Questionnaire

Distinguishing Cristobalite and Opal

Summary of results based on 37 out of 66 responses

Tallied 12 Febuary 1997

This questionnaire has been prepared to determine how accredited analytical laboratories test samples and report results involving the family of crystalline cristobalite minerals and the family of non-crystalline opal minerals, opal-CT and opal-C. Please answer the questions as presented and add any supportive remarks in the space available or on a separate sheet keyed to the question code. All answers will remain confidential with respect to individual laboratories. Only cumulative results will be published in any report.

1. Method of analysis for crystalline silica phases

A. How many analyses for crystalline silica do you do in an average year

__1_ 0-10, _13__ 11-100, _19_101-1000, __4_ 1001-10000

B. What type of samples do you analyze

__11_ Air filter ____ Bulk _26_ Both

C. What method of analysis is your primary technique for quantifying crystalline silica

_22_ X-ray Diffraction _14_ Infrared __3_Chemical

_1__ Optical __2_ Other _Colorimetric__

D. What is the average level of silica in analyzed samples

_23_0-10 wt.% _10__10-50 wt.% ____ 50-100wt.%

E. Indicate the percentage of analyzed samples that are dominantly

____ quartz ____ cristobalite/tridymite ____opal 30 labs do >95% quartz

F. Problems encountered with silica analyses

_12_ Insufficient sample

_9__ Difficult samples

_17_ Interferences among the silica phases

_24_ Interferences from other phases in sample

_7__ Difficulties with identification of silica phases

_13_ Limitations on accuracy in the methods of analysis

_0__ Other

G. Which of the following methods do you employ for the detection ofcrystalline silica

_23_ X-ray diffraction _14_ Infrared Analysis

_7__ Chemical (*e.g.* Phosphoric acid digestion) ____ Differential Thermal Analysis

_1__ Scanning electron microscopy _1__ Transmission electron microscopy

_6__ Optical microscopy __2_Other __Colorimetric_____

H. Which of the following methods do you employ for the quantification of crystalline silica

_22_ X-ray diffraction _14_ Infrared Analysis

__4_ Chemical (*e.g.* Phosphoric acid digestion) ____ Differential Thermal Analysis

____ Scanning electron microscopy ____ Transmission electron microscopy

__4_ Optical microscopy __2_Other ___Colorimetric_____

I. General questions

Can you quantify crystalline silica phases at the 0.10% level

_18_ Yes _15_ No

Do you employ preconcentration methods

_11_ Yes _23_ No

What standards do you use for sample and instrument calibration

Sample_____AIHA PAT (7)_____Internal Ag (2)_____

_____MinUSil (11)_____Pre-spiked Stds. (2)_____

_____NIST (17)_____CanMet (1)_____

_____NIOSH (7)_____In-House (3+)_____

_____Arkansas (3)_____

_____Outside suppliers (6)_____

Instrument_____This question was misinterpreted to mean instrument type_____

_____not instrument calibration. Calibartion standards are listed above._____

_____

_____

_____

2. Distinguishing cristobalite and opal

A. Do you recognize cristobalite and opal as distinct silica phases

_7__ Yes _30_ No

(*If answer is "No", go to section 3*)

B. Which of the following criteria do you use to distinguish the two phases.

_6__ Value of d-spacing of peak at 22° 2θ (d = 4.04 - 4.20A)

_5__ Full width at half maximum of peak at 22° 2θ (FWHM = 0.12 - 3.5°)

_3__ Presence of double peak at 22° 2θ (d = 4.08, 4.20A)

_4__ Asymmetry of peak at 22° 2θ

_4__ Presence of peak at 36° 2θ (d = 2.50A)

_2__ Recognition of more than two diffraction peaks

_1__ Recognition of more than four diffraction peaks

____ Recognition of more than seven diffraction peaks

____ Tests for presence of water in the phase

_3__ Changes in diffraction pattern with heating

_3__ Confirmation with IR measurements or optical microscopy

____ Confirmation of analysis beyond computer produced values
_1__ TEM
_2__ Other ___Opal removed by H_3PO_4_____

3. Reporting of silica results

A. Do you recognize quartz, tridymite, cristobalite and opal as distinct minerals in reports

_19_ Yes (Except for opal where indicated) _17_ No

B. How do you report final results.

_16_ Percent abundance by individual silica mineral species
_2__ Mass Loading
_12_ Total abundance of all crystalline silica species as one number

____ Do you supply copies of the analytical data to requestors

_1__ Yes _7_ No

_24_ Only if specifically requested

4. Please complete the following information and return in the supplied envelope

Name and address of analytical laboratory

_____All but one respondend identified lab____

_____

_____

_____

Name of person responding to questionnaire

_____

5. Use this section for further comments on any answer. (Indicate the section by number and letter.)

_____Does IR. Where XRD required contracts with another AIHA lab_____

_____Never have considered opal. Never has been requested_____

_____Only analyzing AIHA PAT samples_____

_____Reports respirable dust and respirable silica_____

_____Employs XRD when quartz is found_____

_____Opal determination requires multiple tests_____

_____Several never see opal____Others comment on never seeing tridymite_____

_____Some samples have low crystallinity._____

_____Only reports quartz based on IR_____

_____Only do in-house analyses_____

Note: This is historic information. This survey is closed.

Please return the questionnaire to

Deane K. Smith
1652 Princeton Drive
State College, PA 16803

FAX number is 1-814-238-4069 or 1-814-863-7845

Thank you for your time responding to this important survey