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1 SCOPE

- 1.1 This method describes a general procedure for the identification of alkyl cyanoacrylate in glues and adhesives applicable to Part 4 of the Consumer Chemicals and Containers Regulations, SOR/2001-269.

2 APPLICABLE DOCUMENTS

- 2.1 NEXUS FT-IR spectrometers, 470 User's Guide, Nicolet.
- 2.2 B.Marchand, P. Lambert. Analysis of glues and adhesives for alkyl cyanoacrylate. Product Safety Laboratory, Project Report No. 99-0549, March 2000.
- 2.3 P. Lambert. Sample: S-1001712, Pro-seal-Future glue gel, March 2000.
- 2.4 B. Séguin "Revision of Method C16 - Analysis of alkyl cyanoacrylate in glues and adhesives", Health Canada, PSL, Project report no. 2002-0713. (2003)

3 REAGENTS AND APPARATUS

- 3.1 Infrared spectrophotometer equipped with Fourier Transform acquisition system
- 3.2 KBr cell
- 3.3 Ethyl 2-cyanoacrylate, E-1505, Sigma-Aldrich Co.
- 3.4 Polystyrene NIST standards
- 3.5 Kimwipes or other suitable non-abrasive tissue
- 3.6 Potassium bromide (KBr), IR grade
- 3.7 Hydraulic press used for preparing KBr pellets (maximum pressure 25 tons)
- 3.8 KBr die kit

4 EXPERIMENTAL PROCEDURE


4.1 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 to a bottle and kept in a dessicator.

Preparation of KBr disc

A blank disc (a disc made 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. Crush and mix the mixture using the mortar and pestle.

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- 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 KBr 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 face 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 face of the press.
- Operate the pump handle until the pressure is 5 tons, wait 5 minutes.
- Release the pressure slowly to zero.
- Operate the pump handle until the pressure is 10 tons, 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 to 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 Preparation of standard and sample

4.2.1 Preparation of a standard disc

- The standard (Ethyl 2-cyanoacrylate) is spread over a KBr cell with Kimwipes or other suitable tissue and placed in the compartment sample of the instrument. Analyse the sample as per section 4.3 to 4.4.

4.2.2 Preparation of a sample disc

- The sample is spread over a KBr cell with Kimwipes or other suitable tissue and placed in the compartment sample of the instrument and the analysis is done following the instructions in section 4.4.

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4.3 FT-IR Instrument set-up parameters

Select the following experiment "C16- Detection of alkyl cyanoacrylate" on the FT-IR which includes all parameters below:

- Number of scans: 200
- 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

Analyse the sample using the FT-IR. Be sure to make all controls of the instruments before every analysis and complete the log book.

Collect the spectrum of the sample to be analysed and compare the spectrum with the library (Hummel polymer and additives and CCAC Product Safety Laboratory).


The characteristic band for the CN group is about 2240 cm^{-1} and for the R-COO-R' group is about at 1750 cm^{-1} . Figure 2 shows the spectrum obtained for the ethyl 2-cyanoacrylate which has been included in the CCAC PSL library as a control (the spectrum in the library is named ethyl 2-cyanoacrylate on KBr disc 20030407).

Note: If you obtain a spectrum with a percent transmittance lower than 60%, you should remake your disc with less sample by following steps 4.2 and 4.3. When the percent transmittance is low, it means that your disc is too opaque.

5 CALCULATION AND REPORTING


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

Sample no.	Specimen no.	Description	Presence of Alkyl cyanoacrylate
			Yes/No

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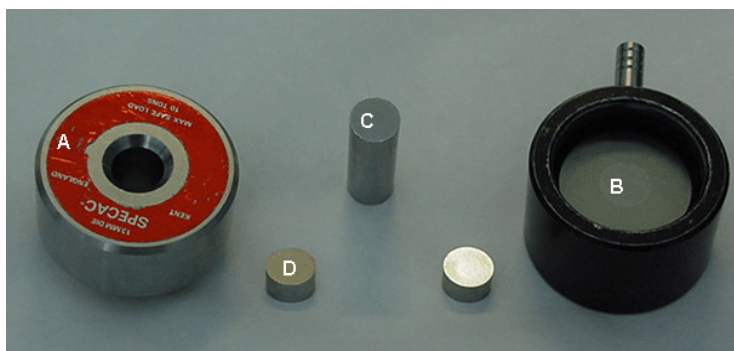
6 QUALITY CONTROL PROCEDURE

- 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 sections of the log book should be complete. If the spectrometer fails the validation tests, the instrument shall be checked or repaired and the validation tests shall be run again to meet the requirements prescribed by the manufacturer.

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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 standard ethyl 2-cyanoacrylate($\text{CH}_2=\text{C}(\text{CN})\text{COOCH}_2\text{CH}_3$).

