Appendix Nr 1

Certificates of analysis

Linseed oil

SIGMA CHEMICAL COMPANY

Certificate of Analysis

LINSEED OIL
Insect Cell Culture Tested
Product No. L-4275
Lot No. 22H01731
CAS No. 8001-26-1
Assay Date: January 1993

Test	Observed Results
Appearance	Hazy yellow-brown liquid
Solubility	Clear faint yellow solution @ 0.1 ml/5 ml $CHCl_3$
IR Spectrum	Consistent with structure
Density	0.93 g/ml
Refractive Index	1.50 at 20°C
Biological Test	Pass

The growth promoting capacity of this product was evaluated by incorporating into insect diets and assessed using an established colony of the southern corn borer. First or second instar larvae were placed on the diet and the number of individuals reaching critical developmental stages, i.e. pupation and eclosion, were determined. During the testing period cultures were examined microscopically for any structural abnormalities that may indicate toxic components in the diet.

Sigma Quality Assurance

Sigma warrants that its products conform to the information contained in this and other Sigma publications. Purchaser must determine the suitability of the product for its particular use. See reverse side of invoice or packing slip for additional terms and conditions of sale.

SIGMA CHEMICAL CO. P.O BOX 14508 ST. LOUIS, MO. 63178 USA
For Technical Service in the USA/Can 800-848-7791 Outside the USA/Canada 314-771-5765 Ext. 3950

Linseed oil

Back To Description / Pricing

Printed on 09/18/2001 06:06:39 AM

Aldrich Product

Product Number: 430021
Product Name: Linseed oil
CAS: 8001-26-1
MDL Number: MFCD00131507
Density: 0.930
Comments: Refractive Index: 1.4795
Flash Point (°F): >230
Product Comments: Stabilized with 0.5% lead naphthenate
ELINCS/EINECS Number: 232-278-6
Miscellaneous: *This chemical is in the EPA inventory under TSCA.*
Label Precautions: Harmful liquid
Reproductive hazard
Irritant
Target organ: nerve
Target organ: blood

Tung oil

ALDRICH

Certificate of Analysis

TEST	LOT {08228BN} RESULTS
Product Name	Tung oil
Product Number	440337
CAS Number	8001205
APPEARANCE	VISCOUS COLORLESS LIQUID
REFRACTIVE INDEX AT 20 DEG C	1.5198
INFRARED SPECTRUM	CONFORMS TO STRUCTURE.
LOSS ON DRYING	0.07% LOSS *
COLOR TEST	6.1 GARDNER COLOR *
ACID VALUE	1.71 *
IODINE VALUE	164.4 *
SAPONIFICATION VALUE	191.9 *
UNSAPONIFIABLE MATTER	0.44% *

* SUPPLIER INFORMATION

David Swessel

David Swessel, Supervisor
Quality Control

Aldrich Product

Product Number: 440337
Product Name: Tung oil
Synonyms: (China wood oil)
CAS: 8001-20-5
MDL Number: MFCD00217929
Density: 0.937
Comments: Refractive Index: 1.5200
Flash Point (°F): >230
Product Comments: Typically *ca.* 80% ester of eleostearic acid and *ca.* 20% esters of linolenic, 9,12-linoleic, oleic, stearic and palmitic acids
Acid value (mg KOH/g); <5; Saponification value (mg KOH/g); 189-195; Iodine value; >220; Gel time; 12 minutes
Applications: Air drying coatings and varnishes.
ELINCS/EINECS Number: 232-272-3
Reference to Aldrich Library of *FT-IR* Spectra: 2(1),998C
Miscellaneous: *This chemical is in the EPA inventory under TSCA.*
Label Precautions: Air sensitive

Boiled linseed oil

SIGMA

Certificate of Analysis

TEST	LOT {046H0993} RESULTS
Product Name	Linseed oil, boiled
Product Number	L3026
CAS Number	8001261
APPEARANCE	SLIGHTLY HAZY YELLOW-BROWN LIQUID
SOLUBILITY	CLEAR YELLOW-GREEN SOLUTION AT 0.1 ML PLUS 5 ML OF CHLOROFORM
REFRACTIVE INDEX	1.4741 (AT 40 DEG C)
DENSITY	0.930 G/ML (AT 25 DEG C)
FATTY ACIDS BY GAS CHROMATOGRAPHY (FROM GLYCERIDES)	52.0% LINOLENIC ACID 13.5% LINOLEIC ACID 22.3% OLEIC ACID
QC ACCEPTANCE DATE	AUGUST 1996

Jeff Heiland, Manager
Analytical Services

Appendix Nr. 1

Boiled linseed oil

Sigma Product

Product Number: L3026
Product Name: Linseed oil, boiled
CAS: 8001-26-1
MDL Number: MFCD00131507
Comments: Stabilizer/extender: Stabilized with 1-5% calcium alkanoate and 1% manganese naphthenate.
Storage Temp: Store at RT.
R&S: R: 20/21/22,36/37/38,43, S: 26,36

Linoliefernis

Bioteknologisk Institut

WOLF'S LINOLIE- OG TRÆTJÆREFABRIK APS
STEVNSVEJ 57A

4660 ST. HEDDINGE

Att.: Hans Wolf

Arkiv nr. 92699.1
Prøve B110160056

Kolding d. 17.10.2001
Side 1 af 1

A N A L Y S E C E R T I F I K A T

Analyse af	Olie
Modtaget	16.10.2001
Påbegyndt	16.10.2001
Prøvens mærke:	**Kogt linolie**

Analyse	Resultat	K_{95} 1)
Fedtsyrefordeling og pct. i fedt	**Resultat vedlagt.**	
AOAC 963.22 + 969.33 (17.ed.)		

*) K_{95} = 95% Konfidensinterval.

Med venlig hilsen
Bioteknologisk Institut

Jens Sondermark

Resultaterne må ikke gengives, undtagen i sin helhed, uden laboratoriets skriftlige godkendelse. Resultaterne gælder udelukkende for den analyserede prøve.

Alle opgaver udføres i henhold til Bioteknologisk Institut's akkreditering/autorisation og almindelige vilkår.

Høibergsvej 10
Postboks 818
DK-6000 Kolding
Tlf.: 75 52 04 33
Fax: 75 52 99 89

Bank: Den Danske Bank
Konto nr. 1771 3711 143978

SE nr.: DK 15231878

Appendix Nr. 1

Linoliefernis

Bioteknologisk Institut

Fedtsyresammensætning 17-10 2001

Prøvemærke: Kogt linolie

Lab. nummer: B110160056

% Fedtsyre af fedt	86 %
Sum af fedtsyrer C:20 og derover	0.7 %
Sum af ω-3 fedtsyrer	54.5 %
Sum af ω-6 fedtsyrer	15.1 %
Sum af mættede fedtsyrer	9.8 %
Sum af enkeltumættede fedtsyrer	19.6 %
Sum af flerumættede fedtsyrer	69.7 %

Fedtsyre	Navn	Rel. fordeling
C-14:0	Myristinsyre	0.1 %
C-16:0	Palmitinsyre	5.9 %
C-16:1 ω 7	Palmitoleinsyre	0.1 %
C-17:0		0.1 %
C-18:0	Stearinsyre	3.4 %
C-18:1 ω 9	Oliesyre	18.5 %
C-18:1 ω 7	Vaccensyre	0.8 %
C-18:2 ω 7.13		0.1 %
C-18:2 ω 6	Linolsyre	14.9 %
C-18:3 ω 3	Linolensyre	54.5 %
C-20:0	Arachinsyre	0.2 %
C-20:1 ω 11+20:1 ω 9	Eicosensyre	0.2 %
C-20:2 ω 6		0.1 %
C-20:3 ω 6	Homogamma Linolensyre	0.1 %
C-22:0	Behensyre	0.1 %
Uidentificerede fedtsyrer		0.9 %

Med venlig hilsen

Jens Søndermark

Appendix Nr. 1

Appendix Nr 2

Method of Wijs' index (or iodine index) measurement

DÉTERMINATION DE L'INDICE D'IODE : I_I
(ou indice de Wijs)

- Indice d'iode I_i : masse de diiode (I_2) en g qui se fixe par addition sur 100 g de lipide.

- $M(I_2) = 253{,}81\ g.mol^{-1}$

1. Protocole expérimental

1.1 Réaction d'addition

Dans une fiole d'Erlenmeyer bouchant émeri placer :

- m # 0,2 g exactement pesé de lipide à analyser que l'on entraînera avec 20 mL de chloroforme (le cyclohexane, non toxique, peut être utilisé à la place du chloroforme).
- 20 mL de réactif de Wijs

Agiter et maintenir à l'obscurité au moins 30 min en agitant de temps en temps.

1.2 Dosage

Ajouter 20 mL de solution de KI à 10% et 100 mL d'eau distillée.
Réduire l'excès d'iode dans la solution aqueuse par une solution de thiosulfate de sodium en *agitant fortement* la fiole afin d'extraire tout l'iode de la phase inférieure organique (rouge-violette).
Soit **V_i mL le volume versé**.

1.3 Témoins (réaliser simultanément 1 essai 1 témoin)

Opérer de même sur 20 mL de solvant (chloroforme ou cyclohexane) et 20 mL de réactif de Wijs puis doser l'iode comme précédemment.
Soit **V'_i mL le volume versé**.

1.4 Résultats

	Thiosulfate C =		
Corps gras (g)	Témoin (mL)	essai (mL)	Indice d'iode
m_1 =	V'_{i1} =	V_{i1} =	I_{i1} =

2. Calcul de l'indice d'iode

2.1 *Définition :*

Indice d'iode I_i : *masse de diiode en g qui se fixe par addition sur 100 g de lipide.*

2.2 *Équations du dosage*

$$R—CH_2=CH_2—R' \quad + \quad I_2 \quad \underset{\text{Excès}}{\leftrightarrow} \quad R—CHI—CHI—R'$$

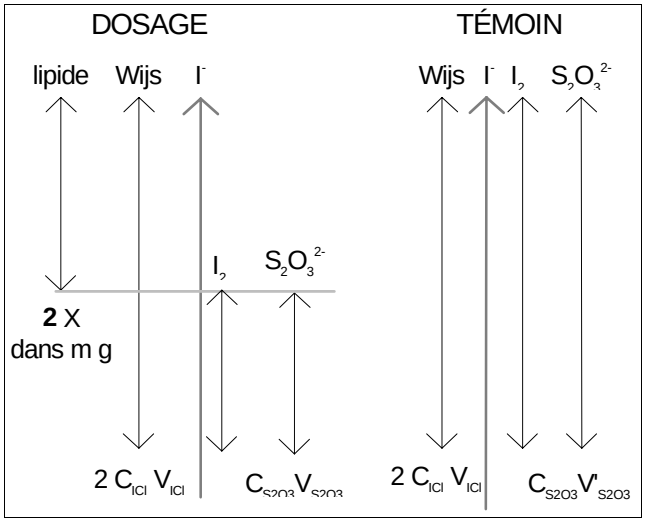
Réaction équilibrée, déplacée par un excès de I_2
Réaction lente (2 h), accélérée par catalyseur (ions Hg^{2+})
Réactions parasites de substitution radicalaires (utilisation de solvants halogénés pour limiter les substitutions, obscurité car la lumière favorise les substitutions et remplacement de I_2 par I→-Cl polarisé qui favorise l'addition).

2.3 *Calculs*

X : nombre de moles d'ICl (ou I_2) fixées par m g de lipide.
Pour le témoin : $C_{S2O3} . V'_{S2O3} = 2 C_{ICl} . V_{ICl}$
Pour le dosage : $2 X + C_{S2O3} . V_{S2O3} = 2 C_{ICl} . V_{ICl}$

D'où : $2 X = C_{S2O3} (V'_{S2O3} - V_{S2O3})$

$$I_i = \frac{C_{S2O3}}{2} . (V' - V) . M_{I2} . \frac{100}{m}$$



L'indice d'iode permet de calculer le nombre de doubles liaisons de l'acide gras (n_Δ)présent dans un triglycéride pur homogène :

nb de moles d'I_2 fixées par 100 g de lipide : $\frac{I_2}{M_{I_2}}$

pour 1 mole de triglycéride pesant $M_{triglyceride}$ g soit :

$$n_\Delta = \frac{I_2}{M_{I_2}} \times \frac{M_{triglyceride}}{100}$$

Appendix Nr 3

Standard Test Methods for Drying, Curing or Film Formation of Organic Coatings at Room Temperature

Designation: D 1640 – 95 (Reapproved 1999)

Standard Test Methods for Drying, Curing, or Film Formation of Organic Coatings at Room Temperature[1]

This standard is issued under the fixed designation D 1640; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the various stages and rates of film formation in the drying or curing of organic coatings normally used under conditions of ambient room temperature.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 202 Test Methods of Sampling and Testing Untreated Paper Used for Electrical Insulation[2]

D 823 Practices for Producing Films of Uniform Thickness of Paint, Varnish, and Related Products on Test Panels[3]

D 1005 Test Methods for Measurement of Dry-Film Thickness of Organic Coatings Using Micrometers[3]

D 2091 Test Method for Print Resistance of Lacquers[4]

2.2 *U.S. Government Standards:*

Fed. Spec. No. CCC-C-440, Cheesecloth[5]

Fed. Spec. No. CCC-C-419b, Type III, Army Duck[5]

2.3 *TAPPI Standards:*[6]

T 402 Standard Conditioning and Testing Atmospheres for Paper, Board, Pulp Handsheets, and Related Products

3. Significance and Use

3.1 These test methods are used to determine the various stages and rates of drying, curing, and film formation of organic coatings for the purpose of comparing types of coatings or ingredient changes, or both. This is significant in the development of organic coatings for various end uses and also for production quality control.

4. Coatings and Recommended Film Thicknesses

4.1 Whenever tests are to be performed on coatings not listed in Table 1, there should be a prior agreement between the purchaser and seller as to the substrate, film thickness, and application method for testing the specific coating involved.

5. Test Conditions

5.1 Conduct all drying tests in a well-ventilated room or chamber, free from direct drafts (Note 1), dust, products of combustion, laboratory fumes and under diffused light (see 5.4). Make all measurements at a temperature of 23 ± 2°C and 50 ± 5 % relative humidity with the coated panels in a horizontal position while drying.

NOTE 1—A device to equalize air change conditions has been developed by F. Scofield.[7] Relative humidity should be controlled for moisture-cured and two-package urethane coatings, since their cure is greatly affected by the existing moisture conditions.

5.2 Tests should be carried out at practical viscosities at which films can be applied to the proper film thickness with resultant good flow and leveling properties. In the absence of any specific material specification, instructions for preparation of the film should be determined and agreed upon between the purchaser and the seller.

5.3 Films to be tested should have practical thicknesses commensurate with performance characteristics expected under actual usage for the type under test. All testing should be done within an area, any point of which is not less than ½ in. (15 mm) from the film edge.

5.4 *Light Conditions*—Illumination of the films during the entire drying test period should be about 25 ft-candles (270 lx)

[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.23 on Physical Properties of Applied Paint Film.

Current edition approved Feb. 15, 1995. Published April 1995. Originally published as D 1640 – 59 T. Last previous edition D 1640 – 83 (1989) ε1.

[2] *Annual Book of ASTM Standards*, Vol 10.01.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 06.02.

[5] Available from Standardization Documents Order Desk, Bldg. 4 Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, Attn: NPODS.

[6] Available from Technical Association of the Pulp and Paper Industry, Technology Park, P.O. Box 105113, Atlanta, GA 30348.

[7] Gardner and Sward, *Paint Testing Manual*, ASTM STP 500, ASTM, 13th edition, 1972, p. 269.

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TABLE 1 Recommended Film Thickness of Materials to be Tested[A]

Material	Dry Film Thickness
Drying oils	1.25 ± 0.25 mil (32 ± 6 μm)[B]
Varnishes	1 ± 0.1 mil (25 ± 2 μm) (See 7.4.2)
Lacquers	0.5 ± 0.1 mil (12.5 ± 2 μm) (See 7.5.2)
Resin solutions	0.5 ± 0.1 mil (12.5 ± 2 μm)
Enamels	1.5 ± 0.25 mil (36.5 ± 6 μm)
Oil paints	1.8 ± 0.2 mil (45 ± 2.5 μm) (See 6.1.2)
Water paints	1 ± 0.1 mil (25 ± 2 μm)

[A]This table is a general guide to be used when nothing more specific is agreed upon between the purchaser and the seller.

[B]See 6.1.2 and 7.5.1. Add driers a minimum of 24 h before test.

from normal laboratory or sky sources, never from direct sunlight or other sources high in nonvisible radiant energy.

6. Preparation of Test Specimens

6.1 Carry out all tests as described in 6.1.1, 6.1.2 and 6.1.3, unless otherwise noted.

6.1.1 All test specimens shall be prepared and tested by one operator properly skilled in the methods to be used. Apply the specimens in duplicate at a time arranged so that examination intervals will fall within the normal working hours of the operator.

6.1.2 Apply the materials to be tested on clean glass panels or other specific substrate of suitable dimensions agreed upon between the purchaser and the seller. Ground-glass plates are more suitable for certain types of coatings that tend to crawl, such as low-viscosity drying oils. Suitable plates can be prepared by roughening the surface of polished glass by grinding a paste of silicon carbide (grit 1-F) and water between two glass plates.

6.1.3 The test films perferably shall be cast with a doctor blade having a clearance sufficient to give the recommended dry film thickness indicated in Table 1. When a suitable doctor blade is not available, or it has been agreed upon to apply the film in some other manner, the various conventional and automatic methods of spray, dip, flow, and brush application may be used, provided dry film thicknesses conform to the requirements given in Table 1. See Practices D 823 for a description of the spray and dip methods of application.

6.1.4 Measure the dry film thickness of test films with the proper film thickness gage. This shall be a micrometer, dial comparator, or dial indicator as described in Test Methods D 1005. When plates of small area are used, measurement of dry film thickness can be made by weighing plates before and after coating and calculating from plate area and coating solids.

7. Procedure

7.1 When test methods or end points other than those listed in 7.2-7.9 are used, there shall be a prior agreement between the purchaser and the seller.

7.2 *Set-To-Touch Time*—To determine set-to-touch time, lightly touch the test film with the tip of a clean finger and immediately place the fingertip against a piece of clean, clear glass. Observe if any of the coating is transferred to the glass. For the purpose of this test, the pressure of the fingertip against the coating shall not be greater than that required to transfer a spot of the coating from ⅛ to 3/16 in. (3 to 5 mm) in cross section. The film is set-to-touch when it still shows a tacky condition, but none of it adheres to the finger.

7.3 *Dust-Free Times*:

7.3.1 *Cotton Fiber Test Method*—Separate a number of individual fibers from a mass of absorbent cotton with the aid of tweezers. At regular drying intervals, drop several of the cotton fibers from a height of 1 in. (25 mm) onto a marked section of the film. The film is considered to have dried dust free when the cotton fibers can be removed by blowing lightly over the surface of the film.

7.4 *Tack-Free Times*:

7.4.1 *Paper Test Method*:

7.4.1.1 *Test Paper*—The test paper shall be K-4 Power Cable Paper[8] that when conditioned in accordance with the TAPPI Standard Method T 402, conforms to the following requirements:

Basis weight (24 by 36/500), lb	90 ± 5
Thickness, mils (μm)	6.65 (17)
Air resistance (s/100 cm^2/$in.^2$)	350
Coefficient of static friction[A]	0.5
Friction angle, °	22
Tensile strength, machine direction/cross direction	119/32
Tear, machine direction/cross direction	180/250
Elongation, machine direction/cross direction, %	3.0/7.0
pH of water extract	7.4
Ash content, max, %	0.6

[A]All tests except this one shall be run in accordance with Test Method D 202. All values for properties are typical values and not specification limits.

7.4.1.2 Lay a 2 by 3-in. (50 by 75-mm) piece of the special test paper on the film and place upon it a steel cylinder 2 in. in diameter, and of such weight 6.28 lb, (2.85 kg), as to produce a pressure of 2 psi (13.8 kPa). At the end of 5 s, remove the cylinder and invert the test panel. The film is considered free from after-tack when the paper drops off of the test film within 10 s.

7.4.2 A variation of the test method described in 7.4.1 using the same test paper can be used to test the tack-free time of insulating varnishes. In this method the piece of paper shall be 1½ in. (40 mm) in width and 6 in. (150 mm) in length. The varnish is considered tack-free when this strip of paper does not adhere to it when it is pressed on the surface of the varnish for 1 min by a cylindrical 1-lb (450-g) weight, 1 in. (25 mm) in diameter. In this test, apply the paper in the vicinity of the center of the specimen at right angles to the length of the coated specimen.

7.4.3 *Mechanical Test Method (Tack Tester[9])*—The tack tester to be used in this method comprises essentially a base or surface-contacting portion 1-in. (25-mm) square and a counter-balancing portion 1 by 2 in. (25 by 50 mm) in area. Both portions are made up from a continuous metal strip 0.016 to 0.018 in. (0.41 to 0.46 mm) in thickness. To prepare the apparatus for use (see 7.4.3.1), fit the base with several thicknesses of masking tape and paper strips to provide a

[8] The sole source of supply of paper Grade R20–34, meeting these requirements known to the committee at this time is the Crocker Technical Papers, Inc., 431 Westminster St., Fitchburg, MA 01420. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[9] The standard tack tester is fully described in the U. S. Patent 2,406,989, Sept. 3, 1946.

means of attaching the aluminum foil and so adjust the angle of the 1 by 2-in. counter-balancing strip so that a weight of 5 g placed in the geometric center of the base portion is just sufficient to overcome the unbalanced force.

7.4.3.1 The tester is prepared for use by carrying out the following steps in sequence:

(1) Wrap the metal base with three thicknesses of masking tape, sticky side out,

(2) Cover the outer layer with a good grade of paper, except for two exposed strips, equally spaced, about ¼by 1 in. (6.4 by 25 mm) in area on the top of the tester, and

(3) Cover the paper on the contact side of the base with one thickness of pressure-sensitive cellulose tape previously fixed to the metal base of the tester. The cellulose tape serves two purposes:

First, to pull the layers of masking tape firmly against the front of the metal base, and

Second, to provide a smooth surface for the foil. Attach the aluminum foil to the base of the tester by pressing gently but firmly a 1 by 2-in. (25 by 50-mm) piece of foil, 0.0005 in. (13 μm) in thickness against one of the ¼ by 1-in. (6.4 by 25-mm) exposed strips of masking tape on the top surface of the base. Wrap the foil tightly and smoothly around the base, exposing the shiny side, and finally press the outer end gently against the remaining exposed strip of masking tape. When it finally becomes necessary to replace wrinkled or soiled aluminum foil, the ends are easily removed from the masking tape by exerting a slow, even, upward pull sufficient to overcome the tack of the tape without tearing the foil.

7.4.3.2 A film is considered to have dried tack-free when the tack tester tips over immediately on removing a 300-g weight allowed to act for 5 s on the counter-weighted metal square base fitted with masking tape and aluminum foil.

7.5 *Dry-To-Touch Time*:

7.5.1 *Drying Oils*—Continue testing after the set-to-touch time has been observed. The film is considered dry when it no longer adheres to the finger and does not rub up appreciably when the finger is lightly rubbed across the surface.

7.5.2 *Lacquers (and Sealers)*—Touch the film lightly at varying intervals of time. The film is considered dry when no pronounced marks are left by the finger touching the film in the same area on each observation. Test sealers on wood or other porous substrates as agreed upon between the purchaser and the seller.

7.6 *Dry-Hard Time*:

7.6.1 With the end of the thumb resting on the test film and the forefinger supporting the test panel, exert a maximum downward pressure (without twisting) of the thumb on the film. Lightly polish the contacted area with a soft cloth. The film is considered dry-hard when any mark left by the thumb is completely removed by the polishing operation.

7.7 *Dry-Through (or Dry-To-Handle) Time*:

7.7.1 Place the test panel in a horizontal position at a height such that when the thumb is placed on the film, the arm of the operator is in a vertical line from the wrist to the shoulder. Bear down on the film with the thumb, exerting the maximum pressure of the arm, at the same time turning the thumb through an angle of 90° in the plane of the film. The film is considered dry-through or dry-to-handle when there is no loosening, detachment, wrinkling, or other evidence of distortion of the film.

7.8 *Dry-To-Recoat*:

7.8.1 A film is considered dry for recoating when a second coat or specified topcoat can be applied without the development of any film irregularities such as lifting or loss of adhesion of the first coat, and the dry time of the second coat does not exceed the maximum specified (if any) for the first coat.

7.9 *Print-Free Time*:

NOTE 2—This procedure is similar to Test Method D 2091, except that the time to reach the print-free condition is determined, while Test Method D 2091 is used to evaluate whether a film is print free at a specified time.

7.9.1 *Test Panels*—Apply the material under test to clean plane panels, at least 3 by 6 in. (75 by 150 mm) in size, made of wood, metal, glass, plastic or other material as agreed upon between the purchaser and the seller.

7.9.2 *Imprinting Fabric*—Eight-ounce Army duck conforming to Type III of U.S. Fed. Spec. No. CCC-C-419b or cheesecloth conforming to Fed. Spec. No. CCC-C-440.

7.9.2.1 A pad should be used with the cheesecloth only, made of nonwoven felt cloth at least 0.05 in. (1.3 mm) thick, weighing 7 oz/yd ^{2} (0.24 kg/m^2) and larger than the plane end of the weight.

7.9.3 *Weights*—Consisting of metal cylinders not less than 2 in. (50 mm) in diameter with plane ends perpendicular to the axis and of a length to give a pressure of ½ or 1 lb/in.^{2} (3.5 or 6.9 kPa).

7.9.4 *Procedure*:

7.9.4.1 Apply the test material to several of the specified or agreed-upon panels by a film applicator, or other specified method, as described in Practices D 823 in either single or multiple coats, as agreed upon between the purchaser and the seller. In the absence of a specified dry film thickness, the values listed in Table 1 should be used.

7.9.4.2 Allow the coated panels to dry under the conditions specified in Section 5, unless otherwise agreed. At appropriate intervals, starting shortly before the coating is expected to be print-free, carry out the print-free test as described in Test Method D 2091, comparing the appearance with the photographic standards appearing therein, until the test shows the coating to be print-free.

8. Frequency of Testing

8.1 It is suggested that test intervals be set at periods of approximately 10 % of the total test time. If frequency varies considerably from the 10 % interval or such time interval is impractical, the intervals used shall be reported.

9. Report

9.1 Reports of tests shall include all applicable conditions that deviated from the standards as outlined or special conditions or tests used and the results of the test.

10. Precision and Bias

10.1 Because of the subjective nature of the drying time tests, the agreement to be expected between laboratories

depends upon their understanding of the terms used, and is difficult to establish with certainty. Within any laboratory, the agreement depends upon the material being tested, some coatings being much sharper in their end point than others, but duplicate determinations should agree within 10 % of the time of drying.[10]

10.2 *Bias*—These test methods have no bias because the value for dry times are defined only in terms of these test methods.

11. Keywords

11.1 drying time

[10] See Prane, J. W., "A Latin Square Drying Time Study," *Paint Industry Magazine* (August 1961), for a study of precision of drying time measurements.

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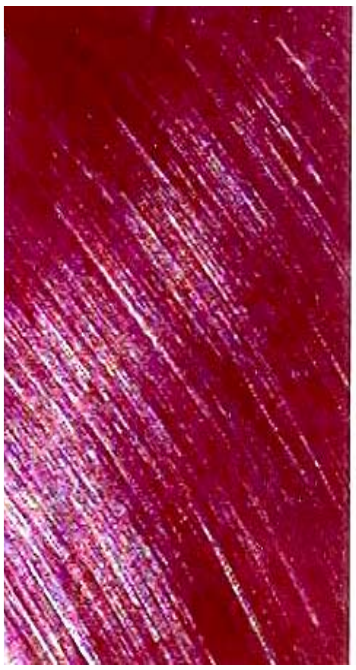
Appendix Nr 4

Photos from optical microscope

Surface quality of samples received from OPERA project.

Bad chamber

Good chamber



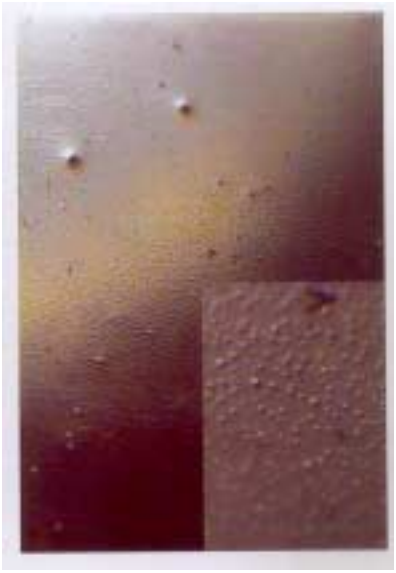
Appendix Nr. 4

Surface quality obtained with different oils, in laboratory.

Boiled linseed oil

Tung oil

Boiled linseed oil + Tung oil



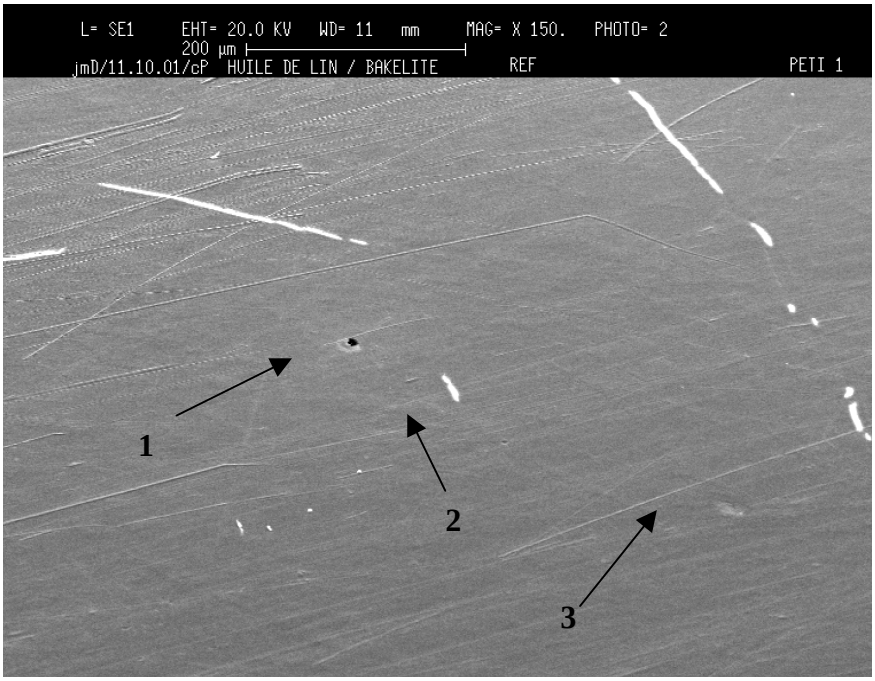
OPERA 1 oil

Linoliefernis

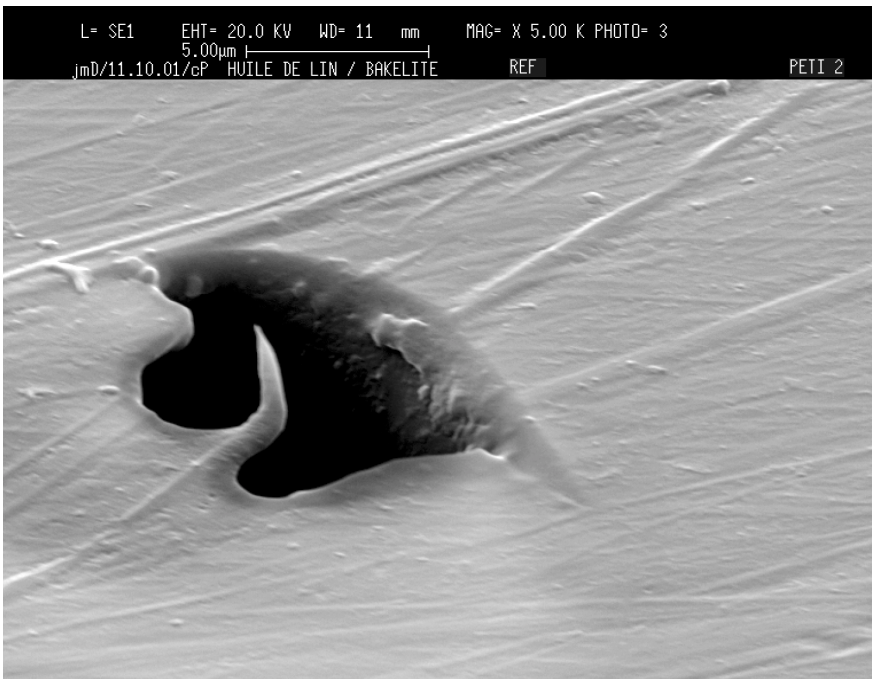
Appendix Nr 5

Photos from Scanning Electron Microscope (SEM)

Uncoated Bakelite sample

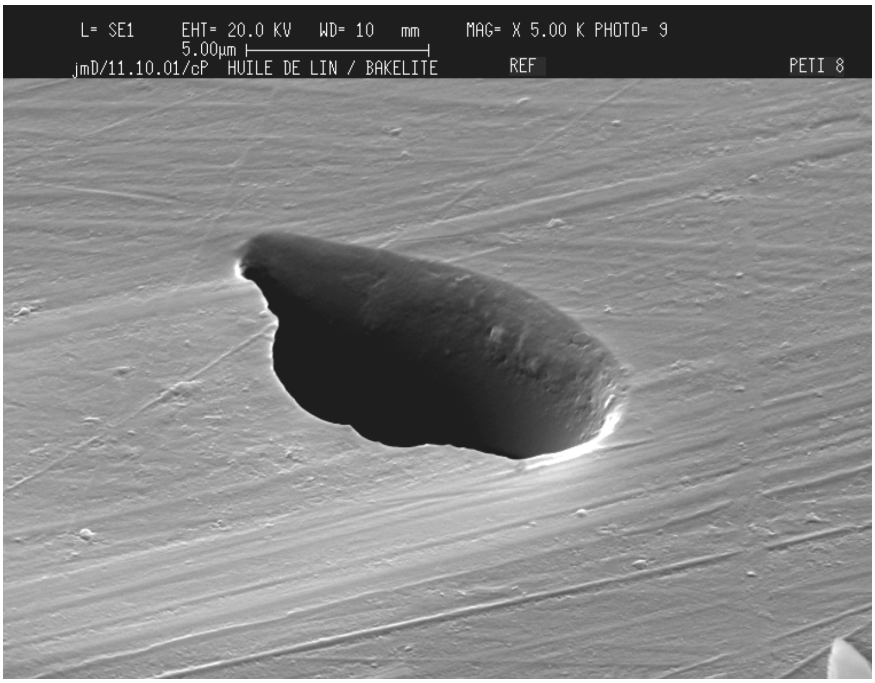


General surface observation (tilt 60°).

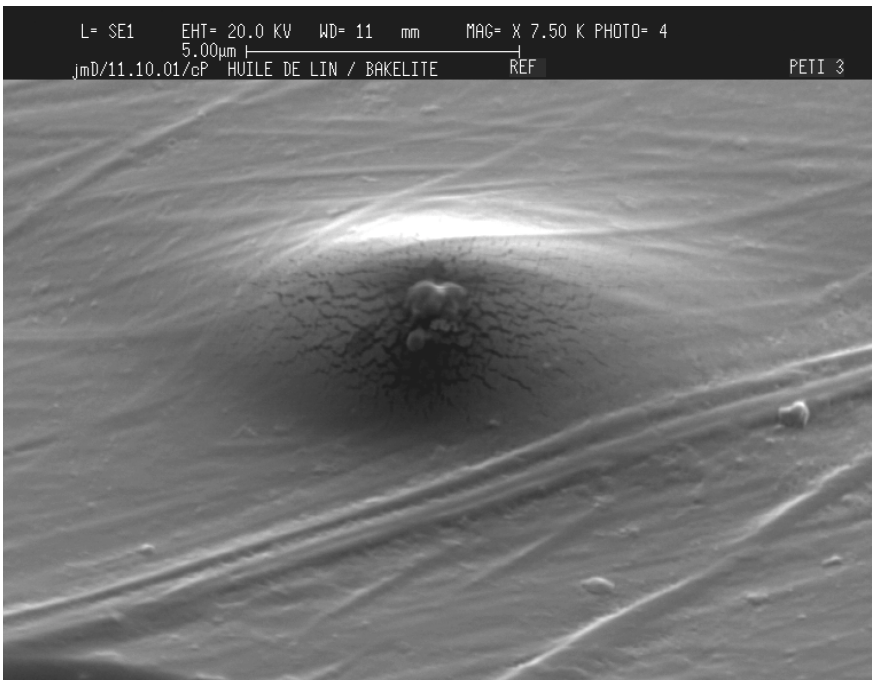


Detail of the flaw 1 : a preexisting surface hole.

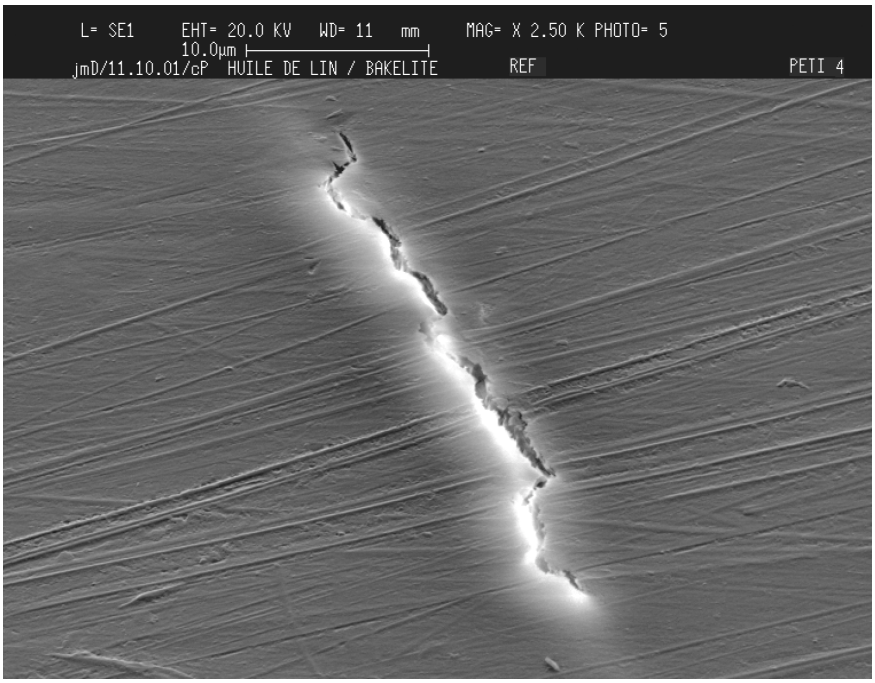
Uncoated Bakelite sample



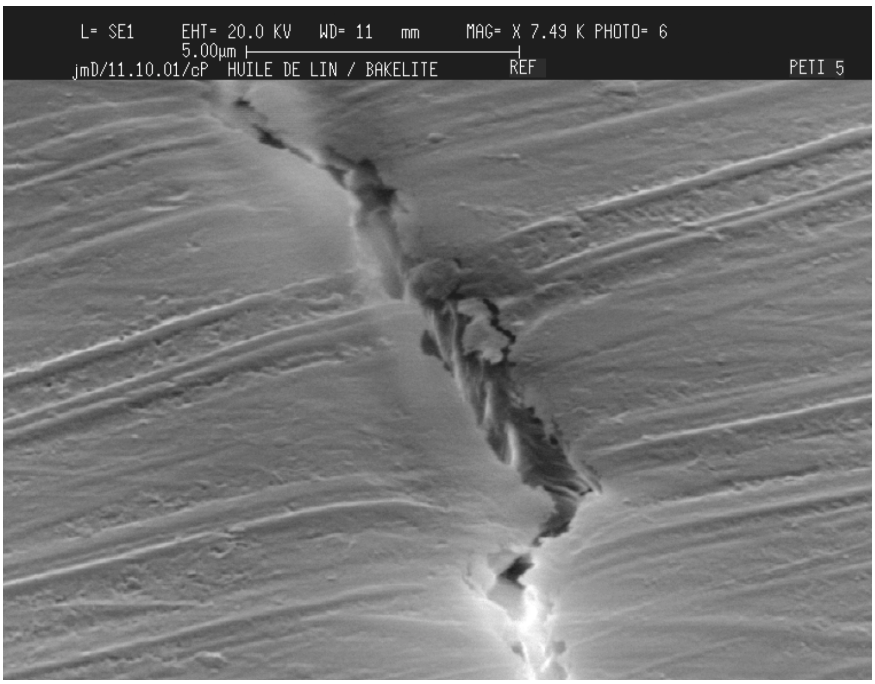
Other hole.



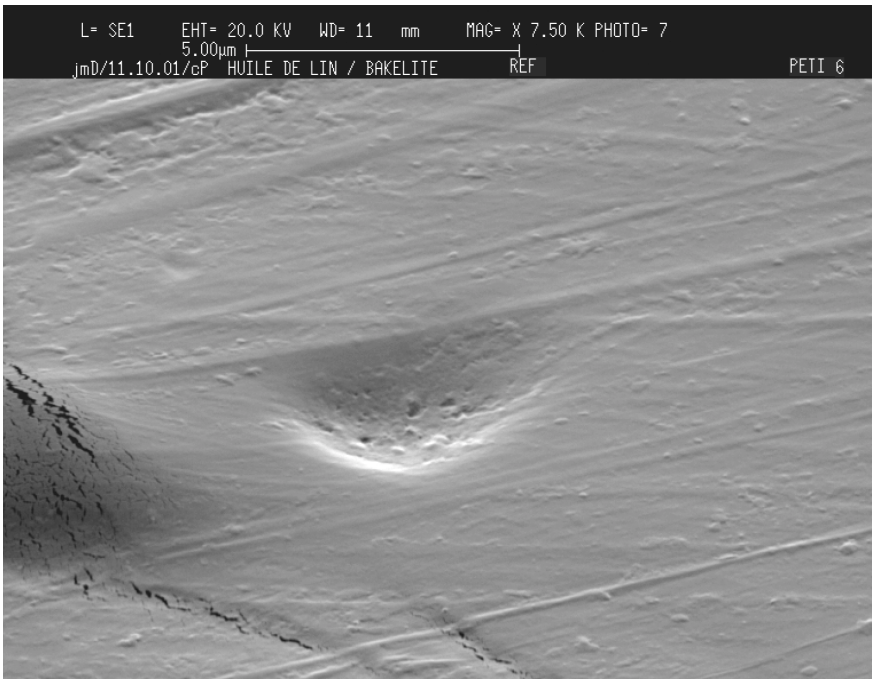
"Heights" are present.



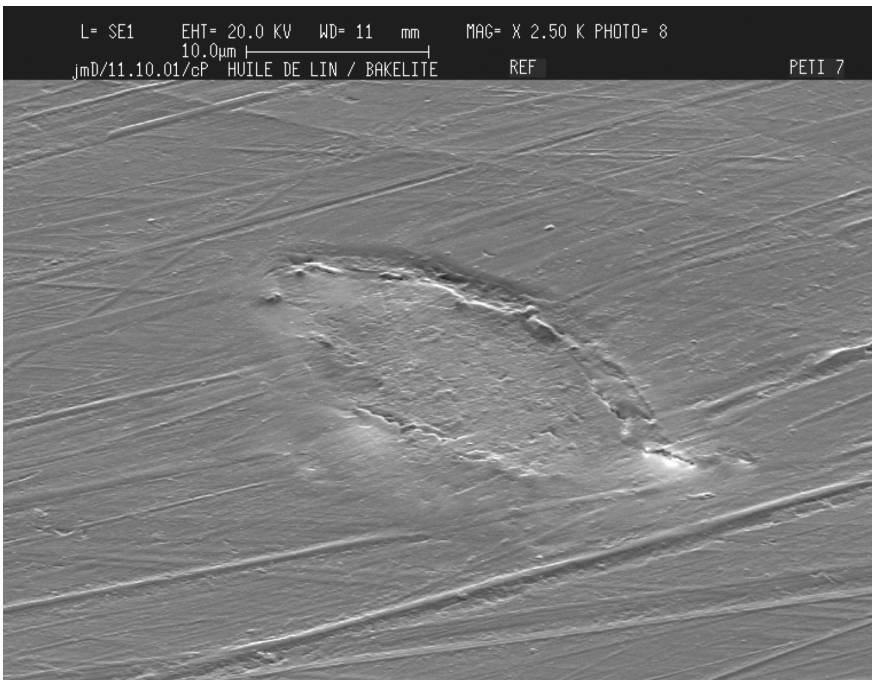
"Linear" cracks in the bakelite outer layer
(detail of the flaw 2 in the first picture).



The above crack magnified 2x.



A convex crater neighbour to a height.



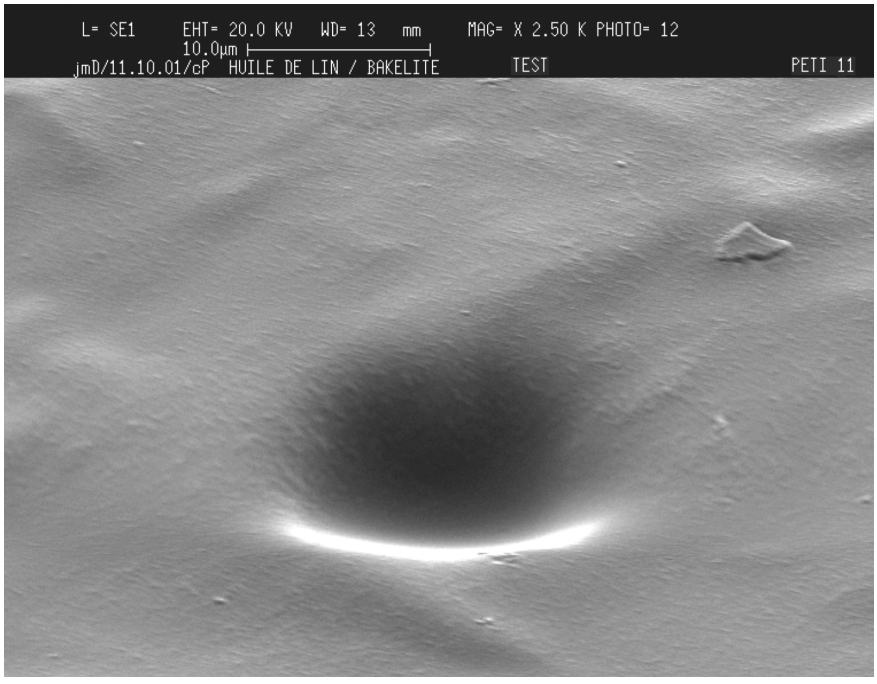
An oval surface non-uniformity : detail of flow 3 of the first photo.

Uncoated Bakelite sample

A surface non uniformity.

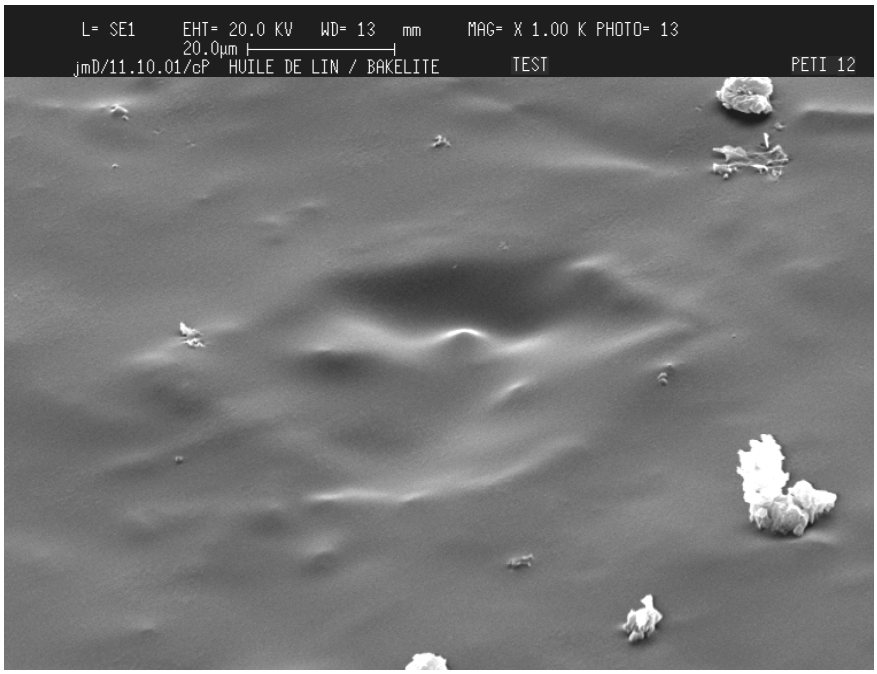
Sample of "bad" chamber received from OPERA

Non-uniform coating and dust particles present.

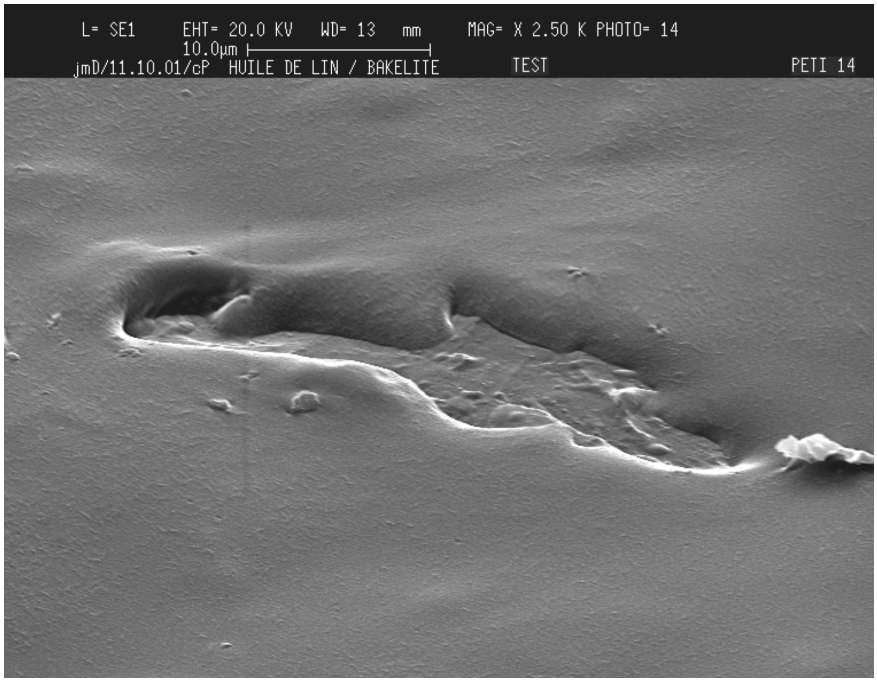


A hole silted-up by the coating layer.

Sample of "bad" chamber received from OPERA

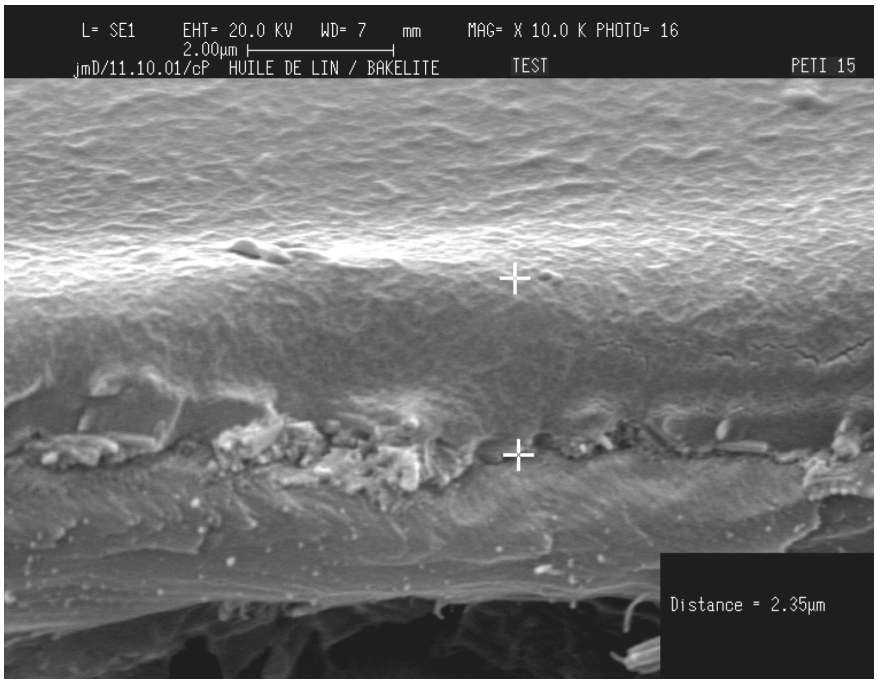


A Bakelite surface non-uniformity covered by the polymerised linseed oil.



Bad surface wettability (thickness of the layer = 2 μm).

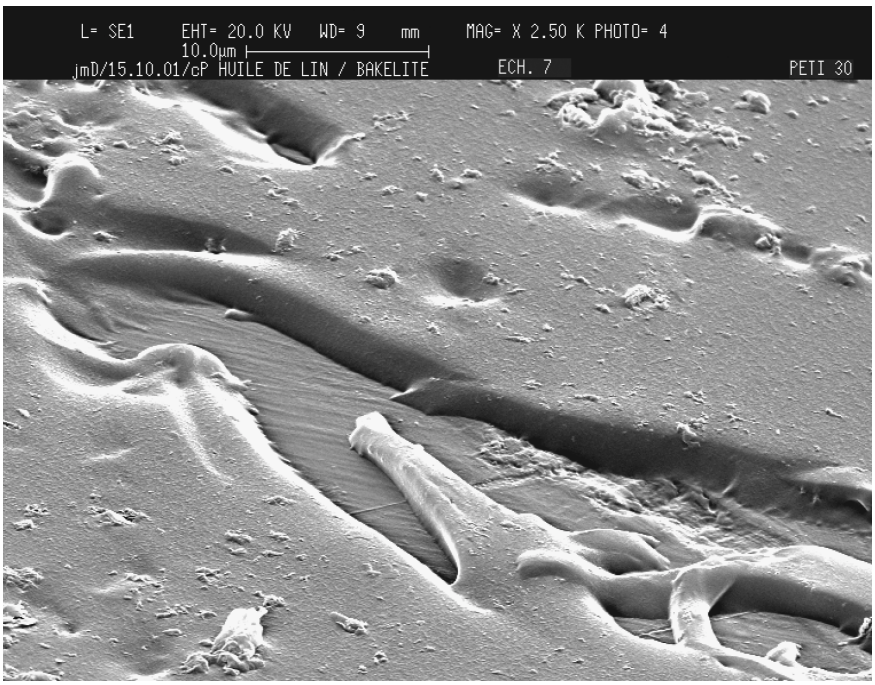
Sample of "bad" chamber received from OPERA



The measurement of the coating layer thickness near a substrate fracture has an estimated thickness of 2.3 µm.

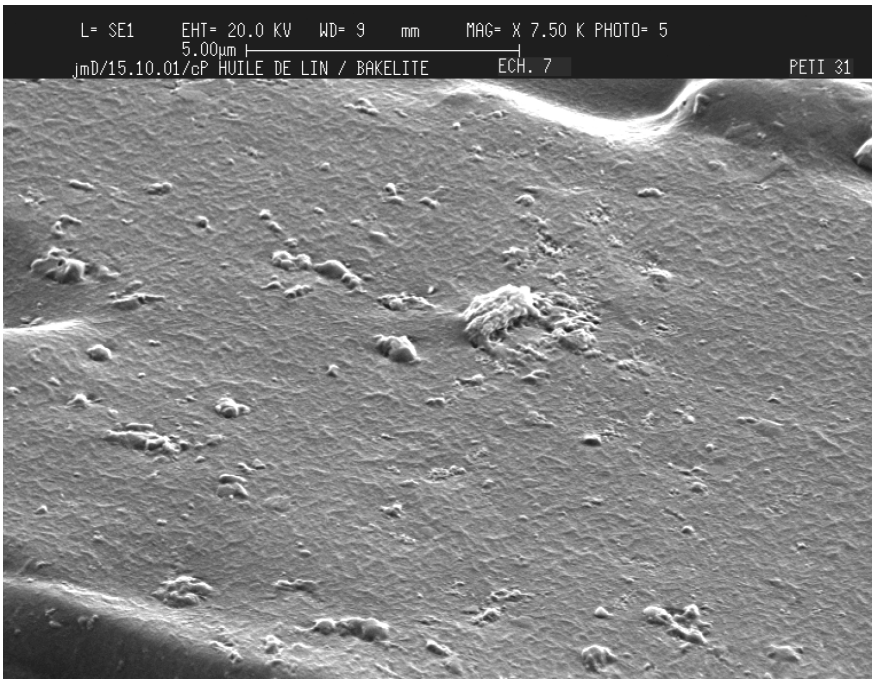
Sample of "good" chamber received from OPERA

On the sample surface many small areas are not coated



Detail of the above photo. The coating layer has an estimated thickness of 2.5 µm.

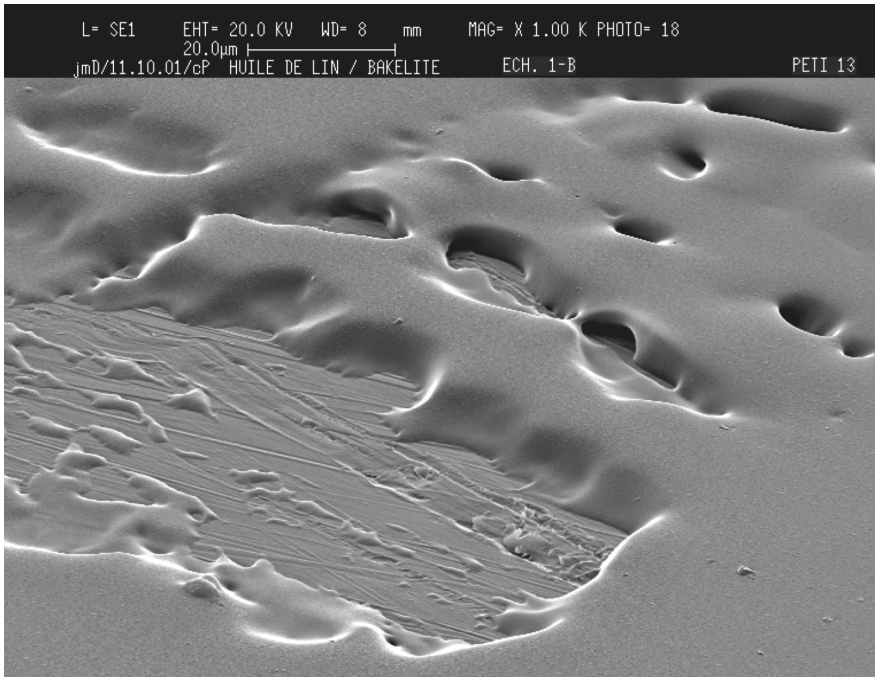
Sample of "good" chamber received from OPERA



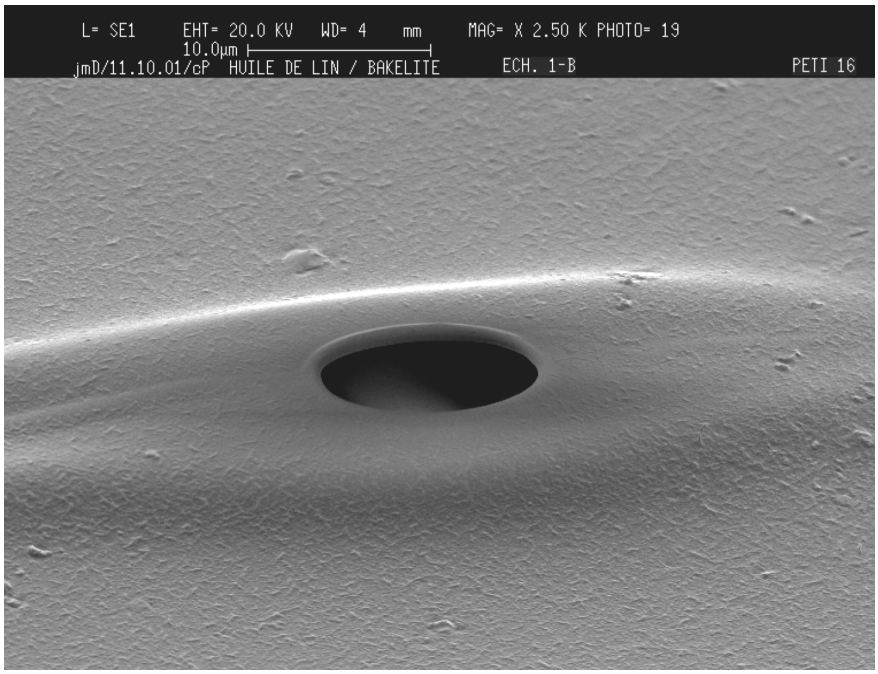
Inclusions are traped in the coating layer.

Sample n° 1: OPERA oil 30 / 70, filtered

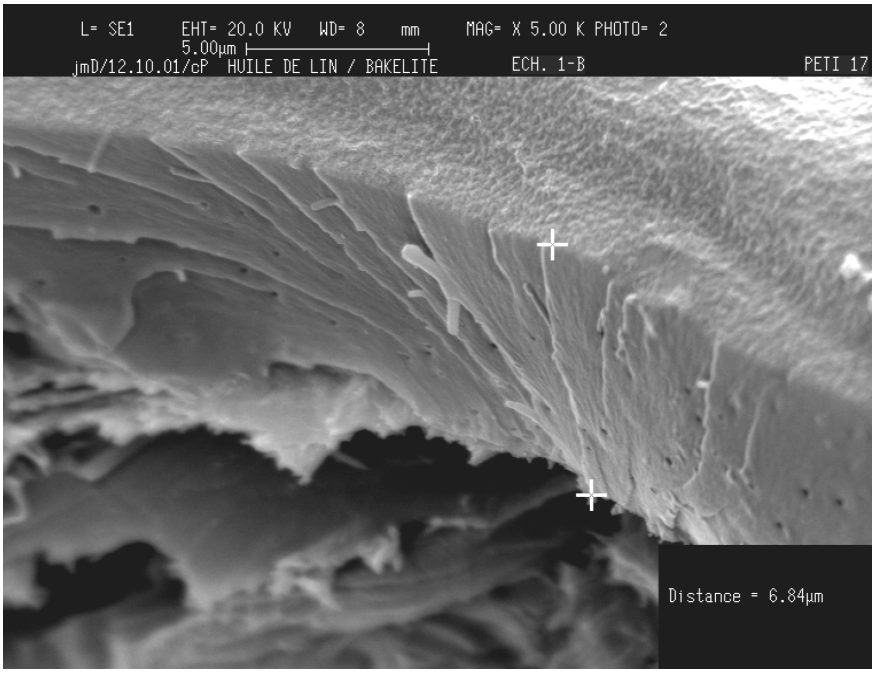
The coating layer is clean and smooth; bad wettability for certain areas.



Detail of the above photo. Thickness = 5.5 µm.

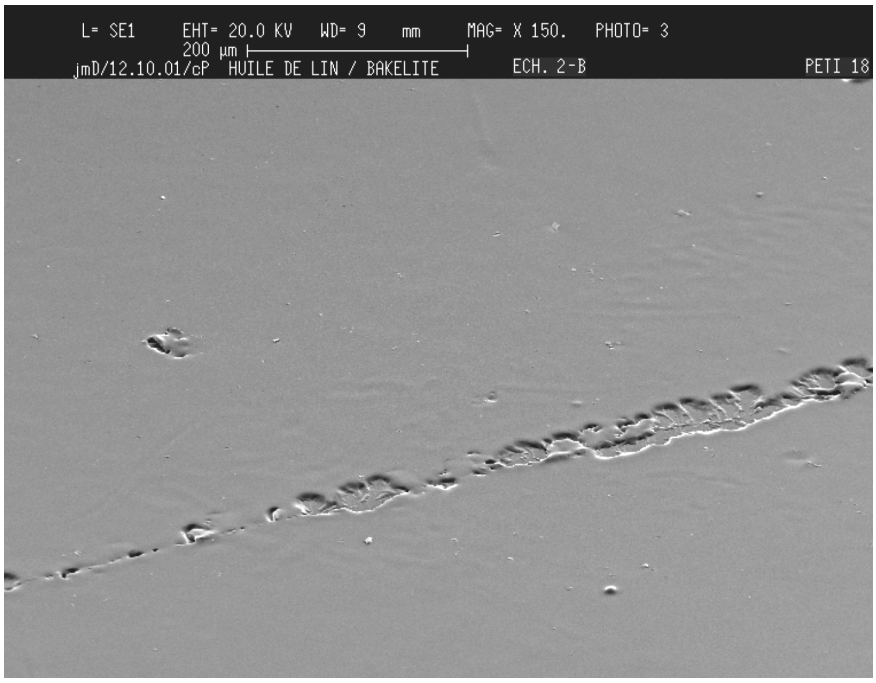


A hole apeared as a results of the surface outgassing.

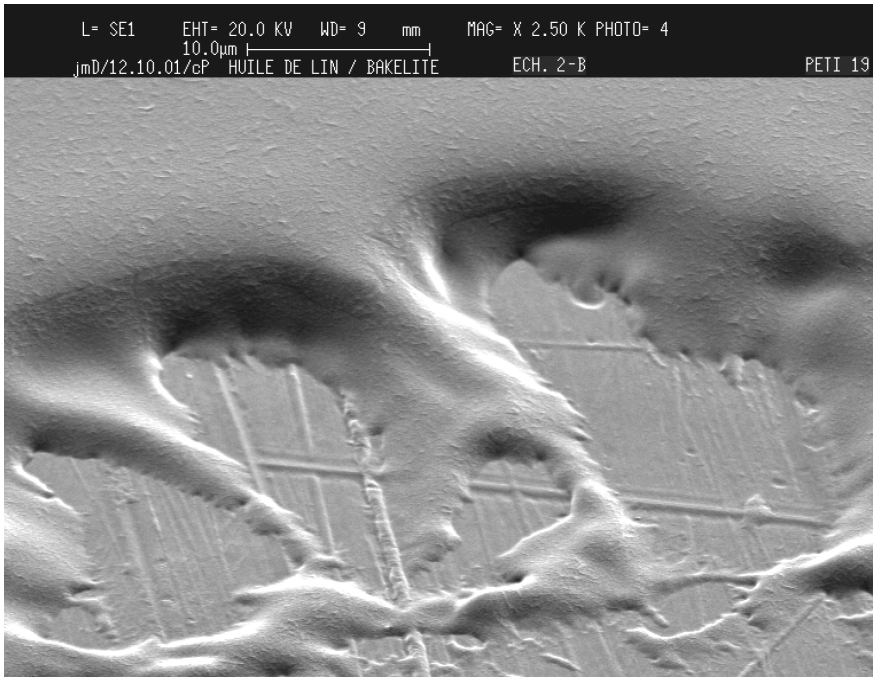


Thickness measurement = 6.8 µm

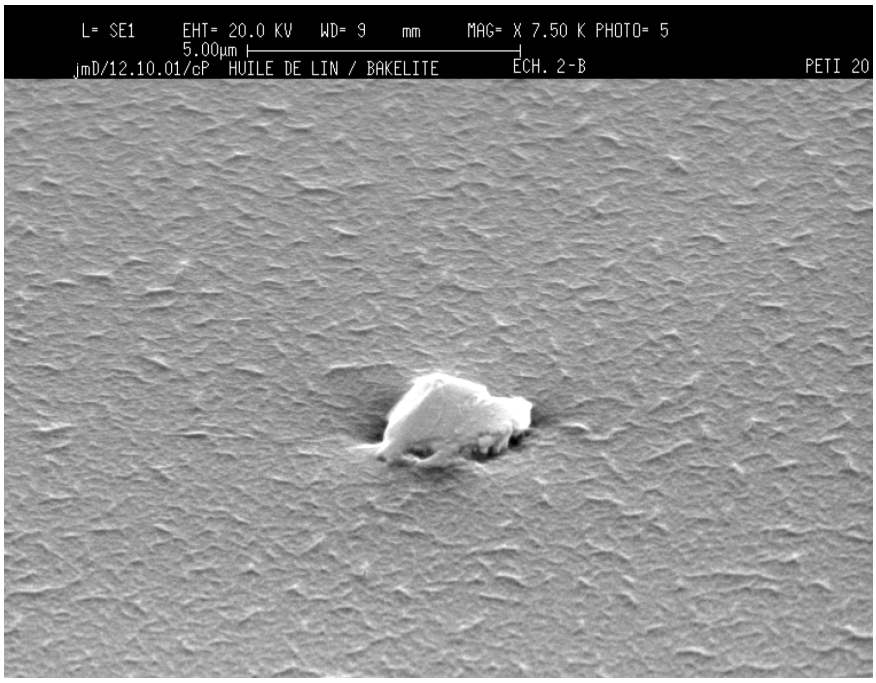
Sample n° 2: OPERA oil 15 / 85, filtered



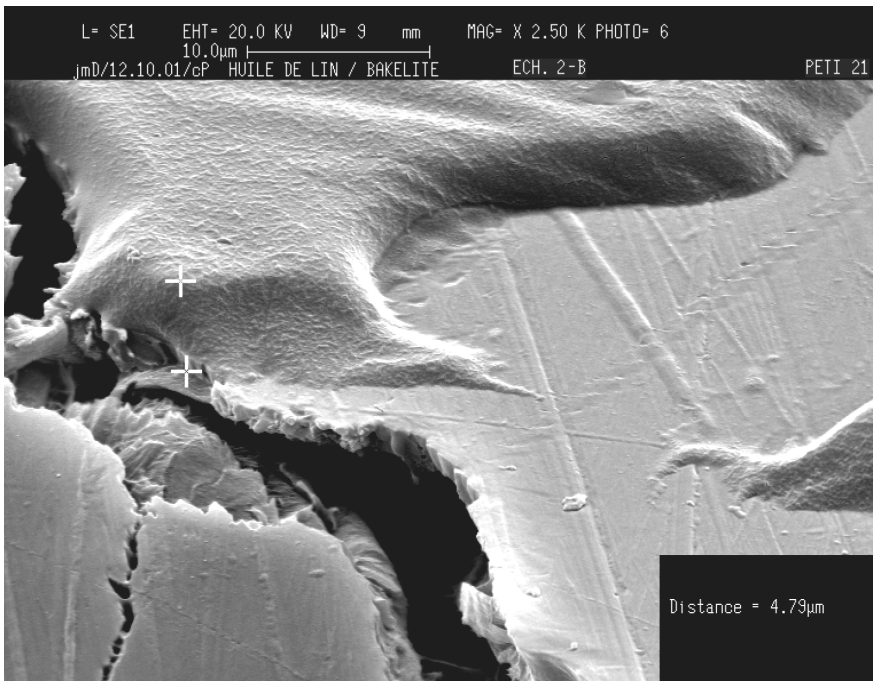
General surface observation. Tilt 60°. Some uncoated areas and inclusions.



Detail of an unwetted area (thickness= 5 µm).

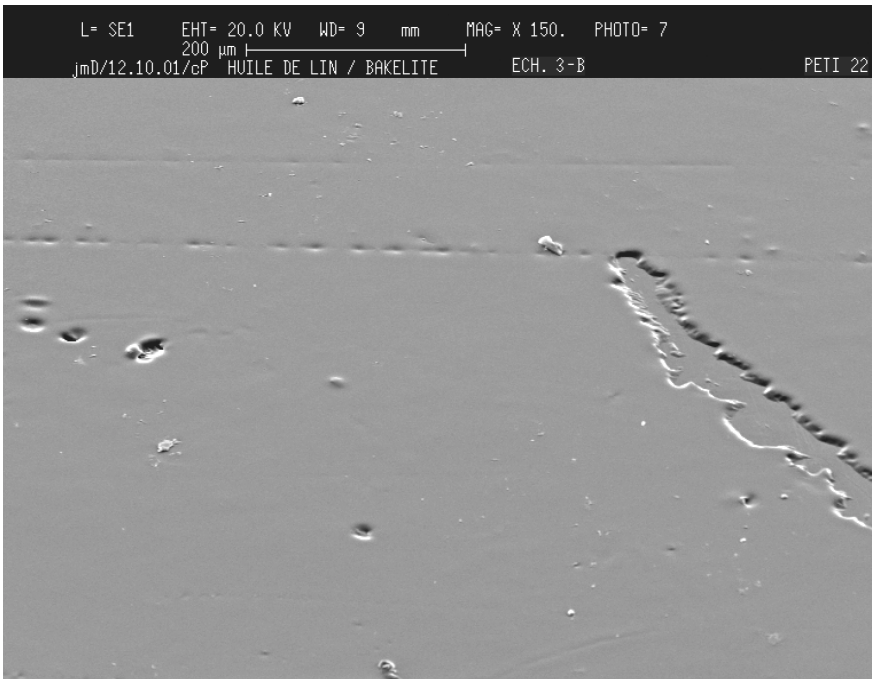


Detail of an inclusion; specific appearance of the surface layer presenting an uniform roughness.

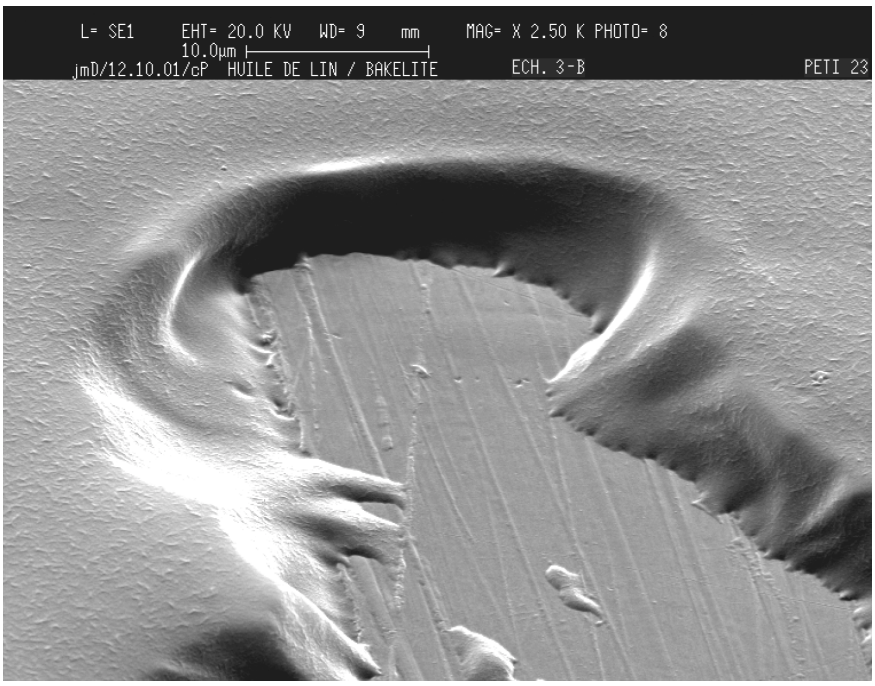


The thickness of the coating layer = 4.8 µm

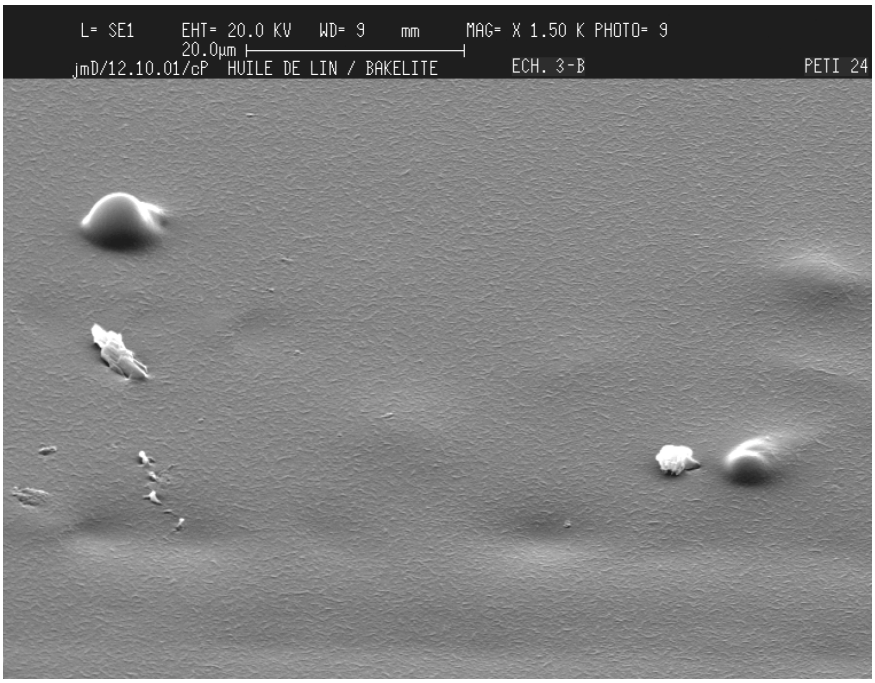
Sample n° 3: boiled linseed oil 30 / 70, filtered



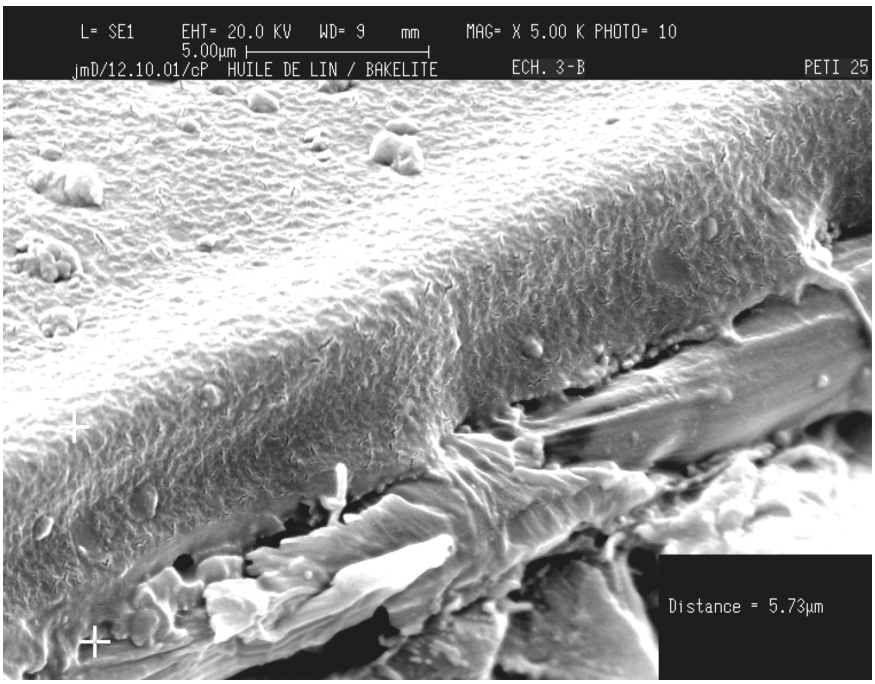
The surface is smooth. Some small areas are not entirely covered. Dust particules are present.



Detail of the above photo.
The thickness of the coating layer = 5.8 µm

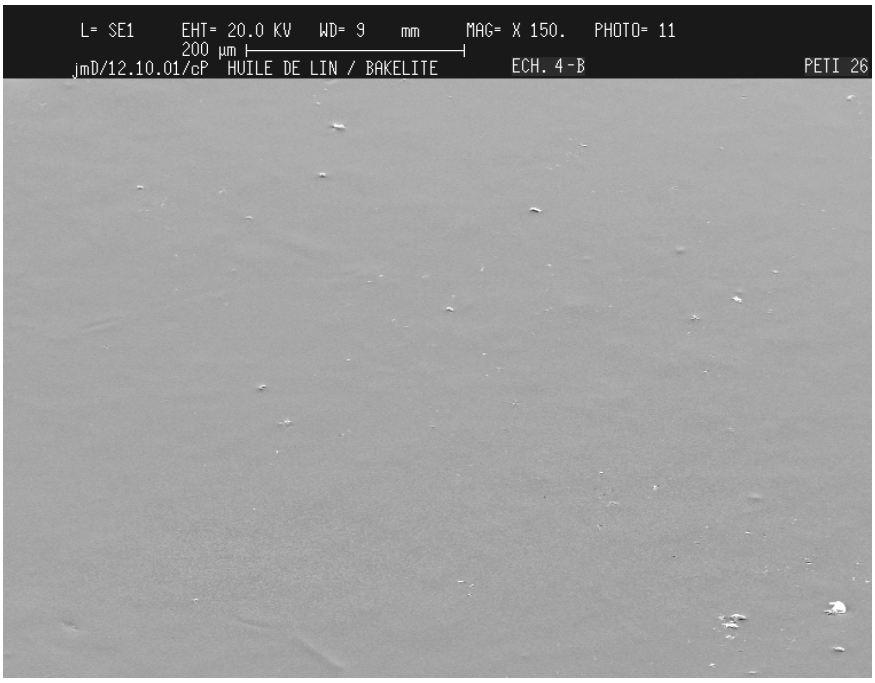


Detail of inclusions and dust particules.

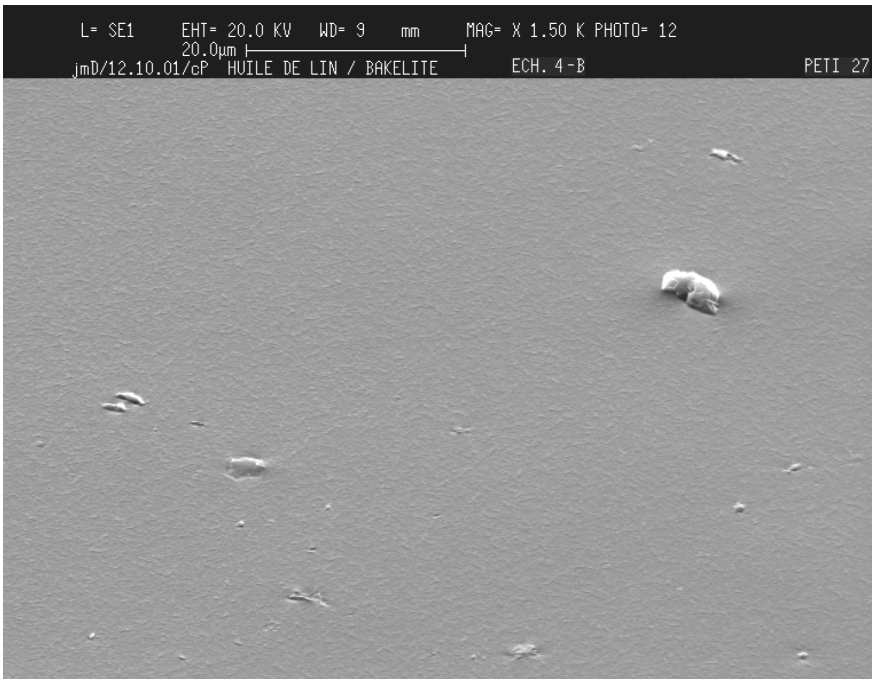


The thickness of the coating layer = 5.7 µm.

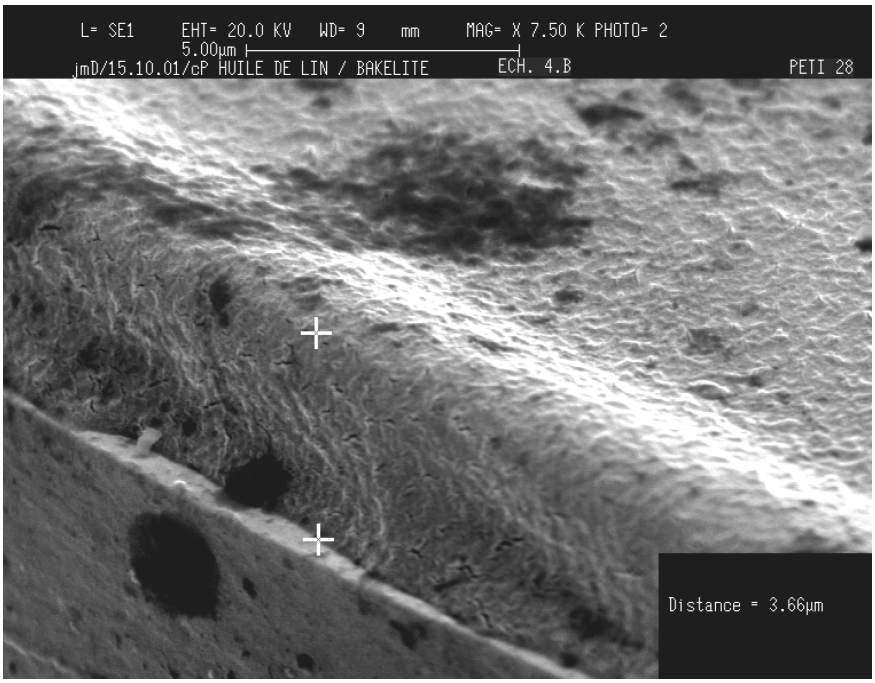
Sample n° 4: boiled linseed oil 15 / 85, filtered



An uniform repartition of the coating layer.

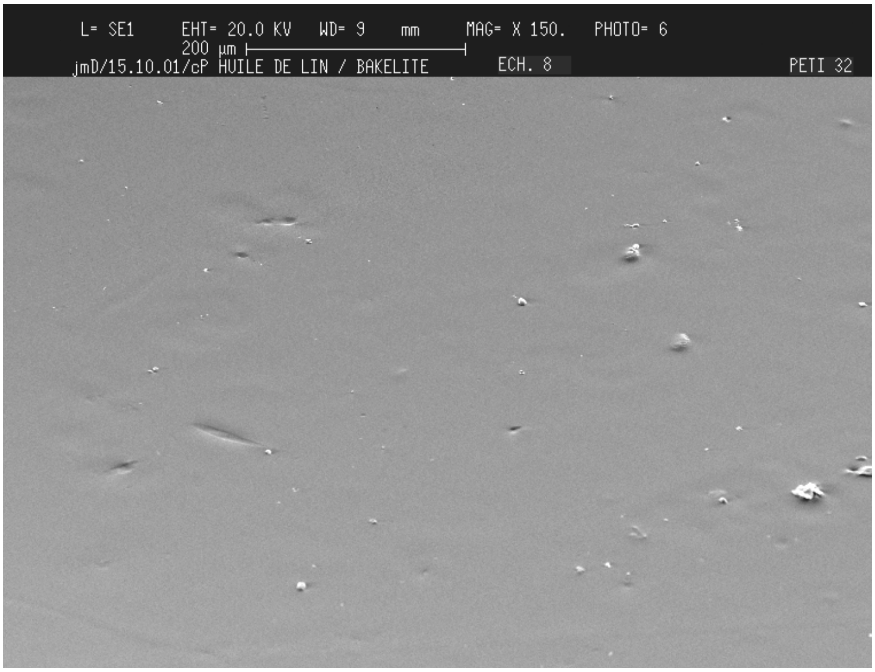


Detail of the inclusions and dust particles.

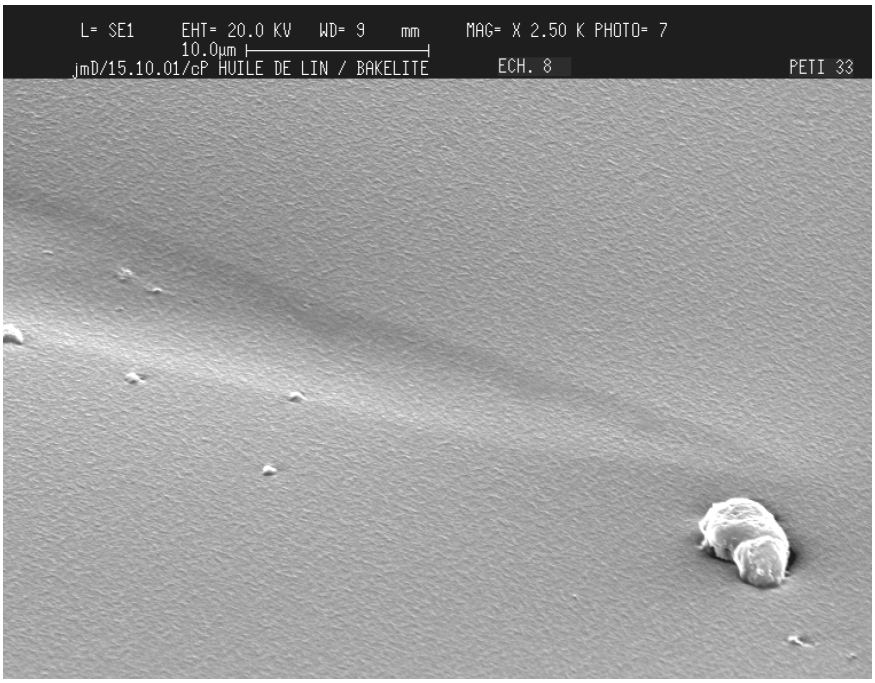


The thickness of the coating layer = 3.7 µm

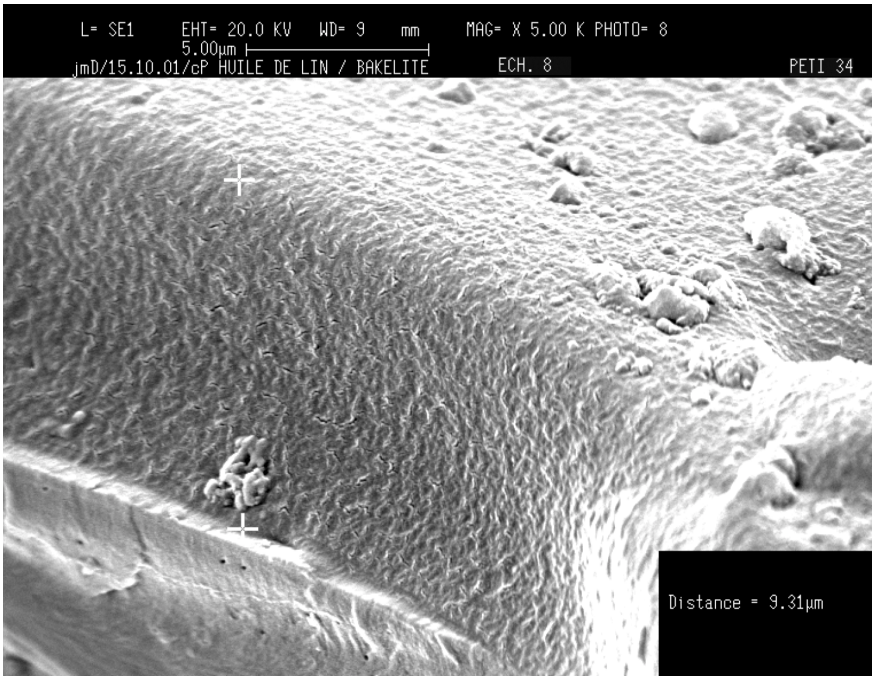
Sample n° 5: OPERA oil dried at room temperature



The surface is smooth, with some non uniformities. Dust particles are present.



Detail of the above photo.



The thickness of the coating layer = 9.3 µm.