



SGS Laboratoire Crépin



PROCEDURE NUMBER :

ME-0177

Department : *SGS LABORATOIRE CREPIN*  
 Service/Section /Agency : *Matières Actives.*

Revision : A (TRAD)

Procedure General

Procedure of organisation

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Procedure Specific

Procedure Technical

Number of Appendices : 2

**DRIOL : ANALYSIS BY HPLC**

**DIFFUSION CONTRÔLÉE**

Revision	Date	Author Name/visa	Inspector Name/visa	Assurance Quality Name/visa	Designation of modification	For approbation Name/visa
A (trad)	04/04/2000	<i>H. LESGOTTE</i>	<i>B. PAUL</i>	<i>I. BEUCHER</i>	English translation ME-0177A	<i>04/04/2000</i> <i>P. BLANFUNE</i>

**DIFFUSION**

FOR APPLICATION : TO STAFF OF UNITE TECHNIQUE MATIÈRES ACTIVES.

FOR INFORMATION :

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Test Method

Date 04/04/2000

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**DRIOL : ANALYSIS BY HPLC/UV**

MÉTHODE CONTRÔLÉE

**1. OBJECT**

This method describes the determination of the Driol content by HPLC/UV.

This method is suitable for fumigant sample and to other formulations or technical material. In this case, it will be necessary to ensure that the method is specific.

A repeatability of the method has been determined on a commercial batch of a fumigant sample (Assay Report n° P200003/0466-1).

**2. OUTLINE OF METHOD**

Driol is determined by reversed phase high performance liquid chromatography (HPLC) with photodiode array detection (PDA – UV detection) using the external standard method.

**3. RECOMMENDATIONS AND CAUTION**

- It's better to prepare sample and standard solutions at the same time and in the same conditions. However, a standard solution which is stored between 0 °C and 5 °C in darkness and whose response factor is every so often followed may be used.
- Caution of toxicity of product; protect yourself with glasses, hoods, gloves, mask.

**4. REAGENTS (OR EQUIVALENTS)**

- Methanol HPLC grade
- Water HPLC grade (Milli Q Plus Millipore)
- Driol certified reference substance

Reference substance used for the assay report n°P200003/0466-1 - Supplier : L.C.B. – CODE 51005 – BATCH N° PAN 14/99 – Purity : 99.5 % (w/w)

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**5. APPARATUS (OR EQUIVALENTS)**

- Ultrasonic bath
- High performance liquid chromatograph(Waters 2690) equipped with a Photodiode array detector (Waters PDA 996).
- Column MERCK LiChroCART 250-4 packed with LiChrospher 100 RP8 (5 $\mu$ m); Ref. : 1.50982.
- Precolumn MERCK LiChroCART 4-4 , LiChrospher 100 RP8 (5 $\mu$ m); Ref.: 1.50956.
- Filter : 0,45  $\mu$ m of porosity (Millex HV Millipore), DURAPORE-PVDF; Ref.: SLHV025NK.
- Volumetric flasks
- Balance at  $\pm$  0.1 mg

RECEIVED BY CONTROLÉE

**6. PROCEDURE**

**6.1. Preparation of calibration solutions**

Prepare calibration solutions in duplicate. Weigh (to the nearest 0.1 mg) about 300 mg of driol ( $m_{\text{standard}}$  mg) into a volumetric flask ( $V_{\text{standard}}$  ml) (100 ml) and dissolve in 80 ml of methanol. Place the volumetric flask in an ultrasonic bath for 10 minutes, allow it to cool to 20°C and fill to the mark with methanol. Dilute 1/20 in methanol.

**6.2. Preparation of sample solution**

Weigh (to the nearest 0.1 mg) 10g of sample fumigant into a volumetric flask and add exactly 400 ml of methanol ( $V_{\text{sample}}$  ml) (400 ml). Place the flask in an ultrasonic bath for 15 minutes, allow it to cool to 20°C. Dilute 1/20 in methanol. Filter the sample solution on 0.45  $\mu$ m filter before HPLC analysis.

**6.3. Determination**



**6.3.1 Chromatographic conditions**

- Column : Column MERCK LiChroCART 250-4 packed with LiChrospher 100 RP8 (5 $\mu$ m)
- Mobile phase : methanol/water (60/40) (V/V)
- Flow rate: 1 ml/min
- Injection volume : 5  $\mu$ L
- Column temperature : 25 °C

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- Detection wavelength : 230 nm
- Retention time : about 5 minutes

All this parameters may be adjusted according to the sample composition to obtain an optimum response, separation and reproducibility.

DI-FUSIÓN CONTROLÉE

### 6.3.2. Determination

- Filter the calibration solution and sample solution
- Inject each calibration solution in duplicate
- Inject each sample solution in duplicate
- The variation coefficient of injection on the areas must be inferior or equalled to 1.5 %

± 25% of variation of RF for the reproducibility of standard solutions injections is acceptable in the same conditions with the same apparatus.

## 7. RESULT EXPRESSION

$$\text{Driol content (\% (w/w))} = \frac{R_{\text{sample}} \times m_{\text{standard}} \times V_{\text{sample}} \times P_{\text{standard}}}{R_{\text{standard}} \times m_{\text{sample}} \times V_{\text{standard}}} \times D$$

With :

- $R_{\text{standard}}$  : area of driol peak in the calibration solution (AU)
- $R_{\text{sample}}$  : area of driol peak in the sample solution (AU)
- $M_{\text{sample}}$  : Mass of sample in the sample solution (mg)
- $V_{\text{sample}}$  : Volume of preparation of the sample solution (mL)
- $M_{\text{standard}}$  : Mass of standard in the calibration solution (mg)
- $V_{\text{standard}}$  : Volume of preparation of the standard solution (mL)
- $P_{\text{standard}}$  : Purity of standard (% (w/w))
- $D$  : Dilution ratio



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## 8 PERFORMANCE OF THE METHOD

The different result and raw data are in the technical unit

### Repetability

Many replicates are realised in the same time on a commercial batch of fumigant sample

Average = 9.9 % (w/w)

S.D. (Standard Deviation) = 0.0495 % (w/w)

R.S.D. (Relative Standard Deviation) = 0.50 %

IF-PLISF-10 CONTROL-LEE

➤ The method is repeatable

### Reproductibility

Many replicates are realised at different time on a commercial batch of fumigant sample

The RSD (Relative Standard Deviation) is less than 2 %.

➤ The method is reproducible

### Specificity

Essay are realised by injection of solvent of dissolution, standard solution and blank matrix

The specificity is validated by the spectrum and the purity plot of the pic

No interference are detected.

➤ The method is specific

Appendix 1 : Chromatogram of a drial standard solution.

Appendix 2 : Chromatogram of a fumigant sample (P200003/0466) solution.

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## APPENDIX 1 : CHROMATOGRAM OF A DRIOL STANDARD SOLUTION.

1 of 1

 Project Name : DIVERS2  
 User Name : System  
 Software Version : 3.05.01

 Report Method Name : RAPPORT CREPIN  
 Current Date : 05/04/00

 RAPPORT SGS LABORATOIRE CREPIN  
 REF CREPIN : P200003\_0466

REVISION CONTROLÉE

 Echantillon : ET DRIOL 1 1/20  
 Flacon : 1  
 Injection : 1  
 Volume d'injection : 5,00 ul  
 Détecteur : 996  
 Durée de l'analyse : 10,0 Minutes

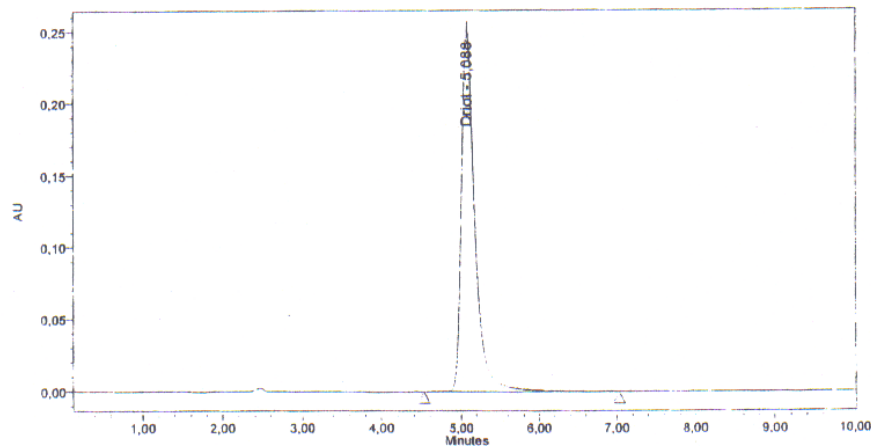
 Type d'échantillon : Unknown  
 Date Acquired : 10/03/00 11:47:11  
 Acq Method Set : DRIOL6040230  
 Processing Method : DRIOL  
 Date Processed : 10/03/00 12:24:1  
 Longueur d'onde d'analyse : PDA 230,0 nm


Table de résultats

Nom	RT (min.)	Aire (µV*sec.)	Hauteur (µV)
1 Driol	5,088	3119806	252934

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**APPENDIX 2 : CHROMATOGRAM OF FUMIGANT SAMPLE SOLUTION.**

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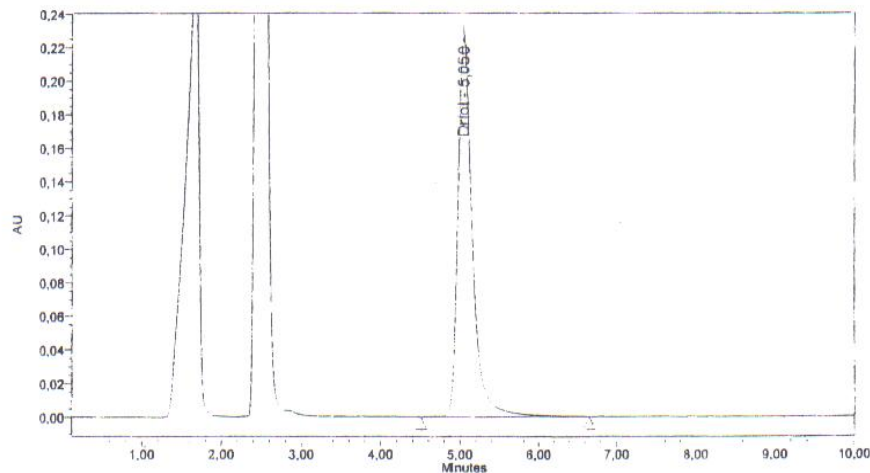
 Project Name DIVERS2  
 User Name System  
 Software Version 3.05.01

 Report Method Name RAPPORT CREPIN  
 Current Date 05/04/00

**RAPPORT SGS LABORATOIRE CREPIN**  
**REF CREPIN : P200003\_0466**

DIFFUSION CONTRÔLÉE

 Echantillon : ECH 466-1A  
 Flacon : 4  
 Injection : 1  
 Volume d'injection : 5,00 ul  
 Détecteur : 996  
 Durée de l'analyse : 10,0 Minutes

 Type d'échantillon : Unknown  
 Date Acquired : 10/03/00 14:48:04  
 Acq Method Set : DRIOL6040230  
 Processing Method : DRIOL  
 Date Processed : 10/03/00 15:19:1  
 Longueur d'onde d'analyse : PDA 230,0 nm

**Table de résultats**

Nom	RT (min.)	Aire (µV*sec.)	Hauteur (µV)
1 Driol	5,050	2900186	229420

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