



Regione Siciliana
Assessorato Agricoltura e Foreste
Dipartimento interventi Infrastrutturali
XI Servizio - Servizi allo Sviluppo

DETERMINAZIONE DELLA GUAZATINA NEGLI AGRUMI: ASPETTI ANALITICI

SERENA LAZZARO *, PAOLO BRANCA **

* U.O.S. n. 34 A.S.C.A. - Analisi e Servizi per la Certificazione in Agricoltura- Ispica (Rg)

** Arpa Piemonte - Polo Alimenti - La Loggia (To)

LA GUAZATINA È UN FUNGICIDA
DA CONTATTO NON SISTEMICO
LARGAMENTE IMPIEGATA SU:

CEREALI

CONCIA DELLE SEMENTI

AGRUMI

IMMERSIONE (bulk dip)

TRATTAMENTI POST

RACCOLTA

FUMIGANTE DURANTE IL

PACKING

APPARTIENE ALLA CLASSE DELLE
GUANIDINE

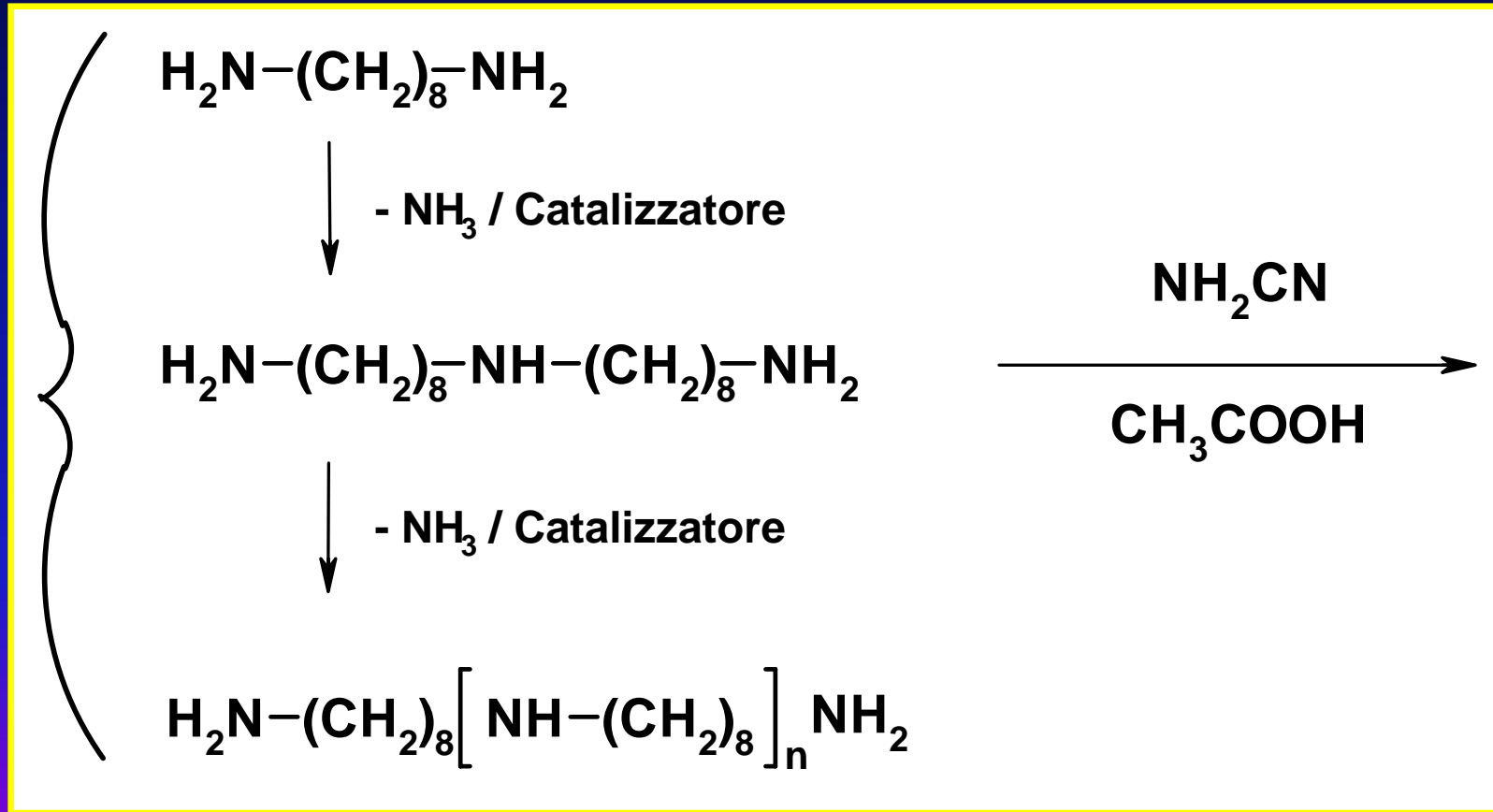


DEFINIZIONE IUPAC:

Una miscela di prodotti di reazione, tra
poliammine - principalmente
octametilendiammine, iminodi
(octameten)diammine, octametenbis
(imino-octameten)diammine - e
cianammide.

Poliammine

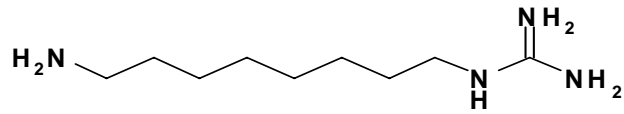
Cianammide



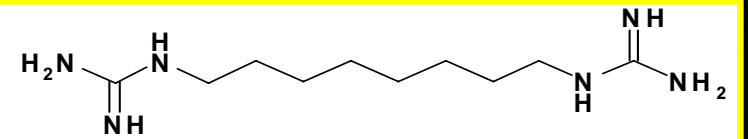
PRODOTTI PRIORITARI DI REAZIONE:
GUAZATINA

UNA MISCELA DI GUANIDINE

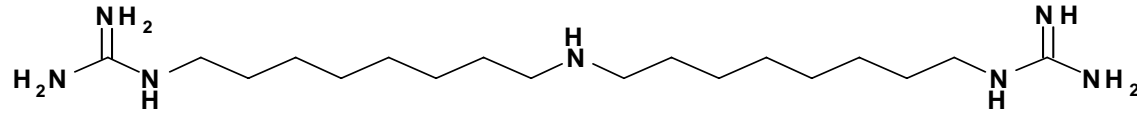
NG



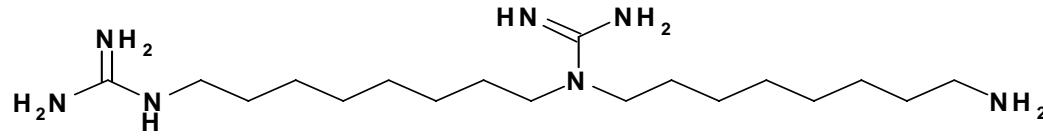
GG



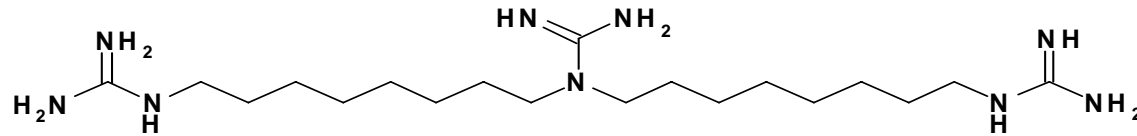
GNG



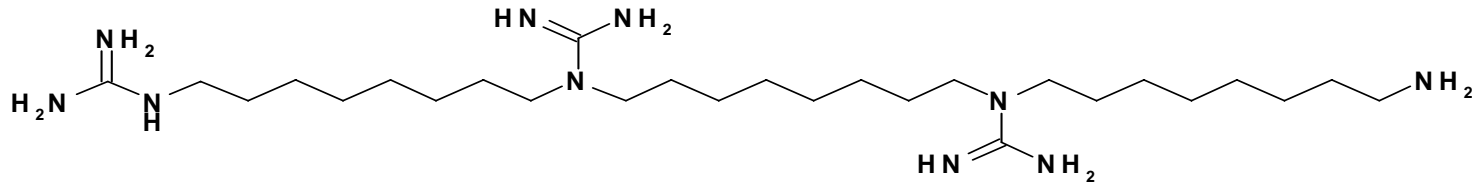
GGN



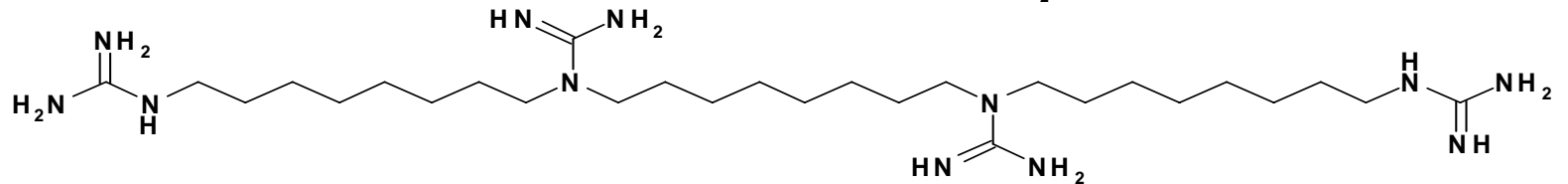
GGG



GGGN



GGGG



N = gruppo amminico

G = gruppo amminico (NH o NH₂) guanidato

COMPOSIZIONE DELLA GUAZATINA

9.8 %

29.5 %

GG

4.5 % **GNG**

89% + OTHER AMINES

8.1 % **GGN**

Report FAO 1997

30.6 % **GGG**

International Portal on Food Safety,
Animal and Plant Health

1.4 %

5.1 %



COMPOSIZIONE DELLA GUAZATINA

9.8 %

29.5 %

GG

4.5 %

GNG



STD

8.1 %

GGN

30.6 %

GGG

1.4 %

5.1 %

Gravimetric Certificate		Dr. Ehrenstorfer	
Product Identification 14057300 Guazatine triacetate Formula N/A Mol.Weight N/A CAS No. 115044-19-4 Please note: The expiry date is valid under recommended storage conditions only.			Reference Materials for Residue Analysis Expiry Date 12.07.2009 Lot Number 70623ME Store at 20°C in the dark
Gravimetric Data Product Name Guazatine triacetate	Conc. (mg/l) 10,000	Purity % 98,0	Weight (mg) 1,024
Solvent Information Solvent Methanol	Lot No. 62153	Exact Quantity (ml) 100,00	
Traceability Data 14057300 21001 14057300 70623ME	neat product 10,000 mg/l		
Analytical Data Detection: LC/MSD Column: ReproSil-Pur ODS3 5µ 250x3 Inj.-Vol.: 10,00 µl Flow: 0,5 ml/min Ret.-Time: 1,70 min.			
Method Details: Acetonitrile:H2O+0,15% Formic acid 2:1			
Identity check RT, MS Comment			
The uncertainty/tolerance of this standard is +/- 1.0 %, calculated in accordance with the EURACHEM/CITAC Guide - Quantifying Uncertainty in Analytical Measurement - Second Edition. The uncertainty given is the expanded combined uncertainty and represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The expanded uncertainty is U which is Uc(y) * K, where K is the coverage factor at the 95% confidence level (K=2). The expanded uncertainty is based on the combination of uncertainties associated with each individual operation involved in the preparation of this product.			
Certified on 15.07.2007			

Lyon 1997, Joint Meeting of the **FAO** panel of experts on pesticide residues in food and environment and **WHO** core assessment group:

Guazatine "moderately hazardous, with an oral LD₅₀ value in rats of 280 mg/Kg bw"

*"The Meeting concluded that the residue data were not adequate for **cistrus fruits** and recommended the withdrawal of the existing **MRL of 5 ppm**"*

Australia

Nuova Zelanda

Austria

Svezia

Finlandia

Norvegia

Spagna

Germania

**REPORT
1997**



Per il **cereali** Guideline Level:

0,05 ppm

Ministero della Salute: "Nociva e pericolosa per l'ambiente" oltre ad essere altamente tossica per gli ambienti acquatici.

Nel 2003, l'assessorato all'ambiente della provincia di Firenze indica la Guazatina tra i fitofarmaci più pericolosi in riferimento al rischio complessivo per l'ambiente (indice ICRA)

27 AGOSTO 2007 LA GUAZATINA IN EUROPA NON E' PIU' AUTORIZZATA.

IT

Gazzetta ufficiale dell'Unione europea

DECISIONE DELLA COMMISSIONE

del 27 agosto 2007

concernente la non iscrizione del triacetato di guazatina nell'allegato I, nell'allegato I A o nell'allegato I B della direttiva 98/8/CE del Parlamento europeo e del Consiglio relativa all'immissione sul mercato dei biocidi

tipo di prodotto 8. Inoltre, la valutazione del rischio ambientale effettuata dalla competente autorità del Regno Unito, sulla base della peggiore delle ipotesi in assoluto, ha evidenziato rischi inaccettabili per l'ambiente.

NON CI SONO EVIDENZE PER AFFERMARE, MA
NON SI PUÓ NEANCHE ESCLUDERE,
CHE LA GUAZATINA SIA CANCEROGENA
NE' CHE SIA PERICOLOSA PER IL SISTEMA
ENDOCRINO E RIPRODUTTORE.

NON INFLUISCE SULLA COLINESTERASI,
MA E' COMUNQUE DEFINITA **TOSSICA**.

PUR ESSENDO UNANIMAMENTE
CONSIDERATA UNA SOSTANZA NOCIVA,
LA GUAZATINA E' RACCOMANDATA
COME ANTIDOTO MIGLIORE CONTRO LA
FUSARIOSI DEL PIEDE DEI CEREALI,
SOPRATTUTTO SE ASSOCIATA AL
TRITICONAZOLO.

RESIDUI DI GUAZATINA SONO STATI RILEVATI IN AGRUMI PROVENIENTI DAL NORD AFRICA, IN PARTICOLARE DAL MAROCCO, MA ANCHE SU AGRUMI NAZIONALI.

RICERCA BIBLIOGRAFICA

LA RISOLUZIONE E L'IDENTIFICAZIONE DEI
COMPONENTI PRINCIPALI
DELLA "MISCELA GUAZATINA"
È STATA ESEGUITA MEDIANTE:

GC-MS

**DERIVATIZZAZIONE CON
ESAFLUOROACETIL ACETONE**

IDENTIFICAÇÃO DOS PRINCIPAIS COMPONENTES DO FUNGICIDA GUAZATINE ATRAVÉS DA CROMATOLOGRAFIA A GÁS E CROMATOLOGRAFIA A GÁS ACOPLADA COM ESPECTROMETRIA DE MASSAS

Arquimedes Lavorenti

Departamento de Química - Escola Superior de Agricultura "Luiz de Queiroz" - Universidade de São Paulo - CP 9 - 13418-900 - Piracicaba - SP

Harry Robinson Hudson, Max Pianka

School of Applied Chemistry - University of North London - 166-220 Holloway Road - London N7 8DB - England.

Recebido em 28/10/96; aceito em 10/11/97

IDENTIFICATION OF THE MAIN COMPONENTS OF GUAZATINE FUNGICIDE BY GAS CHROMATOGRAPHY AND GAS CHROMATOGRAPHY-MASS SPECTROMETRY. Hexafluoroacetylaceton derivatives of some synthesized components of the guazatine fungicide were prepared, and their separation and identification performed through gas chromatography and gas chromatography-mass spectrometry. The chromatogram obtained from hexafluoroacetylaceton derivative of the commercial guazatine presented 14 major peaks, and 9 of them were identified through both techniques mentioned above.

GC-MS

DERIVATIZZAZIONE CON ESAFLUOROACETIL ACETONE

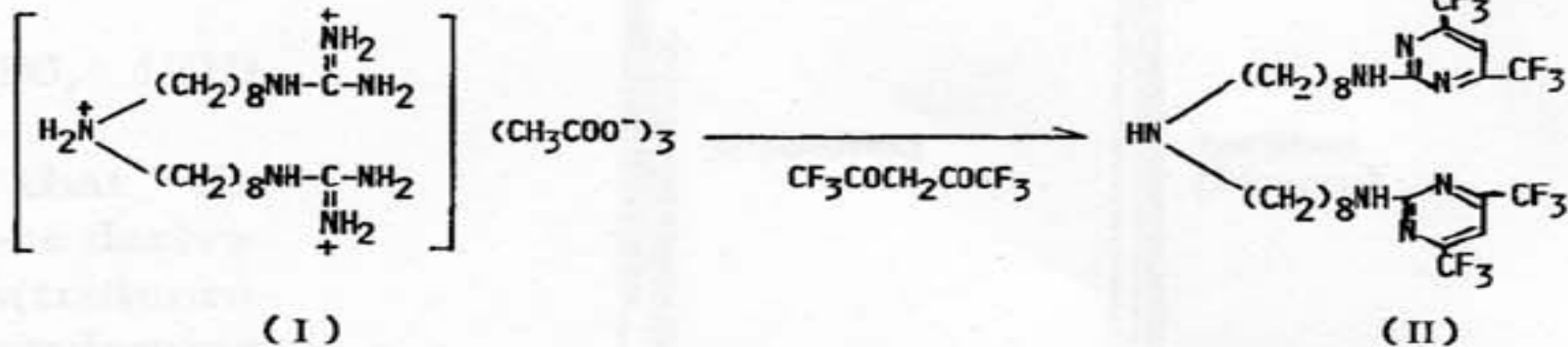


Fig. 1 Derivatization of Guazatine with hexafluoroacetylacetone.

HPLC-ESI-MS

Single-quadrupole

- COLONNA 250 X 10 mm
- ISOLAMENTO DEI COMPONENTI PRIORITARI
- QUANTIFICAZIONE

LC/ESI/MS Method for the Quantitative Detection of Guazatine Residues in Cereals

ELENA DREASSI,* ALESSANDRA TANIA ZIZZARI, ASSUNTA ZANFINI,
GIANFRANCO CORBINI, AND MAURIZIO BOTTA

Università degli Studi di Siena, Dipartimento Farmaco Chimico Tecnologico – via A. Moro,
53100 Siena, Italy

Guazatine is a fungicide used in agriculture to control a wide range of seed-borne diseases of cereals and other vegetable foods. In this work, a LC-ESI-MS method was developed for the quantitative detection of guazatine residues in maize and hard wheat. Quantitative data were determined for the residues of the main diamines, triamines, and tetramines that cover more than 87% of the total contents of the mixture. The mean recoveries from the fortified cereals at 0.050 mg/kg ranged from 81 to 86%, with the coefficients of variation (CVs) ranging from 0.9 to 5.5% ($n = 5$). At 0.025 mg/kg, the recoveries ranged from 78 to 87%, with the CVs ranging from 0.8 to 6.3% ($n = 5$). The limits of quantification have been estimated to be 0.010, 0.004, 0.002, 0.002, 0.005, and 0.002 mg/kg, respectively, for GN, GG, GNG, GGN, GGG, and GGGG in maize and hard wheat (S/N ratio > 10).

HPLC-ESI-MS

Single-quadrupole

- COLONNA 250 X 10 mm
- ISOLAMENTO DEI COMPONENTI PRIORITARI
- QUANTIFICAZIONE

LC/ESI/MS Method for the Quantitative Detection of Guazatine Residues in Cereals

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53100 Siena, Italy

The separation and identification of the main components of guazatine was performed through LC-MS analysis. We searched for better conditions to obtain single component separation through LC-MS analysis. RP analytical or ionic exchange columns did not provide good separation (data not shown), while better results were obtained using a semipreparative column.

1° OBIETTIVO

RISOLUZIONE CROMATOGRAFICA
MEDIANTE UNA COLONNA
ANALITICA

HPLC - ESI MS
ION TRAP

SI È PROVVEDUTO
ALL'ACQUISTO DI:

STANDARD DELLA GUANIDINA GNG

9.8 %

29.5 %

GG

4.5 %

GNG



8.1 %

GGN

30.6 %

GGG

1.4 %

5.1 %

Gravimetric Certificate		Dr. Ehrenstorfer	
Product Identification 14057300 Guazatine triacetate Formula N/A Mol.Weight N/A CAS No. 115044-19-4 Please note: The expiry date is valid under recommended storage conditions only.			Reference Materials for Residue Analysis Expiry Date 12.07.2009 Lot Number 70623ME Store at 20°C in the dark
Gravimetric Data			
Product Name	Guazatine triacetate	Conc. (mg/l)	Purity %
		10,000	98,0
			Weight (mg)
			1,024
Solvent Information			
Solvent	Methanol	Lot No.	Exact Quantity (ml)
		62153	100,00
Traceability Data			
14057300 21001	neat product		
14057300 70623ME	10,000 mg/l		
Analytical Data			
Detection:	LC/MSD	Method Details:	
Column:	ReproSil-Pur ODS3 5µ 250x3	Acetonitrile:H2O+0,15% Formic acid 2:1	
Inj.-Vol:	10,00 µl		
Flow:	0,5 ml/min		
Ret.-Time:	1,70 min.		
Identity check RT, MS			
Comment			
The uncertainty/tolerance of this standard is +/- 1,0 %, calculated in accordance with the EURACHEM/CITAC Guide - Quantifying Uncertainty in Analytical Measurement - Second Edition. The uncertainty given is the expanded combined uncertainty and represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The expanded uncertainty is U which is Uc(y)·K, where K is the coverage factor at the 95% confidence level (K=2). The expanded uncertainty is based on the combination of uncertainties associated with each individual operation involved in the preparation of this product.			
Certified on 15.07.2007			

Standard Analitico di GUAZATINA miscela di guanidine

SIGMA-ALDRICH **Riedel-de Haën**

**CERTIFICATE OF ANALYSIS /
INSPECTION CERTIFICATE 3.1 acc.**

Sigma-Aldrich Laborchemikalien GmbH D-30918 Seelze
Telefon: +49 5137 8238-150

Seelze, 16.05.2006/757088

Order-No.:
Customer-No.:
Order-Code:
Quantity:

Production date: 30.Aug.2005
rec. Retest Date: 30.Aug.2008

Article/Product: 37915 Batch : 5242X

Guazatine acetate PESTANAL® (mixture of reaction products from polyamines), 100 mg

Reference Material (RM)

1. General Information

Formula: Molar mass: 535.7 g/Mole
CAS-NO.: [115044-19-4] Recomm. storage temp.: room
Usage : Fungicide

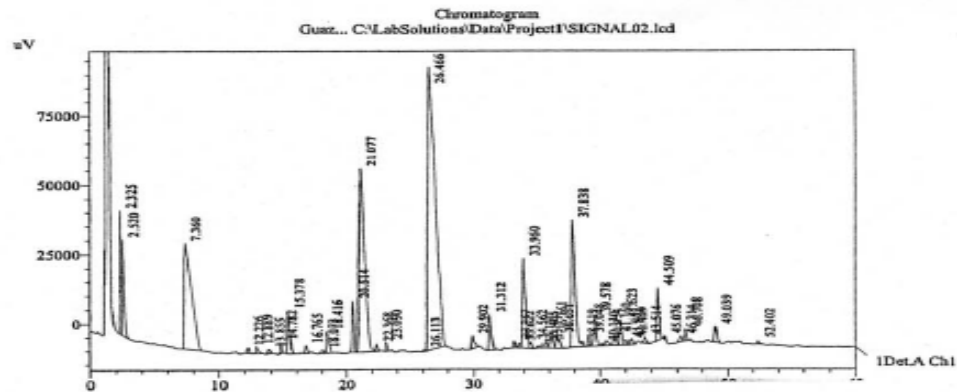
Batch Analysis

Identity (LC-MS)	complying	
acetate (HPLC)	19.5	%
<u>sum of ident.guazat.HPLC uncorr.</u>	<u>79.4</u>	<u>%</u>
Water (Karl Fischer)	38.2	%
Date of Analysis	12.Oct.2005	

Article : Guazatine acetate
Article-No : 37915
Batch : 5242X

Column : L=150mm, ID=2,1mm; Discovery C-18 5µm
Eluent A : Acetonitril
Eluent B : Water + 0,1% Methanesulfonic acid
Gradient : time(min) % A % B
20 5 95
50 2,5 75
60 Step

Flow : 0,4ml/min
Detector : UV-210nm
Injection-Volume : 20µl
Sample-Preparation : 2mg/ml
Linearity : checked
Evaluation : Normalisation (uncorrected); identified by LC-MS (ESI)
Operator : Schowe



1 Det.A Ch1 / Ret. Time	Area	Area %	Name	Ret. Time	Area	Area %	Name
2.325	275347	2.198		22.368	28004	0.224	
2.520	387452	3.093		23.090	35668	0.285	
7.360	1604096	12.806		26.113	11105	0.089	
12.226	22226	0.177	GG	26.466	4450972	35.533	GGG
12.889	37202	0.297		29.502	70066	0.559	
13.855	17661	0.141		31.312	190332	1.519	
14.782	40589	0.324		31.257	24157	0.193	
15.378	206663	1.650	GNN	33.622	26291	0.162	
16.765	37571	0.300		33.960	575159	4.592	NNNNN
18.073	19187	0.153		34.562	31850	0.254	
18.416	133327	1.064	GNG	35.140	11261	0.090	
20.514	208338	1.663		35.465	24646	0.197	
21.077	1932449	15.427	GGN	35.792	29678	0.237	
				36.161	98648	0.788	NNNN
				36.651	82072	0.655	
				37.838	941067	7.513	GNNNN
				38.519	10145	0.081	
				39.048	39973	0.319	
				39.578	173513	1.385	
				40.110	10947	0.087	
				40.454	33353	0.266	
				41.160	59780	0.477	
				41.623	112049	0.902	
				41.952	21522	0.172	
				42.193	21181	0.169	
				42.429	28242	0.225	
				43.514	31028	0.248	
				44.509	248529	1.984	
				45.076	12415	0.099	
				46.314	34464	0.275	
				46.708	49723	0.397	
				49.039	75391	0.602	
				52.402	16204	0.129	
					12526447	100.000	

Formulato Commerciale PANOCTINE L - FUNGICIDA



Composizione:

- GUAZATINA 30.00%
- GLICOLE ETILENICO 0.00%
- NON SPECIFICATI 70.00%

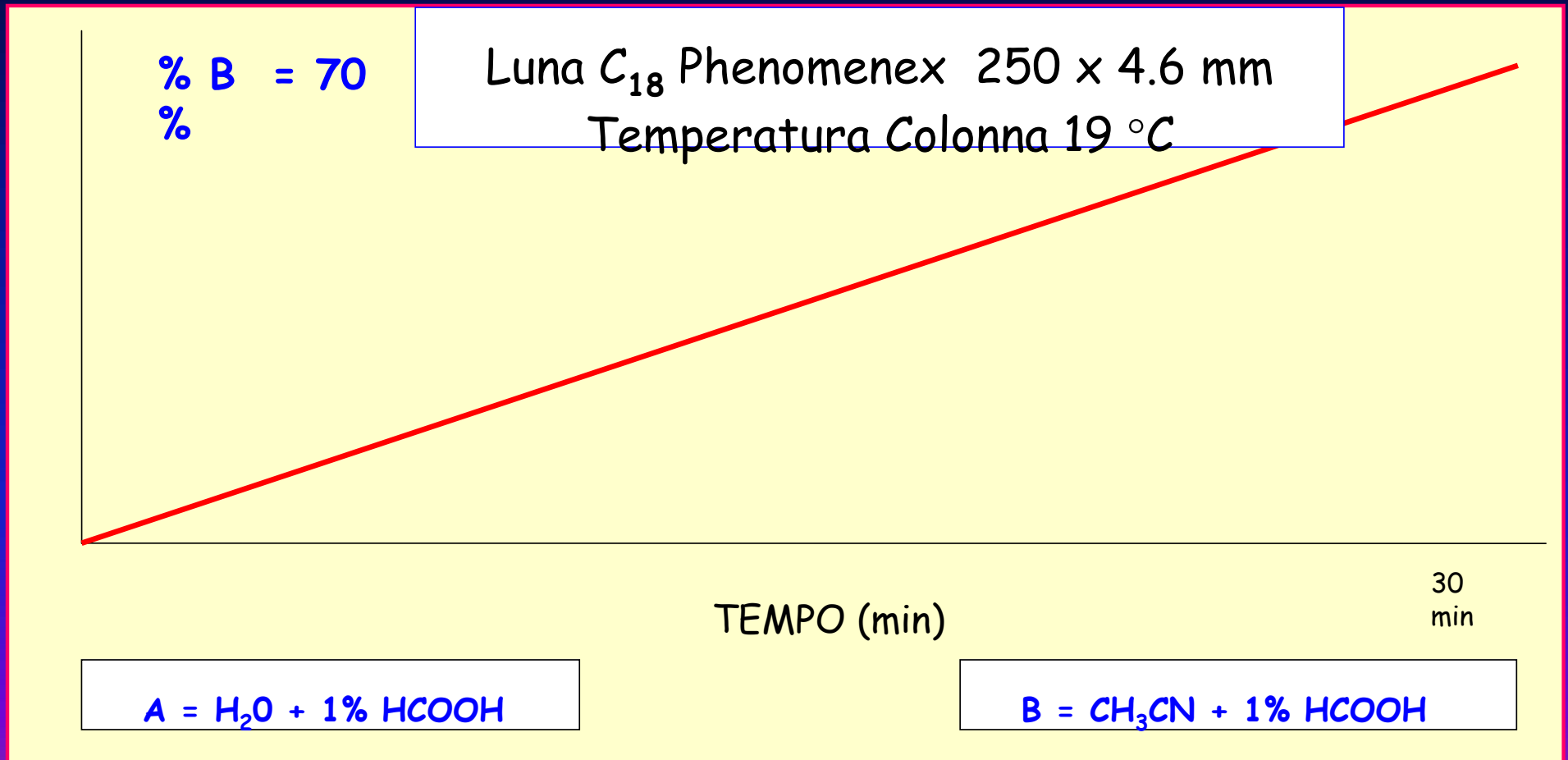


Registrazione n° 5890 del 25/04/1984 del Ministero della Sanità.

Società Detentrici: makhteshim agan italia

Distributore: • makhteshim agan italia

L' ANALISI DEI COMPENENTI DELLA GUAZATINA È STATA ESEGUITA MEDIANTE **COLONNA ANALITICA**



L' ANALISI DEI COMPONENTI DELLA GUAZATINA È STATA ESEGUITA MEDIANTE **COLONNA ANALITICA**

**% B = 70
%**

Luna C₁₈ Phenomenex 250 x 4.6 mm
Temperatura Colonna 19 °C

Flow 300 µl/min

TEMPO (min)

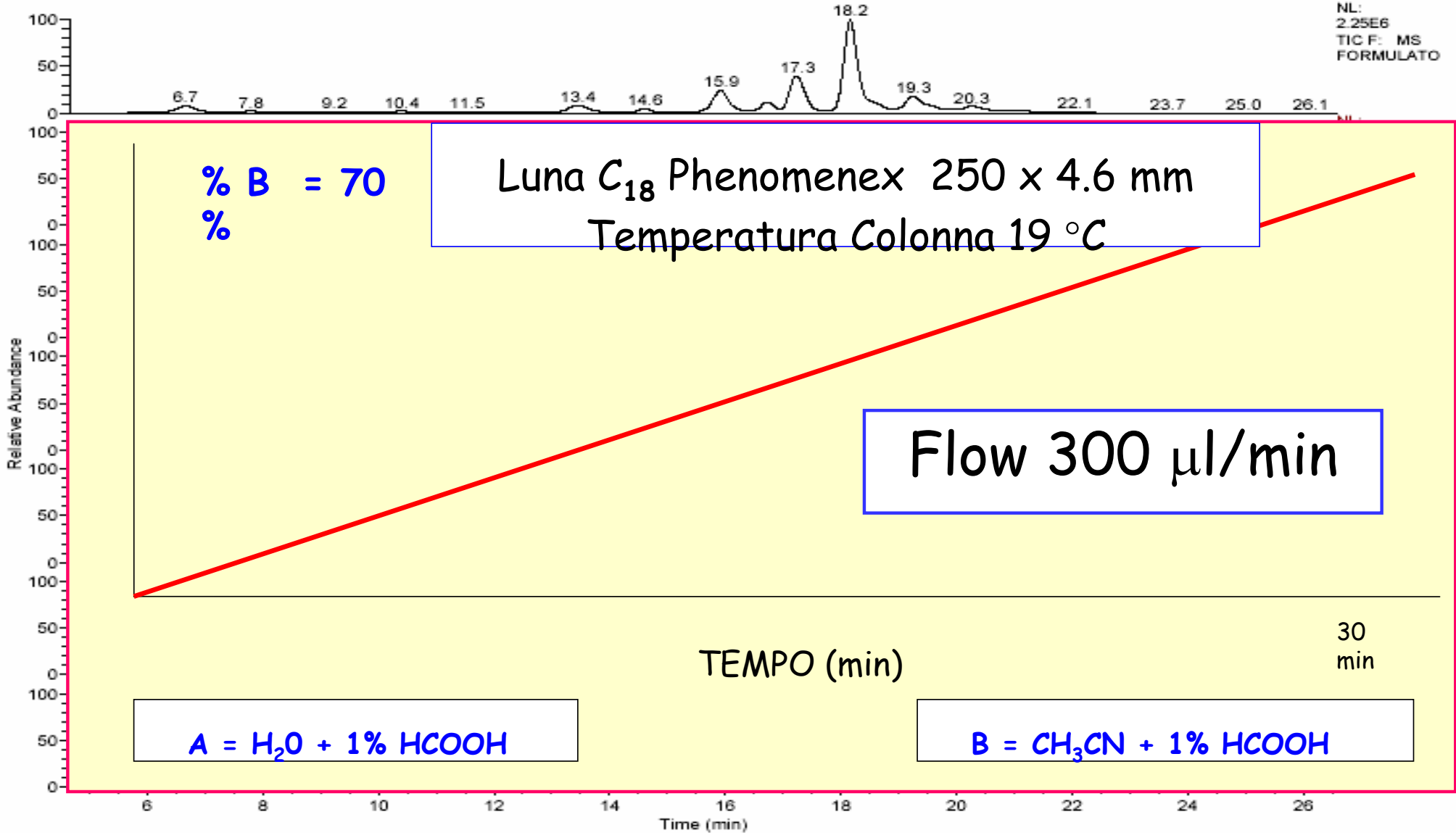
30
min

A = H₂O + 1% HCOOH

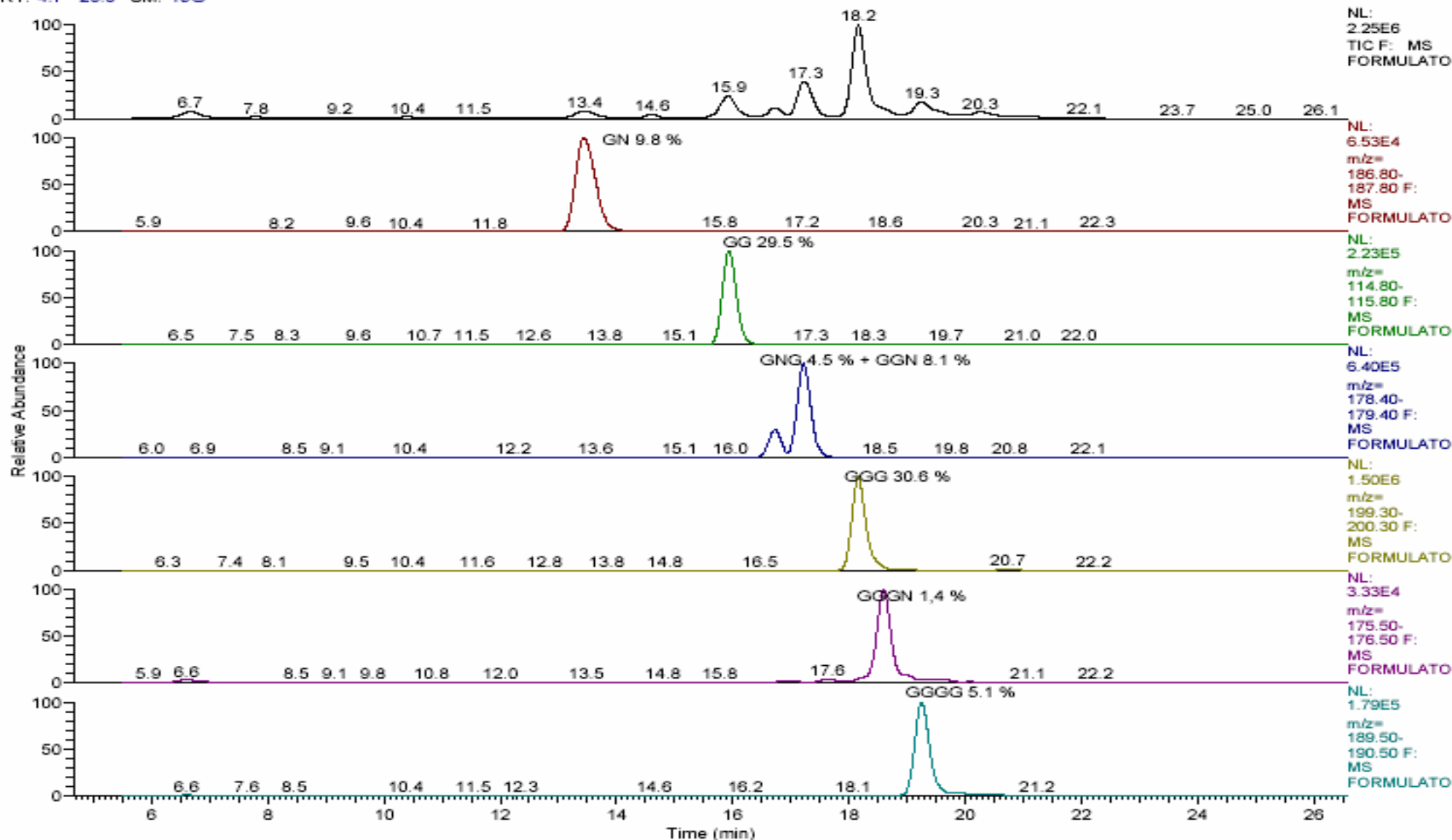
B = CH₃CN + 1% HCOOH

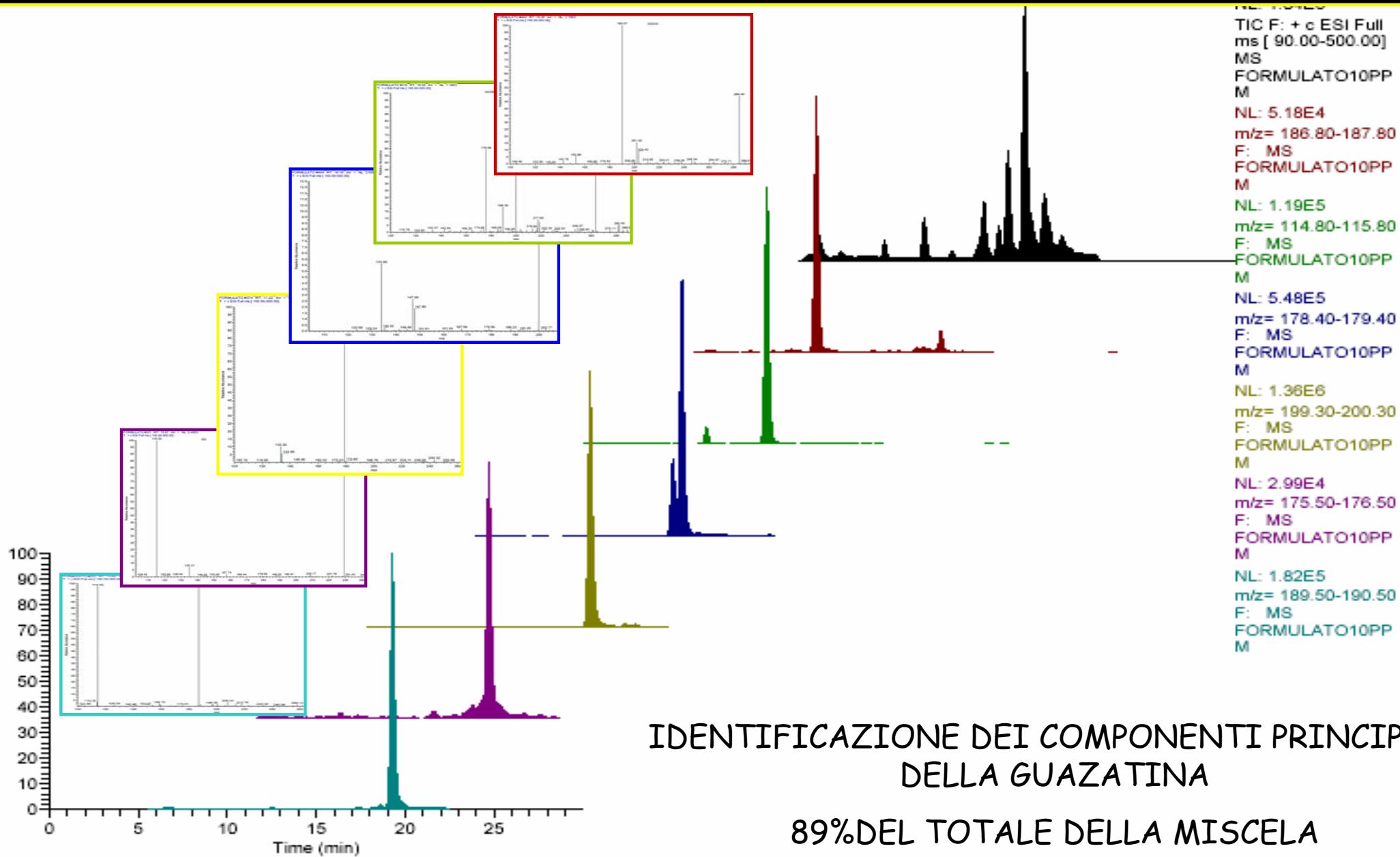
RT: 4.7 - 26.6 SM: 15G

NL:
2.25E6
TIC F: MS
FORMULATO



RT: 4.7 - 26.6 SM: 15G

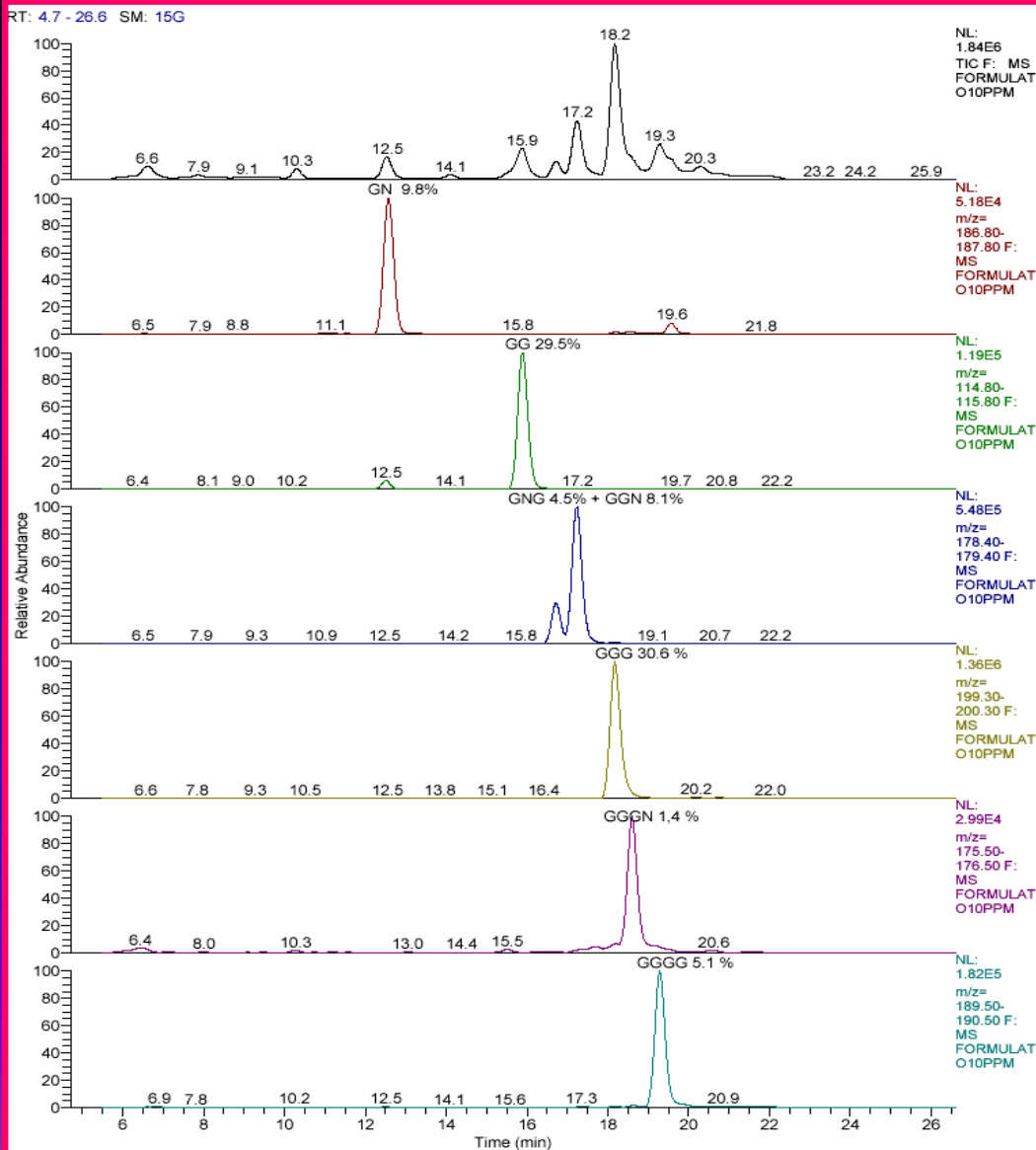




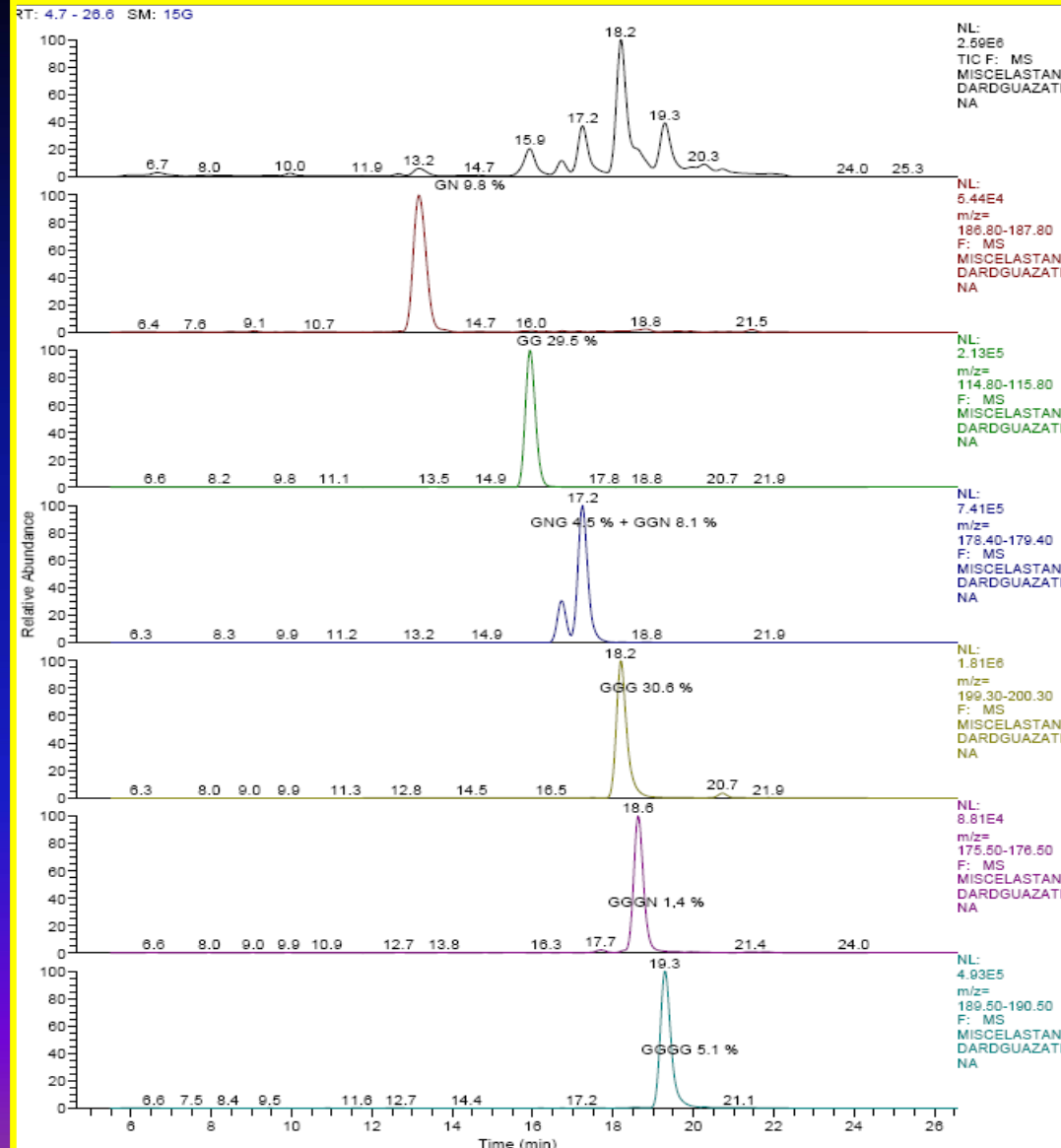
IDENTIFICAZIONE DEI COMPONENTI PRINCIPALI
 DELLA GUAZATINA

89% DEL TOTALE DELLA MISCELA

FORMULATO



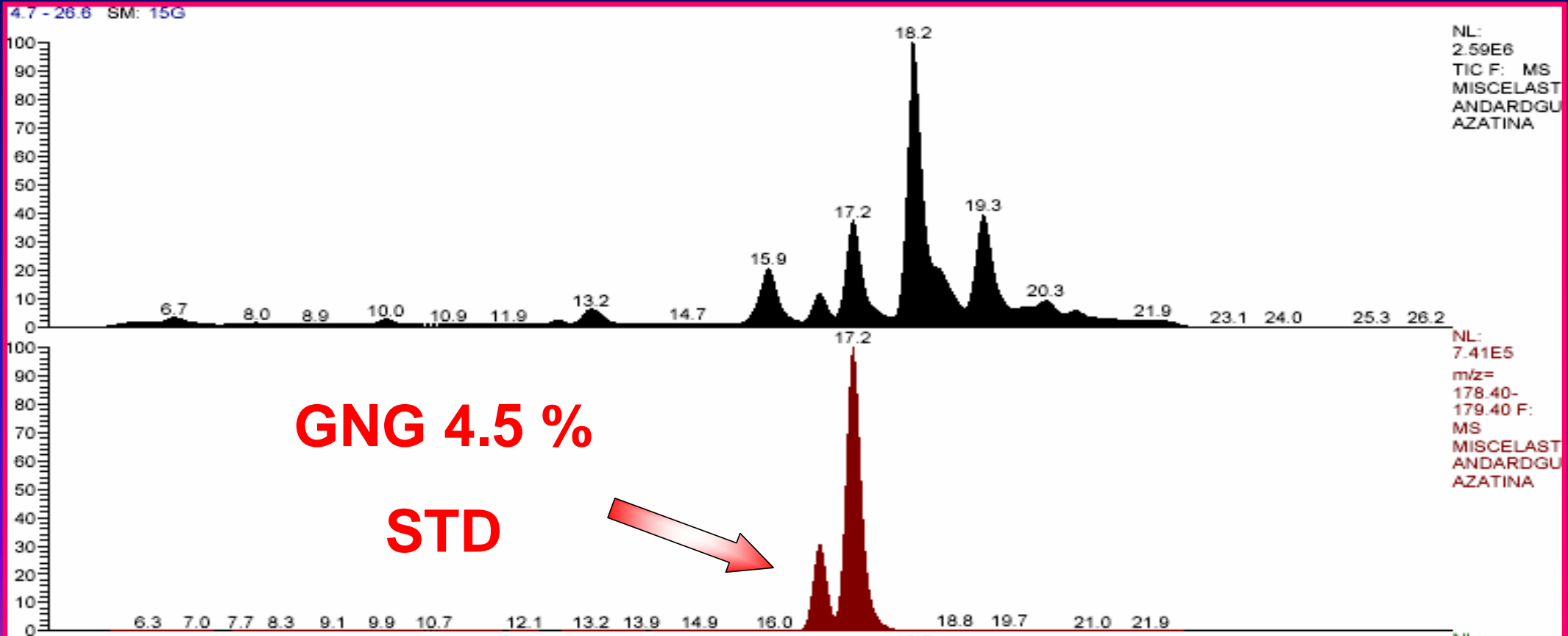
STANDARD ANALITICO



STANDARD ANALITICO

2° OBIETTIVO

DETERMINAZIONE DELLA PUREZZA %



DETERMINAZIONE DELLA PUREZZA % DELLO STANDARD ANALITICO

SE LO STANDARD ANALITICO FOSSE PER IL 100 % COSTITUITO DA GUAZATINA IN UNA SOLUZIONE

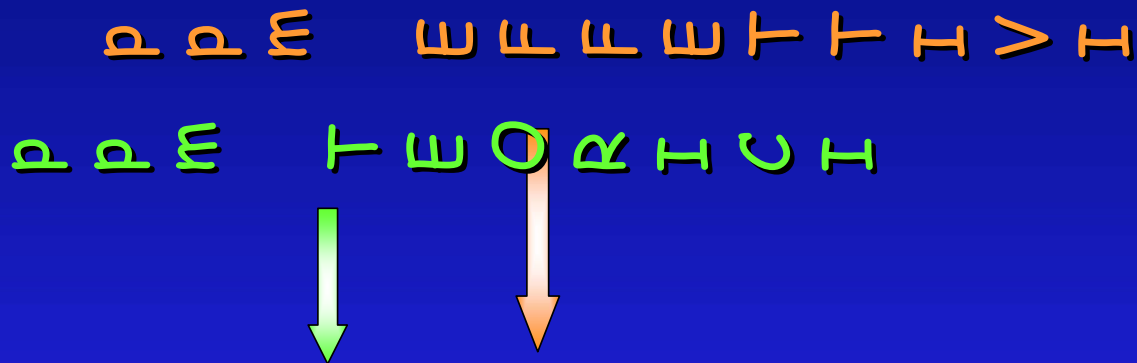
34 ppm DI GUAZATINA IL CONTENUTO DI GNG DOVREBBE ESSERE:

$$4.5 : 100 = x : 34$$

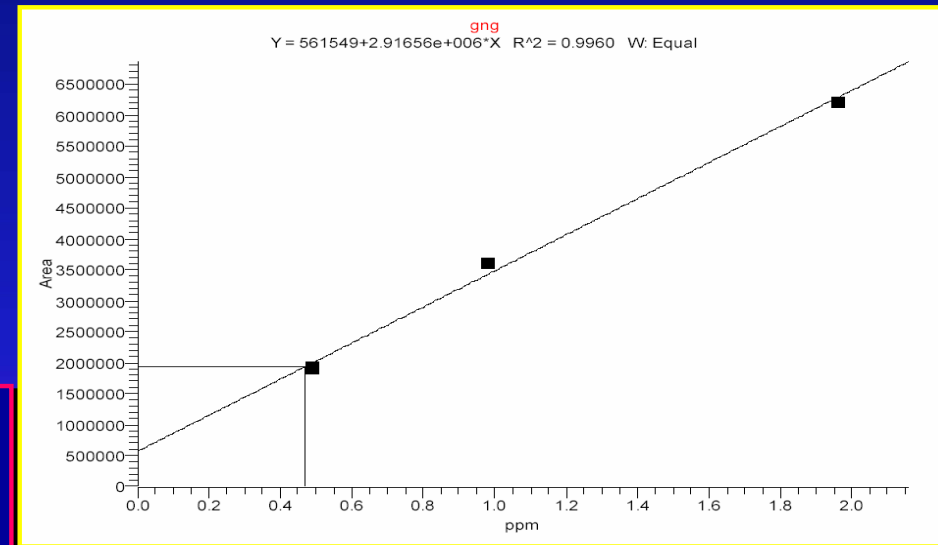
$$x = 1.5 \text{ ppm TEORICI}$$

NELLA SOLUZIONE DI GUAZATINA IL CONTENUTO IN ppm DI **GNG** CALCOLATO CON IL METODO DELLO STANDARD ESTERNO RISULTA PARI A **0,60 ppm**

LA PUREZZA % DELLO STANDARD ANALITICO SARA':



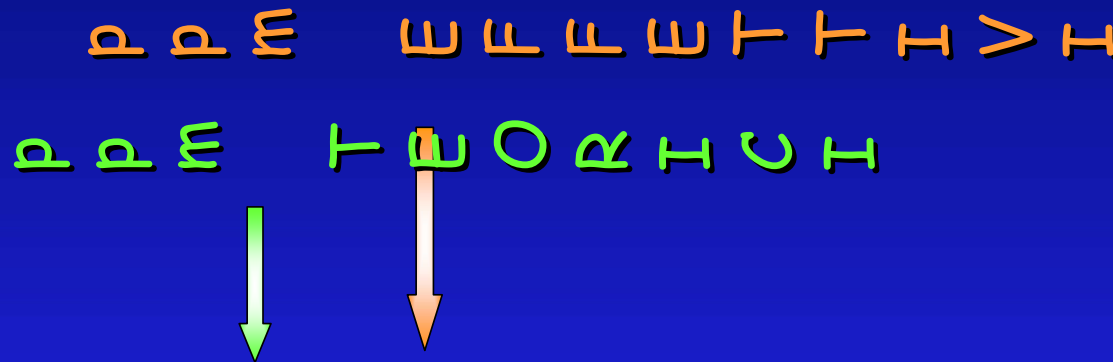
$$0,6 : 1,53 = X : 100$$



RETTA DI CALIBRAZIONE GNG

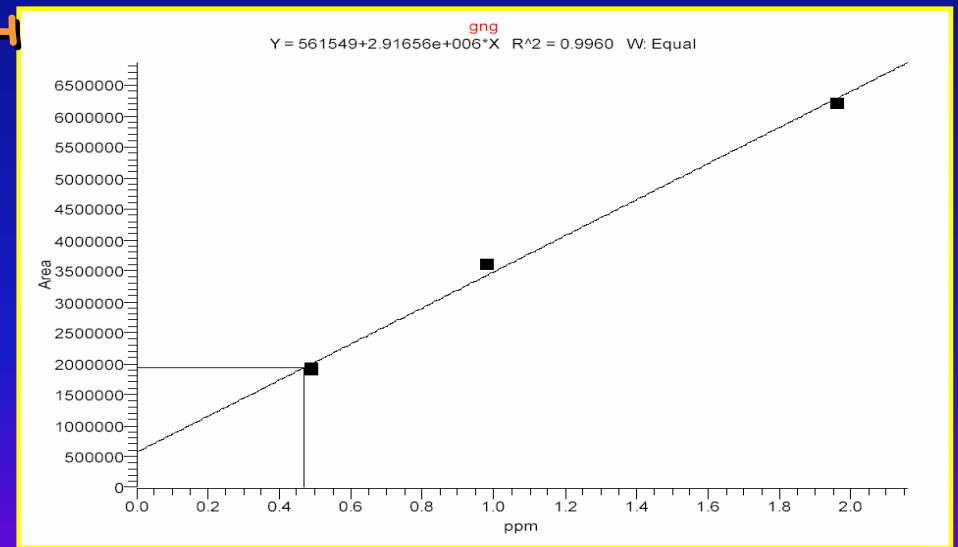
NELLA SOLUZIONE DI GUAZATINA IL CONTENUTO IN ppm DI **GNG** CALCOLATO CON IL METODO DELLO STANDARD ESTERNO RISULTA PARI A **0,60 ppm**

LA PUREZZA % DELLO STANDARD ANALITICO SARA':



$$0,6 : 1,53 = X : 100$$

$$X = 39 \%$$



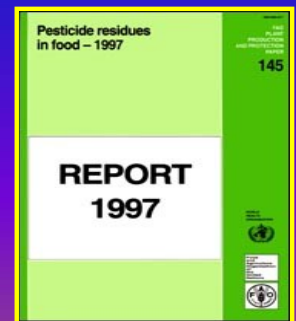
RETTE DI CALIBRAZIONE GNG

3° OBIETTIVO

QUANTIFICAZIONE DELLA GUAZATINA

**1997 JOINT MEETING OF THE FAO PANEL OF
EXPERTS ON PESTICIDE
RESIDUES IN FOOD AND THE ENVIRONMENT
AND THE WHO CORE
ASSESSMENT GROUP**

Lyon (IARC), 22 September-1 October 1997



3° OBIETTIVO

QUANTIFICAZIONE DELLA GUAZATINA

The use of such a complex mixture as guazatine presents a problem in choosing a residue analytical method.

It is not considered practical to attempt the determination of all the components so some alternative is necessary.

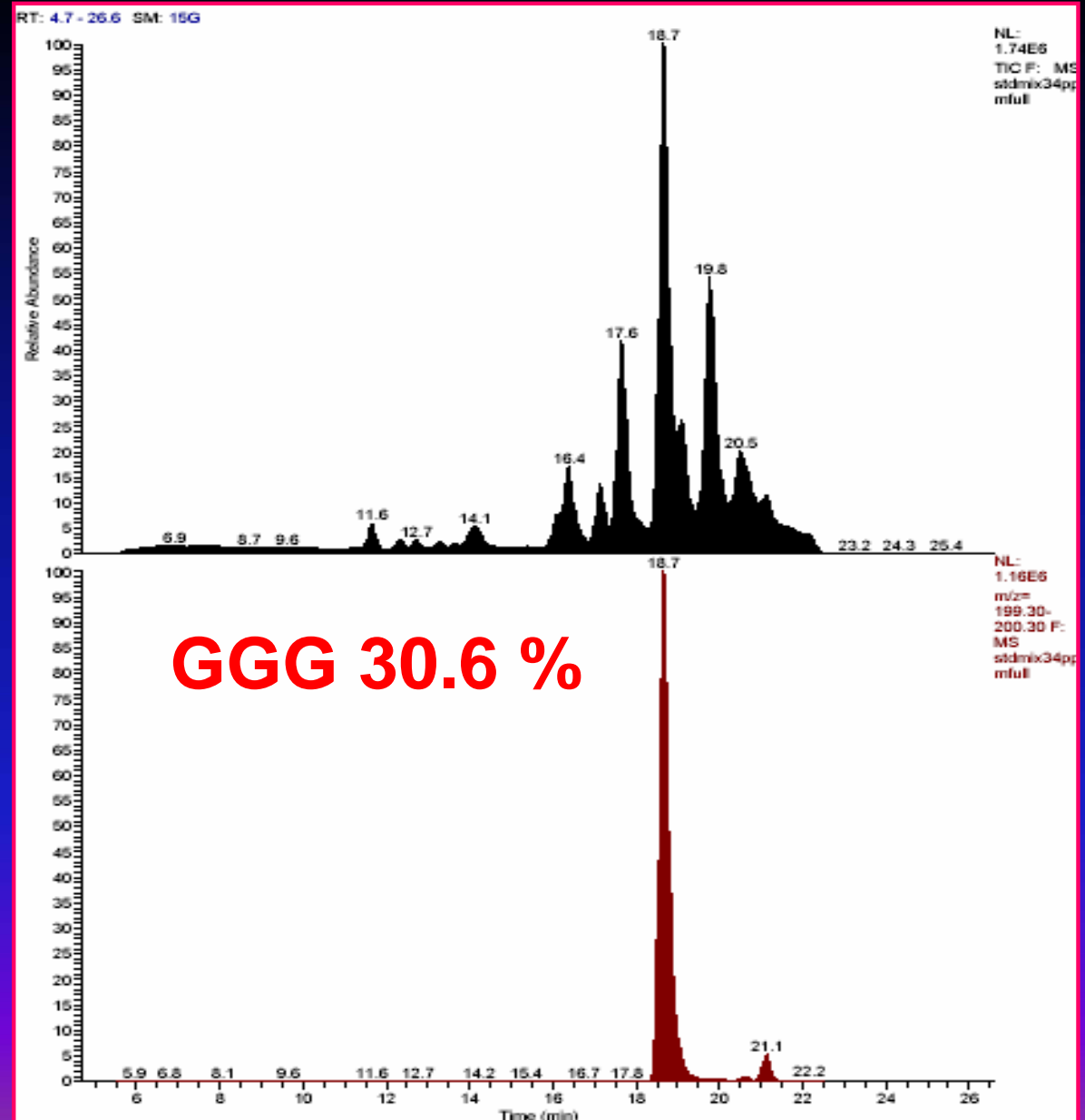
Two approaches may be applicable:

- 1. Development of a 'total residue' method by conversion to a single compound.
- 2. **The choice of a major component as a 'marker', with the inclusion of a correction factor to give the total residue.**

STANDARD ANALITICO

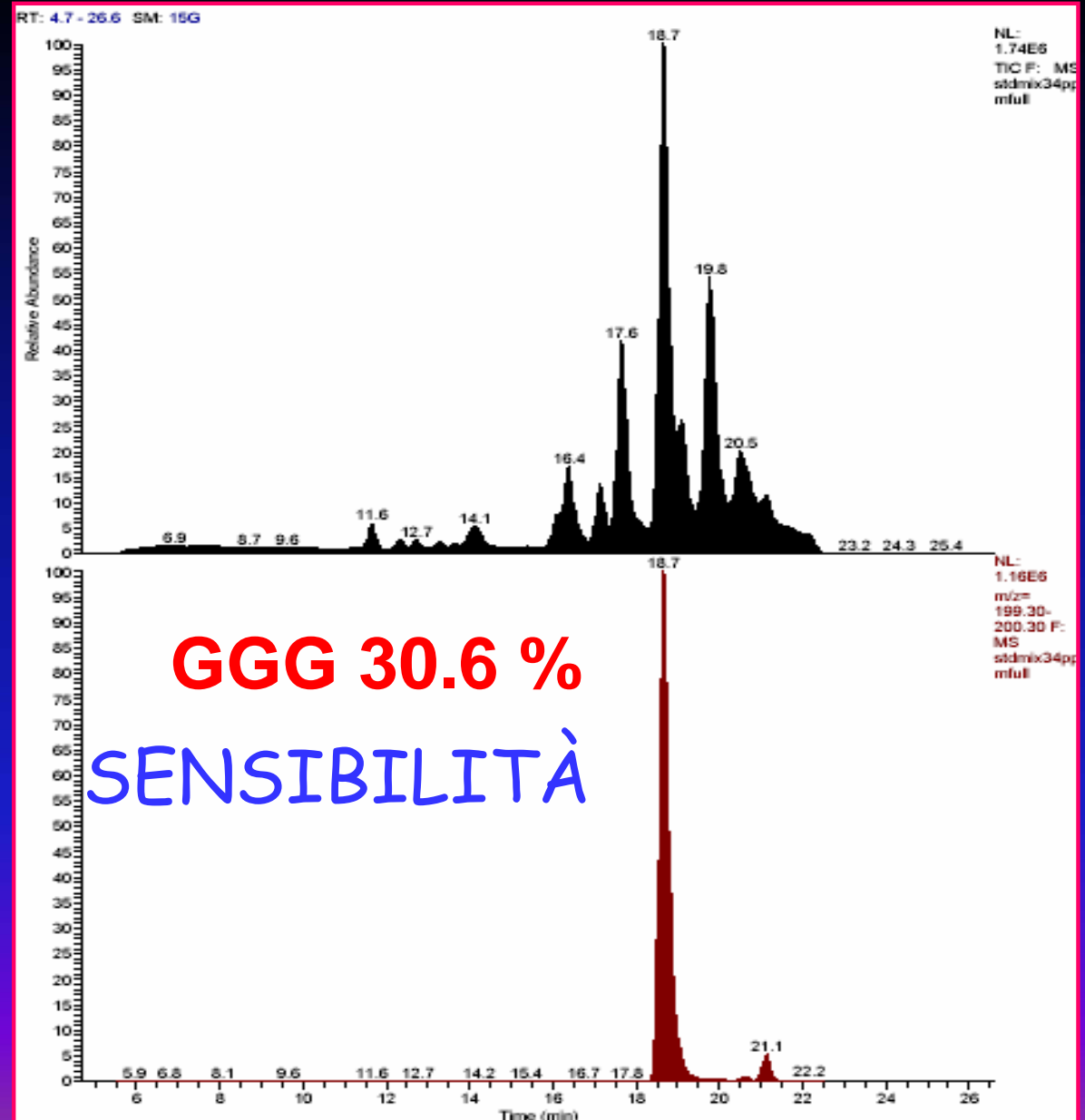
Marker = GGG

FULL SCAN 100 - 400 uma
IONE ESTRATTO GGG



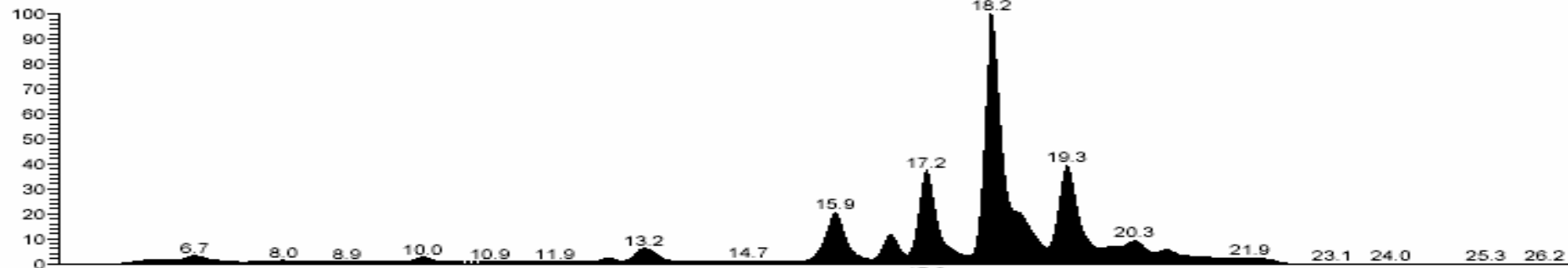
STANDARD ANALITICO

Marker = GGG

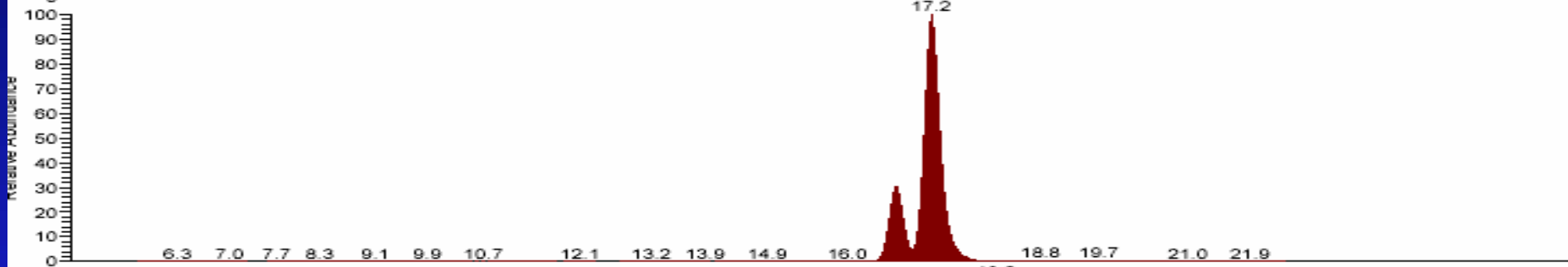


FULL SCAN 100 - 400 uma
IONE ESTRATTO GGG

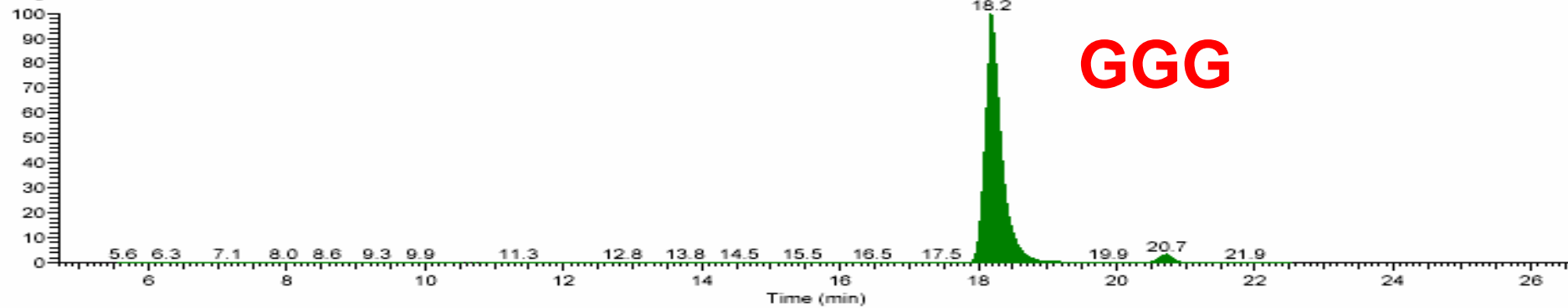
T: 4.7 - 26.6 SM: 15G



NL:
2.59E6
TIC F: MS
MISCELAST
ANDARDGU
AZATINA

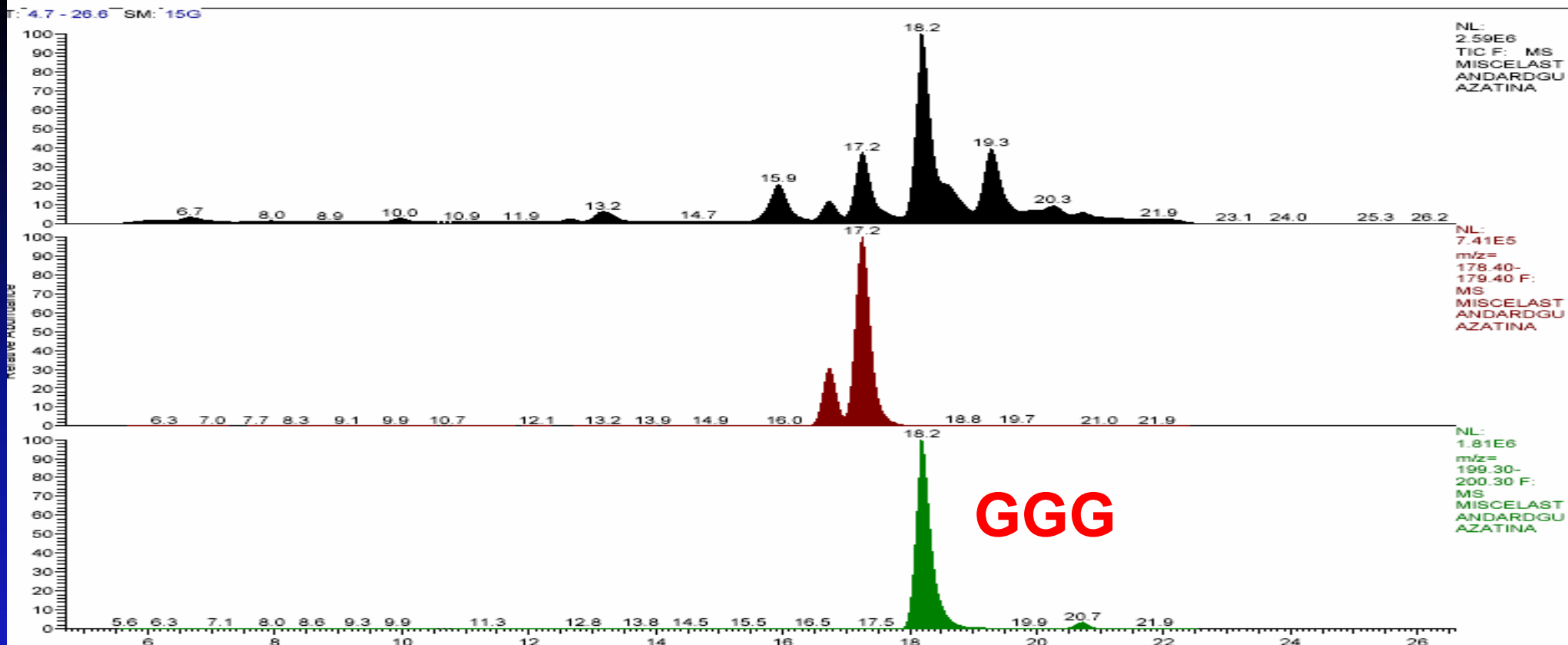


NL:
7.41E5
m/z=
178.40-
179.40 F:
MS
MISCELAST
ANDARDGU
AZATINA



NL:
1.81E6
m/z=
199.30-
200.30 F:
MS
MISCELAST
ANDARDGU
AZATINA

GGG



WASTE

WASTE

SOURCE

17,5 -19 min

30 min

ION TRAP EVENTS

IONI
QUALIFICATORI

CHE ASSICURI

SCAN EVENT 1



SELETTIVITÀ

ION TRAP EVENTS

IONI
QUALIFICATORI

CHE ASSICURI

SCAN EVENT 1



SELETTIVITÀ

SCAN EVENT 2



SENSIBILITÀ

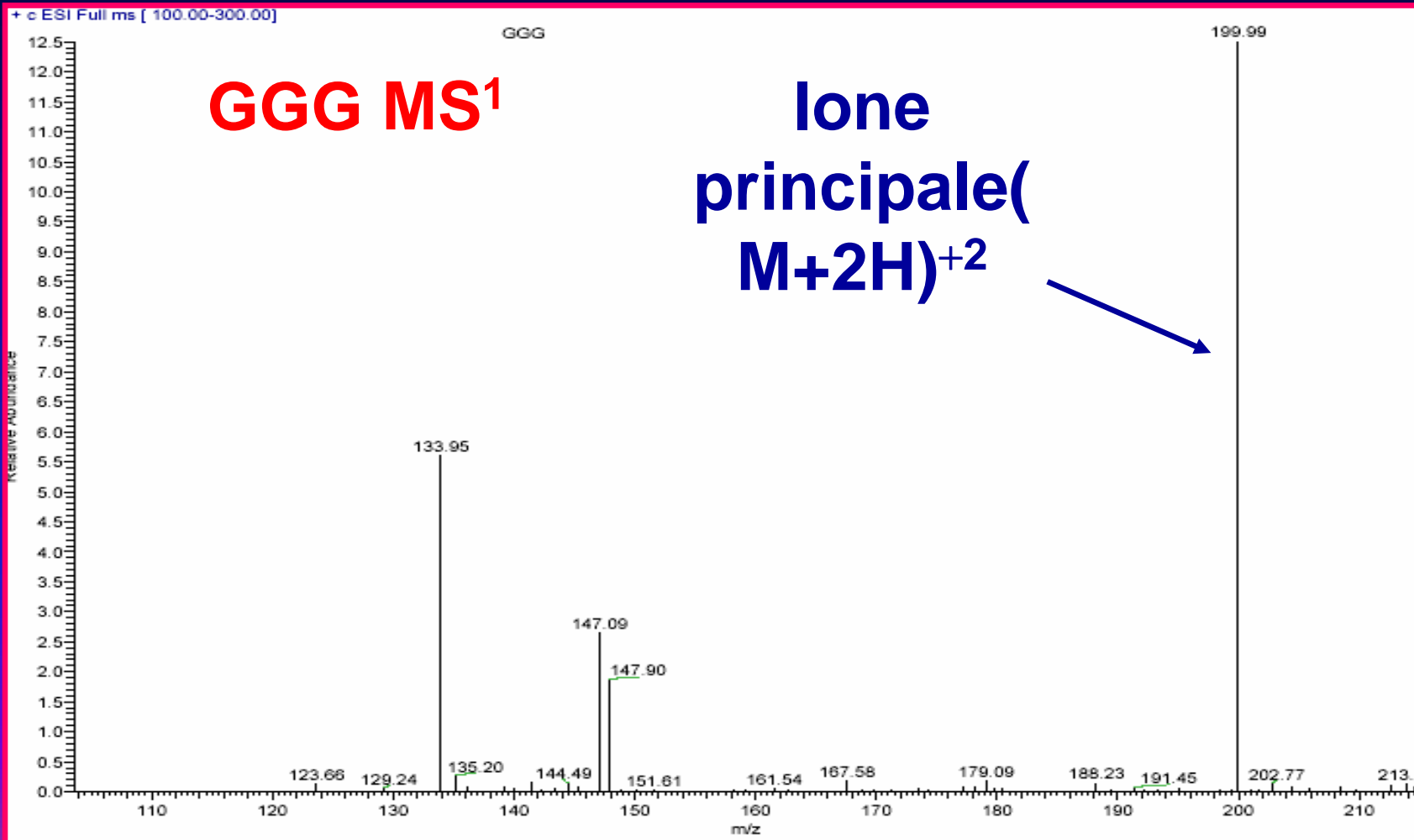
IONE
QUANTIFICATORE

SCAN EVENT 1



SELETTIVITÀ

INDIVIDUAZIONE DI IONI QUALIFICATORI



GGG MS¹

**lone
principale(
M+2H)⁺²**

200

C.E. 42 %



GGG MS²

**lone
principale(
M+2H)⁺²**

179

GGG MS¹

lone
principale(
M+2H)⁺²

200

C.E. 42 %



GGG MS²

lone
principale(
M+2H)⁺²

179

C.E. 36 %



IONI
QUALIFICATORI

158

170

GGG MS³

SCAN EVENT 1

GGG MS¹

lone
principale(
M+2H)⁺²

200

C.E. 42 %



GGG MS²

lone
principale(
M+2H)⁺²

179

C.E. 36 %



158

170

GGG MS³

MS³ + CRM

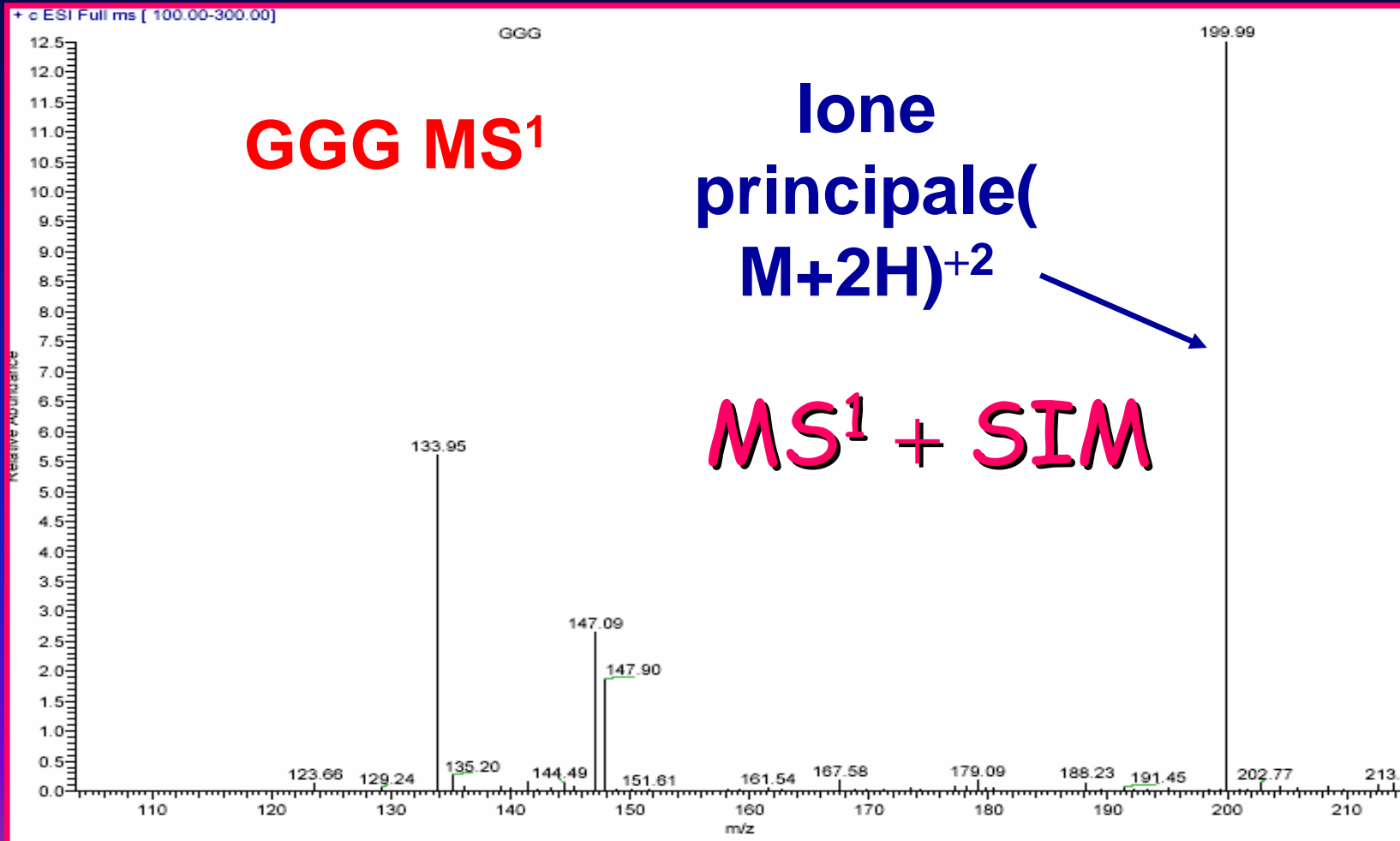
SCAN EVENT 1

SCAN EVENT 2



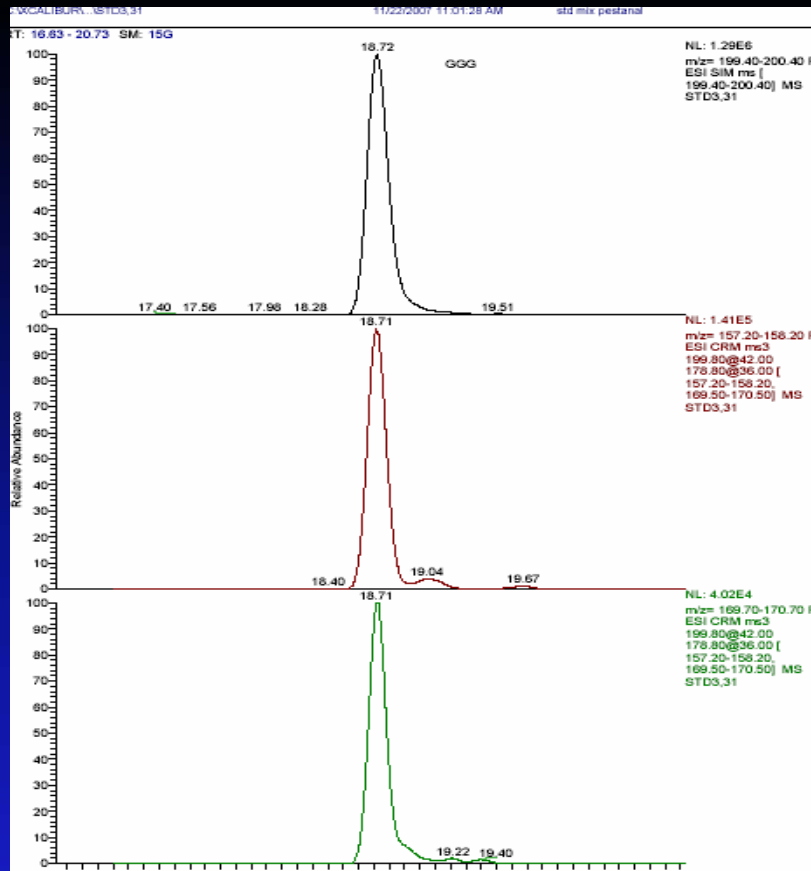
SENSIBILITÀ

INDIVIDUAZIONE IONE QUANTIFICATORE



$MS^1 + SIM$

$MS^3 + CRM$



200

158

170

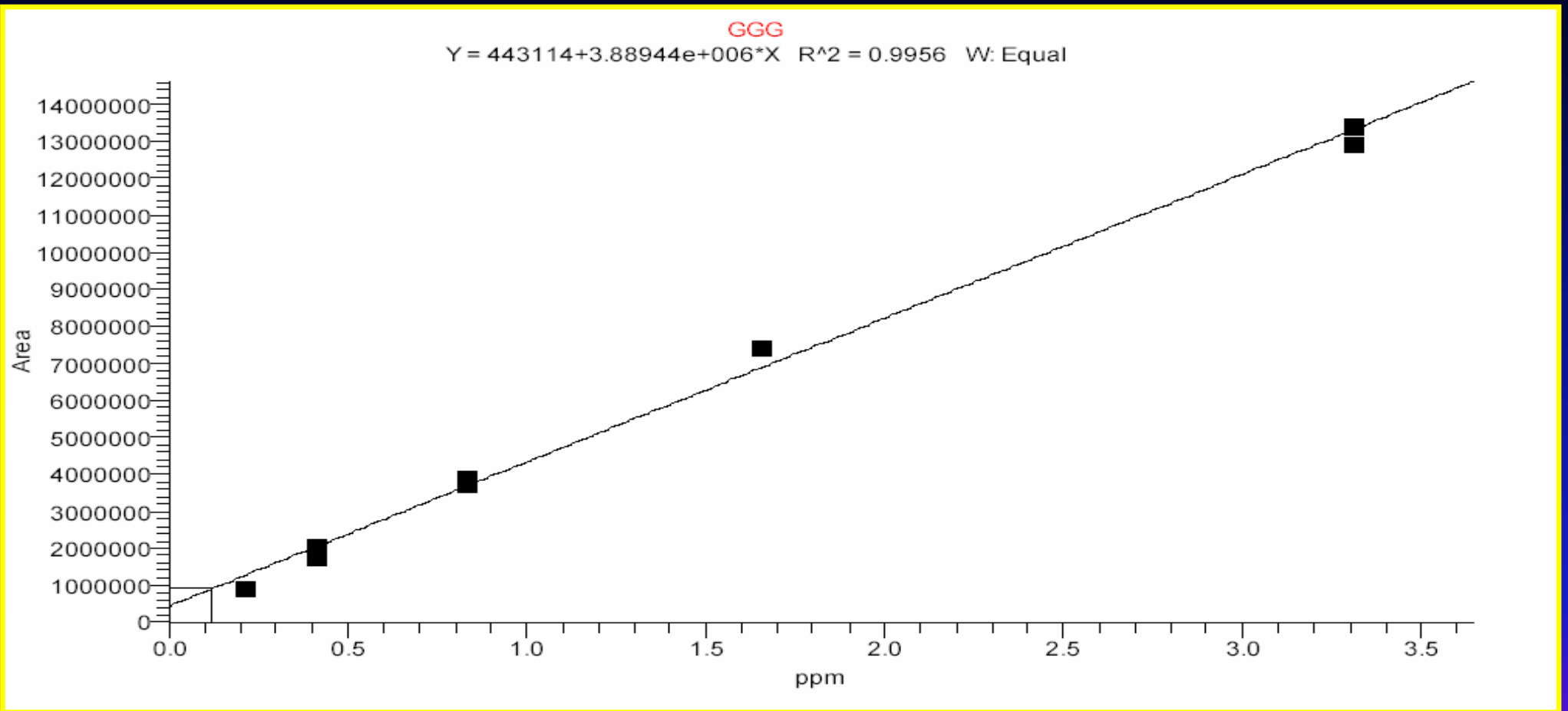
WASTE

WASTE

SOURCE

17,5 -19 min

30 min



RANGE DI CALIBRAZIONE = 0.2 - 3.3 ppm

4° OBIETTIVO

QUANTIFICAZIONE DELLA GUAZATINA NEGLI AGRUMI

4° OBIETTIVO

QUANTIFICAZIONE DELLA GUAZATINA NEGLI AGRUMI

LA MAGGIOR PARTE DEI METODI
RIPORTATI IN LETTERATURA SONO
FINALIZZATI ALLA
DETERMINAZIONE DELLA
GUAZATINA NEI CEREALI.

J. Pesticide Sci. 2, 427–430 (1977)

Gas Chromatographic Determination of Guanidino
Fungicide, Guazatine, in Rice Grain.

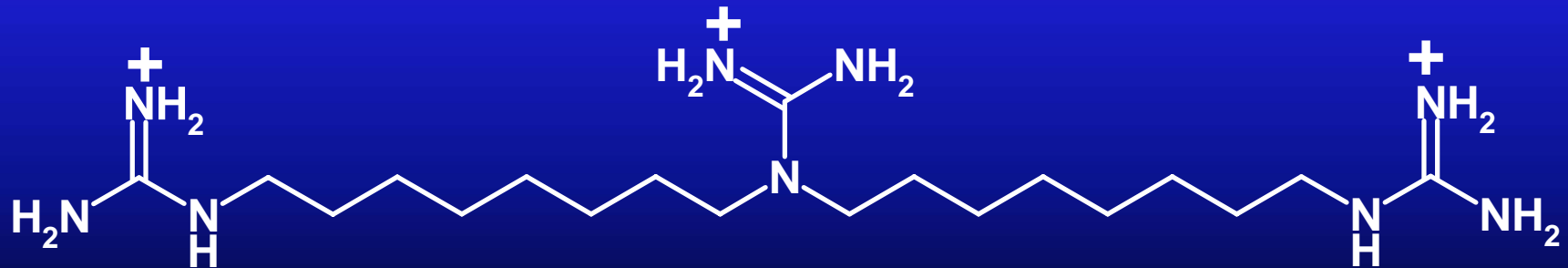
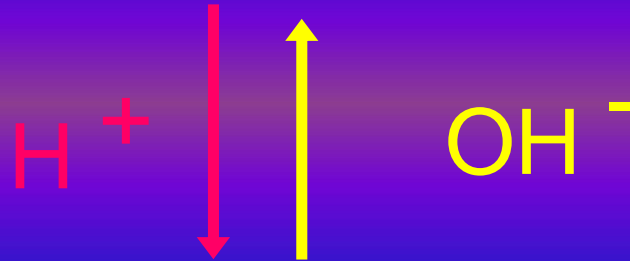
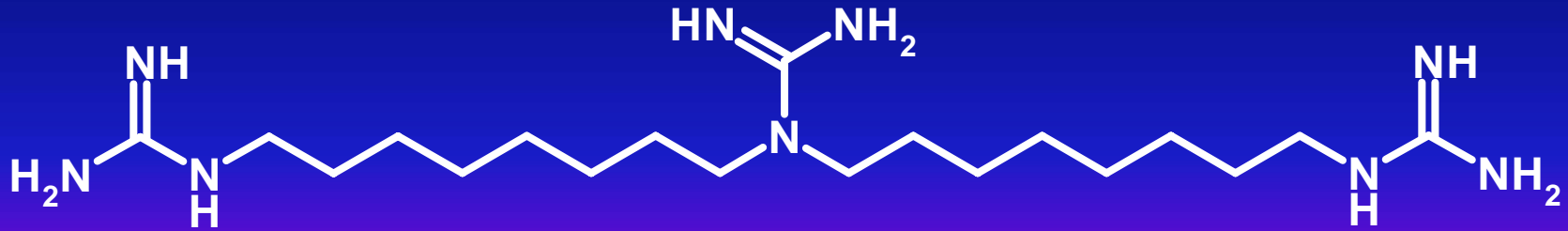
Hiroko KOBAYASHI, Osami MATANO, and Shinko GOTO

Institute of Environmental Toxicology, Kodaira, Tokyo 187, Japan

(Received April 26, 1977)

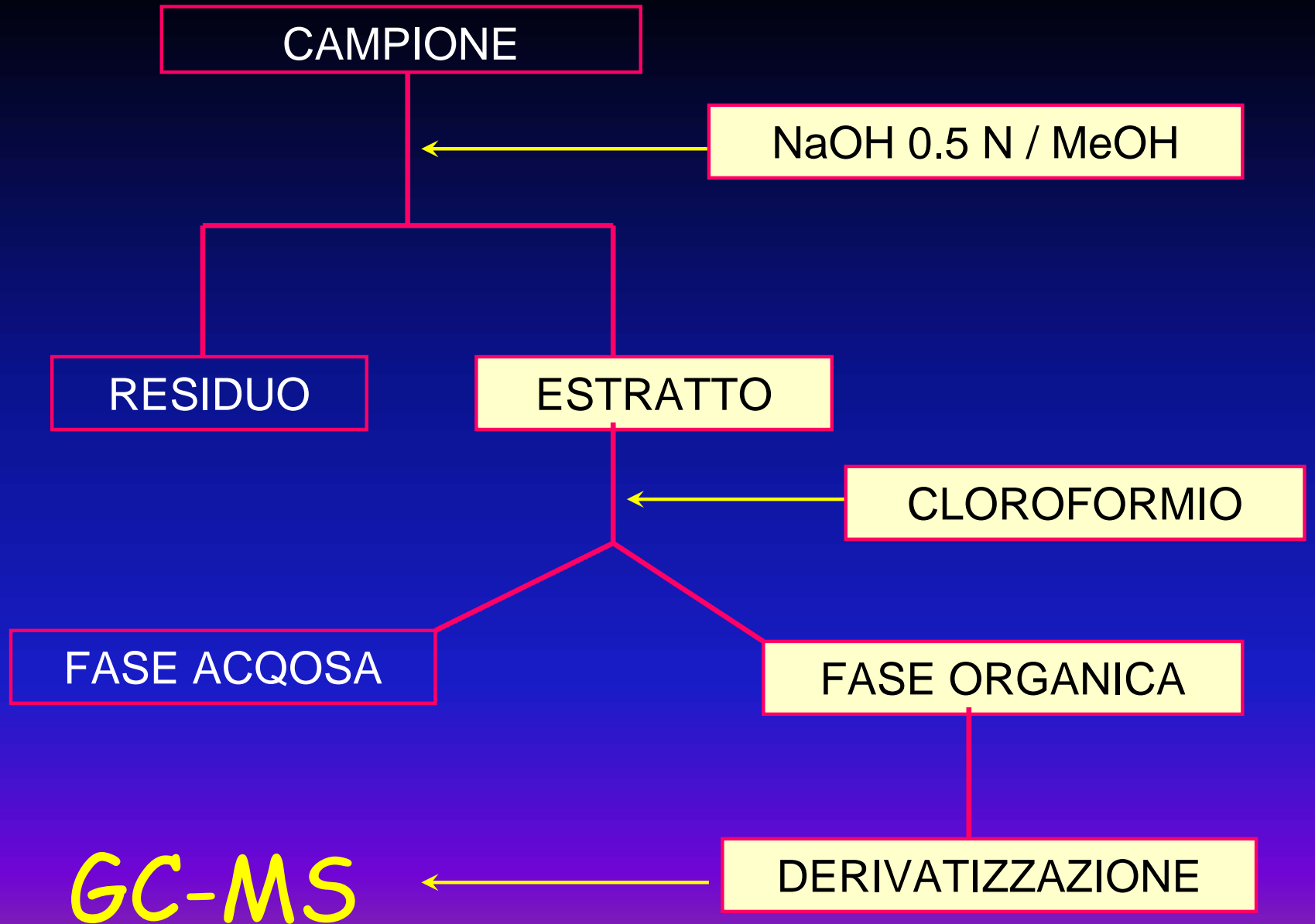
ESTRAZIONE GUAZATINA

SOLUBILE IN FASE
ORGANICA



SOLUBILE IN FASE
ACQUOSA

CEREALI



GC-MS

DERIVATIZZAZIONE CON ESAFLUOROACETIL ACETONE

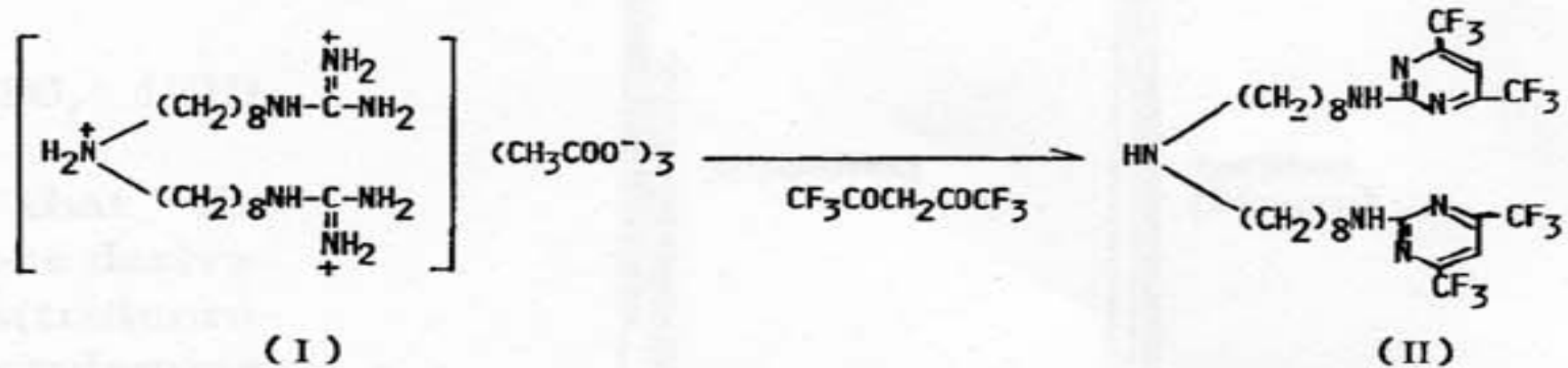


Fig. 1 Derivatization of Guazatine with hexafluoroacetylacetone.

MELE UVA CETRIOLI

J. Pesticide Sci. **9**, 449–453 (1984)

Original Article

Interaction between Guazatine Triacetate and Fructose in Basic Media

Hiroko KOBAYASHI, Osami MATANO and Shinko GOTO

*Chemistry Division, Institute of Environmental Toxicology,
Suzuki-cho, Kodaira 187, Japan*

(Received December 15, 1983)

Constituents in apple and grape which bound guazatine were surveyed, and interactions between the constituents and guazatine were studied. Fructose was a responsible constituent for the binding in basic media, and the active sites of fructose for guazatine were the hydroxyl and carbonyl groups on C₁- and C₂-position.

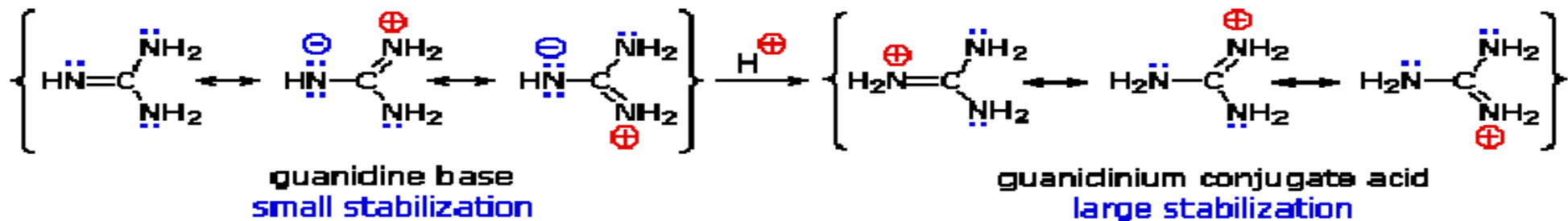
PER LE MATRICI AD ALTO CONTENUTO
DI FRUTTOSIO OCCORRONO DEI
REAGENTI DI RILASCIO

REAGENTI DI RILASCIO MELE

Table 2 Effect of releasing reagents on extraction of guazatine triacetate from apple sample.

Additive	Amount of reagent g (mm)	n	Recovery (%) ^{a)}
None	—	4	1.9 ± 0.5
EDTA-2Na	1 (27)	2	0.8
Thiourea	1 (13)	2	12.7
Thiourea	4 (52)	4	44.6 ± 13.8
Guanidine-HCl	1 (10)	6	87.3 ± 5.5
Methylguanidine-HCl	1 (9)	3	87.7 ± 7.6
Glycocyanine	1 (8.5)	3	46.1 ± 7.6
Dicyandiamide	1 (12)	3	61.6 ± 4.6
Dicyandiamidine sulfate	1 (3)	3	5.0 ± 1.1

GUANIDINA CLOROIDRATO



La **guanidina** è una base molto forte.

La sua notevole basicità viene attribuita al fatto che la carica positiva dello ione guanidinio, che si forma per protonazione, è delocalizzata in ugual misura su tre atomi di azoto.

Original Article

Effect of Guanidine Hydrochloride on the Recovery of Guazatine Triacetate from Vegetables and Fruits

Hiroko KOBAYASHI, Osami MATANO and Shinko GOTO

*Chemistry Division, Institute of Environmental Toxicology,
Suzuki-cho, Kodaira, Tokyo 187, Japan*

(Received April 23, 1982)

Effect of guanidine hydrochloride on the residue analysis of guazatine triacetate [1,1'-iminodi(octamethylene)diguanidine triacetate] is described. Guazatine triacetate residue in vegetables and fruits was extracted with 2N NaOH in MeOH with or without guanidine hydrochloride. The extract was transferred into chloroform and reacted with hexafluoroacetylacetone (HFAA) to form the bis(trifluoromethyl)pyrimidine derivative, which was determined by a gas chromatograph equipped with N-P FID. The value of recovery ($87.3 \pm 5.5\%$) of guazatine triacetate (20 ppm) from the guanidine hydrochloride-treated apple was significantly greater ($p < 0.001$) than that ($1.9 \pm 0.5\%$) from untreated apple. The limit of detection was 0.02 ppm.

REAGENTI DI RILASCIO

CONFRONTO MATRICI

Table 1 Recovery of guazatine triacetate with or without guanidine hydrochloride.

Sample	Sample size (g)	Fortification level (ppm)	Recovery (%) ^{a)}	
			Without guanidine-HCl	With guanidine-HCl
Apple	50	20	1.9 ± 0.5 (4) ^{b)}	87.3 ± 5.5 (6)
Grape	50	20	0.3 ± 0.1 (3) ^{c)}	77.3 ± 8.0 (3)
Cucumber	50	20	42.0 ± 2.8 (3) ^{b)}	89.9 ± 5.6 (3)
Soil	20	50	56.9 ± 4.3 (3) ^{b)}	82.6 ± 4.0 (4)

The recoveries of guazatine triacetate from apple and grape were drastically lower than those from cucumber and soil (Table 1). These differences of recovery among plant species may be dependent on the presence of certain plant-components interfering with the recovery of guazatine.

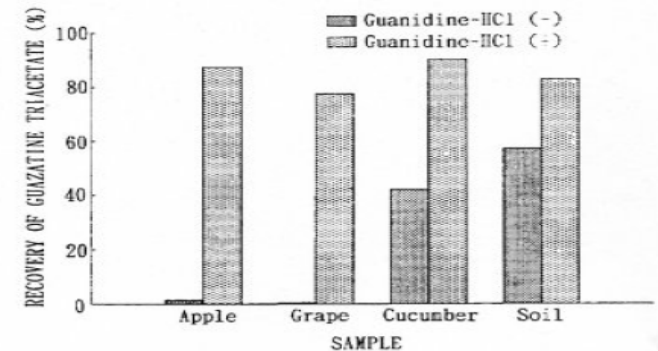
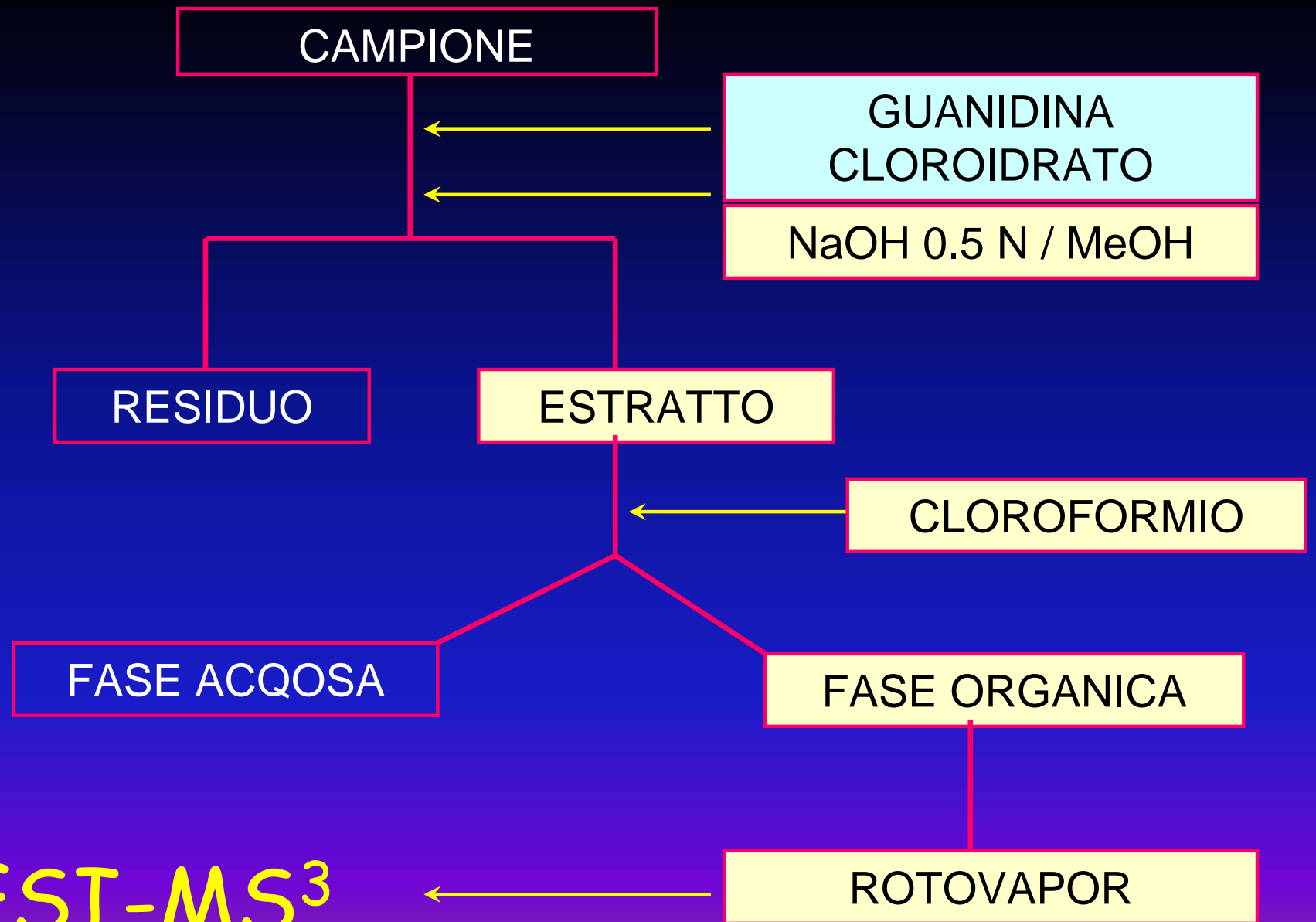


Fig. 10 Recovery of guazatine triacetate from samples added with or without guanidine hydrochloride.

ARANCE

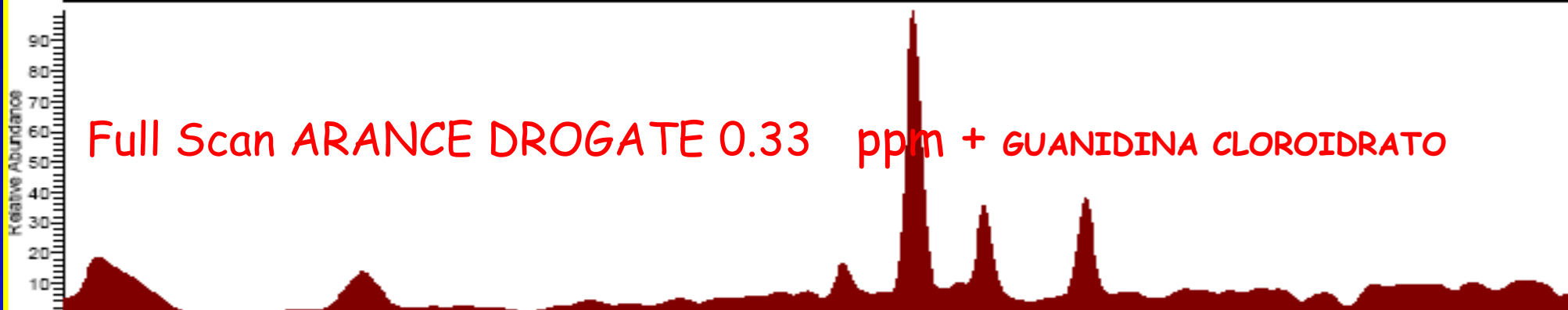


HPLC-ESI-MS³

Full Scan

ARANCE BIANCO

NL: 1.93E5
TIC F: MS
BIANCOAR
ANCE



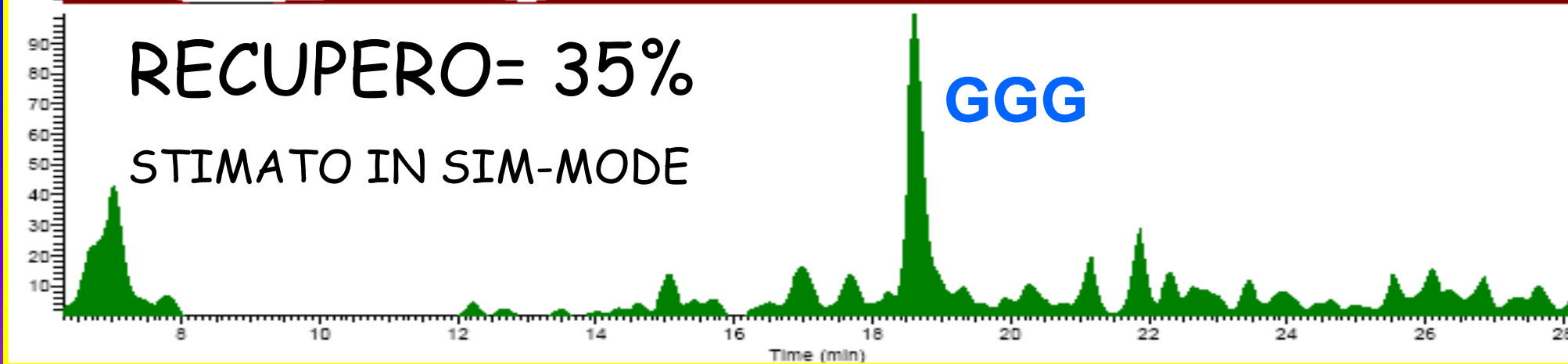
Full Scan ARANCE DROGATE 0.33 ppm + GUANIDINA CLORIDRATO

NL: 2.20E5
TIC F: MS
ARANCEM
EOHDROG
3,33

RECUPERO= 35%

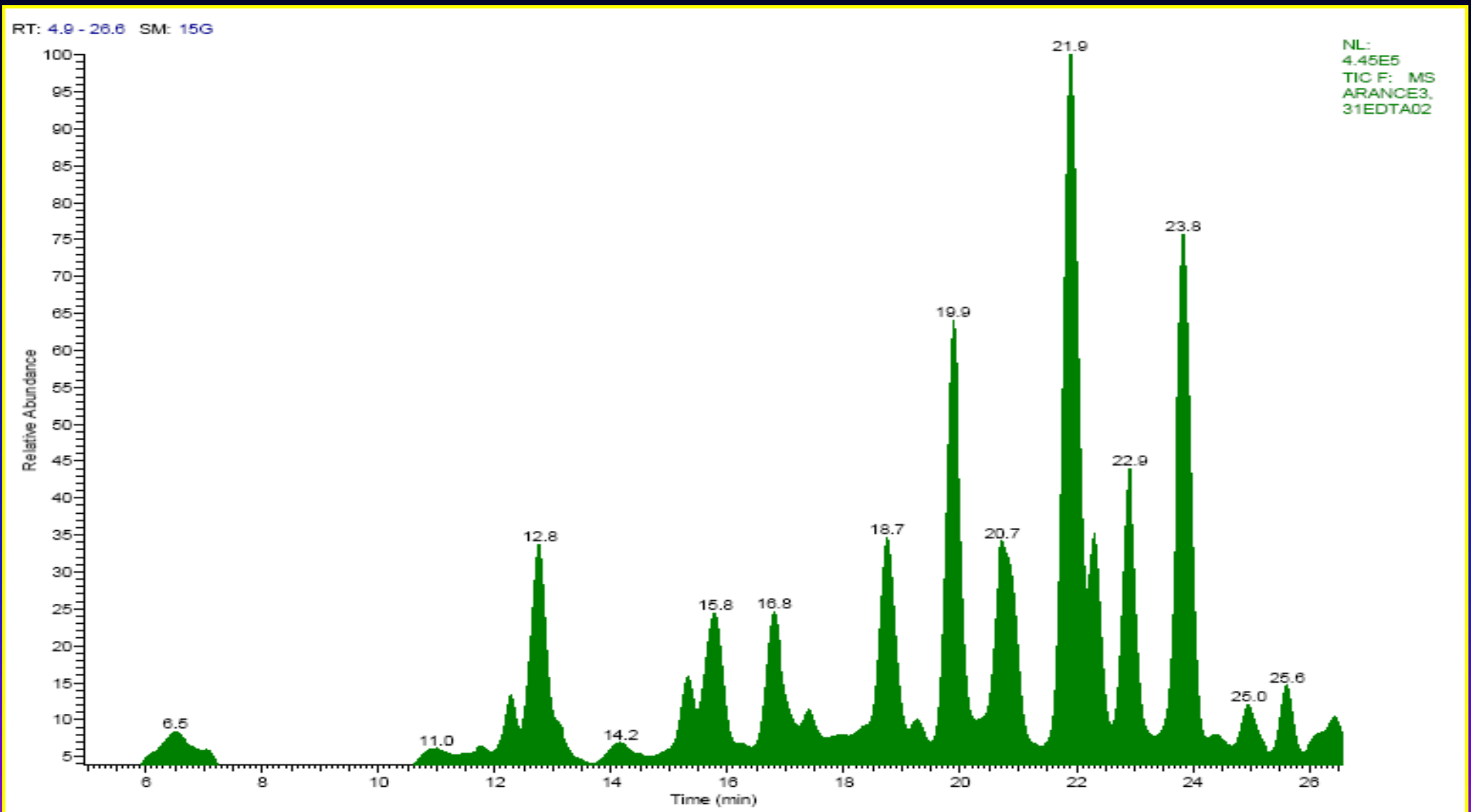
STIMATO IN SIM-MODE

GGG

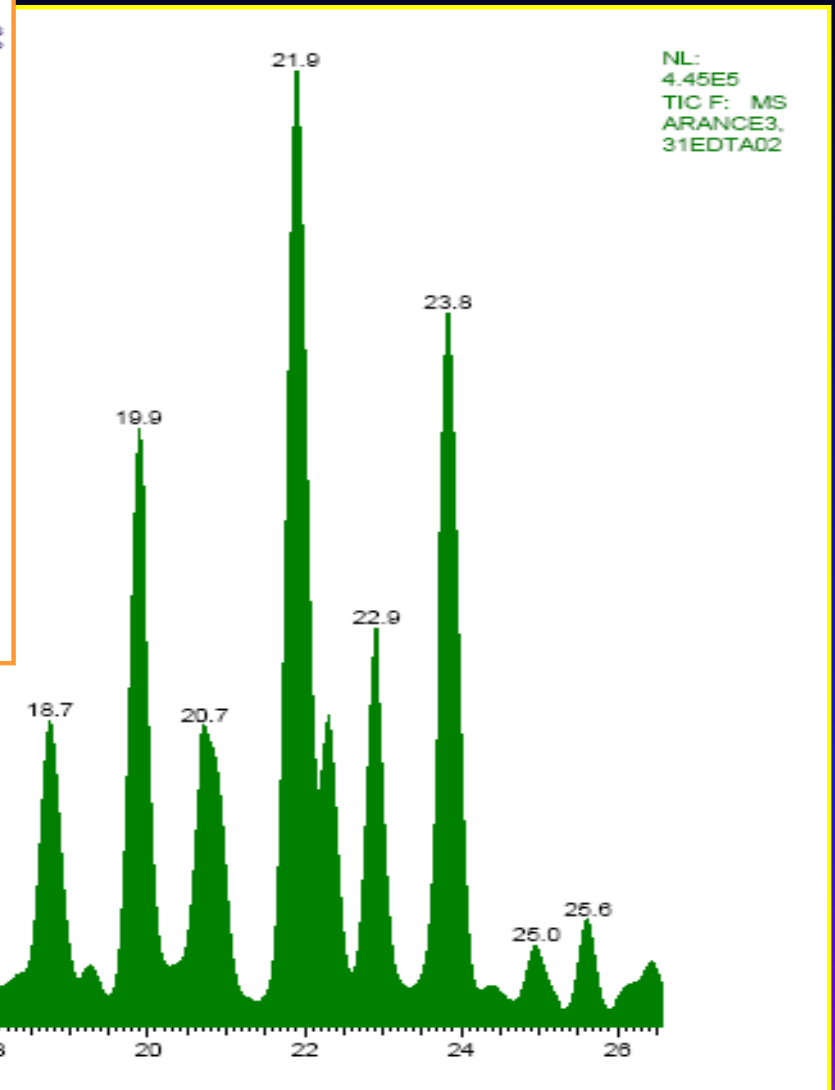
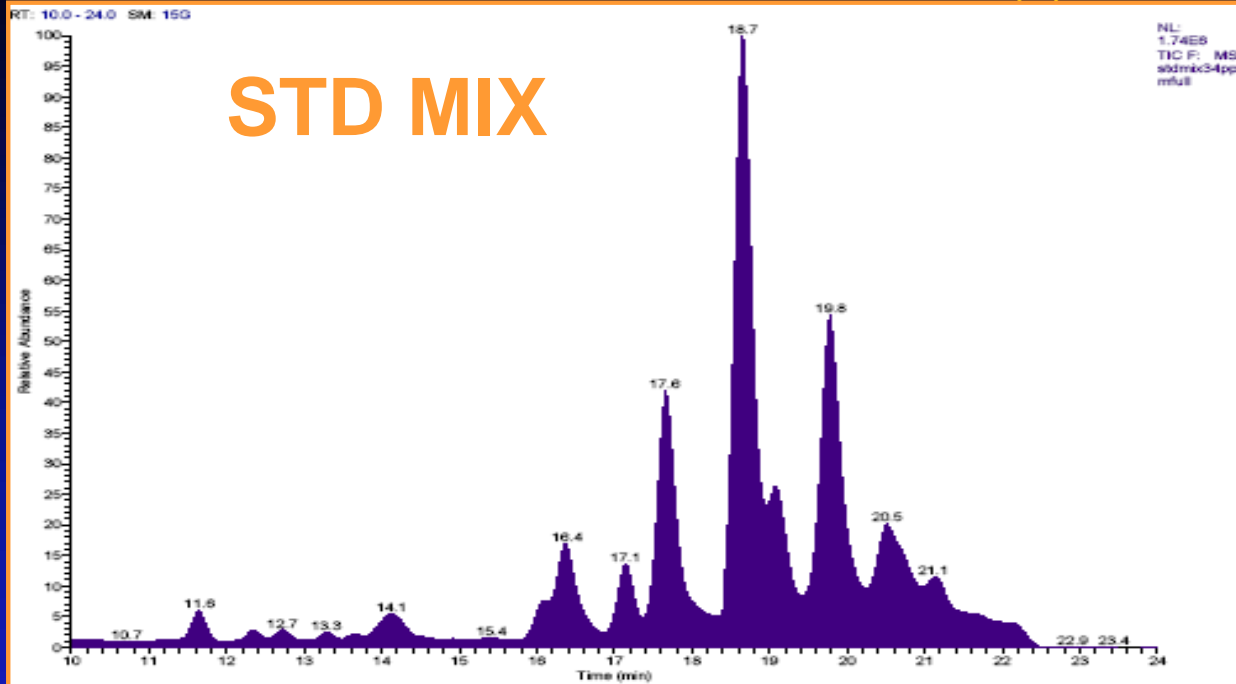


NL: 2.83E3
m/z= 199.40-
200.40 F:
MS
ARANCEM
EOHDROG
3,33

ARANCE DROGATE A 0,33 ppm in presenza di EDTA



ARANCE DROGATE A 0,33 ppm in presenza di EDTA



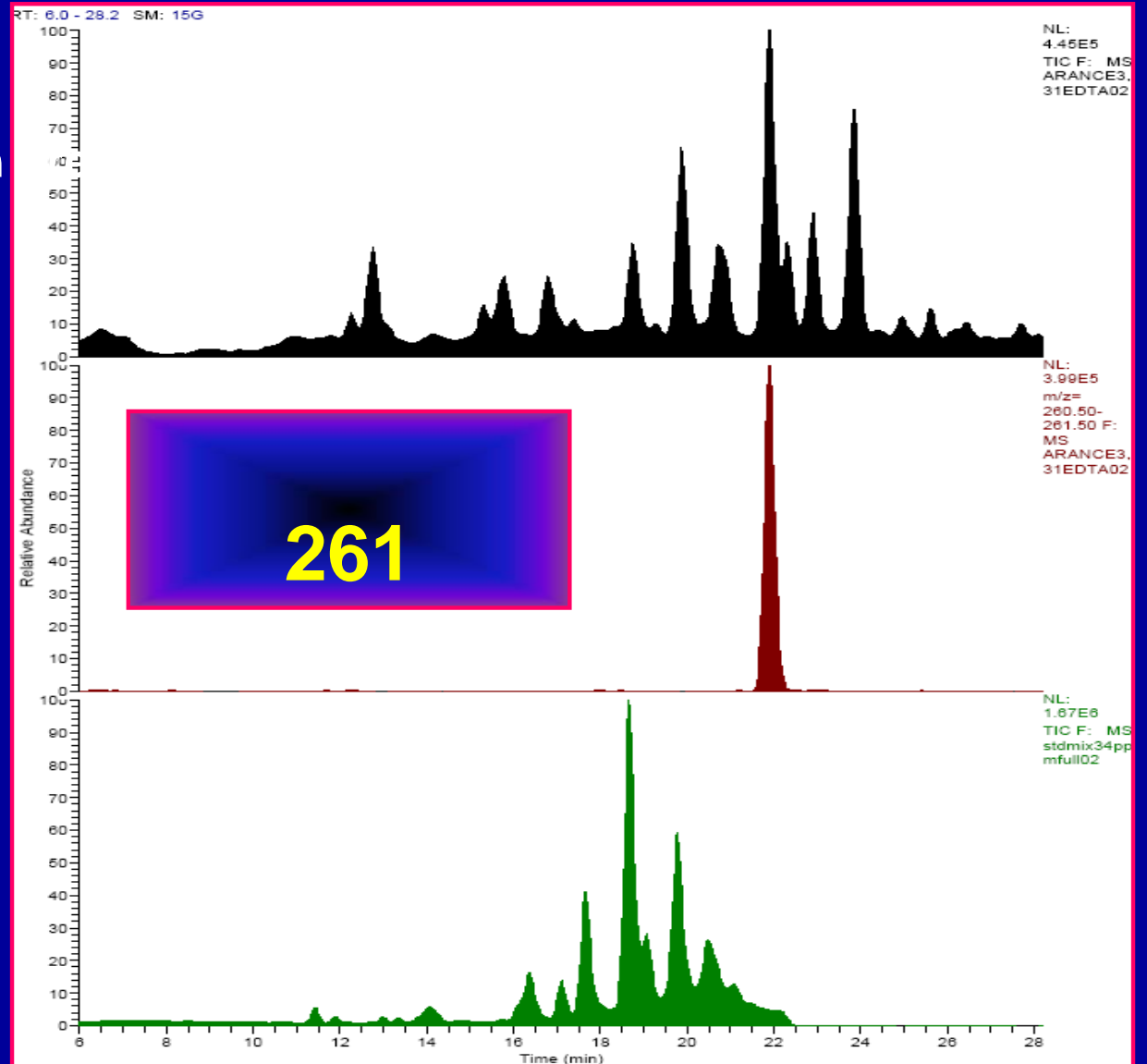
L'EDTA COMPLESSA LE
GUANIDINE

L'EDTA COMPLESSA LE GUANIDINE

ARANCE DROGATE A 0,33 ppm in

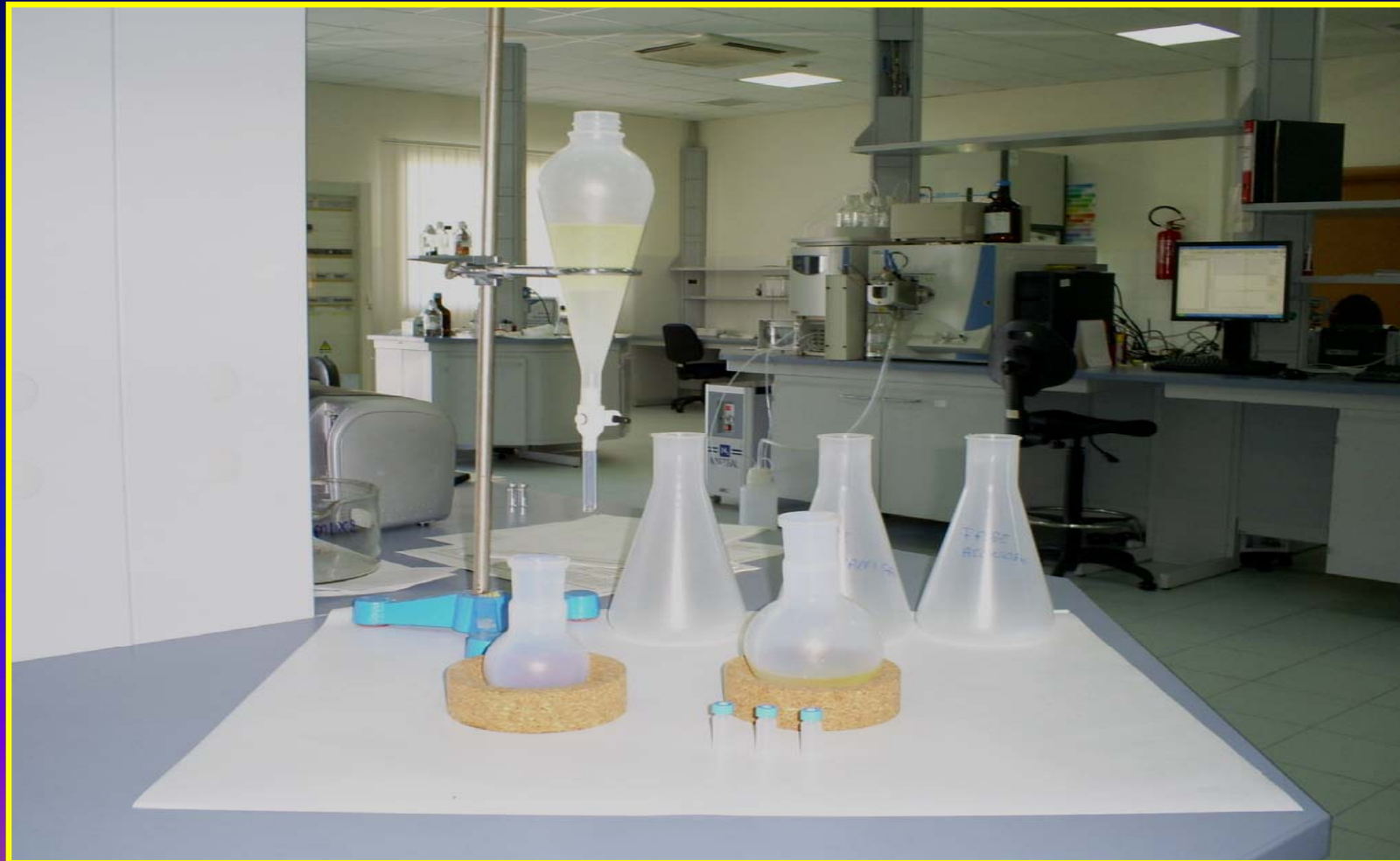
**COMPLESSO
GGG + EDTA**

STD MIX



CONSIDERAZIONI

1. LA GUAZATINA SI DEGRADA A CONTATTO CON IL BOROSILICATO



1. LA GUAZATINA SI DEGRADA A CONTATTO CON IL BOROSILICATO

J. Pesticide Sci. **10**, 91–100 (1985)

Original Article

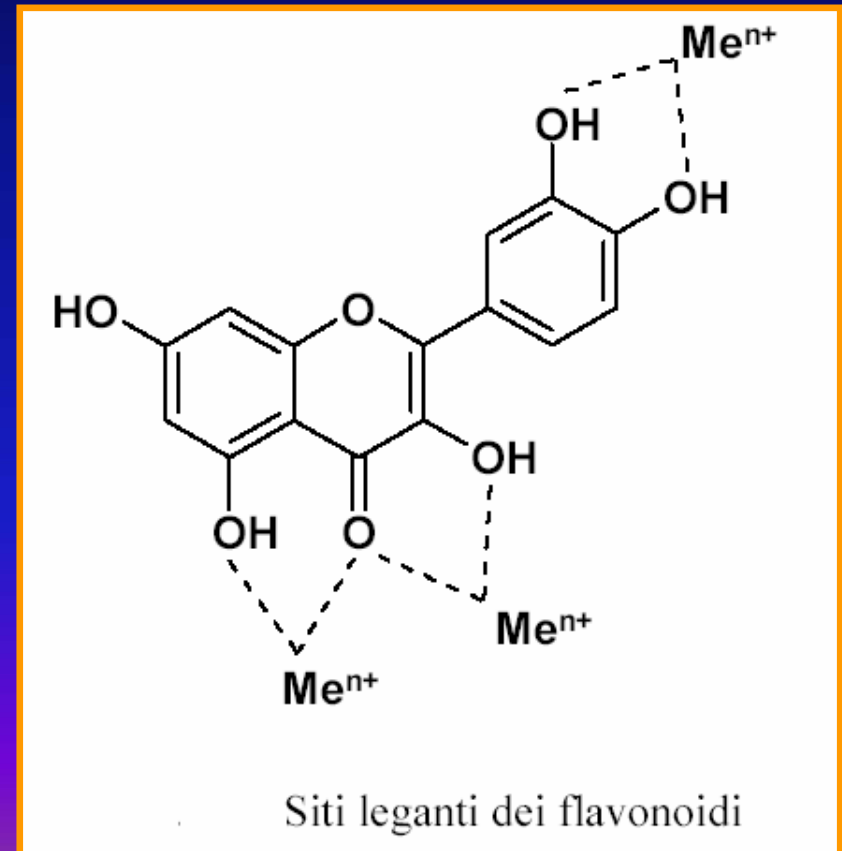
Photolysis of Fungicide Guazatine on Glass Surfaces

Kiyoshi SATO, Yasuhiro KATO, Shin-ichi MAKI, Osami MATANO
and Shinko GOTO

CONSIDERAZIONI

2. LA GUAZATINA VIENE COMPLESSATA DA ALCUNI COSTITUENTI DELLE ARANCE

FLAVONOIDI ?



CONSIDERAZIONI

2. LA GUAZATINA VIENE COMPLESSATA DA ALCUNI COSTITUENTI DELLE ARANCE
3. L'EDTA COMPETE CON TALI COSTITUENTI

PROSSIMO OBIETTIVO

QUANTIFICAZIONE DELLA GUAZATINA
NEGLI AGRUMI MEDIANTE L'IMPIEGO DI
SOLUZIONI STANDARD PREPARATE

CON AGGIUNTA DI EDTA



Regione Siciliana
Assessorato Agricoltura e Foreste
Dipartimento interventi Infrastrutturali
XI Servizio - Servizi allo Sviluppo

Grazie per l'attenzione

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