NBS TECHNICAL NOTE 840

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Reference Materials for Collaborative Tests of Air Quality Methods

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REFERENCE MATERIALS FOR COLLABORATIVE TESTS OF AIR QUALITY METHODS

Richard H. Johns and John K. Taylor

Reference materials and associated distribution apparatus were developed for seven ambient air contaminants and for two smokestack contaminants. These established the reference base for collaborative tests of ASTM procedures for ambient air quality and smokestack emissions carried out under a three-year program known as Project Threshold.

Key words: Air pollution; chemical analysis; Standard Reference Materials.

1. Introduction

Analysis of the atmosphere for ambient-level contaminants demands meticulous care in the handling of the sample. Gaseous contaminants such as the oxides of nitrogen and sulfur are typically encountered at concentrations of 10-100 parts per billion; at these concentrations they are prone to adsorption or condensation within the sampling equipment and reaction with its materials of construction. Similarly, particulate contaminants are subject to sampling losses and physical alteration within the sample collection train. Furthermore, a group of contaminants may react among themselves at some stage of the sampling operation adding further complication to the measurement. As a consequence of these problems, ambient air samples are almost never stored; rather the contaminant of interest is collected from a flowing air stream by filtration, absorption or chemical reaction, using the simplest and most inert plumbing arrangements possible.

Reference materials for atmospheric contaminants are subject to the same stability problems as the samples themselves. References are of considerable importance to this field because only through their use may air quality measurements from widely separated laboratories be soundly compared. Because of the storage problems of trace contaminant reference mixtures, development effort has been focused on systems wherein the contaminant is generated at the point of use.

Reference materials are ordinarily employed in chemical analysis in one or the other of two modes. In most laboratory procedures, the standard is used in a control analysis to ascertain that the procedure is producing accurate results.

This approach has the advantage of simplicity but is subject to problems if the real sample has a complex matrix. The matrix problem is largely circumbented when reference materials are used in the standard addition mode. By this procedure, the real sample is analyzed twice: At first alone, and again after the addition of a known quantity of the reference. The difference between the two results is due to the reference and since its value is known accurately the real sample can be evaluated by a simple ratio. It is by this standard addition or spiking principle that reference materials were employed in Project Threshold. It has the important advantage that the reference spike is exposed to the same matrix (the atmosphere) as the contaminant being measured.

Seven reference materials were provided for the first phase of Project Threshold through the ASTM Research Associateship at National Bureau of Standards. For two of the contaminants, NBS-certified Standard Reference Materials were employed. Standards for the remaining contaminants were developed under the program, along with equipment and techniques for their use. In addition, spectrophotometer calibration service, using certified standards, was provided at the test sites. The succeeding subsections of this report describe the development, calibration and testing of these reference materials.

2. Permeation Tubes

The permeation tube has emerged as a useful source for the generation of trace contaminant reference standards. This device consists simply of a sealed plastic capsule containing the contaminant of interest as a liquefied gas. The contaminant permeates the wall of the capsule at a constant rate which may be measured by weight loss. Rates are typically 1-10 micrograms/minute. The capsule is usually made from FEP fluorocarbon because of its inertness. Sulfur dioxide permeation tubes are available from the National Bureau of Standards as a Standard Reference Material (SRM 1625) and these were used for generation of the sulfur dioxide reference "spikes" in the first phase of Project Threshold. Nitrogen dioxide permeation tubes (not yet available in SRM form) were obtained from a commercial source, calibrated at the Bureau, and used to generate the nitrogen dioxide reference spikes. Calibration data for these devices showed them to possess permeation rates in the range of 1-10 μg/min.

Field application of permeation devices is complicated by the requirement for rigid temperature and flow control. In use, the tube is placed in a simple, thermostated glass generator and swept with dry air at a metered rate. The generator and dilution apparatus used in the field for spiking is diagrammed in Figure 1. For an exemplary spike, a permeation tube possessing a rate of 5 μg/min might be swept with dry air at 0.2 l/min and the resulting stream diluted with ambient air into a total flow of 100 l/min, to provide a spike of 50 $\mu g/m^3$. The total flow must be measured with great accuracy and the dilution apparatus must provide perfect mixing of the sample before it is delivered to the collaborating laboratories.

The orifice flow meters used in the dilution manifold are accurate to ± 1 percent. Since permeation rates are a function of temperature to the extent of nearly 10 percent per degree centigrade, the generator temperature must be controlled within narrow limits to maintain the calibration accuracy of the permeation device. In practice, temperature was easily maintained within ± 0.05°, corresponding to ± 0.4 percent constancy in output of the devices.

The fact that the dilution apparatus operates under a slight positive pressure (10 cm water) raises the question whether system pressure might affect the output rates of permeation tubes. To answer this question, an NBS-certified sulfur dioxide tube was recalibrated gravimetrically after exposure to flowing conditions at ≃ 0, 1 and 2 atmospheres absolute pressure, respectively, for 2-day periods. No change in calibration was observed under these exaggerated pressure conditions.

3. Ozone

Ozone is the least stable of the atmospheric contaminants measured in these studies. It may be present in the atmosphere at concentrations of 20-200 $\mu g/m^3$ at ground level and approaches a maximum of 2,000 $\mu g/m^3$ at 20,000 feet altitude. It is produced by the action of solar ultraviolet radiation on atmospheric oxygen, and its altitude profile arises from the opposing effects of air density and the shielding effect of the lower atmosphere toward ultraviolet energy.

An ozone spiking system was developed, calibrated and tested under the ASTM Program, although collaborative testing of ozone methods has been deferred to a later date. Ozone reference standards must of necessity be generated at the point of use. Ozone generators are based on the irradiation of air or oxygen by ultraviolet light, gamma radiation, or an

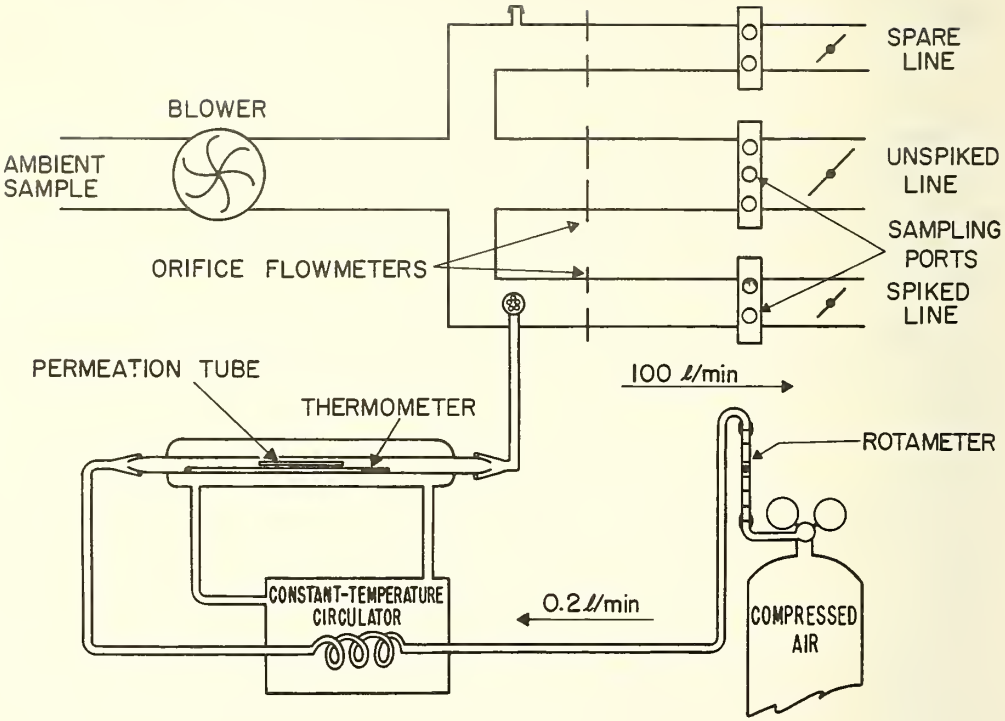

Figure 1. Spike Generator and Sampling Manifold

electric discharge. Commercial equipment for generating low atmospheric-level concentrations of ozone has recently appeared on the market, and such an instrument was purchased for Project Threshold. The ozone generator which was obtained employs a carefully-regulated ultraviolet source acting on a metered stream of air to produce ozone concentrations of 20-2,000 $\mu g/m^3$. The ultraviolet lamp is provided with thermal compensation of its power input, a photometric intensity reference, and telescoping shutters for control of ozone concentration. At maximum output, the instrument will produce 5 l/min of ozone at a concentration 2,000 $\mu g/m^3$ in air. This stream is to be diluted with ambient air into a total flow of 100 l/min for distribution to the collaborators. The resulting spike is 100 $\mu g/m^3$, a suitable level.

The instrument was tested at the Bureau and found to be reproducible in its output to the 5 percent precision which is claimed. Because the distribution manifold operates under a slight positive pressure (10 cm water), it was felt necessary to examine the effect of pressure on the generator output. Measurements with an electrochemical oxidant meter, Figure 2, showed that there is no measureable change in oxone production over the pressure range of 0-25 cm water at the generator output tap. The generator was calibrated over its output range using the neutral potassium iodide method in preparation for its use in the field. Calibration data are given in Table I.

4. Off-Stream Reference Spikes

In the procedures for nitrogen dioxide, sulfur dioxide and ozone, collaborators were provided with live, on-stream spiked ambient samples. For these analyses, their samples were drawn from distributors on two parallel mixing manifolds (Figure 1), differing only in the respect that one carried the injected contaminant spike.

The spiking technique was not feasible for five of the procedures of Project Threshold. Two of these are "effects methods" which require month-long sampling periods. Two others involve particulate reference standards, the problems of which are legion. Finally, the spiking of atmospheric lead vapor would require a level of a few parts per trillion for a 5-day period.

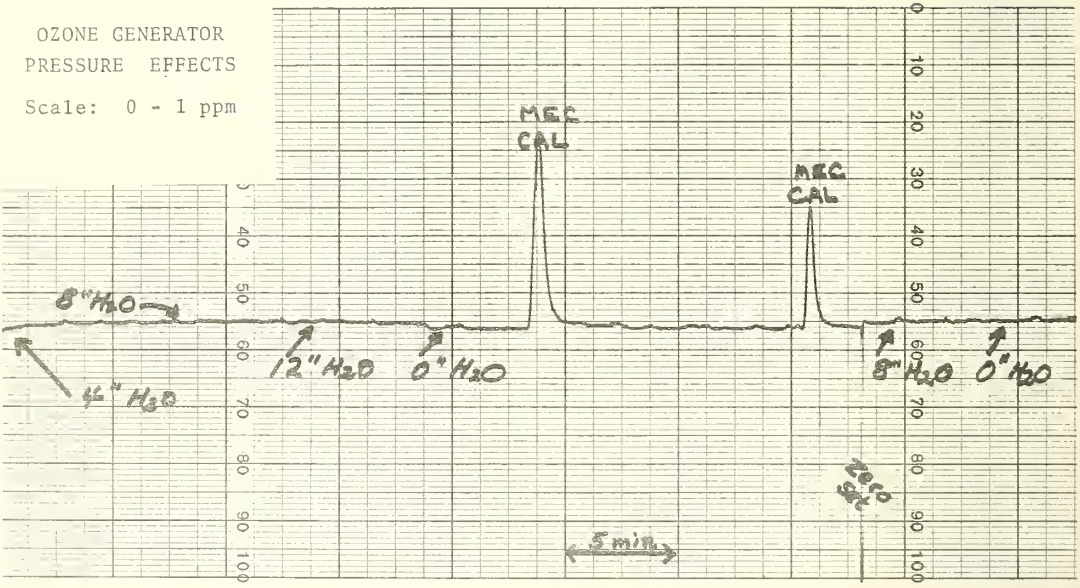

Figure 2. Ozone Generator, Pressure Data.

Table I

Ozone Generator
Calibration Data

Instrument as Received

Indicated Output, ppb	Sample Liters	Measured[a] Ozone, ppb
100	15.1	128
300	15.2	389
500	12.1	654
500	12.2	647

After Final Recalibration

500	10.0	486
500	10.0	498
500	10.0	526
500	11.0	510
500	10.0	499
500	10.0	481
500	5.0	505
500	mean	501

[a]Neutral bufferred potassium iodide method (D 2912)

For these latter methods, procedures were developed at NBS for spiking the collected sample rather than the ambient air. Collaborators were given prepared, packaged spikes for addition to their collected samples prior to analysis. The development and testing of these reference materials is related in subsequent paragraphs.

5. Sulfation

The sulfation procedure (ASTM D 2010), an effects method, is based on the exposure of a reactive "candle" which collects atmospheric sulfur compounds as lead sulfate over an exposure period of a month. After exposure, the loaded candle is digested and analyzed for sulfate by a classical gravimetric procedure. For reference spikes, measured quantities of solid potassium sulfate were provided for the collaborators, to be added to the digestion mixtures prior to analysis. The samples were packaged in gelatin capsules, packed with filter pulp, so that no further quantitative transfer measurements would be required.

The spiking procedures were tested at NBS with unexposed candles and found to give good sulfate recovery. The potassium sulfate standard, a reagent commercial chemical, was assayed and found to be 99.3 percent pure.

6. Dustfall

This simple but useful effects method (ASTM D 1739) is almost explicit in its name. Settleable atmospheric particulate is collected in a pail of water over a period of a month. Using the known mouth area of the bucket, the result is calculated in grams/sq meter/month of collected dust.

The method involves separation of the collected material into water-soluble, water-insoluble and benzene-soluble fractions by a laboratory procedure. An off-stream spike was devised to check this separation. Known mixtures of salt, sand and granular polystyrene were provided to the collaborators in vials. Alternate buckets were spiked by addition of the entire contents of vials containing this material, prior to analysis.

7. Airborne Particulate (Tape Sampler)

This second particulate method was subjected to collaborative testing during the program. With the tape sampler, ambient air is drawn at a measured rate through a circular area on a filter paper tape, which is fed mechanically between supply

and take-up spools. A dirt spot results and is quantitated by the reduction in light transmission of the paper, as measured by a simple photometer. The instrument is fully automatic and is provided with an adjustable timer for selection of its sampling period---ordinarily in the range of a fraction of an hour to several hours.

Neither on-stream nor off-stream spiking seemed feasible for this procedure, owing to uncertainties in the preparation and dispersion of uniform particulate samples. It was decided instead to provide a calibration service for the collaborating instruments. A number of Schott neutral density filters were obtained and precisely calibrated in absorbance at the Bureau of Standards. For tape sampler calibration, filters were positioned over the tape, in the light path of the instrument, to check photometric accuracy. The results were discouraging. Reflection from the tape caused erroneous measurements, and light losses resulted from the displacement of the photometer due to the insertion of the filters. Although no further work is anticipated, it would be possible to retrieve and calibrate the samplers if a better procedure could be devised.

8. Lead

Atmospheric lead is measured by a procedure (ASTM D 2681) which provides for segregation of the sample into particulate and vaporous fractions at the point of collection. The particulate material, which generally accounts for more than 95 percent of the lead, is collected on an in-line filter; vaporous lead is trapped in a charcoal absorber placed immediately downstream of the filter. The collected samples are digested and analyzed separately by a dithizone colorimetric procedure.

Separate spikes were provided for particulate and vaporous lead. For the particulate spike, NBS certified Orchard Leaves SRM 1571 was used. This material is a natural particulate containing 44 μg lead per gram of the standard. Considering the problems of dispersion and control associated with on-stream particulate spiking, the standard was used instead to spike the collected sample. For this purpose, weighed quantities of the standard were packaged in coded gelatin capsules. Each collaborator was then able to combine for analysis an encapsulated spike and his collected particulate sample, without the need for quantitative transfer of the reference material. Tests of this spiking procedure showed good recovery of lead from the SRM, as shown in Table II

For spiking of the vaporous lead fraction, samples of lead-doped charcoal were added to the respective absorbers

Table II

Test of Lead Standards

ASTM D-2681

Lead Recovery from Orchard Leaves (SRM-1571)

Sample	Lead, µg
Reagent Blank	0.9
Empty Capsule	1.9
Empty Capsule	1.3
Ink-Coded Capsule	1.1
Ink-Coded Capsule	0.9
Standard "0" (30.1 µg Pb)	28.4
Standard "6" (26.0 µg Pb)	26.7

Lead Recovery from Doped Carbon

Sample	Lead, µg
Reagent Blank	1.0
Reagent Blank	1.0
Reagent Blank	1.0
Carbon Blank (10 g)	1.2
Carbon Blank (10 g)	1.4
Carbon Blank (10 g)	1.4
1.00 ml TML-Methanol	10.2
1.00 ml TML-Methanol	9.8
1.00 ml TML-Methanol	10.4
1.00 ml TML-Methanol	10.5
Carbon, 10.1 µg lead	10.0
Carbon, 10.1 µg lead	8.7
Carbon, 10.1 µg lead	8.6
Carbon, 10.1 µg lead	8.7

prior to sample collection. The doping material was a solution of tetramethyl lead in methanol prepared by the Ethyl Corporation. The material was analyzed by the supplier and checked at the Bureau and found to contain 10.1 μg lead per milliliter. This material was pipetted onto 2-gram samples of a charcoal which had been previously analyzed for residual lead. These samples were supplied to the collaborators in coded vials to be added to the spiked absorbers prior to sample collection. A number of these spikes were tested for lead recovery both at the Bureau and at Ethyl Corporation. Results of these tests are also shown in Table II.

9. Reference Materials for Stack Emissions

The development of reference standards for source-monitoring analytical procedures is a considerably different problem from that of the ambient standards used in the first phase of Project Threshold. Nitrogen and sulfur oxides, the prominent gaseous contaminants, are present in process stacks at concentrations which are larger than ambient by a factor of about 10^5. Compared to atmospheric sampling, which can be done under near-laboratory conditions, source sampling must be done in an industrial environment which may be dirty, noisy, and unprotected from the weather. Moreover, the variety and concentration of interfering substances in stacks, and the elevated temperature of the effluent mixture, make interfering reactions more rapid and more significant.

Several procedures have been devised for applying reference standards to source measurements. The gaseous contaminant may simply be diluted with air or nitrogen and stored in a pressure vessel, to be used later as a reference standard. Aside from the problem of deterioration during storage, such a standard fails to represent the contaminant under true stack conditions. Another procedure is to build a standard emissions generator -- that is, a small furnace -- but conditions are difficult to control and the equipment is unwieldy for use in the field. For Project Threshold, a "spiking" or standard-addition procedure was desired which would permit the reference standard to be placed in the same matrix as the contaminant itself, the matrix in this case being the stack gas mixture. By way of example, parallel samples of stack gas are analyzed for sulfur oxides, but to one of these samples a known amount of pure sulfur dioxide is added as an internal standard. The difference in the response of the method to the two samples is that which is due to the known spike and this provides calibration of the method, given that response is linear with concentration.

The spiking of an industrial facility fueled at rates measured in tons per hour is impractical. Accordingly, field tests on such facilities were confined to the determination of repeatability and reproducibility -- the precision of the methods under test. Accuracy tests, using a spiking system developed at NBS, were conducted in a small pilot plant at Battelle Columbus Laboratories. The system is illustrated in Figure 3. A portion of the effluent from a furnace was piped to an adjoining laboratory via a branched line whose flow parameters could be measured accurately. To one of these branches was added a gaseous spike at a flow rate controlled by calibrated metering orifices. Previously-analyzed high purity cylinder gases were used for the purpose. The pilot plant effluent was spiked with nitric oxide and sulfur dioxide for the respective tests at spiking rates ranging up to one mole per hour. Analyses of the standard gases are given in Table III.

Table III

Analysis of Reference Gases

(Battelle, Mass Spectrometer)

Impurity	Nitric Oxide	Sulfur Dioxide
CO_2	0.08 percent	0.16 percent
N_2	0.36 percent	0.04 percent
N_2O	0.33 percent	--
Assay	99.23 percent	99.80 percent

Fabrication and calibration of the metering orifices were major tasks in design of a reliable spiking system. Four orifices were fabricated from watch jewels having bore diameters of 0.06 to 0.14 mm. These established gas flows ranging from 75-500 milliliters per minute from a 15-psig source. The spike was subsequently diluted in a duct system flowing at 200-300 liters per minute, to produce spiking levels from two hundred to several thousand parts per million.

The metering orifices were calibrated by a gravimetric procedure. Attempts at calibration by chemical and volumetric means were fraught with experimental difficulties. The gravimetric method proved simple and reliable. A small cylinder of the pure contaminant gas was fitted with a regulator, a precision pressure gage and the orifice undergoing calibration. Gas pressure was adjusted to 15 psig, the spiking condition, and the entire assembly was placed on a 10-kg top-loading

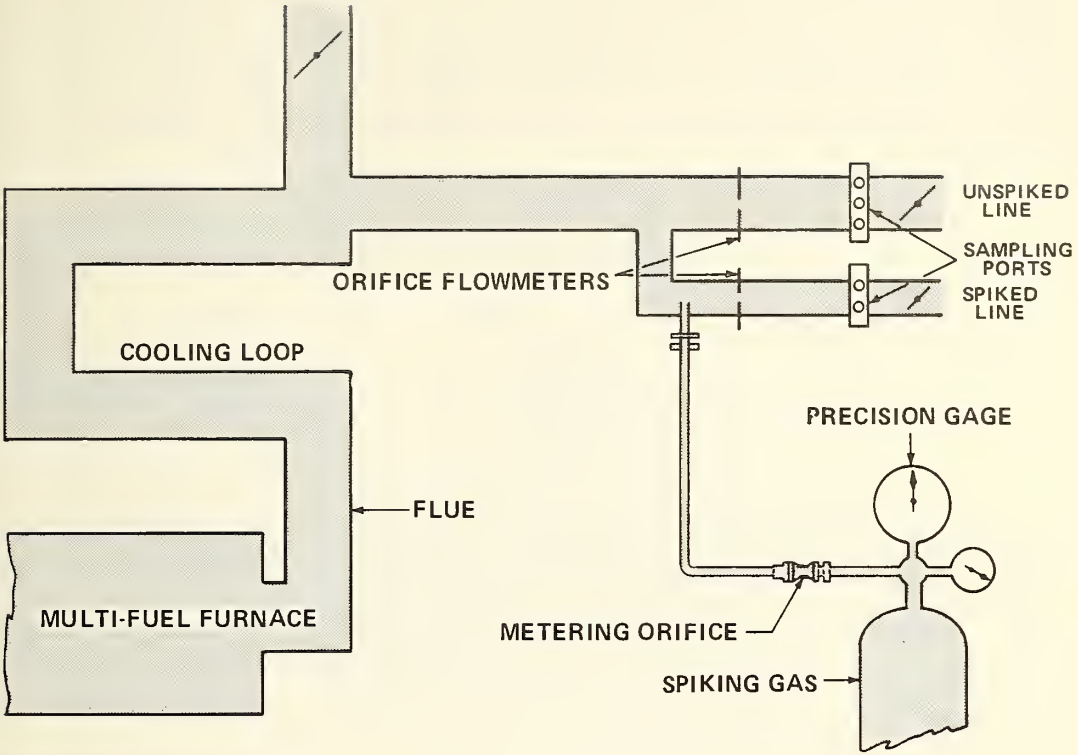

Figure 3. Stack Spiking System

balance. A balance-reading device was devised to reduce parallax error. The weight loss of the assembly was monitored manually over a period of several hours and the resulting data used to determine a gravimetric rate for the orifice using a least squares fit. Rates for the four orifices are shown in Table IV. In a separate experiment, it was determined that the orifice rates are unaffected by a downstream back-pressure of 12 inches of water, a pressure several times that existing in the duct system. A subsequent experiment demonstrated that the rates are independent of ambient temperature over the range of 15-30 °C.

Table IV

Orifice Rates, grams/second

Orifice, mm	NO, 15 psig	SO_2, 15 psig
0.06	0.001464	(not used)
0.08	0.002349	0.003390
0.12	0.004751	0.006998
0.14	0.006468	0.009702

In addition to the pilot plant spiking procedure, it was desired to assess the precision and accuracy of the analytical procedures for nitrogen and sulfur oxides, apart from the respective sampling operations. For this purpose, simulated stack samples were prepared as aqueous solutions of inorganic salts. The samples were packaged in 20-ml ampoules for distribution to the collaborators. Test samples were checked through the respective analytical procedures at NBS for NO_x and SO_x and were also checked for accuracy by a gravimetric procedure.

Each collaborator was supplied with triplicate ampouled samples representing three discrete levels of nitrogen and sulfur oxides chosen to be typical of the samples collected in the field. The sets of coded samples were distributed to the collaborators at three times during the program to be run in parallel with actual stack samples collected at industrial sites. Analysis of the resulting data provided an assessment of the accuracy of the chemical analysis procedures of the collaborators, independent of the sampling procedures in the field.

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