Technical Procedure for Microspectrophotometry

1.0 Purpose – This technical procedure shall be followed for the operation of the CRAIC QDI 2010 microspectrophotometer.

2.0 Scope – This procedure applies to the CRAIC microspectrophotometer in the Trace Evidence Section.

3.0 Definitions – N/A

4.0 Equipment, Materials, and Reagents

4.1 Equipment

- CRAIC QDI 2010 microspectrophotometer
- Standardized filter set containing quartz reference slide, holmium oxide, didymium oxide, and neutral density filters

4.2 Materials

- Quartz slides and cover slips
- Glass slides and cover slips
- Lens paper

4.3 Reagents

- Glycerol
- Xylene substitute
- Spectroscopy grade isopropanol or acetone

5.0 Procedure

5.1 Start-Up

5.1.1 Turn on the microscope, spectrophotometer, and the light sources that are going to be used, one at a time (transmitted, reflected, fluorescence). Wait for all green lights to illuminate before turning on the next light source. Allow the lamps to warm up and stabilize for approximately 30 minutes.

5.1.2 Rotate the turret below the microscope oculars to 1 (Transmission). Ensure the TL electronic shutter light is on while the RL electronic shutter light is off.

5.1.3 Start the LambdaFire software.

5.2 Daily Performance Check

5.2.1 A performance check shall be performed prior to the analysis of any evidence samples and shall include checking the wavelength and photometric accuracy with holmium oxide, didymium oxide, and neutral density filters (0.1OD, 0.5OD, 1.0OD). A sample

log shall be maintained that includes the date, initials of the Forensic Scientist, type of analysis performed and that the instrument passes the performance check.

5.2.2 To perform the performance check, select "Auto Calibration – Transmission" from the "Tools" menu.

5.2.3 Run Autoset Optimize, Dark Scan, and Reference Scan.

5.2.4 Following the instrument prompts, perform the wavelength and photometric calibration checks.

5.2.5 The instrument will indicate if all performance checks are within manufacturer's established limits. If all of the performance checks are within the manufacturer's calibration limits, the instrument shall be ready for use in casework. If it is not within those limits, the instrument shall not be used for casework.

5.2.5.1 If the instrument is not within the limits, ensure the filters are lying flat and complete the performance check again.

5.2.5.2 If it still does not pass, perform a Köhler illumination adjustment on the microscope and repeat the instrument wavelength and photometric calibration check steps. See the Trace Evidence Section Technical Procedure for Microscopes for Köhler illumination adjustment.

5.2.5.3 Do not proceed with casework samples until all of the filter peak mark values are within an acceptable range of the NIST certified reference values (as determined by the computer). Contact CRAIC Technical Support as needed.

5.2.5.4 Once maintenance has been performed, the performance check shall be done. If the samples pass the performance check, the instrument may be used for casework.

5.3 New Instrument Setup and Performance Verification

5.3.1 New instruments shall be installed by a certified engineer according to the manufacturer's guidelines.

5.3.2 The samples utilized in the daily quality control performance check shall be analyzed. If the samples pass the performance check, the instrument can be used for casework.

5.4 Analysis of Casework Samples

5.4.1 Samples can be analyzed in Transmission, Reflection, or Fluorescence.

5.4.2 Place the microscope slide containing the colored sample on the stage and focus on a sample. The sampling aperture (black square on monitor) shall be completely contained in the area of the sample to be measured.

5.4.2.1 If analyzing only in the visible range, samples may be mounted on glass slides with/without glass coverslips and using a non-fluorescing mounting medium.

5.4.2.2 If analyzing in the UV and visible ranges, samples shall be mounted on quartz slides with/without quartz coverslips and using a non-fluorescing mounting medium.

5.4.3 Move the sample away from the aperture area, until a reference (clear) area is found on the microscope slide. Run Autoset Optimize, Dark Scan, and Reference Scan.

5.4.4 Reposition the aperture on the sample and collect a sample scan choosing the correct light source and objective. All casework samples shall be run with a resolution factor of 6 and shall average a minimum of 100 scans.

5.5 Shutdown

5.5.1 Turn off the microscope, spectrophotometer, and all light sources.

5.5.2 The computer may remain on.

5.6 Maintenance – Maintenance shall be performed as needed

5.7 Standards and Controls

5.7.1 A QC check shall be performed prior to the analysis of any evidence samples and shall include checking the wavelength and photometric accuracy with holmium oxide, didymium oxide, and neutral density filters.

5.7.2 The holmium oxide, didymium oxide, and neutral density filters shall be certified.

5.7.3 There are no special storage requirements for the holmium oxide, didymium oxide, and neutral density filters.

5.7.4 Wavelength filters can be cleaned as needed with spectroscopy grade isopropanol or acetone and lens paper.

5.8 Sampling and Sample Selection – N/A

5.9 Calculations – N/A

5.10 Uncertainty of Measurement – N/A

6.0 Limitations

6.1 The microspectrophotometer cannot determine how many individual dye components are present.

6.2 The microspectrophotometer cannot differentiate between dyes with the same chromophore but slightly different chemical structures.

6.3 The microspectrophotometer cannot be used on opaque fibers that have not been reduced in cross section before analysis or fibers with a colorant level that is insufficient for detection.

6.4 Sample thickness contributes to the intensity of the absorption.

6.5 The uptake of the dye by a fiber can vary along the length of a fiber and therefore cause variation in the absorption.

7.0 Safety

7.1 The switch between the reflective and fluorescence lamps may be hot.

7.2 Halogen and mercury lamps may be hot. Care shall be exercised when using this equipment.

7.3 Refer to Appendix 1 for chemical hygiene and safety precautions.

8.0 References

8.1 ASTM/SWG Guidelines

SWGMAT. "Forensic Fiber Examination Guidelines." *Forensic Science Communications* 1.1 (1999). Chapter 3: Visible Spectroscopy of Textile Fibers

8.2 Books/Manufacturer Information

Caddy B, ed. *Forensic Examination of Glass and Paint*. New York: Taylor & Francis, 2001. Chapter 8: The Role of Colour and Microscopic Techniques for the Characterisation of Paint Fragments.

CRAIC QDI 2010 Microspectrometer User's Manual, Version 2.7, CRAIC Technologies, San Dimas, CA, 2002-2007.

Robertson, J. and M. Grieve, eds. *Forensic Examination of Fibres.* 2nd Ed. London: Taylor and Francis, 1999. Chapter 10: Microspectrophotometry/Colour Measurement

8.3 Journal Articles

Grieve, M.C., J. Dunlop and P. Haddock. "An Investigation of Known Blue, Red, and Black Dyes Used in the Coloration of Cotton Fibers." *Journal of Forensic Sciences* 35.2 (1990): 301-315.

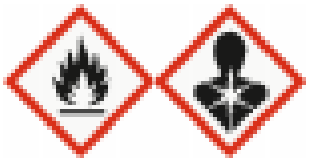
9.0 Records

- Performance Check and Use Log
- Maintenance Log

10.0 Attachments – N/A

Revision History		
Effective Date	Version Number	Reason
06/01/2020	4	Updated header to Trace Evidence Section, issuing authority to Trace Evidence Section Forensic Scientist Manager Updated all references in procedure from Trace Unit to Trace Evidence Section 5.1.3 – Removed "CRAIC CCD Image software and the CRAIC Data Acquisition" and replaced it with LambdaFire 7.3 and Appendix 1 – Added chemical hygiene information

Appendix 1

Xylene Substitute **DANGER: HIGH RISK SUBSTANCE***		
	**HEALTH**	**2**
	FLAMMABILITY	**3**
	REACTIVITY	**0**
Detection of Release	Colorless liquid; Odorless	
Signs/Symptoms of Exposure	Breathing difficulties. Skin irritation.	
PEL	OSHA TWA 500 ppm	
Associated Hazards (2)	Highly flammable liquid and vapor. May be fatal if swallowed and enters airways.	
Controls (8.2)	Use under fume hood. Avoid contact with skin, eyes and clothing. Wash hands before breaks and immediately after handling the product. Use eye protection. Handle with gloves. Wear lab coat.	
Safe handling, storage, disposal (4)(7)(13)	Handling: Use protective equipment. Do not get in eyes, on skin, or on clothing. Use only under a chemical fume hood. Do not breathe vapors or spray mist. Do not ingest. Keep away from open flames, hot surfaces and sources of ignition. Use only non-sparking tools. Use explosion-proof equipment. Take precautionary measures against static discharges. To avoid ignition of vapors by static electricity discharge, all metal parts of the equipment must be grounded. Storage: Keep containers tightly closed in a dry, cool and well-ventilated place. Keep away from heat and sources of ignition. Dispose in Hazardous Chemical Waste.	
Emergency Procedures (4.1)(6)	**Eye Contact:** Flush eyes with water as a precaution. **Inhalation Exposure:** If breathed in, move person into fresh air. If not breathing, give artificial respiration. Consult a physician. **Ingestion:** Do NOT induce vomiting. Never give anything by mouth to an unconscious person. Rinse mouth with water. Consult a physician. **Skin Contact:** Wash off with soap and plenty of water. Consult a physician. **Spills:** Avoid breathing vapors, mist or gas. Ensure adequate ventilation. Remove all sources of ignition. Evacuate personnel to safe areas. Beware of vapors accumulating to form explosive concentrations. Vapors can accumulate in low areas. Small contained spill: wearing appropriate PPE, collect with non-combustible absorbent material, (e.g. sand, earth, diatomaceous earth, vermiculite) and place in container. Dispose in Hazardous Chemical Waste. Large spills: Evacuate area and call 911 (Haz Mat).	