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Analytical Report N°. 14-WES0002

Determination of Dinotefuran in Bait samples formulations by LC/UV

TAMI order N°: 104062
Customers order N°: 330165794
Purchase order N°: WEST.6.3.17
WBS N°: TS-01.00765
Samples characterization: brown viscous liquid
Number of samples: 10
Arrival date: 03.04.17
Analyses dates: 18-20.04.17

| | Analysts name | Remarks |
|---------------------|--------------------------------|----------------|
| LC/UV: | Carmela Carmi , Yulia Kilchtok | |
| Written by : | Carmela Carmi | |
| Supervisor : | Igal Gozlan | |

*Declaration: the analyses report are related only to the samples which were obtained for analysis

Analytical Report

Samples of Bait formulations (table 1) with the active ingredient Dinotefuran (fig. 1) were obtained in order to perform LC/UV quantitative analysis.

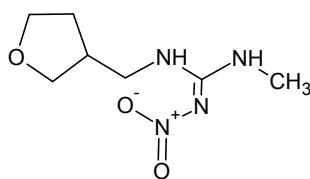
The CIPAC LC/UV method was modified for Dinotefuran concentration in samples formulations.

Calculations were carried out using UV detector at $\lambda=270$ nm (table 1).

The LOQ value is 25 ppm (fig. 2).

The LOD value is 5 ppm (fig. 2).

Fig. 1: Dinotefuran structure



1-methyl-2-nitro-3-(tetrahydrofuran-3-ylmethyl)guanidine

Molecular Formula: $C_7H_{14}N_4O_3$
Monoisotopic Mass: 202.10659 Da

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Fig. 1: Dinotefuran structure

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Fig. 4: LC/UV chromatograms of samples "F-1, 2, 3, 4 and 5" - Fresh formulation

Table 1: Samples information and quantitative results

| No. | Sample name | Description | TAMI name | Dinotefuran Result (ppm) | LC/UV (HPLC-7) Data file name: LMS4836\ DINOTEFURAN000.D |
|-----|--|--------------------------------------|------------------------|--------------------------|--|
| 1 | AS#1 | Accelerated storage, 8 weeks at 40°C | 2017-104062/1 LMS4836 | 746 | 9-10 |
| 2 | AS#2 | | 2017-104062/2 LMS4837 | 772 | 11-12 |
| 3 | AS#3 | | 2017-104062/3 LMS4838 | 690 | 13-14 |
| 4 | AS#4 | | 2017-104062/4 LMS4839 | 767 | 15-16 |
| 5 | AS#5 | | 2017-104062/5 LMS4840 | 759 | 17-18, 48 |
| 6 | F#1 | Fresh formulation | 2017-104062/6 LMS4841 | 677 | 20-21 |
| 7 | F#2 | | 2017-104062/7 LMS4842 | 694 | 22-23 |
| 8 | F#3 | | 2017-104062/8 LMS4843 | 718 | 24-25 |
| 9 | F#4 | | 2017-104062/9 LMS4844 | 665 | 26-27 |
| 10 | F#5 | | 2017-104062/10 LMS4845 | 699 | 28-29, 50 |
| 11 | Dinotefuran STD 98.5%, Sigma-Aldrich P/N#32499-50MG Lot#BCBR7776V, Exp.04/2022 (white powder) | - | 2017-104062/11 LMS4846 | - | 31-32 37-46 |

Fig. 2: LC/UV chromatograms of Blank and Dinotefuran STD (C=0.0001 and 0.0005 mg/ml in water)

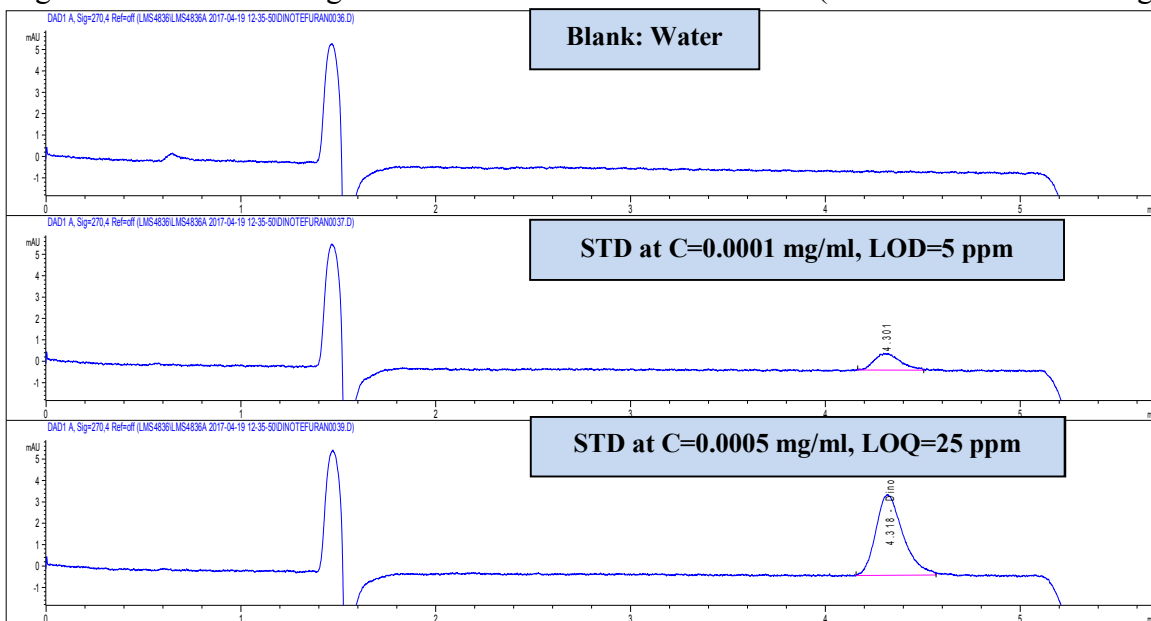


Fig. 3: LC/UV chromatograms of samples "AS-1, 2, 3, 4 and 5" - Accelerated storage, 8 weeks at 40°C

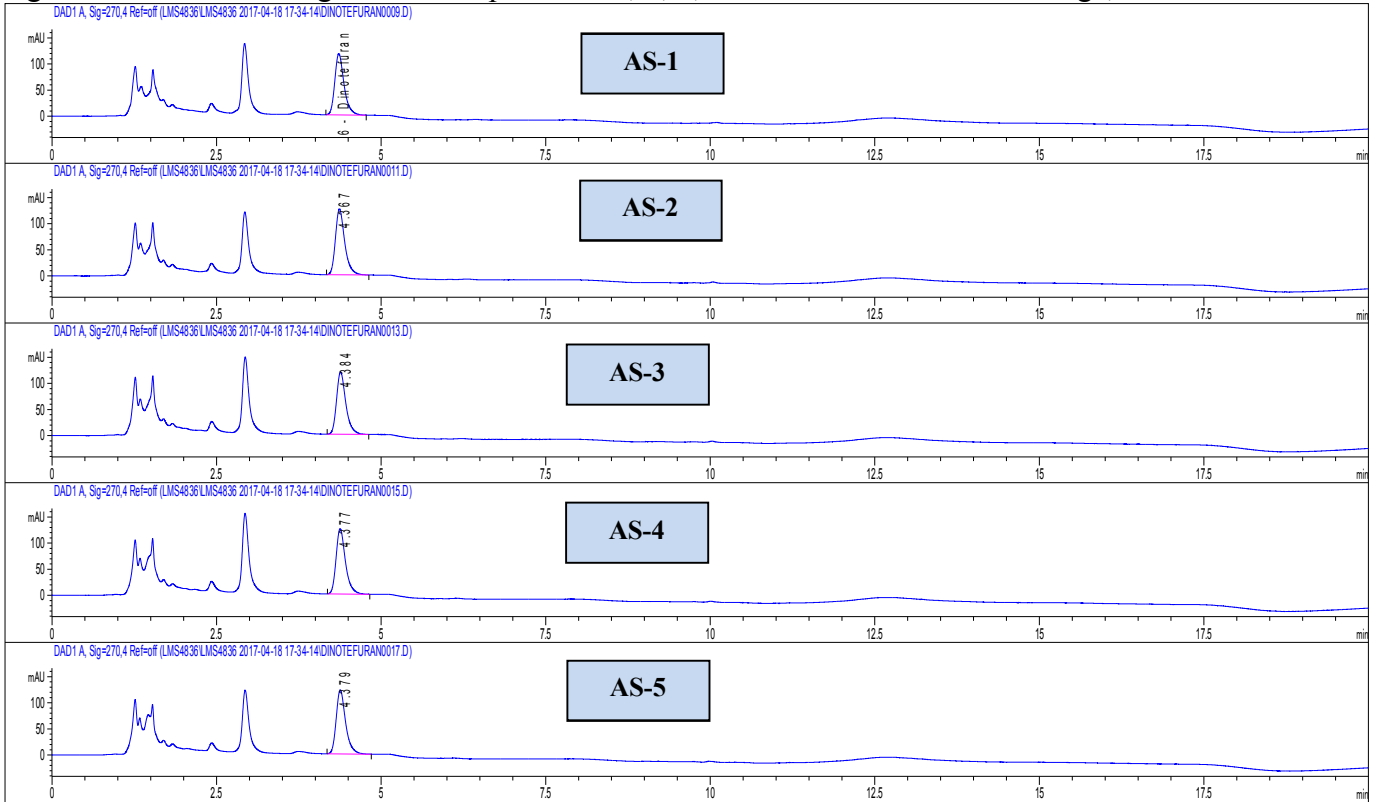
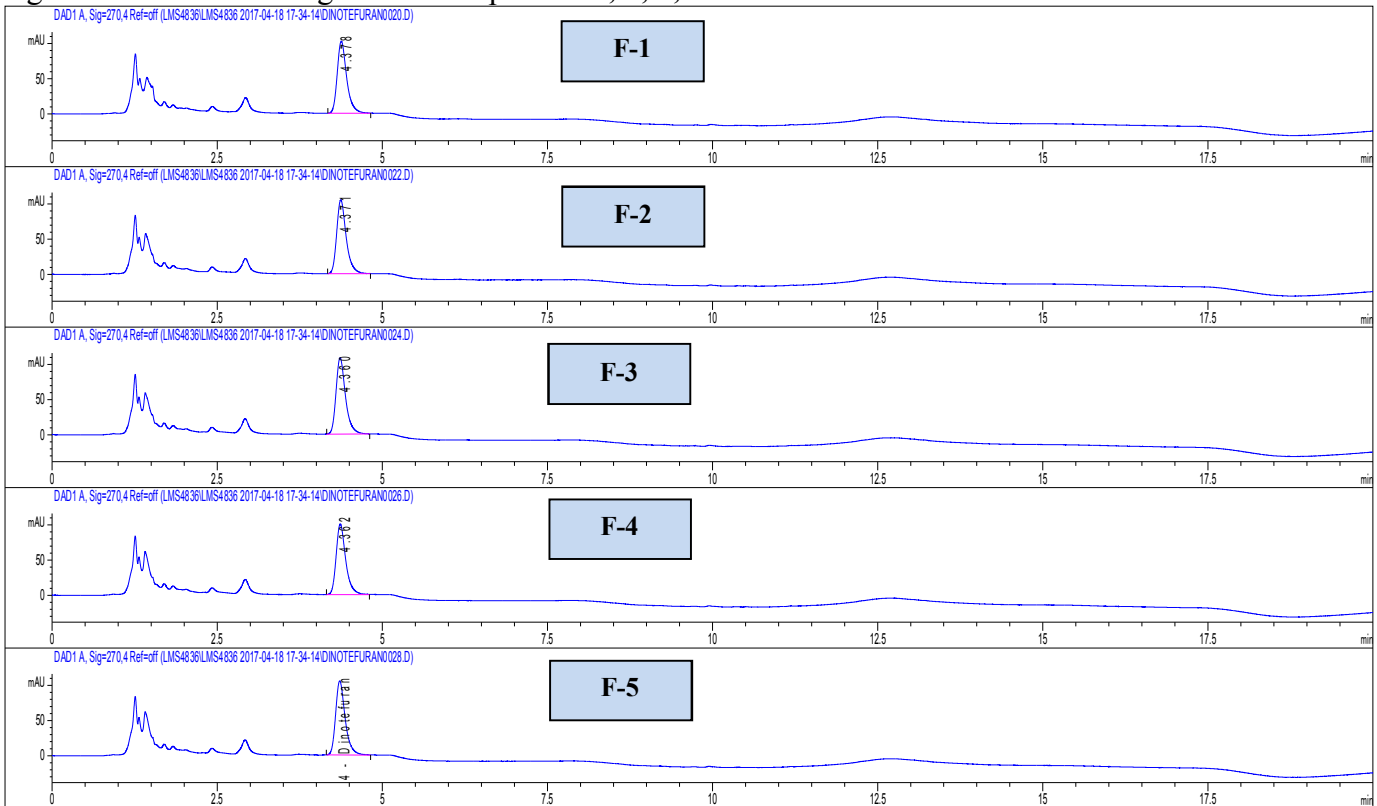


Fig. 4: LC/UV chromatograms of samples "F-1, 2, 3, 4 and 5" - Fresh formulation



Materials, Method and Instrumentation

Methanol: Bio lab, HPLC supra-gradient, Cat. No. 001368350100 (MeOH)

Water: Bio lab, HPLC Grade, Cat. No. 002321060200 (H₂O)

LC/UV (HPLC-7, Critic#32-150-05)

Pumping system: Agilent-Technologies model 1200 series

UV Detector: Agilent-Technologies model 1100 series

Chromatographic Conditions

Method name: LMS4836.M

Wavelength: λ=270 nm

Column: Waters, SymmetryShield RP8 5μm, 150mm*2.1mm ID*5μm,
Part#WAT094245, Lot#0168370241, Serial#01683702417410
+ Pre Column C8

Mobile phase: [C] H₂O: [D] MeOH

Gradient profile:

| <u>Time</u> (min) | <u>H₂O</u> (%) | <u>MeOH</u> (%) | <u>Flow</u> (ml/min) |
|----------------------|------------------------------|--------------------|-------------------------|
| 0 | 90 | 10 | 0.3 |
| 5 | 90 | 10 | 0.3 |
| 5.1 | 90 | 10 | 0.5 |
| 10 | 10 | 90 | 0.5 |
| 15 | 10 | 90 | 0.5 |
| 20 | 90 | 10 | 0.5 |

Post time: 10 min

Injection volume: 10 μl

Column temperature: 40⁰C

Sample temperature: 25⁰C

Rt of Dinotefuran: 4.3 min

Standard preparation

1. Weight 25 mg of Dinotefuran STD into 25 ml volumetric flask and fill up to line with MeOH (solution A) (C=1 mg/ml).
2. Transfer 0.1 ml solution A into 10 ml volumetric flask and fill up to line with water (solution B) (C=0.01 mg/ml).
3. Transfer 0.13 ml solution A into 10 ml volumetric flask and fill up to line with water (solution C) (C=0.013 mg/ml).
4. Transfer 0.15 ml solution A into 10 ml volumetric flask and fill up to line with water (solution D) (C=0.015 mg/ml).
5. Transfer 0.1 ml solution B into 10 ml volumetric flask and fill up to line with water (solution E) (C=0.0001 mg/ml) (LOD).
6. Transfer 0.25 ml solution B into 5 ml volumetric flask and fill up to line with water (solution F) (C=0.0005 mg/ml) (LOQ).
7. Inject each standard solution twice to the LC/UV.

Sample preparation

1. Insert samples into stirred water bath at ~40°C in order to mix well and homogenize sample before weight.
2. Weight 2 gr of sample into 100 ml volumetric flask and fill up to line with hot deionized water at ~40°C (C=20 mg/ml).
3. Inject each sample solution twice to the LC/UV.

Supervisor: I. Gozlan Ph.D. – Head of Analytical Instrumentation Department

Signature: _____

