## **DEPARTMENT OF HEALTH AND HUMAN SERVICES**

PUBLIC HEALTH SERVICE
CENTER FOR DISEASE CONTROL
ATLANTA, GEORGIA 30333

CDC--MOUNT ST. HELENS VOLCANO HEALTH REPORT #7

June 17, 1980

Identification of Free Crystalline Silica in Settled-Dust Samples of Volcanic Ash

(Submitted by Donald D. Dollberg, Ph.D., and Charles L. Geraci, Jr., Ph.D., Research Chemists, Measurement Support Branch, Division of Physical Sciences and Engineering, NIOSH, CDC)

As reported in Bulletins 10, 13, and 18 of the Mount St. Helens Technical Information Network (FEMA), the National Institute for Occupational Safety and Health is particularly concerned with the identification and quantitation of free crystalline silica. Although a number of laboratories are performing analyses of volcanic ash, it is important to emphasize that NIOSH is concerned with the analysis of the respirable (<10 µm) fraction of the dust. This factor, as well as the sample collection location, method of sampling, and method of analysis, must be taken into consideration when comparing results from various laboratories.

The chemical compound silicon dioxide, chemical formula SiO<sub>2</sub>, is commonly referred to as silica. However, this compound exists in both crystalline and non-crystalline (amorphous) forms. It is the crystalline form that is of particular concern with regard to health effects. For an in-depth discussion of this aspect, the reader is referred to "Criteria for a Recommended Standard... Occupational Exposure to Crystalline Silica, 1974" (HEW Publication No. (NIOSH) 75-120). Because of different arrangements of the silicon and oxygen atoms within the crystal lattice, it is possible to obtain several different crystal arrangements for the same chemical compound. For crystalline silica there are 3 "polymorphs": quartz, cristobalite, and tridymite. Identification and quantitation of crystalline silica can be accomplished by several techniques:

1) light microscopy and electron microscopy; 2) wet chemical analyses; 3) spectrophotometry--visible and infrared (IR); and 4) x-ray powder diffraction (XRD). Only infrared spectrophotometry and x-ray powder diffraction are capable of identifying and quantitating the 3 polymorphic forms of crystalline silica.

The Division of Physical Science and Engineering, NIOSH, has examined 3 settled-dust samples collected at Ellensburg, Yakima, and Spokane. Each settled-dust sample was wet-sieved (isopropanol) to pass the <10  $\mu$ m particles in order to obtain a sample characteristic of respirable dust. X-ray powder diffraction patterns of the sieved samples were similar and complex. Using computerized search/match techniques based on the Joint Committee on Powder Diffraction Standards (NIH/EPA Chemical Information System), several plagioclase minerals (sodium and calcium aluminum silicate) were identified as components of the sample. These results are in agreement with the microscopic examination, and infrared spectrophotometric analysis, as well as results reported by other laboratories.

Of significant importance to the identification of the silica polymorphs is the overlap of the diffraction pattern of the plagioclase minerals with the diffraction pattern of cristobalite and to a lesser extent with that of quartz. Thus, the presence of the plagioclase minerals makes the identification of cristobalite extremely difficult, if not impossible. To circumvent this problem it is necessary to remove the plagioclase minerals from the sample. This has been accomplished by chemical digestion of the plagioclase minerals using hot phosphoric acid. The procedure was developed by Talvitie (Sweet DV, Wolowicz FR, Crable JV. Spectrophotometric determination of free silica. Am Ind Hyg Assoc J 1973;34:500-6) and is the basis for the standard colorimetric method for crys-

talline silica (NIOSH Manual of Analytical Methods, 2nd ed., Volume 1, Method #P&CAM 106, HEW Publication No. (NIOSH) 77-157A).

Application of the phosphoric acid treatment was very successful. The residue from the sample digestion was re-examined by XRD and IR; both techniques confirmed the presence of quartz and cristobalite at about 2% and 4%, respectively, of the <10  $\mu$ m fraction, although the residue was not pure crystalline silica, as evidenced by small amounts of calcium, aluminum, iron, etc. NIOSH chemists are currently studying the phosphoric acid digestion procedure to optimize sample cleanup.

Moses Lake Questionnaire Survey--Preliminary Analysis

A symptom questionnaire study was carried out in the week beginning June 2, 1980, by a CDC team in Moses Lake, Washington. One hundred ninety-three house-holds were randomly chosen, and questionnaires were distributed to be completed for every member of the household. Completed questionnaires have so far been received for 150 (78%) of households, or 406 residents (an approximately 4% sample of the total population of Moses Lake). Among the 186 males and 216 females (sex of 4 subjects unknown), 109 (27%) were under 14 years of age, and 50 (12%) were aged 65 or over.

A preliminary inspection of the data indicates that an increase in complaints of cough and mild irritation of the eyes, nose, and throat occurred in the 2 weeks following the May 18 eruption. Haemoptysis was reported in 2 people, both of whom had received heavy exposures. There was little evidence of an increase in symptoms of skin irritation, diarrhea, headaches, or fever. Twenty people (5%) had visited a physician, emergency room, or clinic since the eruption, 3 for respiratory complaints and 17 for other problems seemingly unrelated to ashfall. Four people (1%) had been admitted to a hospital, only 1 for respiratory disease.

About 40% of respondents, including children, had regularly worn masks. Further analyses are to follow, as well as results of a study of a separate group of municipal workers involved in the ash cleanup at Moses Lake.

CDC--Mount St. Helens Volcano Health Reports are now being issued on a twice-a-week basis (Tuesdays and Fridays). Information in these reports represents the latest data reported to CDC; much of the information is preliminary in nature and subject to confirmation and change. It is distributed for the purpose of providing up-to-date health data from CDC and the many other groups involved in public health assessment. We hope to continue to receive relevant reports and data from others working on this problem.

Henry Falk, M.D., Peter J. Baxter, M.D., Roy Ing, M.D., Jean French, Ph.D., Chronic Diseases Division, Clark W. Heath, Jr., M.D., Director, Chronic Diseases Division, CDC, Atlanta, Georgia. James A. Merchant, M.D., Director, Division of Respiratory Disease Studies, NIOSH, CDC, Morgantown, West Virginia.