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EU Proficiency test EUPT-CF10 – Incurred and Spiked Pesticide Residues in Rye Flour

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Introduction: The EURL-CF has in March/April 2016 organised a proficiency test (PT) on pesticide residues in rye. Participants are listed in Table 1

Target Pesticide List: The list included 134 compulsory pesticides, of which 17 was new. Additionally, 7 voluntary pesticides were on the list.

Production of test material: The treatments of the rye are listed in Table 2.

Preliminary assigned values: The preliminary assigned values were calculated as Algorithm A means of the results from EU and EFTA participants, reporting that water was added to the sample prior to extraction. The assigned values ranged from 0.032-0.414 mg/kg, see Figure 1.

Alg A standard deviation: The average robust relative standard deviation (Alg A RSD) was 17 % and ranged from 14 to 23 %, see Figure 2.

False negatives and positives: No false positive results were reported and 42 false negative results of 14 different pesticides.

Acceptable z scores: The distribution of z scores are shown in Figure 3. Also in this EUPT some laboratories reported very high results for carbendazim, see Figure 4. This is due to the low solubility of the compound in organic solvent. Do not prepare stock solutions of carbendazim at concentrations >0.1 mg/ml.

Ion ratios: Reported information showed that 1.3% of the correct reported results did not fulfil the 30% criteria from SANTE/11945/2015. See Figure 5.

Retention times: Reported information showed that 1.6% of the correct reported results did not fulfil the +/- 0.1 min criteria from SANTE/11945/2015. See Figure 5.

Recoveries: Reported information on recoveries showed that 2.4% of the recoveries carried out in the same batch was outside the 70-120% criteria from SANTE/11945/2015. See Figure 5.

Table 1 Participants in EUPT-CF10. Malta was represented by UK

Country	# labs	Country	# labs	Country	# labs
Algeria	1	France	8	Poland	16
Argentina	1	Germany	24	Portugal	2
Australia	1	Greece	3	Romania	9
Austria	2	Hungary	4	Serbia	2
Belgium	3	Iceland	1	Singapore	1
Brazil	3	India	2	Slovakia	2
Bulgaria	2	Indonesia	1	Slovenia	3
China	1	Ireland	1	Spain	22
Croatia	7	Israel	1	Sweden	2
Cyprus	2	Italy	25	Switzerland	1
Czech Republic	3	Latvia	1	Tanzania	2
Denmark	1	Lithuania	1	Thailand	1
Egypt	1	Luxembourg	1	United Kingdom	3
Estonia	1	Netherlands	6		
Finland	2	Norway	2	Total	178

Table 2. Pesticides applied the field and/or spiked in the laboratory. Lambda-cyhalothrin and deltamethrin were also applied in the field and resulted in low level (0.01 mg/kg).

Pesticide	Field application	Spike in laboratory	Formulation or standard
Azoxystrobin	x		Amistar
Bixafen	x		Aviator
Boscalid	x		Viverda
Buprofezin		x	Analytical standard
Carbendazim	x		Benlate
Cypermethrin	x	x	CyberB
Epoxiconazole	x		Viverda
Fenpropidin	x		Term
Fluopyram	x		Propulse
Isocarbofos		x	Analytical standard
Metrafenone	x		Flexity
Pencycuron		x	Analytical standard
Pirimicarb-desmethyl		x	Analytical standard
Prothioconazole	x		Aviator and Propulse
Pyraclostrobin	x		Viverda
Tebuconazole	x		Folicur
Tetramethrin		x	Analytical standard
Heptachlor		x	Analytical standard

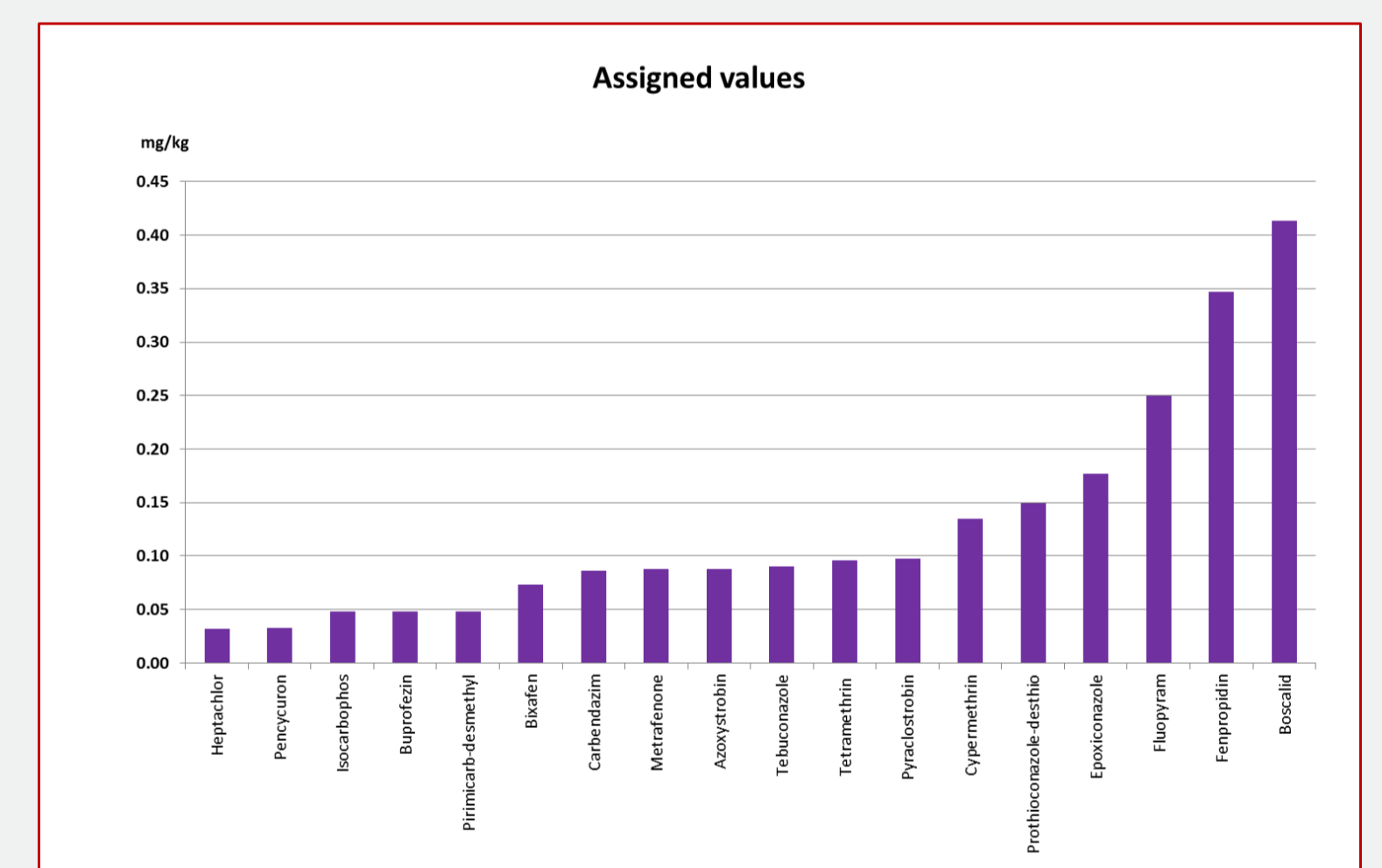


Figure 1 Assigned values of the 18 pesticides

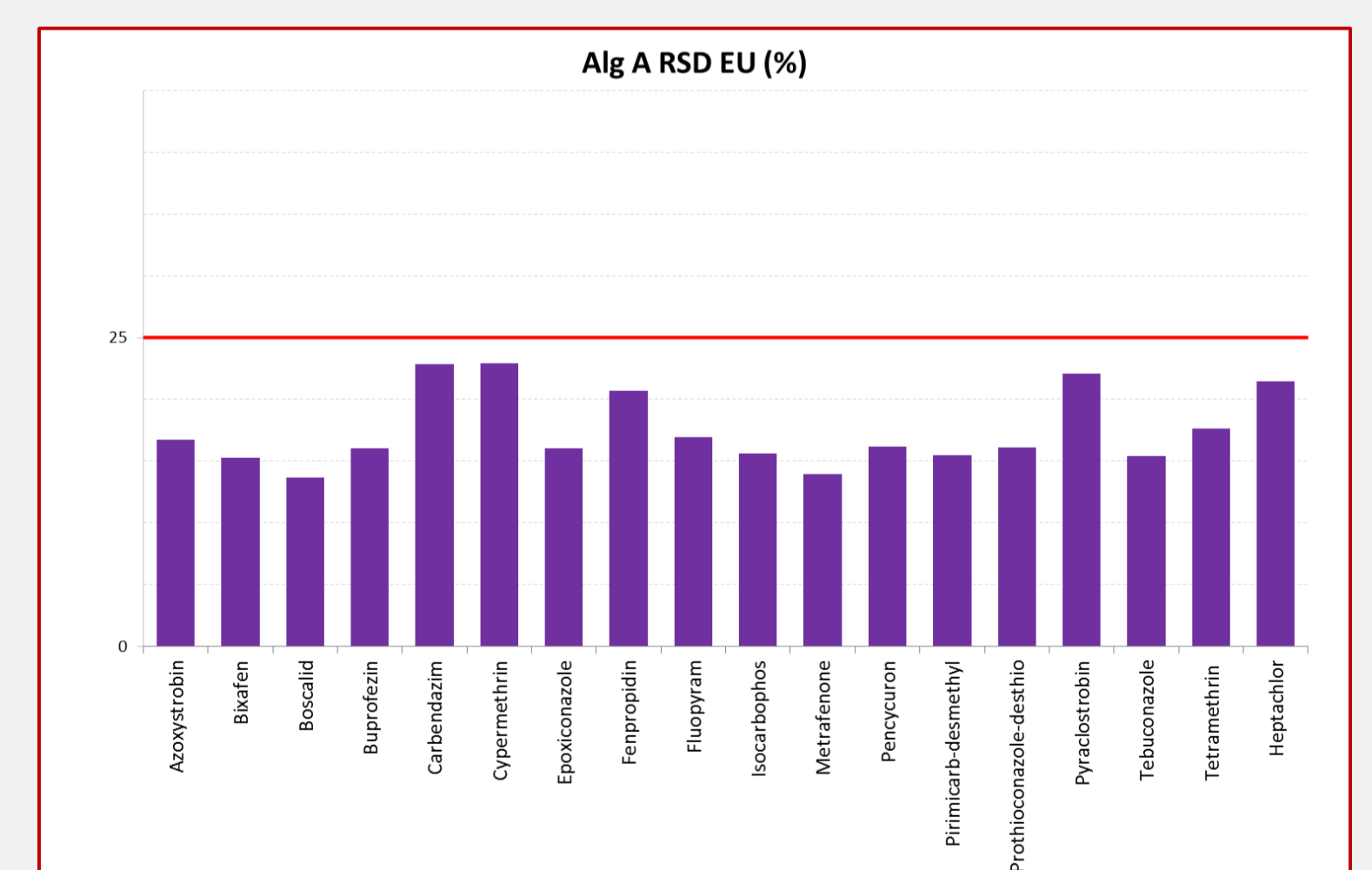


Figure 2 Alg A robust standard deviations

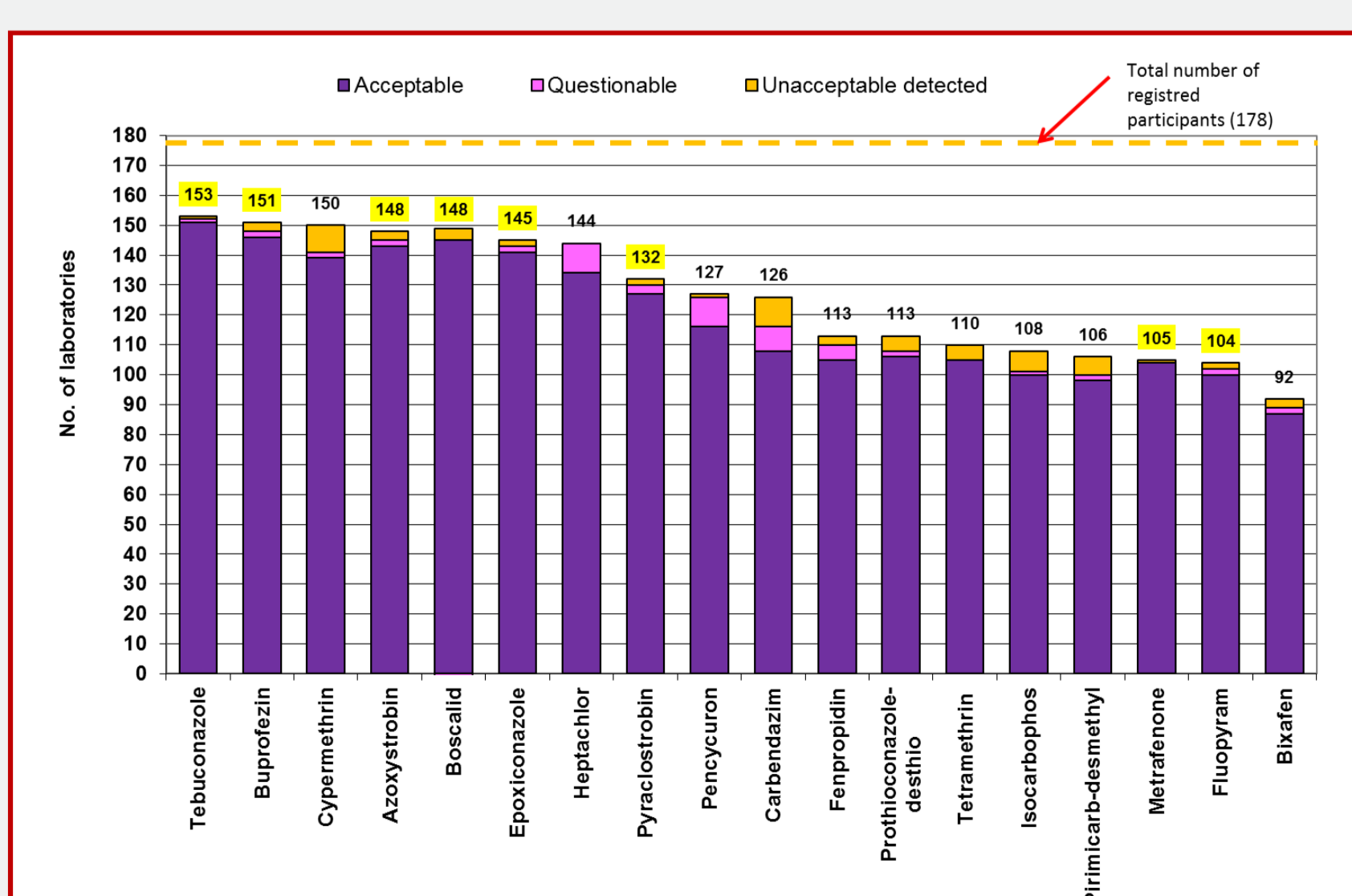


Figure 3 Acceptable, questionable, unacceptable z-scores

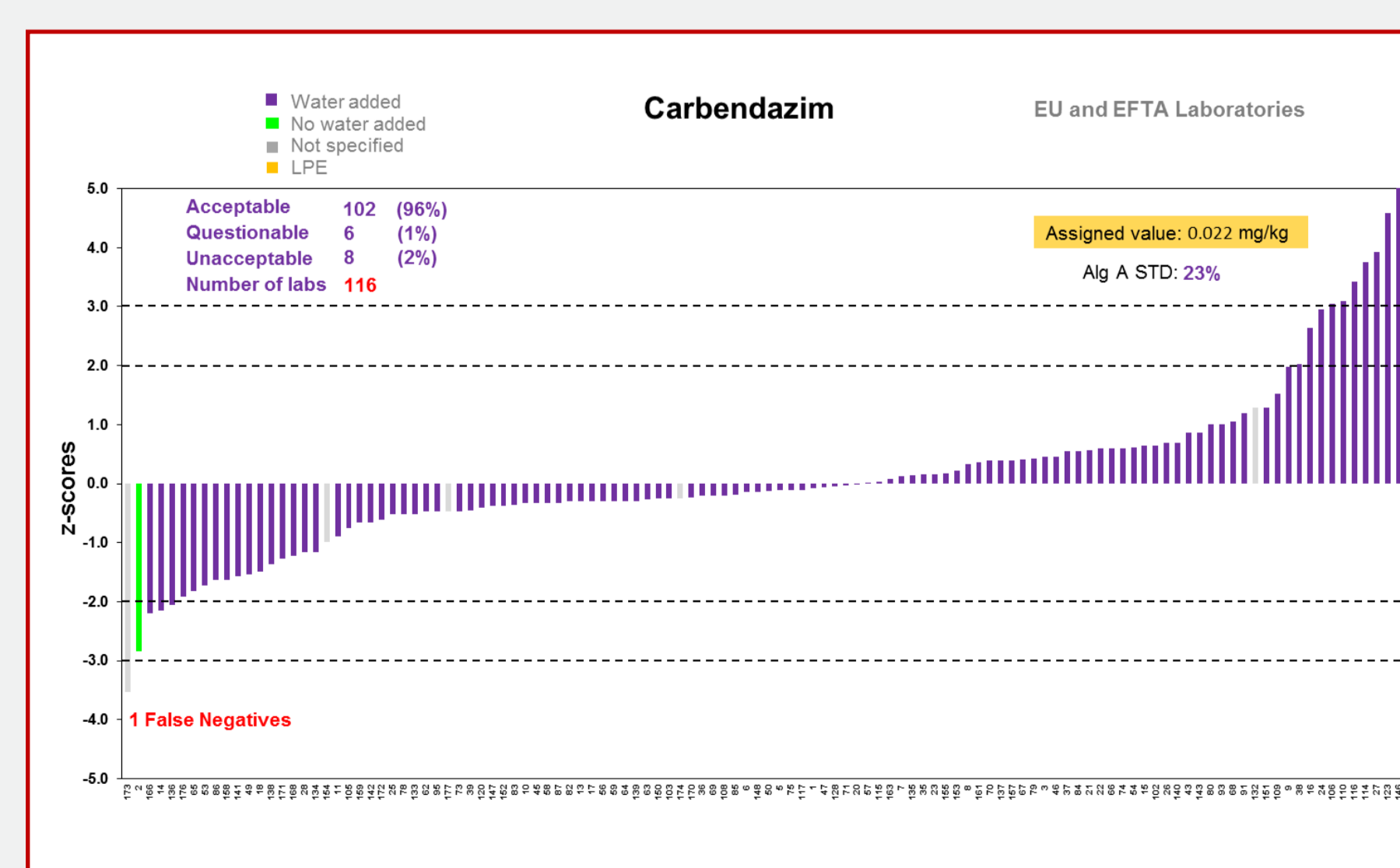


Figure 4 Histogramme of z-scores for carbendazim

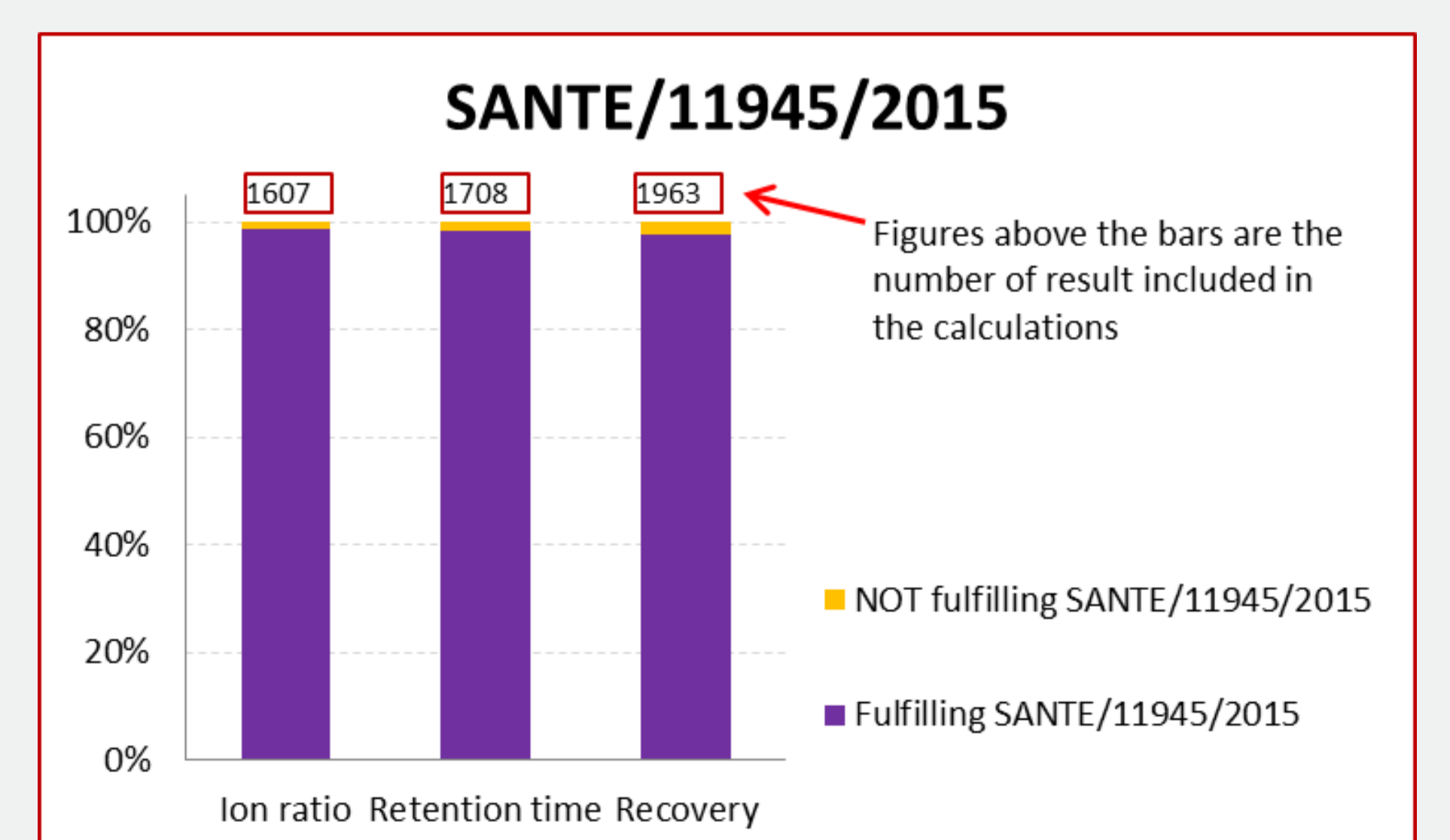


Figure 5 Results that fulfilled the criteria for ion ratio, RT and Recovery