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Bitumen and bituminous binders - Visualisation of polymer dispersion in polymer modified bitumen

Bitumes et liants bitumineux - Visualisation de la dispersion des polymères dans les bitumes modifiés par des polymères

Bitumen und bitumenhaltige Bindemittel - Visualisierung der Polymerverteilung in polymermodifiziertem Bitumen

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Foreword

This document (EN 13632:2010) has been prepared by Technical Committee CEN/TC 336 “Bituminous binders”, the secretariat of which is held by AFNOR/BNPé.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2010, and conflicting national standards shall be withdrawn at the latest by November 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 13632:2003.

Annex A is informative.

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1 Scope

This European Standard specifies a method for visualisation of the polymer distribution in polymer modified bitumen by fluorescent microscopy.

The method is applicable for most of the commercially used polymers, but before the method is used it should be examined whether the test is applicable for the actual polymer.

The method should only be used for identification purposes, i.e. in connection with production control.

NOTE Sample preparation and treatment have an important influence on the test results and it is essential to follow strictly the method described to achieve comparable results.

WARNING — The use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 58, *Bitumen and bituminous binders — Sampling bituminous binders*

EN 12594, *Bitumen and bituminous binders — Preparation of test samples*

3 Principle

A sample of polymer modified bitumen is homogenised by gentle stirring and poured into a preheated mould. After a controlled cooling procedure to ambient temperature the sample is cooled below – 20 °C for a minimum period of time. The bitumen layer is broken into small pieces and the freshly broken surface is viewed through a microscope with a magnification of 25 times to 500 times. The views can be stored photographically or electronically.

4 Apparatus

4.1 Epifluorescence microscope, (incident light excitation) with an appropriate light source and filter systems.

NOTE Examples of light source and filter system are given in Annex A.

4.2 Freezer or solid carbon dioxide.

4.3 Sharp tool.

Scissors shall not be used.

4.4 Aluminium basin, disposable, approximate height 35 mm, approximate diameter 70 mm.

4.5 Porcelain evaporating basin, diameter 150 mm, height 63 mm (approximate volume: 600 ml).

4.6 Fine aggregate (sand).

4.7 Temperature regulated sand bath.

5 Preparation of test samples

Take the sample in accordance with EN 58. Prepare it in accordance with EN 12594. Homogenise the sample by gentle hand stirring for at least 1 min and not more than 5 min. Pour the material into the aluminium basin placed in a sand bath (4.7) preheated to the same temperature as the sample during homogenising. The sand bath is an evaporating basin (4.5) containing sand (4.6). The aluminium basin (4.4) shall be totally surrounded by the sand and there shall be 20 mm of sand between the bottom of aluminium basin and the evaporating basin.

The sample is cooled to ambient temperature by switching off the heating of the sand bath. After cooling to ambient temperature, cool the sample to – 20 °C or lower if the sample needs to become brittle. Using a freezer (4.2), a cooling time of 3 h minimum is required; using solid carbon dioxide (4.2) in a dewar, 10 min is sufficient.

NOTE The cooling procedure can be essential for the morphology. By cooling in the sand bath, a steady cooling rate is assured.

6 Procedure

Prepare small pieces of the frozen material by breaking or cutting with appropriate sharp tool (4.3). This preparation shall be done rapidly to avoid warming the sample. Inspect the freshly broken or cut surface within 1 h. The inspection can be done through a protecting glass or directly on the surface. Using a protecting glass turn the freshly broken surface towards a microscope cover glass and arrange for the freshly broken or cut surfaces to stick to this without disturbing the surface by moving. Turn the cover glass over and place it on the hole of the rigid support such that a perfectly horizontal and flat surface is observed through the glass.

Select the magnification according to the particle size and distribution; magnifications of 25 to 500 are suitable. Analyse each polymer modified bitumen on a minimum of 3 independently prepared surfaces scanning the entire surface before collecting the typical picture.

Provide a reference scale with the picture.

NOTE The picture can be obtained and stored by photographic or electronic systems.

7 Expression of results

Express the binder morphology either as a picture, a picture number (according to Annex A) or a combination of the characterisation letters shown in Annex A. Intermediate numbers are not allowed. If no fluorescent emission can be detected and the sample appears all black, note this as O.

NOTE Numbers are used as a simple tool for comparing pictures and neither assessing the quality, nor the performance of the binder.

8 Test report

The test report shall contain at least the following information:

a) type and complete identification of the sample under test;

b) reference to this European Standard;

c) light source and filter system;

d) present the picture with a reference scale and if required a combination of the characterisation letters given in Annex A (see Clause 7);

e) any deviation, by agreement or otherwise, from the procedure specified;

f) date of the test.

Annex A
(informative)

Bitumen modified by polymers

A.1 Introduction

Multiphase systems like bitumen polymer blends need a certain distribution of the discontinuous phase to maintain their structure over a long life time. The modification of bitumen with polymers can reach two principal phases.

— In the first case, the polymer is more or less dispersed in the bitumen matrix. Depending on the chemical composition of the materials, their compatibility and the input of dispersing energy as well as factors like molecular weight and polarity, the system remains homogenous over a certain period of time. The level of distribution of the polymer phase has an important influence on the physical properties of the binder system.

— In the second case, the polymer phase becomes the continuous phase in which the bitumen is more or less finely dispersed, acting as a plasticiser or extender. Here the distribution of the bitumen in the polymer phase is of importance for the physical properties of the system.

Beside the two principal cases, there are blends existing where phase inversion starts and both systems are visible at the same time. This phenomenon is also dependent on temperature and time.

A way to assess the homogeneity of a blend is to view it under a fluorescence microscope when illuminated by UV light: most of the polymers produce a yellow fluorescence while the bitumen remains dark. The particle size or distribution can be assessed under different magnifications. Special preparations are necessary to obtain a proper picture. The best way seems to be to cool the sample to a temperature of at least – 20 °C. Small pieces of the material are then broken or cut off, warmed up to ambient temperature and placed under the microscope for examination of the freshly broken surface. The distribution of the polymer phase or bitumen can be assessed by measuring the particle size distribution or by comparison with standard pictures.

If no structure is visible but a fine, homogeneous, slightly yellow light is detected, the product should be called homogeneous.

Compatibility, stability and compliance can all be assessed by epifluorescence microscopy.

It should be noted that fluorescence microscopy is not able to give a final quality assessment or indication of the binder performance. It provides important information for understanding of the system and parameters which might have an influence on the final quality.

A.2 Light source and filter system

Example of light source and filter system:

Light source: High pressure Xenon lamp, 75 W

Excitation filter: BP 420/490 (transmit wavelengths from 420 nm – 490 nm)

Beam splitter filter: RKP 510 (reflects wavelengths shorter than 510 nm and transmits light of longer wavelengths)

Barrier filter: LP 515 (transmits wavelengths longer than 515 nm)

From the light source the light falls on the excitation filter, which transmits light from 420 nm to 490 nm. Light with this wavelength falls then on the surface of the specimen. Unabsorbed exciting light and emitted light is then reflected to the chromatic beam splitter filter, which reflects light shorter than 510 nm and transmits light of longer wavelength. According to Stokes' Law emitted light will have a longer wavelength than its exciting light, hence the beam splitter filter effectively reflects exciting light and transmits emitted light, in this way separating the two. To eliminate any residual exciting light, the emitted/transmitted light with wavelength longer than 510 nm finally falls on the barrier filter, which has a high transmission for wavelengths longer than 515 nm.

A.3 Polymer bitumen pictures

The following pictures show some typical dispersions of polymer in bitumen. The size of the pictures are 1,00 mm x 0,75 mm (approximately).

The distribution can be described through a code of characterisation letters:

Continuous phase: P: Continuous polymer phase

B: Continuous bitumen phase

X: Both phases are continuous (inter – twisted phases)

Description of the phase: H: Homogeneous

I: Inhomogeneous

Description of size: S: small (< 10 µm)

M: medium (between 10 µm and 100 µm)

L: large (> 100 µm)

Description of shape: r: roundish

s: stripes

o: other

Some typical pictures are shown hereafter but not all possible morphologies are covered in these pictures. The pictures are characterised by a number and a letter code according to the system described above.

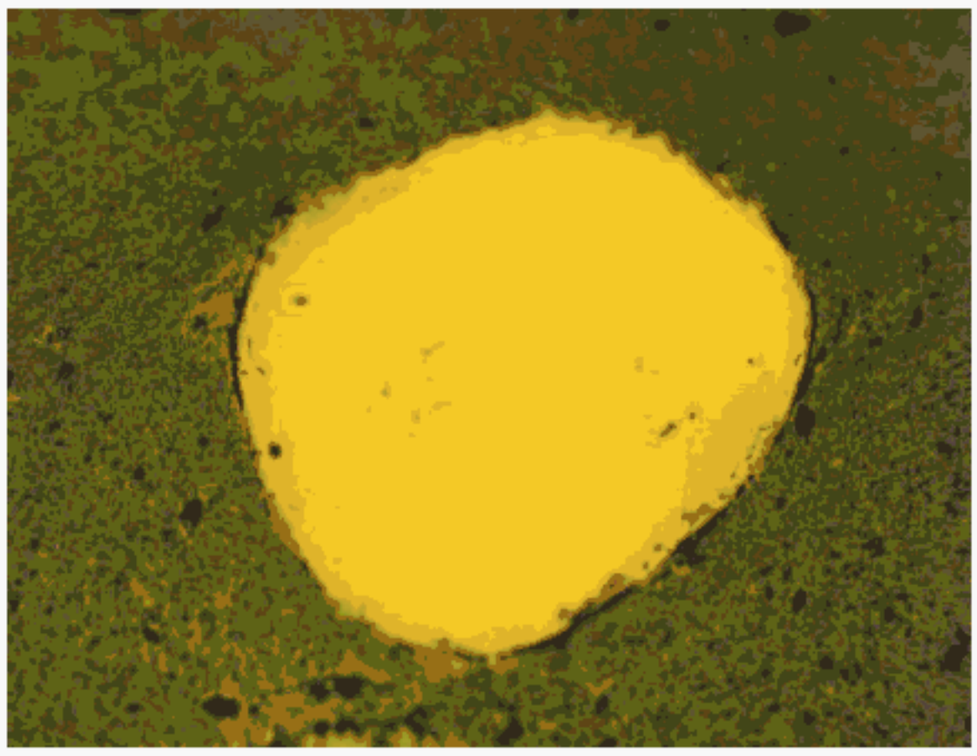

Figure A.1 — P/I/L/r

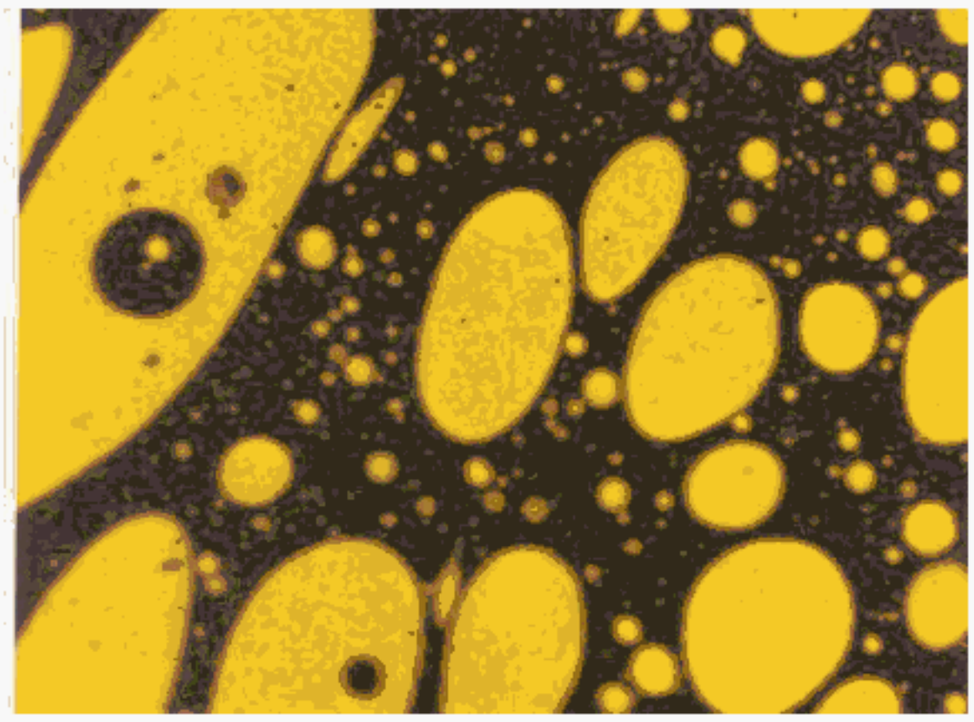

Figure A.2 — B/I/SL/r

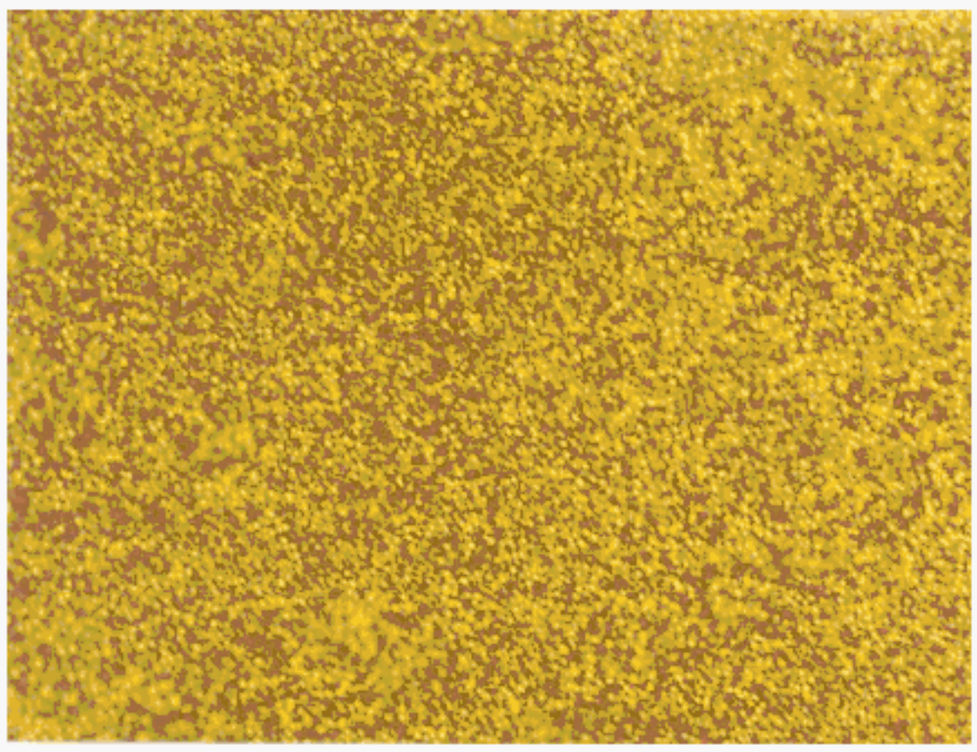

Figure A.3 — B/H/S/r

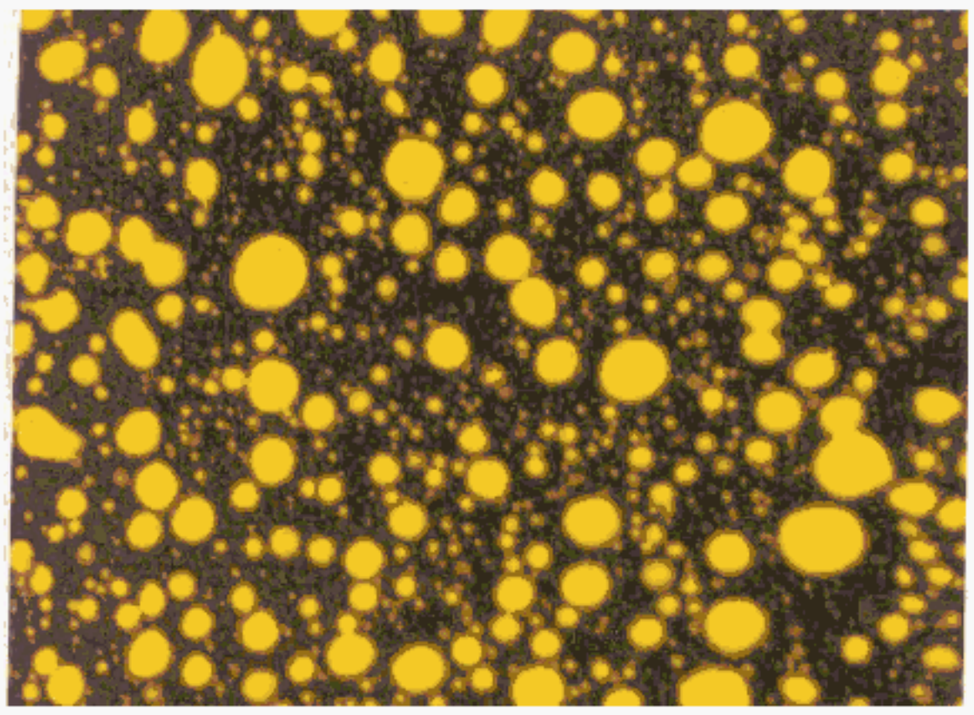

Figure A.4 — B/H/SL/r

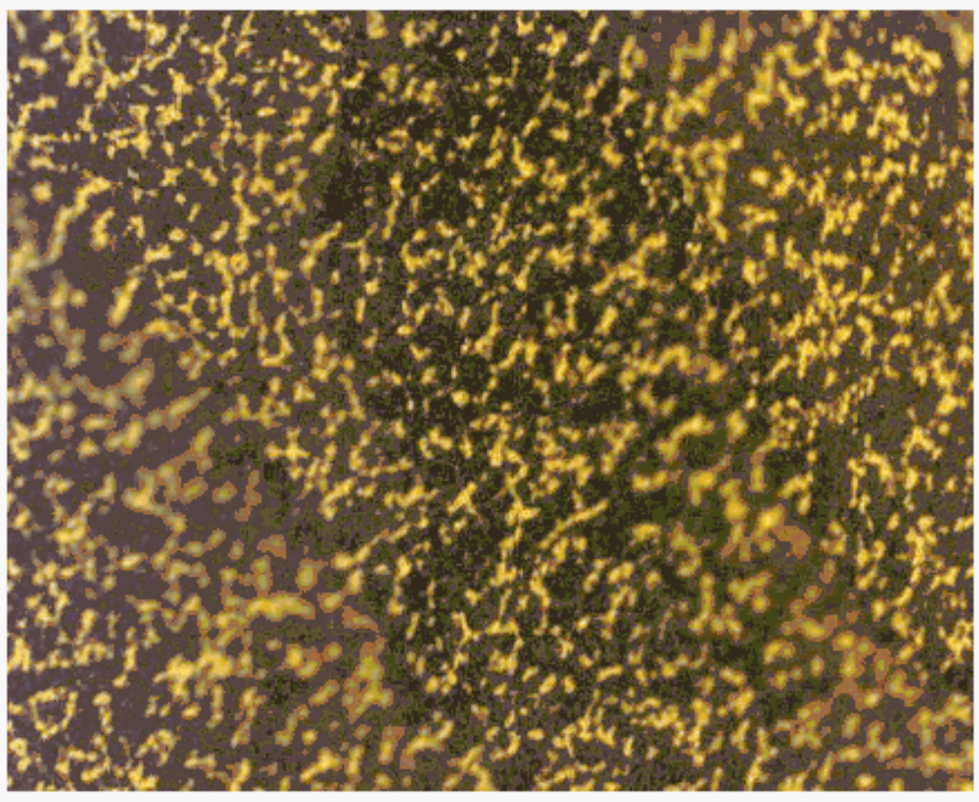

Figure A.5 — B/I/L/o

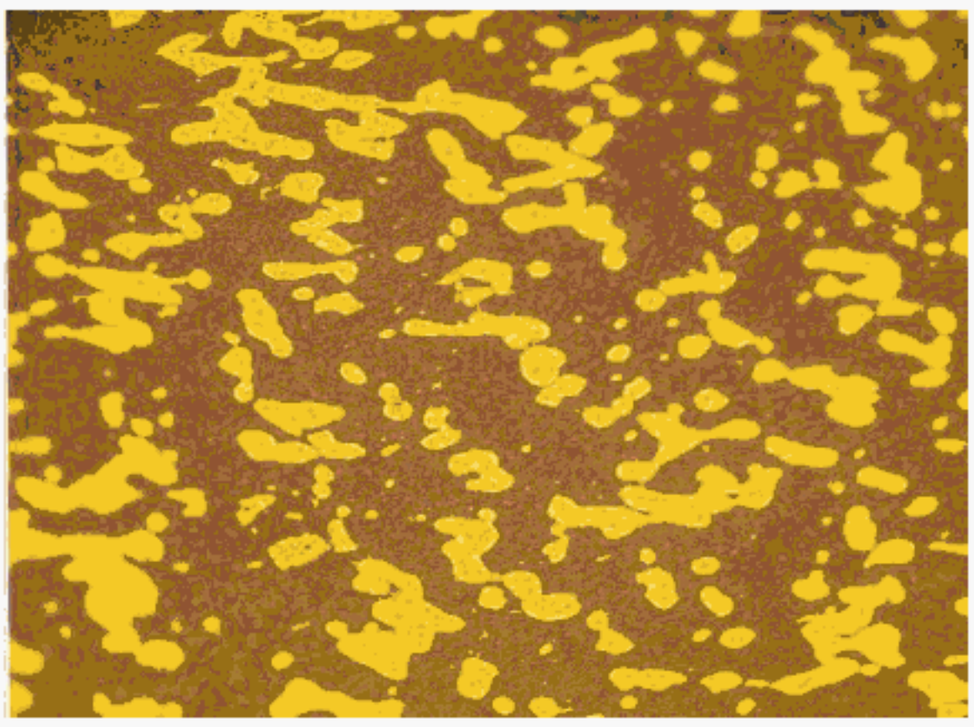

Figure A.6 — B/I/SL/o

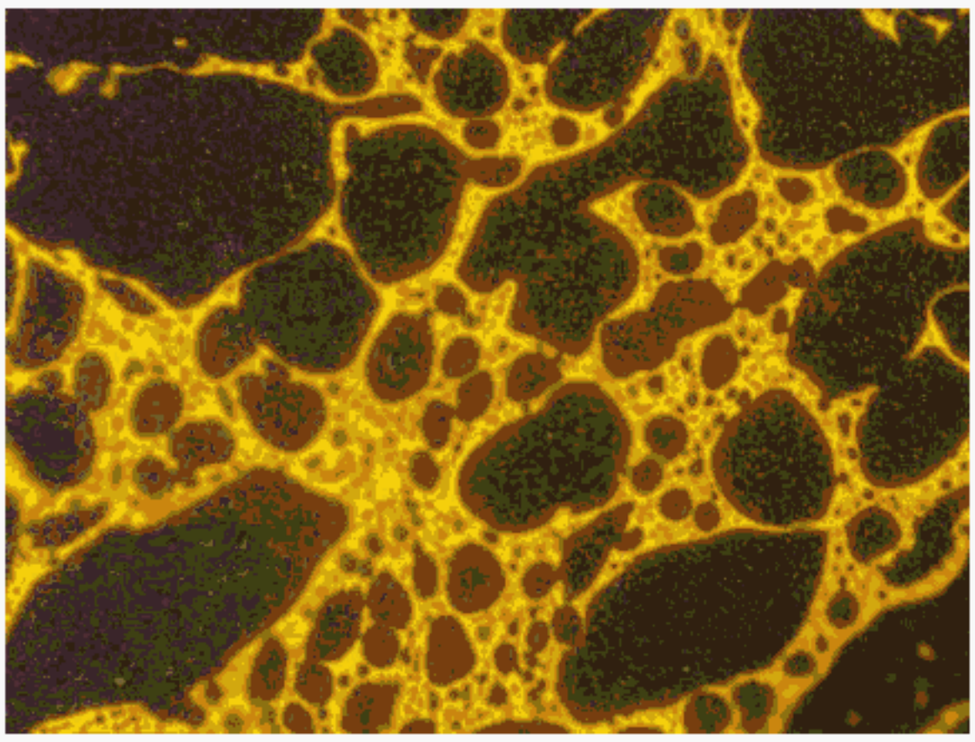

Figure A.7 — P/I/SL/o

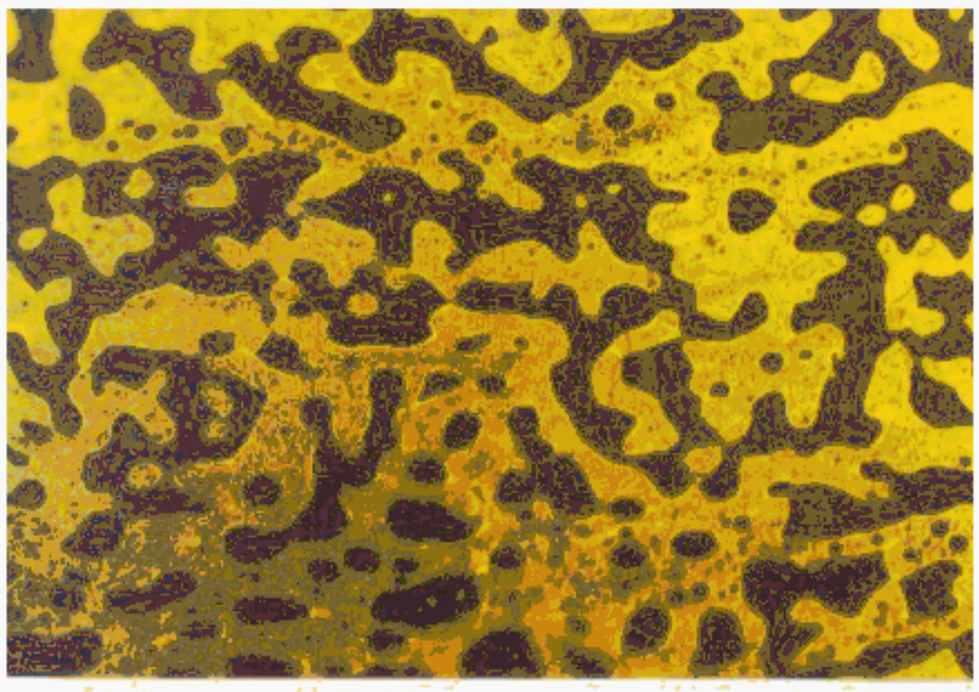

Figure A.8 — P/I/SL/o

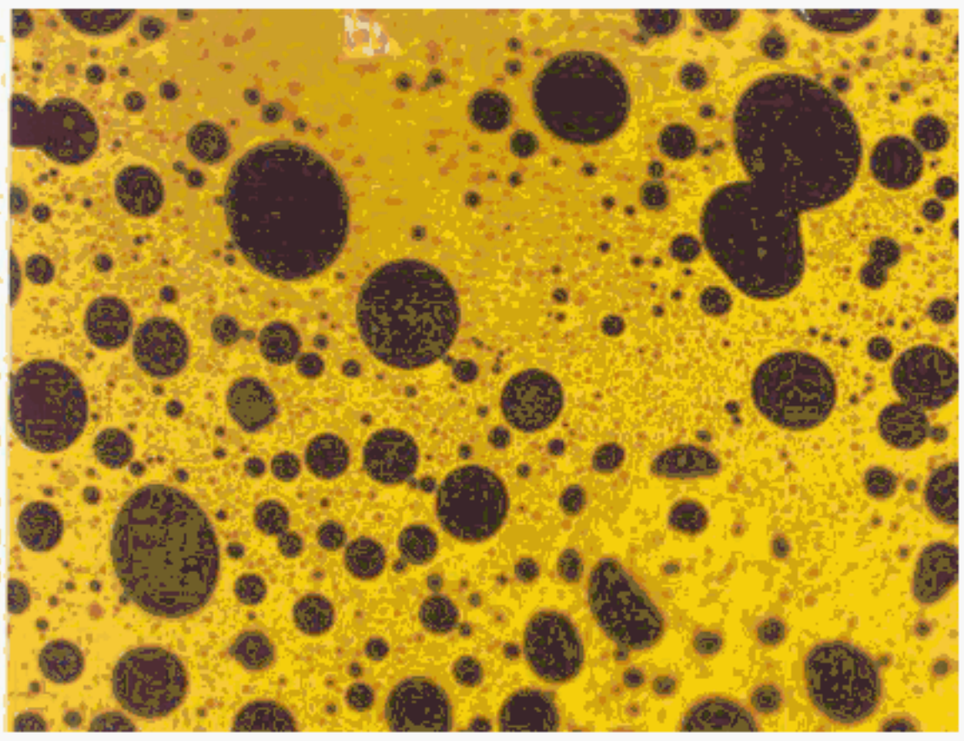

Figure A.9 — P/I/SL/r

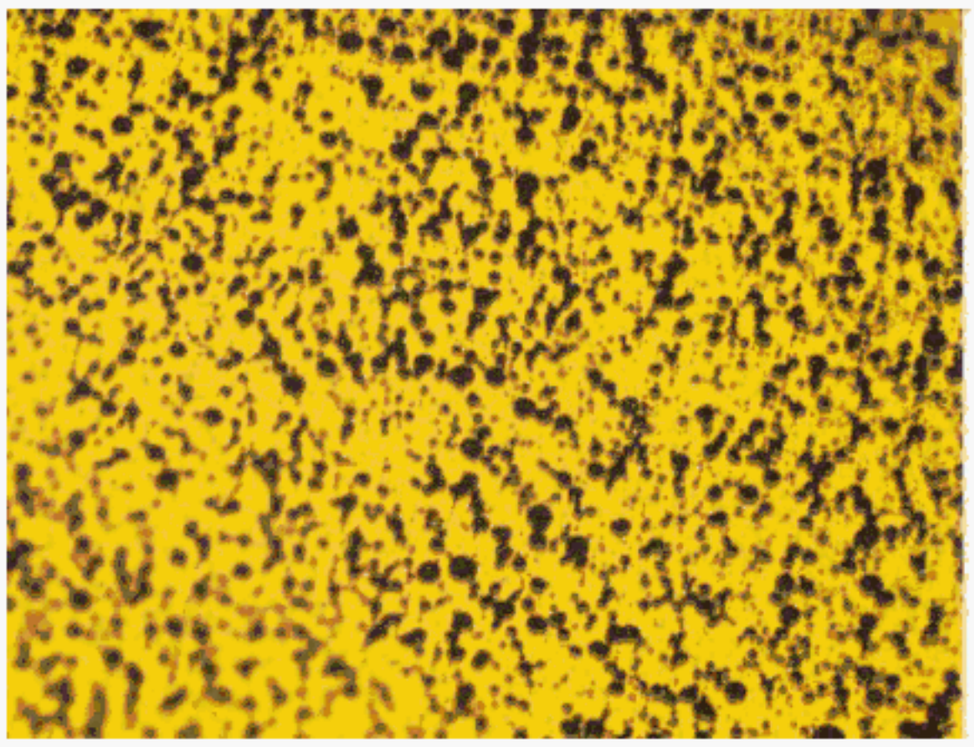

Figure A.10 — P/H/S/r

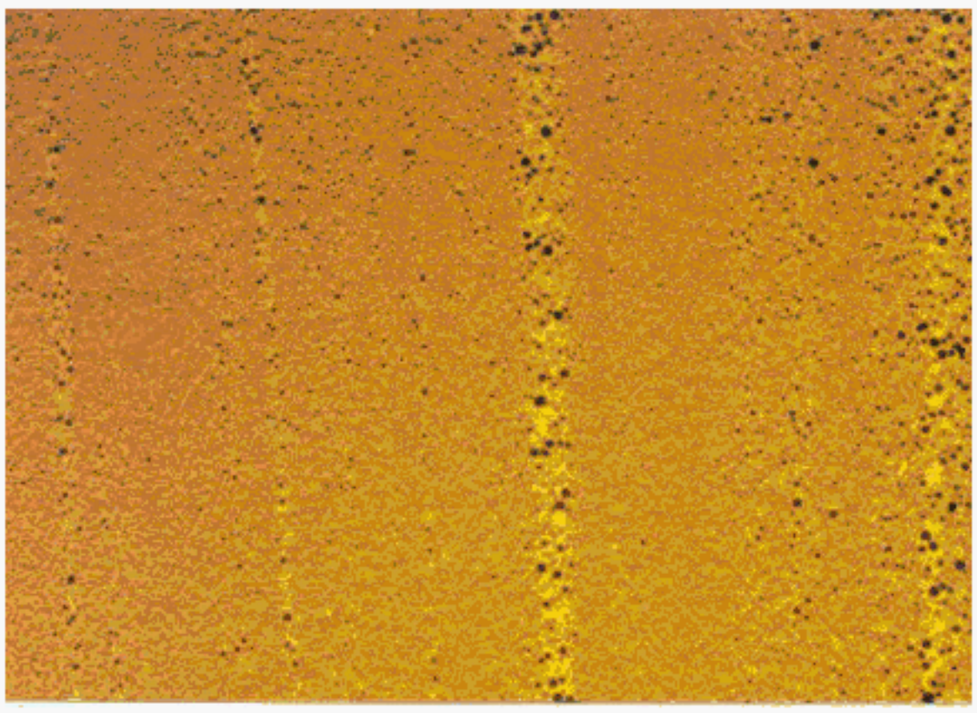

Figure A.11 — P/I/S/s

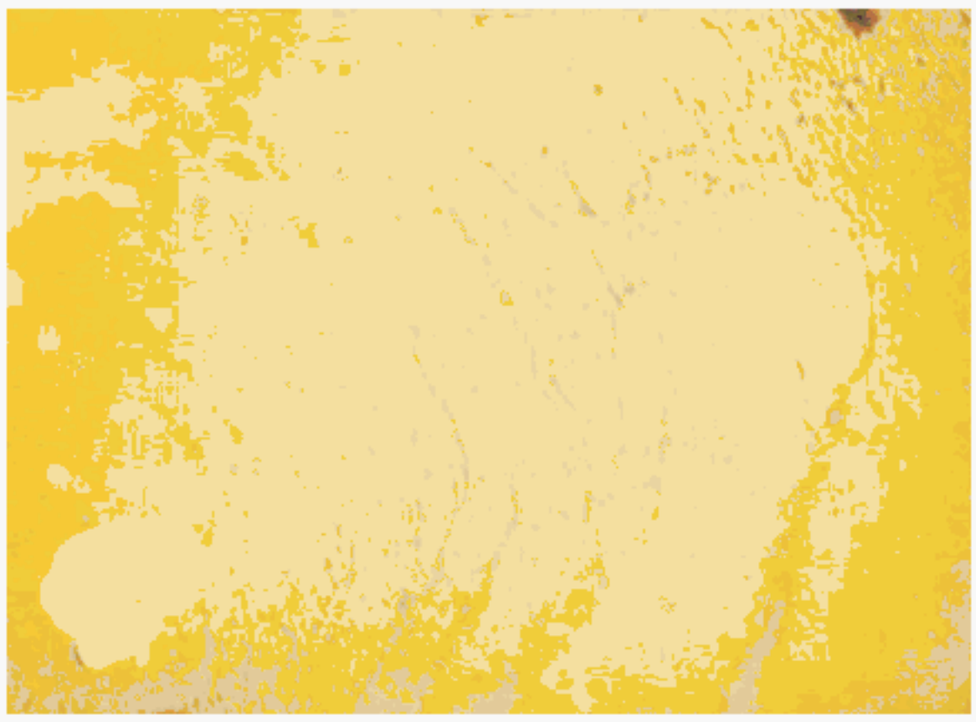

Figure A.12 — P/H/S/o