表層部分を決定した。結果として試料の最表層(8mm)から最下層(19mm)では9回の地磁気逆転が確認できた。地磁気逆転がみられる深度と古地磁気極性年代表を対比し年代および、成長速度を推定した。推定した成長速度は3.1±0.1 ㎜/Maとなり、これは18Be/16Be法で推定された成長速度3.5±0.3 ㎝/Maと比べ整合的な値となった。

キーワード：SQUID顕微鏡、鉄マンガンクラスト、成長速度、残留磁化
Keywords: scanning SQUID microscope, ferromanganese crust, growth rate, remanent magnetization
Regional and temporal variability in deposition of marine mangense minerals: A cooperative study in the Northwestern Pacific seamounts.

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We report the findings of geological and geochemical analyses of the marine manganese deposits collected during the cruises of the last years and our interpretation on parameters controlling the grade and abundance of the deposits over the areas and depths. We have found small- and regional-scale occurrence and compositions in relation to oceanographic and geological environments. We used ROVs, submersibles and surface ships, jointly with JAMSTEC, JOGMEC, GSJ and other organizations. The program is planned for next two-three years.

Keywords: nodule, ferromanganese, pacific

キーワード：nodule, ferromanganese, pacific

Keywords: crust, manganese, northwestern
Stratiform manganese deposits have been known to occur in the Triassic to Jurassic bedded cherts sequence of the Chichibu Belt in Southwest Japan, which are considered to have accumulated in a mid-oceanic basin of the Panthalassa Ocean. To constrain the stratigraphic age and depositional setting of these manganese deposits, we describe the field occurrence, stratigraphy, and radiolarian age of the chert-hosted manganese deposits from the Triassic to Jurassic bedded chert succession of the Chichibu Belt, defined as a Jurassic subduction-generated accretionary complex in Southwest Japan. The Triassic to Jurassic bedded cherts in the Chichibu Belt are considered to be deep-sea sediments that accumulated in an open-ocean realm of the Panthalassa Ocean. Our biostratigraphic analysis of radiolarians reveals that the stratiform manganese deposits intercalated in the bedded cherts were deposited in the Late Triassic and Early Jurassic. Upper Triassic manganese deposit occurs associated with the massive cherts which appear to have been formed by hydrothermal activity. The red bedded chert above the manganese deposit yields radiolarian fossils, including *Trialatus longicornutus* and *Trialatus megacornutus*. These radiolarians indicate that age of manganese deposits can be correlated with the late Carnian age. Lower Jurassic manganese deposit occurs intercalated within the gray to dark gray bedded cherts. Detailed biostratigraphic analysis of radiolarians reveals that manganese deposit is embedded in the upper Pliensbachian to Toarcian (*Mesosaturnalis hexagonus* Zone - *Parahsuum* (?) *grande* Zone). Chemical compositions of Upper Triassic deposits are characterized by enrichments in Mn and depletion of Co, Ni and Zn. These geochemical features are similar to those of modern submarine hydrothermal manganese deposits from hydrothermal activity. In contrast, early Jurassic manganese deposits were triggered by an influx of warm, saline and oxic water into a stagnant deep ocean floor basin. It is likely that the deposits are considered to have formed by oceanic anoxic event.

Keywords: Stratiform manganese deposits, Chichibu belt, radiolarian, Late Triassic, Early Jurassic
We described the litho- and biostratigraphy of the Neogene strata distributed in the Fukaura area, western Tsugaru Peninsula. The Neogene sequence in this area is subdivided into the Odose, Tanosawa, and Akaisi Formations in ascending order. The Odose Formation is composed mainly of andesite lava and rhyolite. The Tanosawa Formation consists of acid pumice tuff, conglomerate, and sandstone with Mn nodule beds. The Akaishi Formation is characterized by acid pumice tuff, conglomerate, silt and sandstones.

Radiolarian biostratigraphy was studied for the Tanosawa and Akaisi Formations. The Tanosawa Formation was assigned to the upper part of the Eucyrtidium inflatum Zone (13.1 to 11.7 Ma) during the middle Miocene within the framework of North Pacific radiolarian zonation. The Akaishi Formation yield radiolarian assemblages that is indicative from the Larcopyle pylomaticus Zone to the lower part of the Cycladophora sakaii Zone (5.2 to 2.7 Ma) during the Pliocene. The radiolarian assemblages of the studied section contained cosmopolitan or mid-to-high latitude species such as Axoprunum acquilonium, Calocyclas motoyamai, Cycladophora sakaii, Hexacontium parviakitaensis, L. pylomaticus and Thecosphaera pseudojaponica, and lack such as low latitude species as Calocyclas costata, Diartus hughesi, Didymocystis penultima, Dorcadospyris alata, Phormostichoartus doliolum, Phormostichoartus fistula, Pterocanium audax and Stichocorys wolffii. Since these characters resemble to those of radiolarian assemblages recognized from the North Pacific Ocean Drilling Program Sites 884 and 887, the Fukaura area was under boreal cool water environments during that interval.

Based on the radiolarian biostratigraphy, this work newly placed the boundary between the Tanosawa and Akaisi Formations of the studied section on the top of a gravelly sand stone bed with Mn nodule. This boundary horizon was lying 5 m below the previously indicated one in the same section and characterized by the absence from the Lychnocanoma magnacornuta to A. acquilonium Zone (11.7 to 5.2 Ma). This result indicated a time gap of 6.5 million years between the two formations. This unconformity was correlated to the widespread unconformity recognized in the northeastern Japan.
Group 6 elements, chromium (Cr), molybdenum (Mo), and tungsten (W), are important trace elements for geochemistry since they are redox sensitive and have several stable isotopes. In particular, Mo has drawn attention in paleoceanography because its geochemical behavior sharply changes depending on the redox conditions (Anbar, 2004). A fractionation for Mo was observed during the adsorption to manganese oxides, i.e. $\delta^{97/95}\text{Mo} = -1.8\%$ (Wasylenk et al., 2011). On the other hand, Cr did not show an isotope fractionation during the adsorption to Fe-(oxyhydr)oxides (Ellis et al., 2002).

Kashiwabara et al. (2011) suggested that the Mo isotope fractionation at water/metal oxides interface is caused by symmetrical change of molybdate from tetrahedral (Td) to octahedral (Oh) structure during adsorption by XAFS analysis. However, this fact was not confirmed well. In this study, we tried to understand the isotope fractionation of group 6 elements accompanying with adsorption on Fe/Mn-(oxyhydr)oxides by means of density functional theory (DFT) calculations and information of adsorption structure obtained by XAFS.

Adsorption structures of Cr, Mo, and W oxoanions were analyzed by XAFS for ferrihydrite, goethite, hematite, and δ-MnO$_2$ (Kashiwabara et al., 2011 and 2013). Isotope fractionations were estimated using the equation based on mass-dependent isotopic differences in vibrational energies (Bigeleisen and Mayer, 1947) with DFT-calculated vibrational frequencies. The several oxoanion structures of hydrated and adsorbed on mineral surface models were examined for DFT calculations.

The results of XAFS analysis suggested that the adsorption structures for chromate and tungstate have $Td$ and $Oh$ symmetries, respectively, and molybdate has both $Td$ and $Oh$ symmetrical adsorption structures with various ratios depending on the minerals. From the DFT calculations, the large isotope fractionation was confirmed by the structural change of oxoanions from $Td$ to $Oh$ symmetry. The evaluated isotope fractionations for each mineral by DFT calculations combined with structural information by XAFS analysis agree with experimental results well. This fact indicates that the isotope fractionations of group 6 elements through the adsorption on minerals are caused by structural change of oxoanions.

References:
Keywords: isotope fractionation, group 6 element, Fe/Mn-(oxyhydr)oxide, adsorption structure
テルル標準試薬中のテルル安定同位体組成
Tellurium Stable Isotope Compositions in Tellurium Standard Regents

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鉄マンガンクラスト等の海底に存在する鉄マンガン酸化物は地殻に対して一万倍以上ものテルル（Te）が濃集しており（Hein et al., 2010）、同じく高濃度で濃集しているコバルト、白金等も含めた将来の鉱物資源として注目されている。地球化学的にも生成時の海洋環境の情報を保持した物質として重要である。近年、Teについて鉄マンガン酸化物に対する吸着構造が明らかにされている（Kashiwabara et al., 2014）。我々のグループでは、鉄マンガンクラスト等の成因について、微量元素の吸着構造やTe安定同位体組成の挙動を明らかにすべく分析手法の開発を行ってきた。このうち、Te安定同位体分析の研究例は、近年になって、ようやく、陸上鉱床由来の自然テルル等の報告例（Fornadel et al., 2014）や、隕石中のTe安定同位体組成の報告（Fehr et al., 2014）が行われたのみである。これらの研究においては、おのおのの実験室のin-house standard溶液をTe安定同位体分析のスタンダードとして使用している。そのため、異なる実験室同士の比較を行うための標準となるTe同位体標準試料は現在のところ存在しない。さらに、異なる製造元の試薬間や同一製造元から供給される別バッチの試薬間のTe同位体組成が均一であるかどうかの報告は未だなされていないのが現状である。本研究では、複数の試薬中のTe同位体組成を明らかにするため、ダブルスパイクを用いたTe安定同位体分析手法を開発し、分析を行った。

Te安定同位体分析にはマルチコレクター型誘導結合プラズマ質量分析計（MC-ICPMS）を使用した。装置はJAMSTEC所有のNEPTUNE（Thermo Scientific社製）を用い、試料導入系として脱溶媒試料導入装置（Aridus II、CETAC社製）を使用した。分析装置内における質量差別効果の補正には、^{125}Te–^{128}Teスパイクによるダブルスパイク法を用いた。In-house standardとしてTe試薬（関東化学）を用いた実験の結果、1測定あたり11ngのTeを使用した分析の繰り返し再現性は、^{130}Te/^{125}Te比について0.2‰ (n =25:2SD)が得られた。比較測定試料として関東化学(#40856-1B)、Alfa Aesar(#44632)、Sigma-Aldrich(#92027)の3社の元素標準試薬と、Alfa Aesarから供給されているTe lump（99.999%, #10758）を用い、本研究で開発したTe同位体分析法を適用した。Sigma-Aldrich社の試薬は誤差の範囲で関東化学社の試薬と一致した。Alfa Aesar社の試薬は、関東化学社の試薬から1.2‰高い値を示した。Alfa Aesar社のTe lump中のTe同位体組成は、Alfa Aesar社の試薬の値と誤差の範囲内で一致した。この結果から、試薬ごとにその原材料の安定同位体組成の差、もしくは製造過程における同位体分別を反映して異なる同位体組成を持つ事が明らかになった。このため、実験室ごとに異なる試薬をin-house standardとして使用した場合、Te同位体組成についての報告値に約1‰程度の偏差が存在することが明らかとなった。以上より、Te安定同位体を広く地球科学に応用するには、Te標準溶液を設定し、研究室間のキャリブレーションを行うことが必要である。

キーワード：テルル、安定同位体、鉄マンガンクラスト
Keywords: Tellurium, Stable Isotope, Fe-Mn Crusts