



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

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1

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⁻¹ and 400 cm⁻¹ (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 μm . 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 μm 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 cm^{-1} to 2600 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 cm^{-1} and O-H bending
145 vibrations at 1650 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 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 μm . 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 μm . 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 cm^{-1}	Toxic, soft, easily damaged
ZnSe	2.4	4000 – 650 cm^{-1}	Toxic, brittle, susceptible to acids and bases
Ge	4.0	4000 – 700 cm^{-1}	Inert, brittle
diamond	2.4	4000 – 400 cm^{-1}	Inert, bands in range 1650 - 2600 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 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⁻¹.

292

293 The standard spectral range used is

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

299

300 It is strongly recommended to use detection down to 650 cm⁻¹ 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⁻¹ and above 3000 cm⁻¹ 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⁻¹ and between 2380 and 2300 cm⁻¹.

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 CO₂ 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 CO₂ absorption bands (2390-2285 and 672-664 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 '1st
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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510

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

Draft document