



PT as an instrument to assess the analytical performance and the methods routinely implemented: the Italian experience for the screening of antibiotic residues in milk in the official control

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INTRODUCTION

Trial (2012) for the Official Control Laboratories (OCLs): satisfactory performance for the screening of β -lactams and sulfonamides; strong criticalities for the detection of tetracyclines.

Cause analysis: general use of screening methods with low sensitivity (2.5xMRL where MRL = Maximum Residue Limit). The need to implement appropriate corrective actions was stressed.

Follow-up trial (2016): PT aimed to verify the OCLs performance for tetracyclines and to update the information concerning the methods used in Italy for the screening of antibiotic residues in milk.

ORGANIZATION

PARTICIPANTS: 31 labs from 8 of 10 OCLs.

PT items: CRMs (freeze-dried UHT cow milk)

- 1 blank sample
- 1 sample spiked with benzylpenicillin (PEN) 1xMRL
- 1 sample spiked with sulfadiazine (SDZ) 1xMRL
- 1 sample spiked with oxytetracycline (OXY) 2.5xMRL
- 1 sample spiked with OXY 4 xMRL

Additional tests: preparation and analysis of 2 samples (OXY 2x and 1xMRL) from dilution of sample 5

RESULTS SUBMISSION: on line via a dedicated web-area using individual pre-arranged forms.

EXPRESSION OF RESULTS:

- Raw data: "+", "-", or *doubtful* ("+/-" or "-/+") depending of the supposed higher probability)
- Reported result: "+" or "-"

EVALUATION CRITERIA

The cumulative evaluation, based on the reported results for samples from 1 to 4, was scored as follows:

Maximum score =1 reduced of 0.25 for each false negative (FN) result and of 0.125 for each false positive (FP) result.

A major penalty was attributed to FN results because of their direct impact on the safety of food products and hence on consumers health.

SCREENING METHODS APPLIED BY OCLs

METHOD APPLIED	N° labs 2012	N° labs 2016	PEN	Claimed Limit of Detection (LOD) $\mu\text{g}/\text{kg}$		
				SDZ	OXY	
Delvotest SP NT	35	26	2	50-65	250-300	
Delvotest T	-	5	1-2	40-50	80-100	
CMT	3	1	1-2	50-100	250-500	
SNAP β -lactam ST	1	1	3	-	-	
SNAP tetracycline test	-	1	-	-	≤ 50	
MRL milk ($\mu\text{g}/\text{kg}$)			4	100	100	

Only 1 lab used more than one method in parallel: Delvotest SP NT + Delvotest T + SNAP β -lactam ST+ SNAP tetracycline test

OCLs PERFORMANCE (BLANK, PEN, SDZ, OXY 250)

Score	FN results n°	Labs n°	FN sample/lab	Method
1	0	26/31	--	--
0.75	1	3/31	OXY 250 OXY 250 PEN 4	CMT Delvotest SP NT Delvotest SP NT
0.5	2	2/31	OXY 250 + SDZ 100 OXY 250 + SDZ 100	Delvotest SP NT Delvotest SP NT

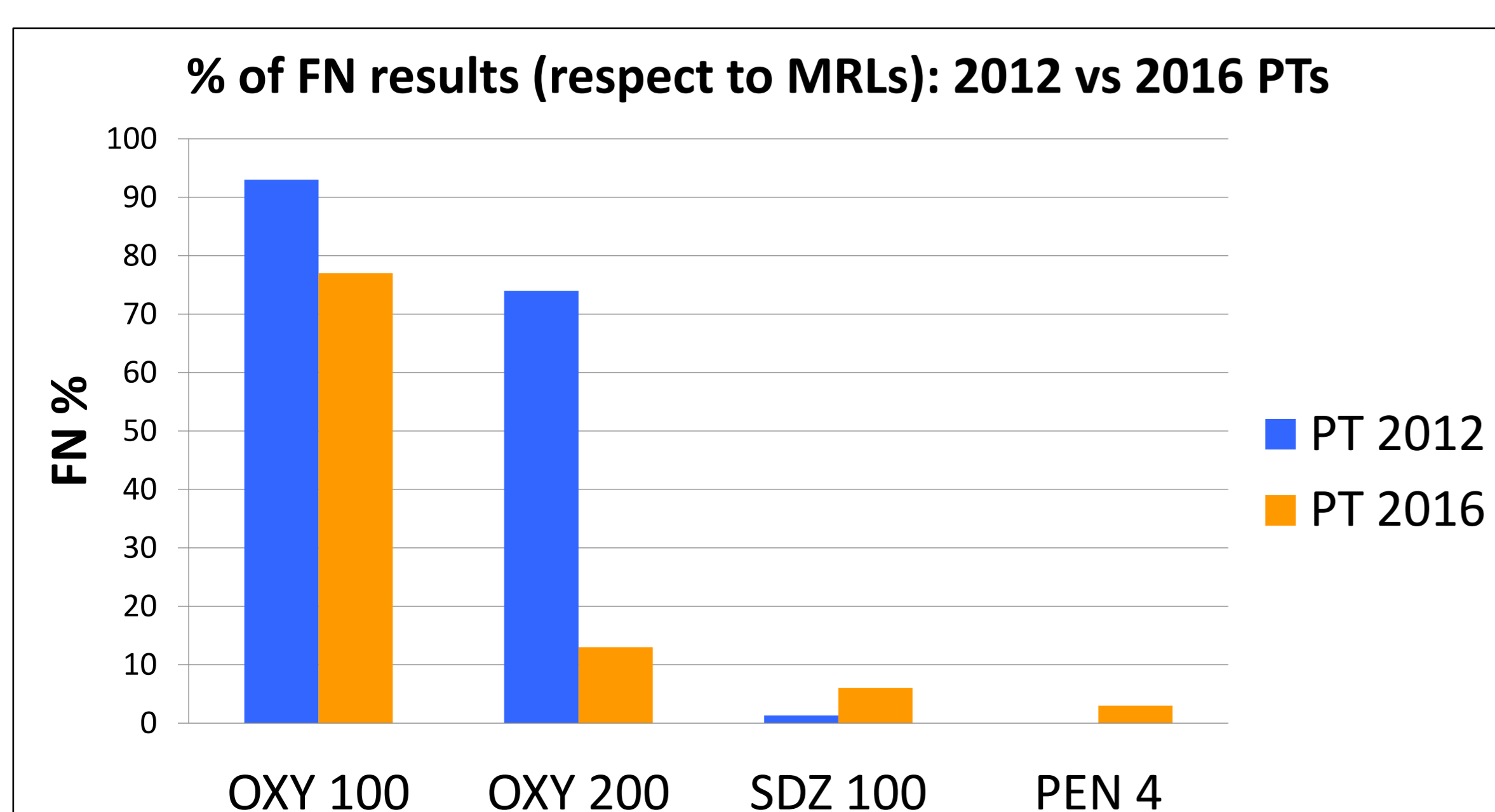
FOCUS ON OXY: FN RESULTS BY METHOD

METHOD	FN results (n°/tot)			
	OXY 400	OXY 250	OXY 200*	OXY 100*
SNAP Tetracycline	0/1	0/1	0/1	0/1
Delvotest T	0/5	0/5	0/5	2/5
Delvotest SP-NT	2/23	4/24	4/23	19/22
CMT	0/1	1/1	0/1	1/1

* Prepared in lab from OXY 400

- false negative respect to the method claimed LOD
- true negative respect to the method claimed LOD
- incongruent results (problem of dilution?)

2012 vs 2016 PT - CONCLUSIONS



The present work reiterates the importance of PTs to highlight critical issues of laboratories.

Due to the action of the NRL the percentage of FN results for OXY is dramatically decreased.

Further improvement is expected in the following PT.