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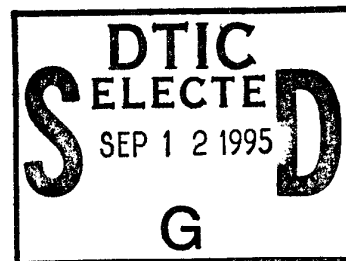
**INVESTIGATING AND SUPPLYING  
HALID FLUX-GROWN KTP CRYSTALS**

**Final Technical Report**

by

Dominique LUPINSKI

July 1995



United States Army  
EUROPEAN RESEARCH OFFICE OF THE U.S. ARMY

London England

**CONTRACT NUMBER DAJA45-93-C-0048**

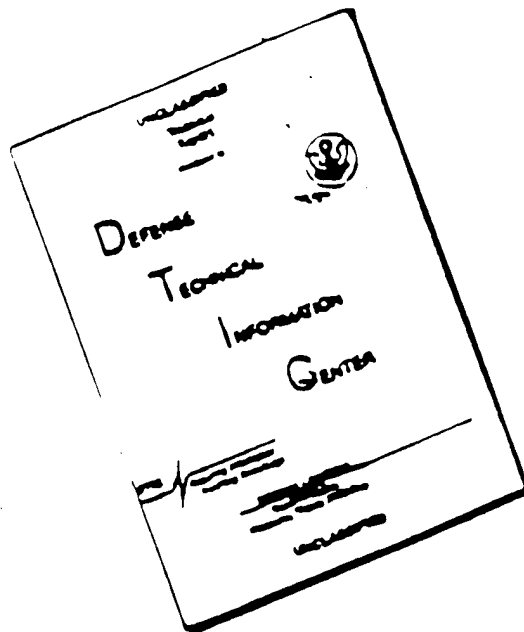
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Therefore, the experiments were performed on crystals from Cristal Laser S.A. The main tests provided for in the contract were performed by qualified scientific personal at Cristal Laser. However, some experiments which required specific equipment had to be realized in external laboratories.

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The 9 AR coated KTP and the 32 mirrors were supplied with the specified control data.

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We wish to thank the US Army Research Laboratory for its support of this important program of production and evaluation of KTP nonlinear material. Under this program, we were able to grow and supply ARL several KTP crystal, and several laser laser mirrors for exciting these materials with laser pump energy at the ARL OPO Laboratory. The COTR for this program was Dr. Albert PINTO, of ARL, who made sure that the material supplied was of the best technical quality we could grow. We wish to thank him for his constant supervision, and also Michael FERRY, who visited us a year ago to discuss requirements and expectations. We also wish to thank Dr. Karl STEINBACH of the US Army European Office in London, for his expert conatructual and administrative support.

Our principal Investigator on this program was Dominique LUPINSKI, but we also include in our list of people to thank Professor Gérard MARNIER, of the Université de Dijon in France, who lent considerable expert advice, Philippe VILLEVAL, who actually grew the crystals, Christophe BONNIN, who processed them, and Hervé ALBRECHT, who analysed them and made sure they met requirements. We also wish to thank the German firm that produced the mirrors, and in particular, Dr. EBBERT, and also the French firm SFIM ODS Mr GEENEN, with whom we interacted during this program.

Finally it should be added that this program was a vital testimony to the success which we can achieve when mutual US-French technical goals are pursued.

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## ABSTRACT

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The 9 AR coated KTP and the 32 mirrors were supplied with the specified control data.

## KEY-WORDS

KTiOPO<sub>4</sub>

KTP

Halide flux-grown

OPO

Absorption

Transparency

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# 1. INTRODUCTION

## 1.1. *Presentation of the contractor*

CRISTAL LASER S.A. is specialized in the growth and processing of inorganic nonlinear crystals and performs also studies and special cuts according to the users' requirements.

The company operates under exclusive licence international CNRS patents<sup>[1]</sup> regarding the synthesis of  $\text{KTiOPO}_4$  and isotypes by flux of alcali metal halide. These patents concretize the research carried out since 1983 by the team of G. Marnier at the Université de Nancy, France, which were transferred to the Université de Bourgogne in 1994.

## 1.2. *Growth of KTP at Cristal Laser*

Potassium Titanyl Phosphate  $\text{KTiOPO}_4$  is an orthorhombic crystal (mm2 space group), optically biaxial, transparent between 0.35 and 4.5  $\mu\text{m}$ , non centrosymmetric and endowed with excellent properties for different nonlinear optical applications. It presents a strong second-order electrical susceptibility, a high optical damage threshold and wide angular and thermal acceptances.

The Second-Harmonic Generation Type II in KTP allows to generate radiations from 0.5 to 1.5  $\mu\text{m}$ . Radiations in the field  $1 \rightarrow 4.5 \mu\text{m}$  can also be obtained by parametric interactions.

The crystals produced for this contract were grown in concentrated solutions of oxides and alcali metal halide raised to high temperature (600°C to 1000°C). This method allows to obtain a high crystalline perfection and low residual absorption (<0.1%/cm @ 1.064  $\mu\text{m}$ ).

## 1.3. *Introduction to the quality controls at Cristal Laser*

The quality criteria of nonlinear crystals are not easy to define. In fact, physical properties of the crystals are strongly correlated with their nonlinear optical properties. So the main problem is to determine the level of an acceptable quality according to the final application. For example, we could not yet define all the significant parameters for Optical Parametric Oscillation (OPO).

Our experience shows that, whatever the application, evaluating the quality level of KTP requires to define and control more than 10 basic criteria during the whole growth-process. Quality parameters such as homogeneity, dark-lines, impurities, optical absorption, transparency, ionic conductivity, thermal stability, extinction ratio, conversion efficiency, life-time, optical damage threshold, depend on the growth conditions :

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<sup>[1]</sup> CNRS-University of Nancy I licence - G. MARNIER, B. BOULANGER, B. MENAERT, M. METZGER.  
US patent n°4,746,396 (28/07/86) - US Patent n°4,961,819 (22/01/88).

stoichiometry, linear growth-speed, temperature of growth, global crystallization efficiency, rotation speed, thermal gradient, thermal shocks, etc...

Lastly a great difficulty is to make the measurements in absolute. Indeed the values which are obtained strongly vary according to the conditions of experimentation (wavelength, polarization of the laser beam, repetition rate, ...).

Regarding KTP as an Optical Parametric Oscillator, which is dealt with this report, we focused on two criteria which seem to be the most significant for this application : the optical absorption at 1.064  $\mu\text{m}$  and the homogeneity.

## 2. LIST OF THE KTP DELIVERED WITHIN THE CONTRACT

Reference	Cutting angle		Size	AR coating	Reference of batch
	$\theta$	$\phi$			
34 00 203	51.5°	0°	5x6x10 mm <sup>3</sup>	1.064 + 2.128 $\mu\text{m}$	173/94/6+83/95/6
34 00 204	51.5°	0°	5x6x10 mm <sup>3</sup>	1.064 + 2.128 $\mu\text{m}$	173/94/6+83/95/6
34 00 206	51.5°	0°	5x6x1.73 mm <sup>3</sup>	1.064 + 2.128 $\mu\text{m}$	173/94/6+83/95/6
40 40 109	90°	0°	5x5x10 mm <sup>3</sup>	1.064 + 1.52-1.58 $\mu\text{m}$	65/95/6+79/95/6
44 00 205	90°	0°	5x5x10 mm <sup>3</sup>	1.064 + 1.52-1.58 $\mu\text{m}$	TM/270795
40 30 724	90°	0°	5x5x2 mm <sup>3</sup>	1.064 + 1.52-1.58 $\mu\text{m}$	65/95/6+79/95/6
40 40 221	90°	90°	5x5x10 mm <sup>3</sup>	1.064 + 1.52-1.58 $\mu\text{m}$	65/95/6+78/95/6
40 40 222	90°	90°	5x5x10 mm <sup>3</sup>	1.064 + 1.52-1.58 $\mu\text{m}$	65/95/6+78/95/6
34 00 209	90°	90°	5x5x2 mm <sup>3</sup>	1.064 + 1.52-1.58 $\mu\text{m}$	65/95/6+79/95/6

## 3. TESTS OF THE KTP

### 3.1. Crystallographic orientation

Performed by : C. BONNIN, Engineer, Cristal Laser.

The crystallographic orientation has been measured with a X-Ray goniometer ensuring  $\pm 3'$  accuracy.

Before the polishing step the precision of the cutting angle reached  $\pm 3'$  for X-cut and Y-cut and  $12'$  for  $\theta = 51.5^\circ$ .

All the finished crystals have a cutting error  $\leq 30'$ . The Z-face is also orientated with less than  $30'$  accuracy.

### 3.2. Flatness and parallelism

Performed by : D. LUPINSKI, principal investigator, Cristal Laser  
C. BONNIN, engineer, Cristal Laser.

Flatness is better than  $\lambda/10$  @ 633 nm. PV values are typically 0.07 or 0.08  $\lambda$ .  
For example, the first surface of the crystal ref. 40 40 109 is  $0.08 \pm 0.01 \lambda$ , including the edge effect. The PV value on the crystal center is below 0.05  $\lambda$  for X-profile (appendix A, fig. 1, 2, 3).

The parallelism is below 10 arc seconds due to the double-side polishing technique used. Parallelism values reach 1  $\mu\text{m}$  for 40 cm.

### 3.3. Optical absorption

Performed by : Dr. V. WILLAMOWSKI, Laser Zentrum Hannover, Germany  
Dr. J. MANGIN, Université de Nancy, France

The principal effect of the optical absorption is a mismatch of the crystals which induces instabilities of the conversion efficiency.

The measurement in absolute is generally critical. It requires very accurate means of measurement because of the low absorption of the KTP, particularly at 1.064  $\mu\text{m}$  (40-100 ppm).

Two ways of measurement have been investigated :

- The calorimetric method <sup>[2]</sup>
- The pyrospectrometric method <sup>[3]</sup>

At 1.064  $\mu\text{m}$ , the values are quite similar with the both techniques, that is to say  $\leq 0.05\%/cm$ .

The absorption of a 5 mm long KTP cut in the SHG direction is respectively 70 and 80 ppm for the two polarizations Kxy and Kz.

With the calorimetric technique, all the crystals measured present an absorption below 0.02%/cm at 1.064  $\mu\text{m}$ .

Seeing these results, the production of Cristal Laser is quite homogeneous. However absolute values of absorption have to be considered cautiously : indeed the very low values measured depend on the method and the control bench used. Hence a comparative

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<sup>[2]</sup> M. Dieckmann, V. Willamowski, D. Ristau, H. Welling - Laser Zentrum Hannover e.V., *Antireflective Coatings on Optical Fibers for High Power Solid-State Lasers*,

<sup>[3]</sup> J. Mangin, A. Khodjaoui and G. Marnier, *Optical absorption of KTP single crystals*, Physica Status Solidi (a) 120 (1990) K111.

study requires obviously the same control bench as well as strictly similar crystals (same size and same crystallographic orientation).

### 3.4. Transmission 0.3-5 $\mu\text{m}$

As for the absorption, the transmission was measured on KTP crystals of similar length, same crystallographic orientation, attending to precise the polarization of the laser beam. The crystals were measured before coating.

A great difference can be observed between 3.2  $\mu\text{m}$  and 3.8  $\mu\text{m}$  at the two polarizations. The idler is polarized along OZ for X-cut and Y-cut.

For X-cut :  $\lambda_i = 3.29 \mu\text{m}$  : the transmission along OZ is higher than along OY.

For Y-cut :  $\lambda_i = 3.5 \mu\text{m}$  : the transmission along OZ is lower than along OX.

The transmission along Y-cut seems generally lower than along X-cut

For equivalent direction and beam polarization, the transmission is quite the same with the different crystals. The transmission graph presents the same profile including the accuracy level of the measurement.

The absorption band at 2.8  $\mu\text{m}$  (OH band) is low compared with other KTP sources, in particular hydrothermally grown crystals.

### 3.5. Homogeneity

In KTP crystals, inhomogeneities can be observed with different devices :

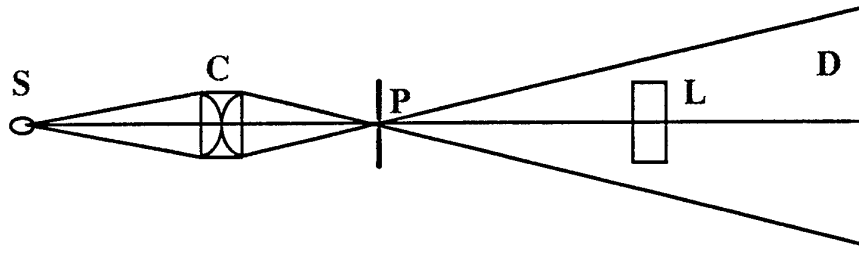
- Inclusions of very small diameter (HeNe laser can pass the crystals and scattering centers can be observed with a microscope)<sup>[4]</sup>
- Larger inclusions can be studied with birefringence interferometry. Along b axis, pictures show {011} and {100} inclusions<sup>[4]</sup>.
- Optical homogeneity has been evaluated by measuring wavefront distortion in transmission with circularly polarized wave. Variations of respectively  $1.6 \cdot 10^{-4}$  and  $7.1 \cdot 10^{-6}$  of  $n_x$  and  $n_{xy}$  refractive indices have been measured<sup>[5]</sup>.
- Variation of the nonlinear effects by scanning the crystals with a tight focused laser beam. The inhomogeneity of refractive indices may induce a variation of the phase-matching angle.

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<sup>[4]</sup> R.J. Bolt, M.H. Van Der Mooren and H. De Hass, *Growth of KTP single crystals by means of Phosphate and Phosphate/Sulfate fluxes out of a three-zone furnace*, Journal of Crystal Growth 114 (1991), 141-152.

<sup>[5]</sup> T. Sasaki, A. Miyamoto, A. Yokotami and S. Nakai, *Growth and optical characterization of large potassium titanyl phosphate crystals*, Journal of Crystal Growth 128 (1993), 950-955.

- The shadow technique : The sample must be a fine polished cube with parallelism better than 10 arc seconds and flatness less than  $\lambda/10$ . A white light is used as a source with lenses to focus on a pinhole. The light is diffracted and goes through the crystal. The projection of the KTP crystal is stored with a fast, continuous tone, contrast graded, black and white paper placed behind the sample (see the figure).



- S : Light source
- C : Lenses
- P : Pinhole
- L : KTP crystal
- D : Paper

When the sample is perfectly homogeneous, the outlines of the crystal can be observed with uniform color. On the contrary, when the crystal shows bulk local defects, the optical path is changed. On the paper, we note complex figures drawn as a variation of the grey tone. It is very difficult to correlate theoretical calculations with the experimental datas. This is the reason why we just compare all the different samples to estimate the quality.

## 4. AR COATINGS

For the OPO application, the KTP have to AR coated at the pump and at the signal wavelength. We selected SFIM ODS as a subcontractor, which performed the following AR coatings :

- n°1 : dual-band AR coating :  $\lambda_p = 1.064 \mu\text{m}$   
 $\lambda_s = 2.128 \mu\text{m}$
- n°2 : dual-band AR coating :  $\lambda_p = 1.064 \mu\text{m}$   
 $1.52 \mu\text{m} \leq \lambda_s \leq 1.58 \mu\text{m}$

Unfortunately the first face of the two batches were not at the maximum efficiency they could reach. The measurement was made directly on KTP for the two indices  $n_y$  and  $n_z$ .

AR COATING 1.064 $\mu\text{m}$ + 2.128 $\mu\text{m}$			
Face	Ref. batch	$\lambda = 1.064 \mu\text{m}$	$\lambda = 2.128 \mu\text{m}$
Face n°1	173/94/6	$1\% \leq R \leq 1.23\%$	$0.13\% \leq R \leq 0.23\%$
Face n°2	83/95/6	Rny = 0.6% Rnz = 0.4%	Rny = 0.1% Rnz = 0.1%

AR COATING 1.064 $\mu\text{m}$ + 1.52-1.58 $\mu\text{m}$			
Ref. batch	$\lambda = 1.064 \mu\text{m}$	$\lambda = 1.52 \mu\text{m}$	$\lambda = 1.58 \mu\text{m}$
65/95/6	Rny = 1.00% Rnz = 1.35%	Rny = 0.35% Rnz = 0.60%	Rny = 1.20% Rnz = 1.65%
78/95/6	Rny = 0.15% Rnz = 0.30%	Rny = 0.50% Rnz = 0.80%	Rny = 0.50% Rnz = 0.70%
79/95/6	$0.1\% \leq R \leq 0.2\%$	$0.4\% \leq R \leq 0.55\%$	$0.6\% \leq R \leq 0.8\%$
TM/270795	$R \leq 0.1\%$	$R \leq 0.15\%$	$R \leq 0.15\%$

## 5. OPO MIRRORS

CaF2 mirrors for testing the KTP in the OPO configuration were provided by a german company which could meet the specifications and the budget of the contract.

### Required specifications :

Substrate : CaF2 1" diameter, 1/4" thick			
	Surface #1	Surface #2	Quantity
M1	AR 1.064 $\mu\text{m}$	AR 1.064 $\mu\text{m}$ HR 1.50-1.65 $\mu\text{m}$	4
M2	HR 1.064 $\mu\text{m}$ 50% $\pm$ 10% @ 1.5-1.65 $\mu\text{m}$	AR 1.5-1.65 $\mu\text{m}$	4
M3	HR 1.064 $\mu\text{m}$ 70% $\pm$ 10% @ 1.50-1.65 $\mu\text{m}$	AR 1.5-1.65 $\mu\text{m}$	4
M4	HR 1.064 $\mu\text{m}$ 90% $\pm$ 10% @ 1.50-1.65 $\mu\text{m}$	AR 1.50-1.65 $\mu\text{m}$	4
M5	AR 1.064 $\mu\text{m}$	AR 1.064 $\mu\text{m}$ HR 1.9-2.3 $\mu\text{m}$	4
M6	AR 1.064 $\mu\text{m}$ 50% $\pm$ 10% @ 1.9-2.3 $\mu\text{m}$	AR 1.9-2.3 $\mu\text{m}$	4
M7	HR 1.064 $\mu\text{m}$ 70% $\pm$ 10% @ 1.9-2.3 $\mu\text{m}$	AR 1.9-2.3 $\mu\text{m}$	4
M8	HR 1.064 $\mu\text{m}$	AR 1.9-2.3 $\mu\text{m}$	4

**Reflection measurements :**

	M1-M4	1064 nm	1500→1650 nm	1520 nm	1570 nm
Type of mirror	M5-M8	1064 nm	1900 → 2300 nm	2100 nm	2160 nm
M1	requested	T > 90%	T < 1%		
	measured	T = 92%	T < 0.5%	T = 0.3%	T = 0.3%
M2	requested	T < 1%	40% ≤ T ≤ 60%		
	measured	T < 1%	37% ≤ T ≤ 68%	T = 58%	T = 40%
M3	requested	T < 1%	20% ≤ T ≤ 40%		
	measured	T < 1%	23% ≤ T ≤ 55%	T = 48%	T = 28%
M4	requested	T < 1%	0% ≤ T ≤ 20%		
	measured	T < 1%	5% ≤ T ≤ 12%	T = 9%	T = 5%
M5	requested	T > 90%	T < 1%		
	measured	T = 89%	T < 1%	T < 1%	T < 1%
M6	requested	T < 1%	40% ≤ T ≤ 60%		
	measured	T < 1%	34% < T < 58%	T = 40%	T = 35%
M7	requested	T < 1%	20% ≤ T ≤ 40%		
	measured	T < 1%	18% < T < 32%	T = 21%	T = 18%
M8	requested	T < 1%	0% ≤ T ≤ 20%		
	measured	T < 1%	8% ≤ T ≤ 16%	T = 12%	T = 10%

# **APPENDIX # 1**

**Flatness data**

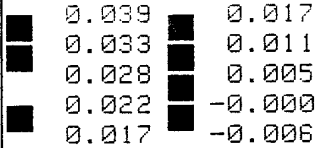
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T P

rms: 0.010  
wv: 633.0nm

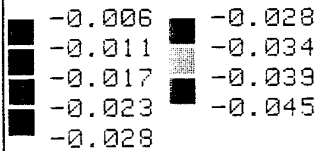
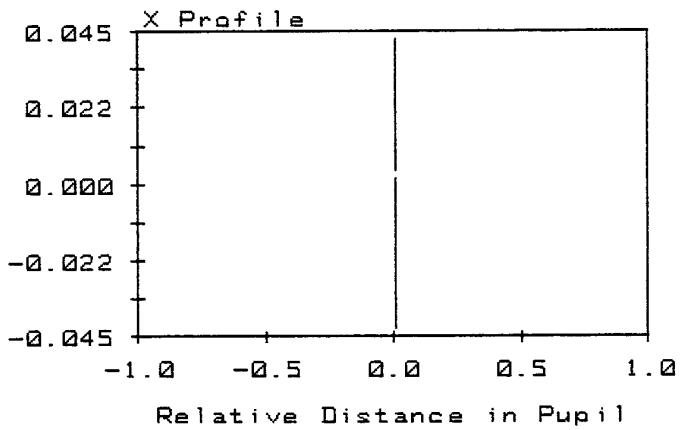
### OPD

p-v: 0.084  
pupil: 90%



x = 125, 0.00  
y = 128, -0.00  
Height = 0.003

rms = 0.007  
p-v = 0.033



SUBSTRAT, A-FLAECHE

WYKO

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T P

rms: 0.008  
wv: 633.0nm

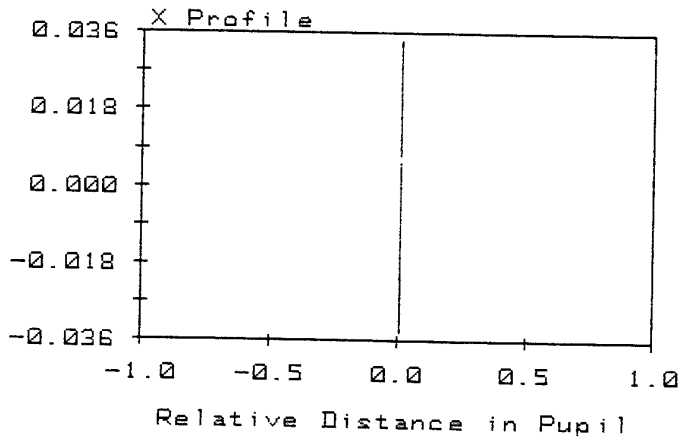
OPD

p-v: 0.071  
pupil: 90%

■	0.035	■	0.016
■	0.030	■	0.011
■	0.026	■	0.007
■	0.021	■	0.002
■	0.016	■	-0.003

x = 125, 0.00  
y = 128, -0.00  
Height = 0.006

rms = 0.005  
p-v = 0.018



■	-0.003	■	-0.022
■	-0.008	■	-0.027
■	-0.012	■	-0.031
■	-0.017	■	-0.036
■	-0.022		

SUBSTRAT , A-FLAECH

WYKO

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T P

rms: 0.022  
wv: 633.0nm

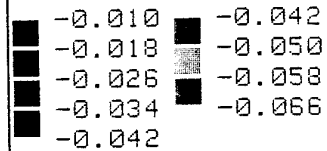
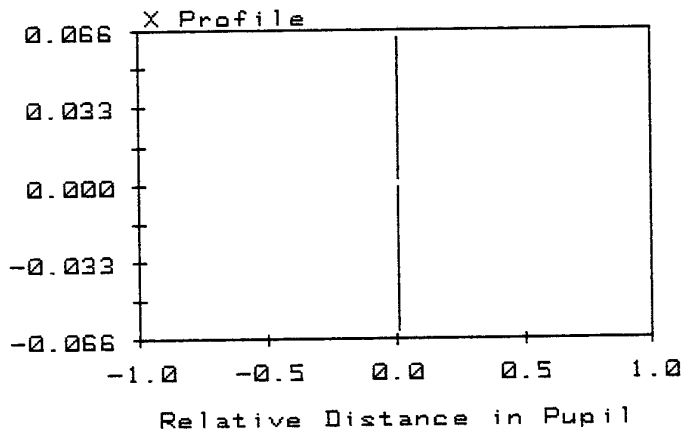
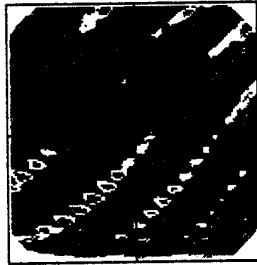
OPD

p-v: 0.120  
pupil: 90%



x = 125.000  
y = 127.000  
Height = -0.002

rms = 0.020  
p-v = 0.065



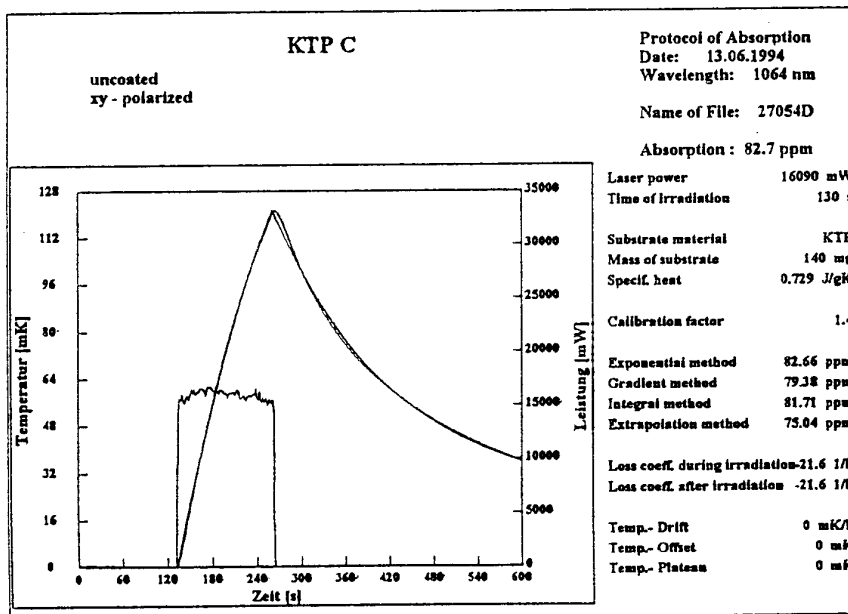
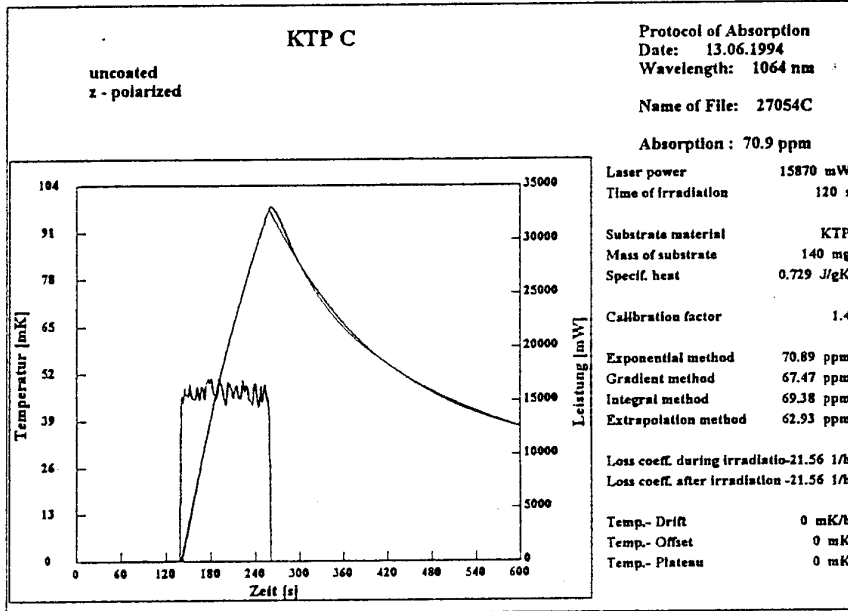
SUBSTRAT , A-FLAECH

WYKO

# **APPENDIX # 2**

**Absorption data**

# KTP 40 30 71 07

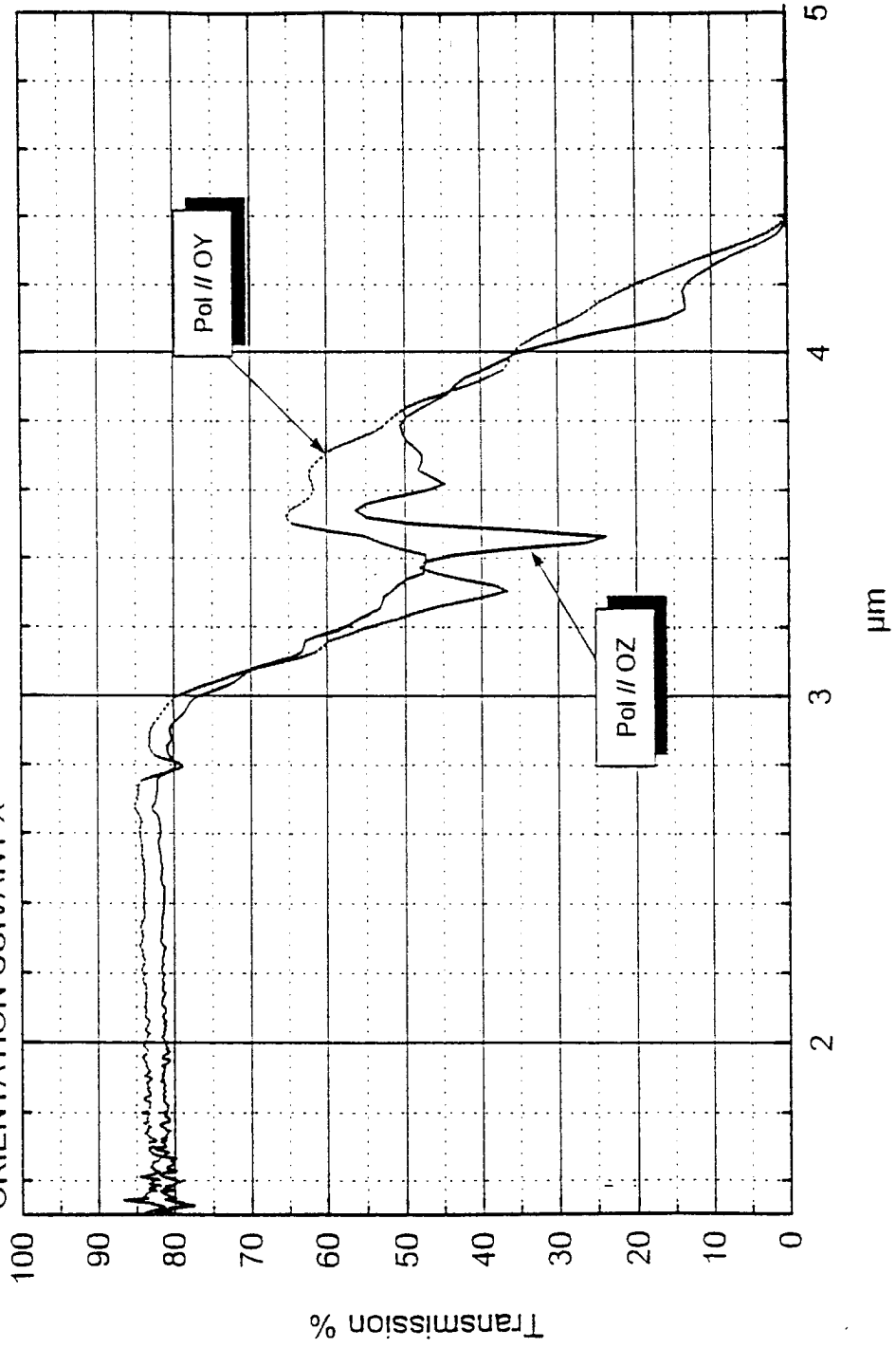


5 MM

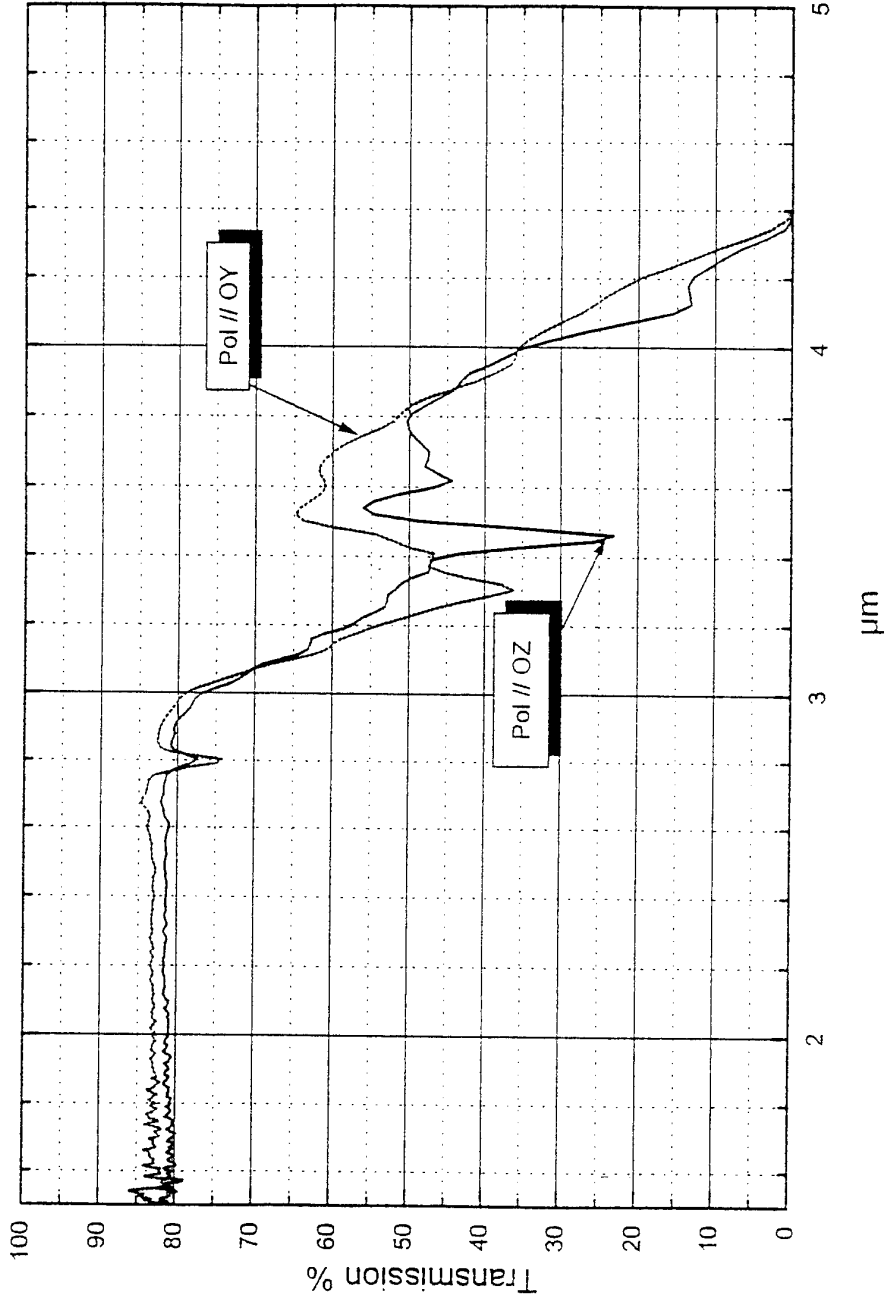
# **APPENDIX # 3**

**Transmission graphes of KTP crystals**

KTP N° 4040109  
ORIENTATION SUIVANT X

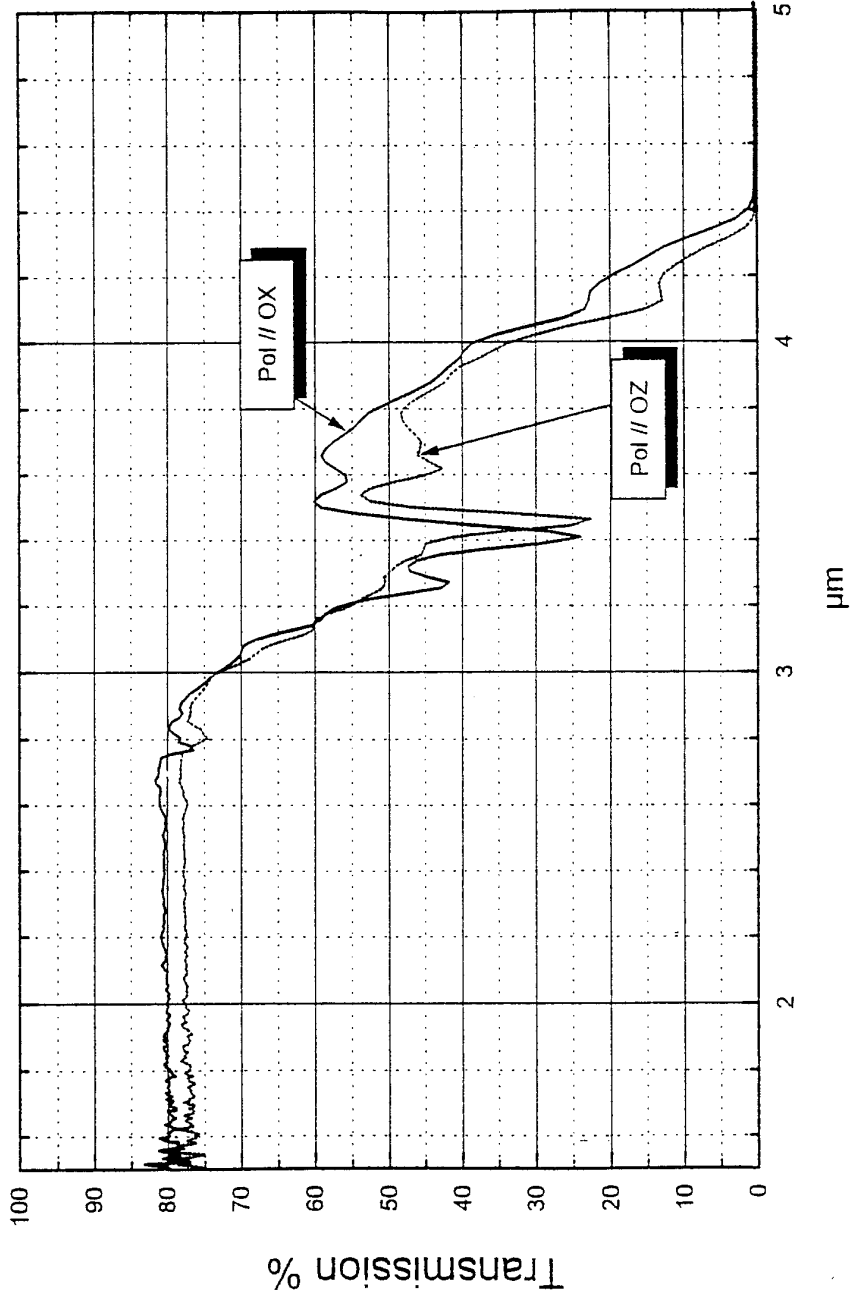


KTP N°4030720  
Orientation suivant X



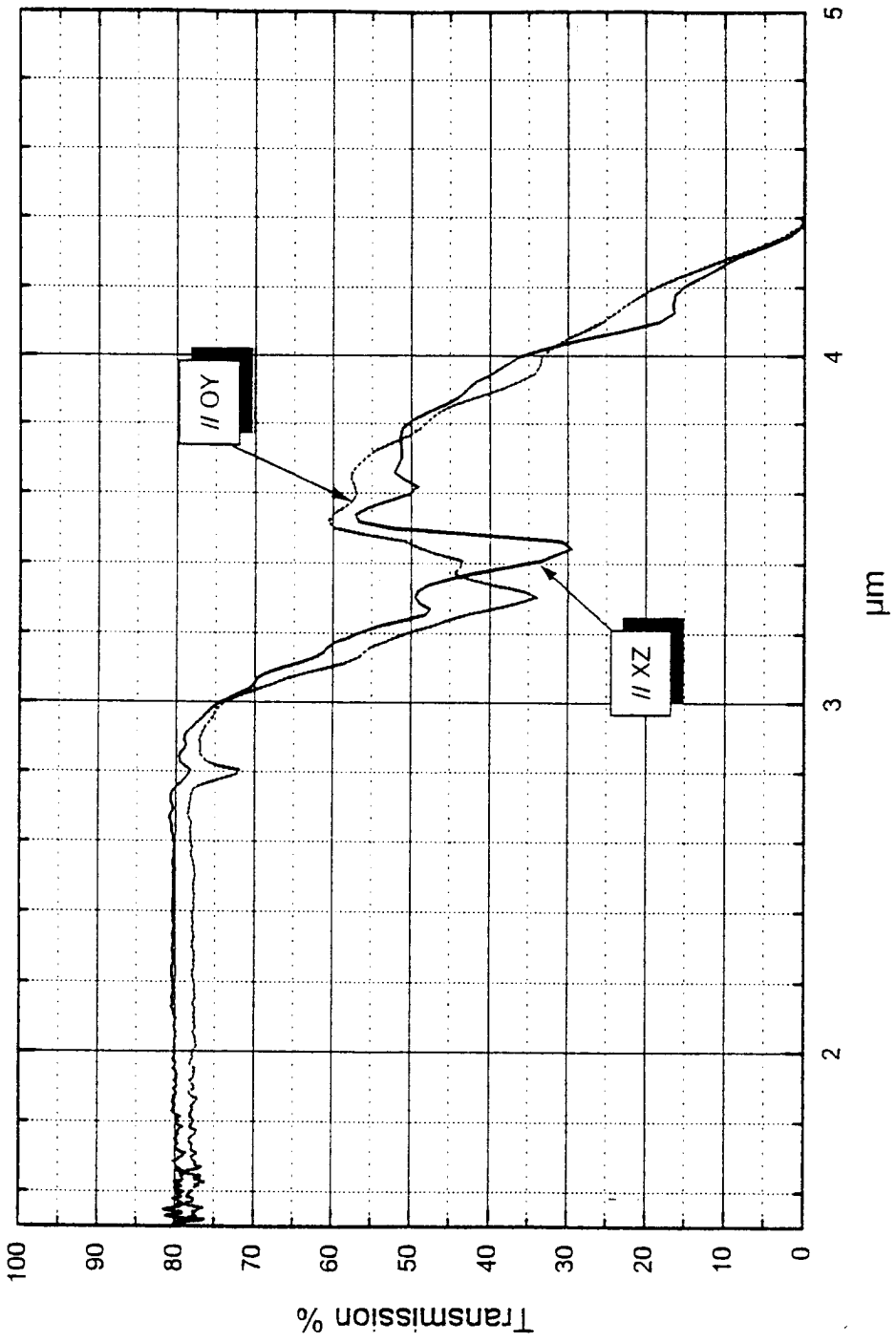
KTP N° 4040222

Orientation suivant Y



# KTP N°3 40 02 04

Orientation suivant XZ



# **APPENDIX # 4**

## **Homogeneity data**

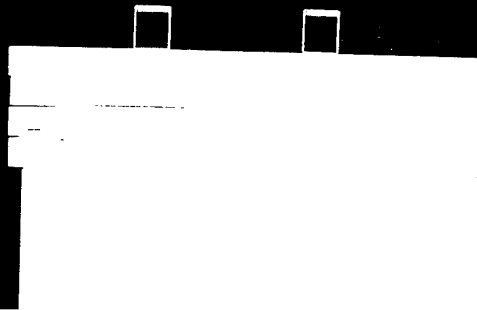
# CRISTAL LASER

34 00 21 04  
 $\theta = 51,5^\circ$

4.94 x 6.06 x 10.00

34 00 21 03  
 $\theta = 51,5^\circ$

4.94 x 6.06 x 10.00





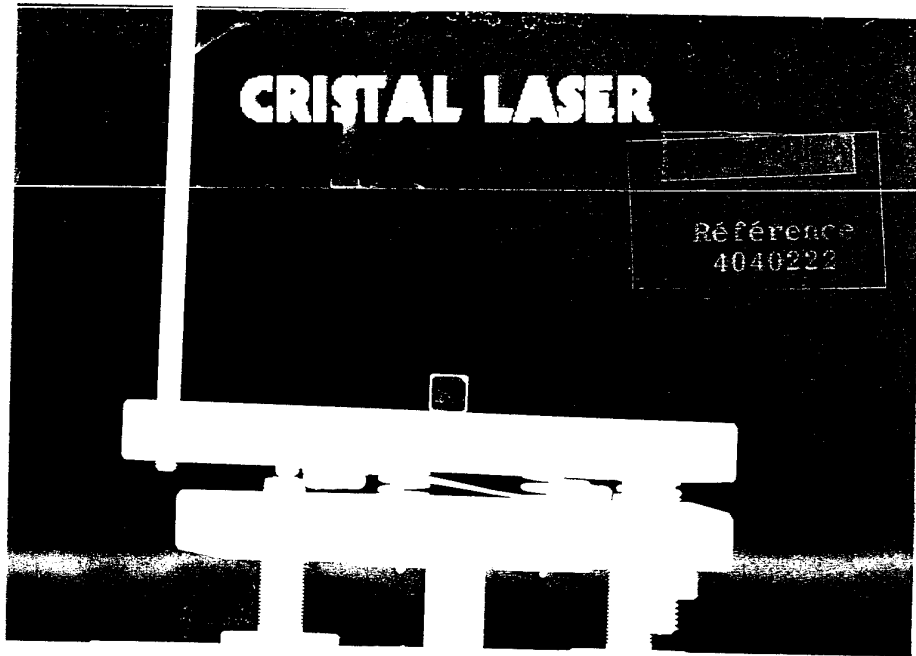
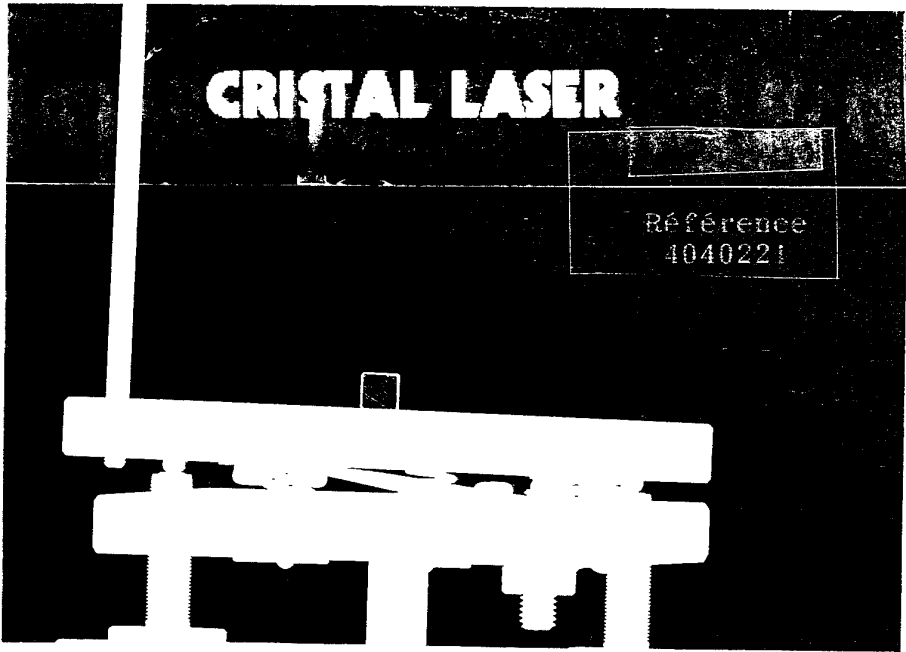
*cristal laser*

12, allée Jean-Rostand  
B.P. 44 - 54230 CHALIGNY  
FRANCE  
Tél. : 83.47.01.01  
Fax : 83.47.22.72

40 40 103  
Ø = 90 g = 0  
5 x 5 x 10.

**CRISTAL LASER**

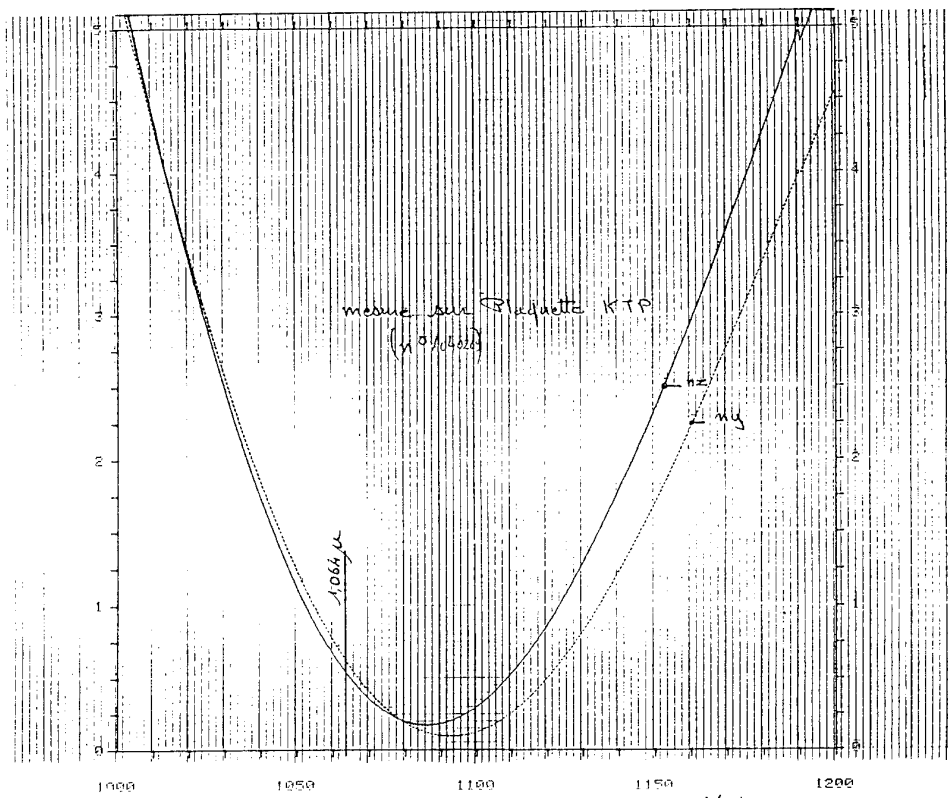
REGISTRATION  
4160205



# **APPENDIX # 5**

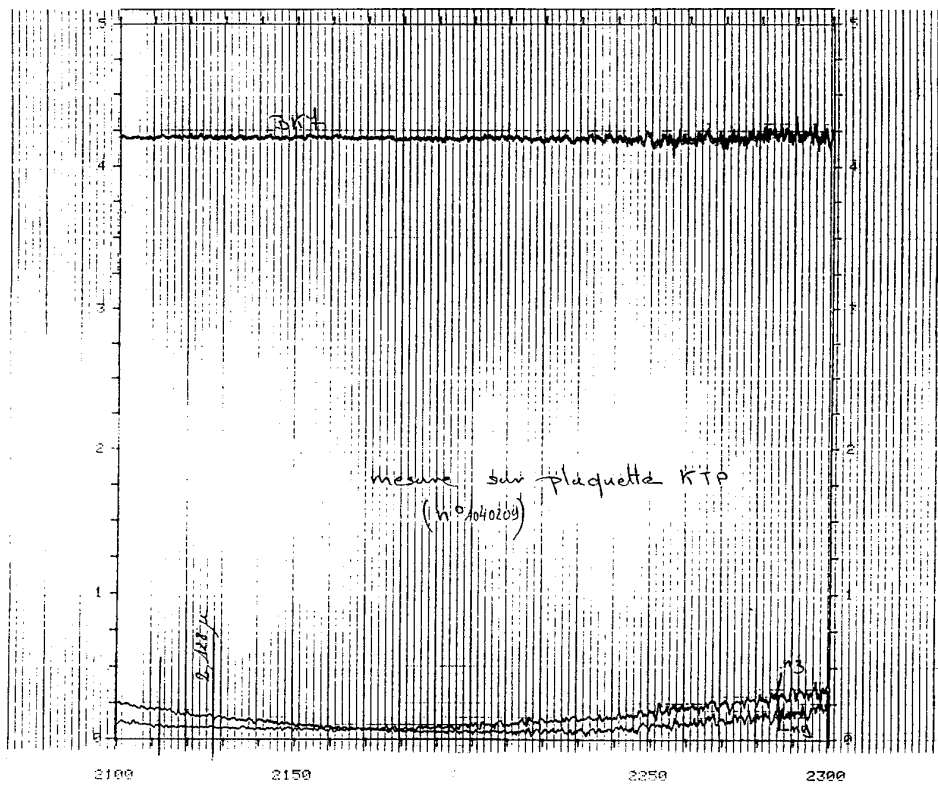
**Reflection graphes of AR coatings**

**1.064+2.128  $\mu\text{m}$**



(code no 1) 113/34/6 - R/8° - existant LASCA - 24/1/95

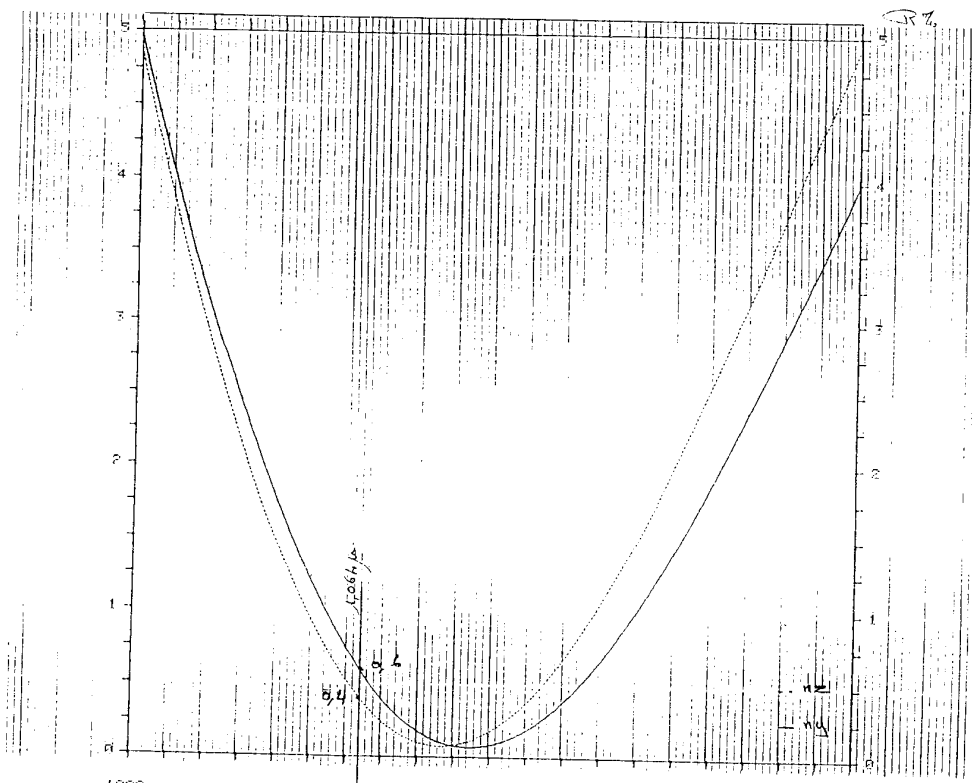
RECHERCHE DE LA LUMIERE



(code no.) 113/94/6

$\theta = 5^\circ$  - CRISTAL LYSER -

24/1/95



83/95/6

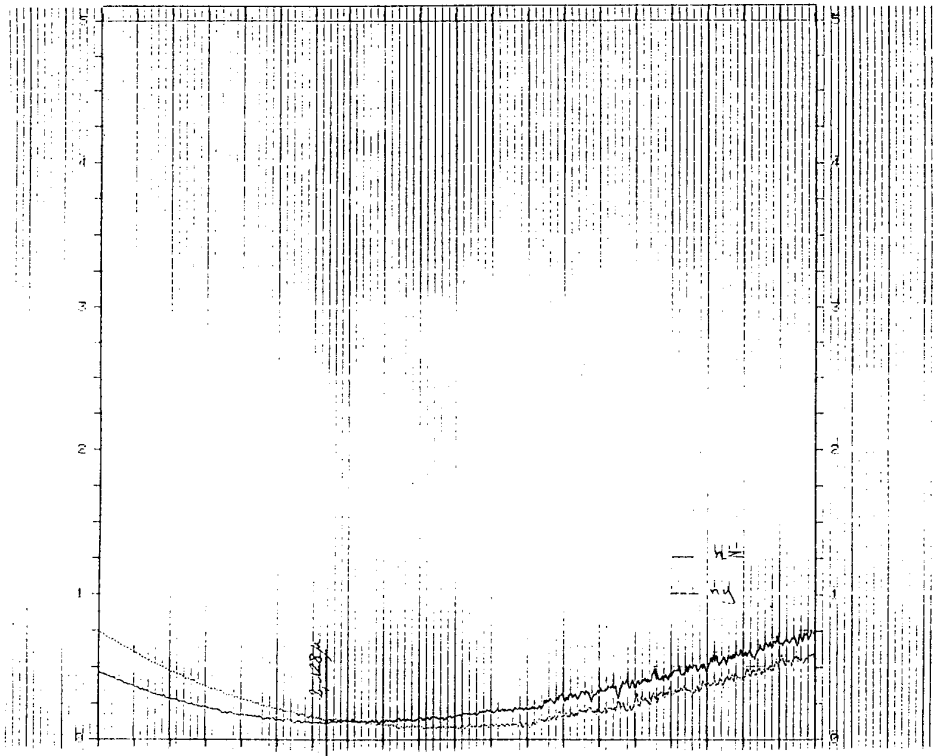
(Bundschuh net 1040209)

R/5°

19/5/95

1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26 27 28 29 30 31 32 33 34 35 36 37 38 39 40 41 42 43 44 45 46 47 48 49 50 51 52 53 54 55 56 57 58 59 60 61 62 63 64 65 66 67 68 69 70 71 72 73 74 75 76 77 78 79 80 81 82 83 84 85 86 87 88 89 90 91 92 93 94 95 96 97 98 99 100

PROCEEDINGS OF THE BOARD OF DIRECTORS

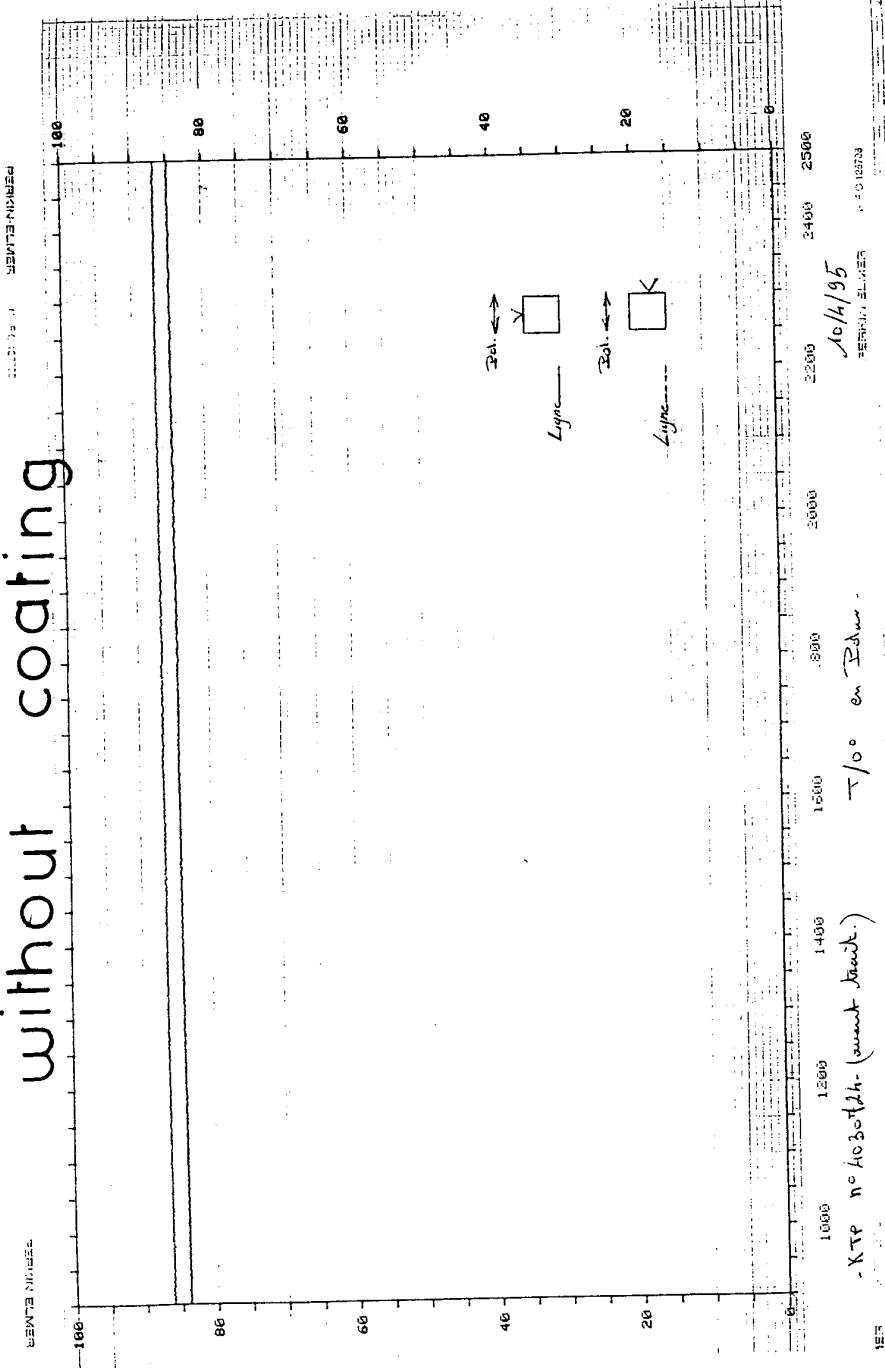


2000 83/95/6 2100 2200 2300 2400  
(Etiopiyalı KTP n° 10/0209) R/5° 13/5/95

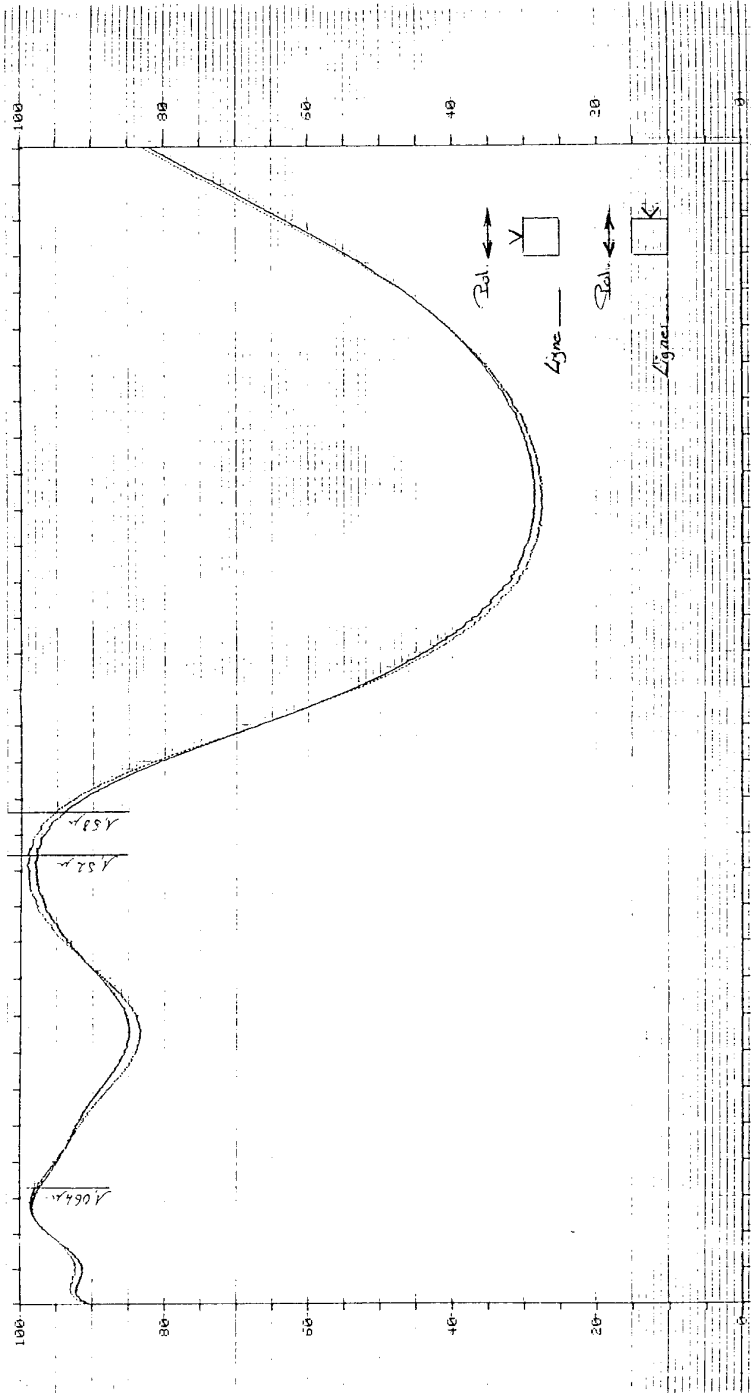
# **APPENDIX # 6**

**Reflection graphes of AR coatings  
1.064 + 1.52-1.58  $\mu\text{m}$**

without coating



PERKIN-ELMER



KTP n° 4030424 (Apres TAR 2<sup>ème</sup>) T/00 en Balan [ 65-79/95/6 ] 19/8/95

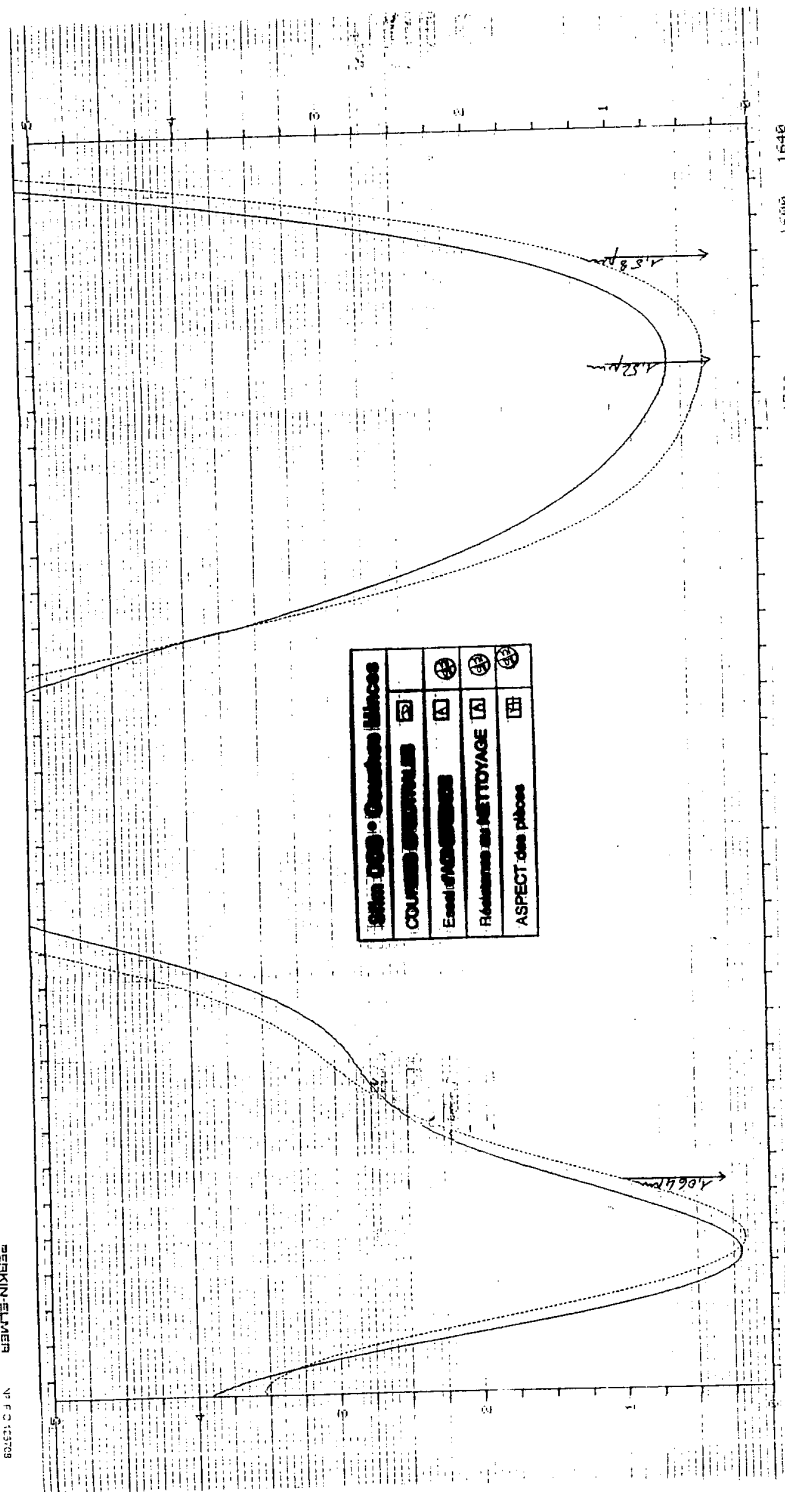
PERKIN-ELMER

PERKIN-ELMER

FO 15722

PERKIN-ELMER

VP F. 15728



Titre DDB - Quantités Mesées	□
COURSUS SPÉCIFIQUES	□
Essai d'ANALYSE	□
Réactions au NETTOYAGE	□
ASPECT des pièces	□

1000 1100 1200 1300 1400 1500 1600 1640

1520 cm⁻¹

1060 cm⁻¹

1200

1300

1100

1000

20/04/56

65/05/6

1000

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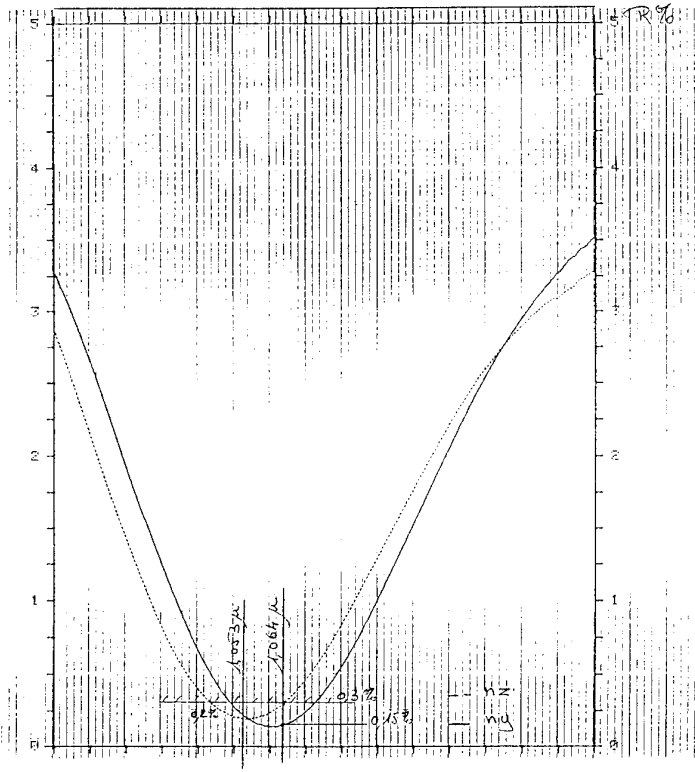
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1000

1100

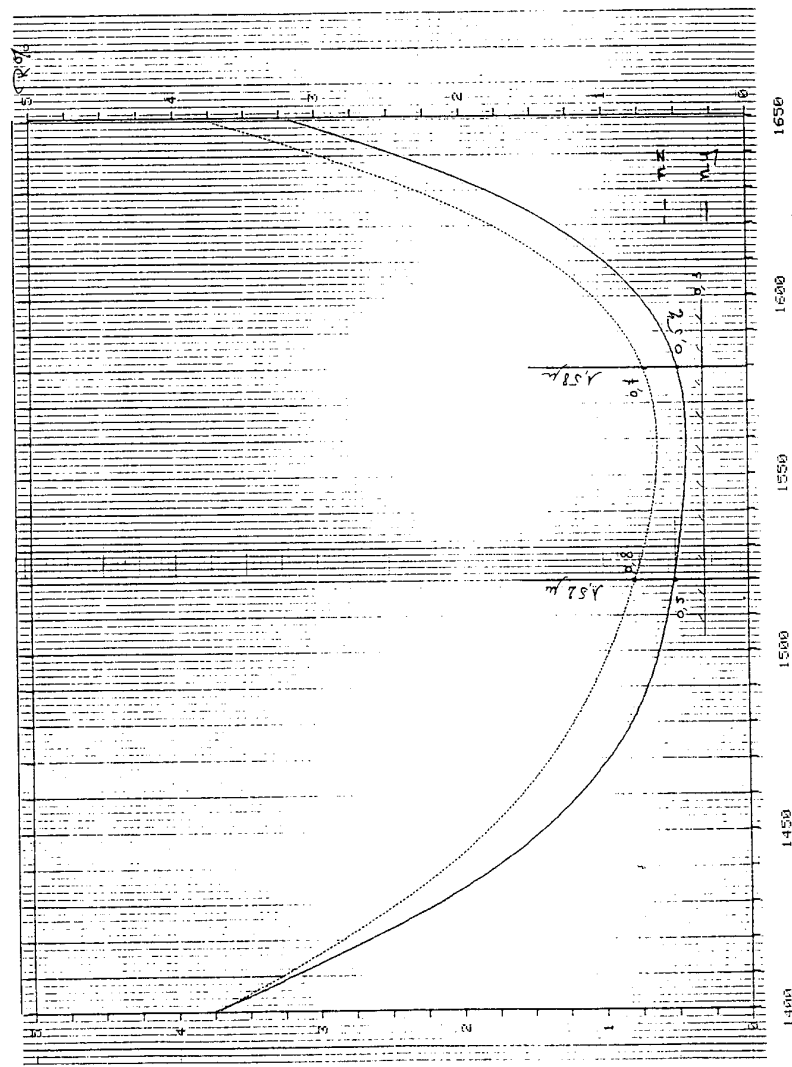
1200

1300



1000 1050 1100 1150  
 18/95/6 CR/5° 19/5/95  
 (Plaque ref. 4040208)

PERFIL BILMENH 10/10/2000



1400 1450 1500 1550 1600 1650

7/8/95/6  
(S) 104 01 00 08

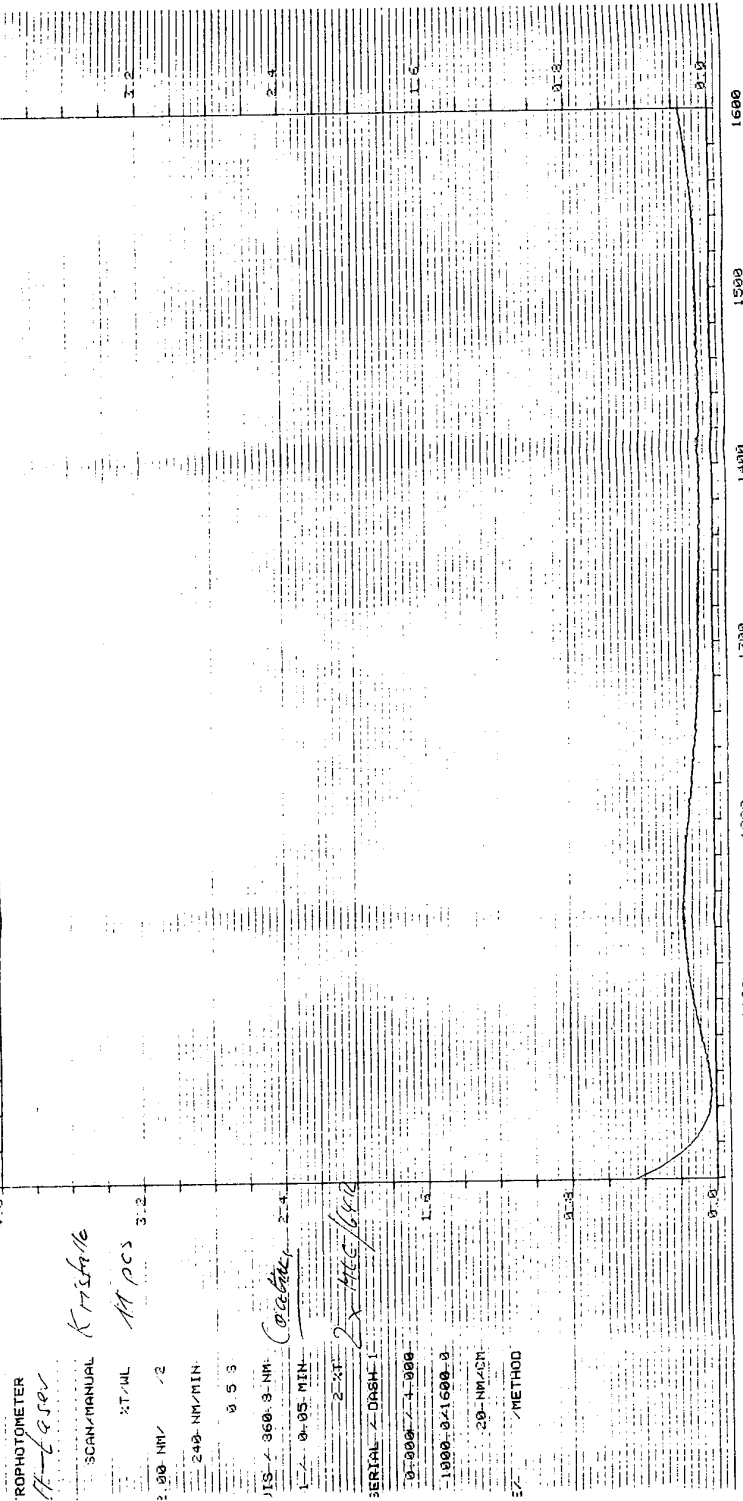
3/5°

10/5/95

PROFIL BILMENH 10/10/2000



TM 270795



1000 1100 1200 1300 1400 1500 1600

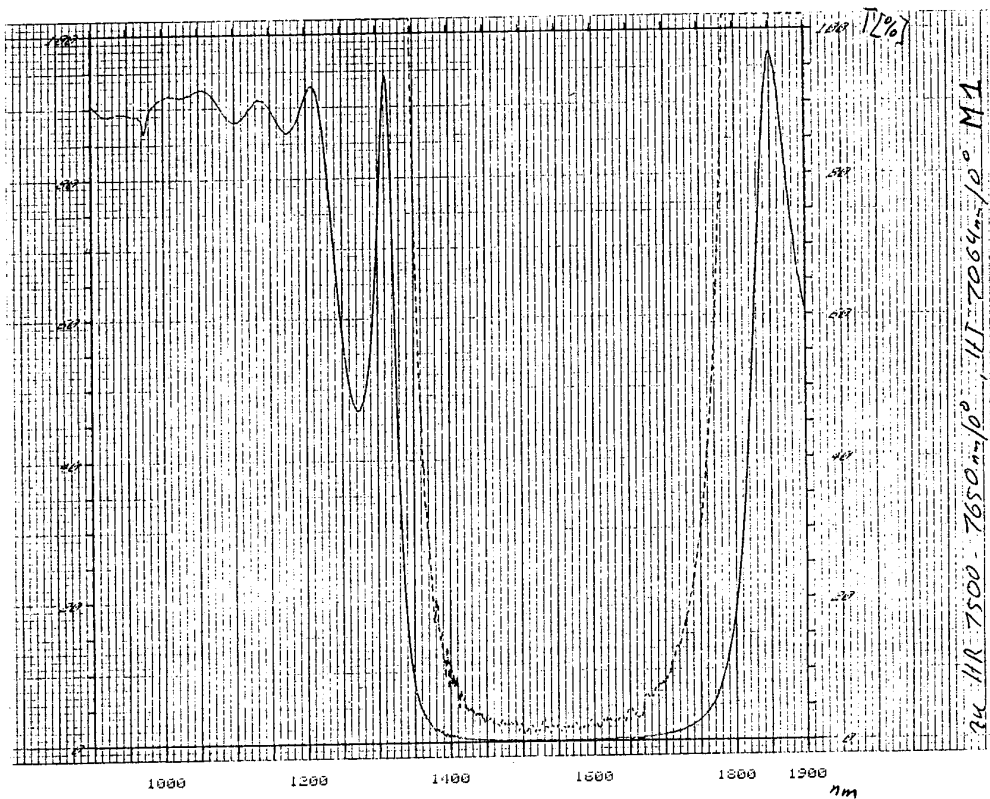
# **APPENDIX # 7**

**Transmission graphes of OPO mirrors**

PERKIN ELMER

PART NO. BO 126708

PERKIN ELMER



PART NO. BO 126708

PERKIN ELMER

PART NO. BO 126708

PERKIN ELMER

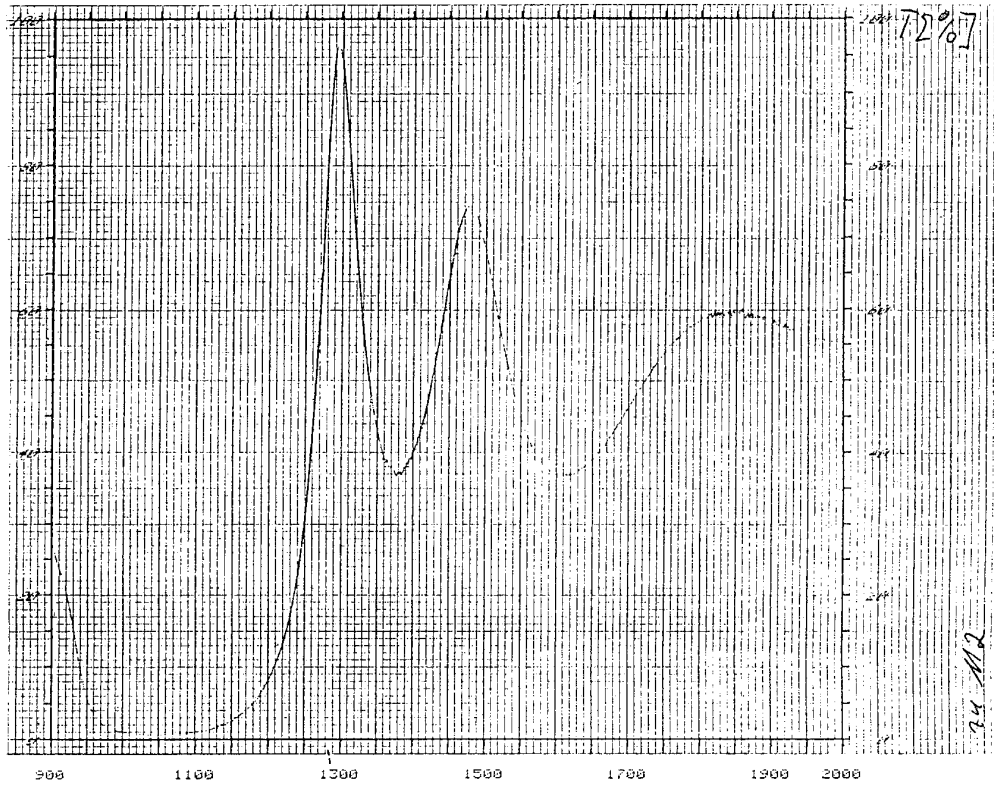
M 1

PERKIN EI

PART NO: BO 126708

PERKIN ELMER

10 126708



5708

PERKIN ELMER

PART NO: BO 126708

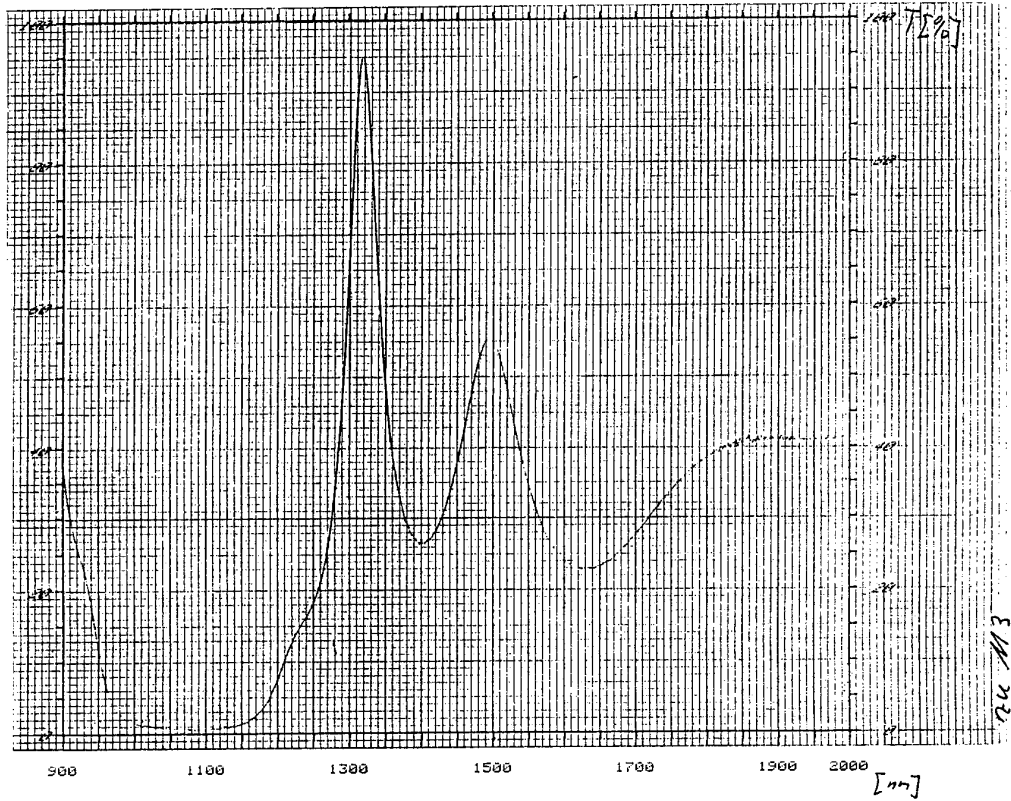
PERKIN ELMER

PERKIN

PART NO. 80 126 708

PERKIN ELMER

PART NO. 80 126 708



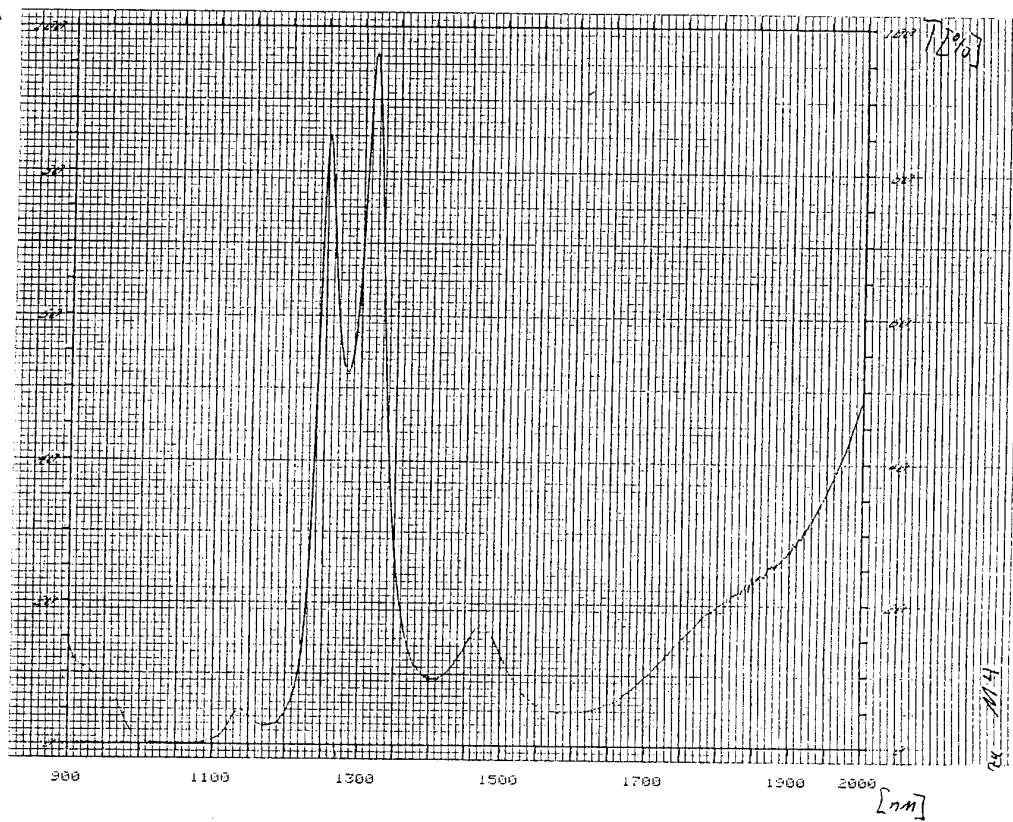
PERKIN-ELMER

PART NO. 80 126 708

PERKIN ELMER

PERKIN ELMER

PART. NO. BO 126 708

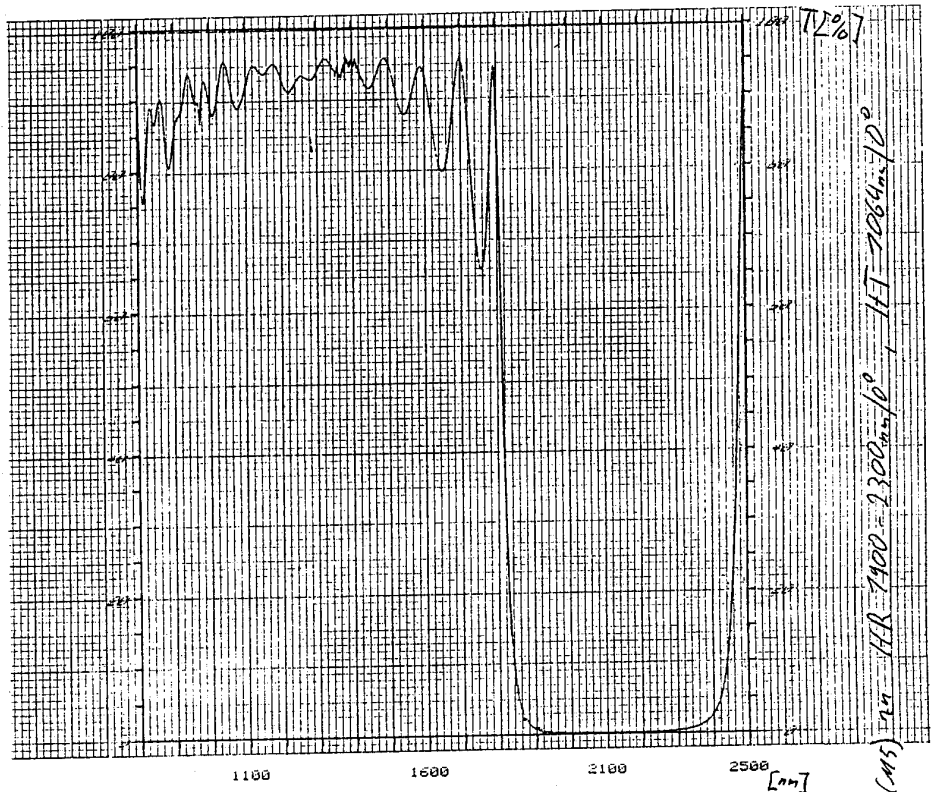


PART. NO. BO 126 708

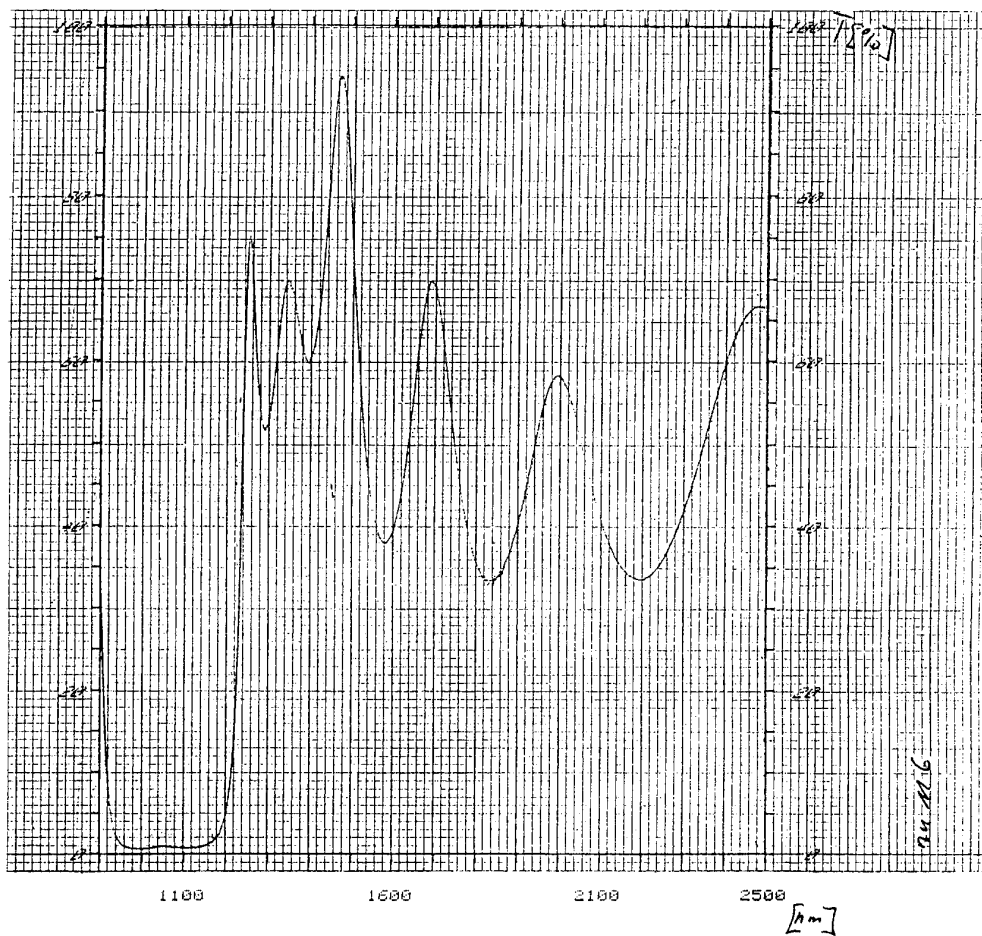
PERKIN ELMER

PA

PERKIN ELMER  
PART. NO. BO 126 708



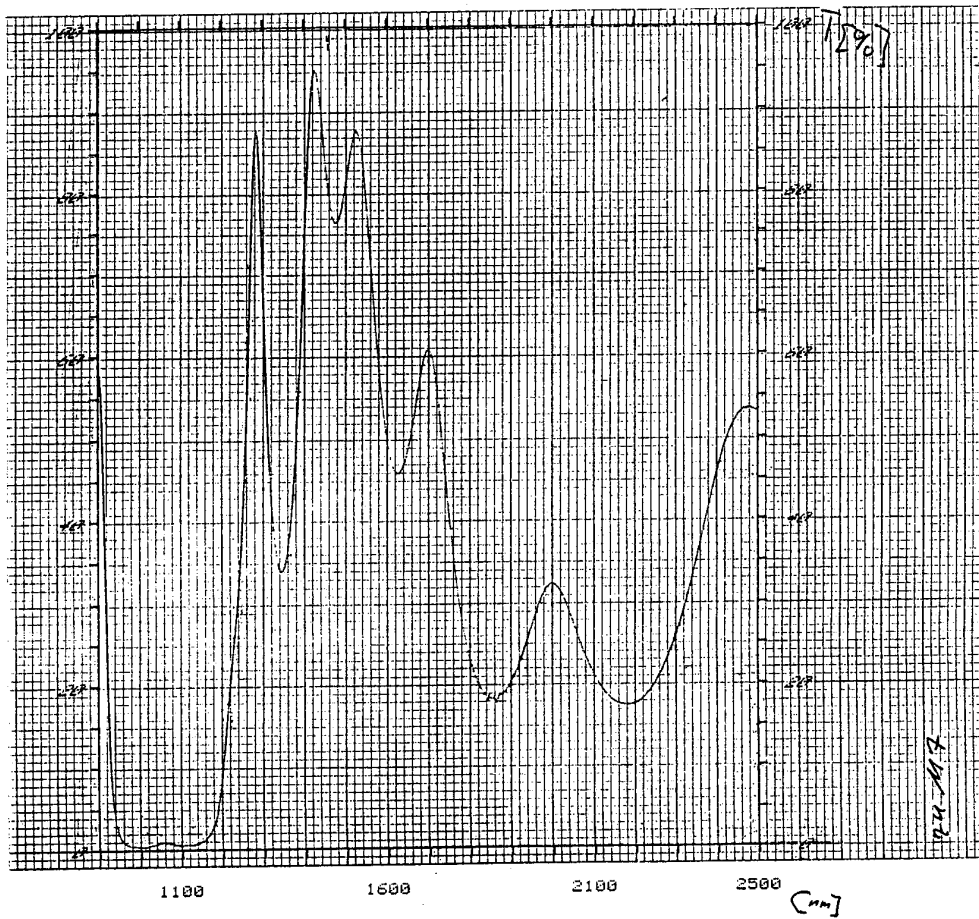
126 708 PERKIN ELMER PART. NO. BO 126 708 PERI



PART NO: BO

PERKIN ELMER

PART NO: BO 126 708



1ER

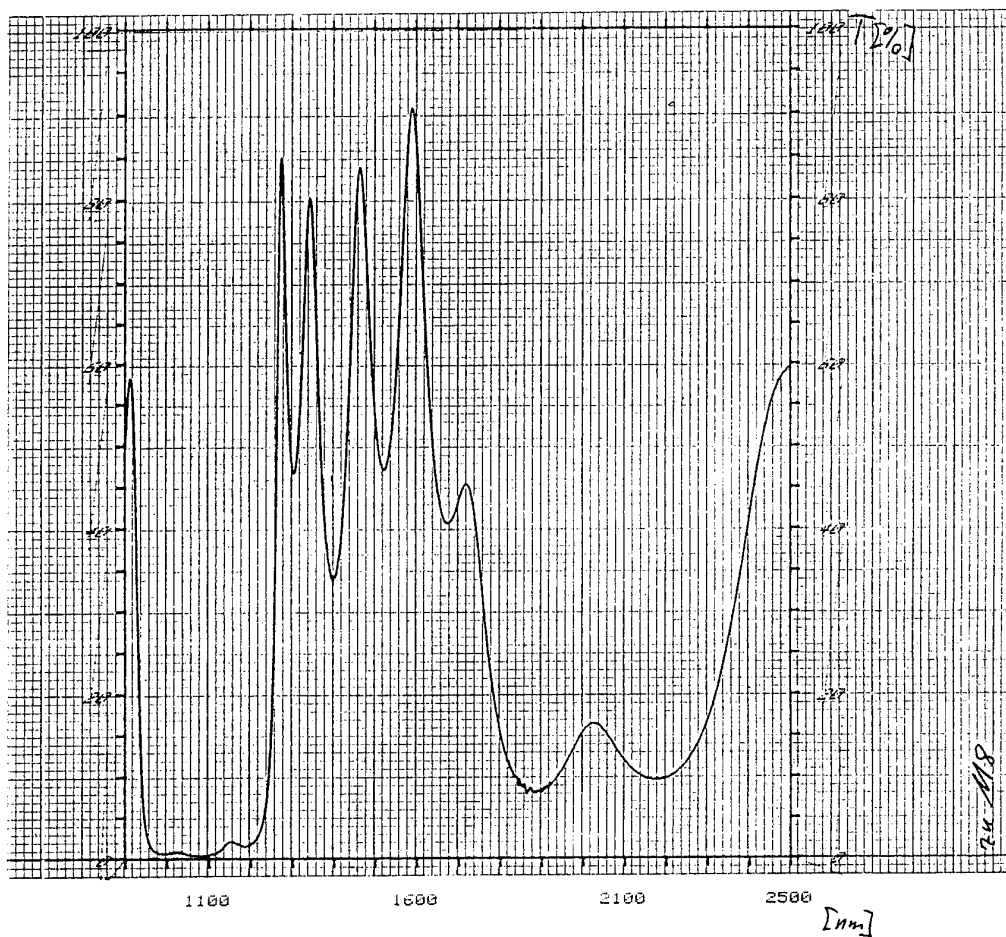
PART NO: BO 126 708

PERKIN ELMER

PER

PART NO.: BO 126 708

PERKIN ELMER



PERKIN ELMER

PART NO.: BO 126 708

PERKIN