



ECOMONDO

THE GREEN TECHNOLOGIES EXPO

Prime evidenze analitiche su prove condotte su residui di frantumazione degli autoveicoli

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INTRODUCTION – BACKGROUND DEFINITION



EU Action Plan promotes measures to stimulate Europe's transition towards a circular economy, covering the whole material cycle:

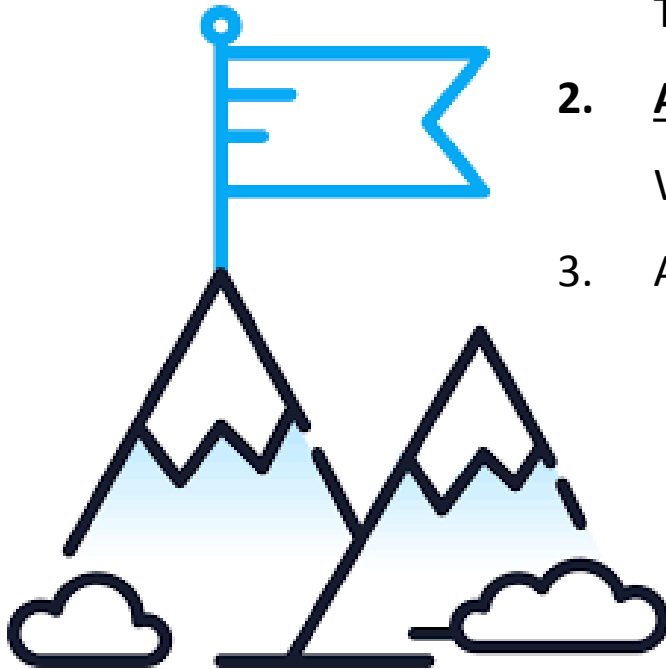
- Sustainable production (promoting the cascade use of by-products)
- **Correct management of residual waste** (e.g. HP14 classification)

POSSIBLE RELEVANT OBSTACLES HINDERING CIRCULARITY

1. Absence of established **End of Waste criteria** for specific type of waste material;
2. Complex Interpretation and Application of **EU Waste Regulation**;
3. **Lab testing requirements not suitable** for the specific material flow

INTRODUCTION – STUDY OBJECTIVES

1. **COMPARE** RESULTS DERIVED FROM THE APPLICATION OF **2 DIFFERENT TESTING PROTOCOLS** TO OBTAIN WATER EXTRACTS TO BE TESTED IN ECOTOXICOLOGICAL BIOASSAYS
2. **ASSESS THE CONSISTENCY** WITH **LANDFILL ACCEPTANCE CRITERIA** for NON HAZARDOUS WASTE (DM 27/09/10 Table 5)
3. ASSESS THE CONSISTENCY WITH ESTABLISHED ECOTOXICOLOGICAL REQUIREMENTS



EN 14735

Preparation of waste samples for ecotoxicity tests
as proposed by Ministry for Solidary and Ecological Transition, France

GHS ANNEX 10

Guidance on Transformation/Dissolution of Metals and Metal compound in aqueous media
as proposed by ISPRA according to
Regulation 1272/2008/EC (CLP) and 440/2008/EC

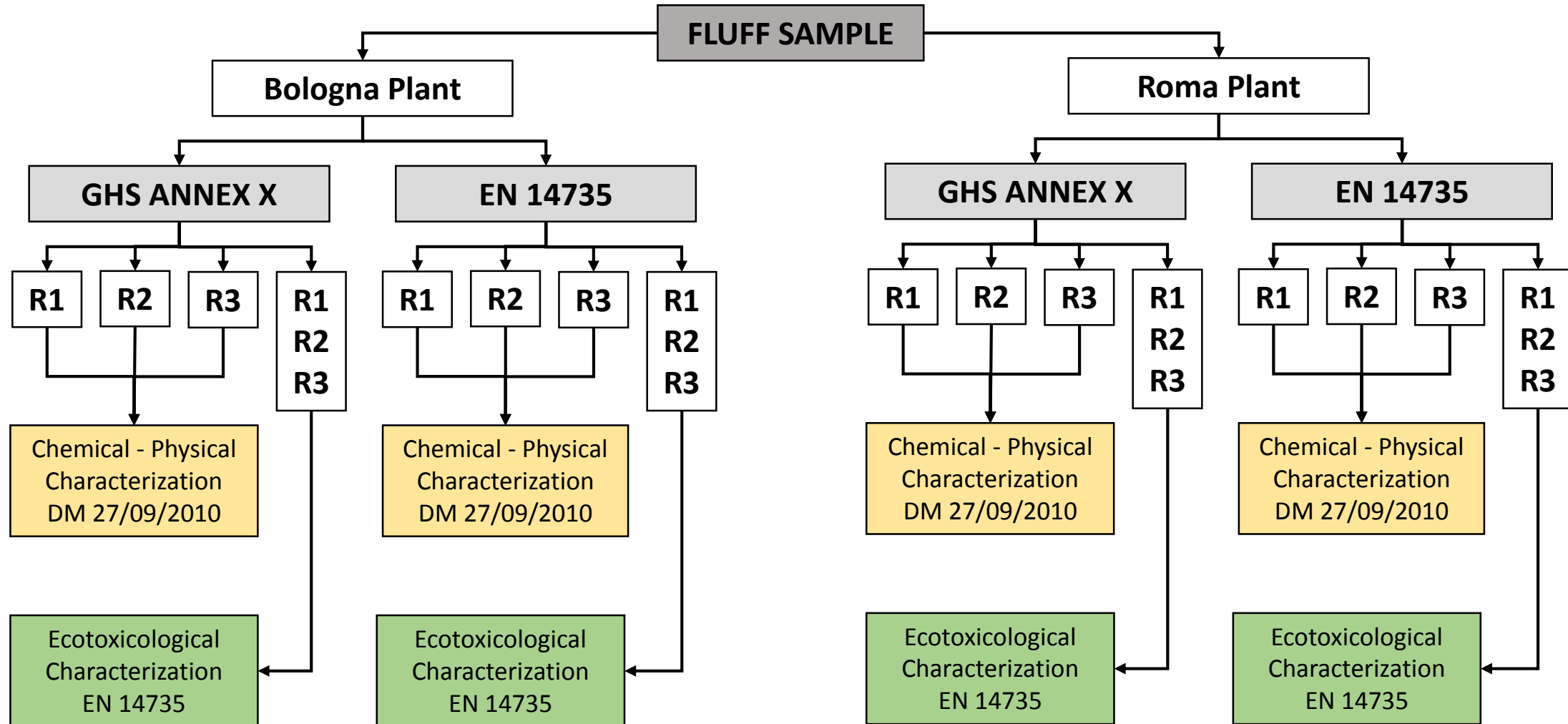
MATERIALS and METHODS – EXPERIMENTAL PROCEDURE

SAMPLING

**I)
LEACHING
TEST**

**II)
CHEMICAL-
PHYSICAL
ANALYSIS**

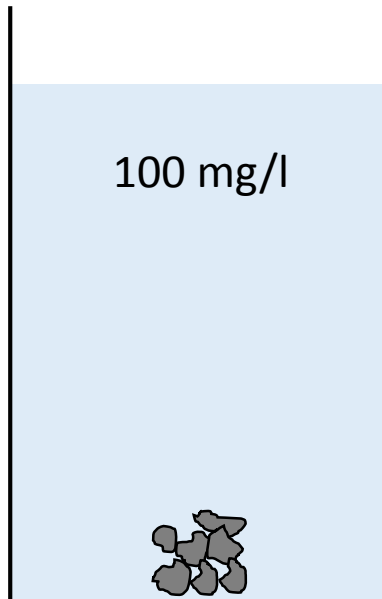
**III)
ECOTOX
ANALYSIS**



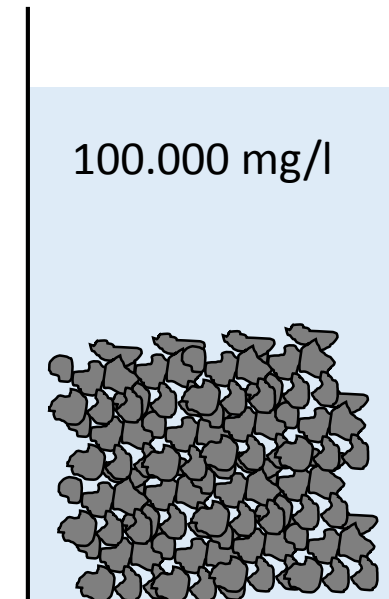
MATERIALS and METHODS REPRESENTATIVENESS

Representativeness is addressed through the sampling design. Representativeness may be considered as the measure of the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition [ANSI/ASQC, 1994].

GHS ANNEX X



EN 14735

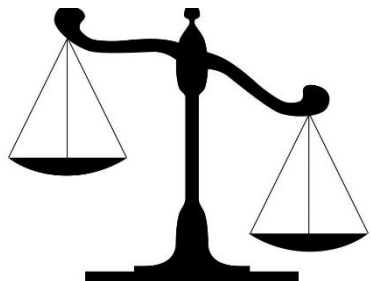


MATERIALS and METHODS

TEST FEATURES INFLUENCE ON CHEMICALS RELEASE IN THE WATER PHASE



	GHS ANNEX X	EN 14735
Particle size reduction	✓	
Test duration	✓	
Liquid-to-Solid Ratio		✓
Test medium	✓	



RESEARCH QUESTION

Which Testing protocols will result to be the
most conservative to determine the potential release of chemicals in the water phase?

MATERIALS and METHODS – CHEMICAL-PHYSICAL ANALYSIS



Landfill Acceptance criteria
used just as reference!

Landfill acceptance criteria
cannot replace waste
hazardousness classification !

Parameter	Limits DM 27/09/10 Tabella 5
EC μS/cm	-
TDS mg/l	10000
pH mg/l	-
Sb mg/l	0,07
As mg/l	0,2
Ba mg/l	10
Cd mg/l	0,1
Cr TOT mg/l	1
Hg mg/l	0,02

Parameter	Limits DM 27/09/10 Tabella 5
Mo mg/l	1
Ni mg/l	1
Pb mg/l	1
Cu mg/l	5
Se mg/l	0,05
Zn mg/l	5
DOC mg/l	100
Chlorides (as Cl⁻) mg/l	2500
Fluorides (as F⁻) mg/l	15
Sulphates (as SO₄²⁻) mg/l	5000

MATERIALS and METHODS – ECOTOXICOLOGICAL BIOASSAYS



1272/2008/EC (CLP)

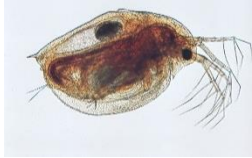
Organism	Standard	Limit
Fish	OCSE/OECD	EC50≤100 mg/l
Crustaceans (Daphnia)	OECD TG 202/C2	EC50≤100 mg/l
Algae	OECD TG 201	EC50≤100 mg/l



- Designed for products
- Fish test difficult to implement
- Scientific community approves EN 14735 for HP14 of WASTE

EN 14735 – Hennebert, 2018

Organism	Standard	Limit
Crustaceans (Daphnia)	EN ISO 6341	EC50≤15,8%
Algae (Pseudokirchnerie lla subcapitata)	EN ISO 8692	EC50≤7,03%
Aquatic Bacteria (Vibrio Fisheri)	EN ISO 11348-3	EC50≤7,95%
Soil Bacteria (Arthrobacter globiformis)	ISO 18187	EC50≤2,25%
Plants (Brassica Rapa)	EN ISO 11269-2	EC50≤13,7%
Soil Invertebrates (Eisenia fetida)	ISO 17512-1	EC50≤3,75%



MATERIALS and METHODS

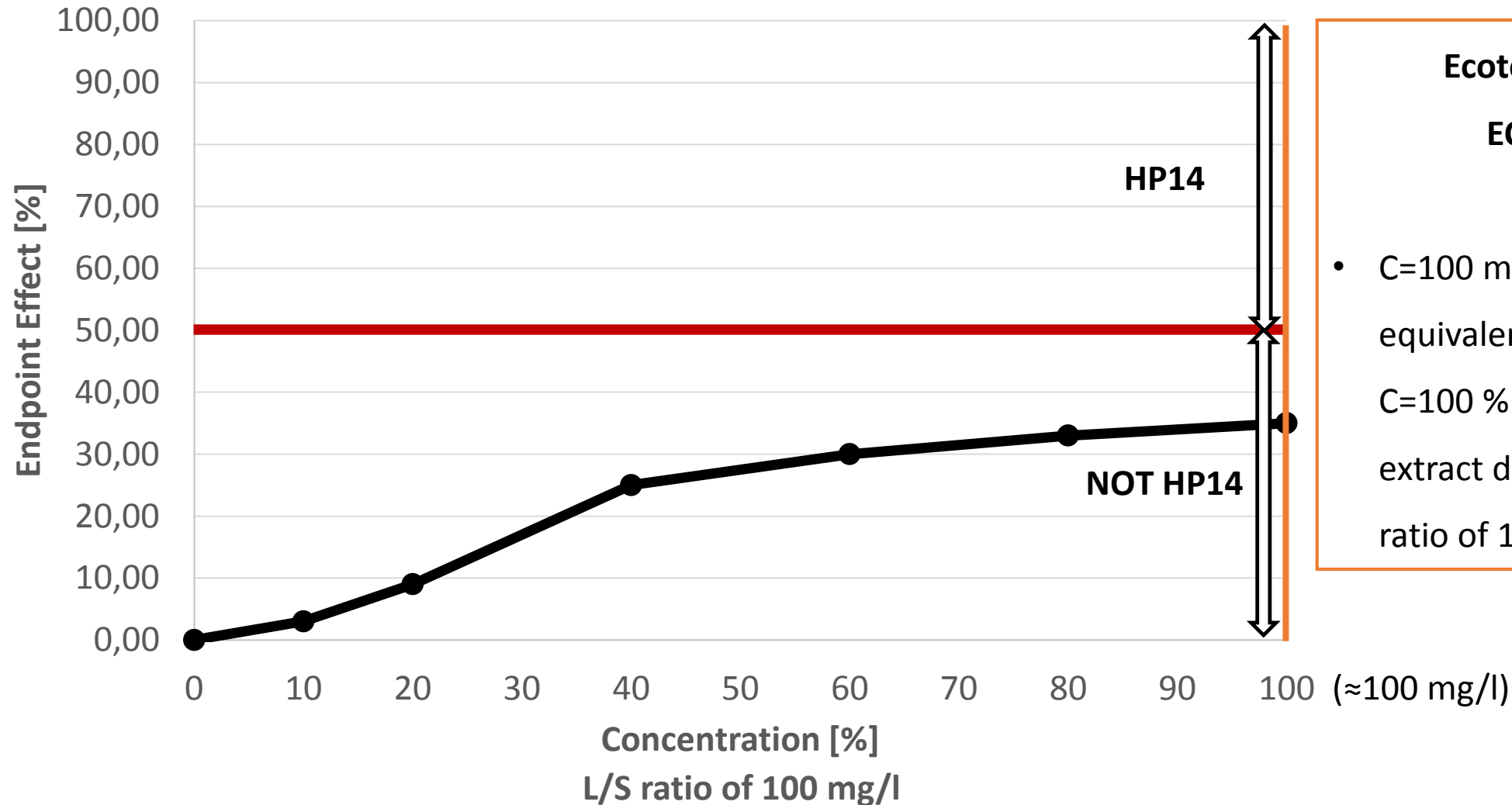
INTERNATIONAL STANDARDS FOR ECOTOX TESTING

	EN ISO 6341	OECD TG 202/C2
Test organism	Daphnids (neonates)	Daphnids (neonates)
Age of test organism	< 24 h	< 24 h
Feeding	None	None
Dilution medium	Freshwater or synthetic reconstituted medium (dechlorinated tap water)	Any water suitable for culturing, natural or reconstituted water
Water hardness	140-275 mg/l CaCO ₃ /L	140-250 mg CaCO ₃ /L
Volume	10 ml	Minimum 10 ml/replicate
Test conditions	(20 ± 2) °C	(20 ± 2) °C
Temperature		
Ph	7 - 8 ± 0,2	6 - 9
Photoperiod	Darkness or 16 h + 8 h light + dark photoperiod	Darkness or 16 of light
Number of containers, number of replicates	5 daphnids per vessel 4 replicates	20/concentration 4 replicates (5 animal each)
Test duration	24 h and/or 48 h	48 h
Validity criteria	- Control mortality ≤ 10 %; - O₂ concentration ≥ 2 mg/l; - sensitivity to K₂Cr₂O₇	≤ 10% individuals immobile or trapped at the surface of the water in control; - Dissolved O₂ ≥ 3 mg/L in all concentrations
Test parameters	Immobilisation	Immobilisation
Endpoints	EC 50	EC 50

- Testing procedures are **comparable**;
- **Minor differences**;
- Further experimental activity needed to **confirm assumptions**



MATERIALS and METHODS – ECOTOXICOLOGICAL LIMITS for CLP



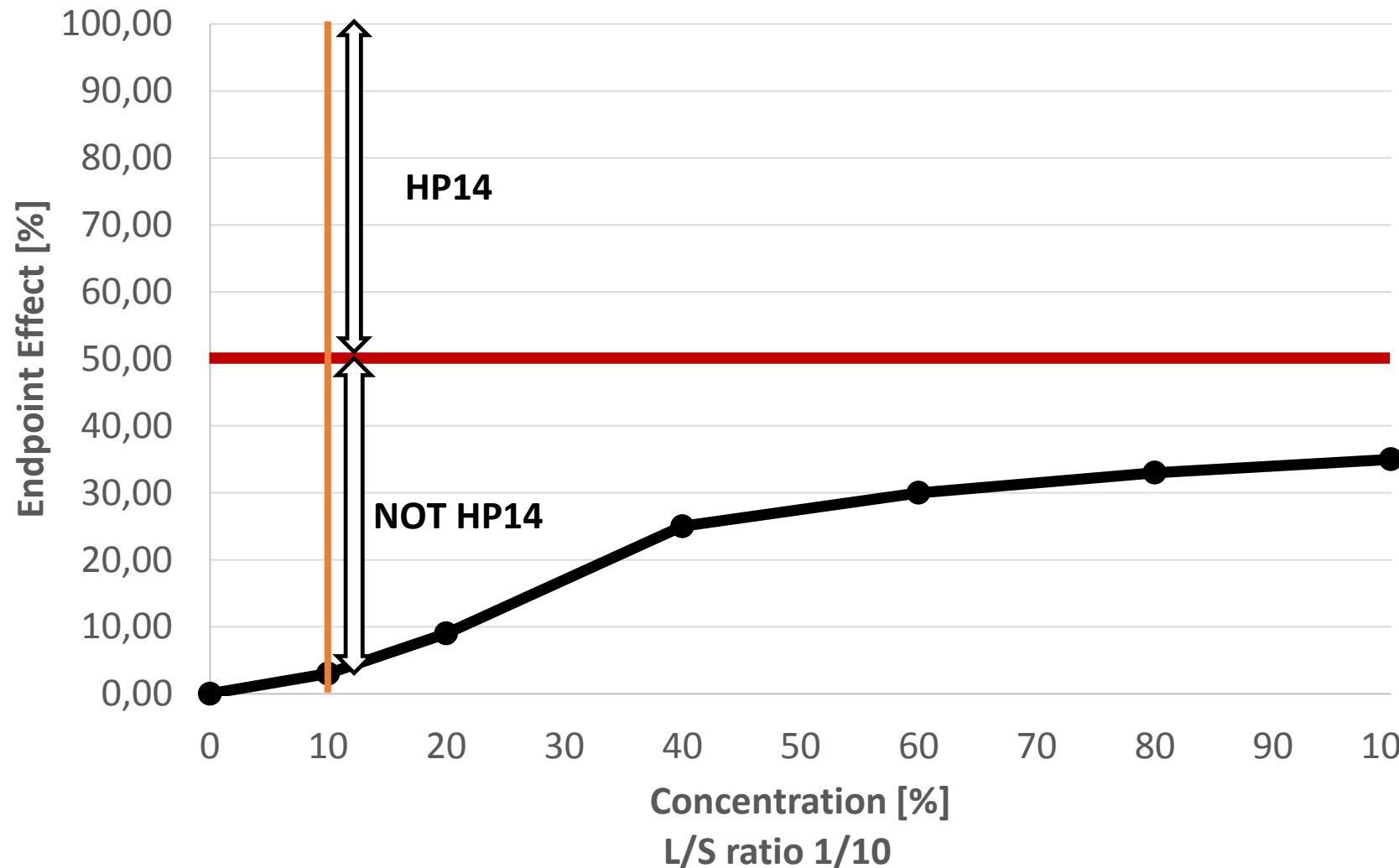
Ecotox Chronic (III) if :

$EC_{50} \leq 100 \text{ mg/l}$

- C=100 mg/l is considered equivalent to a concentration C=100 % (vol/vol) of a water extract derived according to L/S ratio of 100 mg/l.

MATERIALS and METHODS –ECOTOXICOLOGICAL

LIMITS for EN 14735 (Pandard and Rombke, 2014 Hennebert, 2018 , ISS/ISPRA, 2011)



HP14 if :

$EC50 \leq 10 \% (vol/vol)$

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Proposal for a "Harmonized" Strategy for the Assessment of the HP 14 Pro

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JECT Oekotoxikologie GmbH, Flön

(Submitted 6 December 2012; Re-

ABSTRACT

In Europe, the decision whether ("ecotoxic") waste that present classified compounds in the waste substances and mixtures and the waste carried out mainly in the waste between hazardous and non-hazardous both effect concentrations (EC) of tests (3 using waste eluate recommended for the ecotoxicological legal requirements for the ecotoxicity

Keywords: HP 14 property

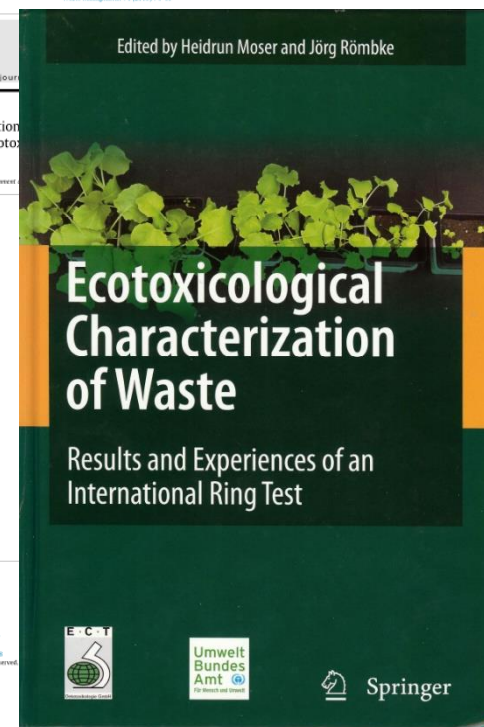
INTRODUCTION

In Europe, the classification of hazardous waste (Commission Decision (EC 2000) amended by Commission Decision (EC 2001)) and the Annex III to the Directive 91/689/EEC (EC 1991) Framework Directive (EC 2006) properties that render waste to be HP 14 property ("ecotoxic") waste: immediate or delayed risks for the environment, recently retained experts as a conclusive factor for more than 80% of hazardous waste. For some of these properties harmful, toxic, corrosive, carcinogenic the evaluation process allowing a waste to be fully defined (EC 2001). EC other properties such as HP 14 established, despite the preponderance of the Framework Directive (EC 2006) for the classification, packaging substances (Annex VI of Commission Decision (EC 1987) and subsequent adapt

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MATERIALS and METHODS – ECOTOX PRELIMINARY ASSESSMENT

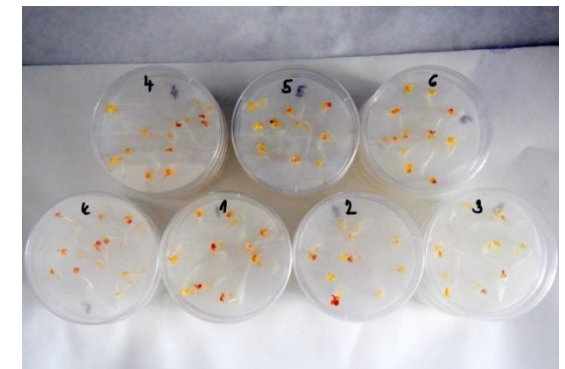
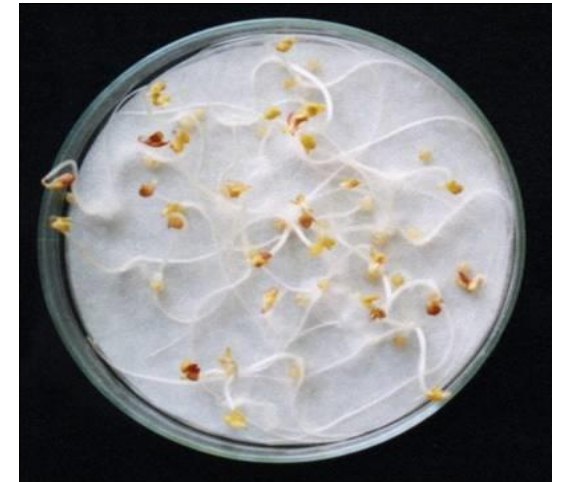


Plants

(Lepidium Sativum)

Seed Germination Bioassay

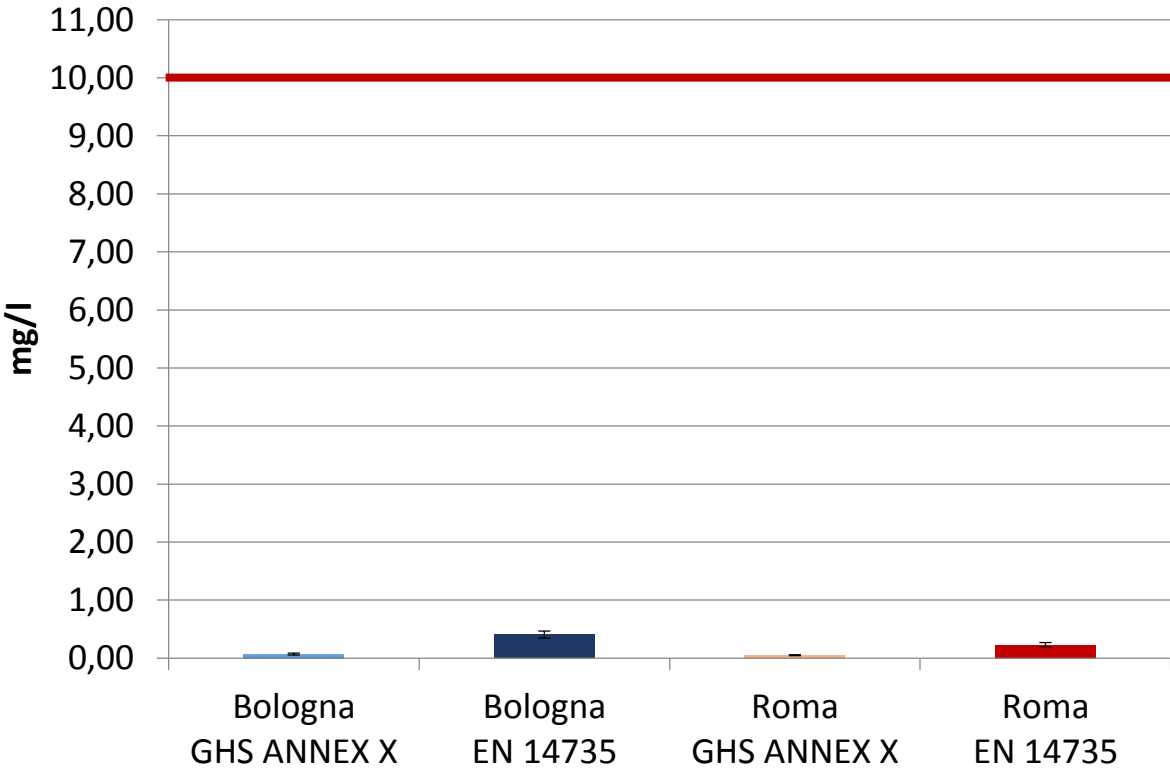
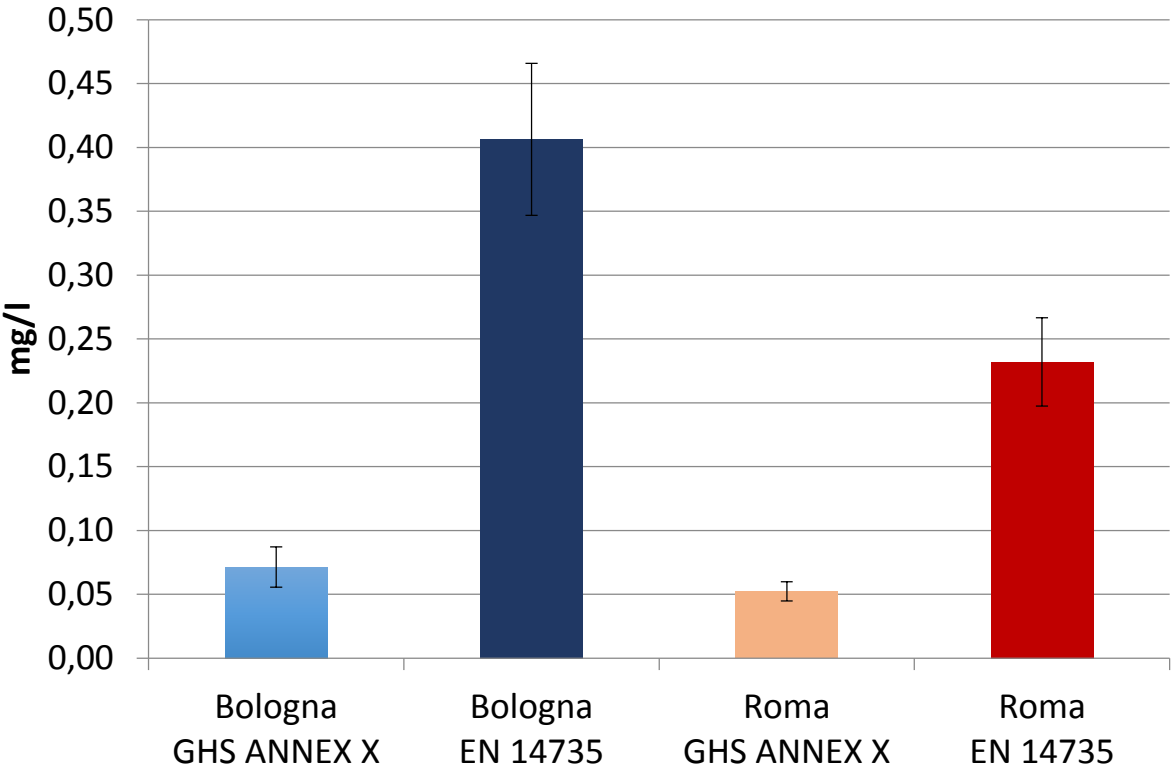
- APAT, 2004 - UNICHIM Metodo 1651/2003
- Endpoints: Germination Index (%)
- 72 hr test
- 7 concentration of water extracts (control included)
- 4 Replicates for each concentration
- 10 seeds per replicate
- 5 ml of tested sample per replicate
- Limits: $EC_{50} \leq 10\%$ (vol/vol)



Ba

RESULTS

Ba (limits 10 mg/l)

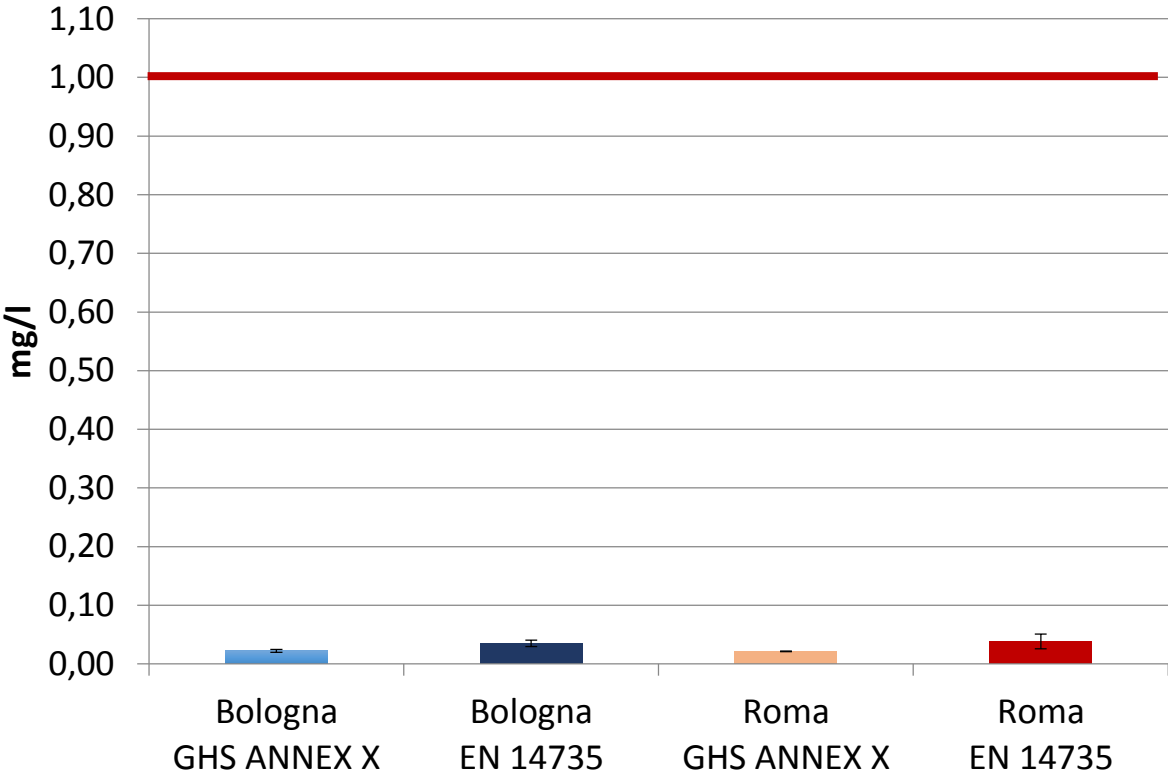
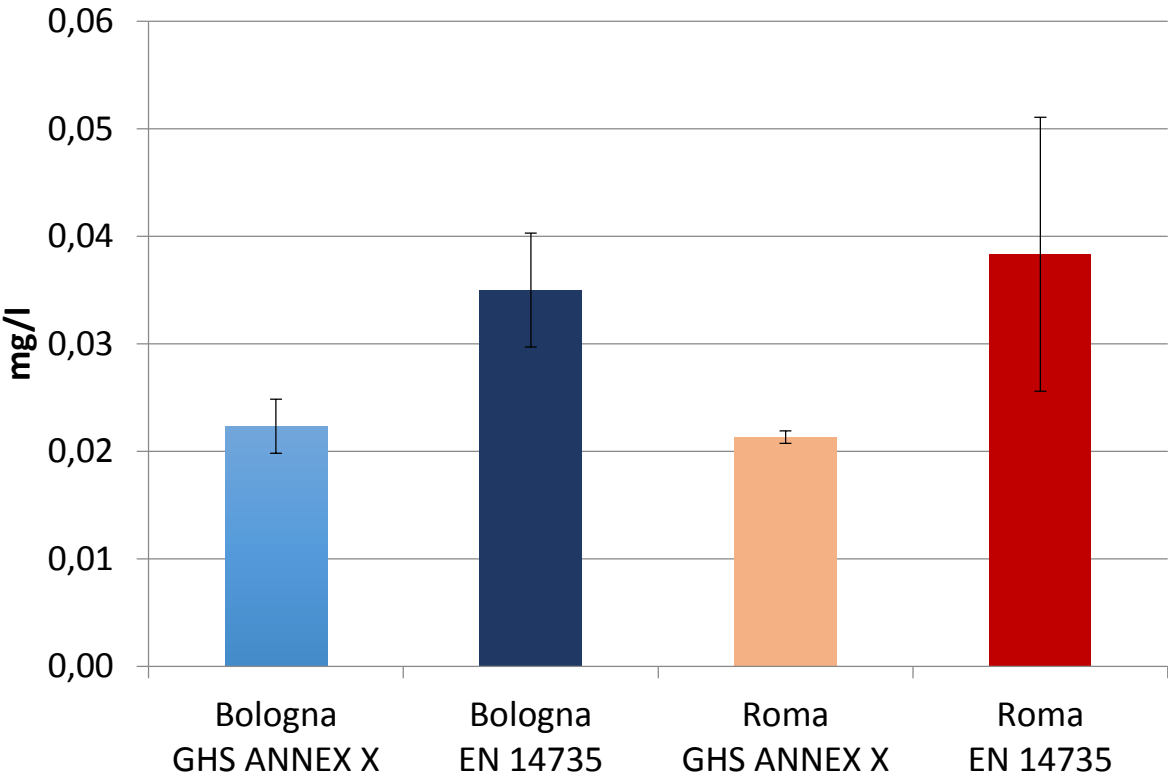


	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Ba mg/l	0,071	0,406	0,052	0,232	0,200	1,657	10
	±0,016	±0,060	±0,008	±0,035	-	-	

Mo

RESULTS

Mo (limits 1 mg/l)

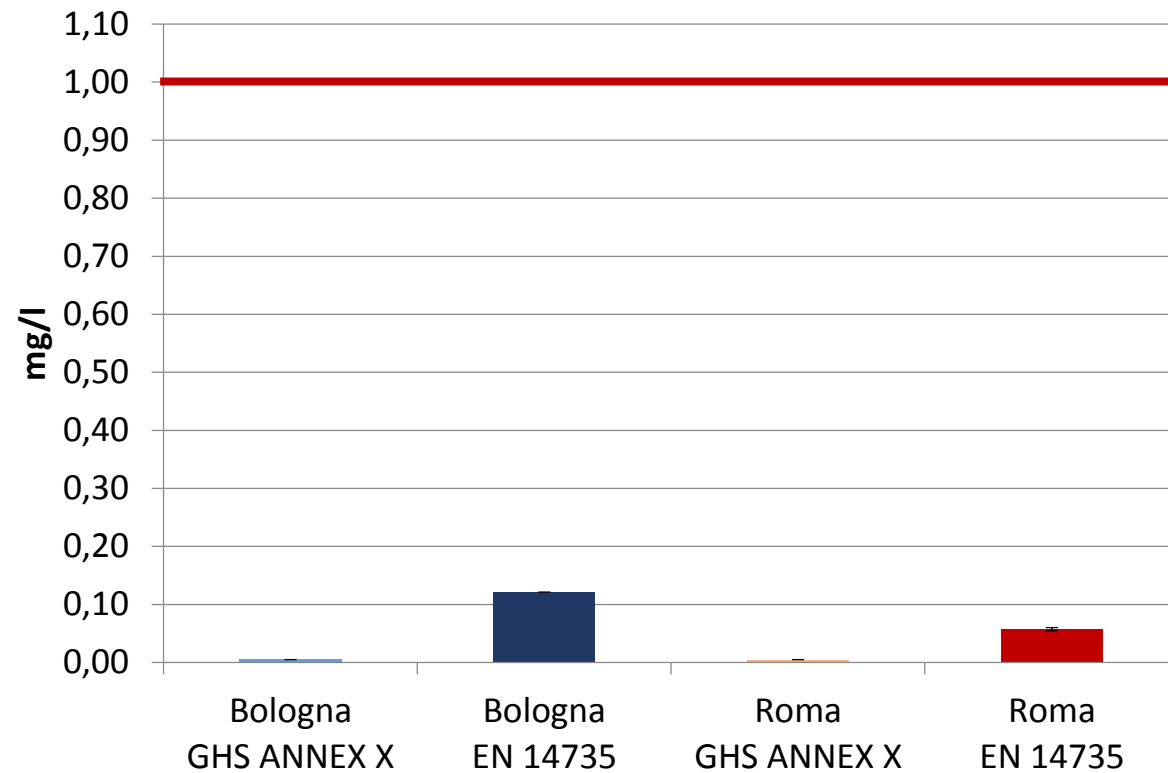
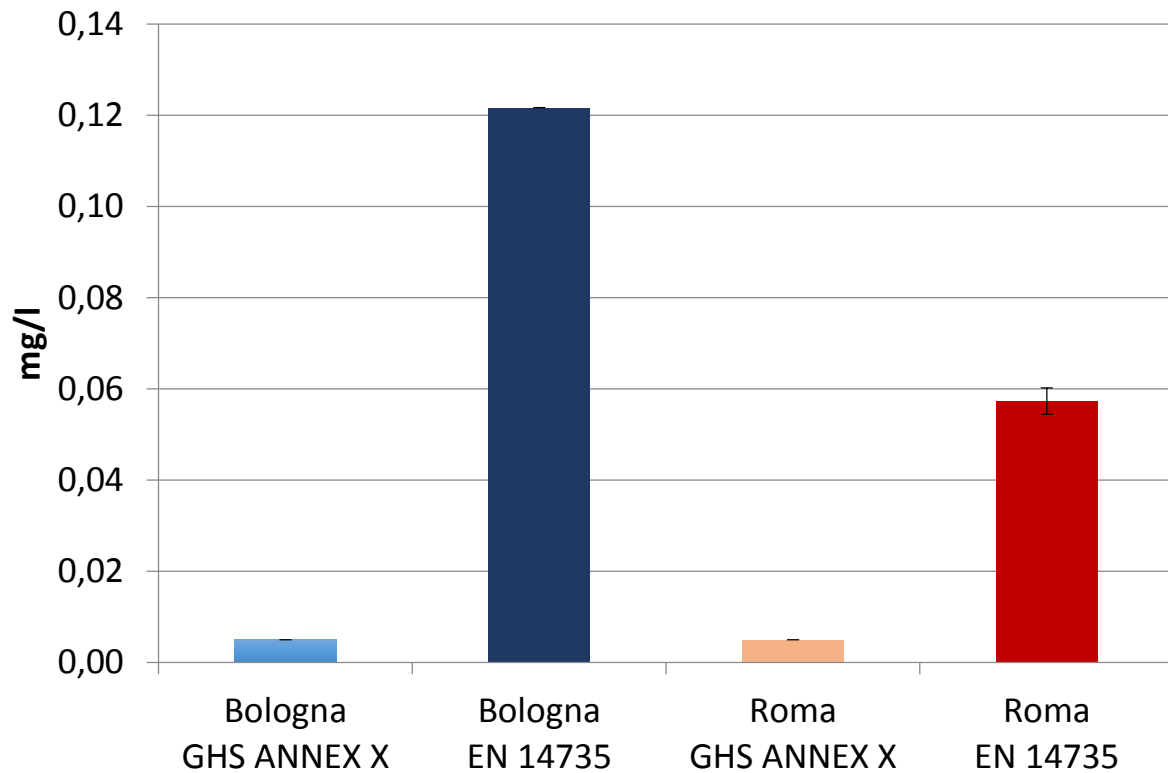


	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Mo mg/l	0,022 ±0,003	0,035 ±0,005	0,021 ±0,001	0,038 ±0,013	0,093 -	0,084 -	1

Ni

RESULTS

Ni (limits 1mg/l)

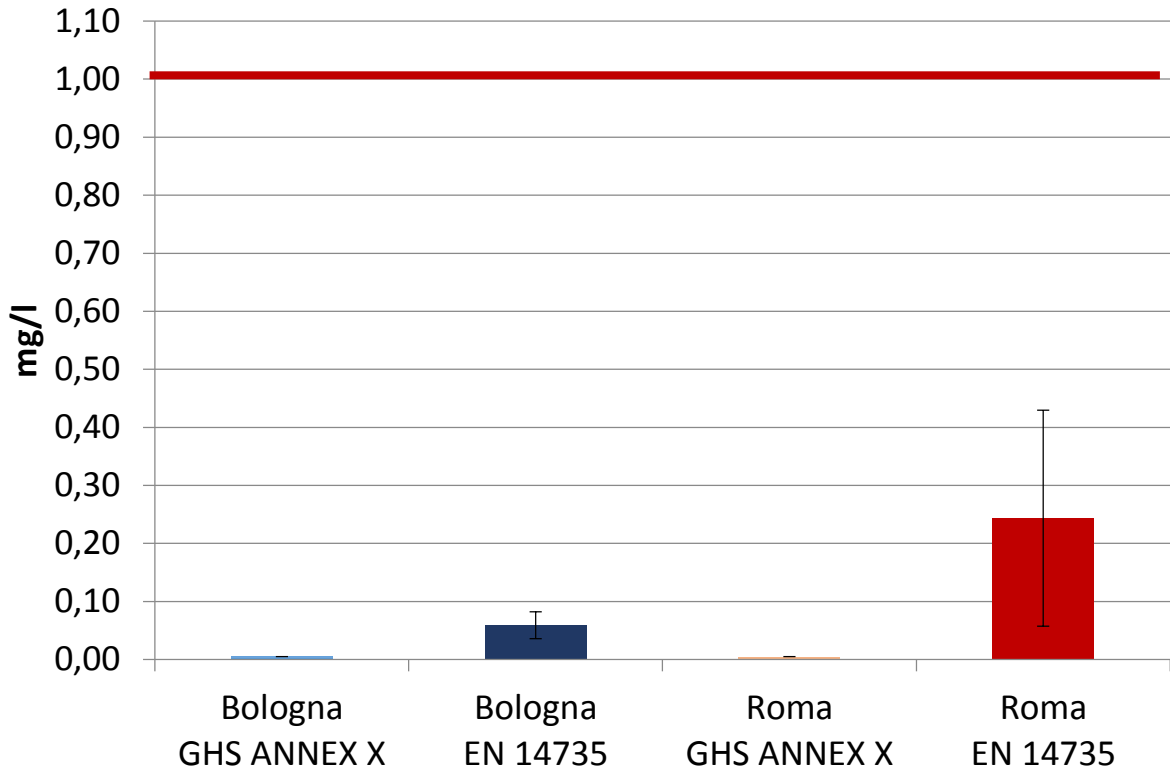
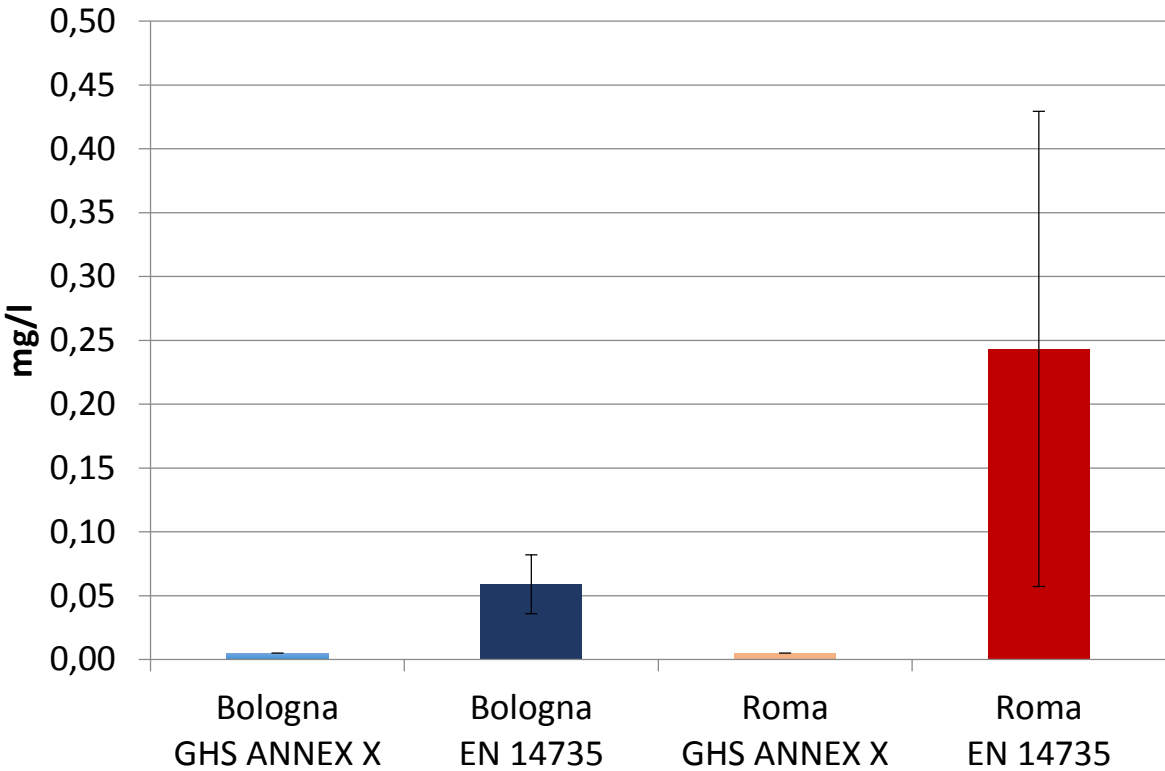


	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Ni mg/l	0,005 ±0,000	0,122 ±0,000	0,005 ±0,000	0,057 ±0,003	0,211 -	0,061 -	1

Pb

RESULTS

Pb (limits 1 mg/l)

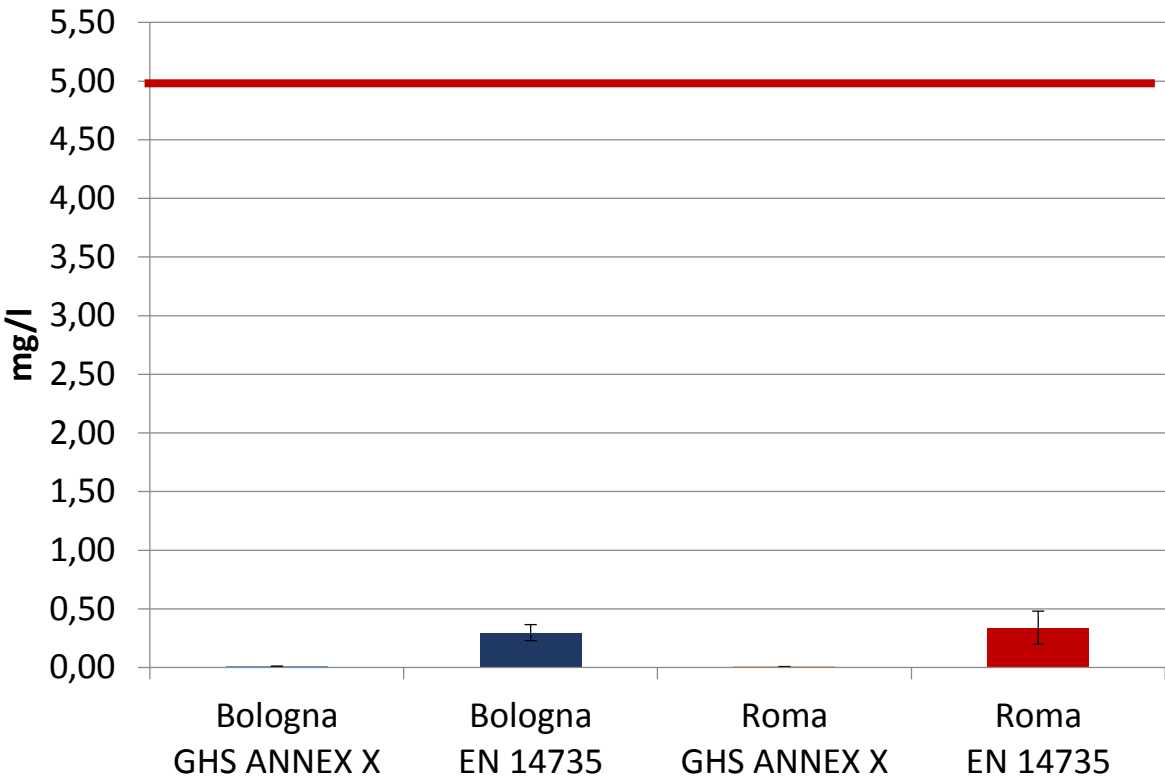
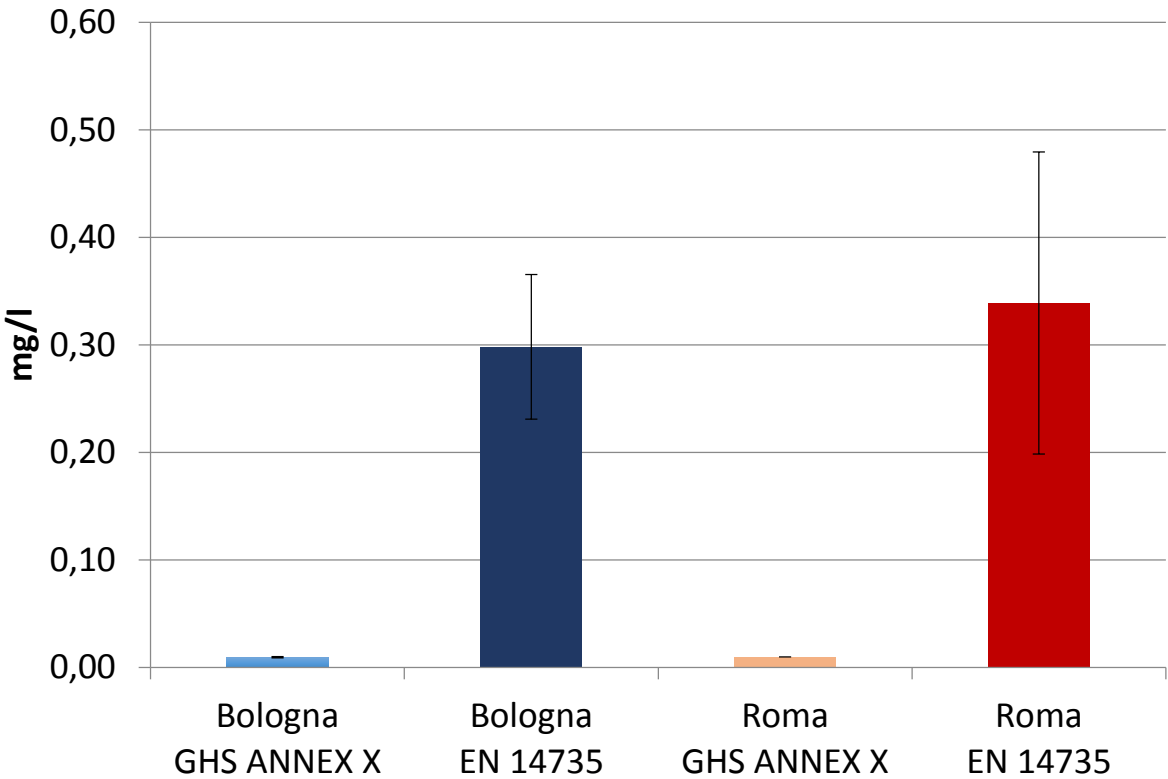


	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Pb mg/l	0,005 ±0,000	0,059 ±0,023	0,005 ±0,000	0,243 ±0,186	0,058 -	0,071 -	1

Cu

RESULTS

Cu (limits 5 mg/l)

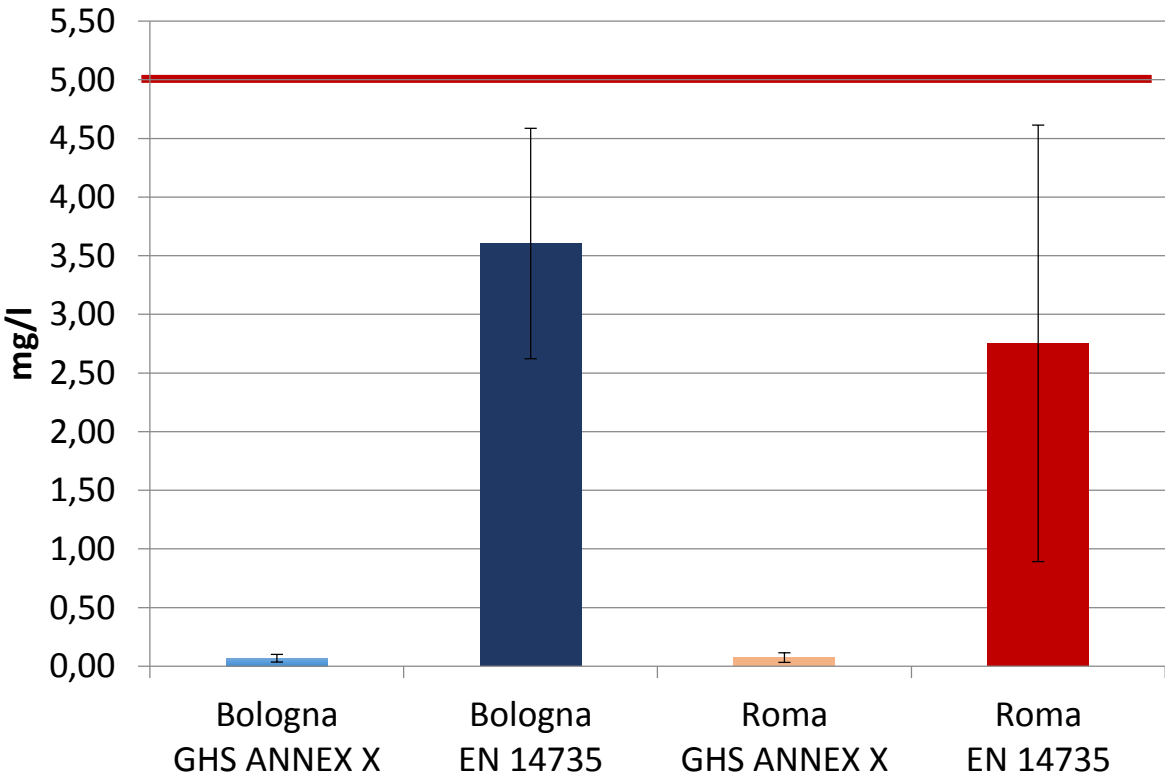
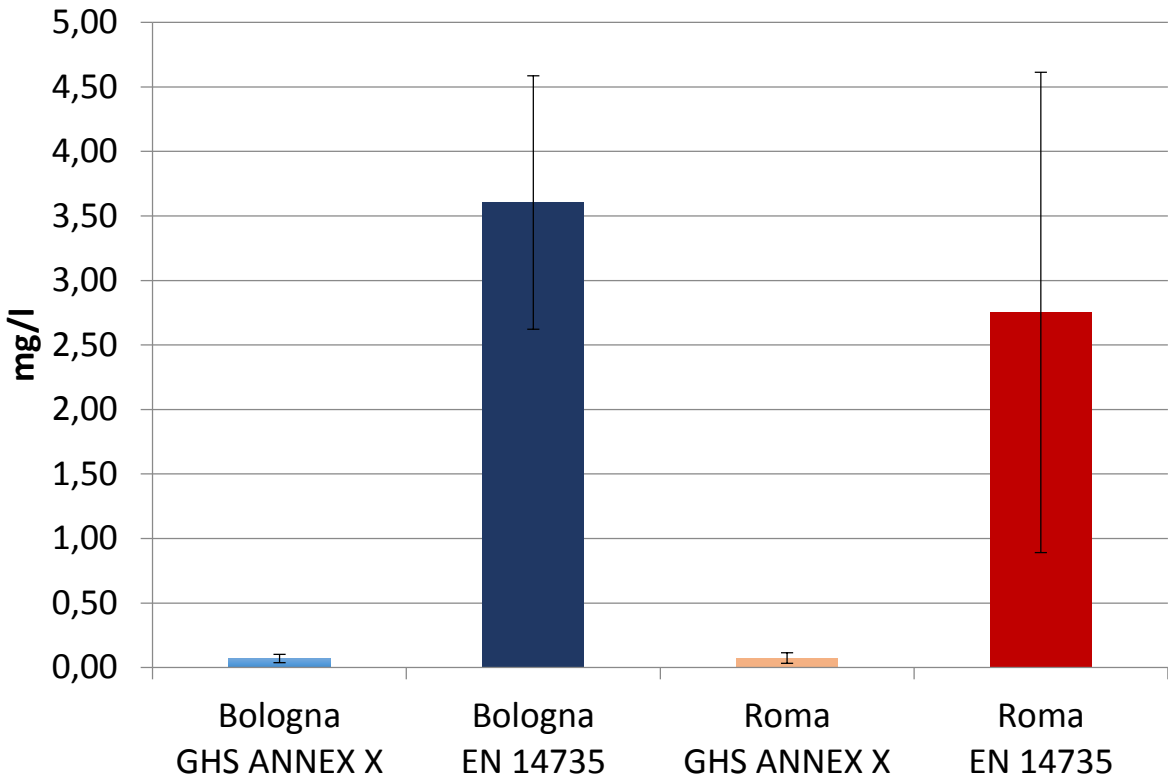


	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Cu mg/l	0,010	0,298	0,010	0,339	0,267	0,112	5
	±0,001	±0,067	±0,000	±0,140	-	-	

Zn

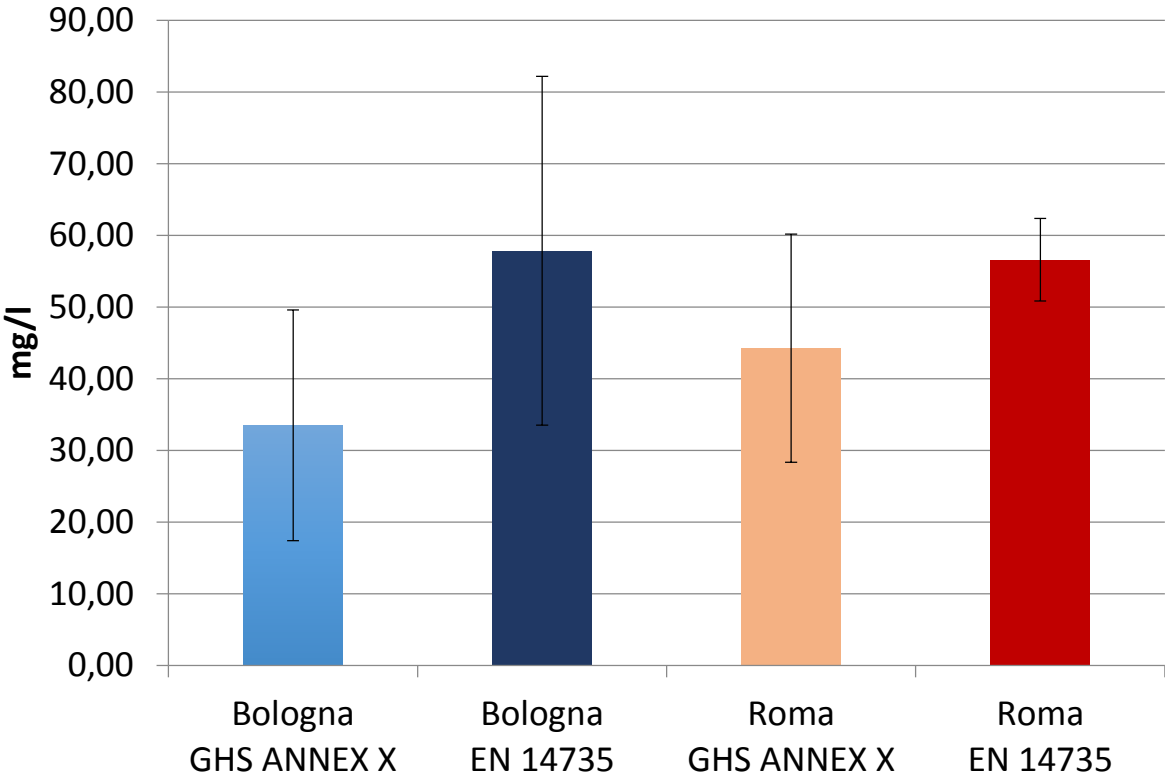
RESULTS

Zn (limits 5 mg/l)



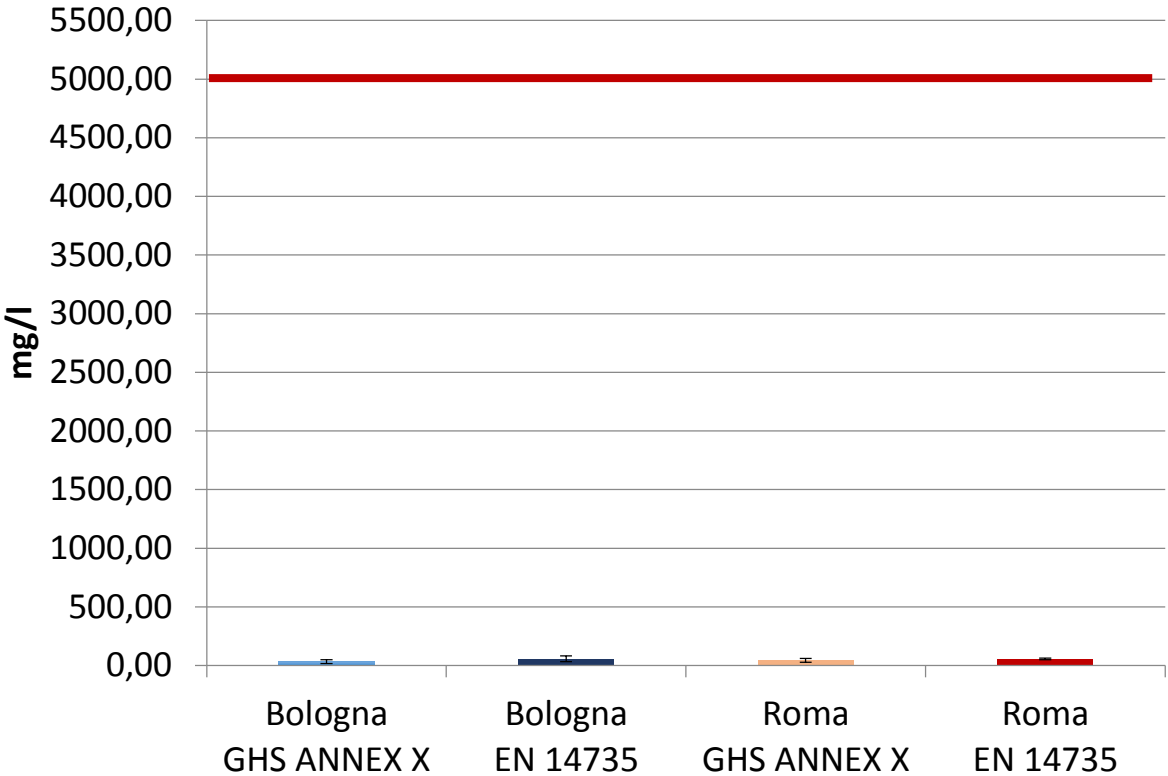
	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Zn mg/l	0,069	3,604	0,074	2,752	2,553	1,085	5
	0,032	0,981	0,041	1,861	-	-	

Sulphates



RESULTS

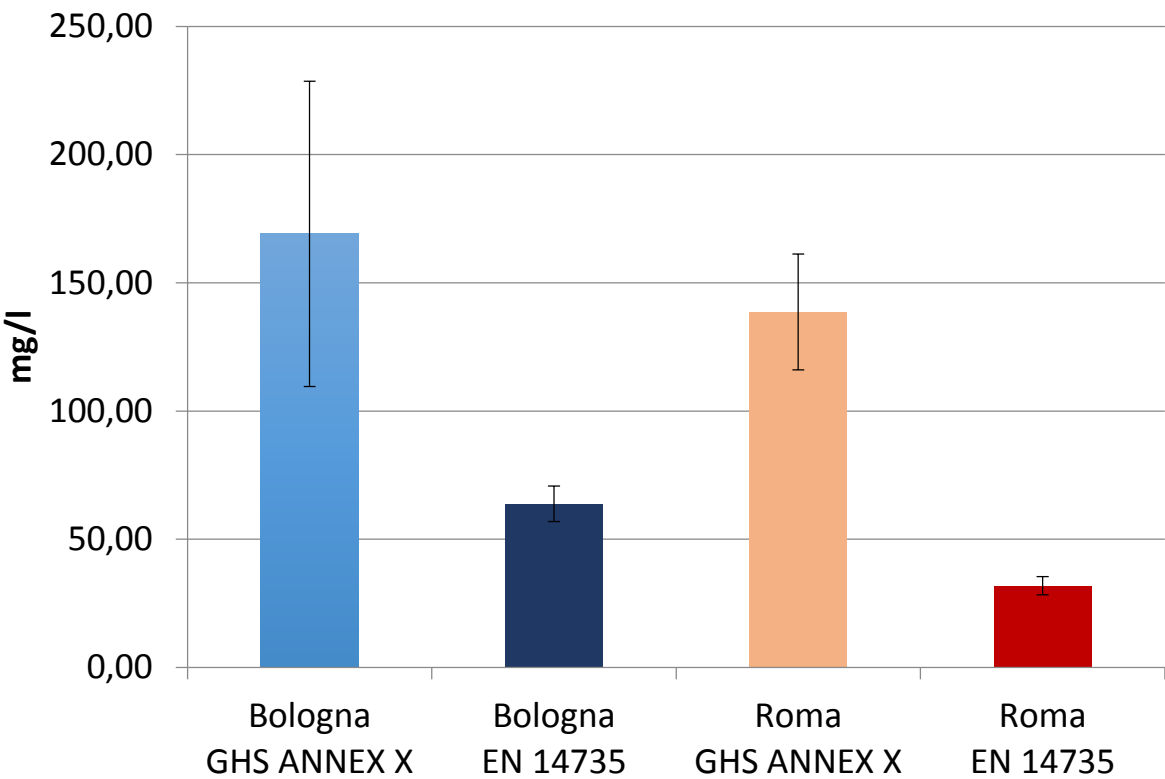
Sulphates (limits 5000 mg/l)



	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Sulphates (SO ₄ ²⁻) mg/l	33,499	57,855	44,270	56,615	71,011	79,538	5000
	±16,100	±24,332	±15,914	±5,761	-	-	

Chlorides

RESULTS



GHS ANNEX X

Average Concentration of Chlorides in the Leaching Media
≈ 150 mg/l

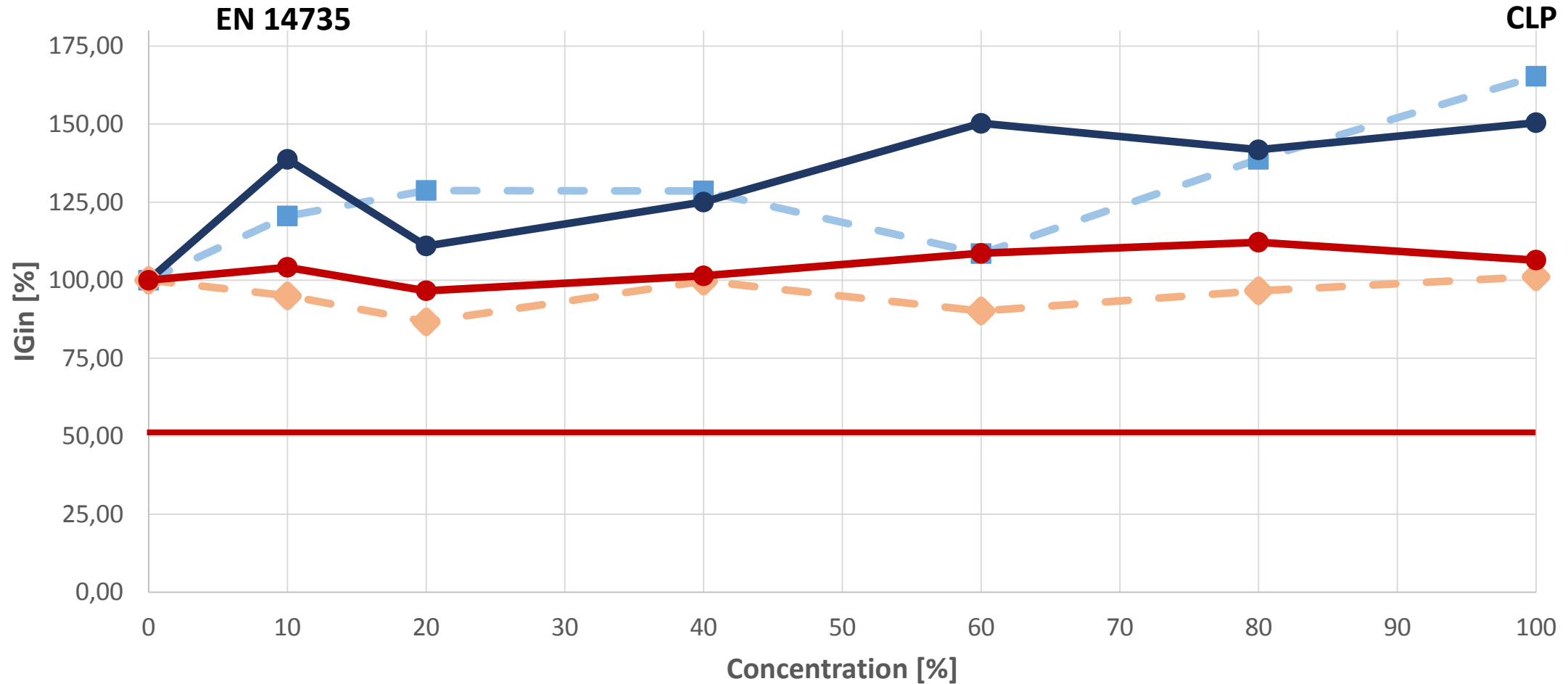
The limits of 2500 mg/l is largely respected

	Bologna GHS ANNEX X	Bologna EN 14735	Roma GHS ANNEX X	Roma EN 14735	Bologna REFERENCE EN 14735	Roma REFERENCE EN 14735	Limits DM 27/09/10
Cloruri (come Cl-) mg/l	169,130 ±59,552	63,846 ±6,876	138,637 ±22,573	31,911 ±3,563	46,921 -	48,893 -	2500

RESULTS

			Cd	Cr	Hg	Ag	Be
Source	Protocol	Replicate	ug/L	ug/L	ug/L	ug/L	ug/L
Bologna	GHS Annex X	1	< 1	< 5	< 0,5	< 5	< 1
Bologna	GHS Annex X	2	< 1	< 5	< 0,5	< 5	< 1
Bologna	GHS Annex X	3	< 1	< 5	< 0,5	< 5	< 1
Bologna	EN 14735	1	1,30	< 5	< 0,5	< 5	< 1
Bologna	EN 14735	2	1,26	< 5	< 0,5	< 5	< 1
Bologna	EN 14735	3	1,98	6,12	< 0,5	< 5	< 1
Roma	GHS Annex X	1	< 1	< 5	< 0,5	< 5	< 1
Roma	GHS Annex X	2	< 1	< 5	< 0,5	< 5	< 1
Roma	GHS Annex X	3	< 1	< 5	< 0,5	< 5	< 1
Roma	EN 14735	1	2,01	18	0,96	< 5	< 1
Roma	EN 14735	2	1,41	16	< 0,5	< 5	< 1
Roma	EN 14735	3	< 1	< 5	< 0,5	< 5	< 1

RESULTS – GERMINATION INDEX



BOLOGNA GHS ANNEX X BOLOGNA EN 14735 ROMA GHS ANNEX X ROMA EN 14735

CONCLUSIONS

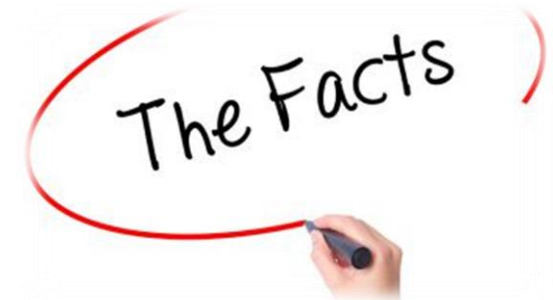
- **Data** obtained by this study **are consistent with reference data** from the period 2016-2018;
- Chemical **concentrations on water extracts obtained from EN 14735 are higher** than chemical concentrations derived from GHS ANNEX X;



Leaching test according to **EN 14735 proved to be more conservative**



- The only case in which the chemical concentration on water extracts is higher than GHS ANNEX X is due to the presence of chlorides in the media;
- Preliminary ecotoxicological tests are **consistent with proposed Limits**.
- **PROCEDURE and LIMITS validated by the scientific community already exist!!!**



REFERENCES

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- **Hennebert, P., 2018.** Proposal of concentration limits for determining the hazard property HP 14 for waste using ecotoxicological tests. Waste Manag. 74, 74–85. <https://doi.org/10.1016/j.wasman.2017.11.048>
- **Pandard, P., Römbke, J., 2013.** Proposal for a “Harmonized” strategy for the assessment of the HP 14 property. Integr. Environ. Assess. Manag. 9, 665–672. <https://doi.org/10.1002/ieam.1447>
- **997/2017/EC** - Council Regulation No 2017/997 of 8 June 2017 amending Annex III to Directive 2008/98/EC of the European Parliament and of the Council as regards the hazardous property HP 14 ‘Ecotoxic’
- **EN 14735:2005**, Characterization of waste – Preparation of waste samples for ecotoxicity tests. CEN, Brussels, Belgium.
- **OECD, 2001.** OECD Series on Testing and Assessment, Number 29. ANNEX 10. Guidance on Transformation/Dissolution of Metals and Metal Compounds in Aqueous Media. S 525–537.
- **ISS/ISPRA 29/09/2011 Prot.** ISS n. 40823



THANKS FOR THE ATTENTION!

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