1 SCOPE

1.1 This method describes a general procedure for the detection of cellulose nitrate in children's optical frames, toys and other consumer products applicable as per items 6 and 7 of Part I of Schedule I of the *Hazardous Products Act*.

2 APPLICABLE DOCUMENTS

2.1 ASTM Standard test method for quantitative determination of cellulose nitrate in alkyd modified lacquers by infrared spectrophotometry, D-3133, 1996.

2.2 J. Haslaw, H.A. Willis and D.C.H. Squirrel, Identification and Analysis of Plastics, 2nd edition, Butlerworth and Co., Chapter 2, 1972.

2.3 Project report 98-0467 "Determination of cellulose nitrate in children's toys."

2.4 B. Séguin "Revision of Method C17 - Detection of cellulose nitrate in children's optical frames, toys, and other consumer products", Health Canada, PSL, Project report no. 2002-0712 (2003).

3 REAGENTS AND APPARATUS

3.1 Infrared spectrophotometer equipped with Fourier Transform acquisition system
3.2 Electric grinder
3.3 Infrared lamp
3.4 Acetone, A.C.S. Certified
3.5 Dichloromethane, Optima Grade
3.6 Tetrahydrofuran, HPLC Grade
3.7 Aluminium platter (weighing pan)
3.8 Potassium bromide (KBr), IR Grade
3.9 Hydraulic Press used for making KBr pellets (maximum pressure 25 tons)
3.10 Cellulose nitrate filters (Whatman 0.45 µm, 25 mmφ)
3.11 KBr die kit
3.12 Polystyrene NIST standards

4 EXPERIMENTAL PROCEDURE

4.1 Cut sample in small fragments. Deposit the sample fragments on an aluminium platter and dissolve them using acetone, dichloromethane, or tetrahydrofuran, depending on the type of plastic. Allow the organic solvent to evaporate at room temperature in order to collect the plastic film that is formed using this procedure. An infrared lamp may be used to reduce the drying time.

4.2 If a film is produced it can be used in the sample holder (section 4.4). If no film is produced, ground in fine particles, the plastic material using an electric grinder and follow the instructions of section 4.2.1.

4.2.1 Disc preparation

For better results, drying of KBr powder is best done by leaving it in a shallow dish in an oven at 120˚C for approximately 24 hours. It may then be transferred in a bottle and kept in a dessicator.

The preparation of a disc without adding any sample is prepared to check the background signal.

- Place 3 micro-spatula scoops (approximately 0.225 g) of KBr in the mortar. Smash and mix the mixture using the mortar and pestle.
- Use the KBr die kit (See Appendix) to form a disc. Before preparing the discs, make sure that all parts of the die assembly are clean and dry. Use a Kimwipe or other suitable tissue and water to clean them.
- Assemble the base and cylinder.
- Place one of the pellets polished face up into the cylinder.
- Place a quantity of powder into the cylinder, enough to cover the pellet.
- Distribute the powder as evenly as possible by lightly shaking.
- Drop the top pellet into the cylinder (polished surface down) and press down lightly with the plunger.
- Place the die under the pellet press and adjust the height by turning the black wheel at the surface of the press.
- Operate the handle until the pressure is 5 tons, wait 5 minutes.
- Release the pressure slowly to zero.
- Operate the handle until the pressure is 10 tons with the handle and wait an additional 10 minutes.
- Release the pressure slowly to zero.
- Remove the die from the press
- Invert and support the rest of the assembly on the plunger
- Remove the base slowly
- On a counter, slowly apply a pressure on the plunger until it moves up through the cylinder lifting the lower pellet and the KBr disc clear of cylinder
- Place the disc on the support for analysis

Note: It is advisable to handle the discs with tweezers, NEVER touch disc with bare hands.

4.2.2 Preparation of a sample disc

- Mix the sample with some KBr in a approximate proportion of 1 part sample: 3 parts KBr (1:3) and follow the instructions as above (section 4.2.1).
- The sample is then ready for analysis (see section 4.4).

4.3 FT-IR Instrument set-up parameters

Select the following experiment "C17- Detection of cellulose nitrate" on the FT-IR which included all parameters below:

- Number of scans: 32
- Resolution: 4
- Spectral Range: 4000 to 400
- Source: IR
- Detector: DTGS KBr
- Beamsplitter: KBr
- Collect a new KBr background every 60 minutes

4.4 Analysis of sample

Be sure to make all controls of the instruments before every analysis and complete the log book. Analyse the background by using a sample free KBr disc.

The infrared spectrum is obtained by depositing the film or the KBr pellet of the sample for analysis on the sample holder of the infrared spectrophotometer. The results obtained are compared to the infrared spectrum of a control sample (cellulose nitrate filters, Whatman 0.45 µm, 25 mmφ).

5 CALCULATIONS AND REPORTING

5.1 The results of analysis are reported in the following format:

Sample no.	Specimen no.	Description	Presence of cellulose nitrate
			Yes/No

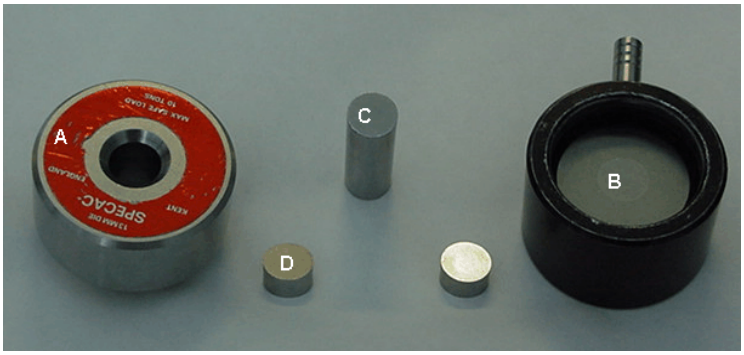
6 QUALITY CONTROL PROCEDURE

6.1 The normal and correct operation of the spectrometer shall be verified according to the following guideline:

6.1.1 Run the system validation of the instrument using the polystyrene NIST standards 1.5 mil and 3.0 mil. If the results pass all the validation tests, the system validation report shall be included in the log book. Also, all the different section of the log book should be complete. If the spectrometer failed the validation tests, the instrument shall be checked or repaired and the validation tests shall be ran again to meet the requirements prescribed by the manufacturer.

APPENDIX

Figure 1: 13 mm KBr die parts (Max Safe Load of 10 tons)



A: Cylinder
B: Base
C: Plunger
D: Two Pellets with a polished face

Figure 2: Typical spectra of the control Cellulose nitrate filters (Whatman 0.45 µm, 25 mmφ)