Report of the Chemistry Working Group, SciMP 23-25 June 2004

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Executive Summary

The Chemistry Working Group (CWG) of SciMP produced this report regarding IODP chemistry laboratories and analytical facilities after soliciting input from the ocean drilling community. In addition to this input, the report also contains the results of discussions between CWG members and other leading geochemists. There are two overall conclusions that SPC and the IOs need to pay particular attention to:

1) Better standardization/calibration should be employed for IODP than was available for ODP;

2) Technician training should be at a higher level than during ODP to maintain the equipment while on-site and also to ensure the data generated is of the highest quality.

The context of the above two unifying points is that the technology has evolved considerably, and the sophistication of the field of geochemistry has been significantly advanced, to the point that the needs of the community have surpassed the abilities of the "ODP-model". The report contains 11 Recommendations and 6 Action Items.

The Chemistry Working Group (CWG) of SciMP has solicited input from the ocean drilling community regarding analytical facilities associated with IODP. This report summarizes the responses to a questionnaire, as described below, as well as discussions within the SciMP and other leading geochemists.

A survey containing 11 questions (Attachment 1) was e-mailed to a list generated by ODP-TAMU for many (not all) co-chiefs, petrologists and geochemists that have sailed on ODP legs since Leg 163, as well as the participants of the "Future Opportunities in Geochemistry for IODP" workshop. In addition, the questionnaire was published in the Geochemical Society's January 2004 issue of "Geochemical News" in order to explore another avenue for community input.

A total of 33 responses were received from a broad international cross section of geochemists and petrologists. Unfortunately over 70 of the e-mail requests were undeliverable because the addresses were no longer active. The breakdown with regard to nationality of the respondents is in Table 1. E-mail was by far the most effective means of soliciting input as only one response was returned from the printed questionnaire in Geochemical News.

Table 1: Nationality of the respondents to the CWG questionnaire.

Country	**USA**	**UK**	**Canada**	**Italy**	**France**	**Japan**	**Netherlands**	**Australia**
	21	4	2	2	1	1	1	1
Total: 33								

Collectively, putting together the responses from the surveys, as well as the SciMP discussions and direct feedback, the overall conclusions of the CWG are:

1) Better standardization/calibration should be employed for IODP than was available for ODP;

2) Technician training should be at a higher level than during ODP to maintain the equipment while on-site and also to ensure the data generated is of the highest quality.

The context of the above two unifying points is that the technology has evolved considerably, and the sophistication of the field of geochemistry has been significantly advanced, to the point that the needs of the community have surpassed the abilities of the "ODP-model".

1. WHAT TYPES OF ANALYSES ARE NECESSARY/RECOMMENDED?

The recommended analytical types are divided into 5 main categories, as described below. Parts 1, 2, and 3, are considered "minimum measurements". Parts 4 and 5 will commonly be dependent on the scientific mission of the expedition and the needs of the Scientific Party.

1) <u>Safety Monitoring</u>.

    Headspace measurement of:

    - Lower molecular weight hydrocarbon gases (methane, ethane, propane, etc.).

- Inorganic gasses (CO_2 and H_2S).
    - i. Measurement by GC-FID, GC-TCD and/or NGA.

  These are probably required for all soft-rock (sediment) expeditions, regardless of platform.

2) Ephemeral components.
    - Measure pH (& Alkalinity), silica, NH_4, NO_3 and PO_4 in pore water samples as soon as possible.
        - i. Measurement with pH sensor, auto titration, colorimeter.
        - ii. Allow for routine sampling for stable isotope determinations in pore waters and headspace gases.
        - iii. For inorganic metal analysis, acidify with ultrapure HNO_3 to ~5% as soon as possible to allow for post-cruise data acquisition.
3) Essential components to decide drilling strategy during a given expedition.
    - Measure major dissolved anions (Cl and SO_4) and all major dissolved cations in pore-water samples (ion chromatograph).
    - Elemental analysis of total carbon, total nitrogen, total sulfur, and inorganic carbon to provide composition of the organic matter in sediments. (CHNS analyzer, Coulometer).
    - Whole-rock major and trace element compositions (ICP-OES, ICP-MS).
    - Laser ablation ICP-MS of glasses for elemental ratio data.
4) Rock/sediment-Specific analyses: these depend upon the type of rock being recovered. For example, sedimentary rocks require characterization through, for example, TOC, carbon, CO_3, major and trace analyses (bulk). For basement rocks, major and trace element compositions are required to characterize the different units recovered.
5) "Shipboard" Measurements to determine the best science overall.
    - This will in all probability be project specific.

2. SAMPLE HANDLING

A variety of samples will be handled and in order that these are not compromised for immediate or future analyses, careful handling/storage procedures need to be followed.

1) Volatile components without air contamination.
    - A headspace sample (one per core, where applicable), taken from the core immediately after retrieval, is placed in a glass vial, sealed with a septum under highly purified N_2 atmosphere.
    - Determine O_2 concentration for the samples using GC-TCD to ensure that air contamination is minimal.
2) Organic matter in sediments.
    - Sediments are freeze-dried and crushed.
    - Powdered samples are weighed for individual future measurements such as biomarker analysis.
3) Gas hydrates.
    - The core section is immediately moved to a freezing room.
    - The hydrate is packed in pressure-resistant containers.
    - One container is filled up by purified N_2 gas and pressurized to 100 atmospheres, while the other is not treated.
    - Keep the containers in a freezer under gas-tight conditions.

- Any pressurized core retrieval system must include the ability to micro-sample pristine pore fluids and for biological material.

4) Pore fluids.
   - Squeeze pore fluid from the sediments immediately after retrieval on deck.
   - The sampled fluid is divided into a clean polypropylene tube for the archive and a clean glass vial that is sealed with a septum.
5) Hard rock and sediments.
   - All personnel to remove jewelry from hands and wrists (precious metal contamination).
   - Composition of drilling mud used must be regularly documented.
   - Unless specified by the science party, do not powder using tungsten carbide - use alumina or clean agate or silicon nitride.
   - Grind away all sawn surfaces on diamond wheel.

> **Recommendation 1**: Sample-handling procedures should be specified for each expedition such that the integrity of the drilled samples are not compromised. This should be discussed and specified during the expedition planning stage between the co-chief scientists and the IO.

3. ANALYTICAL INSTRUMENTATION

The analytical capabilities requested by the survey respondents were wide ranging. The list in Table 2 shows instrumentation in addition to that available during ODP or would require a significant upgrade to that available on the *JOIDES Resolution*. Some requested items were considered to be too sophisticated for on site operations (i.e., electron microprobe).

The requested instrumentation (Table 2) would be used to influence the scientific output during each expedition, influence drilling strategy, and also be used to ensure safety of the drilling staff, technicians, and scientists. Permanent labs can be set up on the riser and non-riser vessels, but would be more problematic for MSP drilling. In the latter case, a modular lab should be considered that could be accordingly equipped from an inventory of analytical equipment for each specific expedition and the goals therein.

> **Action Item 1**: SciMP will work with the IOs to investigate the modular lab concept for MSP operations.
> *Action to be taken by SciMP (Petrophysics WG, Chemistry WG, and Microbiology WG) and IOs.*

Table 2: Requested Instrumentation.

Measurement	**Data Type**	**Sample Type**
DBD and porosity*	Physical Properties	Whole Core
Core temperature change*	Physical Properties	Whole Core
Electrical Conductivity* (Formation factors)	Physical Properties	Sediments
In situ interstitial water sampling	Geochemistry	Sediments
UV Excitation	Organic Geochemistry	High molecular wt. hydrocarbons
O, N, C, H isotopes*	Geochemistry	Gasses, waters, and sediments
C (organic and inorganic) and S (Coulometer* and CHNS analyzer*)	Geochemistry	Sediments and waters
Gas chromatography/GC-MS*	Geochemistry	Waters, gasses, sediments (?)
X-ray diffraction*	Mineralogy	Minerals
Cathodoluminescence	Mineralogy	Minerals
ICP-OES*	Geochemistry	Waters, sediments, hard rocks
ICP-MS*	Geochemistry	Waters, sediments, hard rocks
Redox-sensitive pore waters	Geochemistry	Sediments
X-ray CT Scanner*	Geochemistry	Whole Core
SEM facility	Microscopy	Sediments and hard rocks

* Equipment included in the Chikyu equipment list, March 2003.

4. MICROSCOPY.
Use of microscopy during any drilling expedition is a vital part of the characterization and science that is undertaken. Applications include micropaleontology, smear slides, petrologic thin sections, microbiology, etc. Several of the respondents to the CWG survey requested that the microscopy facilities in IODP be significantly upgraded from ODP; this includes both microscopes and thin section making capabilities. Round-the-clock operation of thin section laboratories is essential for sample throughput, which in turn could influence drilling and, therefore, the scientific return of a given expedition. It is essential that a sufficient number of microscopes (3-5?) are available for each specific use and that technical staff be available to maintain these important instruments. Each microscope should be equipped with both transmitted and reflected light capabilities (with a sufficient number of objective lenses), be able to work up to 1600X total magnification in air (and, as much as possible, oil), as well as have the ability to take digital images. The computer shall be equipped with a properly designed photo-capture, annotation, and filing program, plus a high-quality image-analysis and statistical (e.g., point-counting/modal analyses) package. We note that many of these items are to be on board

the Chikyu and it will therefore increase programmatic consistency - in addition to enabling the best science possible - to have these facilities accessible throughout the entire IODP. Microscopes dedicated for a specific purpose should not be used for anything else.

> **Recommendation 2**: SciMP recommends that there be a sufficient number of microscopes configured for each specific use to achieve the scientific objectives of a given expedition, that they be equipped with both transmitted and reflected light capabilities, be able to work up to 1600X total magnification in air (and, as much as possible, oil), as well as have the ability to take and store digital images.

With better modular scopes Cathodoluminescence could be added, which is especially important for highlighting mineral zonation (e.g., plagioclase) and alteration effects. Also, an Environmental Scanning Electron Microscope (E-SEM) can have a profound effect on drilling strategy. For example, on Leg 193 it would have been extremely useful to have an E-SEM available on board, which would have allowed better decision-making (e.g., how to log core, drilling priorities, etc.). Reliance on XRD and thin sections, of what turned out much finer-grained assemblages than expected, meant delays and inaccuracies (i.e., inefficient drilling).

> **Action Item 2**: The Chemistry Working Group of SciMP will work with the various IOs to explore the possibility of adding Environmental SEM and Cathodoluminescence capabilities to the microscopy facilities on the various platforms and affiliated shore-based laboratories.
> *Action to be taken by SciMP (Chemistry WG) and IOs.*

5. ANALYSIS TYPES

5.1. Safety Analyses. While safety is a site survey/safety panel issue, it is recognized that certain analyses are important, although the quality of such analyses during ODP was questioned. The types of analyses that could be conducted include organic and headspace gas measurements (e.g., H_2S, methane and other hydrocarbons), and in some cases, water analyses (organics). It was also recognized that such analyses would need to be conducted "on-site" as samples would degrade and would lose some information if saved for shore-based studies.

5.2. Organic Analyses. In addition to analyses that address safety issues, organic analyses are vitally important for a number of scientific goals. For example, source rock evaluation (organic richness, organic mater types – gas or oil producing, maturity of organic matter) is crucial for drilling strategy to predict the existence of oil and gas reservoirs in further drilling processes.

- Total organic carbon (TOC) by elemental CHN analyzer;
- Hydrogen Index (H.I.) and Oxygen Index (O.I.) by Rock-Eval pyrolysis;
- T_{max} value by Rock-Eval pyrolysis.
- UV excitation for the monitoring of petroleum-type higher-molecular-weight hydrocarbons.

Biomarker analysis using GC and GC/MS is useful for source rock evaluation as well as good science. It provides more detailed information on source rock properties than Rock-Eval, although it requires a longer analytical time. GC and GC/MS have already been installed in Riser and non-Riser platforms. Supporting instruments, such as a rapid solvent extractor, will also be installed in raiser platform. Although the biomarker analysis should not be an obligatory analysis, it will be a highly recommended analysis at on-site.

The community recognizes that improvements in the determination of organic and inorganic carbon need to be made over that conducted during ODP. It has been suggested that carbonate continue to be measured on a coulometer, yet that C-org be measured on an acidified sample with a CHN (elemental) analyzer, as this would give better reproducibility. Additionally, it is expected that the analysis of volatile metabolic compounds using GC and GC/MS (attached with a headspace sampler?) would be useful for microbiology studies – this issue will be dealt with by the Microbiology WG.

We note that Riser drilling uses circulation mud, which contains powdered lignite and other chemicals. Biodegradable drilling muds may also be used although safe drilling may preclude this option (we need more communication with the drilling community on this issue); these often contain animal fats and/or starch, which are another cause of contamination for organic geochemical analysis. It is important that samples of the drilling mud used be analyzed along with the samples in order to assess the contribution of such muds to the organic content and possibly to inorganic analyses (e.g., Ba) of the samples.

5.3. Inorganic Analyses. The change from X-ray fluorescence (XRF) to Inductively Coupled Optical Emission Spectroscopy (ICP-OES) during ODP for inorganic analyses facilitated a greater diversity of samples that could be analyzed (rocks, sediments, and waters) and sample throughput was increased. However, certain sacrifices were made, especially with regard to quantifying the petrogenetically important trace elements (e.g., Nb). For IODP it is recommended that both ICP-OES and quadrupole ICP Mass Spectrometry (ICP-MS) be used (as is the case for the Chikyu) because the combination of both instruments will allow a full suite of major and trace elements to be quantified for almost all sample types. Each instrument is necessary, as each quantifies many elements that the other cannot. Autosamplers should be used with both the quadrupole ICP-MS and ICP-OES to improve throughput. However, the complexity of these machines requires a greater level of technician training than was available during ODP; the technicians should be able to trouble shoot and fix minor problems while at sea (see Section 8 for specific recommendations regarding IODP technicians). Even with the ICP-OES in the latter stages of ODP, the lack of suitably qualified technicians was a major issue.

A number of procedures need to be put in place in order for the ICP-OES and ICP-MS analytical techniques to work well and consistently so:

- Sample preparation – all rock and sediment samples should be crushed and ground in alumina to avoid trace element contamination from tungsten carbide (the high field strength elements, platinum-group elements, etc.) and agate (e.g., Pb). However, some scientists may prefer W-C, so other powdering methods need to be available.
- Samples of drilling mud should be analyzed. The data generated can be used to assess

the extent of sample contamination by the mud (e.g., Ba).

- Clean laboratory facilities with ultrapure acids should be available.
- Digestion vessel cleaning procedures are required (Teflon bombs are expensive so need to be reused).
- Hydrofluoric (HF) acid capable fume hoods are required with sufficient hot-plate space to process tens of samples at one time, along with facilities to cope with a spill of HF.
- The flux-fusion method of sample preparation for ICP-OES required the addition of a flux to the powdered sample followed by fusion to breakdown the silicate matrices. This may dilute some critical trace elements in the samples below detection. Therefore, whole-rock digestion methods are required, which include the use of HF acid.
- The IODP should examine microwave digestion apparatus, such as marketed by CEM and Milestone, as these can easily digest most sediments as well as MORB. Their use dramatically enhances safety, uniformity, and sample throughput.
- Efficiency in sample preparation is essential to make the ICP-MS an effective on-site analytical tool.

Action Item 3: The CWG will explore the suitability of microwave digestion in the preparation of rock and sediment samples for various geochemical analyses, such as ICP-OES and ICP-MS, as a way of increasing sample throughput, safety, and the uniformity of the preparation technique across different platforms and related shore-based labs.
Action to be taken by the Chemistry WG.

The addition of a **laser ablation** (LA) facility that would interface with the quadrupole ICP-MS has been discussed in some detail. It is evident that the new laser systems (e.g., the New Wave UP-213 nm) are very powerful and relatively simple to operate. Quantitative data may not be possible because major element data, which are used as internal standards, will not be determined while on site. However, as long as the external standardization procedure is robust, diagnostic elemental ratios may be obtained from glass and mineral samples that could be used to influence drilling. These analyses do not require digestion nor is a polished section necessary. Rather, a flat sample surface is needed. Therefore, sample throughput is much quicker than for bulk rock analyses. Furthermore, electron microprobe data can be obtained during shore-based studies and the LA-ICP-MS data gathered on site can then be quantified. Samples that could be analyzed are glasses, minerals, and microfossils (i.e., individual foraminifera).

Recommendation 3: SciMP recommends that a laser ablation facility (with radiation of 213 nm or less) be available on the Riser & non-Riser platforms for interfacing with a quadrupole ICP-MS.

Having both ICP-OES and quadrupole ICP-MS capabilities will require a lot of Argon gas and sufficient storage space is required for gas cylinders. In addition, the sensitivity of LA-ICP-MS is greatly enhanced by bleeding in Helium as the carrier gas from the sample chamber to the torch.

Therefore, He gas cylinders are required to be taken on every expedition.

One caveat to the potential addition of quadrupole ICP-MS to the inventory of IODP analytical facilities is that it must be tested on a moving platform. The ICP-MS requires the plasma to be focused through the tiny orifice of the sample and skimmer cones, through a differential vacuum, and into the quadrupole mass spectrometer. If the plasma oscillates (moves) due to platform movement, the stream of ions generated will vary in intensity giving a highly unstable signal. This will produce data with large errors. This was noted to be an issue with the ICP-OES (Quintin et al., 2002), but prudent operation of the instrument in terms of weather conditions was able to solve the problem.

Quintin L. L., Faul K., Lear C., Graham D., Peng C., and Murray R. W., 2002, Geochemical analysis of bulk marine sediment by ICP-emission spectrometry on-board the JOIDES Resolution. *Proc. of the Ocean Drilling Program, Initial Reports,* **199**, http://www-odp.tamu.edu/publications/199_IR/chap_07/chap_07.htm.

> **Action Item 4**: The Chemistry Working Group of SciMP recommends that the IOs of the various platforms examine the potential problem of an oscillating (moving) plasma when using a quadrupole ICP-MS on a moving platform. SciMP further recommends that the IOs report the results if their investigations to SciMP at the December 2004 meeting. SciMP will be conducting independent investigations of this issue and will also report their findings at the December meeting.
> *Action to be taken by the Chemistry WG and IOs.*

There needs to be better major and trace element analytical facilities for S-rich samples. For ICP-MS, sulfur is a signal suppresser and severely reduces machine sensitivity. The ICP-OES analyses on ODP had problems handling S-rich samples, partly because prior tests of the calibration and sample preparation procedures were not as thorough as planned. This will require specialized procedures to be thoroughly tested and implemented throughout the IODP analytical facilities.

5.4. Analysis of Fluids. Expanding the capabilities of pore water sampling is essential for IODP, especially given the nature of some of the first expeditions planned. In all cases, a thorough documentation of how the samples have been taken and the analytical procedures is essential. These capabilities should include:

4.4.1. Centrifuging - this allows temperature control, which has some advantages (e.g., the best technique for sulfide concentration measurements), but it is not good for DIC and sub-mM methane concentrations. The centrifuge could also be used for phyllosilicate (and other minerals) separation schemes for XRD.

4.4.2. Squeezing - in order to study redox-sensitive elements, squeezing under anaerobic conditions should be available. It is also important to develop a method for extracting fluids from indurated (non-squeezable) sediments.

4.4.3. In situ Sampling - a combination of the old WSTP and the so-called DVTP should receive serious attention as such *in situ* sensors, optrodes, and sampling would be of the utmost importance to any serious pore water program. A big step forward would be the ability to sample high temperature fluids (including gases) with a wire-line sampler.

A lot of on-site data for pore fluids (and gases) are of importance in publications and only on board ship can we get high quality data on these components, prior to storage for other work. However, some samples may require immediate isotopic characterization (see above).

5.5. Stable Isotope Analyses. If samples are taken for safety analyses, important scientific data could be obtained from these *provided* the proper equipment was available and the technicians were properly trained in sampling and analytical techniques. For example, stable isotope compositions (C, H, maybe N, and possibly S although a dedicated machine is required for the latter if a lot of sulfur isotope analyses are required) would give invaluable data from ephemeral samples, especially gas hydrates and other organic-rich targets. Furthermore, stable isotope (C, N, O) analyses of bulk sediments would help with stratigraphic correlation and ensure the recovery of complete sections. In addition, some pore water studies would benefit from on-site stable isotope analytical capabilities. Note that the survey respondents suggested that the "on board" stable isotope analyses be restricted to ephemeral samples and those that may affect drilling strategy.

A number of questions arose from within the CWG on this issue. For example, will the mass spectrometer be configured for the analysis of hydrogen? What is the best balance of needs versus resources (financial and technical)? There is broad variance among the options in terms of "what should be done on board" versus "what would be nice to do on board." Selection and operation of an onboard carbonate device in conjunction with the gas source mass spectrometer may be difficult.

That having been said, we must maximize the utility of any onboard gas-source isotope ratio mass spectrometry (IRMS). To that end, the instrument must include the peripheral devices that are specific to (1) the highest priority onboard analyses (e.g., headspace gases [GC-IRMS] and pore waters [$\delta^{18}O_{H2O}$, δD_{H2O}, $\delta^{13}C_{DIC}$—GasBench II or equivalent]) and (2) other perceived needs (e.g., $\delta^{18}O_{CaCO3}$ and $\delta^{13}C_{CaCO3}$ [carbonate device]; $\delta^{13}C_{TOC}$, $\delta^{34}S_{\text{sulfide or sulfate}}$, $\delta^{15}N_{TOM}$ [on-line Elemental Analyzer (EA) for analysis by continuous flow]; $\delta^{18}O_{sulfate}$ [TC-EA]). Each of these devices will increase the range of expertise necessary for the technician and the routine and non-routine repairs and maintenance. Additionally, many of these procedures, particularly those for non-ephemeral components, will require sophisticated and/or time-consuming sample preparation (e.g., foram picking and sediment extractions).

Action Item 5: The Chemistry Working Group of SciMP recommends that the feasibility of having a gas-source stable isotope mass spectrometer on both the Riser and non-Riser platforms be explored. The function of this mass spectrometer would primarily be to undertake analyses of ephemeral samples such as headspace gases and pore waters. SciMP recognizes that in order for this to work, peripheral on-line devices must be included as dictated by scientific need (e.g., GC and an Elemental Analyzer).
Action to be taken by the Chemistry WG.

6. QA/QC ISSUES.

The CWG is working from the following position:

There is no substitute for data of the highest quality.

By adhering to this premise, it is anticipated that the data obtained on different platforms will be of the highest quality, such that they will be able to influence drilling decisions *and be publishable in scientific journals*. With IODP operating multiple platforms and analytical facilities, data quality is an extremely important aspect that requires careful consideration in order for data generated while on site to be used in scientific publications. Where analytical facilities are duplicated on platforms and in shore-based labs, each should have the same suite of reference materials available.

> **Recommendation 4**: Standards/reference materials for each analytical facility be uniform across the different platform and IODP-affiliated shore-based laboratories.

It is essential that the data generated while on site be of the highest quality such that it can be used in scientific publications. This is especially critical for ephemeral samples where the only data obtained will effectively be during the drilling operation (e.g., pore waters, head space gases, gas hydrate samples, etc.). The survey results showed that the community would use the on-site data *if* it could be shown to be of the highest quality. This can be achieved by regular blank, reference material, and replicate sample analyses, along with a thorough error analysis of all data generated. Replicate sample analyses should be conducted during the same run and at least one sample already analyzed being re-analyzed during a later run. This would demonstrate reproducibility of data and consistency in the sample preparation and analytical procedures. As was highlighted in Section 5.4 for pore waters, documentation of how *all* samples were processed is critical in this process.

All blank, reference material, and sample data (especially duplicate analyses) should be readily available from the data repository. Each datum should include a date and who the analyst was. These data should be regularly scrutinized (see below), problems highlighted, and solutions given. During ODP, routine analysis of Standard Reference Materials (SRMs) that were run as *unknowns* during a normal sample batch was discouragingly rare. It must become routine practice to incorporate a wider array of SRMs (and especially those for porewaters) than was done during the ODP.

> **Recommendation 5**: Routine analysis of reference materials as unknowns during every analytical run must become common practice on all IODP platforms and related shore-based labs.

If there is an occasion to use third party equipment (defined as specialized analytical facilities not in the IODP inventory), its suitability should be demonstrated *prior to the expedition* by reference material and duplicate sample analyses. All sample, reference material, and blank data need to be uploaded to the data repository and be available for scrutiny.

Recommendation 6: If third party analytical equipment is to be used on any IODP platform, its suitability should be demonstrated by the analysis of relevant reference materials *prior* to the start of the expedition.

Blind calibration tests could be conducted by each analytical facility on an unknown sample (commonly a reference material) given to the laboratory manager. Such tests could be given at the beginning of each expedition (prior to core being recovered), once a year, or as problems are seen to be developing from regular blank and reference material analyses. The data for these unknowns would be uploaded to the central data repository and scrutinized by a panel of experts (see below). If these blind calibration tests were given at the beginning of every expedition, any problems would be immediately apparent and attempts at remedying them could be made before samples are acquired. However, the feasibility of this process needs to be studied further.

Action Item 6: The Chemistry Working Group of SciMP will study the issue of "blind calibration tests" and formulate a policy on this matter to be presented at the December 2004 meeting.
Action to be taken by the Chemistry WG.

6.1. Implementation: Deciding on the suite of reference materials that should be available in the IODP analytical labs needs to be achieved as soon as possible. The CWG suggests two courses of action: 1) that CWG be the committee to draft this reference material list; 2) that CWG form the core of a committee supplemented with people of the requisite expertise. In either case, the committee reports directly to SciMP.

Monitoring the QA/QC of platform- and shore-based labs requires an oversight committee that has access to the requisite data (outlined above). This could be done either by the existing working groups of SciMP or by a special committee containing the requisite expertise. Either way, this committee is required as a guarantor of high quality data produced by IODP analytical facilities. Regular status reports of the IODP analytical facilities should be made at each SciMP meeting along with actions taken/proposed by the working group/committee. Coordination should be through the co-chairs of SciMP and the respective IOs. Critical in this endeavor is traceability of all data uploaded to the data repository. Each analysis should include the date of the analysis, sample type, the analyst, platform, etc.

Recommendation 7: SciMP will advise the IOs on the development of analytical and sample preparation protocols, as well as their implementation on the various IODP platforms and in shore-based laboratories. SciMP will also oversee and advise on QA/QC issues (and in the mitigation of problems) as they relate to geochemical analyses.

7. ANALYTICAL ACCURACY

This requires sensitive and specialized analytical equipment and low-blank reagents. Furthermore, accurate weighing of the samples and any added reagents is essential for accurate and precise data. As has been seen on the *JR*, this is difficult on a moving ship, and introduced significant errors into the analyses both directly (through weighing errors) and indirectly (through conducting sample preparations by volume measurements rather than weight). We recommend that a balance be isolated (using a gimble or gyroscope system) for such accurate weighing. For low-blank reagents, these can either be purchased or generated by distillation on-board.

> **Recommendation 8**: The CWG of SciMP recommends that facilities for accurate weighing on a moving ship be made available on the Riser and non-Riser platforms. Such facilities will greatly increase the quality of geochemical data generated on these platforms, enhancing their usability in scientific publications.

8. TECHNICAL SUPPORT.

Technician training and ability is a critical part of obtaining the highest quality data, not only in sample preparation and analysis, but also in maintaining and trouble-shooting problems with individual pieces of machinery. The CWG recommends that all IODP technicians should have at least a Masters degree in analytical chemistry, geochemistry, or related fields. However, this alone will not guarantee that quality data will continue to be produced from each analytical facility over the life of IODP. It is essential that the technicians understand the various sample preparation techniques and be able to adequately judge data quality and the best way to do this is to give the technicians training is an IODP-related research laboratory (e.g., Kochi, Bremen, TAMU) or visiting university laboratories for 2-4 weeks. We therefore, the Chemistry Working Group of SciMP proposes the following recommendations:

> **Recommendation 9**: All IODP technicians should have at least a Masters degree and/or equivalent experience or training in analytical chemistry, geochemistry, or related fields. This is essential to ensure that the technician is skilled enough to deviate from a prescribed set of procedures should a given situation require it.

> **Recommendation 10**: Each laboratory technician should undergo training with the respective manufacturer of the analytical facility they are to be responsible for. Such training should include maintenance, trouble-shooting, and software. There should be regular (annual?) refresher courses that would allow the technicians to stay up-to-date with hardware and software developments.

> **Recommendation 11**: Each laboratory technician should undergo training at IODP-related or where applicable, university research laboratories in order to understand how to judge data quality and the problems associated with obtaining data that are of the highest quality.

Community Questionnaire from the CWG.

The Chemistry Working Group of the **Sci**entific **M**easurement **P**anel (SciMP) of the IODP is requesting input from the community regarding the types of geochemical analyses to be conducted "on-site" during IODP. The term "on-site" reflects analyses performed during the drilling phase of any IODP expedition (analogous to the shipboard analyses of ODP) and is inclusive of riser and non-riser platforms as well as Mission Specific Platforms (MSPS). We recogniz*ed* that analyses performed during drilling with a MSP may not be as extensive as with shipboard drilling.

The purpose of this short questionnaire is to ensure that the correct analyses are performed on all IODP platforms, data quality is high, and safety is not compromised. Please respond to Clive Neal (neal.1@nd.edu), on behalf of the Chemistry Working Group.

On previous ODP ocean drilling legs that you participated in, what material(s) and analyses were important? (check all that apply)

<u>Materials</u>

Hard rock
Soft Rock
Metamorphic
Water
Gas
Extracts

<u>Analyses</u>

Organic
Inorganic
Major
Trace
Isotopic
Petrographic

Please specify the types of analyses that ***were*** performed on-board ship:

Given your answer to the first question, what types of analyses ***are*** required to fully characterize materials that are important to your research?

What types of analyses do you consider are necessary to influence drilling strategy?

In your experience, what types of analyses are required to ensure safe drilling and core handling?

Using your experience with ODP, what other "on-site" analyses would be critical during the drilling phase?

Again, using your experience with ODP, how could "on-site" analyses be improved?

Would you consider using data gathered "on-site" in scientific publications?

If you answered "no" to the question above, what would it take for the "on-site" data to be considered usable by you in scientific publications?

Please feel free to provide any other feedback to us.

Many thanks,

Clive Neal
Urumu Tsunogai
Rick Murray