



## GUIDELINE for the forensic examination of paint by Fourier-transform infrared spectroscopy

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### 2 GENERAL REMARK

3 This guideline assumes prior knowledge in the forensic discipline. It is based on consensus  
4 among the relevant forensic experts and reflects the accepted practices at the time of writing.  
5 The requirements of the judicial systems are addressed in general terms only.

6

7

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12

### 1. AIMS

13 This guideline applies to the analysis of coating materials and is a part of the European Paint  
14 Group Paint Analysis Guidelines. It is intended to provide an understanding of the  
15 requirements, benefits and limitations of Fourier-transform infrared spectroscopy available for  
16 use by forensic paint examiners.

17

18

19

### 2. SCOPE

20 This guideline provides recommendations and basic information about FTIR-spectrometer  
21 components and accessories, performance assessment and test routines, sample  
22 preparation, spectral recording, data presentation and interpretation. It is intended to provide  
23 an understanding of the requirements, benefits, limitations and proper use of IR-spectroscopy  
24 and sample preparation techniques available for use by forensic paint examiners.

25

26 **3. DEFINITIONS AND TERMS**

27 For the purposes of this guideline, the relevant terms and definitions given in ENFSI  
28 documents, in ILAC G19 [1], in ISO/IEC 9000 [2], ISO/IEC 17020 [3] and ISO/IEC 17025 [4]  
29 standards, and in ASTM E1610-18 [5] are applied.

30  
31 Specific technical terms used in this guideline are defined in ASTM E2937-18 [6].  
32  
33

34 **4. BASIC PRINCIPLES**

35 Atoms in molecules are made to vibrate by the supply of energy in the spectral range  
36 between 4000 cm<sup>-1</sup> and 400 cm<sup>-1</sup> (mid-IR radiation). Depending on the molecular structure  
37 (the size of the atoms and the strength of the bonds between them), radiation of  
38 characteristic wavelengths is absorbed. By measuring the intensity of absorption within the  
39 selected spectral range an IR spectrum is obtained.  
40

41 Mid-IR spectra of coating materials are generally complex and provide numerous absorption  
42 maxima and minima that can be used for the identification, classification and comparison of  
43 coating materials by determination of chemical components like binders, fillers, pigments or  
44 additives.  
45

46  
47 **5. SAMPLE PREPARATION**

48 Initial inspection, search and recovery of forensic paint evidence are treated in EPG-  
49 GUIDELINE-001 [7].  
50

51 Any impurity on the sample surface has to be noted, to be removed and, if forensically  
52 significant, to be analysed. These impurities can be removed from the paint either by  
53 scraping with a scalpel or washing with a piece of tissue soaked with water or a non-  
54 aggressive solvent like ethanol or methanol. Other organic solvents have to be avoided  
55 because of possible chemical reaction with or dissolution of the sample.  
56

57 Sampled areas should be large enough to be representative for the item under analysis. To  
58 check for inhomogeneity of the sample, material from different areas should be taken and  
59 analysed.  
60

61 Specific preparation of paint samples for their analysis by FTIR spectroscopy include the  
62 preparation of thin sections, the use of diamond compression cell, the pressed-disk method  
63 and sample flattening.  
64

65 Sample preparation techniques should be the same for all samples being compared if  
66 possible.  
67

68 **5.1 Preparation of thin sections**

69  
70 This is the method of choice when measuring multilayered samples in relation to the EUCAP  
71 databases. It is limited to samples of at least 0,5 mm in size.  
72

73 The sample is embedded in Technovit 2000 LC resin [8] (or similar embedding medium).  
74 This monocomponent resin is hardened by irradiating with blue (300 - 400 nm) light

75 according to the instructions given by the supplier (Technotray). Always close the resin  
76 container to avoid polymer degradation by moisture take-up.

77  
78 Some samples, e.g. some refinishing paints, may react with the embedding resin, often  
79 visible as a change in colour of the sample and penetration of the embedding resin in the  
80 sample. The embedding technique should not be applied in these circumstances.

81  
82 The surface of the ensuing blocks is washed with alcohol in order to remove residual non  
83 hardened material. Samples are cut with a microtome using a tungsten carbide hardened  
84 steel knife to a thickness of 2 – 10  $\mu\text{m}$ . Depending on the orientation of the sample during  
85 embedding, either thin cuts perpendicular to the paint surface or cuts parallel to the surface  
86 are being obtained.

87  
88 Thin cuts perpendicular to the surface allow an overview of the layer system and provide  
89 easy accumulation of spectra of the individual layers from a single cut. However paint layers  
90 of 10  $\mu\text{m}$  thickness or lower are near to the diffraction limit and therefore notoriously difficult  
91 to measure in this way. These cuts may also be used and documented with microscopy for  
92 illustrative purposes.

93  
94 Cuts parallel to the surface require several cuts in order to cover the entire paint system and  
95 thus measurement is more time consuming. However they provide a larger measurable  
96 surface area and spectra of significantly better quality. Because the individual layers are  
97 sampled chronologically the inter-layer contamination issue is avoided. These cuts may also  
98 be used for MSP or Raman measurements.

99  
100 Paint chips can also be cut in ice, by fixing the sample between two plastic plates, or without  
101 an embedding step.

102  
103 Cross-sections are usually mounted free in air in order to measure them without interference  
104 of a support. In case they are too brittle and tend to shatter, they can be supported by a KBr  
105 disk or a diamond window.

## 106 107 5.2 Diamond compression cell

108  
109 This preparation method is preferred for single layer paint samples (or easily separable  
110 layers of paint), and is intended to reduce the sample thickness and thus its absorbance by  
111 applying pressure to the sample.

112  
113 The diamond anvil cell is a micro sampling accessory for use on the stage of an IR  
114 microscope or in the focal compartment of an FTIR spectrometer. The diamond windows  
115 (type II a quality) are transparent to most of the infrared spectrum except in the region of  
116 about 1650  $\text{cm}^{-1}$  to 2600  $\text{cm}^{-1}$ . Their contribution to the spectrum is minimal when using the  
117 appropriate background. If high pressure is exerted, this can alter the sample morphology  
118 and possibly its molecular characteristics such as crystallinity and orientation. The windows  
119 can be cleaned after use by wiping with a tissue moistened with ethanol.

120  
121 The paint sample is mounted on one of the diamond windows using a stereo microscope.  
122 The second diamond window is then meticulously assembled and pressure is applied by  
123 tightening the screws. The sample is now flattened and its thickness reduced.

124

125 In case of resilient materials the sample is measured using the complete cell setup. In this  
126 way the sample thickness is kept constrained at the risk of generating interference fringes.

127

128 Paint sample thickness reduction is generally sufficiently permanent so the cell can be  
129 disassembled and the sample measured through only one window. The resulting beam will  
130 be more intense and interference fringes are avoided.

131

### 132 5.3 KBr pressed disk method

133

134 A portion of the paint layer to be analyzed is isolated and placed in a small mortar. One drop  
135 of e.g. dried dichloromethane can be added and the sample gently ground with a clean  
136 pestle. Approximately half of the required KBr can be added along with another drop of e.g.  
137 dichloromethane and the mixture thoroughly mixed. A pellet is formed from the mixture using  
138 a pellet press and following the instructions.

139

140 In order to obtain a good spectrum the sample must be ground to a particle size below the  
141 shortest wavelength of light - otherwise the spectrum quality will be considerably reduced  
142 due to scattering (Christiansen effect).

143

144 KBr is hygroscopic and therefore O-H stretching vibrations at  $3450\text{ cm}^{-1}$  and O-H bending  
145 vibrations at  $1650\text{ cm}^{-1}$  can be found in spectra taken from samples prepared as KBr pellets.  
146 The KBr and the prepared pellets should be kept in desiccators to minimize these water  
147 peaks.

148

149 13 mm pellets can be analyzed without a beam condenser, 3 mm pellets have to be  
150 measured by using a beam condenser.

151

### 152 5.4 Sample flattening

153

154 Samples can be pressed using a scalpel blade or rolled using a suitable tool, and then placed  
155 directly on a suitable support. This technique is useful when performing reflection – absorption  
156 measurements.

157

158 This technique requires special care during spectral acquisition in order to avoid mixed  
159 spectra of adjacent layers.

160

161

## 162 6. **ANALYTICAL TECHNIQUES AND OPERATING CONDITIONS**

163 Paint fragments and especially traces and smears are often small and require a beam  
164 condensing or focussing device.

165

166 Both transmission and reflectance measurements may be used for the analysis of coatings,  
167 but transmission methods are preferred. The latter spectra are less prone to distortions due  
168 to variations in collection parameters. Most of the reference data of coatings, binders,  
169 pigments and additives consist of transmission spectra.

170

171 Certain types of coatings, including automotive undercoats and many types of architectural  
172 coatings, contain significant amounts of inorganic pigments. These pigments tend to have  
173 most of their characteristic infrared absorption in the lower frequency range, and several  
174 have all their absorptions in the region below  $700\text{ cm}^{-1}$ , e.g. metal oxides and silicates.

175

176 6.1 Main bench

177

178 It is possible to measure paint fragments using a diamond compression cell mounted in a  
179 beam condenser. Using the DTGS detector has the advantage of a lower wavenumber cut-  
180 off, thus enhancing the assessment of inorganic constituents.

181

182 This procedure however is not capable of discriminating areas of interest on the sample, and  
183 likewise it is not possible to visualize the sample as-measured.

184

185 6.2 FTIR microscope, transmission measurements

186

187 Infrared microscopes act as a beam condenser permitting the selection and documentation  
188 of the area of interest on a sample. It is possible to analyse very small samples (less than 1  
189 mm) quickly, reproducibly and with a high degree of sensitivity.

190

191 The sample is presented either as a thin section mounted on a sample holder in a free-  
192 standing way, supported on a salt window, or as a fragment flattened on a diamond window.

193

194 Transmission measurements on free-standing thin sections is the standard method of  
195 obtaining the EUCAP database spectra and provides the optimal data to compare with these  
196 libraries.

197

198 The use of a microscope enables the selection of the area of analysis by using field  
199 apertures for selective sample illumination. Pairs of knife edges can be used to closely  
200 match the form and size of a paint layer of the fragment or thin section. Redundant  
201 aperturing ensures that the generation of stray light is minimized. Nevertheless all spectra of  
202 individual layers should be examined to determine if absorptions of adjacent layers are  
203 contributing to the spectrum.

204

205 The detector measurement aperture width should be adjusted to just slightly less than the  
206 width of the paint sample, but preferably not less than 10  $\mu\text{m}$ . The aperture length may vary  
207 with sample geometry, but should be large enough to allow the detector to be saturated  
208 when acquiring a background spectrum.

209

210 The illumination field aperture should be adjusted so that the image of its edges coincides  
211 with those of the detector measurement aperture. The size and position of the apertures  
212 should be the same for sample and background data acquisition.

213

214 Samples should be focused as close to the center of the sample volume as possible and  
215 centered on the optical axis of the system. The condenser should be focused and re-  
216 centered if necessary (This is best accomplished using a circular pin-hole field aperture).

217

218 Objective and condenser should be adjusted for any IR window that lies between the optic  
219 and the sample in the beam path, to reduce spherical aberration and to permit more  
220 accurate focus.

221

222 6.3 FTIR microscope, reflection-absorption measurements

223

224 Reflection-absorption spectra are obtained using the reflectance mode of an infrared  
225 microscope.

226

227 The sample is mounted on a non-absorbing reflective surface, such as polished metal. The  
228 incident radiation passes through the sample, reflects off the substrate and passes through  
229 the sample a second time, i.e. double-pass transmission. A background spectrum is taken  
230 on a clean portion of the reflective surface.

231

232 The sample can be presented as a flattened particle or as a thin section stretched out using  
233 alcohol and tweezers.

234

235 Reflection-absorption (double transmittance) is carried out at near normal incidence (16  
236 degrees) where it is very efficient (80%). It is a transmission type measurement, collected in  
237 a reflection mode, and so spectral distortions are few. However, for highly reflective  
238 samples, specular reflections from the sample surface may distort the strong bands in the  
239 absorption spectrum (Kramers-Kronig conversion should not be applied to spectra obtained  
240 by reflection-absorption, because it is only applicable to true specular reflectance spectra).

241

242 The use of the detector measurement aperture is similar to transmission measurements.

243

244 Because of the high sensitivity of this method, the sample should be sufficiently flattened to  
245 avoid saturation of the spectrum.

246

#### 247 6.4 Attenuated total reflection (ATR)

248

249 Attenuated total reflectance (ATR) spectroscopy produces a spectrum by reflecting infrared  
250 radiation internally through a high refractive index crystal (Ge, KRS-5, diamond or ZnSe) and  
251 measuring that portion of the radiation absorbed by the area of a sample that is pressed  
252 against the crystal. It is a surface analysis technique yielding wavenumber dependent  
253 penetration depths up to a few  $\mu\text{m}$ . It is therefore used to measure coatings smears or trace  
254 material that cannot be separated from its support.

255

256 For good results it is very important that the sample is in intimate contact with the crystal.

257

258 Suitable crystals include:

Crystal	R.I.	Range	
KRS-5 (ThBr-ThI)	2.37	4000 – 400 $\text{cm}^{-1}$	Toxic, soft, easily damaged
ZnSe	2.4	4000 – 650 $\text{cm}^{-1}$	Toxic, brittle, susceptible to acids and bases
Ge	4.0	4000 – 700 $\text{cm}^{-1}$	Inert, brittle
diamond	2.4	4000 – 400 $\text{cm}^{-1}$	Inert, bands in range 1650 - 2600 $\text{cm}^{-1}$

259

260 As a control, the substrate itself (assuming it is not a metal) should also be analyzed to verify  
261 that its absorptions are not contributing to the spectrum of the smear. Any contribution from  
262 the substrate should be ignored.

263

264 ATR spectra are to be used with care as they present some important differences to  
265 transmission spectra. Band shifts to the higher wavelengths of up to 10 – 15  $\text{cm}^{-1}$  are  
266 observed. Bands at lower frequency are more intense compared to transmission spectra.  
267 There is enhancement in the fingerprint region and hence minor spectral differences may be  
268 more readily distinguished.

269

270 ATR may be used for comparison casework and for identification, but only if other methods  
271 fail. ATR spectra can be compared to ATR spectra but not to transmission libraries such as  
272 EUCAP.

273

#### 274 6.5 Operating conditions

275

276 Equipment shall be handled and used according to the manufacturer's recommendations.  
277 Moisture shall be kept at a minimum by efficient desiccant cartridges and/or purging with dry,  
278 dust and oil free air or nitrogen. The air (nitrogen) pressure has to be adapted to the system  
279 in order to avoid vibrations of spectrometer components.

280

281 The spectrometer and the microscope must have reached overall stability, especially thermal  
282 stability and thermal equilibrium, before starting any operational runs. This may take up to 2  
283 hours after switch-on depending on the thermal mass of the instrument and the laboratory  
284 conditions. Continuous operation is better for performance, stability and longer life time of  
285 the IR-source. It is recommended to leave the system in the stand-by mode for 24 hours a  
286 day.

287

288 Liquid nitrogen cooled detectors should be allowed to equilibrate for approximately 20  
289 minutes after the addition of liquid nitrogen.

290

291 FTIR spectra of coatings materials are collected at a resolution of 4 cm<sup>-1</sup>.

292

293 The standard spectral range used is

- 294 - 4000 – 400 cm<sup>-1</sup> with a DTGS detector (main bench)
- 295 - 4000 – 600 cm<sup>-1</sup> with a middle-band MCT detector, liquid nitrogen cooled
- 296 - 4000 – 450 cm<sup>-1</sup> with a broad-band MCT detector, liquid nitrogen cooled
- 297 - 4000 – 670 cm<sup>-1</sup> with a thermoelectrically cooled MCT detector
- 298 - 4000 – 900 cm<sup>-1</sup> with a FPA detector.

299

300 It is strongly recommended to use detection down to 650 cm<sup>-1</sup> or lower where characteristic  
301 bands of inorganic constituents may be present.

302

303 Samples are preferentially measured using a microscope in transmission mode, using 15x  
304 objective/condensor (or higher magnification). Measuring the samples without support yields  
305 spectra of better quality, although the use of salt or diamond windows is possible if required.

306

307 It is recommended to use automatically optimized gain settings.

308

309 The Blackman-Harris apodization algorithm is recommended [10].

310

311 Multi-layered coating samples that are intended for inclusion in the EUCAP databases are  
312 prepared as thin sections and measured using a microscope in transmission mode.

313

314 ATR measurements can be used when transmission measurements are not possible. They  
315 should only be compared to other ATR spectra or ATR databases and not to transmission  
316 measurements.

317

318

319 6.6 Background measurement

320

321 FTIR is a single beam technique and therefore a background measurement is needed to  
322 eliminate absorbance contributions of all system components, environment and sample  
323 holder. It consists of a reference absorption spectrum measured without sample and may be  
324 taken before or after the sample spectrum.

325

326 The system parameters for background spectra should be identical to the parameters used  
327 for sample spectra (with the possible exception of gain). These parameters include any  
328 automatic instrument control, pre-set frequency limits for spectra, wave number range,  
329 resolution, number of scans, and spectrometer aperture size.

330

331 If one background is used for more than one sample, the spectral baseline has to be  
332 checked carefully. The installation of new equipment, changing water or carbon dioxide  
333 concentrations in the air or baseline alterations (in shape, offset) will require a new  
334 measurement of the background.

335

336 Interference fringes in spectra for the diamond cell technique can be avoided if the  
337 background measurement is done with KBr between the two halves of the diamond cell.

338

339

340 **7. QUALITY CONSIDERATIONS**

341 In order to ensure good quality spectra, the functioning of equipment and the quality of the  
342 spectra have to be assessed.

343

344 7.1 Instrument performance tests

345

346 Depending on the frequency of use, quality assurance tests have to be made at least once  
347 per month. In continuous operation, a daily performance check should be made.

348

349 ASTM Standard Practice E1421-99 [9] describes how to measure the performance of  
350 Fourier Transform mid-IR spectrometers. Most equipment manufacturers offer automatic  
351 sequences incorporating these tests.

352

353 The following tests as described in ASTM E1421-99 are recommended:

- 354 - Signal-to-noise ratio
- 355 - 100% line
- 356 - Interferogram amplitude
- 357 - Energy test
- 358 - Wavenumber calibration
- 359 - Transmission reproducibility

360

361 7.2 Spectral quality criteria

362

363 7.2.1 Transmission range

364

365 The optimal transmission (absorbance) range is 100% to 10% (0 – 1 AU).

366

367 Values in excess to 100% (negative absorbance) do not make spectroscopic sense and may  
368 be caused by an incorrectly acquired background spectrum.

369



370 Scattered and reflected radiation may cause a baseline shift to lower transmittance (higher  
371 absorbance). If the baseline is lower than 80%T (higher than 0.1 AU) the reasons for this  
372 displacement should be considered.

373  
374 If the transmission is lower than 10% T (higher than 1 AU) the corresponding peaks may not  
375 be properly resolved. This would require a new preparation with less sample thickness.

#### 376 377 7.2.2 Water and carbon dioxide

378  
379 Water bands at 1650 cm<sup>-1</sup> and above 3000 cm<sup>-1</sup> should be minimal, otherwise interference  
380 with peaks of inorganic or organic compounds may occur.  
381 Minimize carbon dioxide to avoid bands at 668 cm<sup>-1</sup> and between 2380 and 2300 cm<sup>-1</sup>.

#### 382 383 7.2.3 Signal-to-noise ratio

384  
385 The signal-to-noise ratio criterion is set during validation of the method. Some samples such  
386 as highly filled metallic basecoats may not reach this criterion due to excessive scattering.

#### 387 388 7.2.4 Interference fringes

389  
390 Interference fringes are not allowed in order to avoid misinterpretation. They can be avoided  
391 by slightly tilting the sample during measurement.

### 392 393 394 8. **CLASSIFICATION, COMPARISON, INTERPRETATION**

395 Paint samples are often inhomogeneous at the microscopic level. It is therefore highly  
396 recommended to measure samples at several spots in order to assess the reproducibility of  
397 the spectral features.

398  
399 These features can be exploited on different levels depending on the purposes of the  
400 analysis and the depth of detail required.

#### 401 402 8.1 Identification of component classes

403  
404 The absorption bands present in the spectrum may be evaluated with respect to band  
405 position, band shape and band intensity. They represent characteristic vibrations of  
406 functional groups, and their combination allows patterns to be assigned to classes of  
407 compounds.

408  
409 All paint constituents contribute their characteristic pattern to the spectrum depending on  
410 their absorptivity and their concentration. This provides for complex spectra with strong  
411 overlapping, precluding quantitative exploitation in most cases.

412  
413 Classification of binder, pigment and filler types is obtained by comparison to functional  
414 group charts and spectra of known materials.

415  
416 Vigilance is required in order to accommodate sample inhomogeneity (especially with  
417 respect to pigment and filler distribution), artefacts and contaminations (from the support, the  
418 embedding resin or the glue of adhesive tape).

419

420 High filler content may result in difficulties for identifying other constituents. This is  
421 particularly the case with calcium carbonate where a treatment with 10 % HCl may dissolve  
422 the filler and help in detecting other constituents.

423  
424 The classification may assist in determining the significance and end use of a coating.  
425

## 426 8.2 Comparison between samples

427  
428 Spectra to be compared shall be manipulated as little as possible. Raw data shall always be  
429 secured and the manipulations shall be temporarily. We recommend to limit manipulations to  
430 flatlining the CO<sub>2</sub> absorptions, to correct baseline slope and to normalize the spectra, when  
431 needed.

432  
433 The comparison is made by superimposing the spectra in either transmission or absorbance  
434 mode. We recommend the following match criteria:

435

- Presence of bands : All bands of the reference spectrum are present in the questioned spectrum
- Positions : Band positions correspond within  $\pm 8 \text{ cm}^{-1}$
- Relative intensities : Pattern shape and relative intensity is similar, taking into account variability of especially pigment and filler signals in the reference spectrum

436

437 Differences due to sample inhomogeneity, artefacts and contaminations shall not be  
438 considered significant.

439

440 Spectra are considered indistinguishable if they do not exhibit significant differences.  
441 Spectra are considered dissimilar if they contain one or more significant differences  
442 If it is impossible to decide whether a difference is significant, the comparison is  
443 inconclusive.

444

## 445 8.3 Comparison of a spectrum to databases

446

447 The Paint & Glass group provides a number of databases on its website that can be  
448 exploited using the KnowItAll software (Wiley, version 2015 or more recent) [10]. These  
449 databases contain various coating samples acquired by European laboratories and can be  
450 used to characterize the questioned sample.

451

452 The software enables the simultaneous search of the FTIR spectrum and of additional  
453 properties stored with the spectrum i.e. colour, use, make, etc.

454 Correction of the questioned spectrum must be temporary. Allowable corrections include  
455 flatlining CO<sub>2</sub> absorption bands (2390-2285 and 672-664  $\text{cm}^{-1}$ ), correcting a sloping  
456 baseline, and a range of optimized corrections [11].

457

458 The search classifies the database spectra according to a hit quality index (0 – 100)  
459 calculated using a choice of algorithms. It is advised to use both the 'Correlation' and the '1<sup>st</sup>  
460 Derivative Euclidian' algorithms as they often give complementary results.

461

462 It is often advantageous to limit the search to those wavenumber ranges that contain  
463 information, i.e. excluding regions of background noise.

464

465 The reference samples do not necessarily originate from the same batch as the questioned  
466 sample. More variation on the relative intensities can therefore be expected. The match  
467 criterion on relative intensities in these conditions would become: within a pattern the highest  
468 band remains the highest.

469

#### 470 8.4 Comparison of an automotive sample to EUCAP databases

471

472 IR spectra of automotive paint samples can be compared to the European Collection of  
473 Automotive Paint (EUCAP) databases using the KnowItAll software (Wiley, version 2015 or  
474 more recent), with the aim of identifying its make or obtaining statistical data on its rarity.

475

476 In addition to the comparison described in 8.3, a simultaneous search of the constituent  
477 layers of a paint sample can be executed, each layer in its corresponding databases.  
478 Additional properties, such as colour, car make, production dates etc., can also be included  
479 in this simultaneous search.

480

481

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511  
512 10. **AMENDMENTS AGAINST PREVIOUS VERSION**  
513  
514 New document  
515  
516 ###  
517

Draft document