UNITED STATES DEPARTMENT OF COMMERCE
National Institute of Standards and Technology
Gaithersburg, Maryland 20899-

January 22, 2010

Mr. David Cobb
U.S. Consumer Product Safety Commission
Directorate for Laboratory Sciences
10901 Darnestown Rd
Gaithersburg, MD 20878

Re: CPSC-I-09-0019-000-000

Dear David,

Please find enclosed the progress report for the first 26 weeks of Interagency Agreement CPSC-I-09-0019-000-000 between the U.S. Consumer Product Safety Commission and the National Institute of Standards and Technology: "Development of Lead and Phthalate Standard Reference Materials for Use in Testing of Children's Products to Ensure Compliance with CPSIA Requirements". The report details research and service activities for the period July 29, 2009 to January 26, 2010.

Sincerely,

John R. Sieber, Ph.D.
Research Chemist
Analytical Chemistry Division

Enclosure

cc: S. Wise
M. Schantz
G. Turk
L. Owens

NIST

This report is submitted in partial fulfillment of the interagency agreement for the performance period July 29, 2009 – January 26, 2010.

NIST Project Officer: John R. Sieber

NIST Contributing Investigators: Michele M. Schantz, John L. Molloy

Progress Report:

During this period and with assistance from the CPSC Project Officer, NIST has accomplished the following:

- begun development of candidate SRM 2569 Lead in Paint for Toys,
- installed a new model HD-1000 XRF spectrometer on loan from XOS, Inc.[1],
- begun arrangements to acquire a state-of-the-art handheld X-ray fluorescence (XRF) spectrometer on loan from ThermoNITON,
- participated in standard test method development in ASTM F40.01 in support of XRF testing of paints on plastics and metals

Activities are itemized below by agreement task including additional details of the accomplishments and activities.

Task 1 Development of Lead Containing Paint Films on Flexible Polymer Substrate SRMs

Progress to date on SRM 2569 includes creation of a statement of work for procurement of candidate materials for three compositions of paint on polyester substrates: blank, 90 mg/kg Pb, and 300 mg/kg Pb. The contract was awarded to Rolla Coatings, Inc. of Rolla, Missouri. The original schedule of the contract called for NIST to receive prototypes by November, 30, 2009. Rolla, provided two pre-prototype samples which turned out to have a problem related to early curing of the chosen coating formulation, an alkyd resin containing red and blue organic dyes. Rolla requested and was granted an extension on the delivery of prototypes until January 15, 2010.

The company delivered the prototype paint films on January 6, 2010. The formula was changed to lacquer films with green and blue pigments. Evaluation of suitability of the films began immediately and included the following tests:

- Homogeneity testing using wavelength-dispersive XRF (WDXRF)
- Thickness uniformity testing using ultrasound gauge,

[1] Certain organizations, commercial equipment, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

- Mass fraction determinations by inductively-coupled plasma optical emission spectrometry (by D. Cobb)
- Evaluation by small spot energy dispersive XRF instruments, both handheld and bench versions.

The goals are to ensure that the films meet contract specifications for homogeneity and withstand moderate handling during use. A decision to proceed with full manufacturing will be issued on or before January 31, 2010.

Modification of the paint production contract schedule means that production will conclude on or before April 15, 2010. Determinations of Pb are slated to begin soon after production is completed by Rolla Coatings. Rolla will provide determinations of Pb and thickness as part of the contract, and they will provide a report of analysis 30 days after completion of production. NIST will perform isotope dilution inductively coupled plasma mass spectrometry (ICP-MS) determinations of total Pb, and CPSC will be asked to perform inductively coupled plasma optical emission spectrometry (ICP-OES) determinations. CPSC will provide determinations of paint thickness using an ultrasonic gauge. It has not been determined whether it is feasible to determine the density of the paint films.

Task 2 Development of Lead Containing Polymer (Plastic) SRMs

During the first six-month period of this IAG, NIST has, with assistance from the CPSC, accomplished the following:

- invested in a new NIST service development project investigate test methods and to specify compositions of polyvinyl chloride (PVC) for SRMs for metals and phthalates to be designated SRM 2859.
- NIST is seeking experts in PVC manufacturing and analysis with which to discuss this project. J. Sieber is a consultant to ASTM D20.70 on Analysis of Plastics and has contacts with several testing laboratories that have expressed willingness to assist. NIST and CPSC have experience with one source of available PVC reference materials, a company known as MAT.

Task 3 Development of Phthalate Containing Polymer (Plastic) SRMs

In addition to the activities in Task 2 above, NIST has begun to critically evaluate sample preparation and gas chromatographic separation methods for phthalate compounds.

Task 4 Evaluation of XRF Spectrometry for Analysis of Lead in Paint

NIST accepted the loan of a commercial X-ray fluorescence spectrometer for small spot measurements of Pb and additional elements in coatings, plastics, and other materials. Arrangements are in progress for NIST to accept the loan of a commercial handheld XRF instrument from a second company. If feasible, the loaned spectrometers will be used for homogeneity testing of the production SRM paint films and for various evaluation activities

designed to help CPSC and NIST familiarize themselves with commercially-offered XRF testing options. Knowledge gained may be summarized in a follow-up report on application of XRF technology to analyses of children's products subsequent to the report issued in March 2009.

This task includes involvement with committees of ASTM International for development of XRF test methods for analyses of plastics and children's products. J. Sieber and D. Cobb are members of F40 on Declarable Substances in Materials. J. Sieber is a consultant to D20.70 on Analysis of Plastics. ASTM method development activities centered on development and balloting of a new standard test method using multiple beam EDXRF to determine Pb in coatings and substrates in small areas (F40.01) and completion of revisions to ASTM D 4627 for analyses of additive elements in plastic using WDXRF (D20.70).