FINAL STUDY REPORT

Study Title

Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)

Test Guidelines

Canadian PMRA Regulatory Directive DIR98-02 and 2010-05
SANCO 3029/99 rev. 4. 11/07/00

Study Director and Report Author
Testing Facility
Sandra J. Carringer
The Carringers, Inc.
1003 Palace Ct.
Apex, NC 27502

Study Completion Date

June 26, 2014

Sponsor

Cheminova A/S
P. O. Box 9
DK-7620, Lemvig
Denmark

Performing Laboratories

Analytical Phase

Morse Laboratories, LLC
1525 Fulton Avenue
Sacramento, CA 95825
Laboratory Project ID: 69869

ABC Laboratories, Inc.
7200 E. ABC Lane
Columbia, MO 65202
Laboratory Project ID: 69869

Field Phase

See Page 10 for Field Test Sites

TCI Study Number

TCI-13-366

Total Number of Pages

245

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

Study Title: Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L (2013)

No claim of CBI is made for any information contained in this document on the basis of the definition of CBI in the PCPA 2002.

Company: Cheminova A/S

Company Representative: Paul Whatling

Title: Director of Regulatory Science
Cheminova, Inc.
Agent for Cheminova A/S

Date: June 26, 2014

Signature: Paul Whatling

These data are the property of Cheminova A/S, and, as such, are considered to be confidential for all purposes other than compliance with PCPA 2002. Submission of these data in compliance with PCPA 2002 does not constitute a waiver of any right to confidentiality, which may exist under any other statute or any other country.

GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

This study was conducted in compliance with U.S. EPA FIFRA (40 CFR Part 160) Good Laboratory Practice Standards, which are consistent with the OECD Principles of Good Laboratory Practice (ENV/MC/CHEM(98)17).

The following is a detailed description of all differences between the practices used in the study and those required by 40 CFR 160. None of the items listed impact the quality and integrity of the study.

1. Weather data were generally not collected following strict adherence to Good Laboratory Practice Standards. [40 CFR 160.1]

2. Maintenance pesticides, test system maintenance, pesticide histories, percent slope of the test sites, seeding of crop, sample weights, GPS coordinates, crop growth stages, and soil characterization data were not always obtained under GLPs for field test sites. [40 CFR 160.1]

3. In Trials TCI-13-366-01 and -06 notebooks, a correction on the ICMS Test and Reference Item Use Log was not made according to GLPs. For Trial TCI-13-366-02, data on page 19 of the field trial notebook for the original trial site were not initialed and dated at the time of entry and on Form 28B of the notebook, data were not initialed at the time of entry. For Trials TCI-13-366-04 and -10, an entry on page 1 of the notebook was not dated, and some entries on page 12 of Trial -04 field trial notebook were not initialed and dated at the time of entry. Additionally, some harvesting information was recorded late on pages 21, 22, and 24 of the field trial notebook. For Trial TCI-13-366-07, some entries on page 20 were dated, but not initialed at the time of entry. For Trial TCI-13-366-10, data recorded on ICMS calibration form were not initialed and dated at the time of entry. For Trial TCI-13-366-11, a non-GLP timing device was used to measure the sprayer pass times for calibration and application. [40 CFR 160.130(e)]

Study Director:

Sandra J. Carringer
The Carringers, Inc.

26 Jun 2014
Study Completion Date

Sponsor/Submitter:

Paul Whatling
Director of Regulatory Science, Cheminova, Inc.
Agent for Cheminova A/S

June 26, 2014
Date

QUALITY ASSURANCE UNIT STATEMENT OF INSPECTIONS

The Quality Assurance Unit of The Carringers, Inc. conducted inspections and reported to the Study Director and the Testing Facility Management as indicated below:

Trial Site/Facility	Type of Inspection	Date of Inspection	Date Reported to Study Director	Date Reported to Testing Facility Management
All	Protocol	25-Apr-13	25-Apr-13	25-Apr-13
All	Field Data	13-16-Jan-14	16-Jan-14	16-Jan-14
All	Draft Study Report	17, 20-21-May-14	21-May-14	21-May-14
All	Final Study Report	25-Jun-14	25-Jun-14	25-Jun-14

The Quality Assurance Unit of ABC Laboratories, Inc. conducted inspections and reported to the Study Director and the Testing Facility Management as indicated below:

Type of Inspection	Date of Inspection	Date Reported to Study Director	Date Reported to Testing Facility Management
In Process	18-Mar-14	19-Mar-14	19-Mar-14
Sample Receival Information	24-Jul-13, 30-Aug-13	14-Mar-14	14-Mar-14
Raw Data	24-30-Apr-14	21-May-14	21-May-14
Raw Data	21-27-Apr-14	22-May-14	22-May-14
Draft Analytical Report	01-02-May-14	27-May-14	27-May-14
Draft Analytical Report and Raw Data	09-10-Jun-14	25-Jun-14	25-Jun-14
Final Analytical Report	24-Jun-14	25-Jun-14	25-Jun-14

QUALITY ASSURANCE UNIT STATEMENT OF INSPECTIONS (continued)

The Quality Assurance Unit of each field test site conducted inspections and reported to the Study Director and the Testing Facility Management as indicated below:

Trial Site	Type of Inspection	Date of Inspection	Date Reported to Study Director	Date Reported to Testing Facility Management
TCI-13-366-01	Application	29-May-13	03-Jun-13	03-Jun-13
	Field Notebook/Raw Data	27-Sep-13	04-Oct-13	04-Oct-13
TCI-13-366-02	Application (Original)	03-Jun-13	17-Jun-13	17-Jun-13
	Application (Restart)	11-Jun-13	17-Jun-13	17-Jun-13
	Field Notebook/Raw Data	03-Dec-13	18-Dec-13	18-Dec-13
TCI-13-366-03	Sampling	13-Sep-13	19-Sep-13	19-Sep-13
	Field Notebook/Raw Data	05-Dec-13	18-Dec-13	18-Dec-13
TCI-13-366-04	Sampling	07-Sep-13	08-Oct-13	08-Oct-13
	Field Notebook/Raw Data	12-Dec-13	18-Dec-13	18-Dec-13
TCI-13-366-05	Application	28-May-13	29-May-13	29-May-13
	Field Notebook/Raw Data	27-Nov-13	02-Dec-13	02-Dec-13
TCI-13-366-06	Application	25-May-13	29-May-13	29-May-13
	Field Notebook/Raw Data	05-Nov-13	06-Nov-13	06-Nov-13
TCI-13-366-07	Application	25-May-13	29-May-13	29-May-13
	Field Notebook/Raw Data	27-Sep-13	04-Oct-13	04-Oct-13
TCI-13-366-08	Application	25-May-13	29-May-13	29-May-13
	Field Notebook/Raw Data	23-Sep-13	26-Sep-13	26-Sep-13
TCI-13-366-09	Sampling	13-Sep-13	08-Oct-13	08-Oct-13
	Field Notebook/Raw Data	13-Dec-13	18-Dec-13	18-Dec-13
TCI-13-366-10	Calibration/Application	12-Jun-13	17-Jun-13	17-Jun-13
	Field Notebook/Raw Data	13-Dec-13	18-Dec-13	18-Dec-13
TCI-13-366-11	Application	28-May-13	10-Jun-13	10-Jun-13
	Field Notebook/Raw Data	13-Dec-13	18-Dec-13	18-Dec-13
TCI-13-366-12	Sampling	19-Sep-13	08-Oct-13	08-Oct-13
	Field Notebook/Raw Data	13-Dec-13	18-Dec-13	18-Dec-13

Ralph P. Rose

Ralph P. Rose
The Carringers, Inc.
Manager, Quality Assurance

25 June 2014
Date

CERTIFICATION OF AUTHENTICITY

We, the undersigned, hereby declare that this study was performed under our supervision according to the procedure described herein, and that this report provides a true and accurate record of the results obtained.

Report By:

Sandra J. Carringer
Study Director
The Carringers, Inc.

26 JUN 2014
Date

Approved By:

Richard D. Carringer, Ph.D.
Testing Facility Management
The Carringers, Inc.

25-JUNE-2014
Date

ABBREVIATIONS AND SYMBOLS

ai	active ingredient
A	acre
ACDS	Agricultural Chemicals Development Services, Inc.
Avg	average
°C	degrees Celsius
CAN	Canada
DAA	days after application
DBH	days before harvest
EPA	Environmental Protection Agency
°F	degrees Fahrenheit
FIFRA	Federal Insecticide, Fungicide, and Rodenticide Act
g	gram
gal.	gallon
GLP	Good Laboratory Practice
HPLC	high-performance liquid chromatography
ID	identification
lb	pound
l	liter
LOD	limit of detection
LOQ	limit of quantitation
µg	microgram
µL	microliter
mL	milliliter
MS/MS	mass spectrometric/ mass spectrometric
M. W.	molecular weight
n	number of replicates
NA	not applicable
ND	not detected; no observable chromatographic response
No.	number
PHI	preharvest interval
PMRA	Pesticide Management Regulatory Agency
ppm	parts per million
RAC	raw agricultural commodity
Ref.	reference
SD	standard deviation
SOP	standard operating procedure
Trt	treatment
U.S.	United States
USA	United States of America
UTC	untreated control
UV	ultraviolet
/	per (as in gallons per acre)
<	less than
>	greater than

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF APPENDICES

STUDY INFORMATION PAGE

TCI Study Number:	TCI-13-366
Study Title:	Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)
Purpose:	This study was conducted to determine the magnitude and decline of residues of clomazone in or on canola raw agricultural commodities (RAC) following one pre-emergence application of Clomazone 360 g/L CS at 0.42 kg ai/ha. The magnitude of residues of clomazone in or on canola processed commodities (PC) was also to be determined following one pre-emergence application at an exaggerated 5× rate if residues were found above the limit of quantitation (LOQ). All residues were <LOQ; therefore, the processing phase was not conducted.
Test Substance:	Clomazone 360 g/L CS 2-(2-chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one or 2-(2-chlorobenzyl)-4,4-dimethylisoxazolidin-3-one (IUPAC)
Study Director/Author & Testing Facility:	Sandra J. Carringer The Carringers, Inc. 1003 Palace Ct. Apex, NC 27502 Tel: 919-387-0825 Fax: 919-387-4161 e-mail: scarringer@carringers.com
Study Sponsor:	Cheminova A/S P.O. Box 9 DK-7620, Lemvig Denmark
Sponsor's Rep.:	Faith Womack Cheminova, Inc. 1600 Wilson Boulevard, Suite 700 Arlington, VA 22209
Study Initiation Date:	May 1, 2013 (protocol signed)
Experimental Start Date:	May 25, 2013 (first application)
Experimental Completion Date:	May 19, 2014 (last instrumentation date)
Study Completion Date:	See date of Study Director's signature on page 3

STUDY INFORMATION PAGE (continued)

The Carringers, Inc. Personnel
Assistant Project Manager: Pamela Bruss

Field Facilities

Laboratory Project ID / Trial No.	Principal Investigator[1]	Company	Facility Address
TCI-13-366-01 TCI-13-366-06 TCI-13-366-07 TCI-13-366-08	Kelly Tiller	ICMS, Inc.	Box 67 Station Main 2375 Saskatchewan Ave. E. Portage la Prairie, Manitoba, Canada R1N 3B2 Tel: 204-857-6609 Fax: 204-239-4478 e-mail: tiller@icms-inc.com
TCI-13-366-02 TCI-13-366-03	Greg Whittington	ICMS, Inc.	334 Packham Ave. Saskatoon, Saskatchewan Canada S7N 2T1 Tel: 306-956-3855 Fax: 306-956-3856 e-mail: whittington@icms-inc.com
TCI-13-366-04 TCI-13-366-09 TCI-13-366-10 TCI-13-366-11 TCI-13-366-12	Dean Ngombe	ICMS, Inc.	334 Packham Ave. Saskatoon, Saskatchewan Canada S7N 2T1 Tel: 306-956-3855 Fax: 306-956-3856 e-mail: ngombe@icms-inc.com
TCI-13-366-05	Taryn Williams	ICMS, Inc.	Box 3270 54474 Range Road 215 Fort Saskatchewan, Alberta Canada T8L 2T2 Tel: 708-992-7983 Fax: 780-992-8499 e-mail: willliams@icms-inc.com

[1]Other field personnel are listed in the detailed study site information (Appendix B).

STUDY INFORMATION PAGE (continued)

Processing Facility[1]

Trial No.	Principal Investigator	Company	Facility Address
TCI-13-366-06	Dick Dusek	GLP Technologies	22723 State Highway 6 South Navasota, TX 77868 Tel: 936-825-2184 Fax: 936-825-7929 e-mail: ddusek@glptech.net

[1]Samples were shipped and held at GLP Technologies for possible processing; however, the processing phase was not conducted since all residues were <LOQ.

Analytical Facility[1]

Trial No.	Principal Investigator[2]	Company	Facility Address
All Trials	Carol Rodgers	ABC Laboratories, Inc. 01-Nov-13 to Present	7200 E. ABC Lane Columbia MO 65202 Tel: 573-777-6054 Fax: 573-777-6033 e-mail: rodgersc@abclabs.com
	Jeri Willoh	Morse Laboratories, LLC 01-May-13 to 10-Oct-13[3]	1525 Fulton Avenue Sacramento, CA 95825 Tel: 916-481-3141 Fax: 916-481-2959 e-mail: willohj@morselabs.com

[1] Morse was a wholly owned subsidiary of ABC Laboratories. In the fall of 2013, the Morse site in California was closed. This study was transferred to the ABC Laboratories site in Missouri.

[2] Other analytical personnel are listed in the analytical summary report (Appendix C).

[3] During the interim between Principal Analytical Investigator's (i.e., October 11, 2013 to October 31, 2013), analytical phase oversight was provided by Management.

CITATION

Carringer, S. [06/26/2014]. Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013). TCI Study Number TCI-13-366. Unpublished study prepared by The Carringers, Inc., 245 pages.

STUDY PURPOSE

This study was conducted to determine the magnitude and decline of residues of clomazone in or on canola raw agricultural commodities (RAC) following one pre-emergence application of Clomazone 360 g/L CS at 0.42 kg ai/ha. The magnitude of residues of clomazone in or on canola processed commodities (PC) was also to be determined following one pre-emergence application at an exaggerated 5× rate if residues were found above the limit of quantitation (LOQ). Residues were <LOQ in all samples; therefore, the processing study was not conducted.

EXECUTIVE SUMMARY

Field trial data have been generated for clomazone on canola. Twelve canola trials were conducted in Canada in PMRA Zones/Regions 5 (one trial), 7 (two trials), and 14 (nine trials) during the 2013 growing season. The number and geographic locations of field trials are adequate to support a Canadian tolerance for residues of clomazone in canola.

One untreated control (Trt 1) and one treated plot (Trt 2) were established at each test site. At Trial -06, two additional treated plots (Trt 3 and Trt 4) were established to produce seed for the possible processing phase. The 1× (Trt 2) rate treated plots received one pre-emergence soil-applied broadcast application of Clomazone 360 g/L CS at the maximum proposed label use rate of 0.42 kg ai/ha ±5%. At Trial -06, the 3× (Trt 3) and 5× (Trt 4) rate treated plots received one pre-emergence broadcast application at 1.26 and 2.1 kg ai/ha ±5%, respectively. Applications were made after the canola was planted, but before emergence. The spray volume ranged from 47 to 104 liters per hectare (l/ha) with three trials selected to target a low spray volume of 47 l/ha ±10%. Spray additives were not included in the spray mixes.

At all test sites, one untreated control RAC sample and duplicate treated canola RAC samples were cut/harvested from each treated plot at maturity 90 to 122 days after the application (DAA). Some trials cut and threshed samples on the same day; however, at four sites (including one decline trial), the crop was cut/swathed and dried for 5 to 14 days and then threshed. Two trials (Trials -04 and -05) also collected treated samples 3-4, 7-8, 14-15, and 21 days after normal harvest maturity to assess residue decline. At Trial -06, duplicate RAC samples were also collected from the 5× rate treated plot (Trt 4), and one untreated control (Trt 1) and one 5× (Trt 4) rate treated bulk grain samples were also collected for possible processing. Since adequate seed were collected from Trt 4, seed from Trt 3 were not collected.

The RAC samples for residue analysis weighed at least 0.5 kg except for the treated samples from Trial -11 which weighed 0.20 and 0.25 kg due to poor crop emergence and crop stand. This did not impact the study since the crop that was present at this site was healthy and normal, and the sample weight collected was adequate for analysis. The bulk grain samples for the processing phase weighed at least 30 kg. All samples were collected without bias from 12 locations in the plots avoiding the edges of the treated plot. Samples were placed in freezer storage within 3.13

hours after collection. Frozen RAC samples were shipped via ACDS freezer trucks with Morse Laboratories, LLC as the original destination; however, due to the announced closing of the Morse facility in Sacramento, the destination was changed in transit and the samples were delivered to ABC Laboratories, Inc. for analysis.

The analytical method used for the determination of clomazone in canola RAC samples was the method found in Eurofins-GAB GmbH Study Code 20061401/01-RVP Final Report, entitled "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape," dated January 17, 2007, with ABC Laboratories method modifications, dated April 23, 2014. The method was validated according to SANCO guidelines on canola seed prior to sample analysis. Concurrent procedural control samples were analyzed in conjunction with each analytical set for quality control purposes. These samples were extracted and analyzed according to the same procedure as the study samples. The validated limit of quantitation (LOQ) was 0.02 ppm for clomazone and the limit of detection (LOD) was estimated to be 0.007 ppm (1/3 the LOQ). The overall range, mean, standard deviation, and relative standard deviation for method validation and concurrent procedural recoveries are summarized below:

Summary of Method Recoveries of Clomazone from Canola				
Matrix	**Fortification Level in ppm (mg/kg)**	**Sample Size (n)**	**Range of Recoveries (%)**	**Mean (%) ± std. dev. (RSD)**
Method Validation Fortifications - Clomazone				
Canola Seed	0.02	5	66-95	81 ± 12 (14)
	0.5	5	73-92	81 ± 7.0 (8.6)
	0.02-0.5	10	66-95	81 ± 9.0 (11)
Concurrent Fortifications - Clomazone				
Canola Seed	0.02-0.5	12	68-112	95 ± 12 (12)

Storage Stability: The canola RAC samples were stored frozen (below freezing at the field sites and at -25 °C to -10 °C at the analytical laboratory) for 166 to 203 days. The frozen storage stability of clomazone residues in canola seed has been demonstrated for at least 6 months in a previous study ((Fiedler E. (2007): Determination of the Storage Stability of Clomazone in Potatoes and Oil Seed Rape at approximately -20 °C. Eurofins-GAB GmbH, Niefern-Öschelbronn, DEU Study No.: 20054075/01-RSS; CHA Doc. No.: 83 CAZ). Additional freezer storage stability testing initiated in this study has been transferred to another study for completion and reporting. The data generated to date indicate that residues should not be impacted by 6.7 months of freezer storage.

Residue Levels in RAC Fractions: The results from these trials show that following one pre-emergence application of Clomazone 360 g/L CS at the maximum proposed label use rate of 0.42 kg ai/ha ± 5%, residues of clomazone ranged from not detected (ND) to <LOD in all canola samples collected 90 to 122 DAA. The results from these trials show that following one pre-emergence application of Clomazone 360 g/L CS at a 5× exaggerated rate application of 2.1 kg ai/ha ± 5%, residues of clomazone ranged from <LOD to <LOQ in the canola samples collected 122 DAA.

Residue Decline Data: Two trial sites (Trials -04 and -05) collected additional treated samples 3-4, 7-8, 14-15, and 21 days after normal harvest maturity. At all sampling events at both sites, residues in the seed were <LOD indicating that residues do not increase with longer preharvest intervals.

COMPLIANCE

The study was conducted according to the GLP standards of 40 CFR Part 160 and/or OECD Principles of Good Laboratory Practice. Signed and dated GLP, Quality Assurance, and Data Confidentiality statements are provided. No GLP deviations occurred which impact the validity of the study.

A. BACKGROUND INFORMATION

The Clomazone 360 g/L CS test substance used in this study contained a nominal 360 g ai/L clomazone in a capsule suspension (CS) formulation. Clomazone 360 g/L CS is a herbicide currently being developed by Cheminova A/S for agricultural use in controlling weeds in a variety of crops.

Based on EPA's review of available plant metabolism data, the Agency concluded that the residue of concern in food/feed items is clomazone, and the residue of concern for tolerance enforcement is clomazone *per se*. As such, all plant magnitude of the residue studies are required to measure residue of this analyte in all matrices.

The purpose of this study was to determine the magnitude and decline of residues of clomazone in or on canola raw agricultural commodities (RAC) following one pre-emergence application of Clomazone 360 g/L CS at 0.42 kg ai/ha. The magnitude of residues of clomazone in or on canola processed commodities (PC) was also to be determined following one pre-emergence application at an exaggerated 5× rate if residues were found above the limit of quantitation (LOQ). Residues were <LOQ in all samples; therefore, the processing study was not conducted.

This report provides a summary of the procedures followed during the field and analytical portions of this study. It also reports the results of the sample analyses and draws conclusions from these results. The results from this study may be used to establish Maximum Residue Limits for clomazone in/on canola raw agricultural and processed commodities in Canada.

See Table 1 for the test compound nomenclature and Table 2 for the physicochemical properties of the test compound.

Detailed information regarding the test substance, including the GLP certificate of analysis, is presented in Appendix A (Test Substance Information).

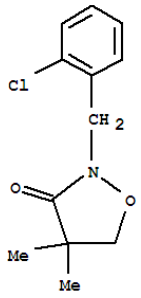
Table 1. Test Compound Nomenclature.	
Compound	Chemical Structure  Clomazone
Common name	Clomazone
IUPAC name	2-(2-chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one or 2-(2-chlorobenzyl)-4,4-dimethylisoxazolidin-3-one
CAS name	2-[(2-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone
CAS #	81777-89-1
End-use product/EP	Clomazone 360 g/L CS

Table 2. Physicochemical Properties.		
Parameter	**Value**	**Reference[a]**
Melting point/range	25 ºC, broad range	MRID 00144241
pH	6.89 ± 14 (supernatant of slurry)	MRID 00144241
Density	1.187 g/mL	MRID 00144241
Water solubility (mg/L at unspecified C)	1100 mg/L	MRID 00144241
Solvent solubility (mg/L at unspecified C)	>200 mg/L in Tenneco 500-100 4.5-5.0 mg/L in Isopar M >90mg/L in refined soybean oil Infinite in dimethyl formamide Infinite in cyclohexanone	MRID 00144241
Vapour pressure at 25 ºC	1.92 x 10^{-2} Pa (1.44 x 10^{-4} mm Hg)	MRID 00144241
Dissociation constant (pK_a)	N/A neither acidic nor basic	MRID 00144241
Octanol/water partition coefficient Log(K_{OW})	350 (log p = 2.54)	MRID 00144241
UV/visible absorption spectrum	Not Available	Not Applicable

[a]Data have been submitted to EPA and this is the EPA MRID number.

B. EXPERIMENTAL DESIGN

1. Study Site Information

Field data tables detailing the field phase of this study are included in Appendix B. A summary of the procedures used during the field phase is given below.

Field trial data have been generated for clomazone on canola. Twelve canola trials were conducted in Canada in PMRA Zones/Regions 5 (one trial), 7 (two trials) and 14 (nine trials) during the 2013 growing season. Climate, soil types, and other conditions were typical of areas where Clomazone 360 g/L CS may be used to control weeds in canola.

One untreated control (Trt 1) and one treated plot (Trt 2) were established at each test site. At Trial -06, two additional treated plots (Trt 3 and Trt 4) were established to produce seed for the possible processing phase. The untreated control and treated plots were laid out and properly identified. The treated plots were a minimum of 19 rows ranging in size from 90 to 360 square meters. The treated plots were separated from the untreated plots by a minimum of 30 meters. At the processing trial, the treated plots were separated by at least 10 meters.

The 1× (Trt 2) rate treated plots received one pre-emergence soil-applied broadcast application of Clomazone 360 g/L CS at the maximum proposed label use rate of 0.42 kg ai/ha ±5%. At Trial -06, the 3× (Trt 3) and 5× (Trt 4) rate treated plots received one pre-emergence broadcast application at 1.26 and 2.1 kg ai/ha ±5%, respectively.

The application techniques and spray volumes were adequate to ensure uniform coverage as required. The spray volume ranged from 47 to 104 liters per hectare (l/ha) with three trials selected to target a low spray volume of 47 l/ha ±10%. Spray additives were not included in the spray mixes. Applications were made using commercial or simulated commercial ground spray equipment which was calibrated prior to each application with the volume/time method.

Phytotoxicity was observed at Trials -01, -02, -03, -06, -07, and -08 shortly after emergence. In all cases, the crop response was short lived, and the crops recovered fully.

The test crops were grown and maintained according to typical agricultural practices for the geographical regions for canola. Irrigation was not used in keeping with normal commercial practice for canola. The conditions at each test site are summarized in Table 3; the use pattern assayed is summarized in Table 4; and the number of trials and locations established in this study are presented in Table 5 and Figure 1. Detailed field data are presented in Appendix B including experimental period weather data compared to historical data. Air temperatures were generally normal during the growing season. Precipitation amounts were generally less than normal at many of the test sites except for the month of June where rainfall was above normal at all sites except for Trials -01, -02, and -06. At Trial -01, rainfall was greater than normal in May and July, and at Trial -02, rainfall was less than normal in July and August. At Trial -06, rainfall was greater than normal in May and normal for all other months. There were no abnormal events that adversely impacted crop yields or crop growth and development.

At all test sites, one untreated control RAC sample and duplicate treated canola RAC samples were cut/harvested from each treated plot at maturity 90 to 122 days after the application (DAA). Some trials cut and threshed samples on the same day; however, at four sites (including one decline trial), the crop was cut/swathed and dried for 5 to 14 days and then threshed. At Trial -06, duplicate RAC samples were also collected from the 5× rate treated plot (Trt 4), and one untreated control (Trt 1) and one 5× (Trt 4) rate treated bulk grain samples were also collected for possible processing. Since adequate seed were collected from Trt 4, seed from Trt 3 were not collected. At two sites (Trials -04 and -05), treated samples were also collected 3-4, 7-8, 14-15, and 21 days after normal harvest maturity to assess residue decline.

The RAC samples for residue analysis weighed at least 0.5 kg except for treated samples from Trial -11 which weighed 0.20 and 0.25 kg due to poor crop emergence and crop stand. This did not adversely impact the study since the crop that was present was healthy and normal, and the sample weights collected were adequate for analysis. The bulk grain samples for the processing phase weighed at least 30 kg. All samples were collected without bias from at least twelve separate areas avoiding the edges of ends of the treated plot. The duplicate treated samples were collected by making separate passes through the treated plots. The untreated plots were harvested before the treated plots to avoid contamination.

Table 3. Trial Site Conditions.

Trial Identification (City, Province, Country/Year)	Soil characteristics				Meteorological Data	
	Type	%OM*	pH*	CEC* (meq/ 100 g)	Overall (Monthly) Rainfall Range (mm)	Overall Temp Range (Monthly Mean Min/Max) (ºC)
TCI-13-366-01 (Portage la Prairie, MB, CAN/2013)	Silty Clay Loam	4.49	7.94	41.3	38.3 - 100.6	5 / 26
TCI-13-366-02 (Dundurn, SK, CAN/2013)	Loam	2.4	6.3	9.21	28.0 - 73.5	8 / 26
TCI-13-366-03 (Saskatoon, SK, CAN/2013)	Silty Clay	5.62	7.87	31.8	14.7 - 115.9	5 / 26
TCI-13-366-04 (Hepburn, SK, CAN/2013)	Loam	4.1	7.4	18.2	14.7 - 115.9	5 / 26
TCI-13-366-05 (Josephburg, AB, CAN/2013)	Loam	6.2	5.8	30	8.5 - 100.5	5 / 24
TCI-13-366-06 (Carberry, MB, CAN/2013)	Sandy Loam	1.88	7.02	15.6	51.0 - 84.2	5 / 25
TCI-13-366-07 (Shilo, MB, CAN/2013)	Sand	1.29	6.37	9.45	22.6 - 149.2	4 / 25
TCI-13-366-08 (Brandon, MB, CAN/2013)	Silty Clay	2.74	7.89	33.8	47.8 - 149.2	4 / 25
TCI-13-366-09 (Alvena, SK, CAN/2013)	Loam	2.2	6.5	20.5	14.7 - 115.9	7 / 26
TCI-13-366-10 (Wakaw, SK, CAN/2013)	Loam	4.5	7.8	NA	20.0 - 181.6	5 / 25
TCI-13-366-11 (Waldheim, SK, CAN/2013)	Loam	2.1	7.5	18.8	14.7 - 115.9	5 / 26
TCI-13-366-12 (Aberdeen, SK, CAN/2013)	Clay Loam	3.8	7.7	25.4	14.7 - 115.9	5 / 26

* These parameters are optional except in cases where their value affects the use pattern for the chemical.

Detailed site information, including maintenance chemical information and weather conditions, is provided in Appendix B.

Table 4. Study Use Pattern for Clomazone.

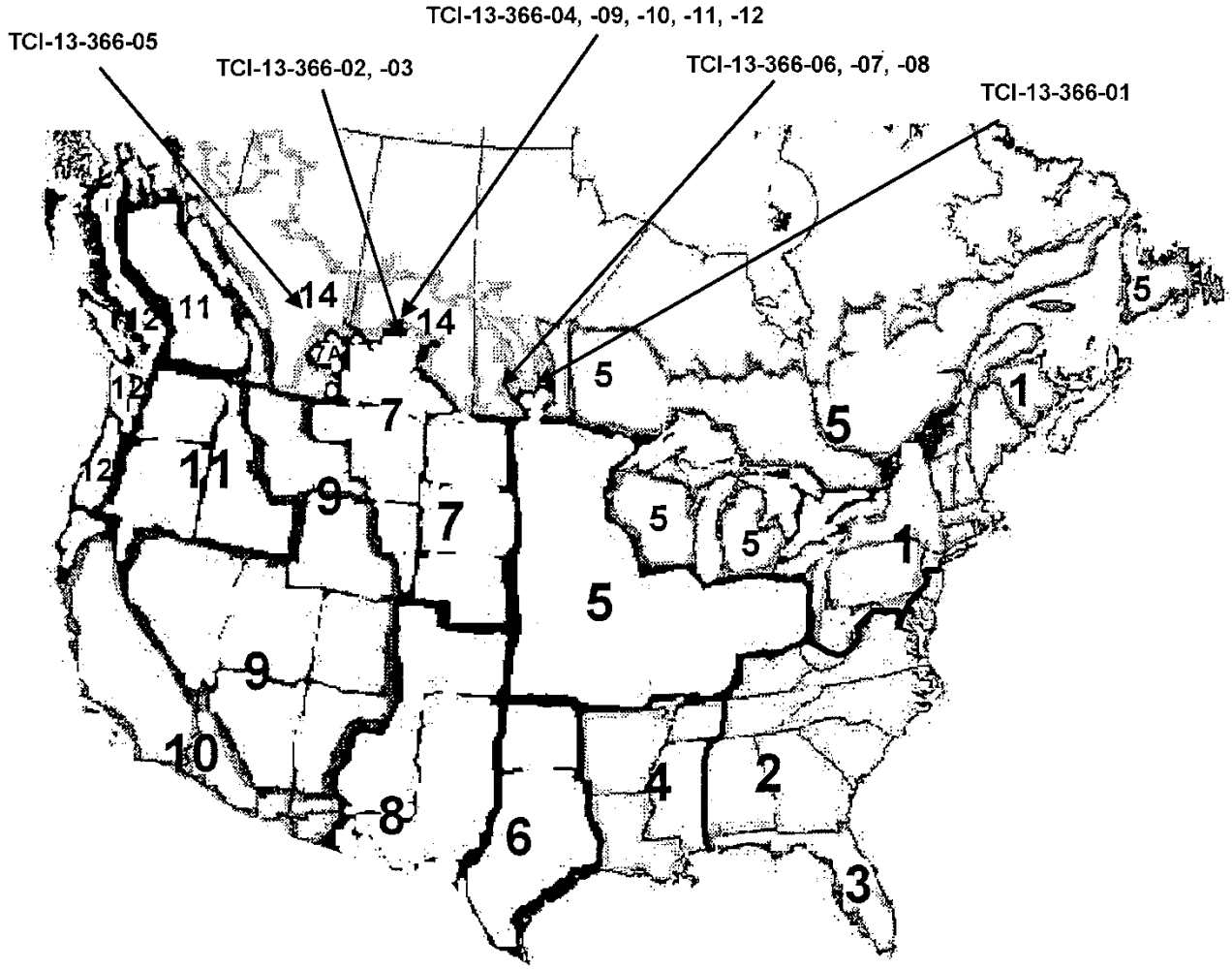
Trial Identification (City, Province, Country/Year)	EP[a]	Application							
		Trt No.	Appl. Timing	Rate, kg ai/ha	RTI[b] (days)	Volume L/ha[c]	Method	Total Rate, kg ai/ha	Tank Mix Adjuvant
TCI-13-366-01 (Portage la Prairie, MB, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.436	NA	103	Soil-applied Broadcast	0.436	None
TCI-13-366-02 (Dundurn, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.425	NA	48	Soil-applied Broadcast	0.425	None
TCI-13-366-03 (Saskatoon, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.436	NA	104	Soil-applied Broadcast	0.436	None
TCI-13-366-04 (Hepburn, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.421	NA	100	Soil-applied Broadcast	0.421	None
TCI-13-366-05 (Josephburg, AB, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.420	NA	47	Soil-applied Broadcast	0.420	None
TCI-13-366-06 (Carberry, MB, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.419	NA	99	Soil-applied Broadcast	0.419	None
		3	Pre-emergence	1.26	NA	99		1.26	
		4	Pre-emergence	2.11	NA	99		2.11	
TCI-13-366-07 (Shilo, MB, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.433	NA	48	Soil-applied Broadcast	0.433	None
TCI-13-366-08 (Brandon, MB, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.424	NA	100	Soil-applied Broadcast	0.424	None
TCI-13-366-09 (Alvena, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.424	NA	101	Soil-applied Broadcast	0.424	None
TCI-13-366-10 (Wakaw, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.404	NA	96	Soil-applied Broadcast	0.404	None
TCI-13-366-11 (Waldheim, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.433	NA	103	Soil-applied Broadcast	0.433	None
TCI-13-366-12 (Aberdeen, SK, CAN/2013)	Clomazone 360 g/L CS	2	Pre-emergence	0.414	NA	99	Soil-applied Broadcast	0.414	None

[a]EP = End-use Product.
[b]RTI = Retreatment Interval.
[c]L/ha = liters/hectare.

Table 5. Trial Numbers and Geographical Locations.		
PMRA Growing Region	**Canola**	
	Requested[a]	**Submitted**
1		
1A		
5	1	1
5A		
5B		
7	2	2
7A		
9		
11		
12		
14	9	9
Total	12	12

[a]The requested number and geographic distribution of field trials for a canola study according to PMRA Regulatory Directive DIR98-02 and 2010-05.

Figure 1. Geographic Location of the Test Sites



2. Sample Handling and Preparation of Field Samples

The canola RAC samples were placed on dry ice or in freezer storage within 3.13 hours after collection from the field. All samples were maintained frozen at the field facilities and were shipped 3 to 38 days later via ACDS freezer truck with Morse Laboratories, LLC as the original destination; however, due to the announced closing of the Morse facility in Sacramento, the destination was changed in transit and the samples were delivered to ABC Laboratories, Inc. for analysis.

All canola RAC samples were received frozen from the field and were stored in a freezer (-25 °C to -10 °C) prior to homogenization and analysis. Canola samples were ground with dry ice to a homogeneous consistency in a Robot Coupe RSI 10Y vertical cutter/grinder. For large samples, representative subsamples were prepared from the ground samples for analysis. The ground samples were placed in frozen storage immediately after grinding. The dry ice was allowed to sublime prior to weighing the samples for extraction.

3. Analytical Methodology

The analytical report detailing the analytical phase of this study is included in Appendix C. A summary of the procedures used during the analytical phase is given below.

The analytical method used for the determination of clomazone in canola RAC samples was the method found in Eurofins-GAB GmbH Study Code 20061401/01-RVP Final Report, entitled "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape," dated January 17, 2007, with ABC Laboratories method modifications, dated April 23, 2014. The method modification eliminated several post extraction steps including several filtration steps and performance of GPC cleanup. A study conducted at the laboratory comparing the original method and the modified method demonstrated equivalence. These data are presented in the analytical report (Appendix C).

To summarize, residues of clomazone were extracted from a 25-g seed sample by homogenization with acetone, acetonitrile, Calflo E, and Celite. An aliquot of the sample was centrifuged, and the supernatant filtered through a 0.45-µ PTFE filter. An aliquot of the filtered sample was combined with acetonitrile:water (1:1, v/v) and submitted for HPLC analysis. Determination and quantitation for clomazone were conducted using high performance liquid chromatography (HPLC) with triple quadrupole mass spectrometric (LC-MS/MS) detection.

Reference substances were used in the study to generate data for both instrument and method performance. Quantitation of residues in all samples was achieved using a validated software application to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line (from a set of standard concentrations in ng/mL versus peak response) and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. Weighting (1/x) was used in the generation of standard curves. The performance of the instrument was evaluated during each injection set. For this study, the correlation coefficient (r) for each calibration curve was equal to or greater than 0.990 (r^2 equal to or greater than 0.98). The performance of the analytical method was evaluated during each sample set by fortifying control matrix with reference standard.

The limit of quantitation (LOQ) in this study was 0.02 ppm and was the lowest level at which control samples were fortified during this study. The limit of detection (LOD) was defined as approximately $^1/_3$ the LOQ or 0.007 ppm.

Detailed information regarding the reference substance, including the GLP certificate of analysis, is presented in Appendix C (Analytical Summary Report). This appendix also includes detailed analytical data such as supporting raw data necessary for re-calculations, representative chromatograms, and example calculations.

C. RESULTS AND DISCUSSION

Analytical Method:

Canola RAC samples were analyzed for residues of clomazone using the analytical method found in Eurofins-GAB GmbH Study Code 20061401/01-RVP Final Report, entitled "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape," dated January 17, 2007, with ABC Laboratories method modifications, dated April 23, 2014. The method was validated according to SANCO guidelines on canola seed prior to sample analysis. The fortification levels were 0.02 (LOQ) and 0.5 ppm. Method validation recoveries ranged 66 to 95% (mean = 81% ± 9.0, n = 10).

The clomazone method was judged acceptable for use in this study. The recoveries from the method validation fortified samples demonstrate acceptable precision. Because the method used triple quadrupole mass spectrometric (LC-MS/MS) detection, it is considered specific for the analyte targeted. The correlation coefficient (r) of the calibration curve (>0.990) generated during method validation demonstrated a valid relationship (linear regression) between standard concentration and instrument response. These data indicated that the method would perform well during the course of this study.

Procedural recovery control samples were analyzed in conjunction with each analytical set for quality control purposes. Fortification levels ranged from 0.02 to 0.5 ppm for clomazone. These samples were extracted and analyzed according to the same procedure as the study samples. Acceptable concurrent recovery data were obtained. Concurrent recoveries ranged from 68-112% (mean = 95% ± 12, n=12). The method recoveries are presented in Table 6.

Recoveries for procedural control samples were corrected, if needed, for apparent residues detectable in the associated controls. Residues in treated samples were not corrected for procedural recovery results or for apparent residues in the associated control samples.

Table 6. Summary of Method Recoveries of Clomazone from Canola.				
Matrix	**Fortification Level in ppm (mg/kg)**	**Sample Size (n)**	**Recoveries (%)**	**Range (%) Mean (%) ± std. dev. (RSD)**
Method Validation Fortifications – Clomazone				
Canola Seed	0.02	5	90, 95, 77, 66, 76	66-95 81 ± 12 (14)
	0.5	5	81, 92, 81, 73, 78	73-92 81 ± 7.0 (8.6)
	Overall	10	--	66-95 81 ± 9.0 (11)
Concurrent Fortifications – Clomazone				
Canola Seed	0.02 0.5	7 5	81, 90, 92, 94, 100, 94, 68 95, 109, 112, 99, 100	68-112 95 ± 12 (12)

Apparent residues of clomazone were below the LOD (<0.007 ppm) in/on all untreated control canola samples. No interferences were noted in control samples.

Storage Stability:

The maximum storage interval from collection to analysis for the RAC samples was 203 days (Table 7). The frozen storage stability of clomazone residues in canola seed has been demonstrated for at least 6 months in a previous study (Ref No. 1). Additional freezer storage stability testing initiated in this study has been transferred to another study for completion and reporting. The data generated to date indicate that residues should not be impacted by 6.7 months of freezer storage.

Table 7. Summary of Storage Conditions.			
Matrix (RAC or Extract)	**Storage Temperature (°C)**	**Actual Storage Duration (days)**	**Limit of Demonstrated Storage Stability**
Canola Seed	Frozen at Field Test Sites ~ -25 °C to -10 °C at the Laboratory	166 - 203 days (5.5 - 6.7 months)	6 month (Ref. No. 1)

Residue Levels in RAC Fractions:

The results from these trials show that following one pre-emergence application of Clomazone 360 g/L CS at the maximum proposed label use rate of 0.42 kg ai/ha ± 5%, residues of clomazone were less than the LOD in the canola samples collected at normal crop maturity, 90 to 122 DAA. Following one pre-emergence application of Clomazone 360 g/L CS at a 5× rate of 2.1 kg ai/ha, residues ranged <LOD to <LOQ. Since residues were <LOQ in all samples collected, including samples from the 5× exaggerated rate treatment, the processing phase was not conducted.

These data are presented in Table 8 and summarized in Table 9.

Residue Decline Data:

Two trials (Trials -04 and -05) also collected treated samples 3-4, 7-8, 14-15, and 21 days after normal harvest maturity to assess residue decline. In both cases, residues were <LOD at all sampling events indicating that residues do no increase with longer PHI.

Residue Definition:

Based on EPA's review of available plant metabolism data, the Agency concluded that the residue of concern in food/feed items is clomazone, and the residue of concern for tolerance enforcement is clomazone *per se*. As such, all plant magnitude of the residue studies are required to measure residue of this analyte in all matrices.

Cultural Practices and Environmental Conditions:

The residue trials in canola were not adversely impacted by the farming practices or environmental conditions. The crops were grown and maintained according to typical agricultural practices for the geographical region. The sites selected provided an appropriate range of soil types, and the crop varieties selected were typical for commercial production in the area.

The trial period temperatures and precipitation amounts, as well as the historical weather data for each test site, are provided in Appendix B. Data show that the air temperatures were generally within normal ranges with typical variations. Precipitation amounts were generally less than normal at many of the test sites except for the month of June where rainfall was above normal at all sites except for Trials -01, -02, and -06. At Trial -01, rainfall was greater than normal in May and July, and at Trial -02, rainfall was less than normal in July and August. At Trial -06, rainfall was greater than normal in May and normal for all other months. This did not have an adverse impact on crop growth and development and yields were in the normal range. Irrigation was not used to supplement rainfall which is typical agronomic practice for canola.

There was phytotoxicity observed at Trials -01, -02, -03, -06, -07, and -08 shortly after emergence. In all cases, the crop response was short lived, and the crops recovered fully.

The number and geographic distribution of the canola trials are adequate to support a tolerance for residues of clomazone in/on canola. Data on clomazone residues in/on canola were provided from a total of 12 trials conducted in the PMRA Zones/Regions 5 (one trial), 7 (two trials) and 14 (nine trials) during the 2013 growing season.

The residue results from the crop field trials with clomazone are presented in Table 8 and are summarized in Table 9. The percent moisture was determined for one canola seed sample from each site. Percent moisture data are presented in Table 4 in the analytical report (Appendix C).

Table 8. Residue Data from Crop Field Trials with Clomazone 360 g/L CS – Canola							
Trial Identification (City, Province, Country/Year)	**PMRA Region**	**Crop/ Variety**	**Total Rate, kg ai/ha**	**PHI[a] (days)**	**Trt No.**	**Commodity**	**Clomazone Residues (ppm)**
TCI-13-366-01 (Portage la Prairie, MB, CAN/2013)	5	Canola / Dekalb RR 73-75	0.436	93	2	Seed	<LOD, <LOD
TCI-13-366-02 (Dundurn, SK, CAN/2013)	7	Canola / Pioneer 45H31	0.425	93	2	Seed	ND, ND
TCI-13-366-03 (Saskatoon, SK, CAN/2013)	7	Canola / Liberty Link L130	0.436	109	2	Seed	<LOD, ND
TCI-13-366-04 (Hepburn, SK, CAN/2013)	14	Canola / Pioneer 45H31	0.421	101	2	Seed	ND, ND
				104	2	Seed	<LOD, ND
				108	2	Seed	<LOD, <LOD
				115	2	Seed	<LOD, ND
				122	2	Seed	ND, ND
TCI-13-366-05 (Josephburg, AB, CAN/2013)	14	Canola / L135C	0.420	100	2	Seed	<LOD, <LOD
				104	2	Seed	ND, ND
				108	2	Seed	ND, ND
				115	2	Seed	ND, ND
				121	2	Seed	ND, ND
TCI-13-366-06 (Carberry, MB, CAN/2013)	14	Canola / Dekalb 73-75	0.419	122	2	Seed	ND, <LOD
			2.11	122	4	Seed	<LOQ, <LOD
TCI-13-366-07 (Shilo, MB, CAN/2013)	14	Canola / Dekalb 73-75	0.433	90	2	Seed	ND, ND
TCI-13-366-08 (Brandon, MB, CAN/2013)	14	Canola / Dekalb 73-75	0.424	90	2	Seed	ND, ND
TCI-13-366-09 (Alvena, SK, CAN/2013)	14	Canola / Liberty Link L130	0.424	102	2	Seed	ND, ND
TCI-13-366-10 (Wakaw, SK, CAN/2013)	14	Canola / Liberty Link L130	0.404	100	2	Seed	ND, ND
TCI-13-366-11 (Waldheim, SK, CAN/2013)	14	Canola / Pioneer 45H31	0.433	101	2	Seed	ND, ND
TCI-13-366-12 (Aberdeen, SK, CAN/2013)	14	Canola / Liberty Link L130	0.414	112	2	Seed	ND, <LOD

[a]PHI=Preharvest Interval.

For clomazone, the LOQ is 0.02 ppm and the LOD is 0.007 ppm (1/3 the LOQ). ND = no detectable peak.

Table 9. Summary of Residue Data from Crop Field Trials with Clomazone.											
					Residue Levels (ppm)						
Commodity	EP[a]	Total Appl. Rate kg ai/ha	Analyte	PHI (days)	n[b]	Min.[b]	Max.[b]	HAFT[b]	Median[b] (STMdR)	Mean[b] (STMR)	Std. Dev.[b]
Canola Seed	Clomazone 360 g/L CS	0.42	Clomazone	90-122	24	ND	<LOD	<LOD	<LOD	<LOD	0.000

[a] EP = End-use Product

[b] n = number of individual specimens,
Min = minimum individual specimen residue,
Max = maximum individual specimen residue,
HAFT = Highest Average residue from one Field Trial; (requested when the raw commodity has associated commercially processed commodities on which tolerances/MRLs could be set),
Median (STMdR) = supervised trial median residue,
Mean (STMR) = supervised trial mean residue,
Std Dev = standard deviation of mean

The LOQ is 0.02 ppm and the LOD is 0.007 ppm (1/3 the LOQ). ND = no detectable peak.

For statistical analysis, ½ the LOD (0.0035 ppm) was used for residues <LOD.

D. CONCLUSION

The residue data produced from the treated plots of this study are appropriate for setting tolerances for the agricultural practices studied. The field practices for this study were consistent and appropriate to obtain good residue samples. Samples were properly taken, stored, and shipped at frozen temperatures. Samples were analyzed for residues using a well-controlled appropriate method. No unusual weather phenomena, agricultural, or analytical practices were experienced during the conduct of this study that significantly impacted the study.

Canola samples were analyzed within 203 days (6.7 months) of collection. Freezer storage stability for canola, indicate that clomazone residues are stable in frozen storage for at least 6 months. Additional freezer storage stability testing initiated in this study has been transferred to another study for completion and reporting. The data generated to date indicate that residues should not be impacted by 6.7 months of freezer storage.

E. STATISTICS AND DATA INTEGRITY

Statistical treatment of the data included determination of the minimum and maximum for the residue data and mean and standard deviation for the procedural recovery data. Validated software program Microsoft Office Excel® 2010 was used to develop these data. At the analytical laboratory, statistical methods used were limited to regression analysis and calculations of the mean, range, and standard deviation. Microsoft Excel® 2007-2010 and validated software program, Applied BioSystems/MDS Sciex Analyst software (version 1.5) were employed to develop all statistical data (Appendix C).

Several measures were taken to ensure the quality and integrity of the study. Quality assurance inspected the field and analytical procedures for compliance with good laboratory practices. The inspection dates are provided in the quality assurance unit statement. Study samples and test and reference substances were maintained in secured storage. Freezer temperatures were continuously monitored and recorded.

F. PROTOCOL, AMENDMENTS, AND DEVIATIONS

All protocol deviations that occurred during the conduct of this study were reported and reviewed by the Study Director. A copy of the protocol and amendments and a list of deviations are provided in Appendix D. None of the changes had an impact on the validity of the study.

G. ARCHIVING

Upon completion of the study, samples of the applicable test and reference substances will be retained by the Sponsor for as long as the quality of the materials affords evaluation or for the duration of the registration, whichever occurs first. All raw data associated with the study (field and analytical portions) are retained in the Sponsor's permanent archival facility, EPL Archives, Inc., 45610 Terminal Drive, Sterling, VA 20166 along with the protocol, protocol amendments, and the final report.

H. REFERENCES

1. Fiedler E. (2007): Determination of the Storage Stability of Clomazone in Potatoes and Oil Seed Rape at approximately - 20 °C. Eurofins-GAB GmbH, Niefern-Öschelbronn, DEU Study No.: 20054075/01-RSS; CHA Doc. No.: 83 CAZ.

APPENDICES

Appendix A - Test Substance Information

Test Compound

Common Name:	Clomazone
IUPAC Name:	2-(2-chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one or 2-(2-chlorobenzyl)-4,4-dimethylisoxazolidin-3-one
CAS Name:	2-[(2-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone
CAS Number:	81777-89-1

Formulations

End-use Product	Clomazone 360 g/L CS
Formulation Type	capsule suspension (CS)
Nominal A.I. Content	360 g ai/L
Lot Number	0001023789
GLP Certified Active Concentration	355 g ai/L (used for spray mix calculations)
Date of Analysis	21-Mar-12
Expiration Date	21-Mar-14

Clomazone 360 g/L CS was the formulated test substance for this study. Characterization and purity was determined prior to the use in this study. The stability of the Clomazone 360 g/L CS test substance under the conditions of use has been verified.

The characterization of the test substance was conducted at Cheminova A/S. The GLP Certification of Analysis is presented below:

CHEMINOVA

Cheminova A/S
P.O. Box 9
DK-7620 Lemvig
Denmark

Phone (+45) 96 90 96 90
Fax (+45) 96 90 96 91
www.cheminova.com
CVR-No. DK12 76 00 43

Certificate of Analysis

TEM 103-05

Test substance certified:

Test substance:	***Clomazone 360 g/L CS***
CHA Code No.:	6710
Batch No.:	0001023789
Origin of test substance:	[ ] Laboratory [ ] Pilot plant [X] Commercial

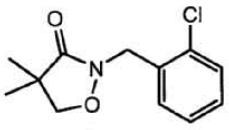
Analysis:

Content of Clomazone:	***33.2% w/w (355 g/L)***
Identified by:	^{1}H-NMR and ^{13}C-NMR Spectroscopy and UV spectroscopy
Quantified by:	Gas Chromatography (VAM 124-01)
Date of analysis:	March 21, 2012

Information of the test substance:

Appearance:	Whitish liquid.
Storage:	Ambient temperature in the dark
Density:	1.07 g/mL at 20°C (according to the recipe)
Expiry date:	March 21, 2014

Information of analyte(s):

Common name:	Clomazone
CAS name:	3-Isoxazolidinone, 2-[(2-chlorophenyl)methyl]-4,4-dimethyl-
CAS No.:	81777-89-1
Molecular formula:	$C_{12}H_{14}ClNO_2$
Molecular mass:	239.7 g/mol
Structure formula:	

Statement of GLP Compliance

The identification and quantification were performed at Cheminova A/S and conducted according to FIFRA Good Laboratory Practice Standards, 40 CFR Part 160 and the OECD Principles of Good Laboratory Practices.

Date: March 29, 2012

Name: Sune Bauer Hansen

Appendix B - Detailed Study Site Information

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-01, Portage la Prairie, Manitoba

Personnel

Principal Investigator:	Kelly Tiller
Affiliation:	ICMS, Inc.
Investigator Street Address:	Box 67 Station Main, 2375 Saskatchewan Ave. E.
Investigator City, Province, Country, Zip Code:	Portage la Prairie, Manitoba, Canada, R1N 3B2
Other Personnel Involved in the Trial:	Clint Ritskes and Katelynn Chabot

Plot Information

PMRA Region Number:	5
Test Site Street Address:	2375 Saskatchewan Ave. E.
City, Province, Country, Zip Code:	Portage la Prairie, Manitoba, Canada, R1N 3B2
County:	R.M. of Portage la Prairie
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Dekalb RR 73-75
Planting Date of Crop:	27-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	85 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	20
Row Spacing:	15 cm
Plant Spacing in Row:	5 cm
Soil Texture:	Silty Clay Loam
Soil % Organic Matter:	4.49
Soil pH:	7.94
CEC (meq/100 g):	41.3

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-01, Portage la Prairie, Manitoba (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Southport, Manitoba (Trial Data) Environment Canada: Portage la Prairie, Manitoba (Historical Data)
Distance of Temperature Data from the Test Site:	~5 km (Trial Data) ~3 km (Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Southport, Manitoba (Trial Data) Environment Canada: Portage la Prairie, Manitoba (Historical Data)
Distance of Precipitation Data from the Test Site:	~5 km (Trial Data) ~3 km (Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Rainfall was greater in May and July, and less in June. Air temperatures were near normal.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	17	19	96.5	47.8	0.0
June 2013	12	11	24	23	47.0	75.8	0.0
July 2013	14	13	25	25	100.6	75.2	0.0
August 2013	14	12	26	25	63.8	70.3	0.0
September 2013	10	6	22	18	38.3	53.9	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
32-16-2 Fertilizer	250 lb/A	16-May-13
Roundup (glyphosate)	0.45 kg ai/ha	28-Jun-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-01, Portage la Prairie, Manitoba (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	29-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	0.0 - 11.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV Mounted Offset Sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	29-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.436
Spray Volume (l/ha):	103

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-01, Portage la Prairie, Manitoba (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	30-Aug-13	12-Sep-13	Canola Seed	BBCH 89	1.0	23-Sep-13	10-Oct-13
02	2	93	30-Aug-13	12-Sep-13	Canola Seed	BBCH 89	0.7	23-Sep-13	10-Oct-13
03	2	93	30-Aug-13	12-Sep-13	Canola Seed	BBCH 89	0.7	23-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	0.42
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #121530
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-02, Dundurn, Saskatchewan

Personnel

Principal Investigator:	Greg Whittington
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Bryn Rees, Brittany Johnson, and Michael Babyn

Plot Information

PMRA Region Number:	7
Test Site Street Address:	Not Applicable (near Evans Road)
City, Province, Country, Zip Code:	Dundurn, Saskatchewan, Canada, S0K 1K0
County:	R.M. #314
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Pioneer 45H31
Planting Date of Crop:	11-Jun-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	56 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	19
Row Spacing:	15.24 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Loam
Soil % Organic Matter:	2.4
Soil pH:	6.3
CEC (meq/100 g):	9.21

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-02, Dundurn, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Outlook PFRA: Outlook, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	47.30 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Outlook PFRA: Outlook, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	47.30 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were normal. Rainfall was slightly lower in July and August
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
June 2013	10	10	22	23	73.5	63.7	0.0
July 2013	11	12	24	25	28.0	57.1	0.0
August 2013	11	11	26	25	28.8	38.3	0.0
September 2013	8	6	23	19	35.7	26.0	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
90-40-00-15 Fertilizer	335 kg/ha	16-May-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-02, Dundurn, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	11-Jun-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	1.0 - 3.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	11-Jun-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.425
Spray Volume (l/ha):	48

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-02, Dundurn, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	12-Sep-13	12-Sep-13	Canola Seed	BBCH 88-89	0.7	24-Sep-13	10-Oct-13
02	2	93	12-Sep-13	12-Sep-13	Canola Seed	BBCH 88-89	0.6	24-Sep-13	10-Oct-13
03	2	93	12-Sep-13	12-Sep-13	Canola Seed	BBCH 88-89	0.5	24-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	2.18 (placed in transport freezer)
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131933
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-03, Saskatoon, Saskatchewan

Personnel

Principal Investigator:	Greg Whittington
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Bryn Rees and Michael Babyn

Plot Information

PMRA Region Number:	7
Test Site Street Address:	Not Applicable
City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7K 3J6
County:	R.M. #344
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Liberty Link L130
Planting Date of Crop:	22-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	69 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	19
Row Spacing:	15.24 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Silty Clay
Soil % Organic Matter:	5.62
Soil pH:	7.87
CEC (meq/100 g):	31.8

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-03, Saskatoon, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Saskatoon RCS/SRC, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	19.0 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Saskatoon RCS/SRC, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	19.0 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were normal. Rainfall was below average in May, July, August, and September.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	21	19	15.2	41.5	0.0
June 2013	10	9	21	23	115.9	60.5	0.0
July 2013	11	12	24	25	35.2	57.3	0.0
August 2013	11	10	26	25	14.7	35.4	0.0
September 2013	7	5	23	18	14.9	28.9	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
90-40-00-15 Fertilizer	335 kg/ha	14-May-13
Liberty (glufosinate-ammonium)	2.0 kg ai/ha	24-Jun-13
Centurion (clethodim)	0.18 kg ai/ha	24-Jun-13
Amigo (vegetable oil surfactant)	0.12 kg ai/ha	24-Jun-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-03, Saskatoon, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	27-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	1.0 - 3.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	27-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.436
Spray Volume (l/ha):	104

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-03, Saskatoon, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	13-Sep-13	13-Sep-13	Canola Seed	BBCH 89	0.7	24-Sep-13	10-Oct-13
02	2	109	13-Sep-13	13-Sep-13	Canola Seed	BBCH 89	0.6	24-Sep-13	10-Oct-13
03	2	109	13-Sep-13	13-Sep-13	Canola Seed	BBCH 89	0.7	24-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	0.10
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131934
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-04, Hepburn, Saskatchewan

Personnel

Principal Investigator:	Dean Ngombe
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Brittany Johnson, Greg Whittington, Evan Megyesi, Michael Babyn, and Brij Verma

Plot Information

PMRA Region Number:	14
Test Site Street Address:	Box 124
City, Province, Country, Zip Code:	Hepburn, Saskatchewan, Canada, S0K 1Z0
County:	R.M. #404
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Pioneer 45H31
Planting Date of Crop:	27-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	6 m x 60 m
Distance between UTC and Trt Plot:	30 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	40
Row Spacing:	15.22 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Loam
Soil % Organic Matter:	4.1
Soil pH:	7.4
CEC (meq/100 g):	18.2

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-04, Hepburn, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	45 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	45 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were slightly above normal. Rainfall was less than normal with the exception of June that had 1.9 times higher precipitation than normal.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	21	19	15.2	43.6	0.0
June 2013	10	9	21	23	115.9	60.5	0.0
July 2013	11	12	24	25	35.2	57.3	0.0
August 2013	11	10	26	25	14.7	35.4	0.0
September 2013	7	5	23	18	14.9	30.6	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
Roundup Transorb (glyphosate)	1.507 kg ai/ha	15-Jul-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-04, Hepburn, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	29-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	6.0 - 8.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	29-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.421
Spray Volume (l/ha):	100

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-04, Hepburn, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	07-Sep-13	07-Sep-13	Canola Seed	BBCH 89	1.1	24-Sep-13	10-Oct-13
02	2	101	07-Sep-13	07-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
03	2	101	07-Sep-13	07-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
04	2	104	10-Sep-13	10-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
05	2	104	10-Sep-13	10-Sep-13	Canola Seed	BBCH 89	0.6	24-Sep-13	10-Oct-13
06	2	108	14-Sep-13	14-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
07	2	108	14-Sep-13	14-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
08	2	115	21-Sep-13	21-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
09	2	115	21-Sep-13	21-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
10	2	122	28-Sep-13	28-Sep-13	Canola Seed	BBCH 89	0.8	04-Nov-13	15-Nov-13
11	2	122	28-Sep-13	28-Sep-13	Canola Seed	BBCH 89	0.7	04-Nov-13	15-Nov-13

Maximum Number of Hours from Sampling until Freezing:	0.50
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131927, #131905
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-05, Josephburg, Alberta

Personnel

Principal Investigator:	Taryn Williams
Affiliation:	ICMS, Inc.
Investigator Street Address:	Box 3270, 54474 Range Road 215
Investigator City, Province, Country, Zip Code:	Fort Saskatchewan, Alberta, Canada, T8L 2T2
Other Personnel Involved in the Trial:	Jennifer Oliver, Abby Czibere, and Ji Cui

Plot Information

PMRA Region Number:	14
Test Site Street Address:	54474 Range Road 215
City, Province, Country, Zip Code:	Josephburg, Alberta, Canada, T8L 2T2
County:	Strathcona
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	L135C
Planting Date of Crop:	28-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	6 m x 45 m
Distance between UTC and Trt Plot:	361 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	39
Row Spacing:	15.24 cm
Plant Spacing in Row:	2.54 cm
Soil Texture:	Loam
Soil % Organic Matter:	6.2
Soil pH:	5.8
CEC (meq/100 g):	30

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-05, Josephburg, Alberta (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Edmonton Namao, Alberta (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	26 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Edmonton Namao, Alberta (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	26 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were normal, but with the exception of June, all other months had below average rainfall.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	21	17	36.0	44.7	0.0
June 2013	9	9	20	21	100.5	88.6	0.0
July 2013	10	11	22	22	66.5	95.7	0.0
August 2013	10	10	24	21	67.5	74.8	0.0
September 2013	6	5	21	16	8.5	39.6	0.0

*The last seed samples were collected early in the day on Oct 1 after drying in the plots since cutting on Sep 26. Oct data, therefore, were not included above.

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
28-7-10-35-0.5 Cu-0.1 Zn Fertilizer	279.4 lb/A	08-May-13
11-52-0 Fertilizer	30 kg/ha	17-May-13
Liberty 150 SL (glufosinate-ammonium)	1.6 L/A	17-Jun-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-05, Josephburg, Alberta (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	28-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	0.0 - 7.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV mounted CO_2 Sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	28-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.420
Spray Volume (l/ha):	47

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-05, Josephburg, Alberta (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	05-Sep-13	19-Sep-13	Canola Seed	BBCH 84	1.4	26-Sep-13	10-Oct-13
02	2	100	05-Sep-13	19-Sep-13	Canola Seed	BBCH 84	0.9	26-Sep-13	10-Oct-13
03	2	100	05-Sep-13	19-Sep-13	Canola Seed	BBCH 84	1.0	26-Sep-13	10-Oct-13
04	2	104	09-Sep-13	19-Sep-13	Canola Seed	BBCH 86-87	0.9	26-Sep-13	10-Oct-13
05	2	104	09-Sep-13	19-Sep-13	Canola Seed	BBCH 86-87	1.0	26-Sep-13	10-Oct-13
06	2	108	13-Sep-13	19-Sep-13	Canola Seed	BBCH 87-89	0.9	26-Sep-13	10-Oct-13
07	2	108	13-Sep-13	19-Sep-13	Canola Seed	BBCH 87-89	0.9	26-Sep-13	10-Oct-13
08	2	115	20-Sep-13	01-Oct-13	Canola Seed	BBCH 89	1.3	06-Nov-13	15-Nov-13
09	2	115	20-Sep-13	01-Oct-13	Canola Seed	BBCH 89	1.3	06-Nov-13	15-Nov-13
10	2	121	26-Sep-13	01-Oct-13	Canola Seed	BBCH 89	1.3	06-Nov-13	15-Nov-13
11	2	121	26-Sep-13	01-Oct-13	Canola Seed	BBCH 89	1.3	06-Nov-13	15-Nov-13

Maximum Number of Hours from Sampling until Freezing:	1.53
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #122849, #134664
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-06, Carberry, Manitoba

Personnel

Principal Investigator:	Kelly Tiller
Affiliation:	ICMS, Inc.
Investigator Street Address:	Box 67 Station Main, 2375 Saskatchewan Ave. E.
Investigator City, Province, Country, Zip Code:	Portage la Prairie, Manitoba, Canada, R1N 3B2
Other Personnel Involved in the Trial:	Katelynn Chabot and Clint Ritskes

Plot Information

PMRA Region Number:	14
Test Site Street Address:	Highway #5
City, Province, Country, Zip Code:	Carberry, Manitoba, Canada, R0K 0H0
County:	R.M. of North Cypress
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Dekalb 73-75
Planting Date of Crop:	22-May-13
Number Treated Plot(s) (excluding controls):	3
Size of Treated Plot(s):	8 m x 100 m
Distance between UTC and Trt Plot:	183 m
Distance between Trt Plots:	10 m
Rows per Treated Plot:	54
Row Spacing:	15 cm
Plant Spacing in Row:	5 cm
Soil Texture:	Sandy Loam
Soil % Organic Matter:	1.88
Soil pH:	7.02
CEC (meq/100 g):	15.6

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-06, Carberry, Manitoba (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Carberry, MB (Trial Data) Environment Canada: Glenboro, MB(Historical Data)
Distance of Temperature Data from the Test Site:	~8 km (Trial Data) ~30 km (Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Carberry, MB (Trial Data) Environment Canada: Glenboro, MB(Historical Data)
Distance of Precipitation Data from the Test Site:	~8 km (Trial Data) ~30 km (Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	Yes
Describe any unusual weather:	None
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	17	20	84.2	55.9	0.0
June 2013	10	10	23	24	72.6	75.9	0.0
July 2013	11	12	24	26	68.2	76.8	0.0
August 2013	11	11	25	25	68.8	70.2	0.0
September 2013	9	6	22	19	51.0	51.3	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
46-0-0 Fertilizer	150 kg/ha	03-Jun-13
Roundup (glyphosate)	0.45 kg ai/ha	17-Jun-13
Reglone (diquat as dibromide salt)	0.41 kg ai/ha	20-Sep-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-06, Carberry, Manitoba (continued)

Application Information

Application No.:	1	1	1
Plot ID/Trt No.:	2	3	4
Application Date:	25-May-13	25-May-13	25-May-13
Retreatment Interval:	Not Applicable	Not Applicable	Not Applicable
Application Timing:	Pre-emergence	Pre-emergence	Pre-emergence
Crop Stage:	Pre-emergence	Pre-emergence	Pre-emergence
Wind Speed at Appl. (km/h):	0.0 - 8.0	0.0 - 8.0	0.0 - 8.0
Application Type:	Soil-applied Broadcast	Soil-applied Broadcast	Soil-applied Broadcast
Application Equipment:	ATV CO_2 Offset Sprayer	ATV CO_2 Offset Sprayer	ATV CO_2 Offset Sprayer
Test Substance (Lot No.):	Clomazone (0001023789)	Clomazone (0001023789)	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None	None	None
Calibration Date:	25-May-13	25-May-13	25-May-13
Method of Calibration:	measured output of nozzles	measured output of nozzles	measured output of nozzles
Rate (kg ai/ha):	0.419	1.26	2.11
Spray Volume (l/ha):	99	99	99

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-06, Carberry, Manitoba (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	24-Sep-13	24-Sep-13	Canola Seed	BBCH 89	1.0	01-Nov-13	15-Nov-13
02	2	122	24-Sep-13	24-Sep-13	Canola Seed	BBCH 89	1.0	01-Nov-13	15-Nov-13
03	2	122	24-Sep-13	24-Sep-13	Canola Seed	BBCH 89	1.0	01-Nov-13	15-Nov-13
04	4	122	24-Sep-13	24-Sep-13	Canola Seed	BBCH 89	0.8	01-Nov-13	15-Nov-13
05	4	122	24-Sep-13	24-Sep-13	Canola Seed	BBCH 89	0.9	01-Nov-13	15-Nov-13
06	1	NA	24-Sep-13	24-Sep-13	Canola Seed (Bulk)	BBCH 89	31.6	01-Nov-13	19-Nov-13
07	4	122	24-Sep-13	24-Sep-13	Canola Seed (Bulk)	BBCH 89	31.0	01-Nov-13	19-Nov-13

RAC

Maximum Number of Hours from Sampling until Freezing:	Placed immediately in transport freezer
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #121544
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Processing

Maximum Number of Hours from Sampling until Freezing:	Placed immediately in transport freezer
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #121529
Shipping Destination:	GLP Technologies 22723 State Highway 6 South Navasota, TX 77868
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-07, Shilo, Manitoba

Personnel

Principal Investigator:	Kelly Tiller
Affiliation:	ICMS, Inc.
Investigator Street Address:	Box 67 Station Main, 2375 Saskatchewan Ave. E.
Investigator City, State, Country, Zip Code:	Portage la Prairie, Manitoba, Canada, R1N 3B2
Other Personnel Involved in the Trial:	Katelynn Chabot and Clint Ritskes

Plot Information

PMRA Region Number:	14
Test Site Street Address:	P. O. Box 760
City, State, Country, Zip Code:	Shilo, Manitoba, Canada, R0K 0H0
County:	R.M. of Cornwallis
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Dekalb 73-75
Planting Date of Crop:	22-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	36 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	20
Row Spacing:	15 cm
Plant Spacing in Row:	5 cm
Soil Texture:	Sand
Soil % Organic Matter:	1.29
Soil pH:	6.37
CEC (meq/100 g):	9.45

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-07, Shilo, Manitoba (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Brandon Airport, Manitoba (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	~20 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Brandon Airport, Manitoba (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	~20 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	Yes
Describe any unusual weather:	None
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	4	4	17	19	59.2	52.7	0.0
June 2013	10	9	24	23	149.2	74.4	0.0
July 2013	11	11	24	25	47.8	75.8	0.0
August 2013	10	10	25	25	68.4	69.2	0.0
September 2013	7	4	22	18	22.6	50.1	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
46-0-0 Fertilizer	150 lb/A	03-Jun-13
Roundup (glyphosate)	0.45 kg ai/ha	18-Jun-13
Roundup (glyphosate)	0.45 kg ai/ha	29-Jun-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-07, Shilo, Manitoba (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	25-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	0.0 - 9.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV Mounted Offset Sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	25-May-13
Method of Calibration:	measured output of nozzles
Rate (lb ai/A):	Not Applicable
Rate (kg ai/ha):	0.433
Spray Volume (l/ha):	48

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-07, Shilo, Manitoba (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	23-Aug-13	05-Sep-13	Canola Seed	BBCH 89	0.5	23-Sep-13	10-Oct-13
02	2	90	23-Aug-13	05-Sep-13	Canola Seed	BBCH 89	0.7	23-Sep-13	10-Oct-13
03	2	90	23-Aug-13	05-Sep-13	Canola Seed	BBCH 89	0.8	23-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	1.75
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #121543
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-08, Brandon, Manitoba

Personnel

Principal Investigator:	Kelly Tiller
Affiliation:	ICMS, Inc.
Investigator Street Address:	Box 67 Station Main, 2375 Saskatchewan Ave. E.
Investigator City, State, Country, Zip Code:	Portage la Prairie, Manitoba, Canada, R1N 3B2
Other Personnel Involved in the Trial:	Clint Ritskes and Katelynn Chabot

Plot Information

PMRA Region Number:	14
Test Site Street Address:	NW 23-11-19W
City, State, Country, Zip Code:	Brandon, Manitoba, Canada, R7A 5Y3
County:	R.M. of Elton
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Dekalb 73-75
Planting Date of Crop:	22-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	36 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	20
Row Spacing:	15 cm
Plant Spacing in Row:	5 cm
Soil Texture:	Silty Clay
Soil % Organic Matter:	2.74
Soil pH:	7.89
CEC (meq/100 g):	33.8

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-08, Brandon, Manitoba (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Brandon Airport, Manitoba (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	~3 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Brandon Airport, Manitoba (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	~3 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	There was more rainfall in June.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	4	4	17	19	59.2	52.7	0.0
June 2013	10	9	24	23	149.2	74.4	0.0
July 2013	11	11	24	25	47.8	75.8	0.0
August 2013	10	10	25	25	68.4	69.2	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
46-0-0 Fertilizer	150 lb/A	03-Jun-13
Roundup (glyphosate)	0.54 kg ai/ha	18-Jun-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-08, Brandon, Manitoba (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	25-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	0.0 - 8.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV Mounted Offset Sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	25-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.424
Spray Volume (l/ha):	100

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-08, Brandon, Manitoba (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	23-Aug-13	30-Aug-13	Canola Seed	BBCH 89	1.0	23-Sep-13	10-Oct-13
02	2	90	23-Aug-13	30-Aug-13	Canola Seed	BBCH 89	0.9	23-Sep-13	10-Oct-13
03	2	90	23-Aug-13	30-Aug-13	Canola Seed	BBCH 89	1.0	23-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	1.75
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #121542
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-09, Alvena, Saskatchewan

Personnel

Principal Investigator:	Dean Ngombe
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, State, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Brittany Johnson, Evan Megyesi, and Andrew Smith

Plot Information

PMRA Region Number:	14
Test Site Street Address:	Box 7
City, State, Country, Zip Code:	Alvena, Saskatchewan, Canada, S0K 0E0
County:	R.M. #402
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Liberty Link L130
Planting Date of Crop:	29-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	43 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	19
Row Spacing:	15.24 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Loam
Soil % Organic Matter:	2.2
Soil pH:	6.5
CEC (meq/100 g):	20.5

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-09, Alvena, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	62.6 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	62.6 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were slightly above normal. Rainfall was less than normal with the exception of June that had 1.9 times higher precipitation than normal.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
June 2013	10	9	21	23	115.9	60.5	0.0
July 2013	11	12	24	25	35.2	57.3	0.0
August 2013	11	10	26	25	14.7	35.4	0.0
September 2013	7	5	23	18	14.9	30.6	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
None	Not Applicable	Not Applicable

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-09, Alvena, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	03-Jun-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	4.0 - 7.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	03-Jun-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.424
Spray Volume (l/ha):	101

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-09, Alvena, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	13-Sep-13	13-Sep-13	Canola Seed	BBCH 89	0.9	24-Sep-13	10-Oct-13
02	2	102	13-Sep-13	13-Sep-13	Canola Seed	BBCH 89	0.9	24-Sep-13	10-Oct-13
03	2	102	13-Sep-13	13-Sep-13	Canola Seed	BBCH 89	1.4	24-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	0.07
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131918
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-10, Wakaw, Saskatchewan

Personnel

Principal Investigator:	Dean Ngombe
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Brittany Johnson, Evan Megyesi, and Brij Verma

Plot Information

PMRA Region Number:	14
Test Site Street Address:	NW 7432 SW 3
City, Province, Country, Zip Code:	Wakaw, Saskatchewan, Canada, S0K 4P0
County	R.M. #401
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Liberty Link L130
Planting Date of Crop:	12-Jun-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	69 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	19
Row Spacing:	15.24 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Loam
Soil % Organic Matter:	4.5
Soil pH:	7.8
CEC (meq/100 g):	Not Available

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-10, Walaw, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Prince Albert A, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	58 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Prince Albert A, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	58 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were normal. June was a very wet month with 2.5 times more precipitation than normal. August and September were drier than normal.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
June 2013	10	9	21	22	181.6	72.6	0.0
July 2013	11	11	23	24	89.6	76.8	0.0
August 2013	10	9	25	23	20.0	58.0	0.0
September 2013	5	4	21	17	20.2	39.5	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
90-40-0-15 Fertilizer	335 lb/A	30-May-13
Poast Ultra (sethoxydim)	1.11 L/ha	09-Jul-13
Merge (surfactant blend)	1 L/100 L	09-Jul-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-10, Wakaw, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	12-Jun-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	6.0 - 8.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	12-Jun-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.404
Spray Volume (l/ha):	96

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-10, Wakaw, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	20-Sep-13	20-Sep-13	Canola Seed	BBCH 89	1.1	24-Sep-13	10-Oct-13
02	2	100	20-Sep-13	20-Sep-13	Canola Seed	BBCH 89	1.0	24-Sep-13	10-Oct-13
03	2	100	20-Sep-13	20-Sep-13	Canola Seed	BBCH 89	0.8	24-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	3.13
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131916
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-11, Waldheim, Saskatchewan

Personnel

Principal Investigator:	Dean Ngombe
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Brittany Johnson, Greg Whittington, Evan Megyesi, and Michael Babyn

Plot Information

PMRA Region Number:	14
Test Site Street Address:	Box 310
City, Province, Country, Zip Code:	Waldheim, Saskatchewan, Canada, S0J 0J0
County:	R.M. #464
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Pioneer 45H31
Planting Date of Crop:	27-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	46 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	19
Row Spacing:	15.22 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Loam
Soil % Organic Matter:	2.1
Soil pH:	7.5
CEC (meq/100 g):	18.8

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-11, Waldheim, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	69 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	69 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were slightly above normal. Rainfall was less than normal with the exception of June that had 1.9 times higher precipitation than normal.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	21	19	15.2	43.6	0.0
June 2013	10	9	21	23	115.9	60.5	0.0
July 2013	11	12	24	25	35.2	57.3	0.0
August 2013	11	10	26	25	14.7	35.4	0.0
September 2013	7	5	23	18	14.9	30.6	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
Roundup Transorb (glyphosate)	1.507 kg ai/ha	15-Jul-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-11, Waldheim, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	28-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	5.0 - 8.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	28-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.433
Spray Volume (l/ha):	103

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-11, Waldheim, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	06-Sep-13	06-Sep-13	Canola Seed	BBCH 89	0.5	24-Sep-13	10-Oct-13
02	2	101	06-Sep-13	06-Sep-13	Canola Seed	BBCH 89	0.2	24-Sep-13	10-Oct-13
03	2	101	06-Sep-13	06-Sep-13	Canola Seed	BBCH 89	0.25	24-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	1.0
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131920
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-12, Aberdeen, Saskatchewan

Personnel

Principal Investigator:	Dean Ngombe
Affiliation:	ICMS, Inc.
Investigator Street Address:	334 Packham Ave.
Investigator City, Province, Country, Zip Code:	Saskatoon, Saskatchewan, Canada, S7N 2T1
Other Personnel Involved in the Trial:	Brittany Johnson, Evan Megyesi, and Andrew Smith

Plot Information

PMRA Region Number:	14
Test Site Street Address:	Box 144
City, Province, Country, Zip Code:	Aberdeen, Saskatchewan, Canada, S0K 0A0
County:	R.M. #373
PMRA Crop Group:	Not Applicable
Test System:	Canola
Variety:	Liberty Link L130
Planting Date of Crop:	27-May-13
Number Treated Plot(s) (excluding controls):	1
Size of Treated Plot(s):	3 m x 30 m
Distance between UTC and Trt Plot:	30 m
Distance between Trt Plots:	Not Applicable
Rows per Treated Plot:	19
Row Spacing:	15.22 cm
Plant Spacing in Row:	Not Applicable
Soil Texture:	Clay Loam
Soil % Organic Matter:	3.8
Soil pH:	7.7
CEC (meq/100 g):	25.4

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-12, Aberdeen, Saskatchewan (continued)

Weather Information

Source of Test Site Temperature Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Temperature Data from the Test Site:	30 km (Trial and Historical Data)
Source of Test Site Precipitation Data:	Environment Canada: Saskatoon RCS, Saskatchewan (Trial and Historical Data)
Distance of Precipitation Data from the Test Site:	30 km (Trial and Historical Data)
Type of Irrigation Used:	None
Was weather normal during the trial period:	No
Describe any unusual weather:	Temperatures were slightly above normal. Rainfall was less than normal with the exception of June that had 1.9 times higher precipitation than normal.
Were yields and crop growth and development normal:	Yes

Weather Period	Mean Min Temp (C)	Mean Min 30 yr Average Temp (C)	Mean Max Temp (C)	Mean Max 30 yr Average Temp (C)	Total Rainfall (mm)	Total Rainfall 30 yr Average (mm)	Total Irrigation (mm)
May 2013	5	5	21	19	15.2	43.6	0.0
June 2013	10	9	21	23	115.9	60.5	0.0
July 2013	11	12	24	25	35.2	57.3	0.0
August 2013	11	10	26	25	14.7	35.4	0.0
September 2013	7	5	23	18	14.9	30.6	0.0

Maintenance Pesticides and Fertilizers

Other Chemicals Applied During the Test Year to the Test Crop:	Rate / Units	Date
Liberty (glufosinate-ammonium)	1.33 kg ai/ha	18-Jun-13
Centurion (clethodim)	0.19 kg ai/ha	18-Jun-13
Amigo (vegetable oil surfactant)	0.5 kg ai/ha	18-Jun-13

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-12, Aberdeen, Saskatchewan (continued)

Application Information

Application No.:	1
Plot ID/Trt No.:	2
Application Date:	30-May-13
Retreatment Interval:	Not Applicable
Application Timing:	Pre-emergence
Crop Stage:	Pre-emergence
Wind Speed at Appl. (km/h):	5.0 - 8.0
Application Type:	Soil-applied Broadcast
Application Equipment:	ATV-mounted CO_2 sprayer
Test Substance (Lot No.):	Clomazone (0001023789)
Adjuvant Name/ Rate (% v/v):	None
Calibration Date:	30-May-13
Method of Calibration:	measured output of nozzles
Rate (kg ai/ha):	0.414
Spray Volume (l/ha):	99

Appendix B – Detailed Study Site Information (continued)
Test Site TCI-13-366-12, Aberdeen, Saskatchewan (continued)

Sample and Shipping Information

Sample No.	Trt. No.	PHI (days)	Harvest Date	Sampling Date	Fraction	Growth Stage	Sample Size (kg)	Shipping Date	Date Received at Lab
01	1	NA	19-Sep-13	19-Sep-13	Canola Seed	BBCH 89	1.0	24-Sep-13	10-Oct-13
02	2	112	19-Sep-13	19-Sep-13	Canola Seed	BBCH 89	1.0	24-Sep-13	10-Oct-13
03	2	112	19-Sep-13	19-Sep-13	Canola Seed	BBCH 89	1.1	24-Sep-13	10-Oct-13

Maximum Number of Hours from Sampling until Freezing:	<0.083
Field Sample Storage Conditions (ambient, frozen):	Frozen
Shipping Carrier:	ACDS #131922
Shipping Destination:	Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, CA 95825
Shipping Conditions (ambient, frozen):	Frozen

Appendix C - Analytical Summary Report

FINAL ANALYTICAL REPORT

STUDY TITLE

Magnitude of the Residue of Clomazone in/on
Canola Raw Agricultural and Processed Commodities Following
One Preemergence Application of Clomazone 360 g/L CS (2013)

RESIDUE CHEMISTRY TEST GUIDELINES

Canadian PMRA Regulatory Directive DIR98-02 and 2010-05
SANCO 3029/99 rev. 4. 11/07/00

SPONSOR

Cheminova A/S
P.O. Box 9
DK-7620 Lemvig
Denmark

STUDY DIRECTOR

Sandra J. Carringer
The Carringers, Inc.
1003 Palace Court
Apex, North Carolina 27502
USA

PERFORMING LABORATORIES

Morse Laboratories, LLC
1525 Fulton Avenue
Sacramento, California 95825

ABC Laboratories, Inc.
7200 E. ABC Lane
Columbia, Missouri 65202

STUDY IDENTIFICATION

Study No.: TCI-13-366
Morse / ABC Laboratories Study No.: 69869

PRINCIPAL ANALYTICAL INVESTIGATOR/AUTHOR

Carol A. Rodgers

REPORT DATE

June 25, 2014

GLP STATEMENT OF COMPLIANCE

All aspects of this study carried out by **ABC LABORATORIES, INC.** were conducted in accordance with the FIFRA Good Laboratory Practice Standards (40 CFR 160).

Carol A Rodgers

Carol A. Rodgers
ABC LABORATORIES, INC.
Principal Analytical Investigator

25 Jun 14
Date

Clark D Chickering

Clark D. Chickering
ABC LABORATORIES, INC.
Director

25 Jun 14
Date

QUALITY ASSURANCE STATEMENT

Morse Laboratories, LLC Project Number: 69869

Protocol Number: TCI-13-366

Quality Assurance inspections were carried out during the execution of the Study by Quality Assurance personnel according to §40 CFR 160.35 of the EPA Good Laboratory Practice Standards to ensure the integrity of the data. The final analytical report, as submitted to the Study Director, reflects the raw data.

Phase Inspected	Dates of Inspection	Date Findings Reported to the Study Director/Management
Procedure: Sample Weighing	3/18/14	3/19/14
Raw Data	7/24/13	3/14/14
	8/30/13	3/14/14
	4/24/14 through 4/30/14	5/21/14
	4/21/14 through 4/27/14	5/22/14
	6/9/14 through 6/10/14	6/25/14
Draft Report	5/1/14 through 5/2/14	5/27/14
	6/9/14 through 6/10/14	6/25/14
Final Report	6/24/14	6/25/14

Jeri Hofen
Manager, Quality Assurance

25 June 2014
Date

ANALYTICAL PHASE IDENTIFICATION

Title:	Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)
Study No.:	TCI-13-366
Sponsor:	Cheminova A/S P.O. Box 9 DK-7620 Lemvig Denmark
Sponsor Representative:	Faith Womack Cheminova, Inc. 1600 Wilson Boulevard, Suite 700 Arlington, Virginia 22209 USA
Study Director and Testing Facility:	Sandra J. Carringer The Carringers, Inc. 1003 Palace Court Apex, North Carolina 27502 USA
Principal Analytical Investigators[1] and Performing Laboratory[2]:	Carol Rodgers (November 1, 2013 to Present) ABC Laboratories, Inc. 7200 East ABC Lane Columbia, Missouri 65202 USA Jeri Willoh (May 1, 2013 to October 10, 2013) Morse Laboratories, LLC 1525 Fulton Avenue Sacramento, California 95825
Date Study Protocol was Signed by the Study Director:	May 1, 2013
Experimental Analysis Initiation Date:	December 5, 2013 (first sample grinding)

Experimental Analysis
Completion Date: May 19, 2014 (last instrumentation date)

Date Final Analytical Report was
Submitted to the Study Director: June 25, 2014

[1] During the interim between PAI's (i.e., October 11, 2013 to October 31, 2013), analytical phase oversight was provided by Management.

[2] In October 2013, ABC Laboratories, Inc. announced plans to move operations of its subsidiary, Morse Laboratories, LLC of Sacramento, CA, to ABC Laboratories in Columbia, MO. In conjunction with this move, ABC rolled Morse under the ABC trade name, and discontinued use of the Morse brand. For this study, sample analyses and generation of the final report were performed at ABC Laboratories, Inc.

AUTHENTICATION

This report is an accurate and authentic representation of the conditions and results of the analytical phase of this study.

Carol A Rodgers
Carol A. Rodgers
Principal Analytical Investigator

25 Jun 14
Date

Clark D Chickering
Clark D. Chickering
Director

25 Jun 14
Date

TABLE OF CONTENTS

1 SUMMARY

This study was conducted to determine the magnitude and decline of residues of clomazone in or on canola raw agricultural commodities (RAC) following one preemergence application of Clomazone 360 g/L CS at 0.42 kg ai/ha, and harvested at normal maturity with additional time points at two trial sites of 3, 7, 14, and 21 ± 1 days after normal maturity. Canola processed commodities (PC) were not analyzed because there were no quantifiable residues (all <LOQ) in the canola seed from the exaggerated rate (5×) plot.

A total of 54 canola seed RAC samples were received at ABC Laboratories from the field test sites on October 10, 2013 and November 15, 2013. All study samples were analyzed for total residues of clomazone.

ANALYTICAL METHOD VALIDATION

The analytical method used to quantify residues of clomazone was validated according to SANCO 3029/99 rev. 4 guidelines on control samples of canola seed prior to analysis of any field samples in this study. The individual recoveries, overall means, standard deviations, and relative standard deviations (RSDs) from the quantitation ion data are summarized below (see Table 2a for more detailed data). Detailed results using confirmation ion data are provided in Table 2b. Comparisons between quantitation and confirmation ion results are provided in Table 2c.

Summary of Clomazone Method Validation Recoveries					
Matrix	**Fortification Level (ppm)**	**Sample Size (n)**	**Recoveries (%)**	**Mean Recovery (%) ± std. dev.**	**RSD (%)**
Canola seed	0.02	5	90, 95, 77, 66, 76	81 ± 12	14
	0.5	5	81, 92, 81, 73, 78	81 ± 7.0	8.6
	Overall	10	66 - 95	81 ± 9.0	11

The method was judged acceptable for use in this study.

PROCEDURAL RECOVERIES

Analytical method performance was monitored through concurrent analysis of freshly fortified control samples along with the field samples. The individual recoveries, overall means, standard deviations, and relative standard deviations are summarized below.

Summary of Clomazone Procedural Recoveries					
Matrix	**Fortification Level (ppm)**	**Sample Size (n)**	**Recoveries (%)**	**Mean Recovery (%) ± std. dev. (as applicable)**	**RSD[a] (%) (as applicable)**
Canola seed	0.02	7	81, 90, 92, 94, 100, 94, 68	88 ± 11	12
	0.50	5	95, 109, 112, 99, 100	103 ± 7.2	7.0
	Overall	12	68 - 112	95 ± 12	12

[a]NA = not applicable.

Residue values were not corrected for procedural recovery results.

RESIDUE ANALYSIS

The limit of quantitation (LOQ) for clomazone residues in or on canola seed was 0.02 ppm. The limit of detection (LOD) was estimated to be 0.0067 ppm (1/3 the LOQ).

No residues of clomazone >LOQ were found in any of the untreated canola seed RAC samples.

The range of residues found in treated samples, excluding decline samples are summarized below.

Clomazone: Magnitude of the Residue-RAC Range of Residues (ppm)				
Matrix	**Sampling Event (nominal)**	**Treatment Number**	**Formulation**	**Residues Found (ppm)[a,b]**
Canola seed	Maturity	2	Clomazone 360 g/L CS	ND – <0.02

[a]<0.0067 ppm represents values <LOD; <0.02 ppm represents values <LOQ
[b]ND = none detected, no chromatographic response.

The residues found in treated decline samples are summarized below.

Clomazone: Residue Decline - RAC (ppm)[a]						
Matrix	**Trial Number**	**Treatment Number**	**Formulation**	**Sampling Event[b] (nominal)**	**Clomazone (ppm)**	**Mean Residue (ppm)**
Canola seed	TCI-13-366-04	2	Clomazone 360 g/L CS	Maturity	ND	ND
		2		Maturity	ND	
		2		Maturity + 3 days	<0.0067	<0.0067
		2		Maturity + 3 days	ND	
		2		Maturity + 7 days	<0.0067	<0.0067
		2		Maturity + 7 days	<0.0067	
		2		Maturity + 14 days	<0.0067	<0.0067
		2		Maturity + 14 days	ND	
		2		Maturity + 21 days	ND	ND
		2		Maturity + 21 days	ND	
Canola seed	TCI-13-366-05	2	Clomazone 360 g/L CS	Maturity	<0.0067	<0.0067
		2		Maturity	<0.0067	
		2		Maturity + 3 days	ND	ND
		2		Maturity + 3 days	ND	
		2		Maturity + 7 days	ND	ND
		2		Maturity + 7 days	ND	
		2		Maturity + 14 days	ND	ND
		2		Maturity + 14 days	ND	
		2		Maturity + 21 days	ND	ND
		2		Maturity + 21 days	ND	

[a]<0.0067 ppm represents values <LOD; <0.02 ppm represents values <LOQ
[b]DALA = days after last application.

STORAGE STABILITY

A storage stability study has been conducted to determine the stability of clomazone in rape seed under deep-freezer conditions (Reference 2). Clomazone residues were stable in rape seed for at least 6 months. Additional storage stability testing will be conducted at ABC Laboratories as part of the on-going ABC Laboratories Study No. 81233 to determine the stability of clomazone in canola under deep-freezer conditions for greater than 6 months. The longest storage for canola seed samples in this study was 6.7 months (203 days).

2 INTRODUCTION

This study was conducted to determine the magnitude and decline of residues of clomazone in or on canola raw agricultural commodities (RAC) following one preemergence application of Clomazone 360 g/L CS at 0.42 kg ai/ha, and harvested at normal maturity with additional time points at two trial sites of 3, 7, 14, and 21 ± 1 days after normal maturity. Canola processed commodities (PC) were not analyzed because there were no quantifiable residues (all <LOQ) in the canola seed from the exaggerated rate (5×) plot.

The analytical portion of this study was conducted by ABC Laboratories, Inc. under Morse / ABC Study No. 69869 in accordance with Residue Study Protocol, Study No. TCI-13-366, entitled "Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)". This report contains the following: information on reference materials, experimental details, summary of the analytical methods, calculations, results and discussion, conclusions, residue data, procedural recovery data, storage stability data and representative chromatograms.

3 SAMPLE RECEIPT, LOGGING AND STORAGE

A total of 54 canola seed RAC samples were received at ABC Laboratories from the field test sites on October 10, 2013 and November 15, 2013.

Upon receipt, all study samples were transferred to a limited-access freezer for storage where they remained until they were processed/ground or weighed for analysis. All samples were logged in according to ABC Labs' SOPs using the original sample numbers assigned to them. Freezer storage temperatures were monitored on a daily basis and were at approximately -25 °C to -10 °C.

4 MATERIALS

4.1 Sample Matrices

The sample matrix investigated in this study was canola seed.

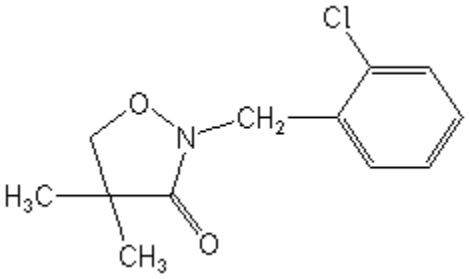
4.2 Reference Materials

The details of the analytical standards used for this study are provided below. No expired standards were used in this study. Certificates of Analysis are found in Appendix III.

4.2.1 *Clomazone*

Common Name: Clomazone
Chemical names:
CAS: 2-[(2-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone

IUPAC: 2-(2-chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one or
2-(2-chlorobenzyl)-4,4-dimethylisoxazolidin-3-one
Structural Formula:



CAS No.: 81777-89-1
Molecular Weight: 239.70 g/mol
Source: Cheminova A/S
Purity: 99.7% w/w
Batch No.: HKA, p. 4204
Receipt Date: May 14, 2010
Expiration Date: March 28, 2014; extended to February 11, 2016
Storage: Freezer (-25 °C to -10 °C)

5 PREPARATION OF STANDARD SOLUTIONS

The concentrations of clomazone standard solutions prepared and used in this study for residue analysis were as follows:

Identification/ Purpose	**Standard Solutions**	**Nominal Concentration**
Stock	Clomazone	1000 µg/mL
Fortification	Clomazone	100 µg/mL
		10 µg/mL
		1.0 µg/mL

Identification/ Purpose	Standard Solutions	Nominal Concentration
Intermediate	Clomazone	100 ng/mL
		20 ng/mL
		5.0 ng/mL
		1.0 µg/mL
Calibration	Clomazone	0.30 ng/mL
		0.15 ng/mL
		0.050 ng/mL
		0.030 ng/mL
		0.010 ng/mL

In accordance with ABC Labs' SOPs, storage stability analyses of standard solutions have been conducted, which demonstrate that the solutions were stable for at least the duration of their use in this study. All standard solutions prepared in this section were stored in the dark, refrigerated (2 °C to 8 °C) when not in use and were not used beyond the period of specified stability.

Details of the preparation of all standard solutions used in this study and their stability when stored as specified are provided in Appendix V.

6 METHODS

6.1 Sample Identification

Identification established in the field was used to uniquely identify each sample in the study. Additional designations such as "+ 0.020," were added to procedural/concurrent recovery samples, as appropriate.

6.2 Sample Preparation

All samples were ground between December 05, 2013 and December 13, 2013.

All samples were ground with dry ice to a homogeneous consistency in a Robot Coupe RSI 10Y vertical cutter/grinder. For large samples, representative subsamples were prepared from the ground samples for analysis.

All ground samples and subsamples were placed in frozen storage immediately after grinding. The dry ice was allowed to sublime prior to weighing the samples for extraction.

All the samples for this study were processed according to ABC Laboratories, SOP# CD-TM 1.11.1, entitled "Procedures for Sample Preparation Equipment."

6.3 Summary of Analytical Method

The full text of the method used in this study, including modifications, can be found in Appendix I.

Samples were analyzed using the method found in Eurofins-GAB GmbH Study Code 20061401/01-RVP Final Report, entitled "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape," dated January 17, 2007 (Reference 1), with ABC Laboratories method modifications, dated April 23, 2014. The method modification eliminated several post extraction steps including several filtration steps and performance of GPC cleanup.

Residues of clomazone were extracted from a 25-g seed sample by homogenization with acetone, acetonitrile, Calflo E, and Celite. An aliquot of the sample was centrifuged, and the supernatant filtered through a 0.45-µ PTFE filter. An aliquot of the filtered sample was combined with acetonitrile:water (1:1, v/v) and submitted for HPLC analysis.

Concurrent procedural recoveries demonstrate method suitability particularly at the LOQ, which was the level below which all of the residues in this study were observed. These methods, therefore, are considered to be equivalent. See Appendix I for complete text of the original method.

Determination and quantitation for clomazone were conducted using high performance liquid chromatography (HPLC) with triple quadrupole mass spectrometric (LC-MS/MS) detection. The LOQ for clomazone in canola seed was 0.02 ppm. The limit of detection (LOD) was estimated to be 0.0067 ppm (1/3 the LOQ).

6.4 Instrumentation

All samples were analyzed by HPLC employing triple quadrupole mass spectrometric (MS/MS) detection (LC-MS/MS). Typical conditions were as follows:

- **Typical Operating conditions**

Instrument: Applied Biosystems/Sciex API 5000 LC/MS/MS System with Acquity UPLC System using Applied Biosystems/MDS Sciex Analyst Software for data collection and system control (version 1.5).

HPLC Column: 50 mm × 2.1 mm i.d. Waters Acquity HSS T3, 1.8 µ particle size

Mobile Phase:

Component A: 0.1% formic acid in water

Component B: 0.1% formic acid in methanol

Gradient:

Time (min)	% A	% B
0.0-6.0	80	20
6.0-7.0	10	90
7.01-8.0	80	20

Flow Rate: 0.5 mL/min.

Interface: TIS (turbo ion spray)

Ionization Mode: Positive (+)

Acquisition Mode: MRM

Source Temperature: 600 °C

Curtain Gas: Nitrogen @ setting of "40"

Collision Gas: Nitrogen @ setting of "10"

Injection Volume: 5 µL

Column Temperature: 40 °C

Autosampler Tray Temperature: 10 °C

Transitions Monitored:

Ion, *m/z* Q1	Ion, *m/z* Q3	Time, *ms*	CE, *v*	
240.1	125.0	200	31	(quantitation)
240.1	89.1	200	65	(confirmation)

Retention Time: ~3.9 minutes

- **Calibration/sample analysis**

A five-point standard curve was prepared by injecting constant volumes of standard solutions at appropriate concentrations. Constant volume injections were used for sample extracts as well. A curve check standard was typically injected every 3-4 sample injections.

6.5 Calculations

Calculations for instrumental analysis were conducted using a validated software application to create a standard curve based on linear regression. The regression functions were used to calculate a best-fit line (from a set of standard concentrations in ng/mL versus peak response) and to determine concentrations of the analyte found during sample analysis from the calculated best-fit line. Weighting (1/x) was used in the generation of standard curves.

The equation used for the least squares fit is:

$$y = mx + b$$

where:

y	=	peak response
m	=	slope
x	=	ng/mL found for peak of interest
b	=	y-intercept

The standard (calibration) curve generated for each analytical set was used for the quantitation of clomazone in the samples. For this study, the correlation

coefficient (R) for each calibration curve for each analyte was equal to or greater than 0.990 (R2 equal to or greater than 0.98).

- **Equations**

The calculation for ppm found and percent recovery were as follows:

1. The amount of clomazone found (in ppm) in the sample is calculated according to the following equation:

$$ppm = ng/mL \times \frac{HPLC\ FV\ (mL)}{sample\ \ wt.\ (g)} \times \frac{ext.solv.(mL)}{aliq.(mL)} \times HPLC\ dil.\ factor \times \frac{1}{1000}$$

where:

ng/mL	=	ng/mL of analyte found as determined by Analyst
HPLC FV (mL)	=	volume of final extract submitted to instrumentation (10 mL)
sample wt. (g)	=	amount of sample taken through the extraction process (25.0 g)
ext. solv. (mL)	=	volume of extraction solvent added (250 mL)
aliq. (mL)	=	volume of extract taken for analysis (0.10 mL)
HPLC dil. factor	=	dilution of sample extract required to produce an analyte response bracketed by standards
1/1000	=	conversion factor (µg/ng)

2. The percent recovery for fortified control samples was calculated as follows:

$$\%\ Recovery = \frac{ppm\ \ found\ in\ \ fortified\ control\ -\ ppm\ \ found\ in\ control}{ppm\ added} \times 100$$

- **Example calculations**

1. ABC ID 69869-006, Canola Seed, Set #1 short, TCI-13-366-02-01, **Control**:

$$0 \text{ peak response} \rightarrow 0.00 \text{ ng/mL}$$

$$ppm = 0.00\ ng/mL \times \frac{10\ mL}{25.0\ g} \times \frac{250\ mL}{0.10\ mL} \times 1 \times \frac{1}{1000}$$

$$ppm = 0.000$$

$$Reported\ ppm = ND$$

2. ABC ID 69869-007, Canola Seed, Set #1 short, TCI-13-366-02-01, **Fortified Control** @ 0.020 ppm:

$$9944\ \ peak\ response \rightarrow 0.0161\ ng/mL$$

$$ppm = 0.0161\ ng/mL \times \frac{10\ mL}{25.0\ g} \times \frac{250\ mL}{0.10\ mL} \times 1 \times \frac{1}{1000}$$

$$\text{ppm} = 0.0161$$

$$\text{Reported ppm} = 0.0161$$

$$\%\ Rec. = \frac{0.0161\ ppm - 0.000\ ppm}{0.020\ ppm} \times 100$$

$$= 81\%$$

3. ABC ID 69869-019, Canola Seed, Set #2, TCI-13-366-06-04, **Field Sample**:

$$8103\ \ peak\ response \rightarrow 0.00727\ ng/mL$$

$$ppm = 0.00727\ ng/mL \times \frac{10\ mL}{25.0\ g} \times \frac{250\ mL}{0.10\ mL} \times 1 \times \frac{1}{1000}$$

$$ppm = 0.00727$$

$$Reported\ ppm = < 0.020$$

6.6 Sample Analysis

The canola seed samples were analyzed in a group referred to as an "analytical set". The set typically consisted of one control sample, two or three fortified control samples and up to 16 field samples.

Moisture determination was conducted on one canola seed sample per trial following ABC Laboratories SOP# CD-TM 2.31.6, entitled, "Determination of Moisture Content." See Appendix II for the SOP.

6.7 Representative Chromatography

For HPLC analyses, example chromatograms of HPLC standards and the associated calibration curve graph are presented in this report as Figures 1 through 6. Example chromatograms of three untreated control, three fortified control (procedural recovery) samples, and ten treated samples are presented as Figures 7 through 22 for canola seed.

Example confirmation chromatograms of HPLC standards and the associated calibration curve graph are presented in this report as Figures 23 through 28 for confirmatory ion (*m/z* 240/89). Example confirmation chromatograms of one control and two fortified controls are presented as Figures 29 through 31.

6.8 Statistics

Statistical methods used were limited to calculations of the mean, range, and standard deviation. Microsoft Excel® 2007-2010 and validated software program, Applied BioSystems/MDS Sciex Analyst software (version 1.5) were employed to develop all statistical data.

7 RESULTS AND DISCUSSION

Data resulting from the analyses in this study are summarized in Tables 2a through 5. Supporting raw data spreadsheets are located in Appendix IV. The chronology of events from sampling to HPLC analysis is presented in Table 1.

7.1 Method Validation Results

The analytical method used to quantify clomazone residues was validated according to SANCO 3029/99 rev. 4 guidelines on control samples of canola seed prior to analysis of any field samples in this study. The individual recoveries, overall means, standard deviations, and relative standard deviations (RSDs) from

the quantitation ion data are summarized below (see Table 2a for more detailed data). Detailed results using confirmation ion data are provided in Table 2b. Comparisons between quantitation and confirmation ion results are provided in Table 2c.

Analytical method performance was monitored through concurrent analysis of freshly fortified control samples along with the field samples.

Summary of Clomazone Method Validation Recoveries					
Matrix	**Fortification Level (ppm)**	**Sample Size (n)**	**Recoveries (%)**	**Mean Recovery (%) ± std. dev.**	**RSD (%)**
Canola seed	0.02	5	90, 95, 77, 66, 76	81 ± 12	14
	0.5	5	81, 92, 81, 73, 78	81 ± 7.0	8.6
	Overall	10	66 - 95	81 ± 9.0	11

The method was deemed acceptable for use in this study. The individual recovery for each fortified control sample was in the range of 70-120% with the exception of one recovery at the 0.02 ppm fortification level (66%). The mean recovery for replicates at each fortification level was in the range of 70-110%, with corresponding RSDs of replicate recovery measurements ≤20%. The residues found in the control samples were less than the LOQ. Based on the precision and accuracy of the recovery data, this method meets European Commission and U.S. EPA guideline requirements.

Because the method used triple quadrupole mass spectrometric (LC-MS/MS) detection, it is considered specific for the analyte targeted. The correlation coefficient (r) of the calibration curve (>0.995) generated during method validation demonstrated a valid relationship (linear regression) between standard concentration and instrument response. These data indicated that the method would perform well during the course of this study.

7.2 Procedural Recovery Results

Analytical method performance was monitored through concurrent analysis of freshly fortified control samples along with the field samples. The individual recoveries, overall means, standard deviations, and relative standard deviations are summarized below.

Summary of Clomazone Procedural Recoveries					
Matrix	**Fortification Level (ppm)**	**Sample Size (n)**	**Recoveries (%)**	**Mean Recovery (%) ± std. dev. (as applicable)**	**RSD[a] (%) (as applicable)**
Canola seed	0.02	7	81, 90, 92, 94, 100, 94, 68	88 ± 11	12
	0.50	5	95, 109, 112, 99, 100	103 ± 7.2	7.0
	Overall	12	68 - 112	95 ± 12	12

[a]NA = not applicable.

Residue values were not corrected for procedural recovery results.

7.3 Control and Sample Residue Results

The limit of quantitation (LOQ) for clomazone residues in or on canola seed was 0.02 ppm.

No clomazone residues >LOQ were found in any of the untreated canola seed RAC.

The range of residues found in treated samples, excluding RAC decline samples are summarized below.

Clomazone: Magnitude of the Residue-RAC Range of Residues (ppm)				
Matrix	**Sampling Event (nominal)**	**Treatment Number**	**Formulation**	**Residues Found (ppm)[a,b]**
Canola seed	Maturity	2	Clomazone 360 g/L CS	ND - <0.02

[a]<0.0067 ppm represents values <LOD; <0.02 ppm represents values <LOQ
[b]ND = none detected, no chromatographic response.

The residues found in treated decline samples are summarized below.

Clomazone: Residue Decline - RAC (ppm)[a]						
Matrix	**Trial Number**	**Treatment Number**	**Formulation**	**Sampling Event[b] (nominal)**	**Clomazone (ppm)**	**Mean Residue (ppm)**
Canola seed	TCI-13-366-04	2	Clomazone 360 g/L CS	Maturity	ND	ND
		2		Maturity	ND	
		2		Maturity + 3 days	<0.0067	<0.0067
		2		Maturity + 3 days	ND	
		2		Maturity + 7 days	<0.0067	<0.0067
		2		Maturity + 7 days	<0.0067	
		2		Maturity + 14 days	<0.0067	<0.0067
		2		Maturity + 14 days	ND	
		2		Maturity + 21 days	ND	ND
		2		Maturity + 21 days	ND	

[a]<0.0067 ppm represents values <LOD; <0.02 ppm represents values <LOQ
[b]DALA = days after last application.

Canola seed	TCI-13-366-05	2	Clomazone 360 g/L CS	Maturity	<0.0067	<0.0067
		2		Maturity	<0.0067	
		2		Maturity + 3 days	ND	ND
		2		Maturity + 3 days	ND	
		2		Maturity + 7 days	ND	ND
		2		Maturity + 7 days	ND	
		2		Maturity + 14 days	ND	ND
		2		Maturity + 14 days	ND	
		2		Maturity + 21 days	ND	ND
		2		Maturity + 21 days	ND	

[a]<0.0067 ppm represents values <LOD; <0.02 ppm represents values <LOQ
[b]DALA = days after last application.

Individual clomazone residue results from field samples are provided in Table 4.

7.4 Freezer Storage Stability

A storage stability study has been conducted to determine the stability of clomazone in rape seed (canola) under deep-freezer conditions (Reference 2). Clomazone residues were stable in rape seed for at least 6 months. Additional storage stability testing will be conducted at ABC Laboratories as part of the on-going ABC Laboratories Study No. 81233 to determine the stability of clomazone in canola under deep-freezer conditions for greater than 6 months. The longest storage for canola seed samples in this study was 6.7 months (203 days).

7.5 Stability of Extracts

The storage stability of the extracts (stored refrigerated at 2 °C to 8 °C) was verified by always storing the procedural recovery samples together with the field samples under the same conditions for each analytical set. Reported recovery results indicate that the extracts were stable during each time interval.

7.6 LOQ and LOD

The limit of quantitation (LOQ) in this study was the lowest level at which acceptable recoveries were achieved during the course of the study (0.02 ppm). The limit of detection (LOD) was defined as ⅓ the respective LOQ or 0.0067 ppm.

7.7 Moisture Determination

Moisture determination was conducted on one canola seed sample from each trial. Results are provided in Table 4.

7.8 Protocol/SOP/Method Deviations

Six protocol deviations were issued for this phase of the study. Two deviations, both dated March 18, 2014, documented one method validation recovery (primary ion transition) at 66%, which is less than the required 70%; and that the Day 0 storage stability will be performed independent of the method verification. The third deviation, dated April 21, 2014, documented one method validation recovery (primary ion transition) at 68%, which is less than the required 70%. The fourth deviation, dated June 2, 2014, documented that the clomazone analytical standard was stored at -10 to -25 °C instead of <-20 °C as required by the CoA. The fifth deviation, dated June 11, 2014, documented a comparison of the original and modified methods without preparing a protocol amendment. The sixth deviation, dated June 16, 2014 documented recoveries for the unmodified method of 68% and 64% (primary ion transition).

Two SOP deviations were issued for this phase of the study, both dated May 12, 2012. The first deviation documented that corrections and additions to original data may have been performed on a day other than the day the original entry was prepared. The second deviation documented that statistical analysis was sometimes performed on rounded numbers in Excel.

The deviations described above are considered to have no impact on the integrity of the study.

8 CONCLUSION

The overall recovery ranges, means, standard deviations, and RSD's for the procedural recoveries analyzed and described in this report indicate that the samples were successfully analyzed for clomazone.

Analysis of three samples from Trial 6 using the original method showed that treated sample results from the modified method and the original method were identical. Supporting data is located in Appendix IV.

9 DISPOSITION OF SAMPLES AND RAW DATA

Samples not consumed in this study, which are judged by the Study Director and Sponsor to be preservable under frozen storage conditions, will be kept in storage at ABC Labs until approval is obtained for transfer or disposal.

All original study-related raw data and the final report will be transferred to the Sponsor's permanent archival facility, EPL Archives, Inc., 45610 Terminal Drive, Sterling, VA 20166. Original facility-related data, as well as verified scans of the raw data and an authentic copy of the Final Analytical Report, will be maintained in the archive facilities of ABC Laboratories, Inc. in Columbia, Missouri.

10 STUDY PERSONNEL

The following personnel were responsible for the conduct of the study:

	Job Title
Sample Preparation:	
Thomas Balko	Sample Prep Technician
Kerri Chapin	Principal Sample Prep Technician
Jessica Matwiczak	Sample Prep Technician
Mark Tunink	Supervisor
Stacy Simpson	Sample Prep Supervisor
Vickie Stith	Sr. Sample Prep Technician
Extractions:	
Elen Tesfai	Assistant Chemist
Christine Ehrmann	Assistant Chemist

Instrumentation:

Jeffery Brendler	Senior Chemist
Marci Staley	Chemist
Scotty Reynolds	Assistant Chemist
Ashley Seifert	Assistant Chemist

Calculations:

Liz Hansen	Laboratory Application Analyst

Data Review:

Amy Gaines	Chemist

Principal Analytical Investigator:

Carol Rodgers	Chemist

Report Author:

Carol Rodgers	Chemist

11 REFERENCES

1. Fiedler, E., Eurofins-GAB GmbH Study Code 20061401/01-RVP, entitled "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape," dated January 17, 2007.

2. Fiedler, E., Eurofins-GAB GmbH Report Number 20054075/01-RSS, entitled "Determination of the Storage Stability of Clomazone in Potatoes and Oil Seed Rape at approximately – 20 °C," dated June 28, 2007.

3. Canadian Pest Management Regulatory Agency (PMRA) Residue Chemistry Guidelines Section 9, Crop Field Trials, Regulatory Directive 98-02, 1998.

4. Canadian Pest Management Regulatory Agency (PMRA) Revisions to the Residue Chemistry Crop Field Trial Requirements, Regulatory Directive DIR2010-05, December 21, 2010.

5. European Commission. Residue Guidance Document - SANCO 3029/99 rev. 4. 11/07/00 - Generating and Reporting Methods of Analysis.

6. Rodgers, C.A., ABC Laboratories Study No. 81233, entitled "Evaluation of Frozen Storage Stability of Clomazone Residues in or on Canola Raw Agricultural Commodities," Ongoing.

TABLES

TABLE OF ABBREVIATIONS

Abbreviations used in Tables 1 through 5 are defined below:

DWB	=	dry weight basis
Fort.	=	fortification
ID	=	identification
LOD	=	limit of detection
LOQ	=	limit of quantitation
MV	=	method validation
m/z	=	mass to charge ratio
n	=	number
N/A	=	not applicable
ND	=	not detected
No.	=	number
ppm	=	parts per million
RAC	=	raw agricultural commodity
RSD	=	relative standard deviation
Std. Dev.	=	standard deviation
Trt.	=	treatment

TABLE 1

Sample Chronology Data

Sample Identification	Trt No.	Matrix	Sample Type	Sampling Date	Lab Receipt Date	Date Sample Ground/ Composited	Extraction Date	Injection Date	Sampling to Extraction Interval (days)
TCI-13-366-01-01	1	Canola seed	Control	12-Sep-13	10-Oct-13	05-Dec-13	14-Mar-14	17-Mar-14	183
TCI-13-366-01-02	2	Canola seed	Treated	12-Sep-13	10-Oct-13	05-Dec-13	14-Mar-14	17-Mar-14	183
TCI-13-366-01-03	2	Canola seed	Treated	12-Sep-13	10-Oct-13	05-Dec-13	14-Mar-14	17-Mar-14	183
TCI-13-366-02-01	1	Canola seed	Control	12-Sep-13	10-Oct-13	05-Dec-13	25-Feb-14	26-Feb-14	166
TCI-13-366-02-02	2	Canola seed	Treated	12-Sep-13	10-Oct-13	06-Dec-13	25-Feb-14	26-Feb-14	166
TCI-13-366-02-03	2	Canola seed	Treated	12-Sep-13	10-Oct-13	06-Dec-13	25-Feb-14	26-Feb-14	166
TCI-13-366-03-01	1	Canola seed	Control	13-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	186
TCI-13-366-03-02	2	Canola seed	Treated	13-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	186
TCI-13-366-03-03	2	Canola seed	Treated	13-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	186
TCI-13-366-04-01	1	Canola seed	Control	7-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	192
TCI-13-366-04-02	2	Canola seed	Treated	7-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	192
TCI-13-366-04-03	2	Canola seed	Treated	7-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	192
TCI-13-366-04-04	2	Canola seed	Treated	10-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	189
TCI-13-366-04-05	2	Canola seed	Treated	10-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	189
TCI-13-366-04-06	2	Canola seed	Treated	14-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	185
TCI-13-366-04-07	2	Canola seed	Treated	14-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	185
TCI-13-366-04-08	2	Canola seed	Treated	21-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	178
TCI-13-366-04-09	2	Canola seed	Treated	21-Sep-13	10-Oct-13	06-Dec-13	18-Mar-14	19-Mar-14	178
TCI-13-366-04-10	2	Canola seed	Treated	28-Sep-13	15-Nov-13	09-Dec-13	18-Mar-14	19-Mar-14	171

TABLE 1 (continued)

Sample Chronology Data

Sample Identification	Trt No.	Matrix	Sample Type	Sampling Date	Lab Receipt Date	Date Sample Ground/ Composited	Extraction Date	Injection Date	Sampling to Extraction Interval (days)
TCI-13-366-04-11	2	Canola seed	Treated	28-Sep-13	15-Nov-13	09-Dec-13	18-Mar-14	19-Mar-14	171
TCI-13-366-05-01	1	Canola seed	Control	19-Sep-13	10-Oct-13	09-Dec-13	18-Mar-14	19-Mar-14	180
TCI-13-366-05-02	2	Canola seed	Treated	19-Sep-13	10-Oct-13	09-Dec-13	18-Mar-14	19-Mar-14	180
TCI-13-366-05-03	2	Canola seed	Treated	19-Sep-13	10-Oct-13	09-Dec-13	18-Mar-14	19-Mar-14	180
TCI-13-366-05-04	2	Canola seed	Treated	19-Sep-13	10-Oct-13	09-Dec-13	21-Mar-14	21-Mar-14	183
TCI-13-366-05-05	2	Canola seed	Treated	19-Sep-13	10-Oct-13	09-Dec-13	21-Mar-14	21-Mar-14	183
TCI-13-366-05-06	2	Canola seed	Treated	19-Sep-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	183
TCI-13-366-05-07	2	Canola seed	Treated	19-Sep-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	183
TCI-13-366-05-08	2	Canola seed	Treated	1-Oct-13	15-Nov-13	10-Dec-13	21-Mar-14	21-Mar-14	171
TCI-13-366-05-09	2	Canola seed	Treated	1-Oct-13	15-Nov-13	10-Dec-13	21-Mar-14	21-Mar-14	171
TCI-13-366-05-10	2	Canola seed	Treated	1-Oct-13	15-Nov-13	10-Dec-13	21-Mar-14	21-Mar-14	171
TCI-13-366-05-11	2	Canola seed	Treated	1-Oct-13	15-Nov-13	10-Dec-13	21-Mar-14	21-Mar-14	171
TCI-13-366-06-01	1	Canola seed	Control	24-Sep-13	15-Nov-13	05-Dec-13	14-Mar-14	17-Mar-14	171
TCI-13-366-06-02	2	Canola seed	Treated	24-Sep-13	15-Nov-13	05-Dec-13	14-Mar-14	17-Mar-14	171
TCI-13-366-06-03	2	Canola seed	Treated	24-Sep-13	15-Nov-13	05-Dec-13	14-Mar-14	17-Mar-14	171
TCI-13-366-06-04	4	Canola seed	Treated	24-Sep-13	15-Nov-13	05-Dec-13	14-Mar-14	17-Mar-14	171
TCI-13-366-06-05	4	Canola seed	Treated	24-Sep-13	15-Nov-13	05-Dec-13	14-Mar-14	17-Mar-14	171

TABLE 1 (continued)

Sample Chronology Data

Sample Identification	Trt No.	Matrix	Sample Type	Sampling Date	Lab Receipt Date	Date Sample Ground/ Composited	Extraction Date	Injection Date	Sampling to Extraction Interval (days)
TCI-13-366-07-01	1	Canola seed	Control	5-Sep-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	197
TCI-13-366-07-02	2	Canola seed	Treated	5-Sep-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	197
TCI-13-366-07-03	2	Canola seed	Treated	5-Sep-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	197
TCI-13-366-08-01	1	Canola seed	Control	30-Aug-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	203
TCI-13-366-08-02	2	Canola seed	Treated	30-Aug-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	203
TCI-13-366-08-03	2	Canola seed	Treated	30-Aug-13	10-Oct-13	10-Dec-13	21-Mar-14	21-Mar-14	203
TCI-13-366-09-01	1	Canola seed	Control	13-Sep-13	10-Oct-13	11-Dec-13	21-Mar-14	22-Mar-14	189
TCI-13-366-09-02	2	Canola seed	Treated	13-Sep-13	10-Oct-13	11-Dec-13	21-Mar-14	22-Mar-14	189
TCI-13-366-09-03	2	Canola seed	Treated	13-Sep-13	10-Oct-13	11-Dec-13	21-Mar-14	22-Mar-14	189
TCI-13-366-10-01	1	Canola seed	Control	20-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	187
TCI-13-366-10-02	2	Canola seed	Treated	20-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	187
TCI-13-366-10-03	2	Canola seed	Treated	20-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	187
TCI-13-366-11-01	1	Canola seed	Control	6-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	201
TCI-13-366-11-02	2	Canola seed	Treated	6-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	201
TCI-13-366-11-03	2	Canola seed	Treated	6-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	201
TCI-13-366-12-01	1	Canola seed	Control	19-Sep-13	10-Oct-13	11-Dec-13	26-Mar-14	26-Mar-14	188
TCI-13-366-12-02	2	Canola seed	Treated	19-Sep-13	10-Oct-13	13-Dec-13	26-Mar-14	26-Mar-14	188
TCI-13-366-12-03	2	Canola seed	Treated	19-Sep-13	10-Oct-13	13-Dec-13	26-Mar-14	26-Mar-14	188

TABLE 2a

Method Validation Recoveries for Clomazone from Canola Seed (Quantitation Ion *m/z* 240/125)

Sample Identification	Set No.	Extraction Date	Injection Date	Fort. Level (ppm)	Residues Found[a] (ppm)	% Recovery
TCI-13-366-02-01	MV2	21-Feb-14	21-Feb-14	N/A	ND	N/A
TCI-13-366-02-01	MV2	21-Feb-14	21-Feb-14	N/A	ND	N/A
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0180	90
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0189	95
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0154	77
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0131	66
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0151	76
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.406	81
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.458	92
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.403	81
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.366	73
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.389	78

[a]Fortified control samples are corrected for mean control contribution, if any.

STATISTICS

Fortification Level:	0.020 ppm	0.50 ppm	Overall
Mean Recovery (%):	81	81	81
Std. Dev. (±):	12	7.0	9.0
RSD (%):	14	8.6	11
Range (%):	66 - 95	73 - 92	66 - 95
n:	5	5	10

TABLE 2b

Method Validation Recoveries for Clomazone from Canola Seed (Confirmation Ion *m/z* 240/89)

Sample Identification	Set No.	Extraction Date	Injection Date	Fort. Level (ppm)	Residues Found[a] (ppm)	% Recovery
TCI-13-366-02-01	MV2	21-Feb-14	21-Feb-14	N/A	ND	N/A
TCI-13-366-02-01	MV2	21-Feb-14	21-Feb-14	N/A	ND	N/A
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0170	85
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0223	112
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0163	82
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0140	70
TCI-13-366-02-01 + 0.020	MV2	21-Feb-14	21-Feb-14	0.020	0.0157	79
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.432	86
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.469	94
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.400	80
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.410	82
TCI-13-366-02-01 + 0.50	MV2	21-Feb-14	21-Feb-14	0.50	0.388	78

[a]Fortified control samples are corrected for mean control contribution, if any.

STATISTICS

Fortification Level:	0.020 ppm	0.50 ppm	Overall
Mean Recovery (%):	86	84	85
Std. Dev. (±):	16	6.3	11
RSD (%):	18	7.5	13
Range (%):	70 - 112	78 - 94	70 - 112
n:	5	5	10

TABLE 2c

Method Validation Recoveries for Clomazone from Canola Seed (Quantitation/Confirmation Ion Comparisons)

	Quantitation Ion (*m/z* 240/125)				Confirmation Ion (*m/z* 240/89)			
Fortification Level (ppm)	**Residues Found[a] (ppm)**	**Recovery (%)**	**Mean Recovery ± RSD**	**Overall Mean[b] ± RSD**	**Residues Found[a] (ppm)**	**Recovery (%)**	**Mean Recovery ± RSD**	**Overall Mean[b] ± RSD**
0.020	0.0180	90	81 ± 14	81 ± 11	0.0170	85	86 ± 18	85 ± 13
0.020	0.0189	95			0.0223	112		
0.020	0.0154	77			0.0163	82		
0.020	0.0131	66			0.0140	70		
0.020	0.0151	76			0.0157	79		
0.50	0.406	81	81 ± 8.6		0.432	86	84 ± 7.5	
0.50	0.458	92			0.469	94		
0.50	0.403	81			0.400	80		
0.50	0.366	73			0.410	82		
0.50	0.389	78			0.388	78		

[a]Corrected for mean control contribution, if any.
[b]Overall mean recovery, calculated from all individual results.

TABLE 3

Laboratory Fortification (Procedural) Recoveries for Clomazone from Canola Seed

Sample Identification	Set No.	Extraction Date	Injection Date	Fort. Level (ppm)	Residues Found[a,b] (ppm)	% Recovery
TCI-13-366-02-01	1 short	25-Feb-14	26-Feb-14	N/A	ND	N/A
TCI-13-366-02-01 + 0.020	1 short	25-Feb-14	26-Feb-14	0.020	0.0161	81
TCI-13-366-02-01 + 0.50	1 short	25-Feb-14	26-Feb-14	0.50	0.477	95
TCI-13-366-01-01	2	14-Mar-14	17-Mar-14	N/A	<0.0067	N/A
TCI-13-366-01-01 + 0.020	2	14-Mar-14	17-Mar-14	0.020	0.0180	90
TCI-13-366-01-01 + 0.50	2	14-Mar-14	17-Mar-14	0.50	0.546	109
TCI-13-366-03-01	4	18-Mar-14	19-Mar-14	N/A	ND	N/A
TCI-13-366-03-01 + 0.020	4	18-Mar-14	19-Mar-14	0.020	0.0184	92
TCI-13-366-03-01 + 0.50	4	18-Mar-14	19-Mar-14	0.50	0.560	112
TCI-13-366-03-01 + 0.020	4	18-Mar-14	19-Mar-14	0.020	0.0187	94
TCI-13-366-07-01	7	21-Mar-14	21-Mar-14	N/A	<0.0067	N/A
TCI-13-366-07-01 + 0.020	7	21-Mar-14	21-Mar-14	0.020	0.0200	100
TCI-13-366-07-01 + 0.50	7	21-Mar-14	21-Mar-14	0.50	0.495	99
TCI-13-366-07-01 + 0.020	7	21-Mar-14	21-Mar-14	0.020	0.0187	94
TCI-13-366-10-01	8	26-Mar-14	26-Mar-14	N/A	<0.0067	N/A
TCI-13-366-10-01 + 0.020	8	26-Mar-14	26-Mar-14	0.020	0.0136	68
TCI-13-366-10-01 + 0.50	8	26-Mar-14	26-Mar-14	0.50	0.499	100

[a]Fortified control samples are corrected for control contribution, if any.
[b]Values <LOD are reported as <0.0067 ppm; values <LOQ are reported as <0.02 ppm.

STATISTICS

Fortification Level:	0.020 ppm	0.50 ppm	Overall
Mean Recovery (%):	88	103	95
Std. Dev. (±):	11	7.2	12
RSD (%):	12	7.0	12
Range (%):	68 - 100	95 - 112	68 - 112
n:	7	5	12

TABLE 4

Residues of Clomazone Found in Canola Seed RAC Samples

Sample Identification	Matrix	Treatment ID	Sampling Event (nominal)	Set No.	% Moisture (DWB)	Residues Found[a] (ppm)
TCI-13-366-01-01	Canola seed	1	Maturity	2	11.87	<0.0067
TCI-13-366-01-02	Canola seed	2	Maturity	2	--	<0.0067
TCI-13-366-01-03	Canola seed	2	Maturity	2	--	<0.0067
TCI-13-366-02-01	Canola seed	1	Maturity	1 short	16.59	ND
TCI-13-366-02-02	Canola seed	2	Maturity	1 short	--	ND
TCI-13-366-02-03	Canola seed	2	Maturity	1 short	--	ND
TCI-13-366-03-01	Canola seed	1	Maturity	4	9.83	ND
TCI-13-366-03-02	Canola seed	2	Maturity	4	--	<0.0067
TCI-13-366-03-03	Canola seed	2	Maturity	4	--	ND
TCI-13-366-04-01	Canola seed	1	Maturity	4	20.55	<0.0067
TCI-13-366-04-02	Canola seed	2	Maturity	4	--	ND
TCI-13-366-04-03	Canola seed	2	Maturity	4	--	ND
TCI-13-366-04-04	Canola seed	2	Maturity + 3 days	4	--	<0.0067
TCI-13-366-04-05	Canola seed	2	Maturity + 3 days	4	--	ND
TCI-13-366-04-06	Canola seed	2	Maturity + 7 days	4	--	<0.0067
TCI-13-366-04-07	Canola seed	2	Maturity + 7 days	4	--	<0.0067
TCI-13-366-04-08	Canola seed	2	Maturity + 14 days	4	--	<0.0067
TCI-13-366-04-09	Canola seed	2	Maturity + 14 days	4	--	ND
TCI-13-366-04-10	Canola seed	2	Maturity + 21 days	4	--	ND
TCI-13-366-04-11	Canola seed	2	Maturity + 21 days	4	--	ND
TCI-13-366-05-01	Canola seed	1	Maturity	4	8.92	<0.0067
TCI-13-366-05-02	Canola seed	2	Maturity	4	--	<0.0067
TCI-13-366-05-03	Canola seed	2	Maturity	4	--	<0.0067
TCI-13-366-05-04	Canola seed	2	Maturity + 3 days	7	--	ND
TCI-13-366-05-05	Canola seed	2	Maturity + 3 days	7	--	ND
TCI-13-366-05-06	Canola seed	2	Maturity + 7 days	7	--	ND
TCI-13-366-05-07	Canola seed	2	Maturity + 7 days	7	--	ND
TCI-13-366-05-08	Canola seed	2	Maturity + 14 days	7	--	ND
TCI-13-366-05-09	Canola seed	2	Maturity + 14 days	7	--	ND
TCI-13-366-05-10	Canola seed	2	Maturity + 21 days	7	--	ND
TCI-13-366-05-11	Canola seed	2	Maturity + 21 days	7	--	ND

[a]Values <LOD are reported as <0.0067 ppm; values <LOQ are reported as <0.02 ppm.

TABLE 4 (continued)

Residues of Clomazone Found in Canola Seed RAC Samples

Sample Identification	Matrix	Treatment ID	Sampling Event (nominal)	Set No.	% Moisture (DWB)	Residues Found[a] (ppm)
TCI-13-366-06-01	Canola seed	1	Maturity	2	18.25	ND
TCI-13-366-06-02	Canola seed	2	Maturity	2	--	ND
TCI-13-366-06-03	Canola seed	2	Maturity	2	--	<0.0067
TCI-13-366-06-04	Canola seed	4	Maturity	2	--	<0.02
TCI-13-366-06-05	Canola seed	4	Maturity	2	--	<0.0067
TCI-13-366-07-01	Canola seed	1	Maturity	7	13.70	<0.0067
TCI-13-366-07-02	Canola seed	2	Maturity	7	--	ND
TCI-13-366-07-03	Canola seed	2	Maturity	7	--	ND
TCI-13-366-08-01	Canola seed	1	Maturity	7	15.41	ND
TCI-13-366-08-02	Canola seed	2	Maturity	7	--	ND
TCI-13-366-08-03	Canola seed	2	Maturity	7	--	ND
TCI-13-366-09-01	Canola seed	1	Maturity	7	8.81	ND
TCI-13-366-09-02	Canola seed	2	Maturity	7	--	ND
TCI-13-366-09-03	Canola seed	2	Maturity	7	--	ND
TCI-13-366-10-01	Canola seed	1	Maturity	8	35.28	<0.0067
TCI-13-366-10-02	Canola seed	2	Maturity	8	--	ND
TCI-13-366-10-03	Canola seed	2	Maturity	8	--	ND
TCI-13-366-11-01	Canola seed	1	Maturity	8	18.37	<0.0067
TCI-13-366-11-02	Canola seed	2	Maturity	8	--	ND
TCI-13-366-11-03	Canola seed	2	Maturity	8	--	ND
TCI-13-366-12-01	Canola seed	1	Maturity	8	13.06	ND
TCI-13-366-12-02	Canola seed	2	Maturity	8	--	ND
TCI-13-366-12-03	Canola seed	2	Maturity	8	--	<0.0067

[a]Values <LOD are reported as <0.0067 ppm; values <LOQ are reported as <0.02 ppm.

TABLE 5

Comparison of Clomazone Found in Canola Seed RAC Samples (Original Method vs Modified Method)

Sample Identification	Trt ID	Original				Modified			
		Set No.	Extraction Date	Injection Date	Residues Found[a,b] (ppm)	Set No.	Extraction Date	Injection Date	Residues Found[a,b] (ppm)
TCI-13-366-06-01	01	10 long	15-May-14	19-May-14	<0.0067	2	14-Mar-14	17-Mar-14	ND
TCI-13-366-06-02	02	10 long	15-May-14	19-May-14	<0.0067	2	14-Mar-14	17-Mar-14	ND
TCI-13-366-06-03	02	10 long	15-May-14	19-May-14	<0.0067	2	14-Mar-14	17-Mar-14	<0.0067

[a]Fortified control samples are corrected for control contribution, if any.
[b]Values <LOD are reported as <0.0067 ppm; values <LOQ are reported as <0.02 ppm.
Note: Recoveries for Set No. 10 long were 68 and 64% for the LOQ and 25× LOQ fortifications, respectively.

CHROMATOGRAMS

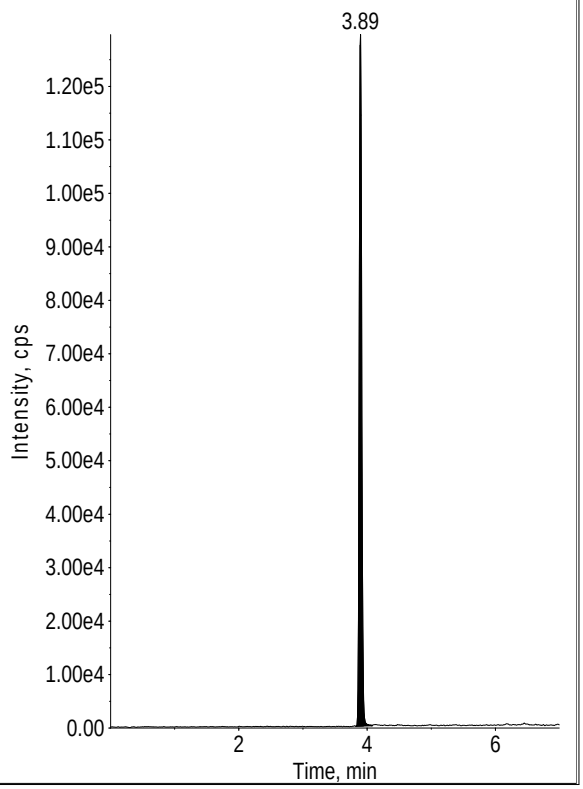
The following chromatograms may show a variation in the analyte retention times from one set to another due to different instrumentation parameters. In all cases, the analytical standard responses correlate with the respective samples run within an analytical set. For specific instrumentation conditions, see Section 6.4.

FIGURE 1

TYPICAL CLOMAZONE HPLC STANDARD
Set #7

Sample Name: "12562-B6" Sample ID: "0.3 ng/mL" File: "H 69869 03212014C.wiff"
Peak Name: "Clomazone" Mass(es): "240.1/125.0 Da"
Comment: "" Annotation: ""

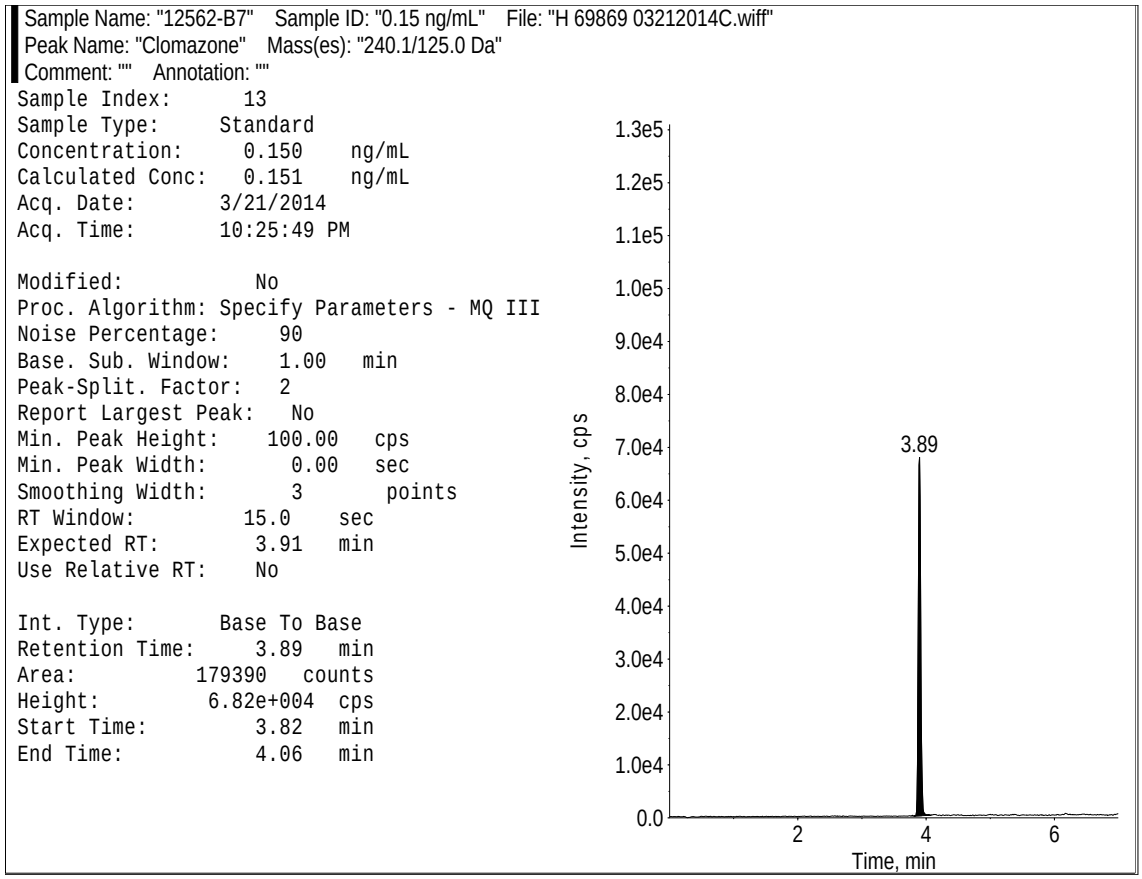
```
Sample Index:       29
Sample Type:      Standard
Concentration:      0.300    ng/mL
Calculated Conc:    0.298    ng/mL
Acq. Date:        3/22/2014
Acq. Time:        12:38:25 AM

Modified:            No
Proc. Algorithm: Specify Parameters - MQ III
Noise Percentage:      90
Base. Sub. Window:     1.00   min
Peak-Split. Factor:    2
Report Largest Peak:    No
Min. Peak Height:     100.00   cps
Min. Peak Width:        0.00   sec
Smoothing Width:        3       points
RT Window:           15.0    sec
Expected RT:          3.91   min
Use Relative RT:     No

Int. Type:         Valley
Retention Time:      3.89   min
Area:           349382   counts
Height:          1.31e+005  cps
Start Time:          3.82   min
End Time:            4.08   min
```



Clomazone Standard, 0.3 ng/mL
Peak Response (area): 349382

FIGURE 2

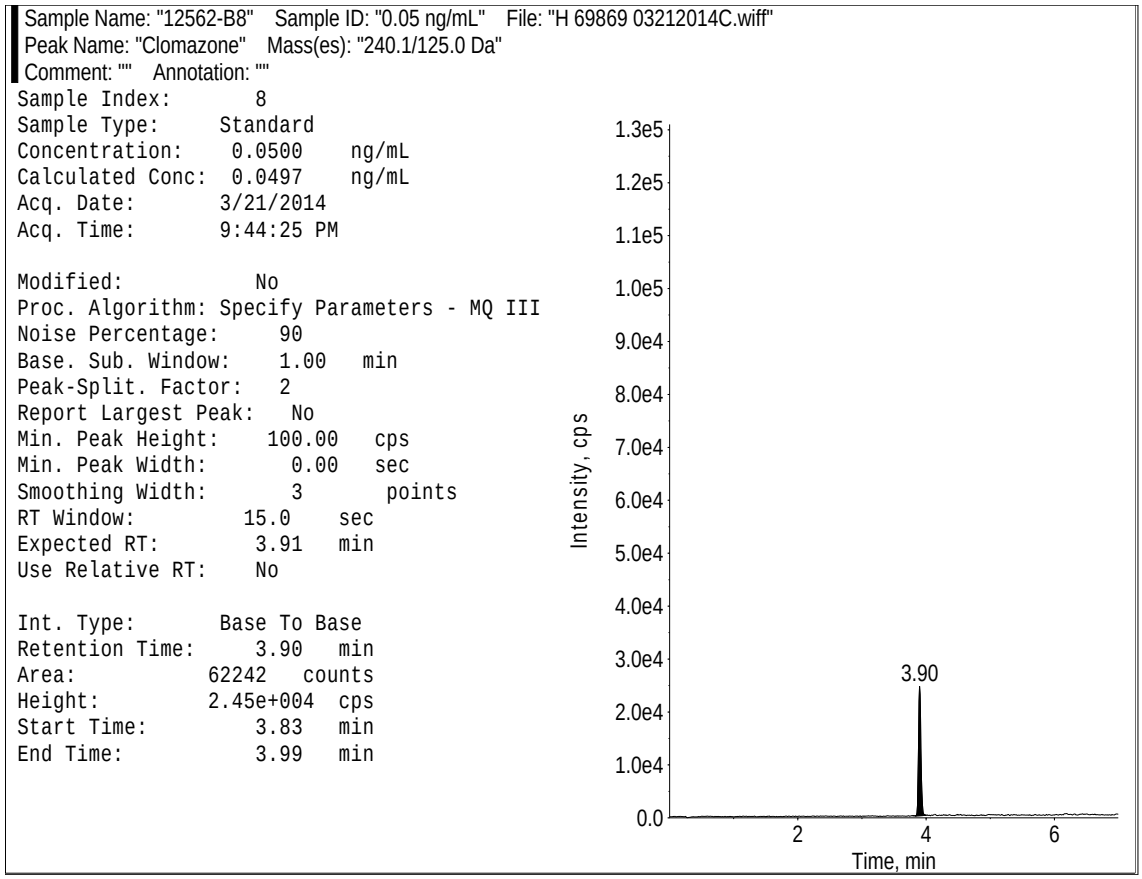
TYPICAL CLOMAZONE HPLC STANDARD
Set #7



Clomazone Standard, 0.15 ng/mL
Peak Response (area): 179390

FIGURE 3

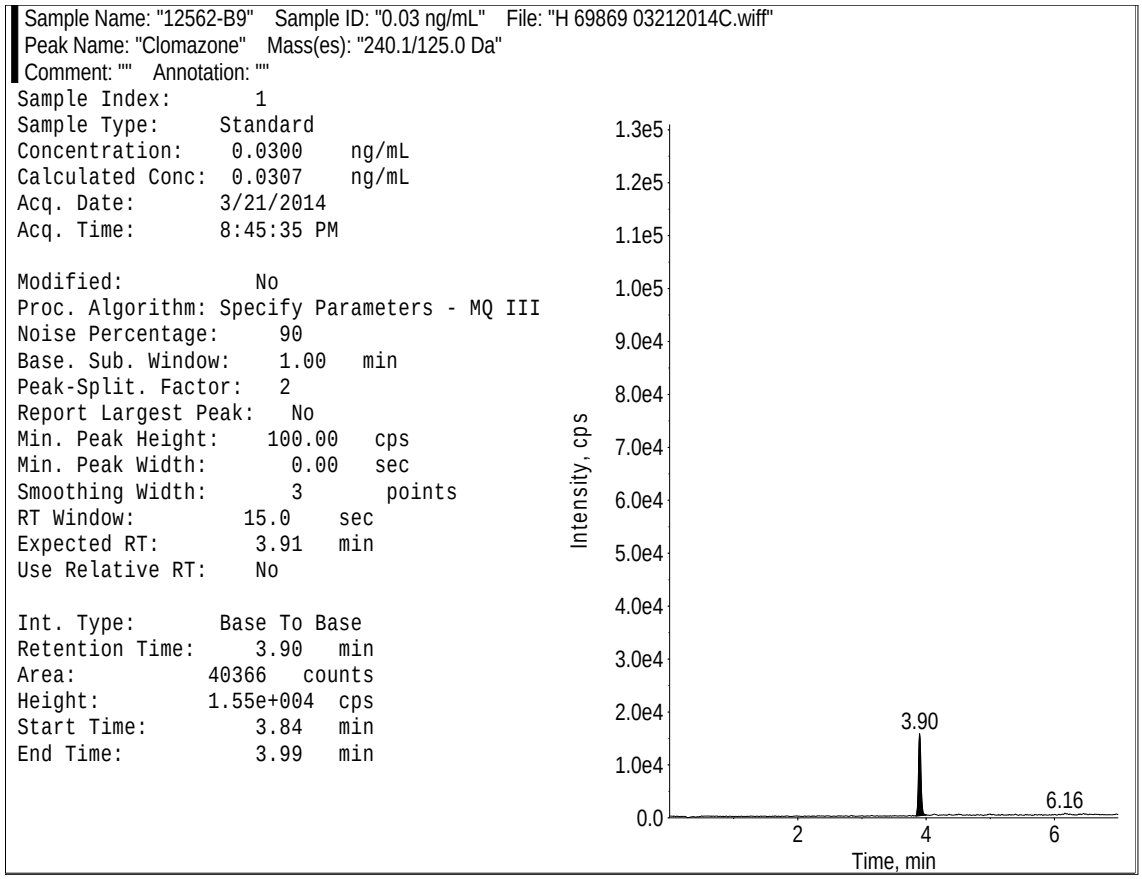
TYPICAL CLOMAZONE HPLC STANDARD
Set #7



Clomazone Standard, 0.05 ng/mL
Peak Response (area): 62242

FIGURE 4

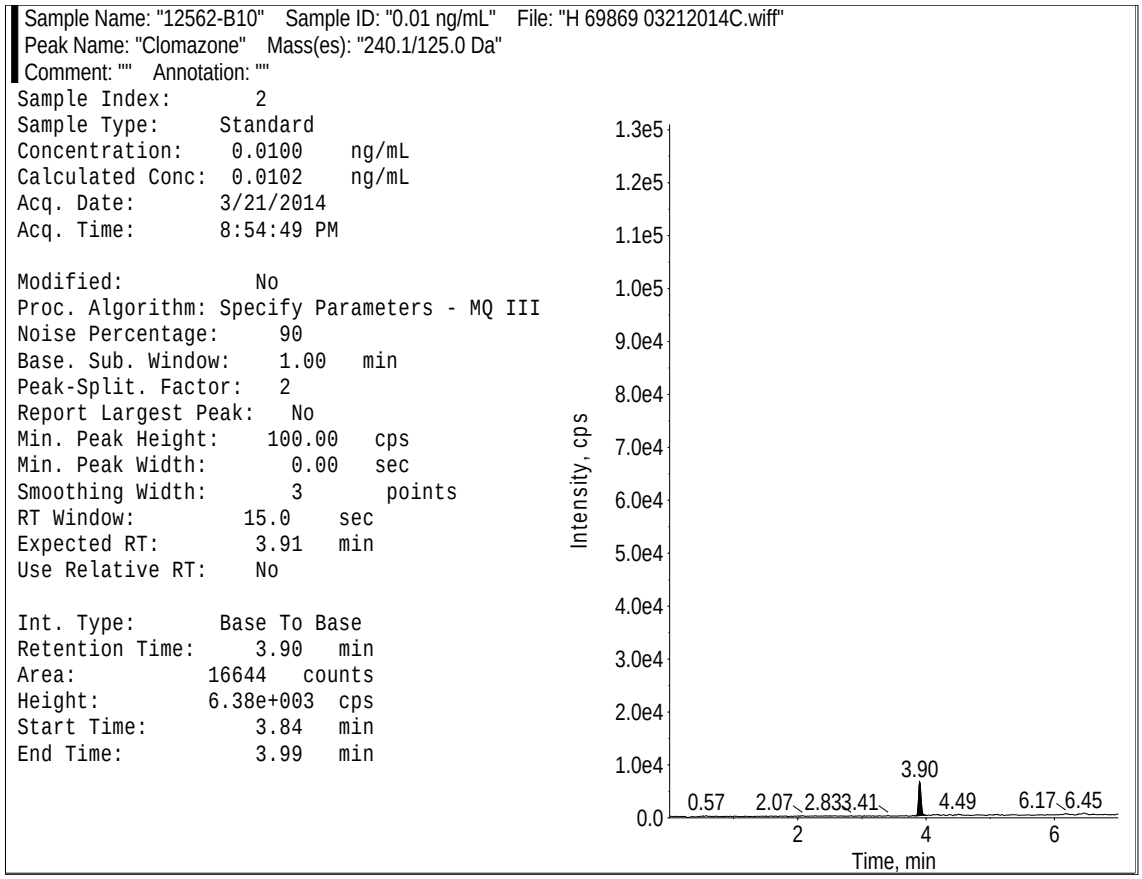
TYPICAL CLOMAZONE HPLC STANDARD
Set #7



Clomazone Standard, 0.03 ng/mL
Peak Response (area): 40366

FIGURE 5

TYPICAL CLOMAZONE HPLC STANDARD
Set #7



Clomazone Standard, 0.01 ng/mL
Peak Response (area): 16644

FIGURE 6

CLOMAZONE CALIBRATION CURVE
Set #7

Calibration Data

Conc. (ng/mL)	Peak Response
0.03	40366
0.01	16644
0.05	62242
0.15	179390
0.05	60191
0.03	39082
0.15	181232
0.3	349382

Calibration Curve

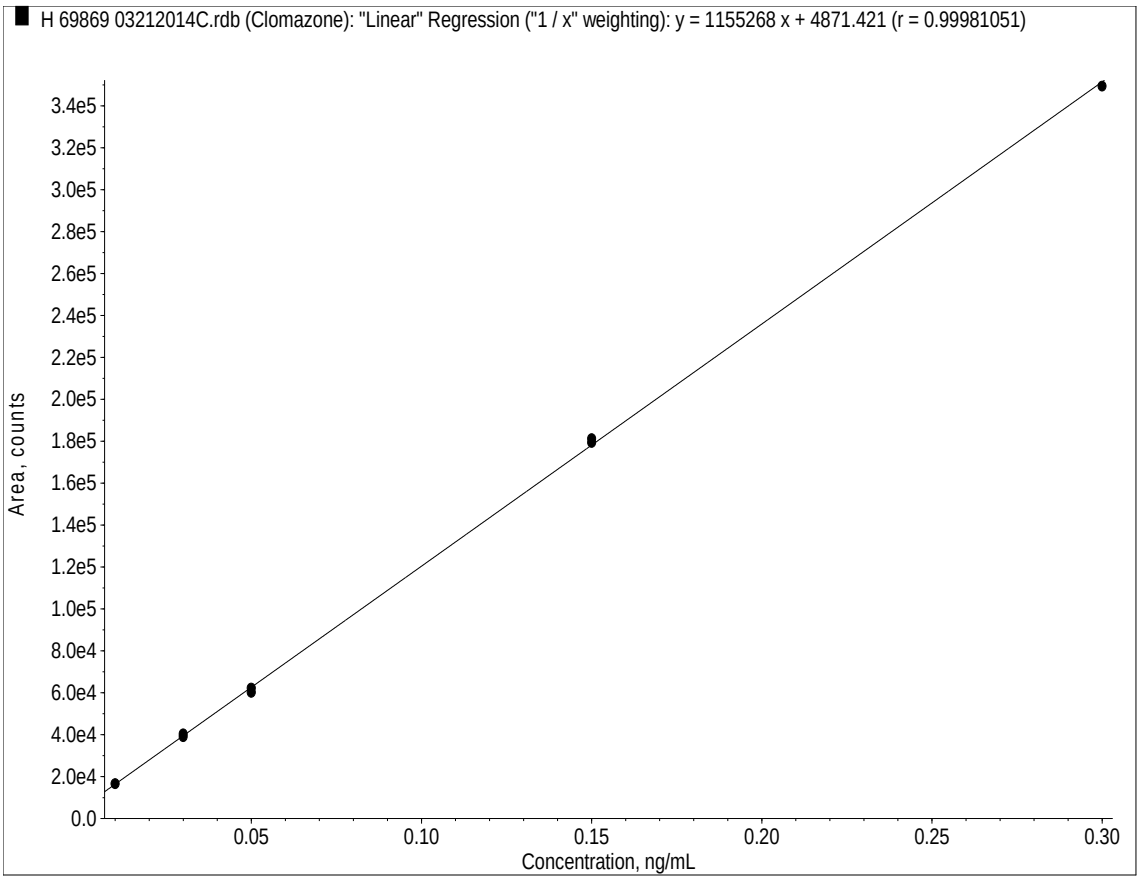
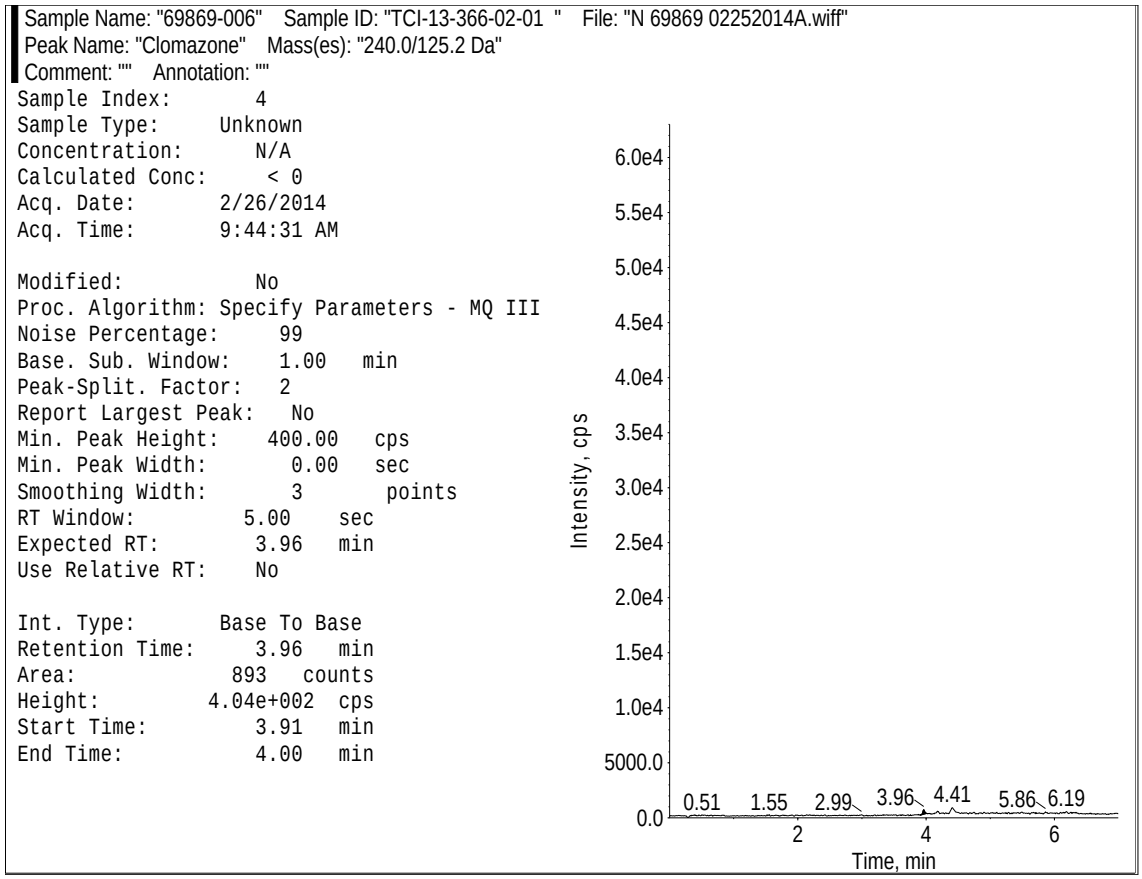


FIGURE 7

CANOLA SEED, CONTROL SAMPLE
Set #1 short



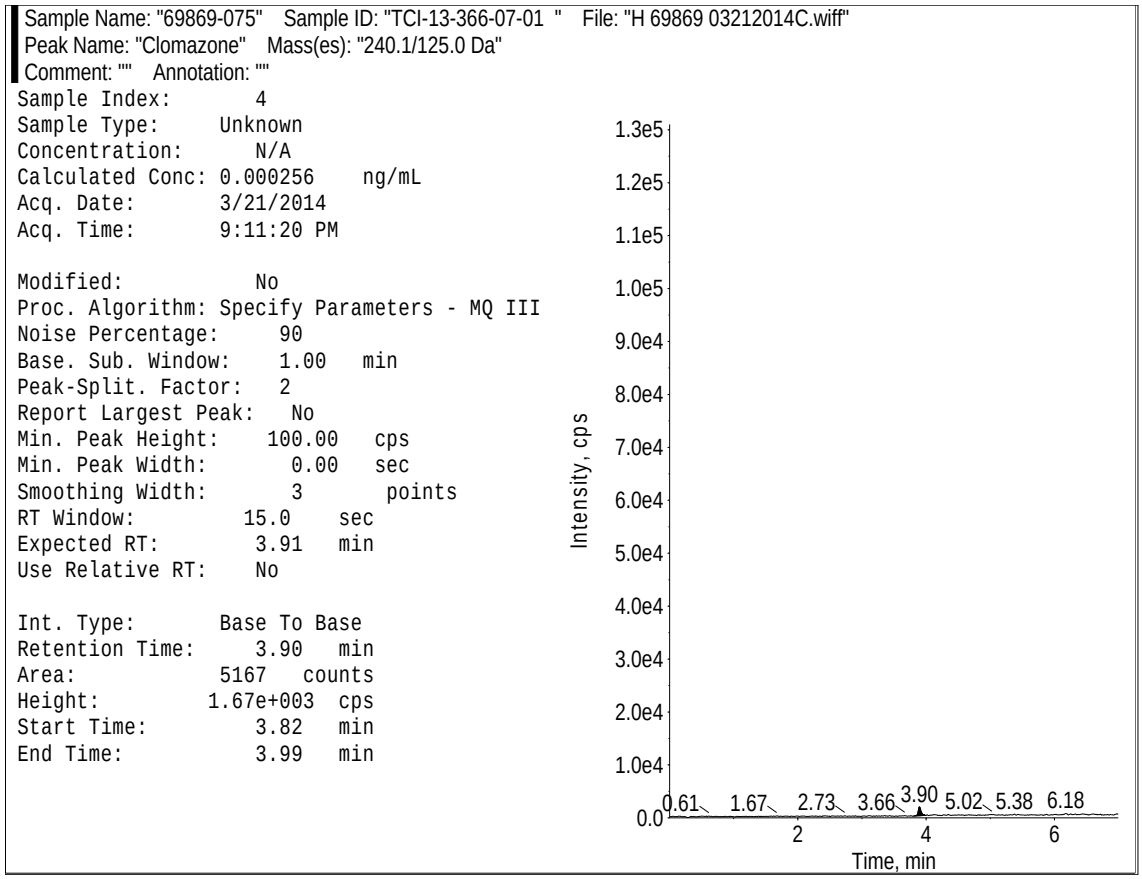
Sample TCI-13-366-02-01

Peak Response (area): 893
Retention Time: 3.96 minutes
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 8

CANOLA SEED, CONTROL SAMPLE
Set #7



Sample TCI-13-366-07-01

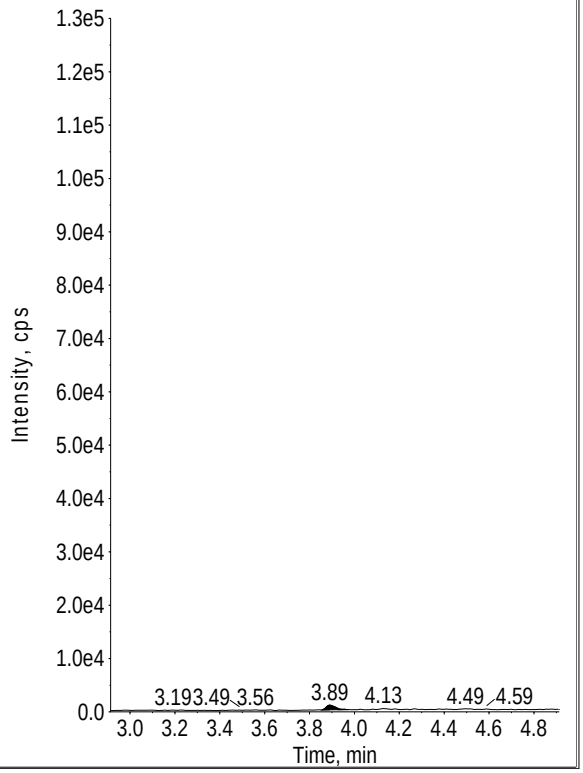
Peak Response (area): 5167
Retention Time: 3.90 minutes
Clomazone Reported: <0.0067 ppm

FIGURE 9

CANOLA SEED, CONTROL SAMPLE
Set #7

Sample Name: "69869-089" Sample ID: "TCI-13-366-08-01 " File: "H 69869 03212014C.wiff"
Peak Name: "Clomazone" Mass(es): "240.1/125.0 Da"
Comment: "" Annotation: ""

```
Sample Index:       21
Sample Type:      Unknown
Concentration:      N/A
Calculated Conc:     < 0
Acq. Date:        3/21/2014
Acq. Time:        11:32:05 PM

Modified:            Yes
Proc. Algorithm: Specify Parameters - MQ III
Noise Percentage:     50
Base. Sub. Window:    1.00   min
Peak-Split. Factor:   2
Report Largest Peak:   No
Min. Peak Height:    100.00   cps
Min. Peak Width:       0.00   sec
Smoothing Width:       3       points
RT Window:          15.0    sec
Expected RT:         3.91   min
Use Relative RT:    No

Int. Type:        Base To Base
Retention Time:      3.89   min
Area:             2508   counts
Height:          8.86e+002  cps
Start Time:          3.86   min
End Time:            3.97   min
```



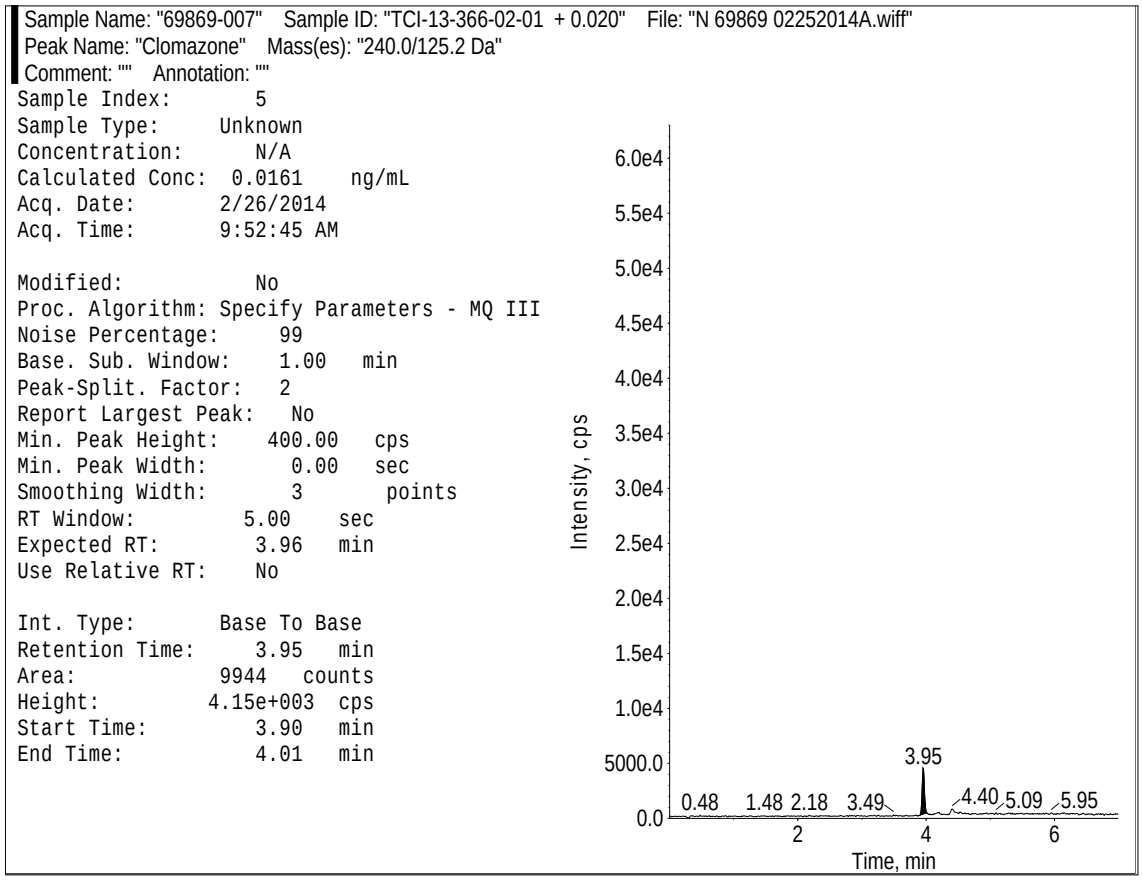
Sample TCI-13-366-08-01

Peak Response (area): 2508
Retention Time: 3.89 minutes
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 10

CANOLA SEED, FORTIFIED CONTROL SAMPLE
Set #1 short

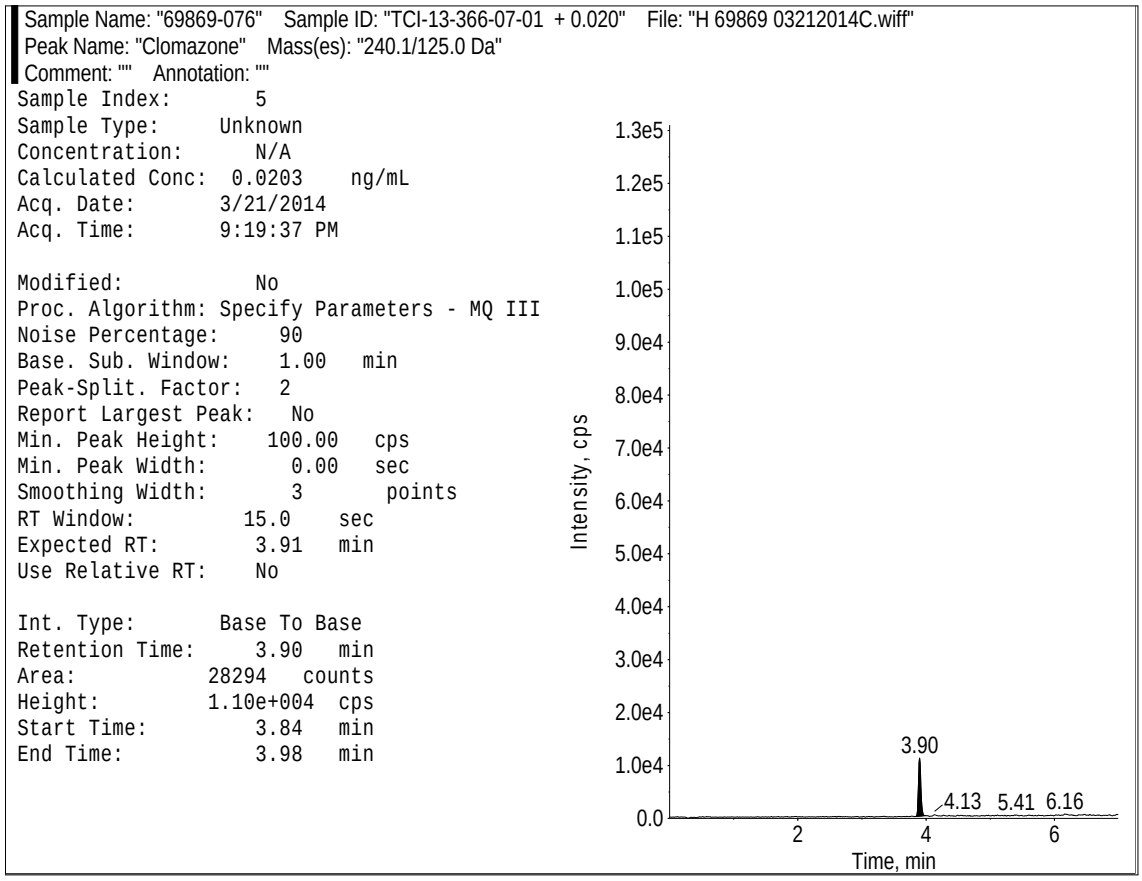


Sample TCI-13-366-02-01 + 0.020, Fortified Control @ 0.020 ppm

Peak Response (area): 9944
Corrected Clomazone Reported: 0.0161 ppm
Percent Recovery: 81%

FIGURE 11

CANOLA SEED, FORTIFIED CONTROL SAMPLE
Set #7

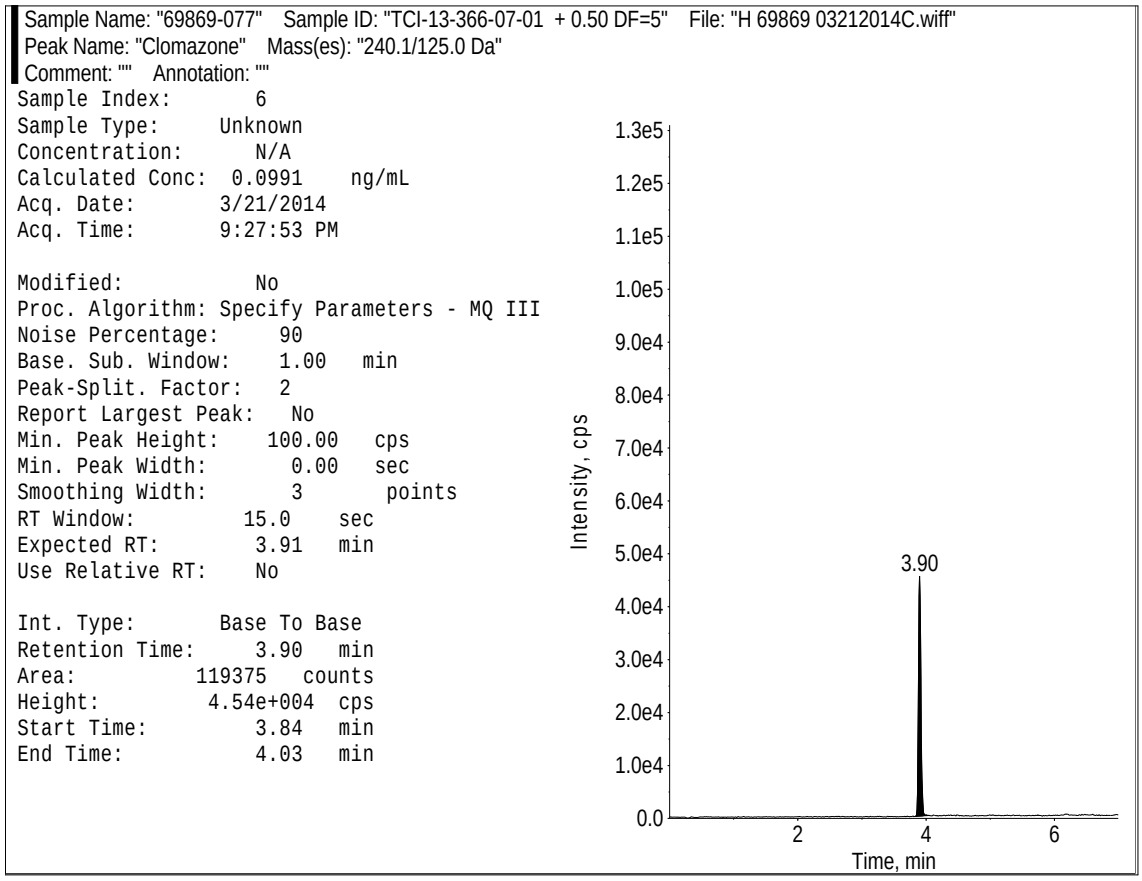


Sample TCI-13-366-07-01 + 0.020, Fortified Control @ 0.020 ppm

Peak Response (area): 28294
Corrected Clomazone Reported: 0.0200 ppm
Percent Recovery: 100%

FIGURE 12

CANOLA SEED, FORTIFIED CONTROL SAMPLE
Set #7

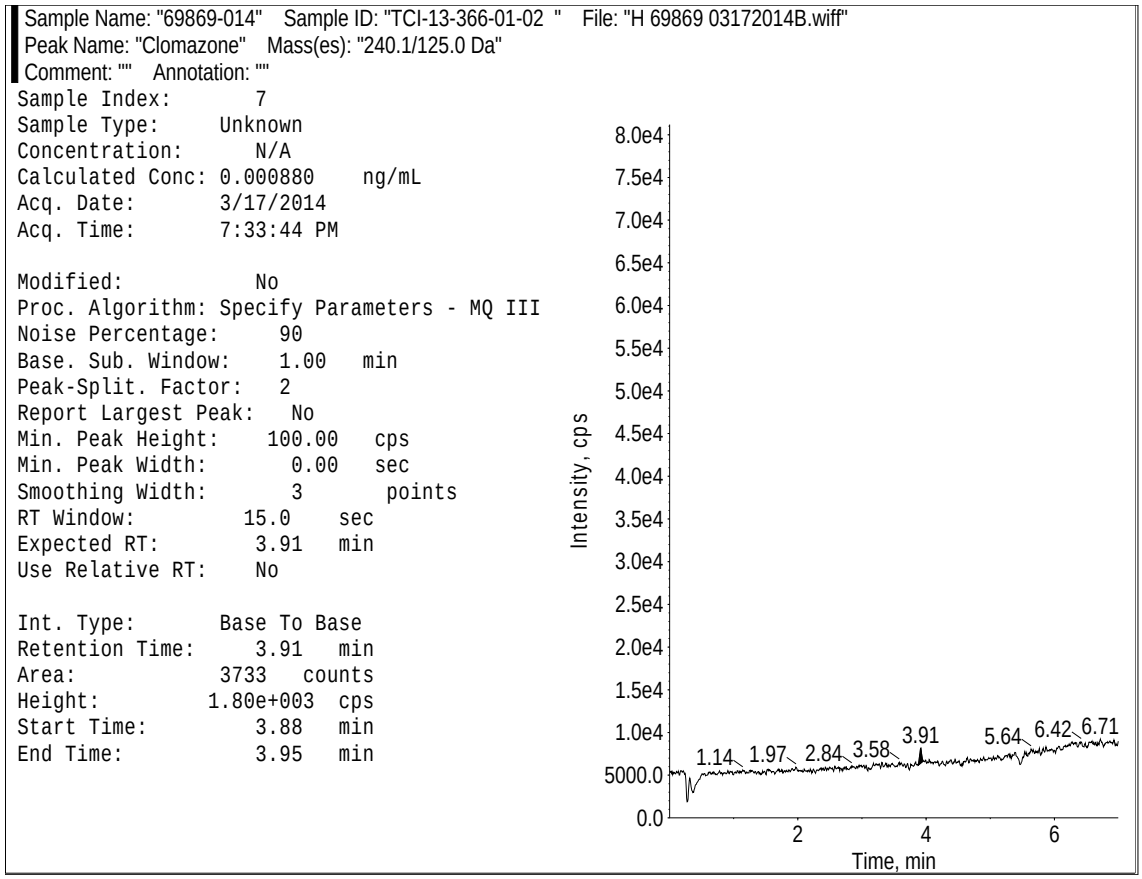


Sample TCI-13-366-07-01 + 0.50, Fortified Control @ 0.50 ppm

Peak Response (area): 119375
Corrected Clomazone Reported: 0.495 ppm
Percent Recovery: 99%

FIGURE 13

CANOLA SEED, TREATED SAMPLE
Set #2

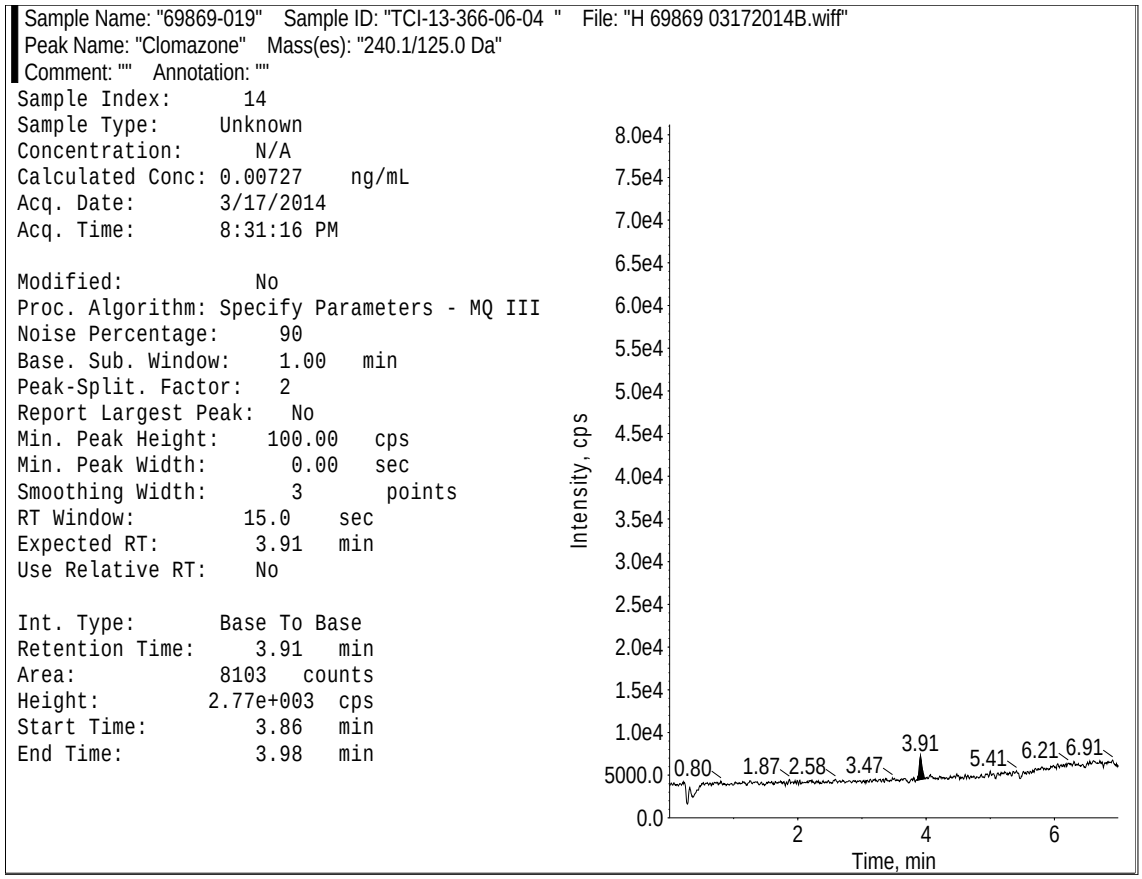


Sample TCI-13-366-01-02

Peak Response (area): 3733
Clomazone Reported: <0.0067 ppm

FIGURE 14

CANOLA SEED, TREATED SAMPLE
Set #2

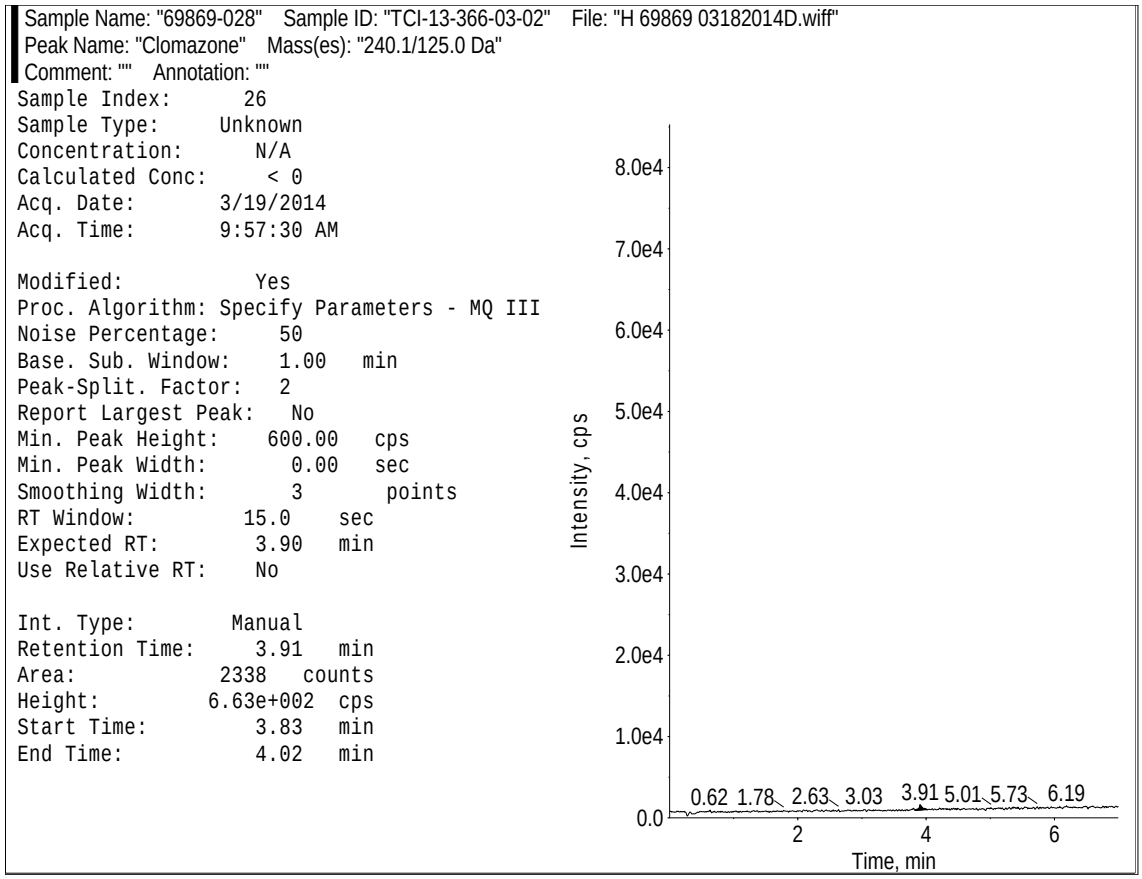


Sample TCI-13-366-06-04

Peak Response (area): 8103
Clomazone Reported: <0.02 ppm

FIGURE 15

CANOLA SEED, TREATED SAMPLE
Set #4

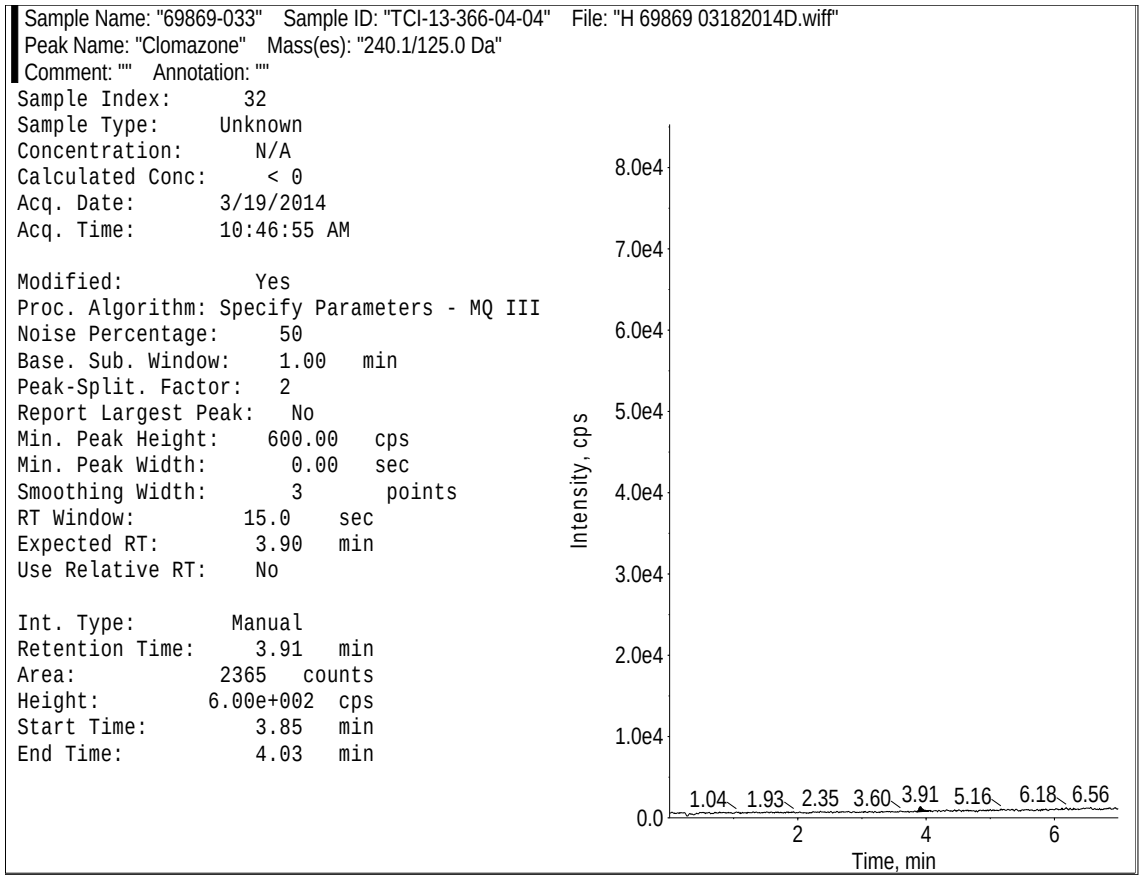


Sample TCI-13-366-03-02

Peak Response (area): 2338
Clomazone Reported: <0.0067 ppm

FIGURE 16

CANOLA SEED, TREATED SAMPLE
Set #4

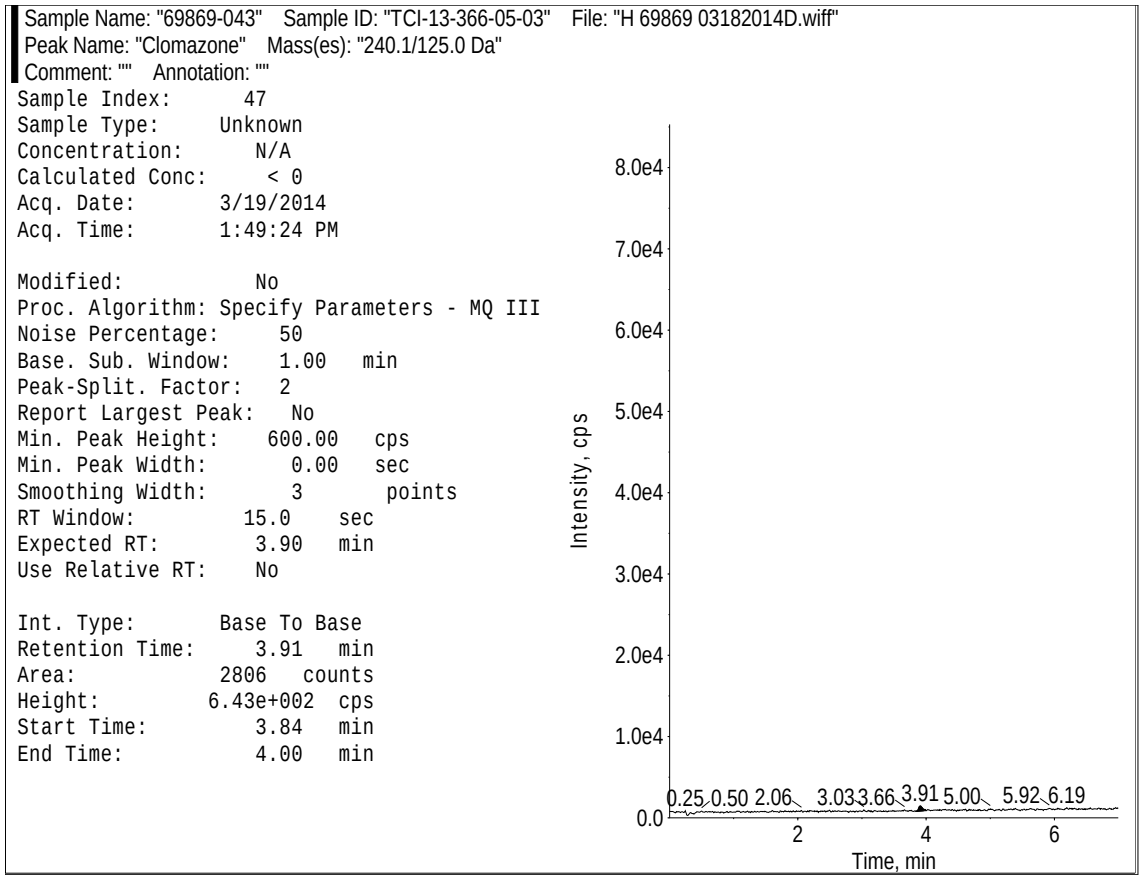


Sample TCI-13-366-04-04

Peak Response (area): 2365
Clomazone Reported: <0.0067 ppm

FIGURE 17

CANOLA SEED, TREATED SAMPLE
Set #4

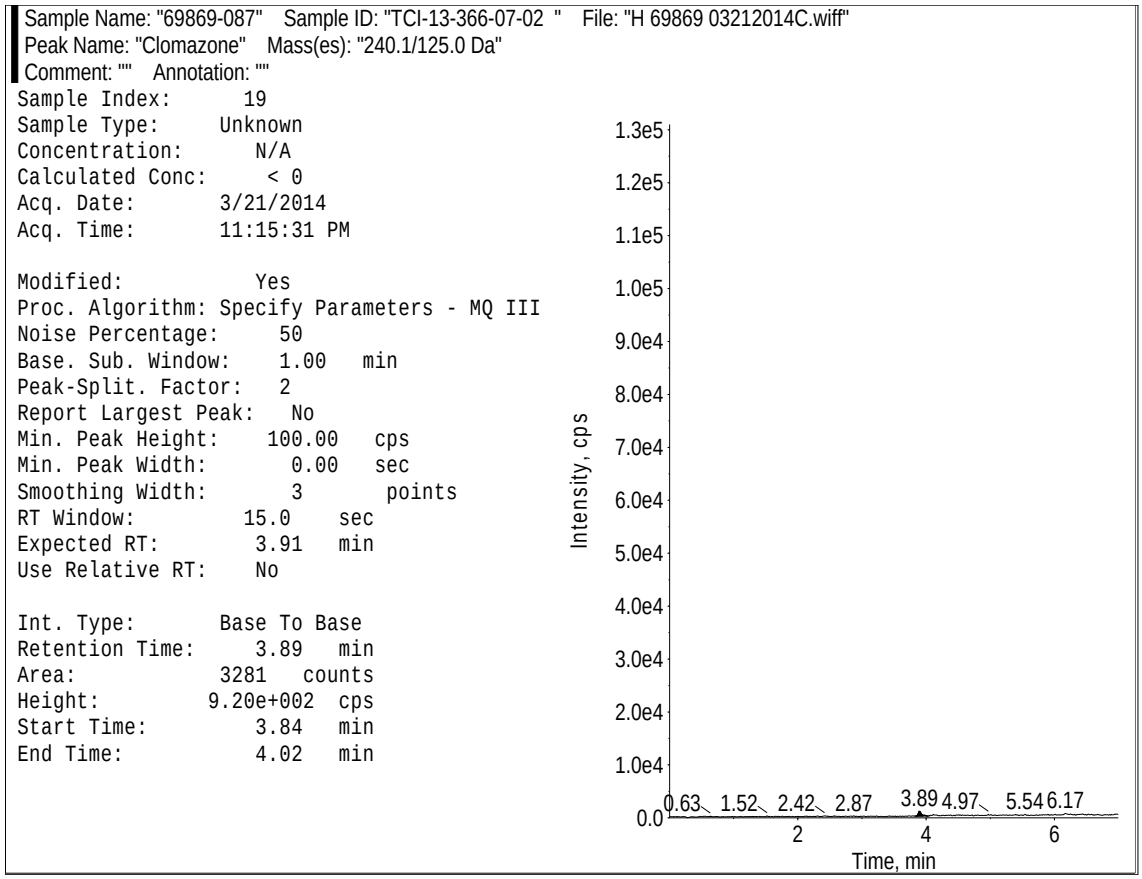


Sample TCI-13-366-05-03

Peak Response (area): 2806
Clomazone Reported: <0.0067 ppm

FIGURE 18

CANOLA SEED, TREATED SAMPLE
Set #7



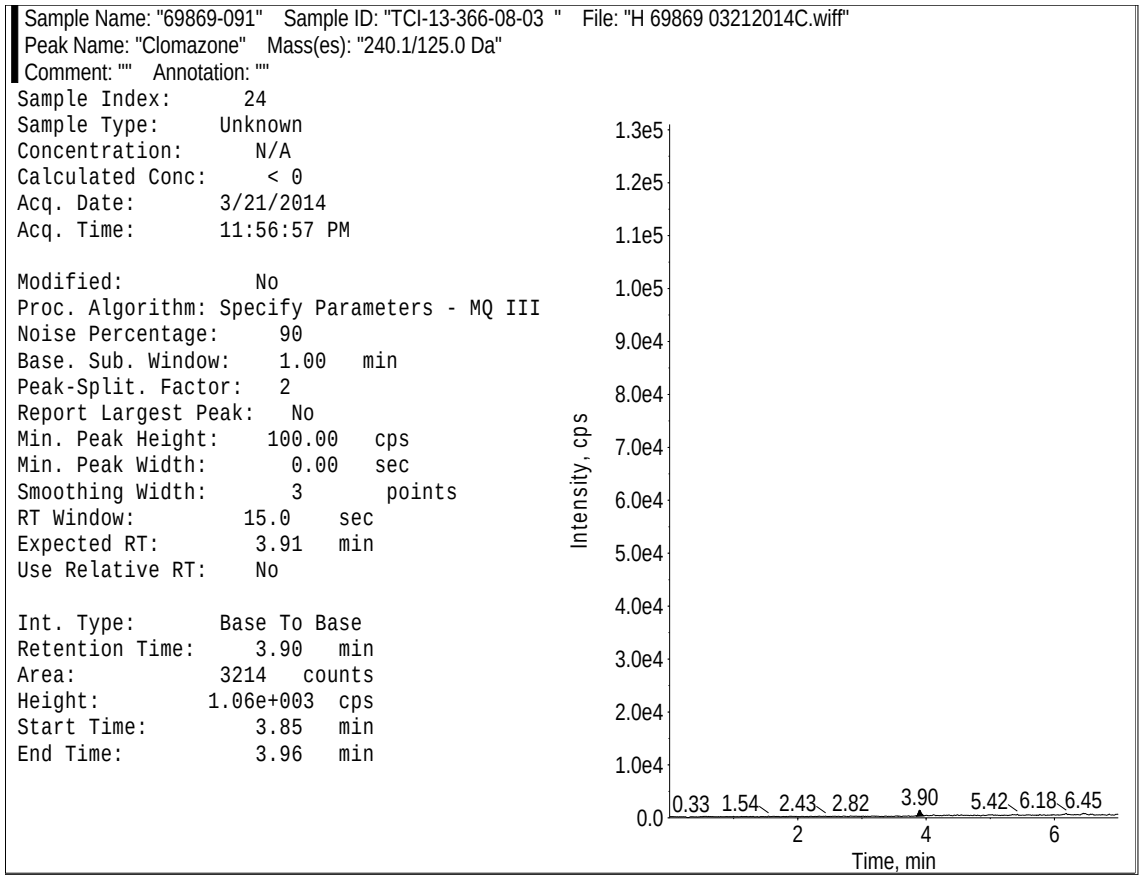
Sample TCI-13-366-07-02

Peak Response (area): 3281
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 19

CANOLA SEED, TREATED SAMPLE
Set #7



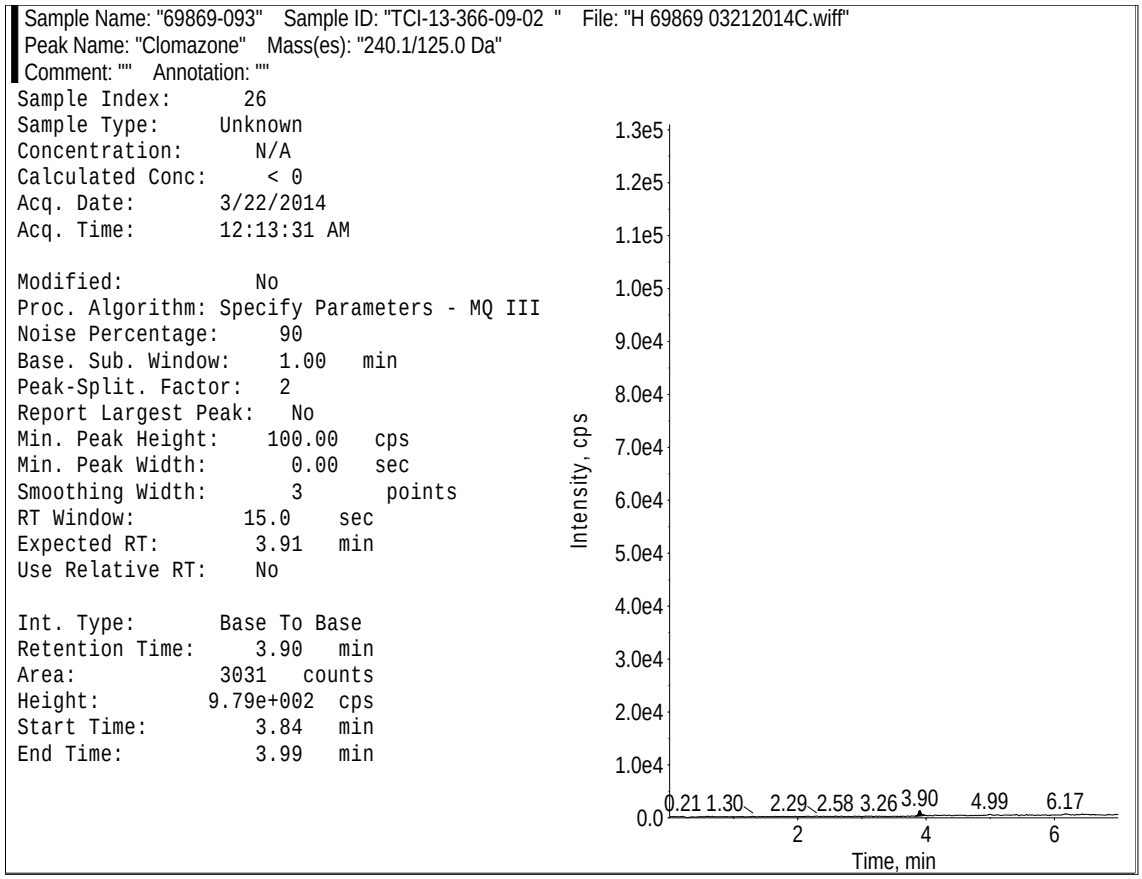
Sample TCI-13-366-08-03

Peak Response (area): 3214
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 20

CANOLA SEED, TREATED SAMPLE
Set #7



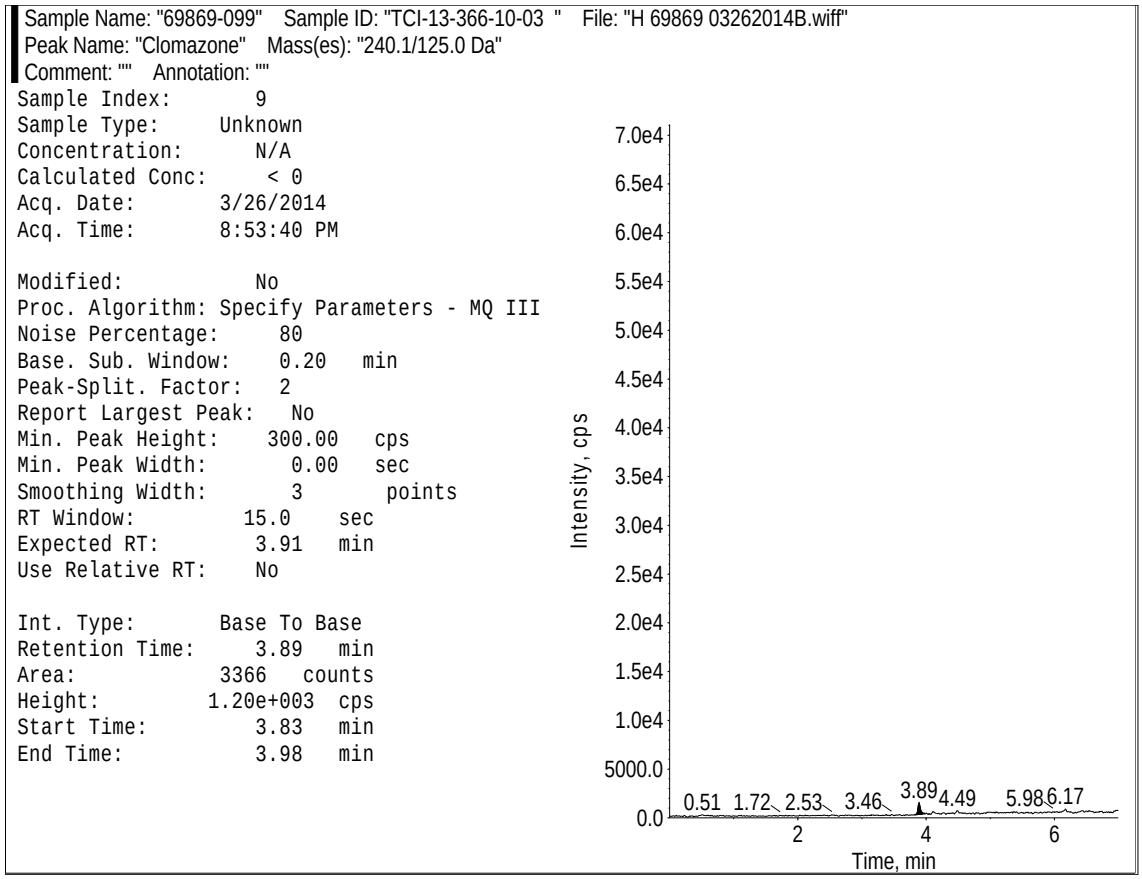
Sample TCI-13-366-09-02

Peak Response (area): 3031
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 21

CANOLA SEED, TREATED SAMPLE
Set #8



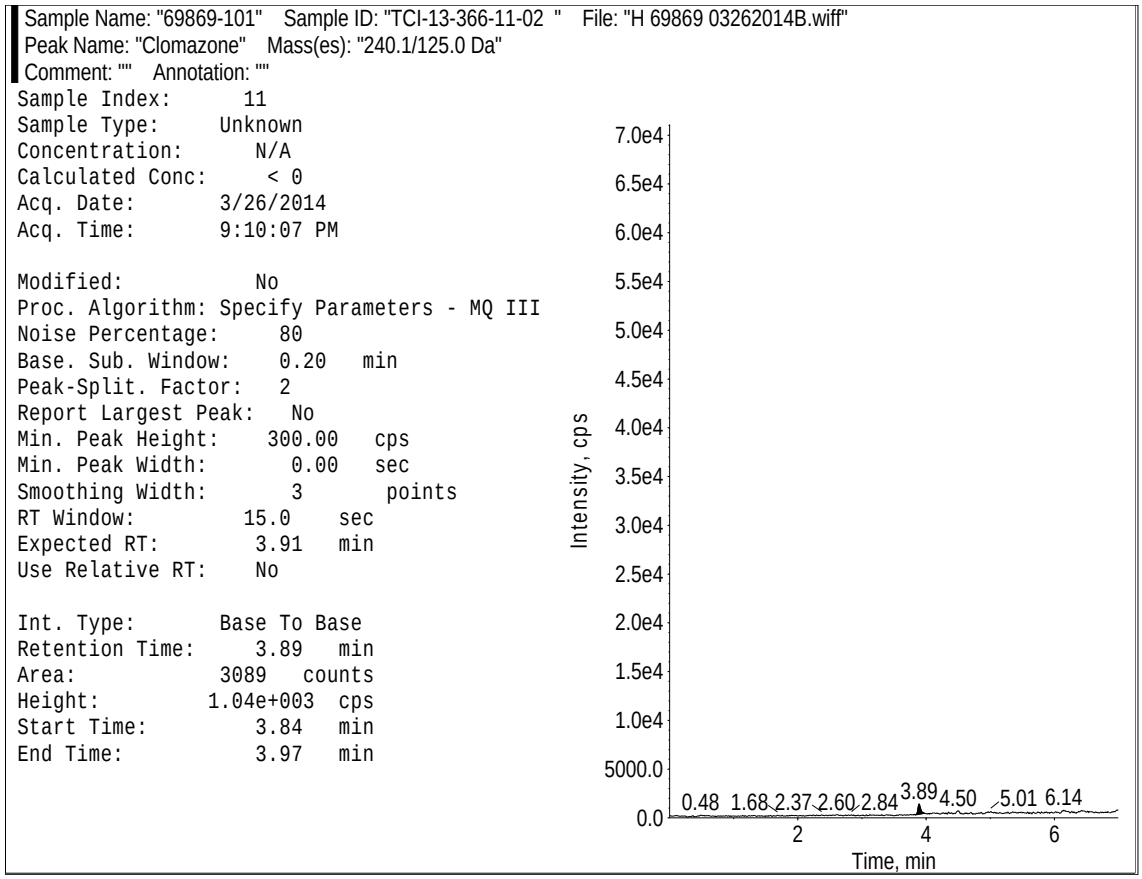
Sample TCI-13-366-10-03

Peak Response (area): 3366
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 22

CANOLA SEED, TREATED SAMPLE
Set #8



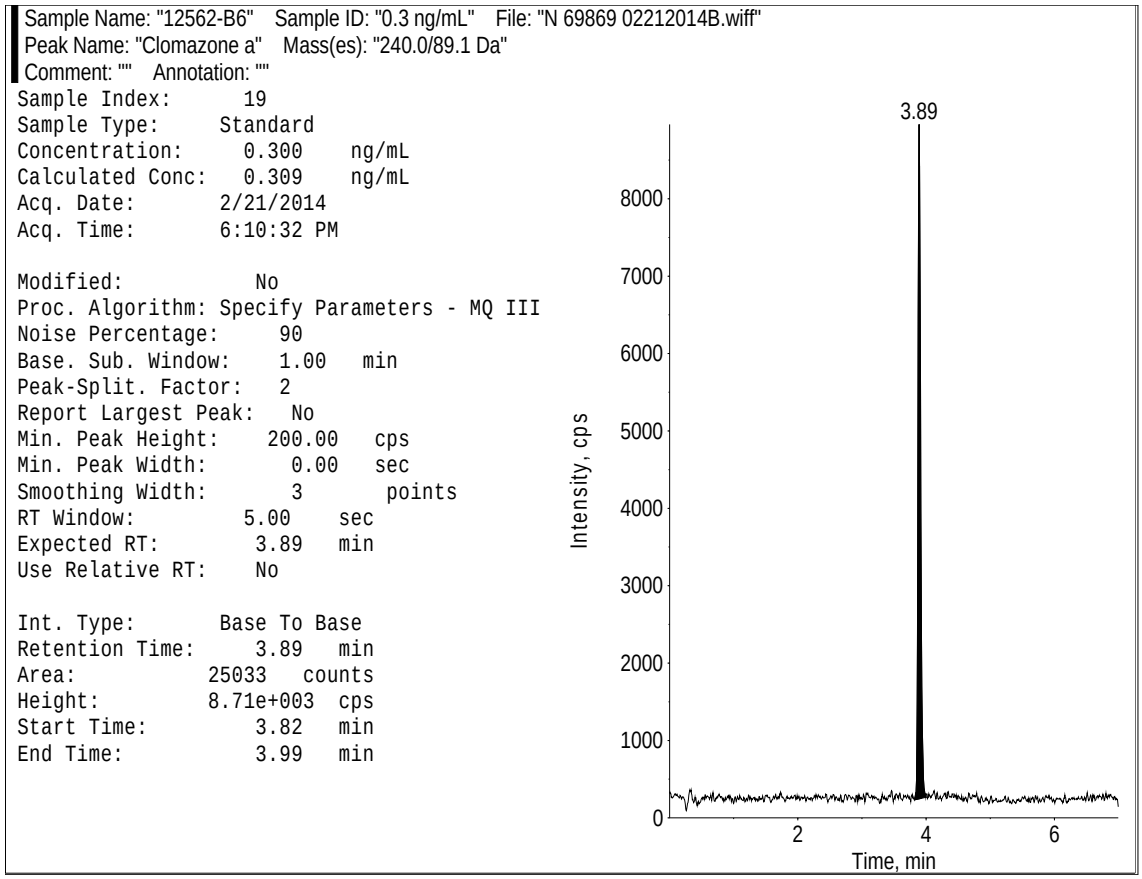
Sample TCI-13-366-11-02

Peak Response (area): 3089
Clomazone Reported: None detected[a]

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

FIGURE 23

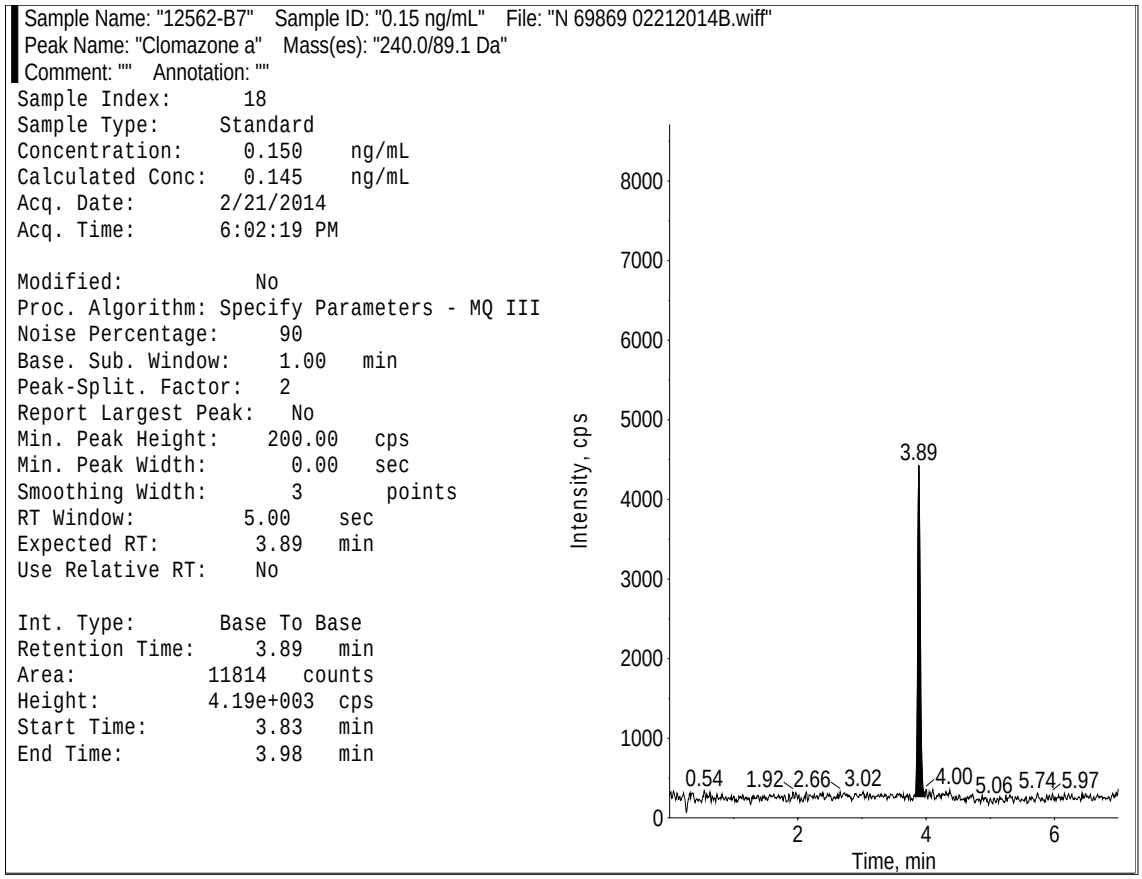
TYPICAL CLOMAZONE HPLC STANDARD
Set #MV2 (Confirmatory Ion)



Clomazone Standard, 0.3 ng/mL
Peak Response (area): 25033

FIGURE 24

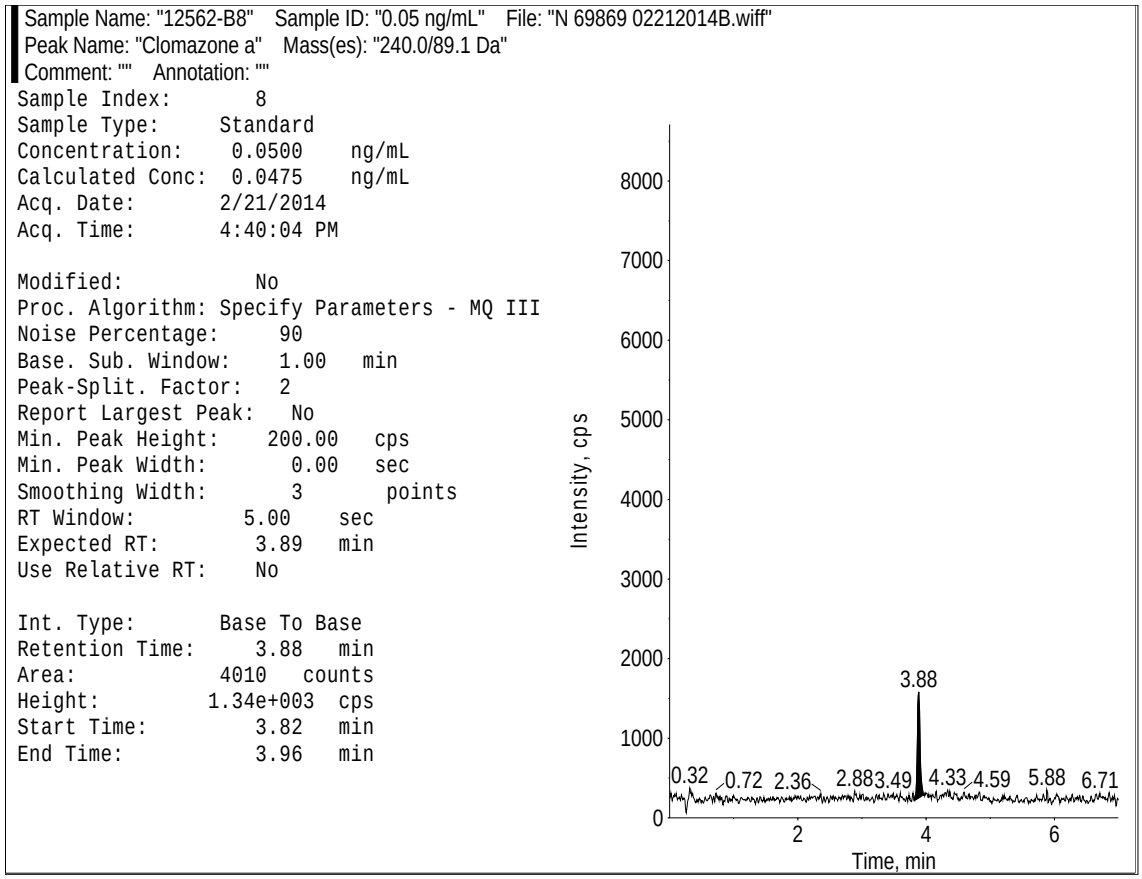
TYPICAL CLOMAZONE HPLC STANDARD
Set #MV2 (Confirmatory Ion)



Clomazone Standard, 0.15 ng/mL
Peak Response (area): 11814

FIGURE 25

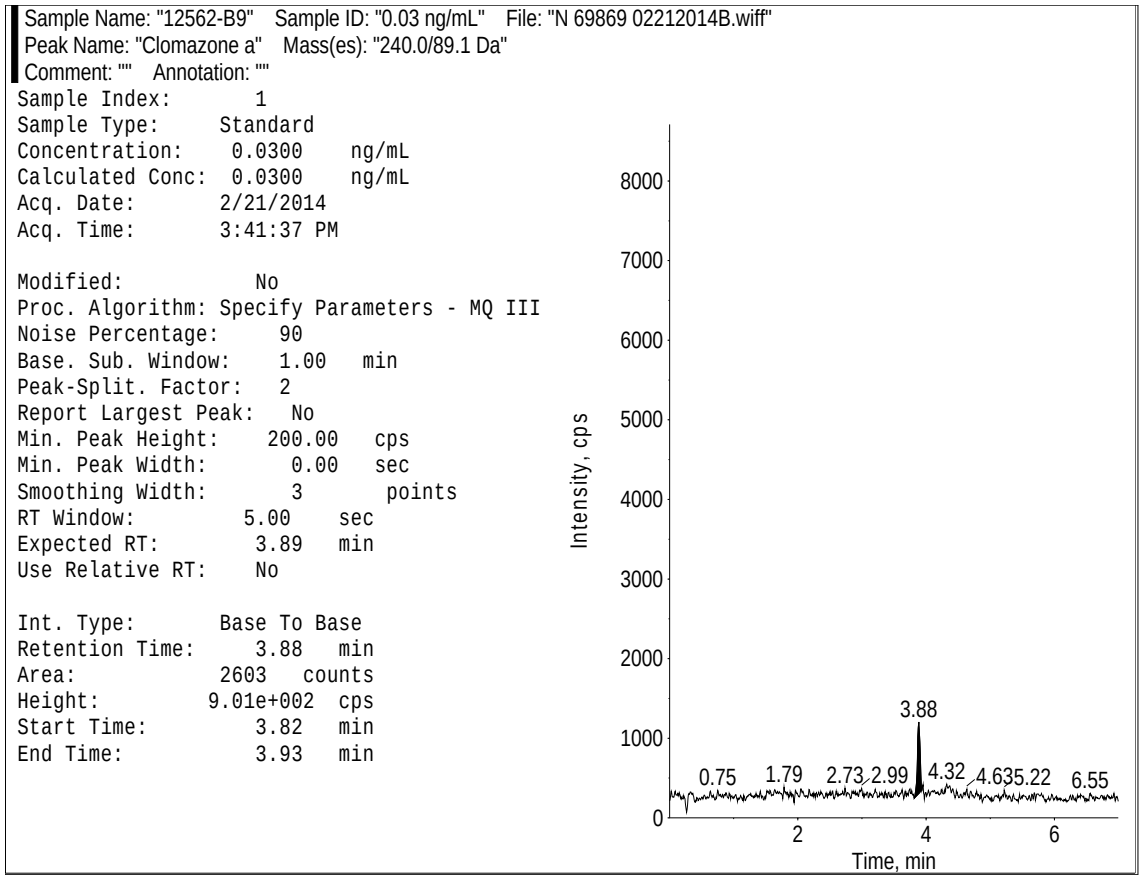
TYPICAL CLOMAZONE HPLC STANDARD
Set #MV2 (Confirmatory Ion)



Clomazone Standard, 0.05 ng/mL
Peak Response (area): 4010

FIGURE 26

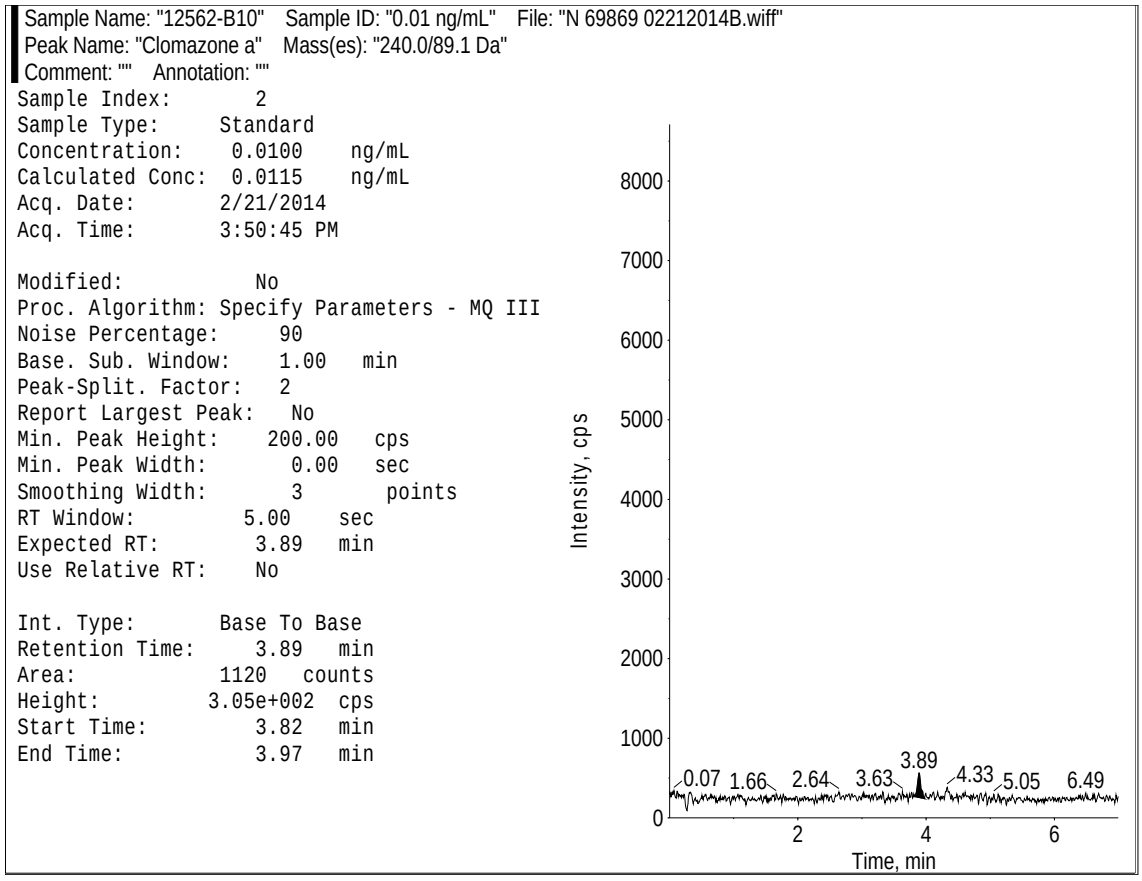
TYPICAL CLOMAZONE HPLC STANDARD
Set #MV2 (Confirmatory Ion)



Clomazone Standard, 0.03 ng/mL
Peak Response (area): 2603

FIGURE 27

TYPICAL CLOMAZONE HPLC STANDARD
Set #MV2 (Confirmatory Ion)



Clomazone Standard, 0.01 ng/mL
Peak Response (area): 1120

FIGURE 28

CLOMAZONE CALIBRATION CURVE
Set #MV2 (Confirmatory Ion)

Calibration Data

Conc. (ng/mL)	Peak Response
0.01	1120
0.03	2603
0.03	2374
0.05	4010
0.15	11814
0.3	25033

Calibration Curve

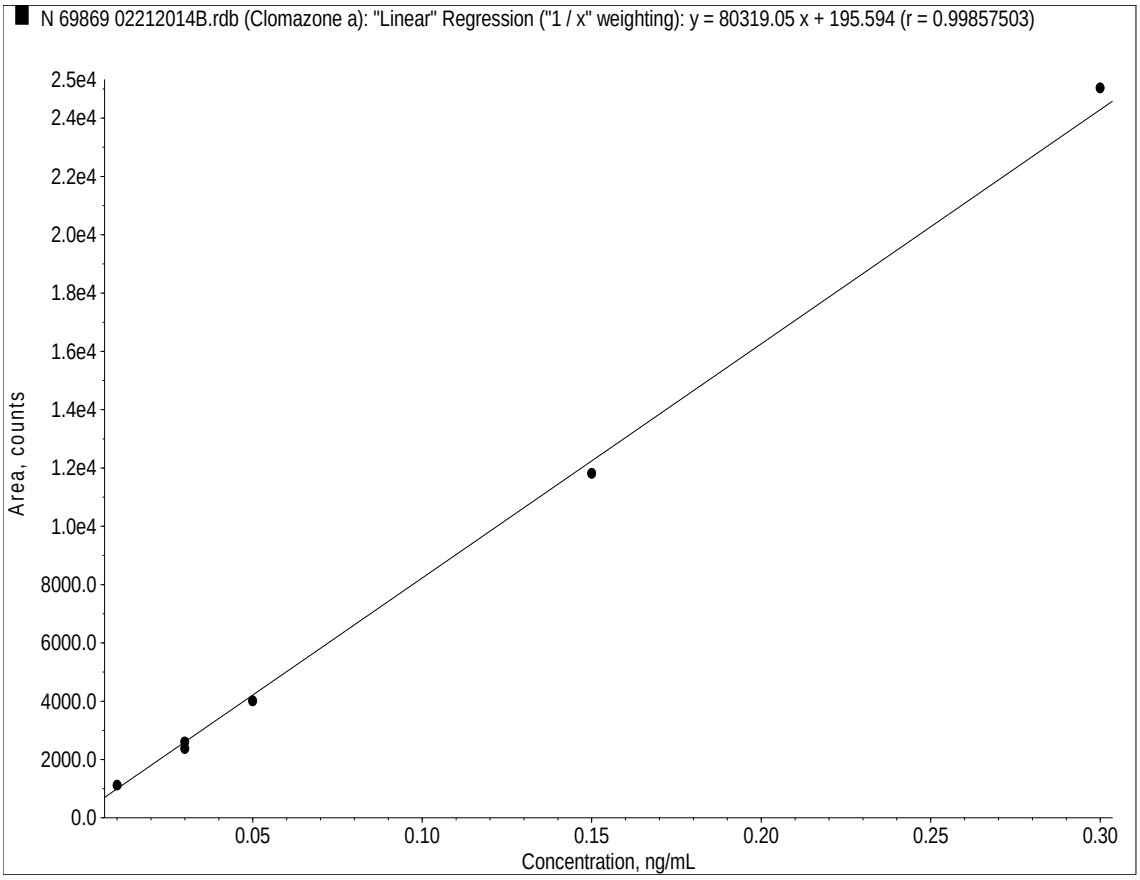
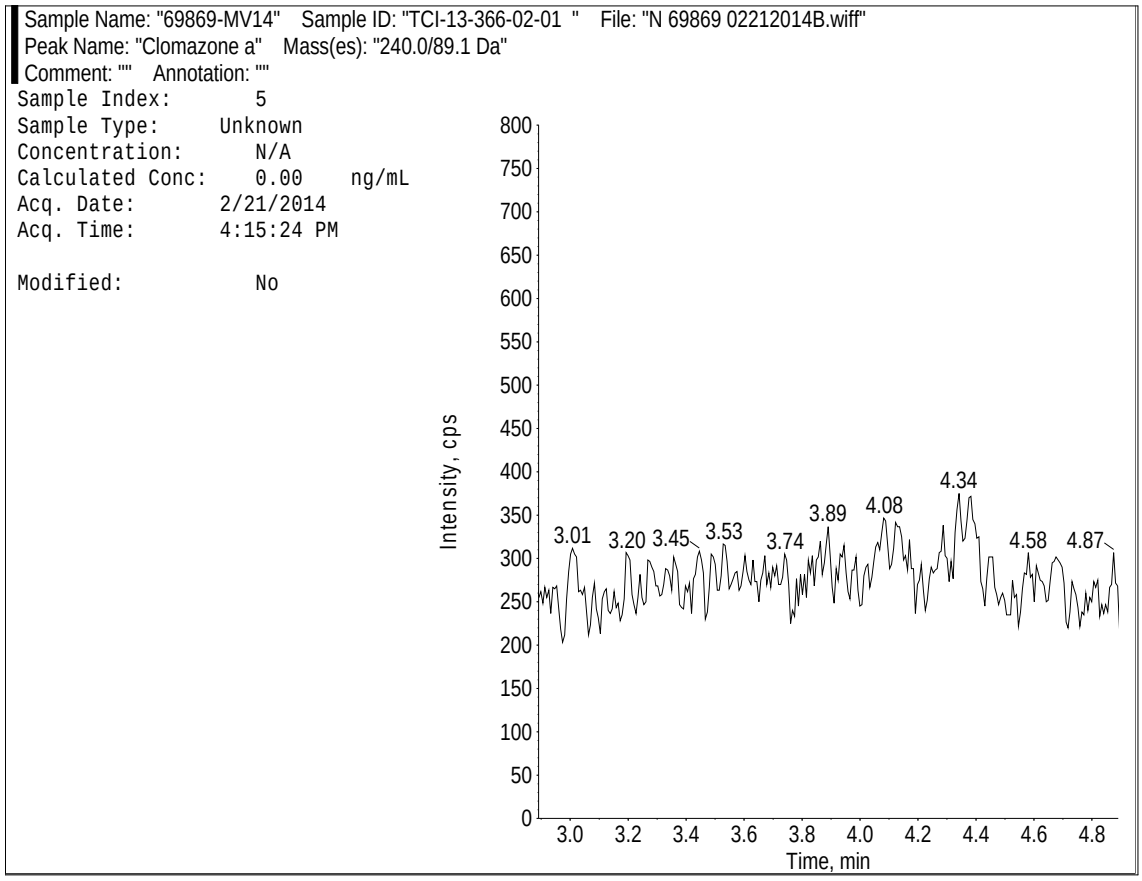


FIGURE 29

CANOLA SEED, CONTROL SAMPLE
Set #MV2 (Confirmatory Ion)

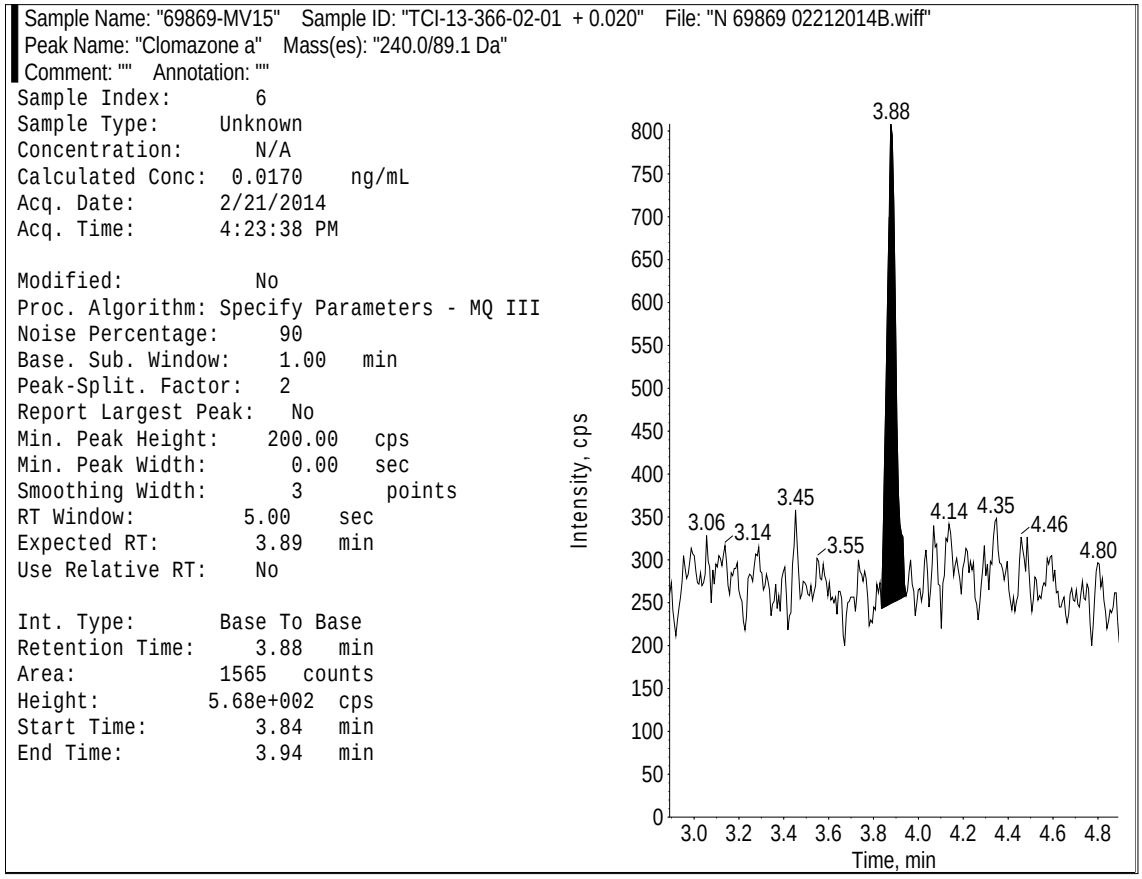


Sample TCI-13-366-02-01

Peak Response (area): 0
Expected Retention Time: 3.90 minutes
Clomazone Reported: None detected

FIGURE 30

CANOLA SEED, FORTIFIED CONTROL SAMPLE
Set #MV2 (Confirmatory Ion)

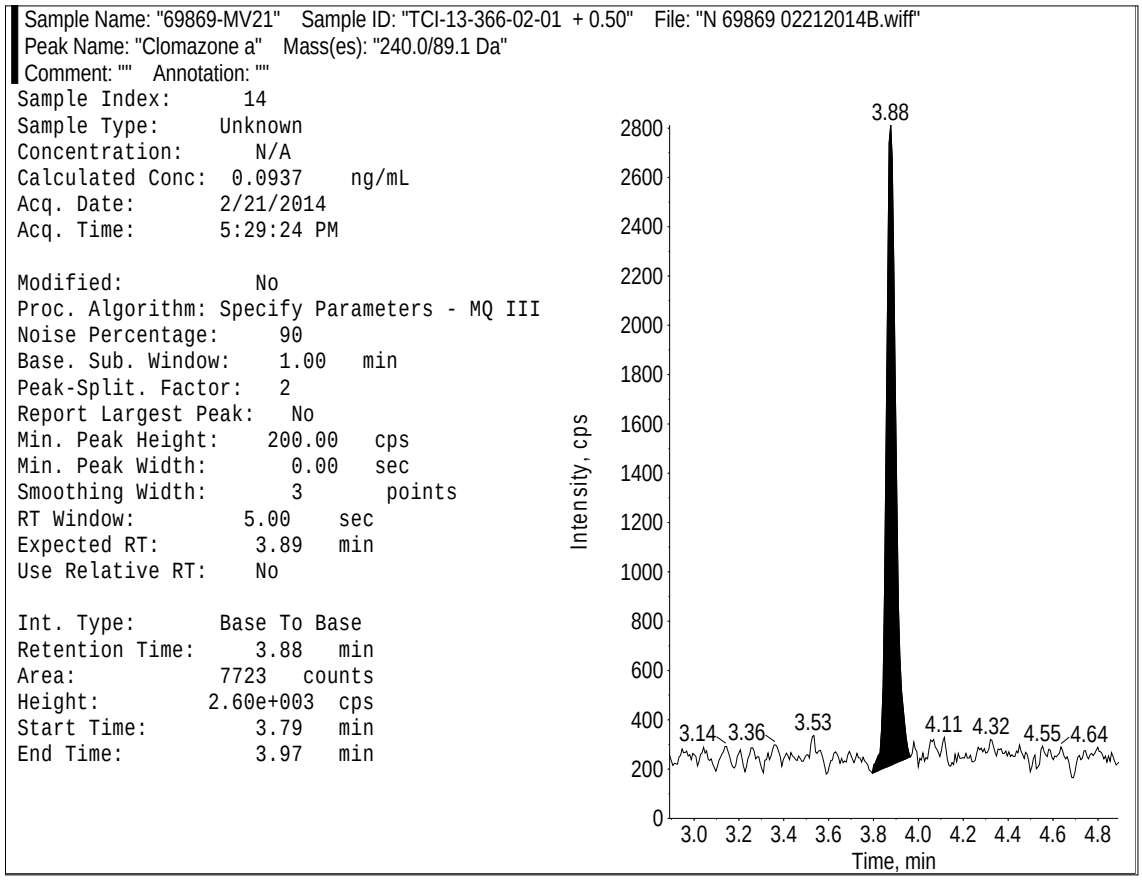


Sample TCI-13-366-02-01, Fortified Control @ 0.02 ppm

Peak Response (area): 1565
Corrected Clomazone Reported: 0.0170 ppm
Percent Recovery: 85%

FIGURE 31

CANOLA SEED, FORTIFIED CONTROL SAMPLE
Set #MV2 (Confirmatory Ion)



Sample TCI-13-366-02-01, Fortified Control @ 0.50 ppm

Peak Response (area): 7723
Corrected Clomazone Reported: 0.469 ppm
Percent Recovery: 94%

APPENDIX I

Analytical Method and Modification

Eurofins-GAB GmbH Report, Study Code 20061401/01-RVP, entitled, "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape" dated January 17, 2007

Method Modification to 20061401/01-RVP, dated April 23, 2014

Doc. No. 22 CAZ

Final Report

Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape

Data Requirement

EC Guidance document on residue analytical methods,
SANCO/825/00 rev. 7 (17/03/2004)

Study Director

Ellen Fiedler

Date

17 January 2007

Testing Facility	**Sponsor**
eurofins-GAB GmbH	Cheminova A/S
Eutinger Str. 24	P.O. Box 9
D-75223 Niefern-Öschelbronn	DK-7620 Lemvig
Germany	Denmark

Study Identification Codes:

Test item: Clomazone

Study code of testing facility: 20061401/01-RVP

Statement of Confidentiality

This report contains confidential and proprietary information of Cheminova A/S that must not be disclosed to anyone except the employees of this company or to persons authorized by law or judicial judgement without the expressed and written approval of the sponsor.

Statement of Compliance with the Principles of Good Laboratory Practice

The study described in this analytical report was conducted in compliance with the most recent edition of:

- The Principles of Good Laboratory Practice (GLP), (Chemical Act, attachment 1, Federal Republic of Germany)
- The OECD Principles of Good Laboratory Practice

The German requirements are based on the OECD Principles of Good Laboratory Practice that are accepted by regulatory authorities throughout the European Community, the United States of America (FDA and EPA) and Japan (MHW, MAFF and MITI) on the basis of intergovernmental agreements.

Head of testing facility:
(Dr. Susanne Timmermann / Dr. Hans Eberhardt) 18.1.07
Date / Signature

Study director:
(Ellen Fiedler) 17.01.07 Ellen Fiedler
Date / Signature

Statement of Quality Assurance Unit

Study code: 20061401/01-RVP

Study title: Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape Seed

Study plan, experimental phase, draft report and final report of this study were audited by the Quality Assurance Unit. The dates are given below:

	Date of audit	Date of report
Study Plan:	21 December 2006	21 December 2006
Experimental Phase:	21 December 2006	21 December 2006
Draft Report:	09 January 2007	09 January 2007
Final Report:	17 January 2007	17 January 2007

Quality assurance:
(Dietmar Feiter)

11.1.2007
Date / Signature

Contents

List of Tables

List of Figures

1 Summary

A method for determination of clomazone in potatoes and oilseed rape seed was successfully validated according to guideline SANCO/825/00 rev. 7.

The principle sample preparation scheme is summarised below:

	Potato	Rape seed
Extraction:	Cold extraction with acetone/water (2:1)	Cold extraction with acetonitrile/acetone in presence of Calflo E and Celite
Clean-up:	Liquid-liquid partitioning into cyclohexane/ethyl acetate (1:1), GPC, chromatography on silica gel	GPC, chromatography on silica gel
Final analysis:	HPLC-MS/MS, APCI positive ion mode	

Limit of quantification: 0.005 mg/kg

Repeatability: 5 % to 9 % relative standard deviation for potato matrix and 4 % to 8 % relative standard deviation for rape seed matrix.

Blanks: In untreated control samples of potatoes and rape seed, clomazone was not detectable (< 30 % of LOQ).

Specificity: The method is highly specific for the clomazone parent compound (mass transition from the positive charged molecule ion at m/z = 240.0 to the fragment ions m/z = 124.9 (quantification) and m/z = 89.1 (qualification) in MS/MS mode).

Linearity: Linear detector response from 1 ng/mL to 150 ng/mL.

Table 1: Summary of recoveries

Mass transition 240.0 → 124.9 (Quantification)				
Matrix	Fortification Level (mg/kg)	n	Mean Recovery ± RSD (%)	Overall Mean Recovery ± RSD (%)
Potato	0.005	5	82 ± 5	84 ± 7
	0.05	5	86 ± 9	
Rape seed	0.005	5	91 ± 4	92 ± 6
	0.05	5	92 ± 8	

Mass transition 240.0 → 89.1 (Qualification)				
Matrix	Fortification Level (mg/kg)	n	Mean Recovery ± RSD (%)	Overall Mean Recovery ± RSD (%)
Potato	0.005	5	86 ± 5	86 ± 7
	0.05	5	86 ± 9	
Rape seed	0.005	5	89 ± 3	89 ± 7
	0.05	5	88 ± 9	

2 Time Schedule

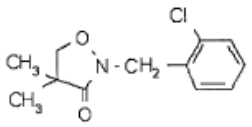
Study initiation date:	20 December 2006
Start of the experimental phase:	21 December 2006
End of the experimental phase:	02 January 2007
Draft Report:	03 January 2007
Study completion date:	17 January 2007

3 Study Objective

The aim of the current study was the validation of an analytical method for the determination of clomazone in potato and rape seed to meet the requirements of guideline SANCO/825/00 rev. 7 (17/03/2004).

4 Materials and Methods

4.1 Test Item

General name:	Clomazone
Structure:	
CAS No.:	81777-89-1
Molecular weight:	239.7
Formula:	$C_{12}H_{14}ClNO_2$
Supplier:	Cheminova A/S
GAB code:	20061401
Lot number:	HKA p. 4174
Purity:	99.7 %
Certificate of analysis:	05 September 2006
Expiry:	20 August 2008
Storage:	Deep frozen (below –18 °C)

A stock solution of the test item was prepared in acetonitrile (1000 µg/mL). From this stock solution a 10 µg/mL dilution and a 1 µg/mL dilution in acetonitrile were prepared and used for fortification. Another 1 µg/mL dilution was prepared in acetonitrile/water (1:1) which was used for for the preparation of calibration standards.

All standard solutions were stored refrigerated (4 °C nominally) in the dark.

4.2 Sample Material

Potato and rape seed matrix not treated with clomazone were taken from control samples of GAB-studies 20054075/F1-FPPO and 20054075/F1-FPRA.

Samples were homogenized with addition of dry ice in a large-scale laboratory mixer. Aliquots of the samples (500 g – 1000 g) were taken as laboratory sub samples.

All samples were stored deep-frozen (nominally at ≤ -18 °C) until analysis.

4.3 Procedure for Determination of Clomazone

4.3.1 Equipment

Analytical balances
Automated gel permeation chromatography system (Abimed Cleanup XL)
Eppendorf pipettes
Hamilton pipettes
HPLC with MS/MS detector (ThermoFinnigan TSQ Quantum)
Laboratory dispenser (Silent Crusher M, Heidolph)
Laboratory standard glassware and equipment
Rotary vacuum evaporator (Heidolph Laborota 4000)
Thermostated oven
Ultrasonic bath

4.3.2 Reagents

Acetone, GC grade (Merck No. 1.00012)
Acetonitrile, gradient grade (Baker No. 9012)
Acetonitrile, gradient grade (Merck No. 1.00030)
Cyclohexane, pestanal (Riedel-de Haën No. 34496)
Ethyl acetate, pestanal (Riedel-de Haën No. 34490)
Iso-Octane, GC grade (Merck No. 1.15440)
Methanol, LC-MS grade (Riedel-de Haën No. 34966)
N-Hexane, pestanal (Fluka No. 52749)
Toluene, pestanal (Riedel-de Haën No. 34494)
Water, for chromatography (Merck No. 1.15333)
Water, deionized (prepared at test site)

Eluent 1 (n-hexane/toluene 65:35 (v/v))
Eluent 2 (toluene)
Eluent 3 (toluene/acetone 95:5 (v/v))

Calflo E (Ehrenstorfer No. E 10935000)
Celite 545 (Merck No. 1.02693)
Silica gel 60, 0.063-0.200 mm particle size (Merck No. 1.07734), deactivated with 1.5 % water
Sodium chloride, p.a. (Riedel-de Haën No. 31434)
Sodium sulfate, anhydrous (Merck No. 1.06649)

Folded filters 595 ½ (Schleicher & Schuell No. 10311647)
Glass fibre filters 691 (VWR No. 516-0036)
Polyethylene frits (Varian No. 12131021)
Single use filters Bulk GHP Acrodisc, 0.45 µm GHP membrane (Pall No. 4562)
Single use filters Minisart SRP 15, 0.45 µm PTFE membrane (Sartorius No. 17559)

4.3.3 Extraction Procedure for Potato

25 g (weight W) of the pre-homogenized sample is accurately weighed into a 500 mL glass bottle. In case of recovery experiments the fortification standard solution is added at this step.

80 mL water is added to the sample in order to adjust the total water content to 100 g. Afterwards the sample is allowed to stand for five minutes.

200 mL acetone and 35 g sodium chloride are added and the sample is homogenized for 2 min at high speed with a laboratory dispenser. Then 100 mL cyclohexane/ethyl acetate (1:1, v/v) is added and mixed again for 1 min. After extraction the sample is allowed to stand for several minutes so that the phases can separate.

200 mL (V_{R1}) of the upper organic phase is measured out using a graduated cylinder and filtered through a cellulose folded filter filled with 100 g anhydrous sodium sulphate. The eluate is collect in a 500 mL round-bottom flask. The filter cake and graduated cylinder is rinsed four times with each ≈ 20 mL ethyl acetate/cyclohexane (1:1, v/v). The combined filtrate is rotary-evaporated (40 °C water bath temperature) to an aqueous oily residue (≈ 1 mL).

The residue is reconstituted in 7.5 mL ethyl acetate and sonicated to dissolve the residue completely. 7.5 mL cyclohexane are added to obtain a total extract volume of 15 mL (V_{R2}). Finally 5 g of a mixture of sodium sulphate/sodium chloride (1:1, w/w) is added and the mixture and swirled. After the salt mixture has settled the extract is filtered through a single-use 0.45 µm membrane filter (0.45 µm into a glass vial for GPC injection.

4.3.4 Extraction Procedure for Rape Seed

25 g (weight W) of the pre-homogenized sample is accurately weighed into a 500 mL glass bottle. In case of recovery experiments the fortification standard solution is added at this step.

25 mL acetone, 225 mL acetonitrile, 20 g Calflo E and 10 g Celite 545 are added and the mixture is homogenised for 2 min at high speed using a laboratory dispenser.

The suspension with suction is filtered through a glass fiber filter in a Buchner porcelain funnel. Vacuum is gently applied to obtain a minimum of 120 mL filtrate.

The filtrate is filtered through a dry fluted cellulose filter paper covered with 3 g Calflo E into a measuring cylinder. 80 mL of filtrate (volume V_{R1}) is transferred to a 500 mL round-bottom flask rinsing the measuring cylinder twice with each 20 mL acetone. The solvent is removed by rotary evaporation (water bath temperature 40 °C) until an oily residue of approx. 1 mL remains.

The residue is reconstituted in 7.5 mL ethyl acetate and sonicated to dissolve the residue completely. 7.5 mL cyclohexane are added to obtain a total extract volume of 15 mL (V_{R2}). Finally 5 g of a mixture of sodium sulphate/sodium chloride (1:1, w/w) is added and the mixture and swirled. After the salt mixture has settled the extract is filtered through a single-use 0.45 µm membrane filter into a glass vial for GPC injection.

4.3.5 Gel Permeation Cleanup

Each extract from 4.3.3 and 4.3.4 is purified using the following GPC conditions:

Column:	Glass column (25 mm i.d. x 580 mm length) filled with 50 g BioBeads S-X3, 200-400 mesh (BioRad)
Flow rate:	5 mL/min ethyl acetate/cyclohexane (1:1, v/v)
Injection volume:	4 mL (V_{R3})
Collection program:	collect 19.0 – 29.0 min

Collect the GPC fraction in a 100 mL round-bottom flask and rotary evaporate to ≈ 1 mL (bath temperature 40 °C). Add 5 mL isooctane and rotary-evaporate to ≈ 1 mL (40 °C), then dissolve residue in 5 mL isooctane.

4.3.6 Silica Gel Cleanup

Preparation of chromatographic columns: In a 5 mL syringe tube with PE frit on the bottom 1.0 g of deactivated silica gel (1.5 % water) is added. Another PE frit is put on the top of the silica gel and a 5 mm layer of anhydrous sodium sulphate is added. The column is rinsed with 5 mL hexane.

The isooctane solution from 4.3.5 is transferred onto the silica gel column. The flask is rinsed with 1 mL hexane and the rinse is added to the column. Afterwards the flask is rinsed with 4 mL eluent 1 (n-hexane/toluene 65:35) which is applied to the column followed by another 4 mL of eluent 1. Then the columns are rinsed with 2 x 4 mL eluent 2 (toluene). All eluates so far are discarded.

Now 50 mL round bottom flasks are placed under the columns and the analyte is eluted with 2 x 4 mL eluent 3 (toluene/acetone 95:5). The collected eluates are evaporated to near dryness (40 °C water bath temperature), the remaining solvent is removed with a slight stream of nitrogen. The residue is reconstituted with 2 mL acetonitrile/water (1:1, v/v) and filtered over 0.45 µm PTFE filters into the HPLC vial.

4.3.7 Determination of Dry Matter

Dry matter of potato matrix was determined by weighing about 50 g sample into an aluminium dish and determining the loss of water after drying overnight at 105 °C in a laboratory oven. The water content of potato matrix was determined to be 82 %.

4.3.8 Analysis by HPLC-MS/MS

HPLC system: Thermo Surveyor MS pump with autosampler

Column: Thermo HyPurity Aquastar, 150 mm x 3 mm i.d., 5 µm mean particle size (No. 22505-153030) and Aquastar guard column

Injection volume: 10 µL

Flow rate: 0.5 mL/min

Temperature: 40 °C

Mobile phase: A: water
B: methanol

Time (min)	% A	% B	Gradient
0.00	80	20	–
6.00	10	90	linear
7.00	10	90	–

Washing the column with 100 % acetonitrile for 2 minutes and afterwards equilibration at initial conditions for 2.5 minutes.

Retention time: Clomazone: approx. 6.1 min

Detector: ThermoFinnigan TSQ Quantum triple quadrupole system

Ionization mode:	APCI
Source polarity:	positive
Vaporizer temperature:	400 °C
Capillary temperature:	250 °C
Capillary offset:	35 V
Sheath gas flow:	37 units
Auxiliary gas flow:	off
Collision gas:	Argon 2.0 mTorr
Quadrupole 1 peak width:	0.7 amu
Quadrupole 3 peak width:	0.7 amu
Scan time:	0.1 sec
Scan width:	0.1 amu

Source and detection parameters for MS/MS experiments:

Compound	Parent m/z	Collision energy (V)	Fragment ions (m/z)
Clomazone	240.0 240.0	42 21	89.1 124.9

Quantification: Peak areas of fragment ions, external standards in acetonitrile/water (1:1, v/v)

Mass transition 240 → 124.9 was used for quantification.

Mass transition 240 → 89.1 was used for confirmation.

At the beginning of the sequence, a calibration curve was constructed by injecting standard solutions within the range from 1 ng/mL to 150 ng/mL (11 calibration points). Injections of sample extracts were interspersed with injections of standards after 2-4 samples to verify the detector response and to adjust the determined concentration to the nominal concentration, if necessary.

4.3.9 Calculation of Residues

Residues of clomazone in potato and rape seed are calculated by the following equation:

$$R = \frac{c \cdot V_{Ex} \cdot V_{R2} \cdot V_{End} \cdot f}{V_{R1} \cdot V_{R3} \cdot W \cdot 1000}$$

R	Residue (mg/kg)
c	Concentration in final extract (ng/mL)
V_{cx}	Volume of organic extract after extraction / partition: 285 mL for potato (100 mL water + 200 mL acetone – 15 mL empirical volume shrinking) respectively 250 mL for rape seeds (25 mL acetone + 225 mL acetonitrile)
V_{R1}	Aliquot of V_{Ex} used for further analysis (200 mL for potato, 80 mL for rape seeds)
V_{R2}	Volume of solution for GPC (15 mL)
V_{R3}	Aliquot of V_{R2} injected onto GPC column (4 mL)
V_{End}	Final volume for HPLC analysis (2 mL)
f	Dilution factor of final extract prior to HPLC-MS/MS measurement
W	Sample weight (25 g)

5 Deviations from the Study Plan

The study was performed according to the study plan dated 20 December 2006.

6 Results

6.1 Recoveries (Accuracy)

Recoveries were obtained by fortification of potato and rape seed samples with the test item prior to extraction. The results are given in Table 2.

Table 2: Recovery of clomazone from potato and rape seed samples

Mass transition 240.0 → 124.9 (Quantification)				
Matrix	Fortification Level (mg/kg)	Recovery (%)	Mean Recovery ± RSD (%)	Overall Mean Recovery ± RSD (%)
Potato	0.005	85 / 85 / 80 / 76 / 86	82 ± 5	84 ± 7
	0.05	89 / 86 / 89 / 73 / 93	86 ± 9	
Rape seed	0.005	88 / 87 / 94 / 96 / 92	91 ± 4	92 ± 6
	0.05	101 / 81 / 87 / 94 / 95	92 ± 8	

Overall mean recovery calculated from individual recoveries.

Mass transition 240.0 → 89.1 (Confirmation)				
Matrix	Fortification Level (mg/kg)	Recovery (%)	Mean Recovery ± RSD (%)	Overall Mean Recovery ± RSD (%)
Potato	0.005	88 / 89 / 84 / 80 / 91	86 ± 5	86 ± 7
	0.05	87 / 86 / 90 / 73 / 92	86 ± 9	
Rape seed	0.005	88 / 87 / 89 / 93 / 90	89 ± 3	89 ± 7
	0.05	102 / 79 / 86 / 87 / 88	88 ± 9	

Overall mean recovery calculated from individual recoveries.

6.2 Blanks

Two untreated control samples were analysed from each matrix. Residues of clomazone were not detectable (i.e. < 30 % of LOQ, see also chromatograms in Appendix C).

6.3 Repeatability (Precision)

The relative standard deviations (RSD) for each fortification level and each commodity was 4 % to 9 % RSD so the requirements of guideline SANCO/825/00 rev. 7 (17/03/2004) are fulfilled (≤ 20 % RSD).

6.4 Limit of Quantification

The limit of quantification is defined as the lowest fortification level with mean recoveries ranging between 70 % and 110 % at a relative standard deviation not exceeding 20 % and blanks not exceeding 30 %. These conditions were fulfilled at the 0.005 mg/kg fortification level for potato and rape seed.

6.5 Linearity

For analysis of clomazone by HPLC/MS-MS the detector response was linear within the range from 1 ng/mL to 150 ng/mL with a correlation coefficient of $r^2 > 0.999$ (see Figure 1 in Appendix A).

6.6 Specificity

A highly specific detection system was used (MS/MS). The retention time of clomazone in solvent matched the retention time in extracts from fortified samples. No significant peak interferences occurred at the retention time of clomazone.

The analytical method can be therefore regarded as highly specific for clomazone parent compound. Since detection is performed by MS/MS, an additional confirmatory method is not necessary according to guideline SANCO/825/00.

6.7 Matrix Effects

To check possible ion enhancement or suppression effects in HPLC/MS-MS analysis, blank extracts from each matrix were spiked with standard solutions. The test item concentration was then quantified against standards in acetonitrile/water (1:1). The results are summarized in Table 3.

Table 3: Checking for possible matrix effects

Sample	Clomazone nominal (ng/mL)	Clomazone determined (ng/mL)		% of nominal	
		Quantifier	Qualifier	Quantifier	Qualifier
Potato	10	9.97	10.46	100	105
	100	97.2	93.8	97	94
Rape seed	5	4.98	4.95	100	99
	50	49.6	49.0	99	98

Quantifier: mass transition 240.0 → 124.9, Qualifier: mass transition 240.0 → 89.1

This indicates that matrix effects are not significant for potato and rape seed, so the quantification could be performed against standards prepared in acetonitrile/water (1:1, v/v).

6.8 Stability of Clomazone in Final Sample Extracts

All rape samples were analysed within 24 hours after extraction, so the storage stability of clomazone in final rape seed extracts was not tested.

Some potato extracts were stored for up to 8 days at approximately 4 °C until analysis. Therefore the storage stability was tested by spiking extracts from untreated samples with the test item at 10 ng/mL and 100 ng/mL, followed by analysis after 8 days of storage at approximately 4 °C.

The measured concentration of clomazone after 8 days was 100 % of the added amount for both fortification levels and both mass transitions. So clomazone can be regarded as stable in final potato extracts for 8 days.

Table 4: Checking for stability of clomazone in potato extracts

Fortification level (potato matrix)	Clomazone determined after 8 days storage (ng/mL)	
	Quantifier	Qualifier
10 ng/mL	10.06 / 10.01	10.01 / 9.99
100 ng/mL	100.7 / 100.0	99.7 / 99.3

7 Discussion and Conclusions

A residue method for determination of clomazone in plant material was developed and successfully validated. The analytical method fulfils all requirements of guideline SANCO/825/00 rev. 7:

- Validation of the method demonstrated that it is specific to clomazone with a LOQ of 0.005 mg/kg in potato and rape seed.
- The results of this study indicate that the recovery efficiency and repeatability were within acceptable limits of 70 % - 110 % for mean recovery and < 20 % RSD at each fortification level.
- Interferences from the control matrix used to validate at the limit of quantification were not detectable.

8 Archiving

For the periods demanded by the principles of GLP the following documents and materials will be archived:

- Study plan, raw data, comments of the sponsor on the draft report and the final report (15 years).
- All documentation generated by the Quality Assurance Unit (15 years).
- A sample of the test item.

All documents and materials will be stored in the archives of eurofins-GAB GmbH. The premises for storing the documents and materials are settled according to the principles of Good Laboratory Practice in the organization of the testing facility.

9 References

SANCO/825/00 rev. 7, Guidance document on residue analytical methods, European Commission – Directorate General Health and Consumer Protection, 17/03/2004.

10 Distribution

Study Plan

Original: Testing facility (1 x)
Sponsor (1 x)

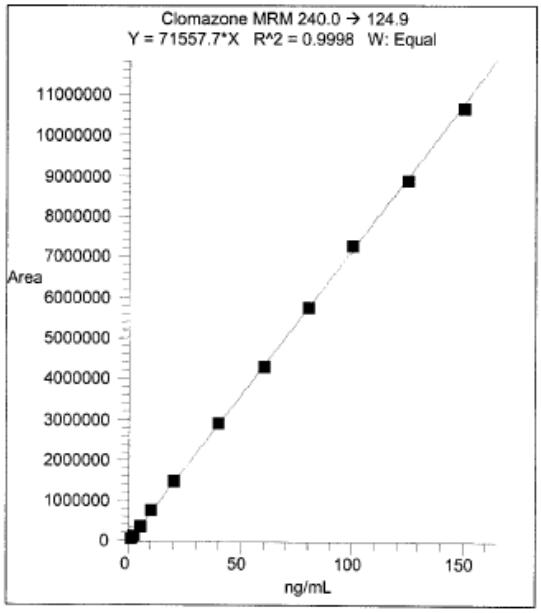
Final Report

Original: Testing facility (1 x)
Sponsor (1 x)

Raw Data

Original: Testing facility

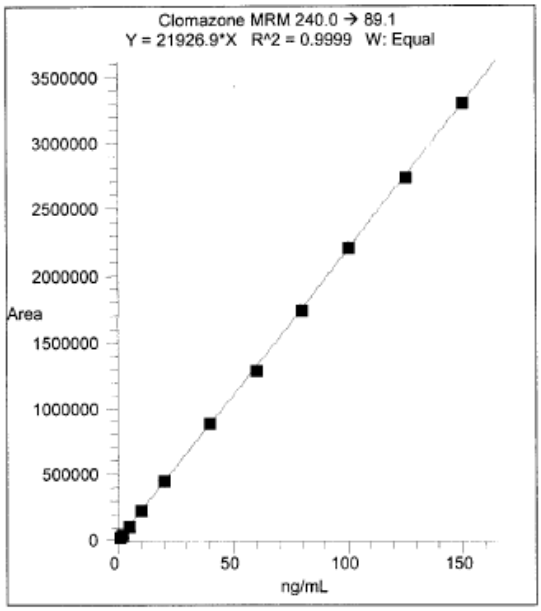
Appendix A: Calibration Data



Clomazone (ng/mL)	Peak area	Calculated concentration (ng/mL)
150	10 669 898	149.109
125	8 890 168	124.238
100	7 268 954	101.582
80	5 742 836	80.255
60	4 288 029	59.924
40	2 919 414	40.798
20	1 486 794	20.778
10	763 172	10.665
5	368 779	5.154
2	144 811	2.024
1	71 513	0.999

Peak areas refer to the area of the fragment ion (m/z = 124.9)

Figure 1: Calibration data for analysis of clomazone in acetonitrile/water (1:1) by HPLC/MS-MS, mass transition MRM 240.0 → 124.9 (quantifier)



Clomazone (ng/mL)	Peak area	Calculated concentration (ng/mL)
150	3 295 898	150.313
125	2 737 443	124.844
100	2 203 935	100.513
80	1 742 806	79.483
60	1 292 961	58.967
40	881 528	40.203
20	454 066	20.708
10	223 968	10.214
5	110 862	5.056
2	43 002	1.961
1	21 718	0.990

Peak areas refer to the area of the fragment ion (m/z = 89.1)

Figure 2: Calibration data for analysis of clomazone in acetonitrile/water (1:1) by HPLC/MS-MS, mass transition MRM 240.0 → 89.1 (qualifier)

Appendix B: Mass Spectrum

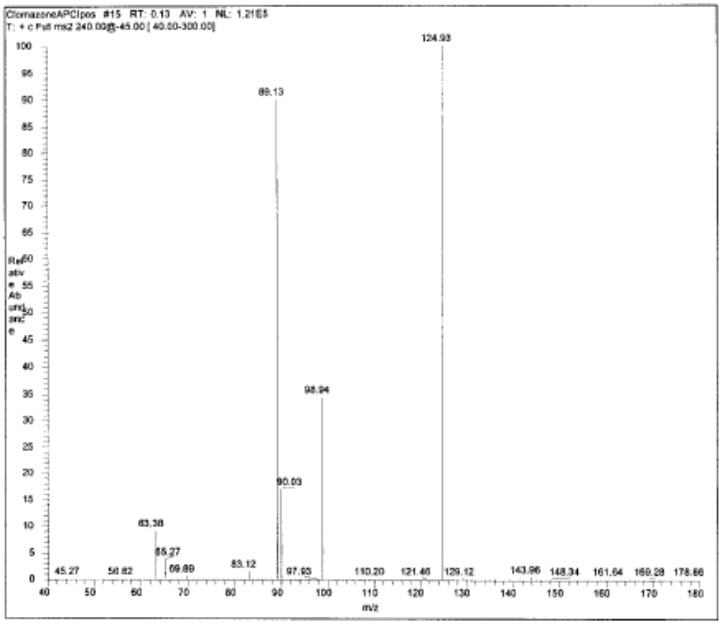


Figure 3: Product ion spectrum of clomazone parent ion (m/z = 240). The daughter ion at m/z = 124.9 was used for quantification, the daughter ion at m/z = 89.1 was used for qualification.

Appendix C: Chromatograms

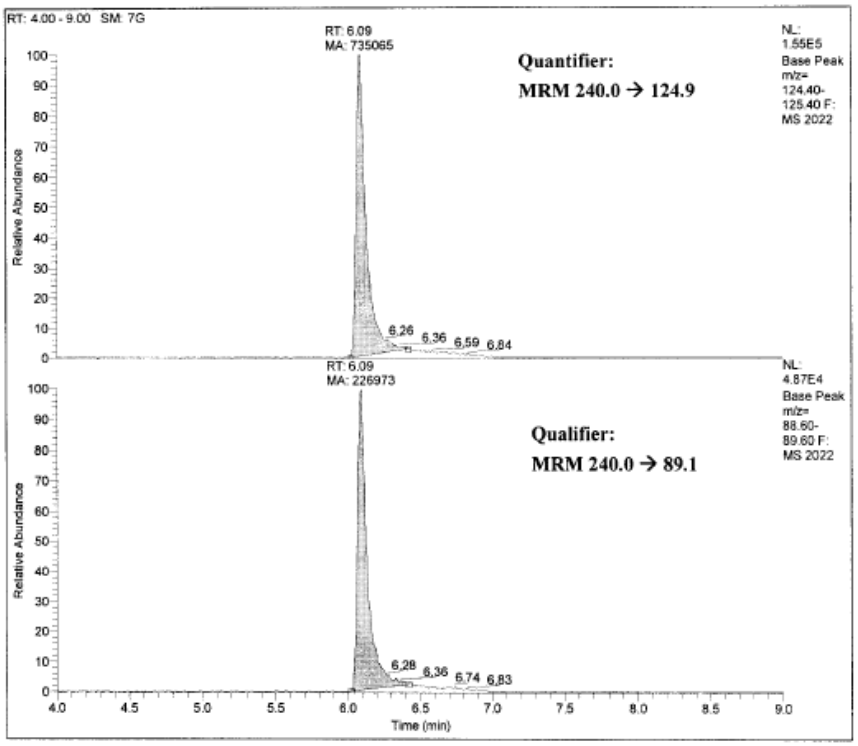


Figure 4: Typical chromatogram of a clomazone 10 ng/mL standard in acetonitrile/water (1:1, v/v)

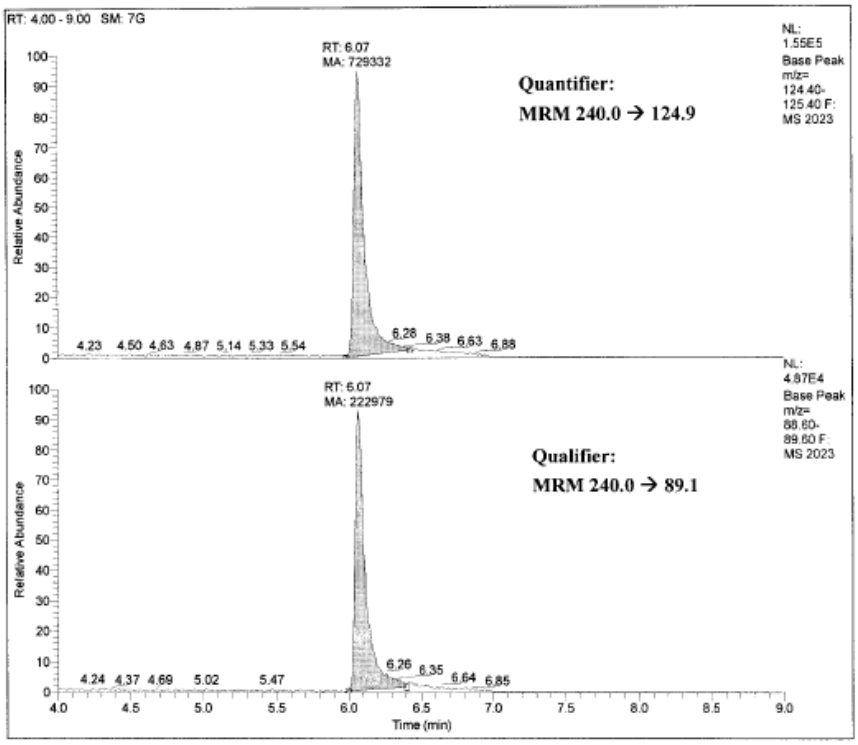


Figure 5: Typical chromatogram of a clomazone recovery sample in potato matrix, fortified at 0.005 mg/kg

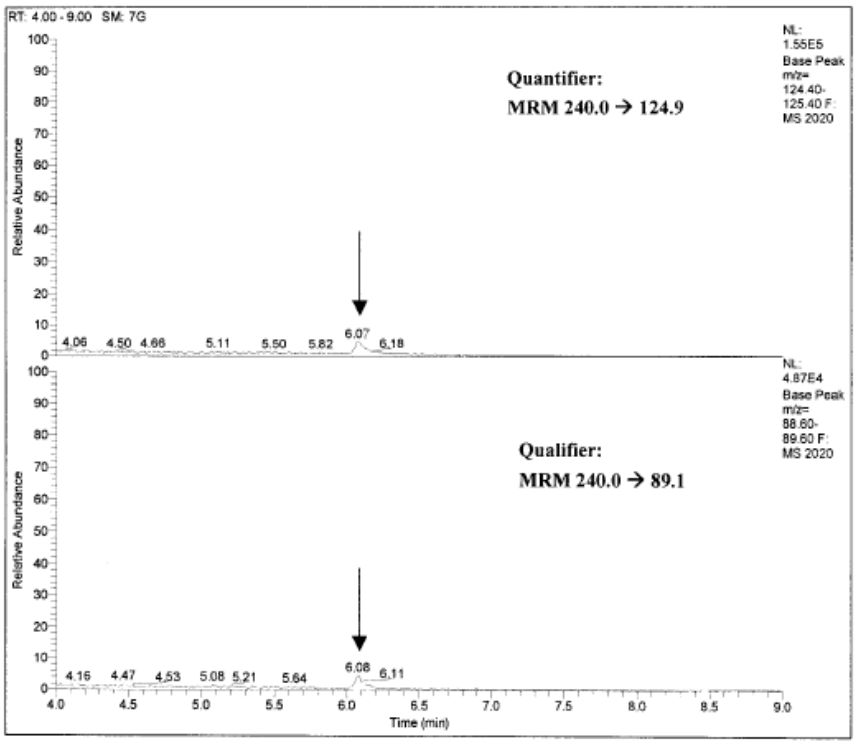


Figure 6: Typical chromatogram of a blank potato sample

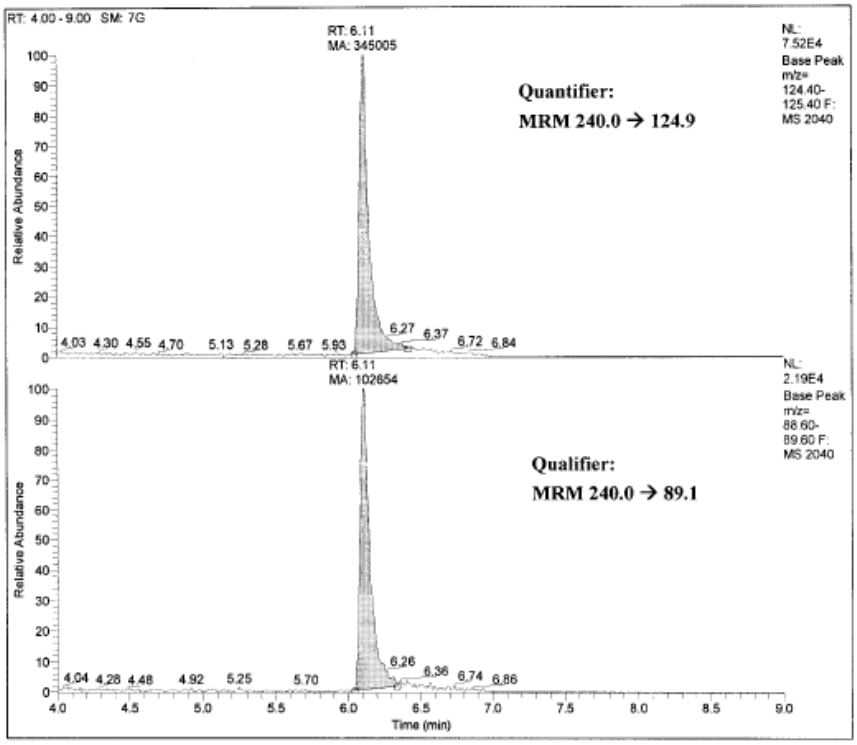


Figure 7: Typical chromatogram of a clomazone 5 ng/mL standard in acetonitrile/water (1:1, v/v)

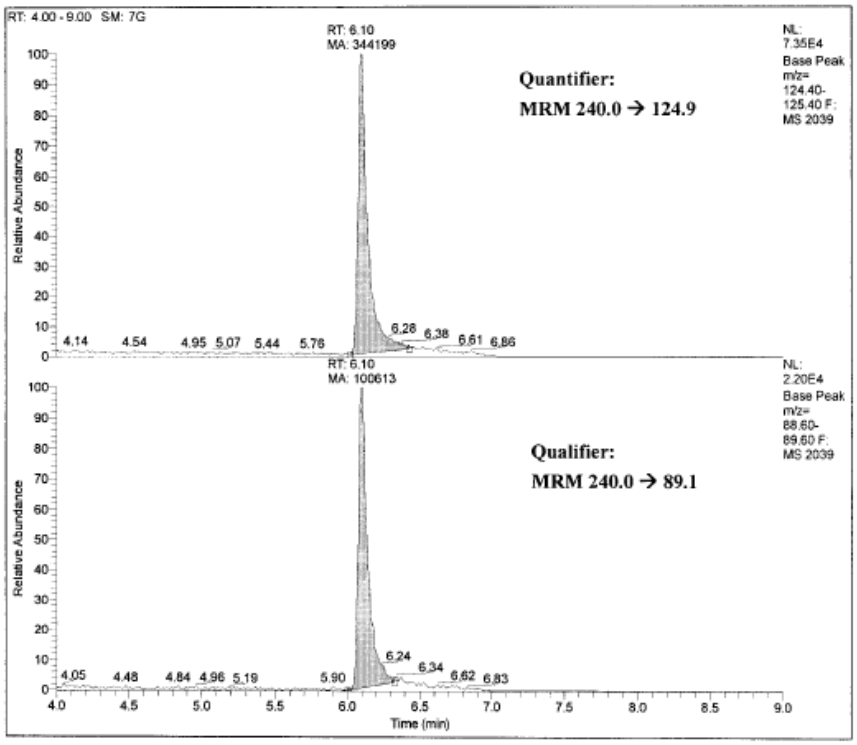


Figure 8: Typical chromatogram of a clomazone recovery sample in rape matrix, fortified at 0.005 mg/kg

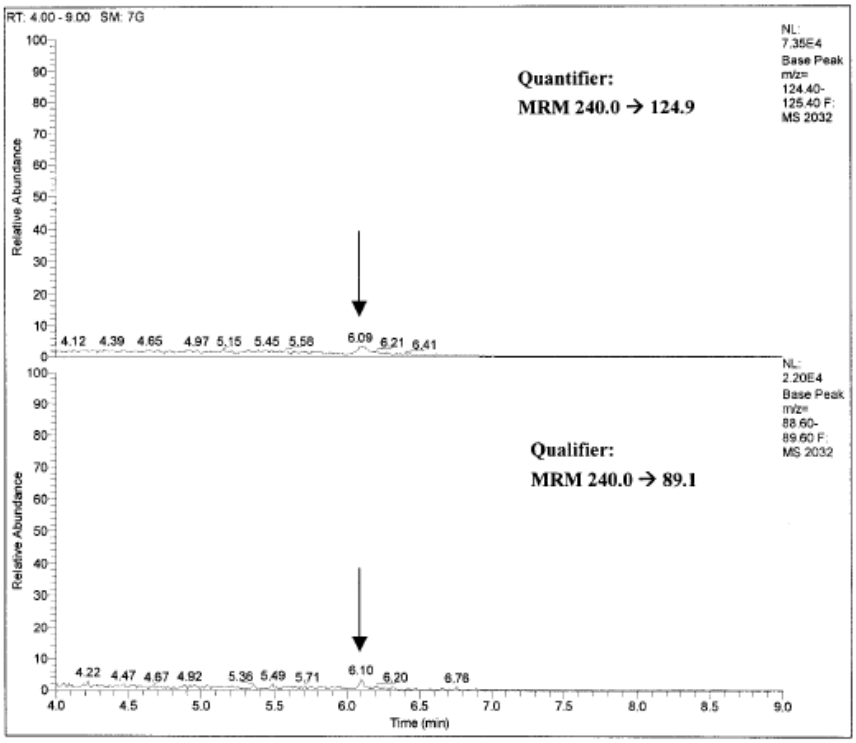


Figure 9: Typical chromatogram of a blank rape sample

Appendix D: Certificates

CHEMINOVA

Cheminova A/S
P.O. Box 9
DK-7620 Lemvig
Denmark

Phone (+45) 96 80 96 80
Fax (+45) 96 80 96 81
www.cheminova.com
CVR-No. DK 12 76 00 43

BATCH ANALYTICAL CERTIFICATE

ARTICLE IDENTIFICATION

Article Name: Clomazone — Reg. Dept. Code:
Manufacturer: Cheminova A/S — Batch No.: HKA, p. 4174
Origin of Production: Commercial [ ]; Pilot plant [ ]; Laboratory [X];

PHYSICAL PROPERTIES

Technical Product [ ]; Preparation of technical Product [ ]; Analytical Standard [X]; Liquid [ ]; Solid [X]; Colour: White

Recommended Storage Conditions

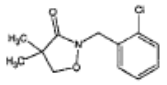
Ambient temperature in the dark ___
In refrigerator ___
In deep freezer X

Expiry Date:
The article is stable at least 2 years from date of analysis/last date of reanalysis when stored at recommended conditions.

Additional Comments:

ACTIVE INGREDIENT IDENTIFICATION

Common Name/ISO-Name: Clomazone
Systematic Name: 3-Isoxazolidinone, 2-[(2-chlorophenyl)methyl]-4,4-dimethyl.
CAS No.: 81777-89-1
Empirical Formula: $C_{12}H_{14}ClNO_2$
Structural Formula:



Molecular Weight: 239.70

Identified by means of:

NMR [X]; IR [X]; UV [X]; MS [X]; Other Methods:

ANALYTICAL DATA

Certified Purity/Content of a.i.: 99.7% w/w
Analytical Method: GC/HPLC profiling
Analytical Report (incl. amendments): RBP 315-01

Date of analysis/ reanalysis (yymmdd)	060820				
-for article stored at -	Cheminova A/S				

GLP-COMPLIANCE

The identification and determination of purity/content of active ingredient were performed at Cheminova A/S and conducted in accordance with FIFRA Good Laboratory Practice Standards, 40 CFR Part 160 and the OECD Principles of Good Laboratory Practices. All raw data, documentation, records, study plans, test articles, reference samples, and report are retained in the GLP archives of Cheminova A/S, Denmark.

Date: September 5, 2006 Signature: Sune Bauer Hansen

Figure 10: Certificate of analysis for clomazone test substance

MINISTERIUM FÜR UMWELT UND VERKEHR
BADEN-WÜRTTEMBERG

Gute Laborpraxis / Good Laboratory Practice

GLP-Bescheinigung / Statement of GLP Compliance

(gemäß /according to § 19b Chemikaliengesetz)

Eine GLP-Inspektion zur Überwachung der Einhaltung der GLP-Grundsätze gemäß Chemikaliengesetz bzw. Richtlinie 88/320/EG wurde durchgeführt in:

Assessment of conformity with GLP according to Chemikaliengesetz and Directive 88/320/EEC at:

☒ Prüfeinrichtung/Test facility ☐ Prüfstandort/Test site

GAB Biotechnologie GmbH / GAB Analytik GmbH
Eutinger Straße 24
75223 Niefern-Öschelbronn

(Unverwechselbare Bezeichnung und Adresse/Unequivocal name and address)

Prüfungen nach Kategorien/Areas of Expertise
(gemäß/according ChemVwV-GLP Nr.5.3/OECD guidance)

1	Prüfungen zur Bestimmung der physikalisch-chemischen Eigenschaften und Gehaltsbestimmungen	6	Prüfungen zur Bestimmung von Rückständen
4	Ökotoxikologische Prüfungen zur Bestimmung der Auswirkungen auf aquatische und terrestrische Organismen	7	Prüfungen zur Bestimmung der Auswirkungen auf Mesokosmen und natürliche Ökosysteme
5	Prüfungen zum Verhalten im Boden, im Wasser und in der Luft; Prüfungen zur Bioakkumulation und zur Metabolisierung	8	Analytische Prüfungen an biologischen Materialien

Zuordnung der Prüfkategorien innerhalb der Prüfeinrichtung:
GAB-Biotechnologie GmbH: Prüfkategorien **1, 4, 5, 7.** GAB Analytik GmbH : Prüfkategorien **1, 5, 6, 8.**

Datum der Inspektion/Date of Inspection
(Tag, Monat, Jahr/day, month, year)

15.09.2003

Die/Der genannte Prüfeinrichtung/Prüfstandort befindet sich im nationalen GLP-Überwachungsverfahren und wird regelmäßig auf Einhaltung der GLP-Grundsätze überwacht.

The above mentioned test facility/test site is included in the national GLP Compliance Programme and is inspected on a regular basis.

Auf der Grundlage des Inspektionsberichtes wird hiermit bestätigt, dass in dieser Prüfeinrichtung/diesem Prüfstandort die oben genannten Prüfungen unter Einhaltung der GLP-Grundsätze durchgeführt werden können.

Based on the inspection report it can be confirmed, that this test facility/test site is able to conduct the aforementioned studies in compliance with the Principles of GLP.

Unterschrift, Datum/Signature, Date

Hofmann Stuttgart, 08.12.2003 / 19.05.2004 (Namensänderung)

(Name und Funktion der verantwortlichen Person/Name and function of responsible person)

Ministerium für Umwelt und Verkehr Baden-Württemberg
Kernerplatz 9, 70182 Stuttgart

(Name und Adresse der GLP-Überwachungsbehörde/Name and address of GLP Monitoring Authority)

Figure 11: GLP certificate of testing facility

www.abclabs.com
4780 Discovery Drive • Columbia, Missouri 65201 800.538.5227 7200 E. ABC Lane • Columbia, Missouri 65202

23 April 2014

Method Modifications to Eurofins-GAB GmbH Method, "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape," found in Eurofins-GAB GmbH Study Code 20061401/01-RVP, dated January 17, 2007

Applicability: Analysis of canola seed with an LOQ of 0.02 ppm

Reasons for Modification:

1. To change the concentration of linearity standards
2. To modify the apparatus used for the study
3. To document modifications to the method
4. To provide calculations used for the study

Modifications to the applicable sections:

4 Materials and Methods

4.1 Test Item

Add the following to the end of the section:

4.1.1 Preparation of Intermediate and Linearity Standards:

The following concentrations of intermediate and calibration standard solutions were stored in the dark typically at 2 °C to 8 °C.

100 ng/mL: 1.0 mL of a 1.0-µg/mL intermediate standard solution was transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

20 ng/mL: 0.20 mL of a 1.0-µg/mL intermediate standard solution was transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

5.0 ng/mL: 0.50 mL of a 100-ng/mL intermediate standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.30 ng/mL: 0.15 mL of a 20-ng/mL intermediate standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.15 ng/mL: 0.30 mL of a 5.0-ng/mL intermediate standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.050 ng/mL: 0.10 mL of a 5.0-ng/mL intermediate standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.030 ng/mL: 1.0 mL of a 0.30-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.010 ng/mL: 0.667 mL of a 0.15-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

4.3 Procedure for Determination of Clomazone

4.3.1 Equipment

Add the following apparatus:

Centrifuge

4.3.4 Extraction Procedure for Rape Seed

Replace this Section with the following:

1. Weigh 25-g sample into a quart glass jar

2. Add 25 mL acetone, 225 mL ACN, 10 g Calflo E, and 10 g Celite 545 and homogenize for 2 minutes at high speed.

3. Place a small aliquot (about 5 mL) in a glass tube and centrifuge at ~2500 rpm for about 5 minutes.

4. Filter the supernatant through a 0.45 µ PTFE filter into a glass tube.

5. Combine 0.1 mL of filtered sample plus 9.9 mL of 1:1 ACN/water in a glass tube and place in a vial for HPLC analysis.

4.3.5 Gel Permeation Cleanup

This Section was deemed unnecessary and was omitted.

4.3.6 Silica Gel Cleanup

This Section was deemed unnecessary and was omitted.

4.3.9 Calculation of Residues

Replace this Section with the following:

A standard curve is generated in Analyst by plotting the standard concentration (in ng/mL) on the x-axis and the respective peak response (area) on the y-axis and using linear regression to calculate a best-fit line. Weighting (1/x) was used in the generation of standard curves.

The calculations for ppm found are:

1. The amount of clomazone found (in ppm) in the sample is calculated according to the following equation:

$$ppm = ng/mL \times \frac{HPLC\ FV\ (mL)}{sample\ \ wt.\ (g)} \times \frac{ext.\ solv.\ (mL)}{aliq.\ (mL)} \times HPLC\ dil.\ factor \times \frac{1}{1000}$$

where:

ng/mL	=	ng/mL of analyte found as determined by Analyst
HPLC FV (mL)	=	volume of final extract submitted to instrumentation (10 mL)
sample wt. (g)	=	amount of soil sample taken through the extraction process (25.0 g)
ext. solv. (mL)	=	volume of extraction solvent added (250 mL)
aliq. (mL)	=	volume of extract taken for analysis (0.10 mL)
HPLC dil. factor	=	dilution of sample extract required to produce an analyte response bracketed by standards
1/1000	=	conversion factor (µg/ng)

2. The percent recovery for fortified control samples was calculated as follows:

$$\%\ Recovery = \frac{ppm\ \ found\ in\ fortified\ control\ -\ ppm\ found\ in\ control}{ppm\ added} \times 100$$

APPENDIX II

ABC Laboratories SOP CD-TM 2.31.6,
entitled "Determination of Moisture"

CD-TM 2.31.6

abc better insight better outcomes	**STANDARD OPERATING PROCEDURE**
	Determination of Moisture Content

EFFECTIVE DATE: DEC 27 2013	**REVISION DUE DATE:** NOV 22 2016

Prepared by/Date:	21 Nov 13
Quality Assurance Signature/Date:	Chris Hughes 21 Nov. 2013
Management Signature/Date:	Andrew Nott for Joe Troxell 22 Nov 2013

Revision History:

CD-TM 2.31.6
1) Triennial Review
2) Updated signature page format

CD-TM 2.31.5
1) Biennial Review

CD-TM 2.31.4
1) Biennial Review

CD-TM 2.31.3
1) Updated Safety Section
2) Editorial Changes

CD-TM 2.31.2
1) Biennial Update

CD-TM 2.31.1
1) Title Change
2) Change equation in Section 7.4.3
3) Minor Corrections

CD-TM 2.31.0
1) New format

Page 1 of 7

CD-TM 2.31.6

1.0 Purpose

To describe the mathematical determination of a sample's moisture content based upon water loss from a dried sample.

2.0 Scope

This Standard Operating Procedure (SOP) applies to any sample matrix that may lose water as a result of heat exposure.

3.0 Responsibility

All personnel that utilize dry weight, moisture content analysis, or correction factors based upon moisture content are responsible for following this SOP.

4.0 Equipment and Materials

4.1 Appropriate balance

4.2 Drying oven

4.3 Moisture analyzer

5.0 Safety

All applicable safety rules and precautions must be followed.

6.0 General

6.1 When calculating chemical concentrations in soils or sediments or when adjusting soil moisture for soil studies, it is necessary to determine the quantitative dry weight of the substrate.

6.2 The quantitative dry weight of a soil or crop sample is necessary in the determination of correction factors for the normalization of the residue concentration results.

6.3 This SOP lists the accepted method when using an oven to dry the samples. Alternatively, a moisture analyzer may be used (see SOP EQ-2 2).

7.0 Procedure

7.1 The moisture content of any sample (e.g., soil, sediment, or crop materials) is determined by recording the water loss of the sample after a specific dehydration period.

7.1.1 Water loss is determined by comparing the wet sample weight versus the weight of the dried sample.

7.1.2 The same balance should be used throughout for all samples in a given set, if possible. All sample weights will be recorded on suitable data sheets with entries dated and initialed.

CD-TM 2.31.6

7.2 A labeled aluminum weigh boat or other suitable container (e.g., a glass beaker) is weighed and its weight is recorded.

7.2.1 The sample is added to the weigh boat and the combined weight is recorded.

7.2.2 Wet samples are typically dried either for 24 hours or overnight at temperatures ranging between 100-110°C (105 ±5°C).

7.2.3 After the drying period, the samples are cooled in a desiccator, weighed again, and the dry weight is recorded.

7.2.4 Extremely wet (muddy) samples should be air dried for 24 hours prior to oven drying.

7.3 When it is necessary to demonstrate that the samples are at a constant dry weight, at least 10% of the samples should be placed back into the drying oven for a second drying period (minimum of 4 hours or overnight), cooled, and weighed.

7.3.1 This second dry weight is also recorded.

7.3.2 If the first and second dry weights vary by more than 1% of the dry sample weight, all of the samples are returned to the oven for another drying period, cooled, weighed, and these weights are also recorded.

7.3.3 If sample dry weights do not vary by more than 1%, a constant dry weight has been achieved.

7.4 When the time constraints are not compatible with the data requirements for the conduct of specific projects, the dehydration time required may be greatly reduced through the use of a preheated (100-110°C) vacuum oven.

7.4.1 A vacuum pump is attached to the oven, which is evacuated for the duration of the dehydration period.

7.4.2 A dehydration period of at least 4 hours is recommended. Following dehydration, the vacuum is released by slowly bleeding ambient laboratory air into the oven. The sample is then cooled in a desiccator and weighed. Multiple drying steps are not necessary following reduced pressure thermal dehydration.

7.4.3 When nondestructive determinations of sample moisture are required, the percent moisture may be determined by directly weighing the tared sample container containing the wet sample. It is necessary to know the dry weight of the sample within the system to use this nondestructive method.

7.5 Moisture Determination - Wet Weight Basis

Moisture content may be calculated by hand or through a suitable computer data reduction program.

Moisture content on a wet weight basis is calculated as follows:

$$\%\,\text{Moisture} = \left(\frac{\text{B - C}}{\text{B - A}}\right) \text{x}\, 100$$

where A = Weight of weigh boat (g)
B = Weight of weigh boat + wet soil (g)
C = Weight of weigh boat + dry soil (g)

7.6 Moisture Determination - Dry Weight Basis

7.6.1 Some analyses are calculated on a dry weight basis, i.e., the percentage of moisture based on the dry soil weight or gravimetric water content.

7.6.2 The laboratory procedure for determining this value is the same as that for any of the wet weight basis determinations; only the calculation is different.

7.6.3 Moisture content on a dry weight basis is calculated as follows:

$$\%\,\text{Moisture} = \left(\frac{\text{B - C}}{\text{C - A}}\right) \text{x}\, 100$$

where A = Weight of weigh boat (g)
B = Weight of weigh boat + wet soil (g)
C = Weight of weigh boat + dry soil (g)

7.6.4 Example 1 - Gravimetric Moisture Determination

7.6.4.1 750 g (wet weight) of soil are used in a study.

7.6.4.2 Triplicate aliquots are taken for moisture analysis.

7.6.4.3 The average wet weight per sample is 5.00 g.

7.6.4.4 After drying, the average weight per sample is 4.00 g.

7.6.4.5 Thus the average weight of the water in the sample was 1.00 g.

7.6.4.6 The percent moisture for the soil follows.

$$\frac{(5.00\text{ g} - 4.00\text{ g})}{4.00\text{ g}} \times 100\% = \frac{1.00\text{ g}}{4.00\text{ g}} \times 100\% = 0.25 \times 100\% = 25\%$$

7.7 Using Moisture Values as Correction Factors

7.7.1 Often, moisture values are determined for use as correction factors to report results on a dry weight basis, thus normalizing results for variable moisture content.

7.7.2 The wet weight basis and dry weight basis values are used differently as correction factors.

7.7.3 To correct a result such as concentration (ppm) for a wet weight basis moisture determination, use the following formula:

$$\text{Result, dry weight basis (ppm)} = \frac{\text{Result on an "as is" basis (ppm)}}{\left(1 - \frac{\%\,M}{100}\right)}$$

7.7.3.1 An equivalent formula is:

where Fraction dryness = (100 - %M)/100

$$\text{Result, dry weight basis (ppm)} = \frac{\text{Result on an "as is" basis (ppm)}}{\text{Fraction dryness}}$$

Note that % M in these cases is on a wet weight basis. Also note that the result being corrected for moisture can be in any relevant units, not just "ppm."

7.7.3.2 To correct a result such as concentration (ppm) for a dry weight basis moisture determination, use the following formula:

$$\underset{\text{dry weight basis}}{\text{Result (ppm)}} = \underset{\text{"as is" basis}}{\text{Result (ppm)}} \div \left(1 + \frac{\%\,M}{100}\right)$$

Note that % M in this case is on a dry weight basis.

Any mathematically equivalent formula may be used.

7.7.4 Example - Correction Factor on a Dry Weight Basis

7.7.4.1 A 25-g soil sample with a 25% gravimetric moisture content was analyzed for test material.

7.7.4.2 The soil was extracted with 100 mL (0.10 L) of solvent and (after appropriate cleanup) was determined to have a concentration of 12 mg/L in the extraction solvent.

7.7.4.3 The calculation of the concentration of the test material in the soil (mg/kg dry soil weight) is as follows.

$$\frac{25\text{ g wet wt.}}{1.25} = 20\text{ g dry weight}$$

$$\frac{12\text{ mg/L} \times 0.10\text{ L}}{20\text{ g soil}} = \frac{1.2\text{ mg}}{20\text{ g}} = 0.06\text{ mg/g}$$

So, the soil concentration is 0.06 mg/g x 10^3 g/kg = 60 mg/kg.

7.8 Using Moisture Values to Hydrate Soils

For soil testing purposes, the moisture valves are used to determine how to hydrate an air dry soil or sediment to the proper hydration level required by the testing guidelines.

7.8.1 Example 1 - Soil Hydration with Dried Soil

7.8.1.1 2.0 kg (2000 g) of soil (wet weight) are needed for a test.

7.8.1.2 Soil moisture will be adjusted to 35%.

7.8.1.3 Therefore, the total weight of the components, soil and water, will equal the weight of dry soil (the basis for 100% calculation) plus the weight of water (35% of the dry soil weight) for a total of 135%.

7.8.1.4 Thus, to calculate the weight of the dry soil the total wet weight is divided by 135% or 1.35.

$$\frac{2000 \text{ g wet soil}}{1.35} = 1481 \text{ g dry weight}$$

7.8.1.5 Therefore the hydrated soil (2000 g) is prepared by mixing 1481 g of dry soil with 519 mL (1.0 g/mL) of water.

7.8.2 Example 2 - Soil Hydration with Inherent Water Content

7.8.2.1 3.20 kg of hydrated soil with a moisture content of 35% of its dry weight is needed for a test.

7.8.2.2 The air-dried soil to be hydrated for the test has a soil moisture of 4% of its dry weight.

7.8.2.3 Figure the dry soil and water constituents of the hydrated soil.

$$\frac{3.20 \text{ kg hydrated soil}}{1.35} = 2.37 \text{ kg dry soil}$$

3.20 kg hydrated soil - 2.37 kg dry soil = 0.83 kg of water

7.8.2.4 Figure the amount of air dried soil needed to produce 2.37 kg of dry soil

2.37 kg + (0.04 x 2.37 kg) = amount of air dried soil needed
= 2.46 kg

7.6.6.5 Figure the amount of inherent water in 2.46 kg of air dried soil

2.46 kg air dried soil - 2.37 Kg dry soil = 0.09 kg inherent water

7.6.6.6

Figure the amount of water to add to 2.46 kg of air dried soil to produce 3.2 kg of hydrated soil with a moisture content of 35% of its dry weight.

0.83 kg water needed - 0.09 kg of inherent water = 0.74 kg of water needed to add to 2.46 kg of air dried soil to produce 3.2 kg of properly hydrated soil.

8.0 Documentation

This should be documented in facility or study records.

9.0 References

SOP EQ-2 2, *Mettler Toledo HR73P Halogen Moisture Analyzer*

APPENDIX III

Certificates of Analysis

CHEMINOVA
Cheminova A/S
P.O. Box 9
DK-7620 Lemvig
Denmark
Phone (+45) 96 90 96 90
Fax (+45) 96 90 96 91
www.cheminova.com
CVR-No. DK12760043

Certificate of Analysis

REF 315-02

Test substance certified:

Test substance:	***Analytical standard of Clomazone***
Batch No.:	HKA, p. 4204
Origin of test substance:	x Laboratory ☐ Pilot plant ☐ Commercial

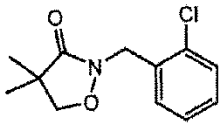
Analysis:

Content:	***99.7% w/w***
Identified by:	^{1}H-NMR, ^{13}C-NMR Spectroscopy, IR Spectroscopy and Mass Spectrometry
Determination of purity by:	High Performance Liquid Chromatography and Gas Chromatography
Date of analysis:	February 11, 2014

Information of the test substance:

Appearance:	White solid
Storage:	< -20°C
Expiry date:	February 11, 2016

Information of analyte(s):

Common name:	Clomazone
CAS name:	3-Isoxazolidinone, 2-[(2-chlorophenyl)methyl]-4,4-dimethyl.
CAS No.:	81777-89-1
Molecular formula:	$C_{12}H_{14}ClNO_2$
Molecular mass:	239.70
Structure formula:	

Statement of GLP Compliance

The identification and determination of purity were performed at Cheminova A/S and conducted according to FIFRA Good Laboratory Practice Standards, 40 CFR Part 160 and the OECD Principles of Good Laboratory Practices.

Date February 19, 2014

(SBH)

CHEMINOVA
Cheminova A/S
P.O. Box 9
DK-7620 Lemvig
Denmark
Phone (+45) 96 90 96 90
Fax (+45) 96 90 96 91
www.cheminova.com
CVR-No. DK 12 76 00 43

Certificate of Analysis

REF 315-02

Test substance certified:

Test substance:	*Analytical standard of Clomazone*
Batch No.:	HKA, p. 4204
Origin of test substance:	x Laboratory ☐ Pilot plant ☐ Commercial

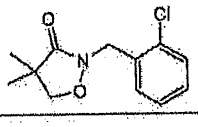
Analysis:

Content:	*99.7% w/w*
Identified by:	^{1}H-NMR, ^{13}C-NMR Spectroscopy, IR Spectroscopy and Mass Spectrometry
Determination of purity by:	High Performance Liquid Chromatography and Gas Chromatography
Date of analysis:	March 28, 2012

Information of the test substance:

Appearance:	White solid
Storage:	< -20°C
Expiry date:	March 28, 2014

Information of analyte(s):

Common name:	Clomazone
CAS name:	3-Isoxazolidinone, 2-[(2-chlorophenyl)methyl]-4,4-dimethyl.
CAS No.:	81777-89-1
Molecular formula:	$C_{12}H_{14}ClNO_2$
Molecular mass:	239.70
Structure formula:	

Statement of GLP Compliance

The identification and determination of purity were performed at Cheminova A/S and conducted according to FIFRA Good Laboratory Practice Standards, 40 CFR Part 160 and the OECD Principles of Good Laboratory Practices.

Date March 28, 2012

Jennifer M. Willett 4/25/13

CofA Received via email dated April 25, 2013

APPENDIX IV

Spreadsheets

69869
TCI-13-366

Residue Results - Method Validation
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	average ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	MV2	B				21-Feb-14						0	0.00					
69869-MV13	TCI-13-366-02-01	MV2	C	25.00		21-Feb-14	21-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-MV14	TCI-13-366-02-01	MV2	C	25.00		21-Feb-14	21-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-MV15	TCI-13-366-02-01 + 0.020	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	6472	0.0180	0.018	0.0180	0.000	0.0180	90
69869-MV16	TCI-13-366-02-01 + 0.020	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	6782	0.0189	0.0189	0.0189	0.000	0.0189	95
69869-MV17	TCI-13-366-02-01 + 0.020	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	5608	0.0154	0.0154	0.0154	0.000	0.0154	77
69869-MV18	TCI-13-366-02-01 + 0.020	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	4833	0.0131	0.0131	0.0131	0.000	0.0131	66
69869-MV19	TCI-13-366-02-01 + 0.020	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	5488	0.0151	0.0151	0.0151	0.000	0.0151	76
69869-MV20	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	27656	0.0811	0.4055	0.406	0.000	0.406	81
69869-MV21	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	31203	0.0916	0.458	0.458	0.000	0.458	92
69869-MV22	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	27465	0.0805	0.4025	0.403	0.000	0.403	81
69869-MV23	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	24981	0.0731	0.3655	0.366	0.000	0.366	73
69869-MV24	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	26533	0.0777	0.3885	0.389	0.000	0.389	78
12562-B9	std. curve 0.03 ng/mL	MV2	S				21-Feb-14						10080						
12562-B10	std. curve 0.01 ng/mL	MV2	S				21-Feb-14						3857						
12562-B8	std. curve 0.05 ng/mL	MV2	S				21-Feb-14						17394						
12562-B9	std. curve 0.03 ng/mL	MV2	S				21-Feb-14						10803						
12562-B7	std. curve 0.15 ng/mL	MV2	S				21-Feb-14						48793						
12562-B6	std. curve 0.3 ng/mL	MV2	S				21-Feb-14						103043						

1/x Weighting
Correlation (r): 0.99944383
Slope (m): 335831.5
Intercept (b): 424.2965

File Name: N 69869 02212014B.rdb

69869
TCI-13-366

Residue Results - Method Validation
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	average ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	MV2	B				21-Feb-14						0	0.00					
69869-MV13	TCI-13-366-02-01	MV2	C	25.00		21-Feb-14	21-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-MV14	TCI-13-366-02-01	MV2	C	25.00		21-Feb-14	21-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-MV15	TCI-13-366-02-01 + 0.02C	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	1565	0.0170	0.017	0.0170	0.000	0.0170	85
69869-MV16	TCI-13-366-02-01 + 0.02C	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	1986	0.0223	0.0223	0.0223	0.000	0.0223	112
69869-MV17	TCI-13-366-02-01 + 0.02C	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	1508	0.0163	0.0163	0.0163	0.000	0.0163	82
69869-MV18	TCI-13-366-02-01 + 0.02C	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	1323	0.0140	0.014	0.0140	0.000	0.0140	70
69869-MV19	TCI-13-366-02-01 + 0.02C	MV2	F	25.00	0.020	21-Feb-14	21-Feb-14	250	0.10	10	5	1	1459	0.0157	0.0157	0.0157	0.000	0.0157	79
69869-MV20	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	7123	0.0863	0.4315	0.432	0.000	0.432	86
69869-MV21	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	7723	0.0937	0.4685	0.469	0.000	0.469	94
69869-MV22	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	6611	0.0799	0.3995	0.400	0.000	0.400	80
69869-MV23	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	6771	0.0819	0.4095	0.410	0.000	0.410	82
69869-MV24	TCI-13-366-02-01 + 0.50	MV2	F	25.00	0.50	21-Feb-14	21-Feb-14	250	0.10	10	5	5	6419	0.0775	0.3875	0.388	0.000	0.388	78
12562-B9	std. curve 0.03 ng/mL	MV2	S				21-Feb-14						2603						
12562-B10	std. curve 0.01 ng/mL	MV2	S				21-Feb-14						1120						
12562-B8	std. curve 0.05 ng/mL	MV2	S				21-Feb-14						4010						
12562-B9	std. curve 0.03 ng/mL	MV2	S				21-Feb-14						2374						
12562-B7	std. curve 0.15 ng/mL	MV2	S				21-Feb-14						11814						
12562-B6	std. curve 0.3 ng/mL	MV2	S				21-Feb-14						25033						

1/x Weighting

Correlation (r): 0.99857503

Slope (m): 80319.05

Intercept (b): 195.594

File Name: N 69869 02212014B.rdb

69869
TCI-13-366

Residue Results
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	1 short	B				26-Feb-14						1200[a]	0.00					
69869-006	TCI-13-366-02-01	1 short	C	25.00		25-Feb-14	26-Feb-14	250	0.10	10	5	1	893[a]	0.00	0	ND			
69869-007	TCI-13-366-02-01 + 0.020	1 short	F	25.00	0.020	25-Feb-14	26-Feb-14	250	0.10	10	5	1	9944	0.0161	0.0161	0.0161	0.000	0.0161	81
69869-008	TCI-13-366-02-01 + 0.50	1 short	F	25.00	0.50	25-Feb-14	26-Feb-14	250	0.10	10	5	5	50639	0.0953	0.4765	0.477	0.000	0.477	95
69869-009	TCI-13-366-02-02	1 short	T	25.00		25-Feb-14	26-Feb-14	250	0.10	10	5	1	1456[a]	0.00	0	ND			
69869-010	TCI-13-366-02-03	1 short	T	25.00		25-Feb-14	26-Feb-14	250	0.10	10	5	1	774[a]	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	1 short	S				26-Feb-14						16858						
12562-B10	std. curve 0.01 ng/mL	1 short	S				26-Feb-14						6847						
12562-B8	std. curve 0.05 ng/mL	1 short	S				26-Feb-14						27779						
12562-B7	std. curve 0.15 ng/mL	1 short	S				26-Feb-14						77058						
12562-B6	std. curve 0.3 ng/mL	1 short	S				26-Feb-14						157377						

1/x Weighting

Correlation (r): 0.99984199

Slope (m): 514199

Intercept (b): 1650.184

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: N 69869 02252014A.rdb

69869
TCI-13-366

Residue Results
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	2	B				17-Mar-14						4358	0.00179					
69869-011	TCI-13-366-01-01	2	C	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	5543	0.00353	0.00353	<0.020			
69869-012	TCI-13-366-01-01 + 0.020	2	F	25.00	0.020	14-Mar-14	17-Mar-14	250	C.10	10	5	1	17818	0.0215	0.0215	0.0215	0.00353	0.0180	90
69869-013	TCI-13-366-01-01 + 0.50	2	F	25.00	0.50	14-Mar-14	17-Mar-14	250	C.10	10	5	5	78595	0.110	0.55	0.550	0.00353	0.546	109
69869-014	TCI-13-366-01-02	2	T	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	3733	0.00088	0.00088	<0.020			
69869-015	TCI-13-366-01-03	2	T	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	3565	0.000634	0.000634	<0.020			
69869-016	TCI-13-366-06-01	2	C	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	2530[a]	0.00	0	ND			
69869-017	TCI-13-366-06-02	2	T	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	3124[a]	0.00	0	ND			
69869-018	TCI-13-366-06-03	2	T	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	5755	0.00384	0.00384	<0.020			
69869-019	TCI-13-366-06-04	2	T	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	8103	0.00727	0.00727	<0.020			
69869-020	TCI-13-366-06-05	2	T	25.00		14-Mar-14	17-Mar-14	250	C.10	10	5	1	3132	0.000000914	0.000000914	<0.020			
12562-B9	std. curve 0.03 ng/mL	2	S				17-Mar-14						25901						
12562-B10	std. curve 0.01 ng/mL	2	S				17-Mar-14						9012						
12562-B8	std. curve 0.05 ng/mL	2	S				17-Mar-14						38946						
12562-B7	std. curve 0.15 ng/mL	2	S				17-Mar-14						106360						
12562-B7	std. curve 0.15 ng/mL	2	S				17-Mar-14						104000						
12562-B6	std. curve 0.3 ng/mL	2	S				17-Mar-14						206151						

1/x Weighting

Correlation (r): 0.99921505

Slope (m): 683450.3

Intercept (b): 3131.65

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03172014B.rdb

69869
TCI-13-366

Residue Results
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	4	B				19-Mar-14						1927[a]	0.00					
69869-024	TCI-13-366-03-01	4	C	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-025	TCI-13-366-03-01	+ 0.020	4	F	25.00	0.020	18-Mar-14	19-Mar-14	250	0.10	10	5	1	15017	0.0184	0.0184	0.000	0.0184	92
69869-026	TCI-13-366-03-01	+ 0.50	4	F	25.00	0.50	18-Mar-14	19-Mar-14	250	0.10	10	5	5	80247	0.56	0.560	0.000	0.560	112
69869-027	TCI-13-366-03-01	+ 0.020	4	F	25.00	0.020	18-Mar-14	19-Mar-14	250	0.10	10	5	1	15202	0.0187	0.0187	0.000	0.0187	94
69869-028	TCI-13-366-03-02		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2338	0.000124	<0.020			
69869-029	TCI-13-366-03-03		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	1974[a]	0.00	0	ND		
69869-030	TCI-13-366-04-01		4	C	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2686	0.000626	0.000626	<0.020		
69869-031	TCI-13-366-04-02		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND		
69869-032	TCI-13-366-04-03		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND		
69869-033	TCI-13-366-04-04		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2365	0.000163	0.000163	<0.020		
69869-034	TCI-13-366-04-05		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2085[a]	0.00	0	ND		
69869-035	TCI-13-366-04-06		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2551	0.000431	0.000431	<0.020		
69869-036	TCI-13-366-04-07		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2268	0.0000231	0.0000231	<0.020		
69869-037	TCI-13-366-04-08		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2409	0.000226	0.000226	<0.020		
69869-038	TCI-13-366-04-09		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND		
69869-039	TCI-13-366-04-10		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	1900[a]	0.00	0	ND		
69869-040	TCI-13-366-04-11		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2159[a]	0.00	0	ND		
69869-041	TCI-13-366-05-01		4	C	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	6207	0.00570	0.0057	<0.020		
69869-042	TCI-13-366-05-02		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2463	0.000304	0.000304	<0.020		
69869-043	TCI-13-366-05-03		4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	2806	0.000799	0.000799	<0.020		
12562-B9	std. curve 0.03 ng/mL		4	S				19-Mar-14						24320					
12562-B10	std. curve 0.01 ng/mL		4	S				19-Mar-14						10189					
12562-B8	std. curve 0.05 ng/mL		4	S				19-Mar-14						35346					
12562-B7	std. curve 0.15 ng/mL		4	S				19-Mar-14						102231					
12562-B8	std. curve 0.05 ng/mL		4	S				19-Mar-14						34119					
12562-B9	std. curve 0.03 ng/mL		4	S				19-Mar-14						21370					
12562-B7	std. curve 0.15 ng/mL		4	S				19-Mar-14						108340					
12562-B6	std. curve 0.3 ng/mL		4	S				19-Mar-14						216722					

1/x Weighting
Correlation (r): 0.99843628
Slope (m): 694314.4
Intercept (b): 2251.838

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03182014D.rdb

69869
TCI-13-366

Residue Results
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	7	B				21-Mar-14						2438[a]	0.00					
69869-075	TCI-13-366-07-01	7	C	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	5167	0.000256	0.000256	<0.020			
69869-076	TCI-13-366-07-01 + 0.020	7	F	25.00	0.020	21-Mar-14	21-Mar-14	250	0.10	10	10	1	28294	0.0203	0.0203	0.0203	0.000256	0.0200	100
69869-077	TCI-13-366-07-01 + 0.50	7	F	25.00	0.50	21-Mar-14	21-Mar-14	250	0.10	10	10	5	119375	0.4955	0.496	0.496	0.000256	0.495	99
69869-078	TCI-13-366-07-01 + 0.020	7	F	25.00	0.020	21-Mar-14	21-Mar-14	250	0.10	10	10	1	26772	0.0190	0.019	0.0190	0.000256	0.0187	94
69869-079	TCI-13-366-05-04	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	4197[a]	0.00	0	ND			
69869-080	TCI-13-366-05-05	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	4567[a]	0.00	0	ND			
69869-081	TCI-13-366-05-06	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	3195[a]	0.00	0	ND			
69869-082	TCI-13-366-05-07	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	4533[a]	0.00	0	ND			
69869-083	TCI-13-366-05-08	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	3108[a]	0.00	0	ND			
69869-084	TCI-13-366-05-09	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	4313[a]	0.00	0	ND			
69869-085	TCI-13-366-05-10	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	3530[a]	0.00	0	ND			
69869-086	TCI-13-366-05-11	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	2921[a]	0.00	0	ND			
69869-087	TCI-13-366-07-02	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	3281[a]	0.00	0	ND			
69869-088	TCI-13-366-07-03	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	2460[a]	0.00	0	ND			
69869-089	TCI-13-366-08-01	7	C	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	2508[a]	0.00	0	ND			
69869-090	TCI-13-366-08-02	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	3437[a]	0.00	0	ND			
69869-091	TCI-13-366-08-03	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	3214[a]	0.00	0	ND			
69869-092	TCI-13-366-09-01	7	C	25.00		21-Mar-14	22-Mar-14	250	0.10	10	10	1	3132[a]	0.00	0	ND			
69869-093	TCI-13-366-09-02	7	T	25.00		21-Mar-14	22-Mar-14	250	0.10	10	10	1	3031[a]	0.00	0	ND			
69869-094	TCI-13-366-09-03	7	T	25.00		21-Mar-14	22-Mar-14	250	0.10	10	10	1	2547[a]	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	7	S				21-Mar-14						40366						
12562-B10	std. curve 0.01 ng/mL	7	S				21-Mar-14						16644						
12562-B8	std. curve 0.05 ng/mL	7	S				21-Mar-14						62242						
12562-B7	std. curve 0.15 ng/mL	7	S				21-Mar-14						179390						
12562-B8	std. curve 0.05 ng/mL	7	S				21-Mar-14						60191						
12562-B9	std. curve 0.03 ng/mL	7	S				21-Mar-14						39082						
12562-B7	std. curve 0.15 ng/mL	7	S				22-Mar-14						181232						
12562-B6	std. curve 0.3 ng/mL	7	S				22-Mar-14						349382						

1/x Weighting
Correlation (r): 0.99981051
Slope (m): 1155268
Intercept (b): 4871.421

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03212014C.rdb

69869
TCI-13-366

Residue Results
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	8	B				26-Mar-14						2681[a]	0.00					
69869-095	TCI-13-366-10-01	8	C	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3740	0.000610	0.00061	<0.020			
69869-096	TCI-13-366-10-01 + 0.020	8	F	25.00	0.020	26-Mar-14	26-Mar-14	250	0.10	10	5	1	11941	0.0142	0.0142	0.0142	0.000610	0.0136	68
69869-097	TCI-13-366-10-01 + 0.50	8	F	25.00	0.50	26-Mar-14	26-Mar-14	250	0.10	10	5	5	63724	0.0999	0.4995	0.500	0.000610	0.499	100
69869-098	TCI-13-366-10-02	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	2859[a]	0.00	0	ND			
69869-099	TCI-13-366-10-03	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3366[a]	0.00	0	ND			
69869-100	TCI-13-366-11-01	8	C	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3614	0.000402	0.000402	<0.020			
69869-101	TCI-13-366-11-02	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3089[a]	0.00	0	ND			
69869-102	TCI-13-366-11-03	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	2989[a]	0.00	0	ND			
69869-103	TCI-13-366-12-01	8	C	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3274[a]	0.00	0	ND			
69869-104	TCI-13-366-12-02	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3263[a]	0.00	0	ND			
69869-105	TCI-13-366-12-03	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	3454	0.000136	0.000136	<0.020			
12562-B9	std. curve 0.03 ng/mL	8	S				26-Mar-14						22774						
12562-B10	std. curve 0.01 ng/mL	8	S				26-Mar-14						9213						
12562-B8	std. curve 0.05 ng/mL	8	S				26-Mar-14						32302						
12562-B11	std. curve 0.15 ng/mL	8	S				26-Mar-14						94932						
12562-B8	std. curve 0.05 ng/mL	8	S				26-Mar-14						33461						
12562-B6	std. curve 0.3 ng/mL	8	S				26-Mar-14						183822						

1/x Weighting
Correlation (r): 0.99965426
Slope (m): 603853.9
Intercept (b): 3371.712

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03262014B.rdb

69869
TCI-13-366

Residue Results
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	1 short	B				26-Feb-14						0	0.00					
69869-006	TCI-13-366-02-01	1 short	C	25.00		25-Feb-14	26-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-007	TCI-13-366-02-01 + 0.020	1 short	F	25.00	0.020	25-Feb-14	26-Feb-14	250	0.10	10	5	1	2440	0.0163	0.0163	0.0163	0.000	0.0163	82
69869-008	TCI-13-366-02-01 + 0.50	1 short	F	25.00	0.50	25-Feb-14	26-Feb-14	250	0.10	10	5	5	12556	0.0979	0.4895	0.490	0.000	0.490	98
69869-009	TCI-13-366-02-02	1 short	T	25.00		25-Feb-14	26-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-010	TCI-13-366-02-03	1 short	T	25.00		25-Feb-14	26-Feb-14	250	0.10	10	5	1	0	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	1 short	S				26-Feb-14						4208						
12562-B10	std. curve 0.01 ng/mL	1 short	S				26-Feb-14						1657						
12562-B8	std. curve 0.05 ng/mL	1 short	S				26-Feb-14						6523						
12562-B7	std. curve 0.15 ng/mL	1 short	S				26-Feb-14						18764						
12562-B6	std. curve 0.3 ng/mL	1 short	S				26-Feb-14						37902						

1/x Weighting

Correlation (r): 0.99991248
Slope (m): 124045.5
Intercept (b): 413.854

File Name: N 69869 02252014A.rdb

69869
TCI-13-366

Residue Results
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	2	B				17-Mar-14						1880	0.00188					
69869-011	TCI-13-366-01-01	2	C	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	1053[a]	0.00	0	ND			
69869-012	TCI-13-366-01-01 + 0.020	2	F	25.00	0.020	14-Mar-14	17-Mar-14	250	0.10	10	5	1	4443	0.0173	0.0173	0.0173	0.000	0.0173	87
69869-013	TCI-13-366-01-01 + 0.50	2	F	25.00	0.50	14-Mar-14	17-Mar-14	250	0.10	10	5	5	20315	0.113	0.565	0.565	0.000	0.565	113
69869-014	TCI-13-366-01-02	2	T	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-015	TCI-13-366-01-03	2	T	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	1377[a]	0.00	0	ND			
69869-016	TCI-13-366-06-01	2	C	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	718[a]	0.00	0	ND			
69869-017	TCI-13-366-06-02	2	T	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	840[a]	0.00	0	ND			
69869-018	TCI-13-366-06-03	2	T	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	1726	0.000958	0.000958	<0.020			
69869-019	TCI-13-366-06-04	2	T	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	2047	0.00289	0.00289	<0.020			
69869-020	TCI-13-366-06-05	2	T	25.00		14-Mar-14	17-Mar-14	250	0.10	10	5	1	705[a]	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	2	S				17-Mar-14						7106						
12562-B10	std. curve 0.01 ng/mL	2	S				17-Mar-14						3150						
12562-B8	std. curve 0.05 ng/mL	2	S				17-Mar-14						9682						
12562-B7	std. curve 0.15 ng/mL	2	S				17-Mar-14						25695						
12562-B7	std. curve 0.15 ng/mL	2	S				17-Mar-14						25770						
12562-B6	std. curve 0.3 ng/mL	2	S				17-Mar-14						52815						

1/x Weighting

Correlation (r): 0.99902813

Slope (m): 166406.7

Intercept (b): 1566.267

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03172014B.rdb

69869
TCI-13-366

Residue Results
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	4	B				19-Mar-14						0	0.00					
69869-024	TCI-13-366-03-01	4	C	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-025	TCI-13-366-03-01 + 0.020	4	F	25.00	0.020	18-Mar-14	19-Mar-14	250	0.10	10	5	1	4165	0.0173	0.0173	0.0173	0.000	0.0173	87
69869-026	TCI-13-366-03-01 + 0.50	4	F	25.00	0.50	18-Mar-14	19-Mar-14	250	0.10	10	5	5	20143	0.111	0.555	0.555	0.000	0.555	111
69869-027	TCI-13-366-03-01 + 0.020	4	F	25.00	0.020	18-Mar-14	19-Mar-14	250	0.10	10	5	1	4170	0.0173	0.0173	0.0173	0.000	0.0173	87
69869-028	TCI-13-366-03-02	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-029	TCI-13-366-03-03	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-030	TCI-13-366-04-01	4	C	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-031	TCI-13-366-04-02	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-032	TCI-13-366-04-03	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-033	TCI-13-366-04-04	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-034	TCI-13-366-04-05	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-035	TCI-13-366-04-06	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-036	TCI-13-366-04-07	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-037	TCI-13-366-04-08	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-038	TCI-13-366-04-09	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-039	TCI-13-366-04-10	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-040	TCI-13-366-04-11	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-041	TCI-13-366-05-01	4	C	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-042	TCI-13-366-05-02	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
69869-043	TCI-13-366-05-03	4	T	25.00		18-Mar-14	19-Mar-14	250	0.10	10	5	1	0	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	4	S				19-Mar-14						5923						
12562-B10	std. curve 0.01 ng/mL	4	S				19-Mar-14						3507						
12562-B8	std. curve 0.05 ng/mL	4	S				19-Mar-14						9429						
12562-B7	std. curve 0.15 ng/mL	4	S				19-Mar-14						25623						
12562-B8	std. curve 0.05 ng/mL	4	S				19-Mar-14						8944						
12562-B9	std. curve 0.03 ng/mL	4	S				19-Mar-14						5613						
12562-B7	std. curve 0.15 ng/mL	4	S				19-Mar-14						27115						
12562-B6	std. curve 0.3 ng/mL	4	S				19-Mar-14						55364						

1/x Weighting

Correlation (r): 0.99610682
Slope (m): 171303.3
Intercept (b): 1201.747

File Name: H 69869 03182014D rdb

69869
TCI-13-366

Residue Results
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	7	B				21-Mar-14						0	0.00					
69869-075	TCI-13-366-07-01	7	C	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1687	0.000790	0.00079	<0.020			
69869-076	TCI-13-366-07-01 + 0.020	7	F	25.00	0.020	21-Mar-14	21-Mar-14	250	0.10	10	10	1	7233	0.0200	0.02	0.0200	0.000790	0.0192	96
69869-077	TCI-13-366-07-01 + 0.50	7	F	25.00	0.50	21-Mar-14	21-Mar-14	250	0.10	10	10	5	30516	0.101	0.505	0.505	0.000790	0.504	101
69869-078	TCI-13-366-07-01 + 0.020	7	F	25.00	0.020	21-Mar-14	21-Mar-14	250	0.10	10	10	1	7093	0.0195	0.0195	0.0195	0.000790	0.0187	94
69869-079	TCI-13-366-05-04	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1148[a]	0.00	0	ND			
69869-080	TCI-13-366-05-05	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1054[a]	0.00	0	ND			
69869-081	TCI-13-366-05-06	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1019[a]	0.00	0	ND			
69869-082	TCI-13-366-05-07	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1342[a]	0.00	0	ND			
69869-083	TCI-13-366-05-08	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
69869-084	TCI-13-366-05-09	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1121[a]	0.00	0	ND			
69869-085	TCI-13-366-05-10	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1084[a]	0.00	0	ND			
69869-086	TCI-13-366-05-11	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
69869-087	TCI-13-366-07-02	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
69869-088	TCI-13-366-07-03	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
69869-089	TCI-13-366-08-01	7	C	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
69869-090	TCI-13-366-08-02	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1113[a]	0.00	0	ND			
69869-091	TCI-13-366-08-03	7	T	25.00		21-Mar-14	21-Mar-14	250	0.10	10	10	1	1101[a]	0.00	0	ND			
69869-092	TCI-13-366-09-01	7	C	25.00		21-Mar-14	22-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
69869-093	TCI-13-366-09-02	7	T	25.00		21-Mar-14	22-Mar-14	250	0.10	10	10	1	1278[a]	0.00	0	ND			
69869-094	TCI-13-366-09-03	7	T	25.00		21-Mar-14	22-Mar-14	250	0.10	10	10	1	0	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	7	S				21-Mar-14						10223						
12562-B10	std. curve 0.01 ng/mL	7	S				21-Mar-14						4562						
12562-B8	std. curve 0.05 ng/mL	7	S				21-Mar-14						15321						
12562-B7	std. curve 0.15 ng/mL	7	S				21-Mar-14						45071						
12562-B8	std. curve 0.05 ng/mL	7	S				21-Mar-14						15159						
12562-B9	std. curve 0.03 ng/mL	7	S				21-Mar-14						9862						
12562-B7	std. curve 0.15 ng/mL	7	S				22-Mar-14						46131						
12562-B6	std. curve 0.3 ng/mL	7	S				22-Mar-14						87641						

1/x Weighting

Correlation (r): 0.99952482

Slope (m): 288698.1

Intercept (b): 1459.161

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03212014C.rdb

69869
TCI-13-366

Residue Results
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	Final Vol mL	Inj Vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	8	B				26-Mar-14						356[a]	0.00					
69869-095	TCI-13-366-10-01	8	C	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	860[a]	0.00	0	ND			
69869-096	TCI-13-366-10-01 + 0.020	8	F	25.00	0.020	26-Mar-14	26-Mar-14	250	0.10	10	5	1	3464	0.0149	0.0149	0.0149	0.000	0.0149	75
69869-097	TCI-13-366-10-01 + 0.50	8	F	25.00	0.50	26-Mar-14	26-Mar-14	250	0.10	10	5	5	16465	0.100	0.5	0.500	0.000	0.500	100
69869-098	TCI-13-366-10-02	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	1025[a]	0.00	0	ND			
69869-099	TCI-13-366-10-03	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	1142[a]	0.00	0	ND			
69869-100	TCI-13-366-11-01	8	C	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	1043[a]	0.00	0	ND			
69869-101	TCI-13-366-11-02	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	1208	0.0000674	0.0000674	<0.020			
69869-102	TCI-13-366-11-03	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	1417	0.00144	0.00144	<0.020			
69869-103	TCI-13-366-12-01	8	C	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	875[a]	0.00	0	ND			
69869-104	TCI-13-366-12-02	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	1025[a]	0.00	0	ND			
69869-105	TCI-13-366-12-03	8	T	25.00		26-Mar-14	26-Mar-14	250	0.10	10	5	1	723[a]	0.00	0	ND			
12562-B9	std. curve 0.03 ng/mL	8	S				26-Mar-14						6308						
12562-B10	std. curve 0.01 ng/mL	8	S				26-Mar-14						2660						
12562-B8	std. curve 0.05 ng/mL	8	S				26-Mar-14						8529						
12562-B11	std. curve 0.15 ng/mL	8	S				26-Mar-14						23773						
12562-B8	std. curve 0.05 ng/mL	8	S				26-Mar-14						8588						
12562-B6	std. curve 0.3 ng/mL	8	S				26-Mar-14						47334						

1/x Weighting
Correlation (r): 0.9992436
Slope (m): 152551.7
Intercept (b): 1197.773

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 03262014B.rdb

69869
TCI-13-366

Residue Results
Analyte: Clomazone
Matrix: Canola Seed

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	GPC vol mL	GPC mL aliq	Final vol mL	Inj vol uL	HPLC Dil Fact	Peak Resp[a]	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	10 long	B				19-May-14								2374	< 0					
69869-111	TCI-13-366-06-01 DF=100	10 long	C	25.00		15-May-14	19-May-14	250	80	15	4	2	5	100	2308	< 0	0	<0.020			
69869-112	TCI-13-366-06-01 + 0.020 DF=100	10 long	F	25.00	0.020	15-May-14	19-May-14	250	80	15	4	2	5	100	72092	0.144	0.0135	0.0135	0.000	0.0135	68
69869-113	TCI-13-366-06-01 + 0.50 DF=10,000	10 long	F	25.00	0.50	15-May-14	19-May-14	250	80	15	4	2	5	10000	22605	0.0343	0.3215625	0.322	0.000	0.322	64
69869-114	TCI-13-366-06-02 DF=100	10 long	T	25.00		15-May-14	19-May-14	250	80	15	4	2	5	100	2476	< 0	0	<0.020			
69869-115	TCI-13-366-06-03 DF=100	10 long	T	25.00		15-May-14	19-May-14	250	80	15	4	2	5	100	2605	< 0	0	<0.020			
12864-B9	0.03 ng/mL	10 long	S				19-May-14								20571						
12864-B8	0.01 ng/mL	10 long	S				19-May-14								10471						
12864-B6	0.05 ng/mL	10 long	S				19-May-14								36601						
12864-B4	0.3 ng/mL	10 long	S				19-May-14								138064						
12864-B5	0.15 ng/mL	10 long	S				19-May-14								74979						
12864-B4	0.3 ng/mL	10 long	S				19-May-14								141473						

1/x Weighting
Correlation (r): 0.9955
Slope (m): 451739.3
Intercept (b): 7116.446

[a]Concentration assumed to be zero when the peak response is below the y-intercept.

File Name: H 69869 05182014C

69869
TCI-13-366

Residue Results
(Confirmatory Transition)
Analyte: Clomazone
Matrix: Canola Seed

ABC Laboratories, Inc.

ABC ID	Sample Identification	Set #	Samp Type	Samp Wt g	Fort Level ppm	Extraction Date	Injection Date	mL solv	mL aliq	GPC vol mL	GPC mL aliq	Final vol mL	Inj vol uL	HPLC Dil Fact	Peak Resp	ng/mL found	ppm	reported ppm	ppm control	ppm corr for control	% Rec
Injection Solvent	Injection Solvent	10 long	B				19-May-14								0	No Peak					
69869-111	TCI-13-366-06-01 DF=100	10 long	C	25.00		15-May-14	19-May-14	250	80	15	4	2	5	100	0	No Peak	0	<0.020			
69869-112	TCI-13-366-06-01 + 0.020 DF=100	10 long	F	25.00	0.020	15-May-14	19-May-14	250	80	15	4	2	5	100	18233	0.150	0.0140625	0.0141	0.000	0.0141	70
69869-113	TCI-13-366-06-01 + 0.50 DF=10,000	10 long	F	25.00	0.50	15-May-14	19-May-14	250	80	15	4	2	5	10000	5645	0.0384	0.36000	0.360	0.000	0.360	72
69869-114	TCI-13-366-06-02 DF=100	10 long	T	25.00		15-May-14	19-May-14	250	80	15	4	2	5	100	0	No Peak	0	<0.020			
69869-115	TCI-13-366-06-03 DF=100	10 long	T	25.00		15-May-14	19-May-14	250	80	15	4	2	5	100	0	No Peak	0	<0.020			
12864-B9	0.03 ng/mL	10 long	S				19-May-14								5145						
12864-B8	0.01 ng/mL	10 long	S				19-May-14								2001						
12864-B6	0.05 ng/mL	10 long	S				19-May-14								8460						
12864-B4	0.3 ng/mL	10 long	S				19-May-14								33945						
12864-B5	0.15 ng/mL	10 long	S				19-May-14								19068						
12864-B4	0.3 ng/mL	10 long	S				19-May-14								34156						

1/x Weighting
Correlation (r): 0.9948
Slope (m): 113073.8
Intercept (b): 1298.89

File Name: H 69869 05182014C

APPENDIX V

Preparation of Standard Solutions

PREPARATION OF STANDARD SOLUTIONS

1 Stock Standard Solutions

Twenty-five (25.0) mg (corrected for purity) of clomazone analytical standard were accurately weighed, quantitatively transferred to a 25-mL volumetric flask, and brought to volume with acetonitrile. The resulting concentration of the solution was 1.0 mg/mL (1000-µg/mL). They were stored in the dark typically at 2 °C to 8 °C. When stored as indicated, these solutions have shown stability for at least two months. Solution stability is on-going as part of ABC Study Number 81233.

2 Intermediate/Fortification Standard Solutions

The following concentrations of intermediate/fortification standard solutions were stored in the dark typically at 2 °C to 8 °C. When stored as indicated, these solutions have shown stability for at least two months.

100 µg/mL: 1 mL of a 1000-µg/mL stock standard solution were transferred to a 10-mL volumetric flask and brought to volume with acetonitrile. The contents were mixed well.

10 µg/mL: 250 µL of a 1000-µg/mL stock standard solution were transferred to a 25-mL volumetric flask and brought to volume with acetonitrile. The contents were mixed well.

1.0 µg/mL: 2.5 mL of a 10-µg/mL standard solution were transferred to a 25-mL volumetric flask and brought to volume with acetonitrile. The contents were mixed well.

3 Calibration Standards Solutions

The following concentrations of intermediate and calibration standard solutions were stored in the dark typically at 2 °C to 8 °C.

100 ng/mL: 1.0 mL of a 1.0-µg/mL intermediate standard solution was transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

20 ng/mL: 0.20 mL of a 1.0-µg/mL intermediate standard solution was transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

5.0 ng/mL: 0.50 mL of a 100-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.30 ng/mL: 0.15 mL of a 20-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.15 ng/mL: 0.30 mL of a 5.0-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.050 ng/mL: 0.10 mL of a 5.0-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.030 ng/mL: 1.0 mL of a 0.30-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

0.010 ng/mL: 0.667 mL of a 0.15-ng/mL calibration standard solution were transferred to a to a 10-mL volumetric flask and brought to volume with acetonitrile:water (1:1, v/v). The contents were mixed well.

When stored as indicated, these solutions have shown stability for at least two months.

Appendix D - Protocol, Amendments, and Deviations

Deviations:

SOP deviations have been documented and are maintained in the study file.

The following protocol deviations were documented, but none adversely impacted the study.

Field Phase

TCI-13-366-02: The trial was originally initiated on June 3, 2013; however, the application rate was too low due to improper spray mixture preparation. The original trial site was abandoned and the trial restarted on June 11, 2013.

TCI-13-366-02: Phytotoxicity was not documented at the time it was observed as required by the protocol.

TCI-13-366-04, -09, -10, -11, and -12: The test substance used and amount remaining after the application was not documented in the field trial notebook log at each event; however, this information was documented after the fact using information from the receipt logs and test substance calculation raw data.

TCI-13-366-05: Due to an error with the lap timers, the pass time for the first pass was not captured. A verification pass time was completed after the second pass to verify the first pass time over the same path using the same settings (cruise control on ATV); therefore, this does not adversely impact the study.

TCI-13-366-11: Due to poor crop emergence and a poor stand, the treated samples weighed 0.20 and 0.25 kg rather than 0.5 kg as the protocol required. The crop that was present was healthy.

Analytical Phase

For method validation, the percent recovery for one 0.02 ppm sample was 66% and not within the range of 70-120% as the protocol required. The other protocol requirements, however, were met (mean recovery at each fortification level in the 70-110% range and the RSD at each level of ≤20%).

The Day 0 storage stability for canola seed was performed independently of the method validation and not in conjunction with method validation as the protocol specified. This does not adversely impact the study since Day 0 was setup along with the other storage samples as required.

For Set 8, the LOQ procedural recovery sample had a recovery of 68% and was not within the range of 70-120% as the protocol required.

The clomazone analytical standard (ABC reference MM-9053-00001) was stored in deep freezer storage at -10 to -25 °C; whereas, the Certificate of Analysis required < -20 °C. This did not adversely impact the study since the temperatures for all freezers used were typically between -15 and -20 °C, and any excursions from the freezer range allowed per ABC SOP were brief in nature and quickly rebounded. The integrity of the compound remained, as demonstrated by response throughout analysis.

A small study comparing the original method and the modified method was performed in this study without outlining this work in the protocol with an amendment. This did not adversely impact the study, but improved the study by providing data to support the changes implemented in the method modifications that were used for field sample analysis.

For the original and modified method comparison, the recoveries for the original method were 68 and 64% (primary ion transition). Since these data were not used for residue reporting, this does not adversely impact the study.

PROTOCOL AMENDMENT IV

I. STUDY IDENTIFICATION:

STUDY No.: TCI-13-366

TITLE: Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)

STUDY DIRECTOR: Sandra J. Carringer

II. CHANGE

D.4.0 ANALYTICAL PARAMETERS, *Storage Stability*, Page 16.

Storage Stability: The freezer stability of clomazone in canola seed initiated in this study (0-day analyses completed and two additional stability sets fortified) will be transferred to a new study for completion and reporting of the stability evaluation.

III. REASON FOR CHANGE:

The storage interval needed to support stability could not be assessed in this study and meet the report submission timelines. The additional freezer storage stability for clomazone in canola seed will be assessed and reported in a separate freezer stability study conducted as ABC Laboratories, Inc. (Study No. 81233).

IV. EFFECT ON STUDY:

Storage stability testing will not be concluded or reported in this study, but will be concluded and reported in a separate freezer stability study at ABC Laboratories, Inc. (Study No. 81233).

V. APPROVALS:

Study Director: _____ Date: 26 APR 2014
Sandra J. Carringer
The Carringers, Inc.

Testing Facility Management: _____ Date: 25-APR-2014
Richard D. Carringer, Ph.D.
The Carringers, Inc.

Sponsor's Representative: Faith A. Womack Date: 4-22-2014
Faith Womack
Cheminova, Inc.

PROTOCOL AMENDMENT III

I. STUDY IDENTIFICATION:

STUDY No.: TCI-13-366

TITLE: Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)

STUDY DIRECTOR: Sandra J. Carringer

II. CHANGE:

Section D.4.0 ANALYTICAL PARAMETERS, Method of Analysis, page 15, changes to the following:

Method of Analysis: Sample analysis will be based on the method found in Eurofins-GAB GmbH Report, Study Code 20061401/01-RVP, entitled "Validation of an Analytical Method for the Determination of Clomazone in Potatoes and Oilseed Rape".

Any method modifications that are necessary will be approved by the Study Director and documented in the study file. The analytical method used will be described in the final report and a copy of the method of analysis will be included in the final analytical report.

III. REASON FOR CHANGE:

The Eurofins method is specific for oilseed rape/canola.

IV. EFFECT ON STUDY:

The change in this amendment does not adversely impact the study.

V. APPROVALS:

Study Director: _____ Date: 07 Feb 2014
Sandra J. Carringer
The Carringers, Inc.

Testing Facility Management: _____ Date: 07-FEB-2014
Richard D. Carringer, Ph.D.
The Carringers, Inc.

Sponsor's Representative: Faith A. Womack Date: February 4, 2014
Faith Womack
Cheminova, Inc.

PROTOCOL AMENDMENT II

I. STUDY IDENTIFICATION:

STUDY No.: TCI-13-366

TITLE: Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)

STUDY DIRECTOR: Sandra J. Carringer

II. CHANGE:

Section C.4.0 PROCESSED FRACTION SAMPLE STORAGE AND SHIPPING, Page 13 and Section D.1.0 PRINCIPAL ANALYTICAL INVESTIGATOR AND ANALYTICAL LABORATORY, page 14.

The Principal Analytical Investigator and analytical laboratory changes to the following:

Carol Rodgers – Principal Analytical Investigator
ABC Laboratories, Inc.
7200 E. ABC Lane
Columbia MO 65202
Tel: 573-777-6054
Fax: 573-777-6033
e-mail: rodgersc@abclabs.com

III. REASON FOR CHANGE:

Morse Laboratories, LLC is closing, and the analytical phase is transferring to ABC Laboratories. The original Principal Analytical Investigator will not be moving to ABC Laboratories.

IV. EFFECT ON STUDY:

The change in this amendment does not adversely impact the study.

V. APPROVALS:

Study Director: _____ Date: 01 Nov 2013
Sandra J. Carringer
The Carringers, Inc.

Testing Facility Management: _____ Date: 01-Nov-2013
Richard D. Carringer, Ph.D.
The Carringers, Inc.

Sponsor's Representative: Faith A. Womack Date: October 31, 2013
Faith Womack
Cheminova, Inc.

PROTOCOL AMENDMENT I

I. STUDY IDENTIFICATION:

STUDY No.: TCI-13-366

TITLE: Magnitude of the Residue of Clomazone in/on Canola Raw Agricultural and Processed Commodities Following One Preemergence Application of Clomazone 360 g/L CS (2013)

STUDY DIRECTOR: Sandra J. Carringer

II. CHANGE:

Section B.1.0 PRINCIPAL FIELD INVESTIGATORS AND TRIAL IDENTIFICATIONS, Pages 6-7. The Principal Field Investigator for Trials -04, -09, -10, -11, and -12 changes to the following:

Principal Field Investigator	Crop	EPA Region	Trial Identification
Dean Ngombe ICMS 334 Packham Ave. Saskatoon, SK Canada S7N 2T1 Tel: 306-956-3855 (Office)/306-260-3061 (Cell) Fax: 306-956-3856 e-mail: ngombe@icms-inc.com	14	SK	TCI-13-366-04 RAC/Decline Trial
Dean Ngombe ICMS 334 Packham Ave. Saskatoon, SK Canada S7N 2T1 Tel: 306-956-3855 (Office)/306-260-3061 (Cell) Fax: 306-956-3856 e-mail: ngombe@icms-inc.com	14	SK	TCI-13-366-09 TCI-13-366-10 TCI-13-366-11 TCI-13-366-12 RAC Trials

III. REASON FOR CHANGE:

This change is made at the request of the field facility management.

IV. EFFECT ON STUDY:

The change in this amendment does not adversely impact the study.

PROTOCOL AMENDMENT I

V. APPROVALS:

Study Director: _____ Date: 21 Aug 2013
Sandra J. Carringer
The Carringers, Inc.

Testing Facility Management: _____ Date: 21-AUG-2013
Richard D. Carringer, Ph.D.
The Carringers, Inc.

Sponsor's Representative: Faith A. Womack Date: August 21, 2013
Faith Womack
Cheminova, Inc.

RESIDUE STUDY PROTOCOL
STUDY NO. TCI-13-366

STUDY TITLE

Magnitude of the Residue of Clomazone in/on
Canola Raw Agricultural and Processed Commodities Following
One Preemergence Application of Clomazone 360 g/L CS (2013)

RESIDUE CHEMISTRY TEST GUIDELINE

Canadian PMRA Regulatory Directive DIR98-02 and 2010-05
SANCO 3029/99 rev. 4. 11/07/00

STUDY DIRECTOR/TESTING FACILITY
Sandra J. Carringer
The Carringers, Inc.
1003 Palace Ct.
Apex, NC 27502
Tel: 919-387-0825
Fax: 919-387-4161
e-mail: scarringer@carringers.com

TESTING FACILITY MANAGEMENT
Richard D. Carringer, Ph.D.
The Carringers, Inc.
1003 Palace Ct.
Apex, NC 27502
Tel: 919-387-0825
Fax: 919-387-4161
e-mail: rcarringer@carringers.com

SPONSOR'S REPRESENTATIVE
Faith Womack
Cheminova, Inc.
1600 Wilson Boulevard, Suite 700
Arlington, VA 22209
Tel: 703-373-8883 (ext 4)
Fax: 703-373-8887
e-mail: faith.womack@cheminova.com

SPONSOR
Cheminova A/S
P.O. Box 9
DK-7620, Lemvig
DENMARK

PROTOCOL APPROVAL:

Sandra J. Carringer — Date: May 1, 2013
Study Director, The Carringers, Inc.

PROTOCOL ACCEPTANCE:

Faith A. Womack — Date: April 30, 2013
Faith Womack
Sponsor's Representative, Cheminova, Inc.

PROTOCOL ACKNOWLEDGEMENT:

Richard D. Carringer — Date: 01-MAY-2013
Richard D. Carringer, Ph.D.
Testing Facility Management, The Carringers, Inc.

Ralph P. Rose — Date: 01 May 2013
Ralph P. Rose
Quality Assurance Unit, The Carringers, Inc.

TABLE OF CONTENTS

A. BACKGROUND INFORMATION

1.0 PURPOSE OF THE STUDY

This study is being conducted to determine the magnitude and decline of residues of clomazone in or on canola raw agricultural commodities (RAC) following one preemergence application of Clomazone 360 g/L CS at 0.42 kg ai/ha. The magnitude of residues of clomazone in or on canola processed commodities (PC) will also be determined following one preemergence application at an exaggerated rate (a 3× or 5× rate if possible) if residues are found above the limit of quantitation (LOQ) following the exaggerated application rate or as directed by the Study Director. The results from this study may be used to establish Maximum Residue Limits for clomazone in/on canola raw agricultural and processed commodities in Canada.

2.0 TEST SYSTEMS

Field Phase: Twelve canola trials will be established in Canada in PMRA Zones/Regions 5 (one trial), 7 (two trials) and 14 (nine trials). The canola will be grown on soil types and under cultural practices typical for commercial canola production.

Processing Phase: One canola test site will be established in PMRA Zone/Region 14 for processing. The variety will be one typical for the area. Canola will be processed following commercial processing procedures to generate meal and refined oil only if residues are found above the LOQ in the seed at the processing trial.

Analytical Phase: Samples of canola seed collected from untreated control plots and from plots treated with Clomazone 360 g/L CS will be analyzed at the analytical laboratory for residues of clomazone. Samples of canola meal and refined oil will also be analyzed for residues of clomazone if the processing phase is conducted.

3.0 JUSTIFICATION FOR SELECTION OF THE TEST SYSTEMS

Selection of the 12 trial sites in PMRA Regions 5, 7, and 14 will fulfill the requirements for canola as specified in PMRA Regulatory Directive DIR98-02 and 2010-05.

4.0 TEST SUBSTANCE NOMENCLATURE

Common Name	Clomazone
Company Experimental Name	6710
IUPAC Name	2-(2-chlorobenzyl)-4,4-dimethyl-1,2-oxazolidin-3-one or 2-(2-chlorobenzyl)-4,4-dimethylisoxazolidin-3-one
CAS name	2-[(2-chlorophenyl)methyl]-4,4-dimethyl-3-isoxazolidinone
CAS Number	81777-89-1
End-use Product	Clomazone 360 g/L CS
Formulation Type	capsule suspension (CS)
Nominal A.I. Content	360 g ai/L
Lot Number	0001023789
GLP Certified Active Concentration	355 g ai/L **(use for spray mix calculations)**
Date of Analysis	21-Mar-12
Expiration Date	21-Mar-14

5.0 STUDY SCHEDULE

Proposed Experimental Start Date (1st field application): May 2013
Proposed Analytical Phase Initiation Date: September 2013
Proposed Analytical Termination Date: March 2014
Proposed Study Completion Date: June 2014

6.0 GOOD LABORATORY PRACTICE COMPLIANCE

This study will be conducted in accordance with EPA Good Laboratory Practice (GLP) Standards for FIFRA (Federal Insecticide, Fungicide and Rodenticide Act) as defined in 40 CFR Part 160 and/or OECD Principles of Good Laboratory Practice. The Standard Operating Procedures (SOPs) of the field, processing, and analytical facilities for specific study procedures must meet the requirements of 40 CFR, Part 160, FIFRA GLP Standards and/or OECD Principles of Good Laboratory Practice. A statement of compliance with EPA/FIFRA and/or OECD GLP Standards will be included in each field trial notebook, processing study files, and analytical study files. The final report will contain a statement of compliance with EPA/FIFRA and/or OECD GLP Standards signed by the Study Director and the Sponsor.

Weather data collection, soil characterization, general land preparation (i.e., tillage, mowing, irrigation, etc.), application of maintenance chemicals during the test, and documentation of chemical history do not require generation under GLPs as long as it is clearly stated as such.

7.0 AMENDMENTS TO AND DEVIATIONS FROM THIS PROTOCOL

Planned changes to the protocol will be documented in the form of protocol amendments, which will be signed by the Study Director and the Sponsor's Representative. Any deviation from the protocol will be documented, stating the reasons for the action(s) and the likely consequences of the action(s). Deviations will be reported to the Study Director so that corrective action can be recommended and documented. The Study Director will sign all protocol deviations and report to the Sponsor's Representative all deviations that adversely impact the study.

8.0 DATA REPORTING AND RETENTION

The Principal Field Investigators (PFI) will complete a field trial notebook, which will be prepared by The Carringers, Inc. (TCI). The processing and analytical facilities will prepare reports that summarize the processing and analytical phases of the study. The testing facility, TCI, will prepare a final report that presents and summarizes the field, processing, and analytical results. The final report will be prepared in accordance with PR Notice 2011-3 (or latest version) or comparable PMRA guideline document, and will contain all information required by, and will be handled per, 40 CFR § 160.185 and/or ENV/MC/CHEM(98)17. TCI's Quality Assurance Unit (QAU) will inspect the final report before issuing the document. Any corrections or additions to the final report will be issued in accordance with 40 CFR § 160.185 (c) and/or ENV/MC/CHEM(98)17 § 9.1.4.

Upon completion of this study, all study records (original raw data), protocol and amendments and the final study report and any amendments, will be transferred to temporary archival storage at TCI and then transferred to the Sponsor's permanent archival facility (to be determined and outlined in the study report). Alternately, data may be transferred directly to an alternate location approved by the Sponsor and as directed by the Study Director.

9.0 QUALITY ASSURANCE

TCI's QAU will have overall responsibility for monitoring the field, processing, and analytical phases of the study. Reports will be submitted to the Study Director and the Testing Facility Management. TCI's QAU will ensure that the individual facilities, equipment, personnel, methods, practices, records, and controls, are designed and function in conformance with 40 CFR Part 160 and/or ENV/MC/CHEM(98)17, the protocol and relevant SOPs. TCI QAU will conduct, as necessary, inspections of the field test sites, processing facility, and analytical facility and prepare written reports of its findings.

The QAU of each field, processing, and analytical testing facility will conduct GLP-required inspections and audits at intervals adequate to ensure the integrity of the study. The field, processing, and analytical test site's QAU will prepare a report of each Quality Assurance inspection, listing details of the operation(s) observed, as well as the information specified by 40 CFR §160.35 (b)(3)(54 FR 34070, 17 August 1989) and/or ENV/MC/CHEM(98)17. Any problems that are likely to affect the integrity of the study will be brought to the attention of the Study Director immediately by telephone, fax, or e-mail. QAU reports will be sent to the Study Director and Testing Facility Management. Inspection dates will be recorded in the Quality Assurance Certification Statement page of the Field Trial Notebook or reports.

10.0 CONFIDENTIALITY

All information regarding the test substance, samples, test plots, processing, sample analysis, and analytical results must be kept strictly confidential. Test plots are to be considered as restricted access areas with measures taken to exclude unauthorized persons from the test area. No plot designations in or around the test area will be made that identify the treatments, chemical, or Sponsor. No raw data, worksheets, data or information summaries, reports, or other information related to this study may be revealed or released to any third party without prior notification and authorization of the Sponsor.

11.0 STATISTICAL METHODS

No statistical analysis of data will be required for the field, processing, or analytical portions of the study other than mean, standard deviation and regression analysis. Additional statistical methods may be used in generation of data for the final study report. These will be described and noted in the report.

B. FIELD PHASE

1.0 PRINCIPAL FIELD INVESTIGATORS AND TRIAL IDENTIFICATIONS

Principal Field Investigator	PMRA Zones/Region	Province	Trial Identification
Kelly Tiller ICMS, Inc. Mail: Box 67, Station Main Shipping: 2375 Saskatchewan Ave. E. Portage la Prairie, MB Canada R1N 3B2 Tel: 204-857-6609 (Office)/204-871-5031 (Cell) Fax: 204-239-4478 E-mail: tiller@icms-inc.com	5	MB	TCI-13-366-01 RAC Trial
Greg Whittington ICMS, Inc. 334 Packham Ave. Saskatoon, SK Canada S7N 2T1 Tel: 306-956-3855 (Office)/306-220-5696 (Cell) Fax: 306-956-3856 E-mail: whittington@icms-inc.com	7	SK	TCI-13-366-02 TCI-13-366-03 RAC Trial
Brittany Johnson ICMS, Inc. 334 Packham Ave. Saskatoon, SK Canada S7N 2T1 Tel: 306-956-3855 (Office)/306-260-3061 (Cell) Fax: 306-956-3856 E-mail: johnson@icms-inc.com	14	SK	TCI-13-366-04 RAC/Decline Trial
Taryn Williams ICMS, Inc. Box 3270, 54474 Range Road 215 Fort Saskatchewan, AB Canada T8L 2T2 Tel: 780-992-7983 (Office) / 780-722-2044 (Cell) Fax: 780-992-8499 E-mail: williams@icms-inc.com	14	AB	TCI-13-366-05 RAC/Decline Trial
Kelly Tiller ICMS, Inc. Mail: Box 67, Station Main Shipping: 2375 Saskatchewan Ave. E. Portage la Prairie, MB Canada R1N 3B2 Tel: 204-857-6609 (Office)/204-871-5031 (Cell) Fax: 204-239-4478 E-mail: tiller@icms-inc.com	14	MB	TCI-13-366-06 RAC/Processing Trial

Principal Field Investigator	PMRA Zones/Region	Province	Trial Identification
Kelly Tiller ICMS, Inc. Mail: Box 67, Station Main Shipping: 2375 Saskatchewan Ave. E. Portage la Prairie, MB Canada R1N 3B2 Tel: 204-857-6609 (Office)/204-871-5031 (Cell) Fax: 204-239-4478 E-mail: tiller@icms-inc.com	14	MB	TCI-13-366-07 TCI-13-366-08 RAC Trials
Brittany Johnson ICMS, Inc. 334 Packham Ave. Saskatoon, SK Canada S7N 2T1 Tel: 306-956-3855 (Office)/306-260-3061 (Cell) Fax: 306-956-3856 E-mail: johnson@icms-inc.com	14	SK	TCI-13-366-09 TCI-13-366-10 TCI-13-366-11 TCI-13-366-12 RAC Trials

2.0 TEST SUBSTANCE RECEIPT, STORAGE, AND CONTAINER HANDLING

The following data should be documented for the test substance: lot/batch numbers(s) received; expiration date, if provided; condition upon receipt; date(s) of receipt; amount of test substance received, used, and remaining after the last application; description of storage location; and the amount and distribution of unused test substance.

The test substance will be stored under label conditions in a temperature monitored pesticide storage area adequate to preserve the identity, strength, purity, and stability of the test substance.

It is not necessary to return remaining test substance. The remaining test substance may be maintained or disposed of according to state and local regulations at trial completion. Retain the test substance containers until notified by the Study Director that you may discard the containers. The empty test substance containers do not need to be stored in an environmentally controlled area.

3.0 TEST SITE SELECTION AND CULTURAL PRACTICES

Site Selection: Each test site will be typical for the intended use of Clomazone 360 g/L CS on canola and will be representative of commercial canola producing areas for each selected zone/region of Canada with respect to crop variety, cultural practices, soil type, and climatic conditions.

One or more of the following criteria should be employed for trials conducted in the same state/province and/or PMRA region by the same Principal Field Investigator to be considered unique:

Different varieties of the same crop
Different soil types (for seed treatment or soil-applied treatments only)
Geographic separation (≥ 40 kilometers and/or different counties)
Temporal separation (significantly different trial periods; e.g., Spring vs. Fall)

Pesticide Use History: For each test site, chemical and cropping data will be made available for the previous two years. **Only those areas where clomazone has not been used in the 1 year prior to this study will be designated as test sites for the field phase of this study.**

Soil Characterization: Soil characterization data are not required for the test site; however, at a minimum the soil type (non-GLP) will be recorded in the field notebook. The percent organic matter, pH, and cation exchange capacity of the soil should be provided if available.

Cultural Practices: The canola will be grown per typical agronomic practices. Adequate and timely irrigation for normal plant growth and development should be maintained throughout the trial period if typical for the region. All cultural practices, e.g. cultivation operations, irrigation (methods, dates, source of water, and approximate amounts), and fertilization, will be documented. Provide cultural practices used prior to the start of the trial if possible.

Maintenance Pesticide Application: During the current growing season, test plots will not be treated with any maintenance pesticides that contain clomazone. All maintenance pesticides will be documented.

Weather: A summary of the trial period and historical weather data should be provided for the months associated with the trial period (first application to final sampling). Data should be provided from the nearest reliable weather station within 40 kilometers of the trial site (if possible). The source of the data, the distance from the test site, and the units of measurement must be clearly identified.

4.0 EXPERIMENTAL DESIGN

Plot Number and Sizes:

RAC Trials and RAC Decline Trials: There will be two plots (Treatments 1 and 2) for these trials. Treatment 1 is the untreated control plot. Treatment 2 is the 0.42 kg ai/ha rate treated plot.

RAC/PC Trial (Trial -06): There will be four plots (Treatments 1, 2, 3, and 4) for this trial. Treatment 1 is the untreated control plot. Treatment 2 is the 1× rate treated plot at 0.42 kg ai/ha. Treatment 3 is the 3× rate treated plot at 1.26 kg ai/ha. Treatment 4 is the 5× rate treated plot at 2.1 kg ai/ha.

The size of the control and treated plots will be determined by the PFI. The plots must be large enough to ensure fulfillment of minimum sampling and sample size requirements. The suggested treated plot size is a minimum of 90 m^2 for trials collecting RAC samples and larger for Trials -04 and -05 where decline samples will be collected and for Trial -06 where bulk samples for processing will be collected. <u>For processing Trial -06, all treated plots (Trt 2, Trt 3, and Trt 4) should be large enough to collect the 30 kg for processing in the event that the exaggerated rates cause crop injury.</u>

Plot Separation: The distance between the nearest treated and untreated plots should be at least 30 meters to ensure that the untreated plots do not become contaminated during the

conduct of the study. The distance between the treated plots should be at least 10 meters. Where possible, the control plot should be upslope and upwind of the treated plot.

Test Site Diagram: A detailed plot diagram is required and will include the following: plot locations, plot orientations, plot dimensions, control plot location relative to treated plot, distance between plots (buffer zones), topographic slope (direction and percent), direction of prevailing wind, plot orientation with respect to north (north arrow), permanent markers (physical markers or GPS coordinates), and any other physical features in the landscape that define the test site. Provide GPS coordinates if available. The location of the permanent markers (physical markers or GPS coordinates) should be documented on the plot map, with plot corners identified in the diagram with sufficient detail such that plots may be re-located with a reasonable degree of accuracy.

Plot Identification: The plots should be clearly identified, with a unique number (such as Study No. and Trt No.), by labeled stakes or other means.

5.0 APPLICATION OF TEST SUBSTANCE

Treatment Summary:

Trt. No.	Application Type	Rate kg ai/ha[1]	Spray Volume l/ha[2]	Application Timing	Spray Additive
1	--	Control	--	--	--
2	Soil-applied Broadcast	0.42	47-189[3]	Preemergence	None
3[4]	Soil-applied Broadcast	1.26	47-189[3]	Preemergence	None
4[4]	Soil-applied Broadcast	2.1	47-189[3]	Preemergence	None

[1]The application rate may vary ±5%.
[2]The spray volume may vary ±10%.
[3]Trials TCI-13-366-02, -05, and -07 must target 47 l/ha spray volume.
[4]Trial TCI-13-366-06 only.

Number and Timing of Applications: Each treated plot will receive one (1) application. The preemergence application is a soil-applied broadcast spray application that will be made after planting but prior to crop emergence. A record of test material usage will be recorded and provided in the raw data.

Phytotoxicity: Phytotoxicity that occurs during the study must be documented. None is expected. No documentation of phytotoxicity will be accepted as indicating none was present.

Spray Additives: None.

Calibration: The applications will be made using commercial or research broadcast ground spray equipment. Equipment will be cleaned and calibrated prior to the application (within one calendar day). Calibration will be based on the speed and distance traveled and the total spray output at a given operating speed and pressure over a measured amount of time (i.e., seconds or minutes). A complete description of the calibration methods, application methods, equipment, and application dates will be included in the raw data.

Application Verification: To verify the application rate, the direction and time of each sprayer pass over the treated area will be documented. It is intended that the application

be within ±5% of the target rate. If the application variance exceeds ±5% of the target rate, the Study Director or his designee will decide on the acceptability of the rate at the time of application.

Environmental and Crop Data at Application: Do not make applications when the wind direction would place the untreated control plot downwind of the treated plot or when the wind speed is in excess of 8 km/hr (if possible). At the time of the application, air temperature, relative humidity, wind speed and direction, and soil temperature (5 and 10 cm) at the test site will be measured and recorded.

6.0 SAMPLING

Sampling Summary:

RAC Trials – (Trials -01, -02, -03, -07, -08, -09, -10, -11, -12)

Trt No.	Sample No.[1]	Matrix	Timing	Sample Size
1 (UTC)	01	Canola Seed	Normal Crop Maturity	0.5 kg
2	02	Canola Seed	Normal Crop Maturity	0.5 kg
2	03	Canola Seed	Normal Crop Maturity	0.5 kg

[1]The trial number will precede the sample number listed above to make each sample identification unique (TCI-13-366-01-01, TCI-13-366-01-02, etc.).

RAC/Decline Trials – (Trials -04 and -05)

Trt No.	Sample No.[1]	Matrix	Timing	Sample Size
1 (UTC)	01	Canola Seed	Normal Crop Maturity	0.5 kg
2	02-03	Canola Seed	Normal Crop Maturity	0.5 kg
2	04-05	Canola Seed	3 Days After Normal Crop Maturity	0.5 kg
2	06-07	Canola Seed	7 Days After Normal Crop Maturity	0.5 kg
2	08-09	Canola Seed	14 Days After Normal Crop Maturity	0.5 kg
2	10-11	Canola Seed	21 Days After Normal Crop Maturity	0.5 kg

[1]The trial number will precede the sample number listed above to make each sample identification unique (TCI-13-366-04-01, TCI-13-366-04-02, etc.).

RAC/PC Trial – (Trial -06)

Trt No.	Sample No.[1]	Matrix	Timing	Sample Size
1 (UTC)	01	Canola Seed	Normal Crop Maturity	0.5 kg
2	02-03	Canola Seed	Normal Crop Maturity	0.5 kg
3 or 4[2]	04-05	Canola Seed	Normal Crop Maturity	0.5 kg
1 (UTC)	06	Canola Seed	Normal Crop Maturity	30 kg
2, 3, or 4[2]	07	Canola Seed	Normal Crop Maturity	30 kg

[1]The trial number will precede the sample number listed above to make each sample identification unique (TCI-13-366-06-01, TCI-13-366-06-02, etc.).

[2]Collect samples from the 5× rate treated plot (Treatment 4), if possible. If there is crop phytotoxicity in the 5× rate plot to the extent that sampling is not possible, the 3× rate plot (Treatment 3) should be sampled. If there is crop phytotoxicity in the 3× rate plot to the extent that sampling is not possible, the 1× rate plot (Treatment 2) should be sampled for processing. If Treatment 2 is used for processing, samples -04 and -05 may not be available for collection.

Schedule: RAC samples and samples for processing will be collected from the untreated and treated plots at normal crop maturity. At decline trials -04 and -05 treated seed samples will also be collected 3 (±1), 7 (±1), 14 (±2) and 21 (±2) days after normal harvest (DANH).

Sample Size: RAC Samples: Collect at least 0.5 kg per sample. Processing Samples (Samples 06 and 07): Collect at least 30 kg per sample.

Number of Samples: See tables above.

Sampling Procedures: All samples should be collected without bias and should be commercially acceptable. All equipment used for sampling will be cleaned prior to its use. The untreated control plot will be sampled first followed by the low rate (Trt 2) and then high rate (Trt 3 or Trt 4 if applicable) treated plot (at processing trial -06 only).

Samples may be collected either by swathing/cutting canola at 40-60% seed color change and then threshing the cut material when the plants and seed have dried down, or collected by straight-cutting (direct harvesting) mature plants. Only one harvest method may be used per trial.

When swathing/cutting is used (recommended for the Decline trials) the date of cutting will follow the decline pattern (i.e. normal maturity, 3, 7, 14, 21 days after normal maturity), and samples may be threshed and frozen as conditions permit. Samples cut on multiple dates may be threshed on the same day, but field drying time (time between cutting and threshing) should not exceed 14 days for any given cut sample. Thresh samples in order from earliest cut date to latest cut date. Each sample obtained in this manner must be collected by cutting plants from at least 12 separate areas of each plot.

In the case of samples collected by straight-cutting, duplicate treated samples must be collected by making two separate passes through the plot or by alternating sample buckets under the combine discharge chute (at least 12x each) as the combine moves through the plot. **Each sample will be collected from at least twelve (12) separate areas of each plot.**

Do not sample from the edges or ends of the treated plot.

Post-harvest Sample Handling: Collect, transport, and store control and treated samples using all necessary precautions to prevent contamination. RAC samples will be placed into labeled plastic-lined cloth residue sample bags (or other approved bag) (with the preprinted sample labels provided). Alternate bags/boxes are acceptable for the large processing samples. Keep all samples cool until they can be transferred to a freezer. Freeze all samples within 4 hours after they are collected. Once frozen, the samples must not be allowed to thaw at any time prior to preparation for analysis. Sample maintenance, storage, and chain of custody records will be retained with the raw data.

Sample Shipping: RAC samples for residue analysis will be shipped frozen and with proper chain of custody documentation via ACDS freezer trucks, on dry ice via overnight carrier (only if approved by the Study Director), or via field test site personnel to the following:

Jeri Willoh – Principal Analytical Investigator
Morse Laboratories, LLC
1525 Fulton Avenue
Sacramento, CA 95825
Tel: 916-481-3141/ Fax: 916-481-2959
e-mail: willohj@morselabs.com

It is acceptable to pack control and treated samples in the same box(es), as long as there is adequate separation to avoid contamination. If overnight carrier is used, use insulated shipping boxes containing at a minimum an approximate ratio of 1 kg of dry ice: 0.5 kg of sample.

Samples for processing will be shipped frozen under chain of custody via ACDS freezer trucks to the following processor:

Dick Dusek–Principal Processing Investigator
GLP Technologies
22723 State Highway 6 South
Navasota, TX 77868
Tel: 936-825-2184 / Fax: 936-825-7929
e-mail: ddusek@glptech.net

Field personnel will notify the laboratory by telephone, fax, or e-mail when samples are shipped. An exact copy of the sample shipping pages will also be faxed or e-mailed to the Study Director when samples are shipped. Documentation of sample shipment will be retained with the raw data.

7.0 CROP DESTRUCTION

Crop destruction is required for this study. Treated crop remaining after collection of all samples is to be destroyed to prevent its potential consumption by humans or livestock. Document the date and method of crop destruction in the field trial notebook.

C. PROCESSING PHASE

1.0 PRINCIPAL PROCESSING INVESTIGATOR AND PROCESSING LABORATORY

Dick Dusek–Principal Processing Investigator
GLP Technologies
22723 State Highway 6 South
Navasota, TX 77868
Tel: 936-825-2184 / Fax: 936-825-7929
e-mail: ddusek@glptech.net

2.0 PROCESSING

Samples will be stored frozen until the Study Director authorizes the processing phase. **If residues are not found in the 5× rate treated samples, the processing phase may not be conducted.**

If processing is authorized, samples will be processed according to current facility SOPs which simulate commercial practices. Details of the procedure used to process the canola will be included in a processing report.

3.0 PROCESSED FRACTIONS

Samples to be collected at the processing facility include the following:

Trt No.	RAC Sample No.[1]	Matrix	Processed Fraction Sample No.	Sample Size[2]
1	06	Whole Seed[3]	08	0.5 kg
1	06	Meal	09	1 kg
1	06	Refined Oil	10	~800 g
2, 3, or 4[4]	07	Whole Seed[3]	11	0.5 kg
2, 3, or 4[4]	07	Whole Seed[3]	12	0.5 kg
2, 3, or 4[4]	07	Meal	13	1 kg
2, 3, or 4[4]	07	Meal	14	1 kg
2, 3, or 4[4]	07	Refined Oil	15	~800 g
2, 3, or 4[4]	07	Refined Oil	16	~800 g

[1]The trial number will precede the sample number listed above to make each sample identification unique (TCI-13-366-06-08, TCI-13-366-06-09, etc.).

[2]The sample sizes listed in the table above are minimum size/weight requirements to be collected. Sample sizes greater than the amounts listed above will not be considered deviations to the study protocol.

[3]Seed samples will be collected immediately prior to sample processing and placed in freezer storage.

[4]Samples will be collected from the 5× rate treated plot (Treatment 4), if possible. If there is crop phytotoxicity in the 5× rate plot to the extent that sampling is not possible, the 3× rate plot (Treatment 3) will be sampled. If there is crop phytotoxicity in the 3× rate plot to the extent that sampling is not possible, the 1× rate plot (Treatment 2) will be sampled. The actual treatment received and processed will be documented in the study files and report.

4.0 PROCESSED FRACTION SAMPLE STORAGE AND SHIPPING

The RAC and processed fraction samples will be packaged, labeled, frozen, and maintained under frozen conditions during storage prior to shipment at the processor facilities according to the appropriate SOP. Frozen RAC and processed fraction samples will be properly packaged and shipped to the following:

Jeri Willoh – Principal Analytical Investigator
Morse Laboratories, LLC
1525 Fulton Avenue
Sacramento, CA 95825
Tel: 916-481-3141/ Fax: 916-481-2959
e-mail: willohj@morselabs.com

Shipping should be done according to the processing facilities shipping SOP or other approved shipping SOP. Briefly, samples may be shipped by freezer truck such as ACDS, Inc., or by Federal Express with overnight delivery (if directed by the Study Director). If overnight carrier is used, use insulated shipping boxes containing at a minimum an approximate ratio of 1 kg of dry ice: 0.5 kg of sample. The laboratory's sample receiving and preparation manager should be notified of the shipping date as soon as possible after shipment.

5.0 PROCESSING REPORT

The processing report will include, but is not limited to the following:

1. A title page
2. A table of contents
3. A GLP compliance statement
4. QA Statement of Inspection
5. A short description of sample handling, storage, etc.
6. A description of the processing procedures and comparisons to commercial processes
7. A material balance of the crop processed and yield of fractions
8. A description of any deviations that occurred

The original, signed final processing report will be submitted to the Study Director.

6.0 RAW DATA RETENTION

The processing facility will maintain a copy of the signed processing report, protocol and amendments, and all documents (letters, memos, notes, etc.) pertaining to the study. Upon completion of the processing phase, the original or verified copy of relevant processing data will be transferred to the testing facility, TCI. Those data and records include, but are not limited to the following:

1. Original laboratory notebooks and/or raw data forms
2. Copies of the SOPs used in processing
3. Original receipt and chain of custody for samples
4. Certified copies of sample storage records
5. Original work sheets and calibration records
6. List of applicable SOPs used during the processing phase

D. ANALYTICAL PHASE

1.0 PRINCIPAL ANALYTICAL INVESTIGATOR AND ANALYTICAL LABORATORY

Jeri Willoh – Principal Analytical Investigator
Morse Laboratories, LLC
1525 Fulton Avenue
Sacramento, CA 95825
Tel: 916-481-3141/ Fax: 916-481-2959
e-mail: willohj@morselabs.com

2.0 TEST SYSTEM INFORMATION

Test System/Crop: Canola

Substrate(s) To Be Analyzed: Canola seed will be analyzed from all field sites. Meal and refined oil will be analyzed if the processing phase is conducted.

Sample Preparation Procedure for Substrates: Each sample, as needed, will be prepared for analysis by grinding in the presence of dry ice or liquid nitrogen.

Sample Storage and Handling: Samples will be kept frozen (< -10 °C) until they are to be prepared and analyzed.

3.0 REFERENCE SUBSTANCE

Reference Substances (Analytical Standard) Identification: The reference substance used in this study will be an analytical grade of the compound clomazone (CAS No. 81777-89-1). The reference substance lot number and certificate of standard purity will be included in the study raw data file. The reference substance will be obtained from the sponsor or obtained from a commercial source.

Reference Substance Solubility: The method utilized for the analysis of samples in conjunction with this study will be verified over a range of fortification levels (µg of material added) anticipated to encompass field-incurred residues. Providing all sample analyses are conducted within the appropriate range, solubility considerations are not an issue for the purposes of this study.

Reference Substance Stability: The analytical standard label information will include an expiration date, and the material will not be used beyond that date. Fresh standard solutions will be prepared as specified in the analytical method. If data demonstrating the stability of analytical standard solutions are not available, these data will be generated by comparing the analytical response of aged standard solutions to those of freshly prepared standard solutions.

Reference Substance Handling: Analytical standard will be stored in separate airtight containers as stated on the label or certificate of analysis when not in use.

4.0 ANALYTICAL PARAMETERS

Analyte to be Determined: Clomazone

Method of Analysis: Sample analysis will be based on the method found in FMC Report No. P-2640 entitled "Residue Analytical Method for the Determination of Clomazone in/on Crop and Processed Part Matrices of Corn, Cottonseed, Soybean, and Tobacco". Any method modifications that are necessary will be approved by the Study Director and documented in the study file. The analytical method used will be described in the final report and a copy of the method of analysis will be included in the final analytical report.

Limit of Quantitation: Target = 0.02 ppm. The actual LOQ will be documented in the final study report.

Method Validation: The method of analysis will be validated on canola seed before any treated canola seed samples are analyzed. **Validation for canola meal and canola refined oil will be conducted only if the processing phase is conducted.**

Validation for clomazone for each matrix will consist of analyzing two untreated control samples, five untreated control samples fortified at the limit of quantification (LOQ), and five untreated control samples fortified at 25× the LOQ. Acceptable individual recoveries will be in the range of 70 to 120% with the mean recovery at each fortification level in the range of 70 to 110% and an RSD at each level of ≤20%. Control sample residues should not exceed 30% of the LOQ.

Routine Analysis: Samples will be analyzed in groups or sets consisting of the number of treated field samples which can effectively be managed through the analytical procedure at one time plus at least one control sample and at least one fortified recovery sample. Fortification levels will range from the LOQ to a level that encompasses the highest residues found. Acceptable recoveries will be in the range of 70 to 120%.

Storage Stability: Storage stability will be determined on canola seed to further support freezer storage stability of residues. If the processing phase is conducted, the processed commodities (meal and refined oil) will be analyzed within 30 days of processing if possible. If analyses are not conducted within 30 days, freezer storage stability data will be required and generated for these matrices as outlined below.

Method verification analyses will serve as zero day analyses; therefore, storage stability sets must be setup on the same day as method verification. One (1) additional set of samples per matrix will be analyzed for storage stability at one time point to include the longest sample storage interval. One (1) additional set of fortified and unfortified samples will be included as contingency samples.

Each stability set will be comprised of 1 control sample, 2 procedural control samples fortified with a known quantity of clomazone, and 2 stored samples fortified with a known quantity of clomazone. The target fortification level will be 25× the LOQ.

Moisture Determination: The percent moisture will be determined for one canola seed sample from each site and for one meal sample (if generated).

Sample Retention: Samples will be retained in frozen storage by Morse Laboratories until the Sponsor and Study Director approves disposal. The Quality Assurance Unit of Morse Laboratories will verify sample disposal.

5.0 GENERAL

Bias Control: Representative sub-samples from homogeneous mixtures of each sample will be taken for analysis. Analytical recovery analyses from each untreated control sample fortified with the appropriate analyte will be conducted with each batch of samples analyzed to assure accurate and precise recovery of the test substance from the samples. At least one untreated control sample will be analyzed with each batch of samples analyzed to demonstrate that there is no significant interference from the sample matrix or analytical reagents.

Statistical Methods: Statistical treatment of the analytical data generated in this study will be limited to regression analysis and measurements of central tendency and dispersion.

Required Analytical SOPs: A list of applicable standard operating procedures will be supplied in the study raw data.

6.0 ANALYTICAL REPORT

The analytical report will include, but is not limited to the following:

1. A title page
2. A table of contents
3. A GLP compliance statement

4. A QA statement of inspections
5. A description of the analytical standards
6. A short description of sample handling, storage, etc.
7. Discussion of results
8. Description of deviations
9. Summary tables of recoveries obtained during method validation or verification with each substrate
10. Summary tables of analytical method recoveries during sample analysis
11. Summary tables of the results of sample analyses
12. Copies of representative chromatograms of untreated control, treated samples, analytical method fortified samples, and analytical standards. OPPTS 860.1000 (5) Requirements for Magnitude of the Residue Studies should be followed for the number of chromatograms to include in the analytical report.
13. Certificates of analysis for the reference materials
14. A copy of the analytical method including the make and model of equipment, column used, various temperatures used, etc.
15. A comprehensive list of all modifications made to the method of analysis and the reasons (justification) for the modifications.

The original, signed final analytical report will be submitted to the Study Director.

7.0 RAW DATA RETENTION

The analytical facility will maintain a copy of the signed analytical report, protocol and amendments, and all documents (letters, memos, notes, etc.) pertaining to the study. All records pertaining to the analytical portion of this study will be submitted to the Study Director with the issuance of the final study report unless directed otherwise by the Study Director.

Appendix E - Critical Dates

Dates for Critical Events in the Analysis for Clomazone in/on Canola Raw Agricultural Commodities Following One Pre-emergence Application of Clomazone 360 g/L CS (2013)

Trial ID. (City, Province, Country/Year)	Appli. Dates	Sample No.	Sample Type	Trt No.	RAC Cutting/ Sampling Date	DALA (days)	Date Shipped to Lab	Date Received At Lab	Extraction Date	Analysis Date	Days in Frozen Storage[1]
TCI-13-366-01 (Portage la Prairie, MB, CAN/2013)	29-May-13	TCI-13-366-01-01	Canola Seed	1	30-Aug-13 / 12-Sep-13	NA	23-Sep-13	10-Oct-13	14-Mar-14	17-Mar-14	183
		TCI-13-366-01-02	Canola Seed	2	30-Aug-13 / 12-Sep-13	93	23-Sep-13	10-Oct-13	14-Mar-14	17-Mar-14	183
		TCI-13-366-01-03	Canola Seed	2	30-Aug-13 / 12-Sep-13	93	23-Sep-13	10-Oct-13	14-Mar-14	17-Mar-14	183
TCI-13-366-02 (Dundurn, SK, CAN/2013)	11-Jun-13	TCI-13-366-02-01	Canola Seed	1	12-Sep-13	NA	24-Sep-13	10-Oct-13	25-Feb-14	26-Feb-14	166
		TCI-13-366-02-02	Canola Seed	2	12-Sep-13	93	24-Sep-13	10-Oct-13	25-Feb-14	26-Feb-14	166
		TCI-13-366-02-03	Canola Seed	2	12-Sep-13	93	24-Sep-13	10-Oct-13	25-Feb-14	26-Feb-14	166
TCI-13-366-03 (Saskatoon, SK, CAN/2013)	27-May-13	TCI-13-366-03-01	Canola Seed	1	13-Sep-13	NA	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	186
		TCI-13-366-03-02	Canola Seed	2	13-Sep-13	109	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	186
		TCI-13-366-03-03	Canola Seed	2	13-Sep-13	109	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	186
TCI-13-366-04 (Hepburn, SK, CAN/2013)	29-May-13	TCI-13-366-04-01	Canola Seed	1	07-Sep-13	NA	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	192
		TCI-13-366-04-02	Canola Seed	2	07-Sep-13	101	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	192
		TCI-13-366-04-03	Canola Seed	2	07-Sep-13	101	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	192
		TCI-13-366-04-04	Canola Seed	2	10-Sep-13	104	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	189
		TCI-13-366-04-05	Canola Seed	2	10-Sep-13	104	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	189
		TCI-13-366-04-06	Canola Seed	2	14-Sep-13	108	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	185
		TCI-13-366-04-07	Canola Seed	2	14-Sep-13	108	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	185
		TCI-13-366-04-08	Canola Seed	2	21-Sep-13	115	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	178
		TCI-13-366-04-09	Canola Seed	2	21-Sep-13	115	24-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	178
		TCI-13-366-04-10	Canola Seed	2	28-Sep-13	122	04-Nov-13	15-Nov-13	18-Mar-14	19-Mar-14	171
		TCI-13-366-04-11	Canola Seed	2	28-Sep-13	122	04-Nov-13	15-Nov-13	18-Mar-14	19-Mar-14	171

[1]Days in Frozen Storage is the number of days between sampling and extraction.
NA = Not applicable.

Dates for Critical Events in the Analysis for Clomazone in/on Canola Raw Agricultural Commodities Following One Pre-emergence Application of Clomazone 360 g/L CS (2013) (continued)

Trial ID. (City, Province, Country/Year)	Appli. Dates	Sample No.	Sample Type	Trt No.	RAC Cutting/ Sampling Date	DALA (days)	Date Shipped to Lab	Date Received At Lab	Extraction Date	Analysis Date	Days in Frozen Storage[1]
TCI-13-366-05 (Josephburg, AB, CAN/2013)	28-May-13	TCI-13-366-05-01	Canola Seed	1	05-Sep-13/ 19-Sep-13	NA	26-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	180
		TCI-13-366-05-02	Canola Seed	2	05-Sep-13/ 19-Sep-13	100	26-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	180
		TCI-13-366-05-03	Canola Seed	2	05-Sep-13/ 19-Sep-13	100	26-Sep-13	10-Oct-13	18-Mar-14	19-Mar-14	180
		TCI-13-366-05-04	Canola Seed	2	09-Sep-13/ 19-Sep-13	104	26-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	183
		TCI-13-366-05-05	Canola Seed	2	09-Sep-13/ 19-Sep-13	104	26-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	183
		TCI-13-366-05-06	Canola Seed	2	13-Sep-13/ 19-Sep-13	108	26-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	183
		TCI-13-366-05-07	Canola Seed	2	13-Sep-13/ 19-Sep-13	108	26-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	183
		TCI-13-366-05-08	Canola Seed	2	20-Sep-13/ 01-Oct-13	115	06-Nov-13	15-Nov-13	21-Mar-14	21-Mar-14	171
		TCI-13-366-05-09	Canola Seed	2	20-Sep-13/ 01-Oct-13	115	06-Nov-13	15-Nov-13	21-Mar-14	21-Mar-14	171
		TCI-13-366-05-10	Canola Seed	2	26-Sep-13/ 01-Oct-13	121	06-Nov-13	15-Nov-13	21-Mar-14	21-Mar-14	171
		TCI-13-366-05-11	Canola Seed	2	26-Sep-13/ 01-Oct-13	121	06-Nov-13	15-Nov-13	21-Mar-14	21-Mar-14	171
TCI-13-366-06 (Carberry, MB, CAN/2013)	25-May-13	TCI-13-366-06-01	Canola Seed	1	24-Sep-13	NA	01-Nov-13	15-Nov-13	14-Mar-14	17-Mar-14	171
		TCI-13-366-06-02	Canola Seed	2	24-Sep-13	122	01-Nov-13	15-Nov-13	14-Mar-14	17-Mar-14	171
		TCI-13-366-06-03	Canola Seed	2	24-Sep-13	122	01-Nov-13	15-Nov-13	14-Mar-14	17-Mar-14	171
		TCI-13-366-06-04	Canola Seed	4	24-Sep-13	122	01-Nov-13	15-Nov-13	14-Mar-14	17-Mar-14	171
		TCI-13-366-06-05	Canola Seed	4	24-Sep-13	122	01-Nov-13	15-Nov-13	14-Mar-14	17-Mar-14	171
	25-May-13	TCI-13-366-06-06	Canola Seed	1	24-Sep-13	NA	01-Nov-13	19-Nov-13	Bulk Sample for Processing		
		TCI-13-366-06-07	Canola Seed	4	24-Sep-13	122	01-Nov-13	19-Nov-13			

[1]Days in Frozen Storage is the number of days between sampling and extraction.
NA = Not applicable.

Dates for Critical Events in the Analysis for Clomazone in/on Canola Raw Agricultural Commodities Following One Pre-emergence Application of Clomazone 360 g/L CS (2013) (continued)

Trial ID. (City, Province, Country/Year)	Appli. Dates	Sample No.	Sample Type	Trt No.	RAC Cutting/ Sampling Date	DALA (days)	Date Shipped to Lab	Date Received At Lab	Extraction Date	Analysis Date	Days in Frozen Storage[1]
TCI-13-366-07 (Shilo, MB, CAN/2013)	25-May-13	TCI-13-366-07-01	Canola Seed	1	23-Aug-13/ 05-Sep-13	NA	23-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	197
		TCI-13-366-07-02	Canola Seed	2	23-Aug-13/ 05-Sep-13	90	23-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	197
		TCI-13-366-07-03	Canola Seed	2	23-Aug-13/ 05-Sep-13	90	23-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	197
TCI-13-366-08 (Brandon, MB, CAN/2013)	25-May-13	TCI-13-366-08-01	Canola Seed	1	23-Aug-13 / 30-Aug-13	NA	23-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	203
		TCI-13-366-08-02	Canola Seed	2	23-Aug-13 / 30-Aug-13	90	23-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	203
		TCI-13-366-08-03	Canola Seed	2	23-Aug-13 / 30-Aug-13	90	23-Sep-13	10-Oct-13	21-Mar-14	21-Mar-14	203
TCI-13-366-09 (Alvena, SK, CAN/2013)	03-Jun-13	TCI-13-366-09-01	Canola Seed	1	13-Sep-13	NA	24-Sep-13	10-Oct-13	21-Mar-14	22-Mar-14	189
		TCI-13-366-09-02	Canola Seed	2	13-Sep-13	102	24-Sep-13	10-Oct-13	21-Mar-14	22-Mar-14	189
		TCI-13-366-09-03	Canola Seed	2	13-Sep-13	102	24-Sep-13	10-Oct-13	21-Mar-14	22-Mar-14	189
TCI-13-366-10 (Wakaw, SK, CAN/2013)	12-Jun-13	TCI-13-366-10-01	Canola Seed	1	20-Sep-13	NA	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	187
		TCI-13-366-10-02	Canola Seed	2	20-Sep-13	100	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	187
		TCI-13-366-10-03	Canola Seed	2	20-Sep-13	100	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	187
TCI-13-366-11 (Waldheim, SK, CAN/2013)	28-May-13	TCI-13-366-11-01	Canola Seed	1	06-Sep-13	NA	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	201
		TCI-13-366-11-02	Canola Seed	2	06-Sep-13	101	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	201
		TCI-13-366-11-03	Canola Seed	2	06-Sep-13	101	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	201
TCI-13-366-12 (Aberdeen, SK, CAN/2013)	30-May-13	TCI-13-366-12-01	Canola Seed	1	19-Sep-13	NA	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	188
		TCI-13-366-12-02	Canola Seed	2	19-Sep-13	112	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	188
		TCI-13-366-12-03	Canola Seed	2	19-Sep-13	112	24-Sep-13	10-Oct-13	26-Mar-14	26-Mar-14	188

[1]Days in Frozen Storage is the number of days between sampling and extraction.
NA = Not applicable.