National Food Institute

Technical University of Denmark

DTU

Extraction efficiency for incurred and spiked pesticides

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Copenhagen, 18 September 2008

CRL Pesticides in Cereals and Feedingstuff Community Reference Laboratory

National Food Institute DTU

DG SANCO EUROPEAN COMMISSION

Water addition to sample prior to extraction – PTC2

mg/kg	Median of all results	Median of results with water addition	Median of results with no water addition	Water / no water
Alpha-cypermethrin	0.076	0.079	0.072	**1.1**
Bifentrin	0.088	0.087	0.090	**1.0**
Chlorpyrifos-methyl	0.110	0.130	0.056	**2.3**
Iprodione	0.265	0.289	0.100	**2.9**
Malathion *)	0.130	0.168	0.102	**1.6**
Prochloraz *)	0.227	0.239	0.160	**1.5**
Azoxystrobin *)	0.217	0.239	0.133	**1.8**
Trifloxystrobin	0.430	0.439	0.376	**1.2**

***) Spiked in the laboratory**

Azoxystrobin PTC1	0.189	0.240	0.074	**3.2**

Extraction efficiency - experimental design

Atmospheric pressure	Solvent		No water	Water 0 min	Water 30 min
	Acetonitril	PTC1	3	3	3
		PTC2	3	3	3
		Recovery	3	3	3
	Ethyl acetate	PTC1	3	3	3
		PTC2	3	3	3
		Recovery	3	3	3
	Ethyl acetate - GPC	PTC1	3	3	3
		PTC2	3	3	3
		Recovery	3	3	3
High pressure (ASE)					
	Acetonitril	PTC1	3		
		PTC2	3		
		Recovery	3		

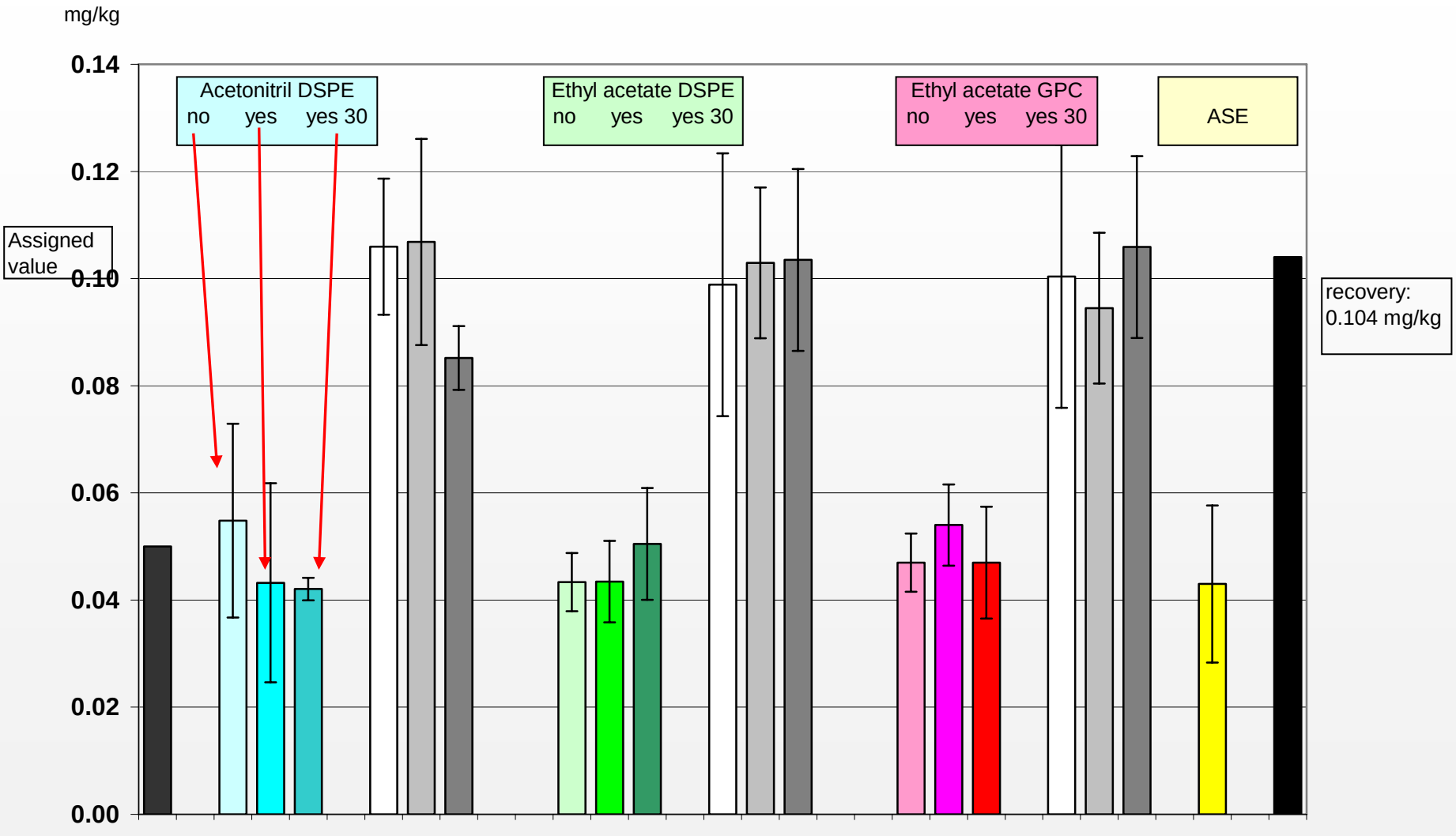
Extraction efficiency of incurred pesticides

		QuEChERS			Modified QuEChERS			FP004			ASE
	Assigned value	No water	Water	Water + 30 min	No water	Water	Water + 30 min	No water	Water	Water + 30 min	No water
Endosulfan	0.049	0.055	0.043	0.042	0.043	0.043	0.050	0.022	0.027	0.027	0.043
Deltamethrin *	0.342	0.291	0.350	0.360	0.384	0.280	0.316	0.310	0.339	0.278	0.287
Alpha-cypermethrin	0.079	0.094	0.109	0.088	0.088	0.072	0.075	0.065	0.084	0.089	0.066
Bifenthrin	0.087	0.097	0.095	0.085	0.106	0.097	0.094	0.090	0.095	0.094	0.117
Diazinon	0.078	0.062	0.099	0.096	0.078	0.065	0.064	0.089	0.114	0.093	0.083
Malathion *	0.168	0.072	0.183	0.073	0.083	0.119	0.024	0.077	0.154	0.031	0.206
Chlorpyrifos-methyl	0.130	0.049	0.202	0.182	0.054	0.132	0.136	0.044	0.130	0.146	0.146
Iprodione	0.289	0.077	0.398	0.353	0.064	0.283	0.290	0.108	0.349	0.339	0.253
Pirimicarb	0.038	0.012	0.052	0.048	0.004	0.038	0.039	0.003	0.038	0.039	0.019
Propiconazole *	0.353	0.264	0.381	0.390	0.245	0.326	0.425	0.228	0.371	0.439	0.314
Difenoconazole *	0.169	0.093	0.203	0.167	0.116	0.193	0.188	0.110	0.180	0.173	0.148
Epoxyconazole	0.176	0.046	0.214	0.180	0.031	0.159	0.146	0.028	0.160	0.162	0.101
Prochloraz *	0.239	0.170	0.319	0.260	0.129	0.252	0.232	0.151	0.280	0.257	0.230
Trifloxystrobin	0.439	0.312	0.481	0.434	0.379	0.458	0.448	0.418	0.482	0.432	0.490
Azoxystrobin *	0.239	0.120	0.246	0.243	0.116	0.249	0.234	0.111	0.276	0.270	0.183
Azoxystrobin	0.240	0.058	0.263	0.269	0.108	0.213	0.222	0.059	0.259	0.250	0.108

* Spiked pesticide

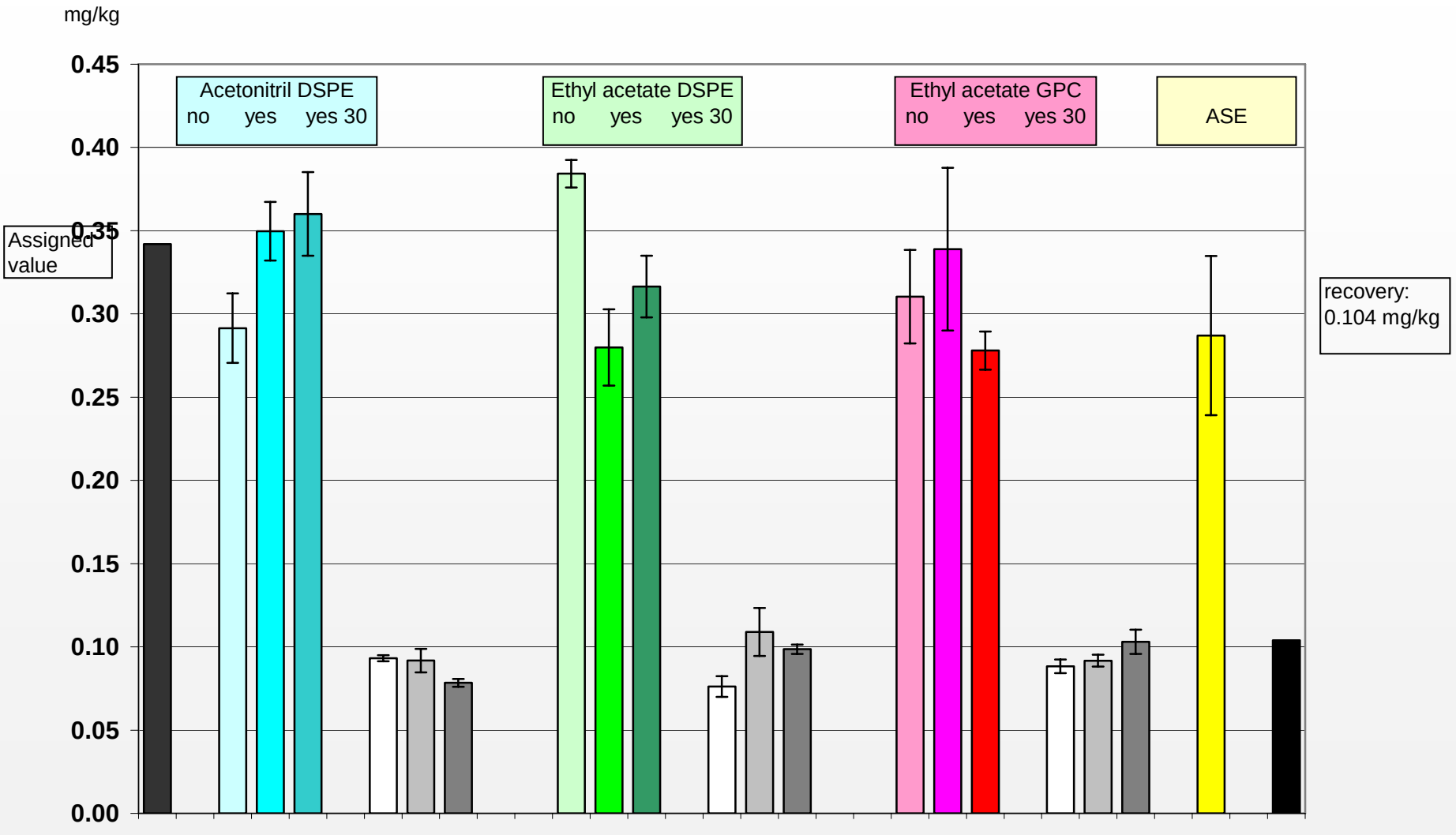
Endosulfan

PTC1 - incurred



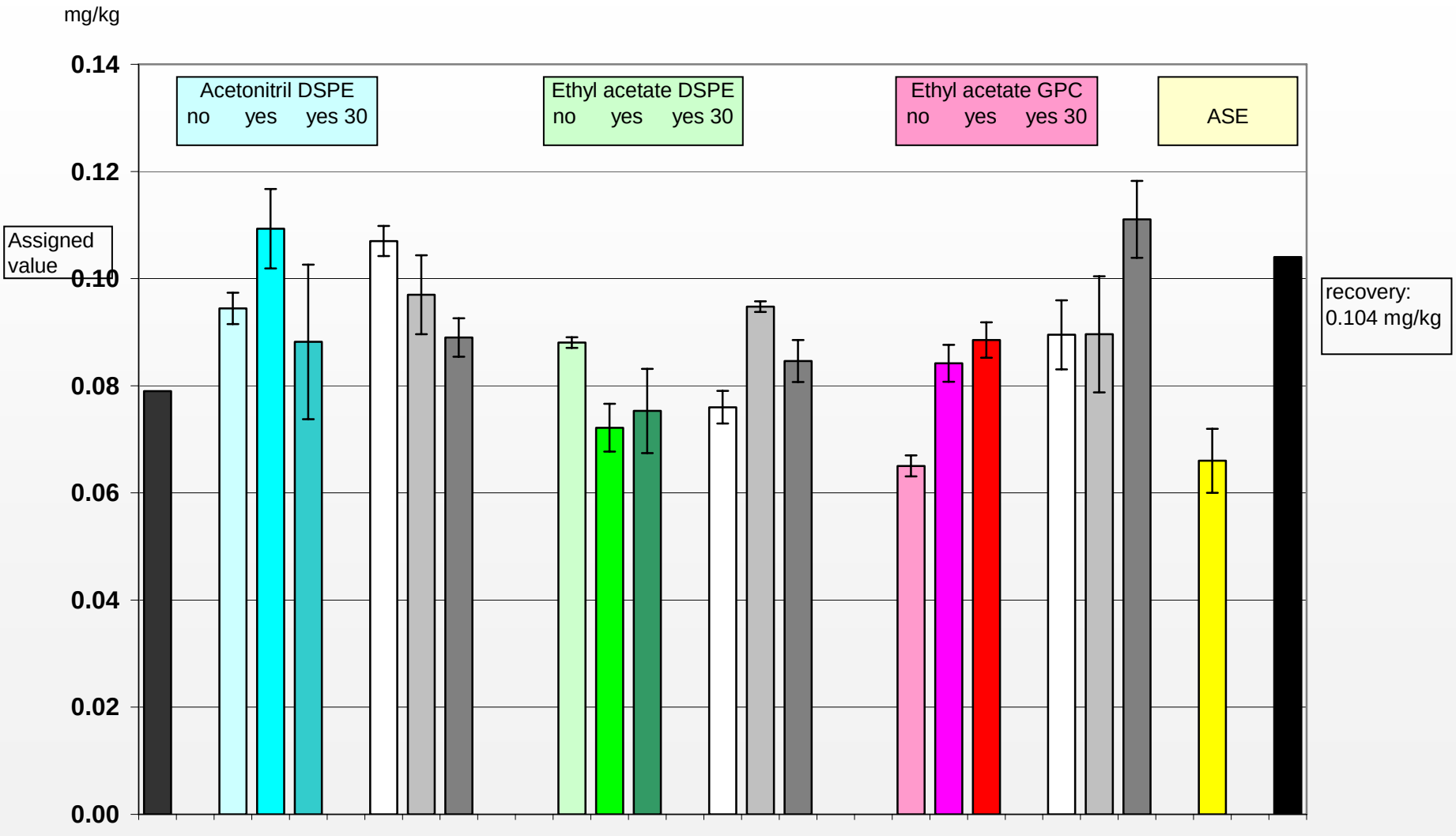
Deltamethrin

PTC1 - spiked



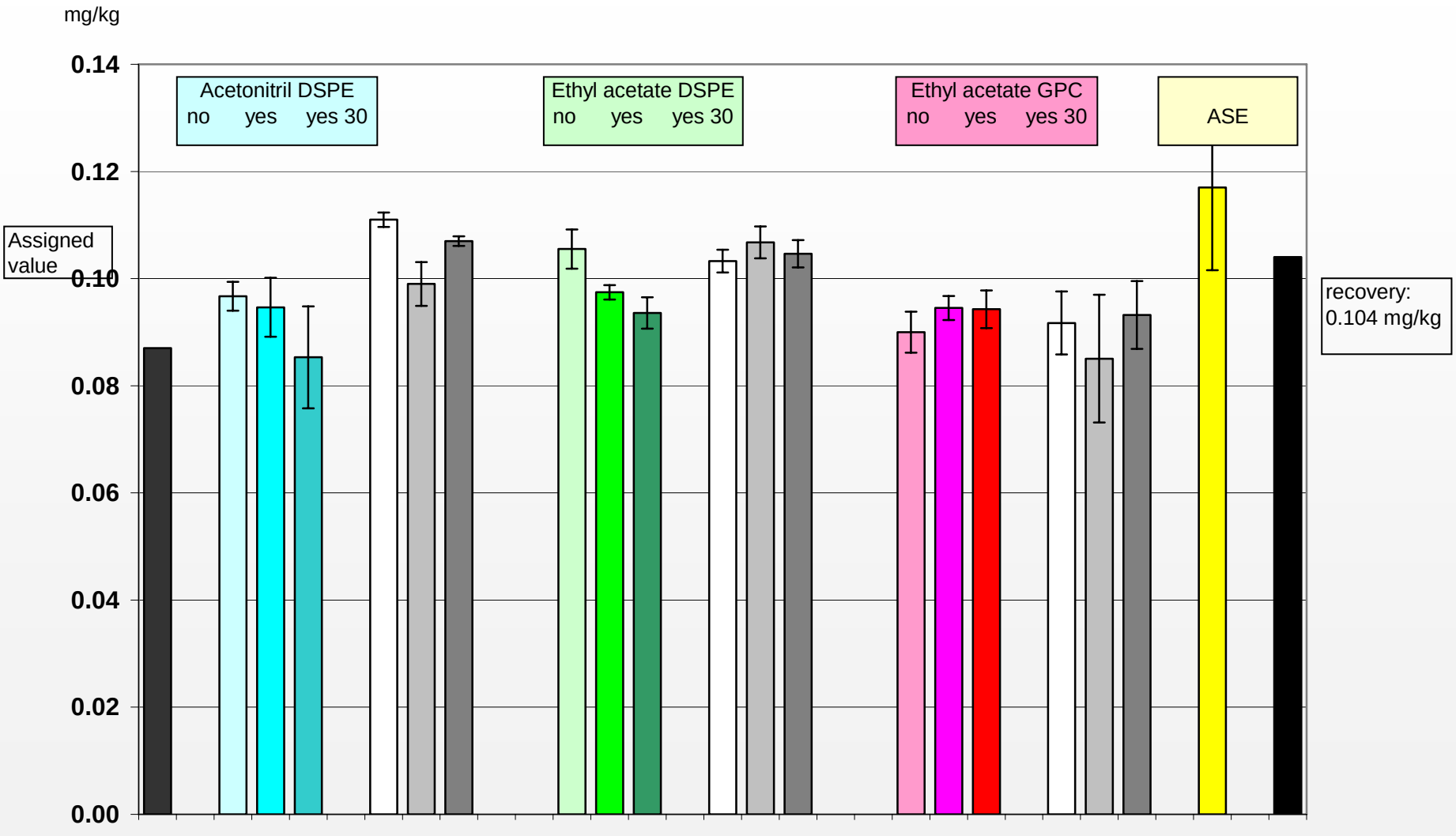
Alpha-cypermethrin

PTC2 - incurred



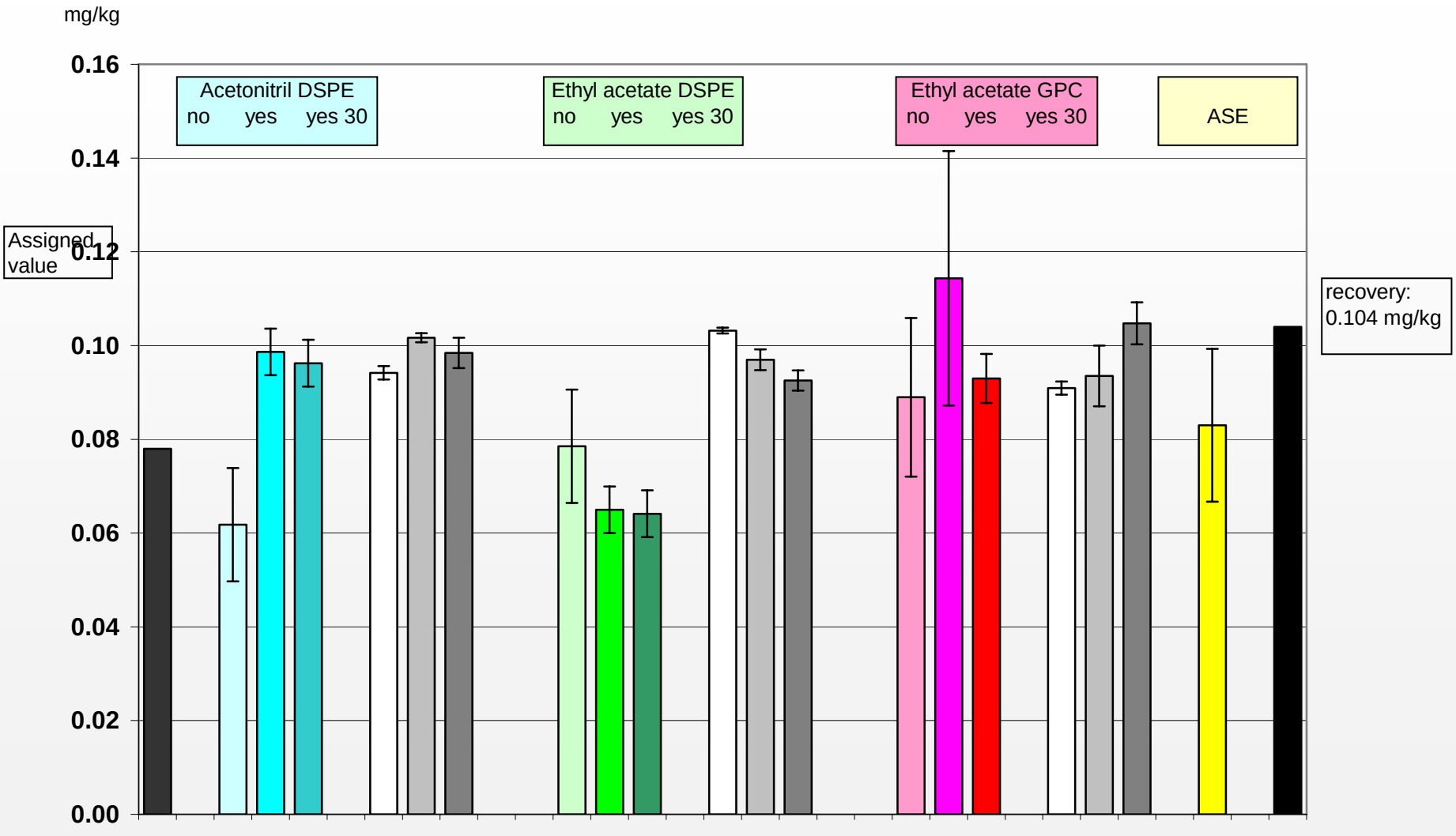
Bifenthrin

PTC2 - incurred



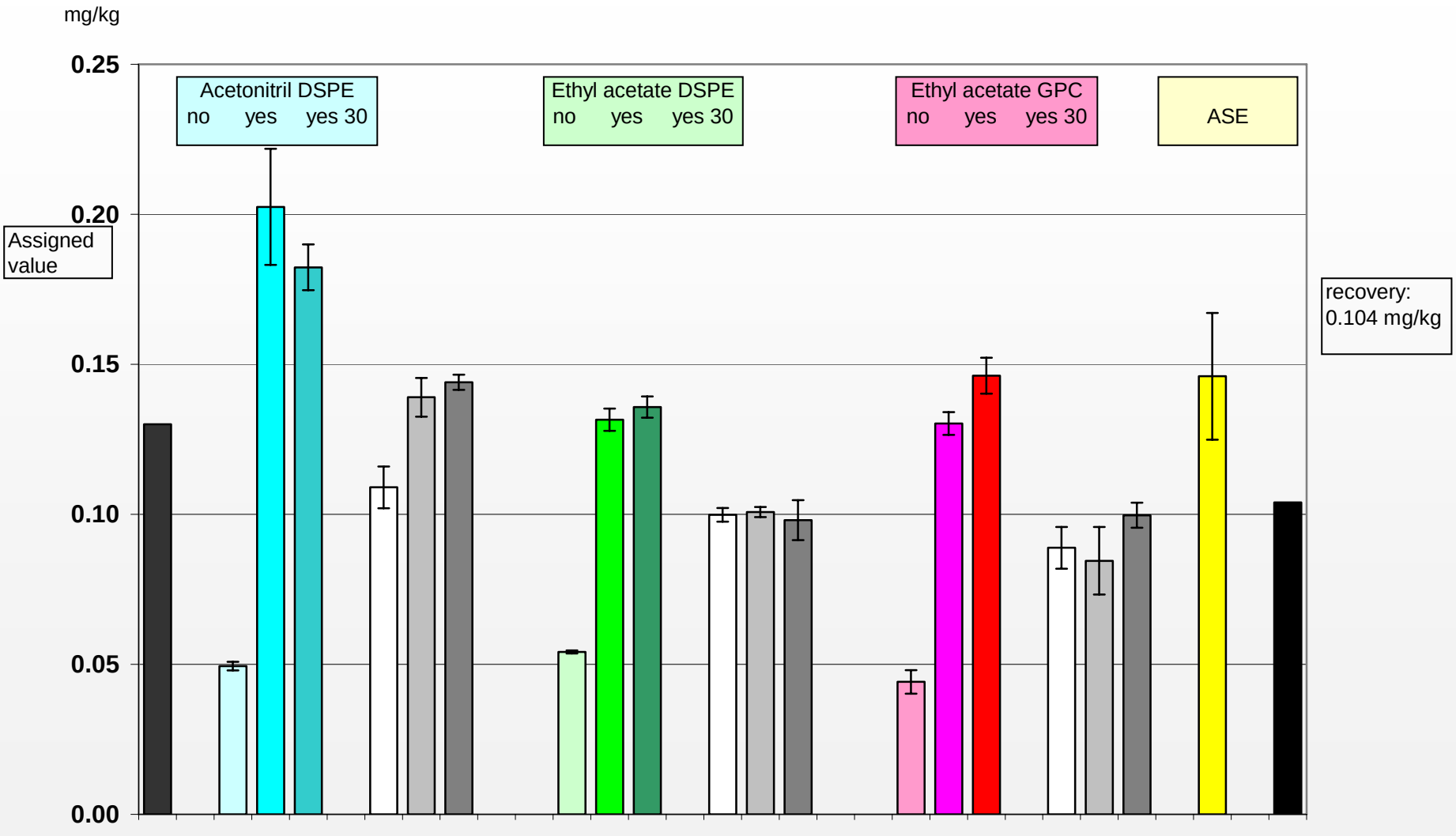
Diazinon

PTC1 - incurred



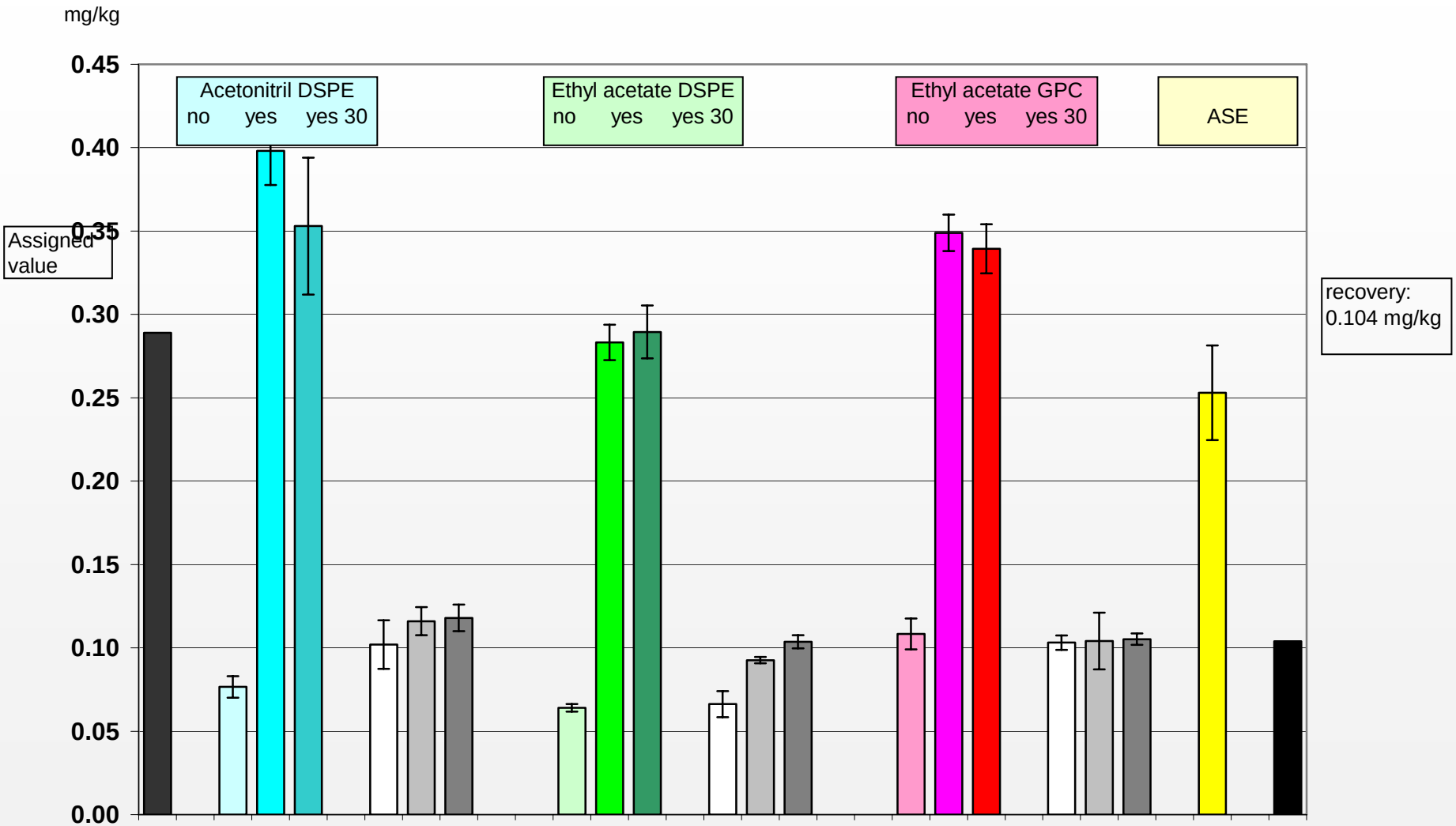
Chlorpyrifos-methyl

PTC2 - incurred



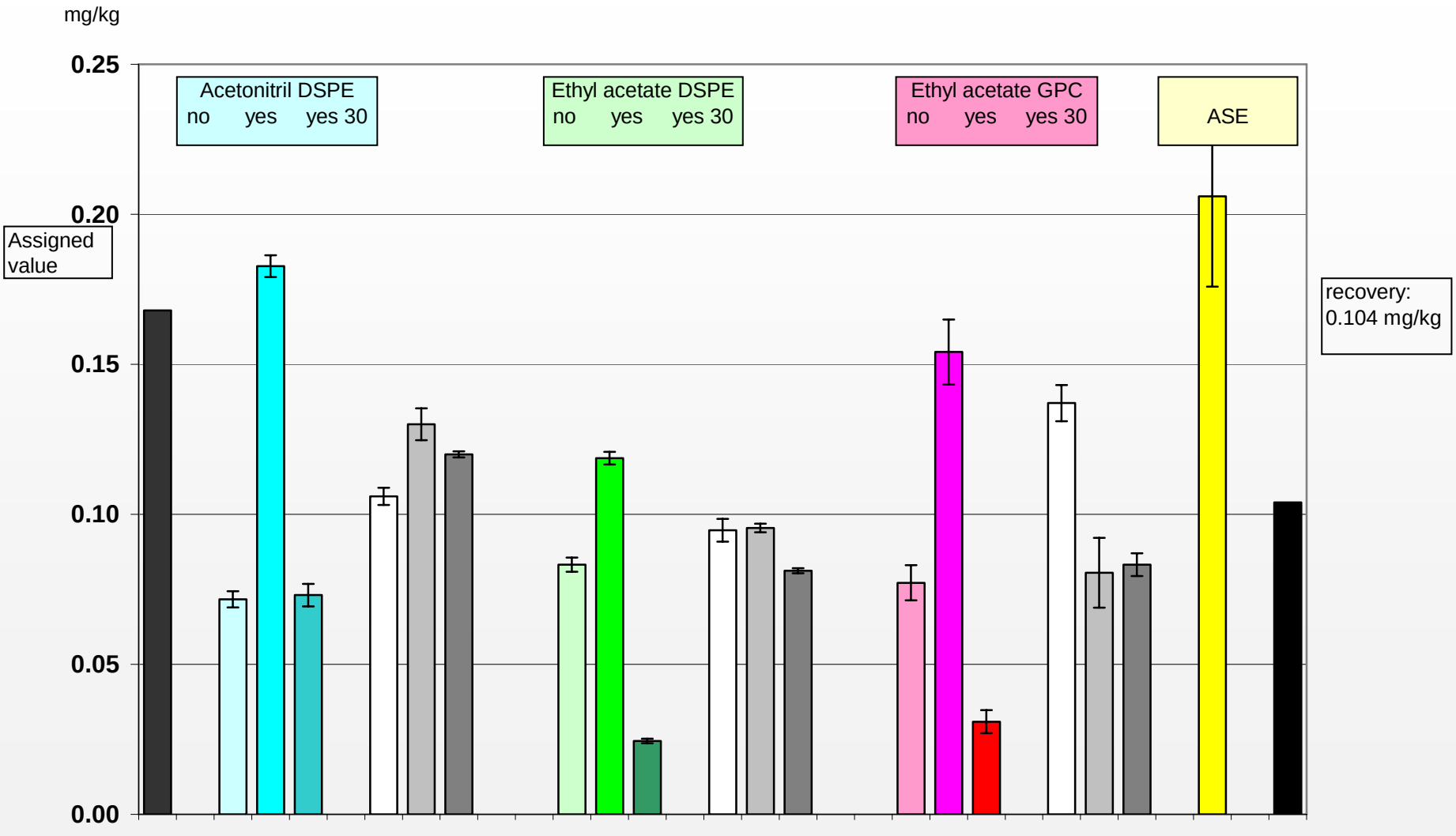
Iprodione

PTC2 - incurred



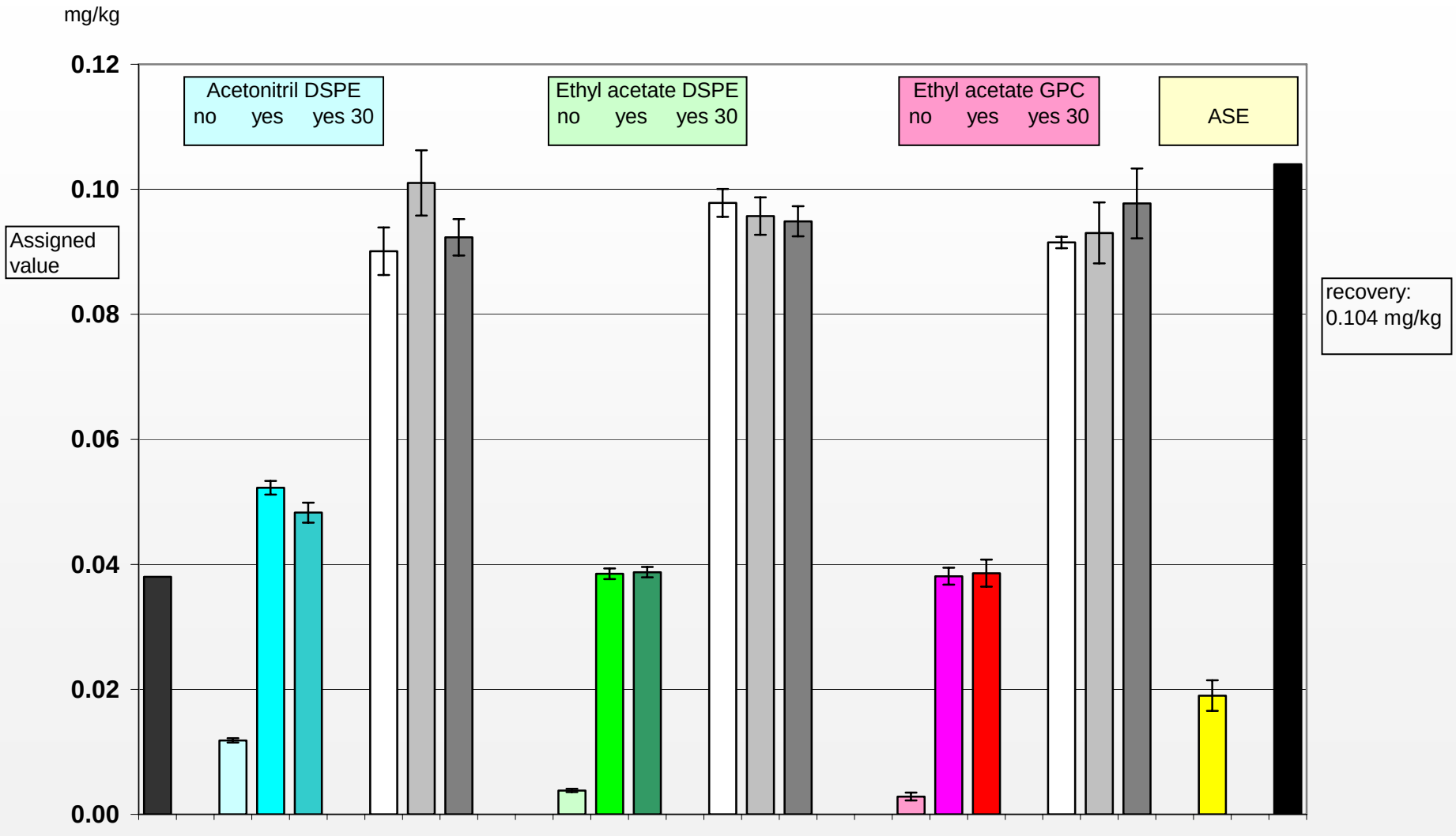
Malathion

PTC2 - spiked



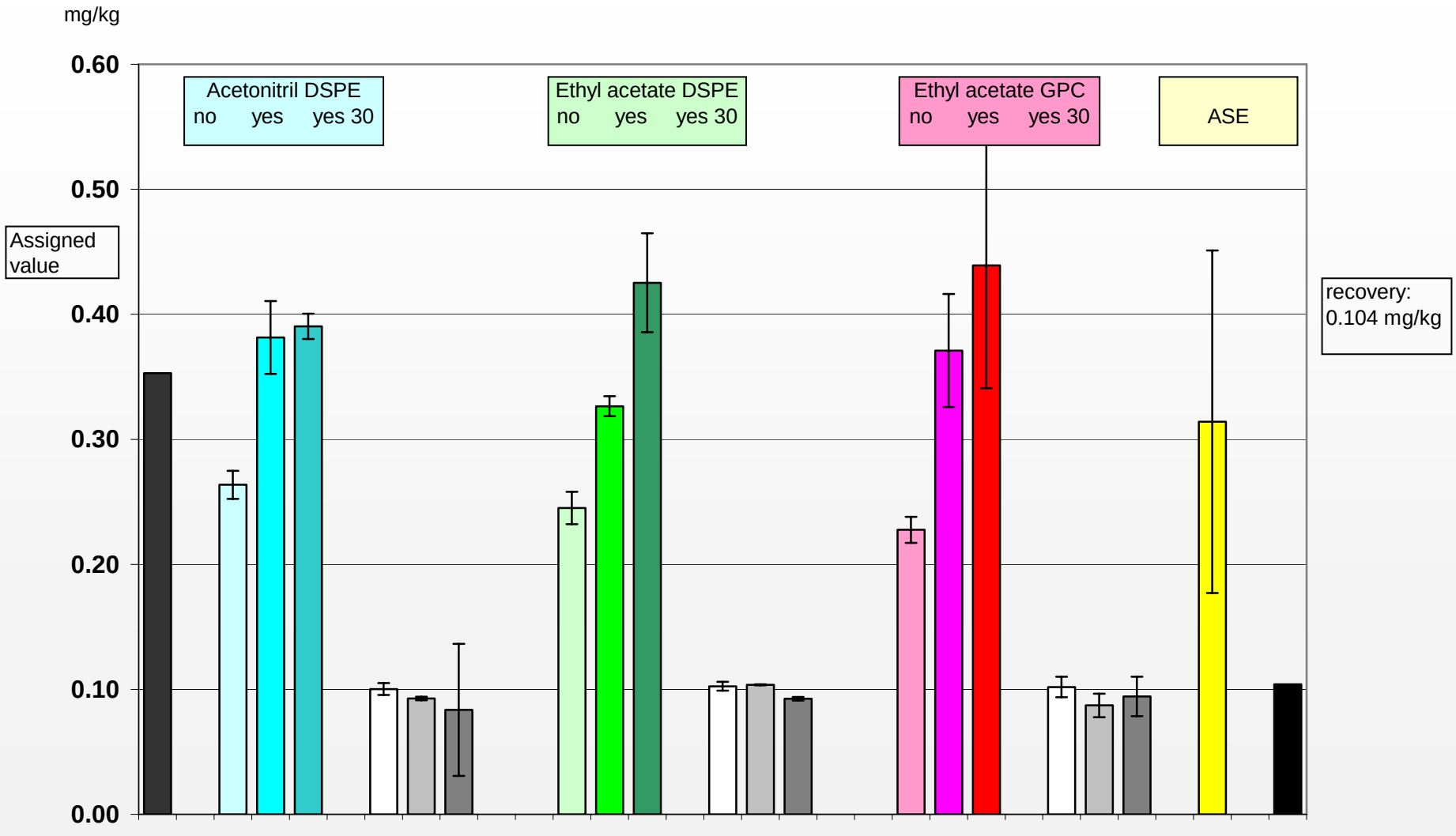
Pirimicarb

PTC2 - incurred



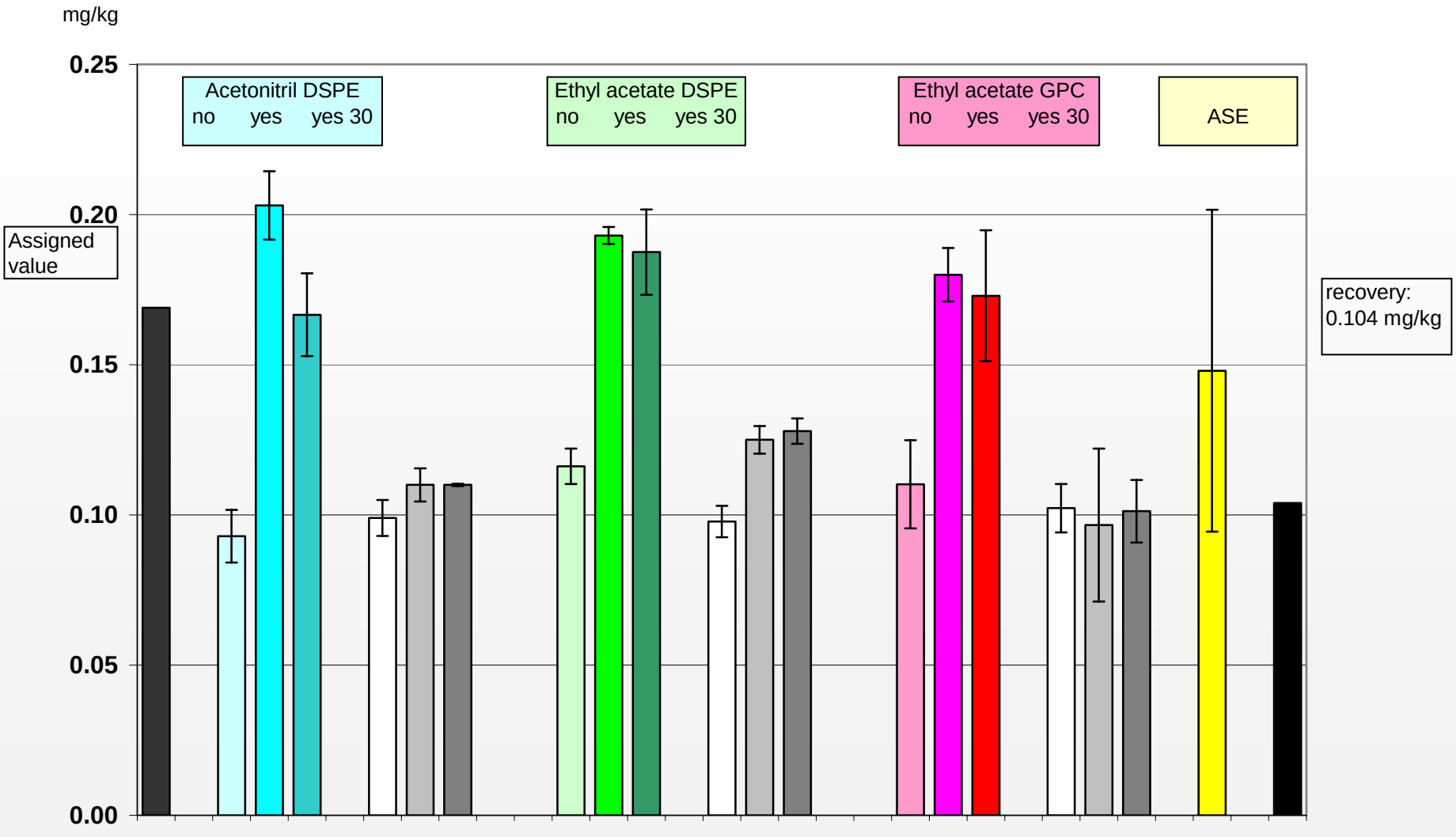
Propiconazole

PTC1 - spiked



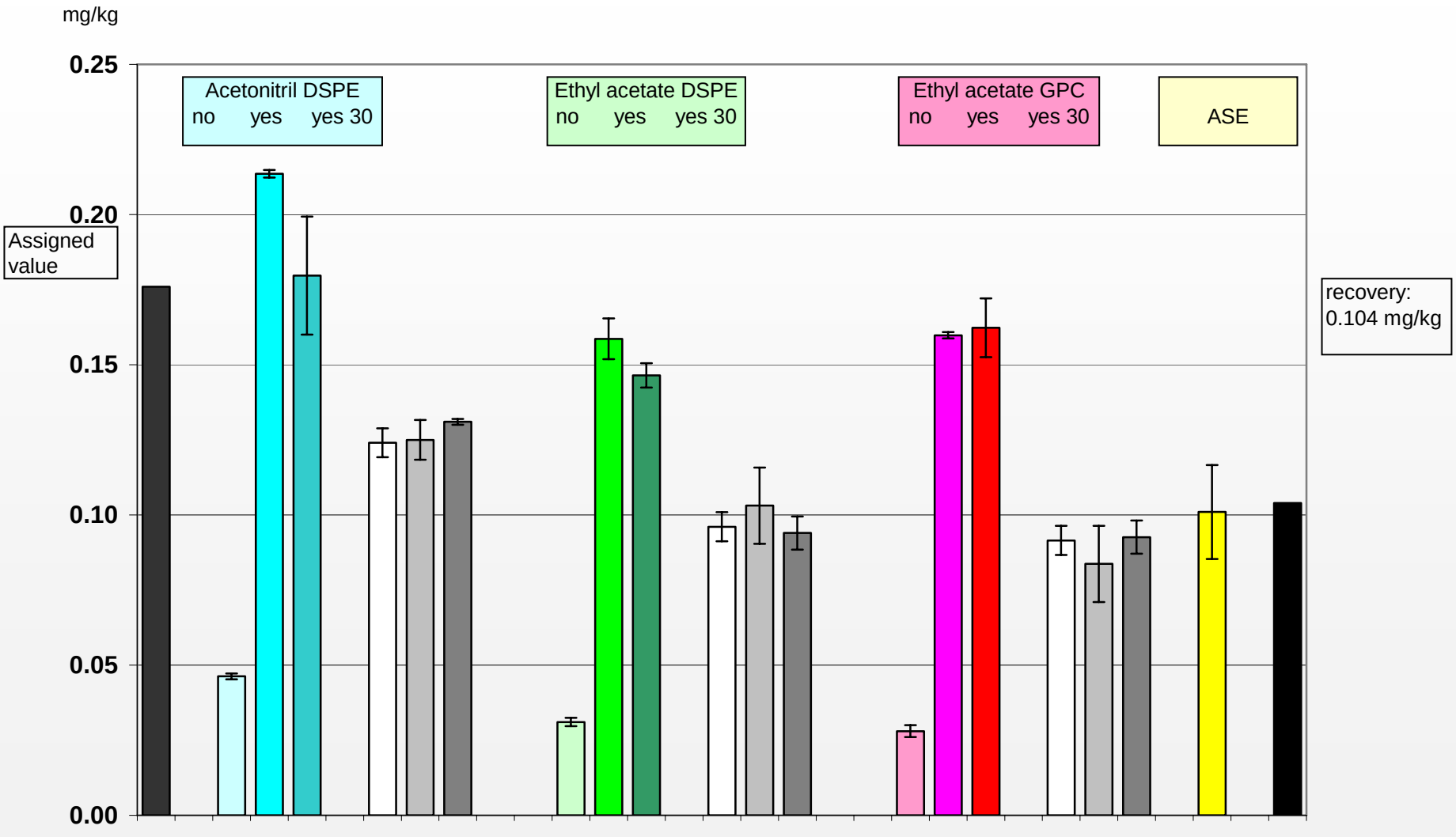
Difenoconazole

PTC2 - spiked



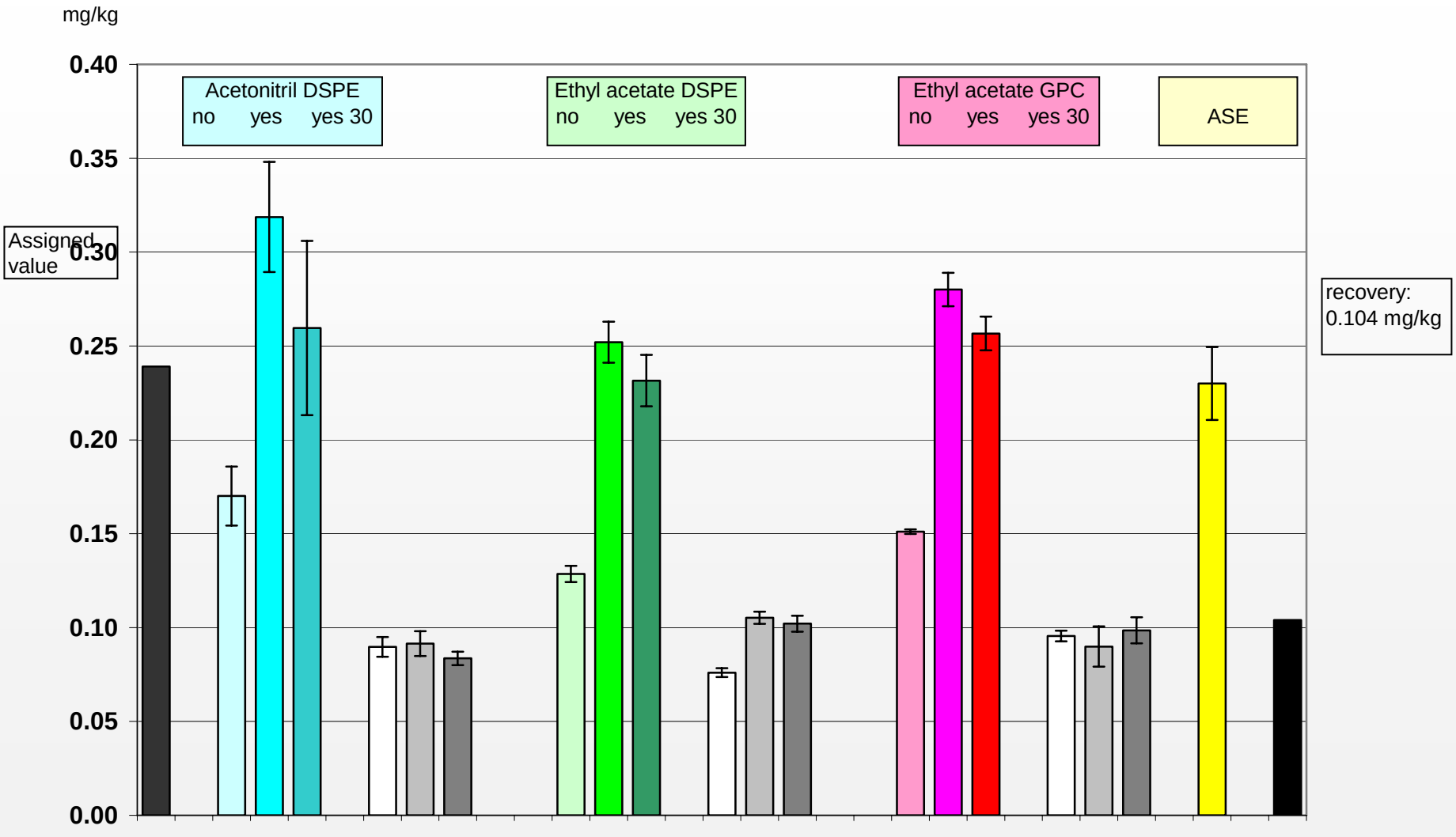
Epoxiconazole

PTC2 - incurred



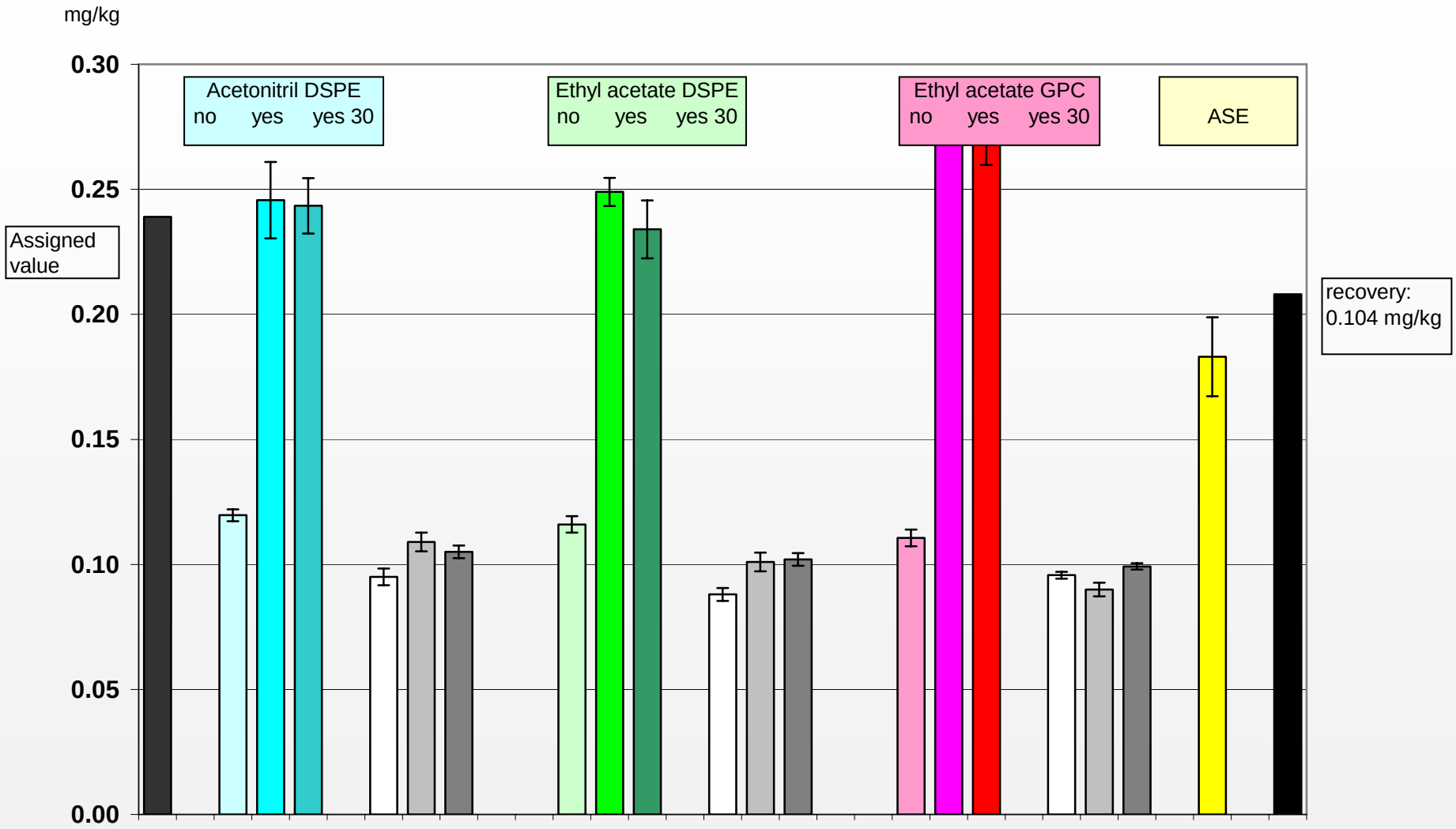
Prochloraz

PTC2 - spiked



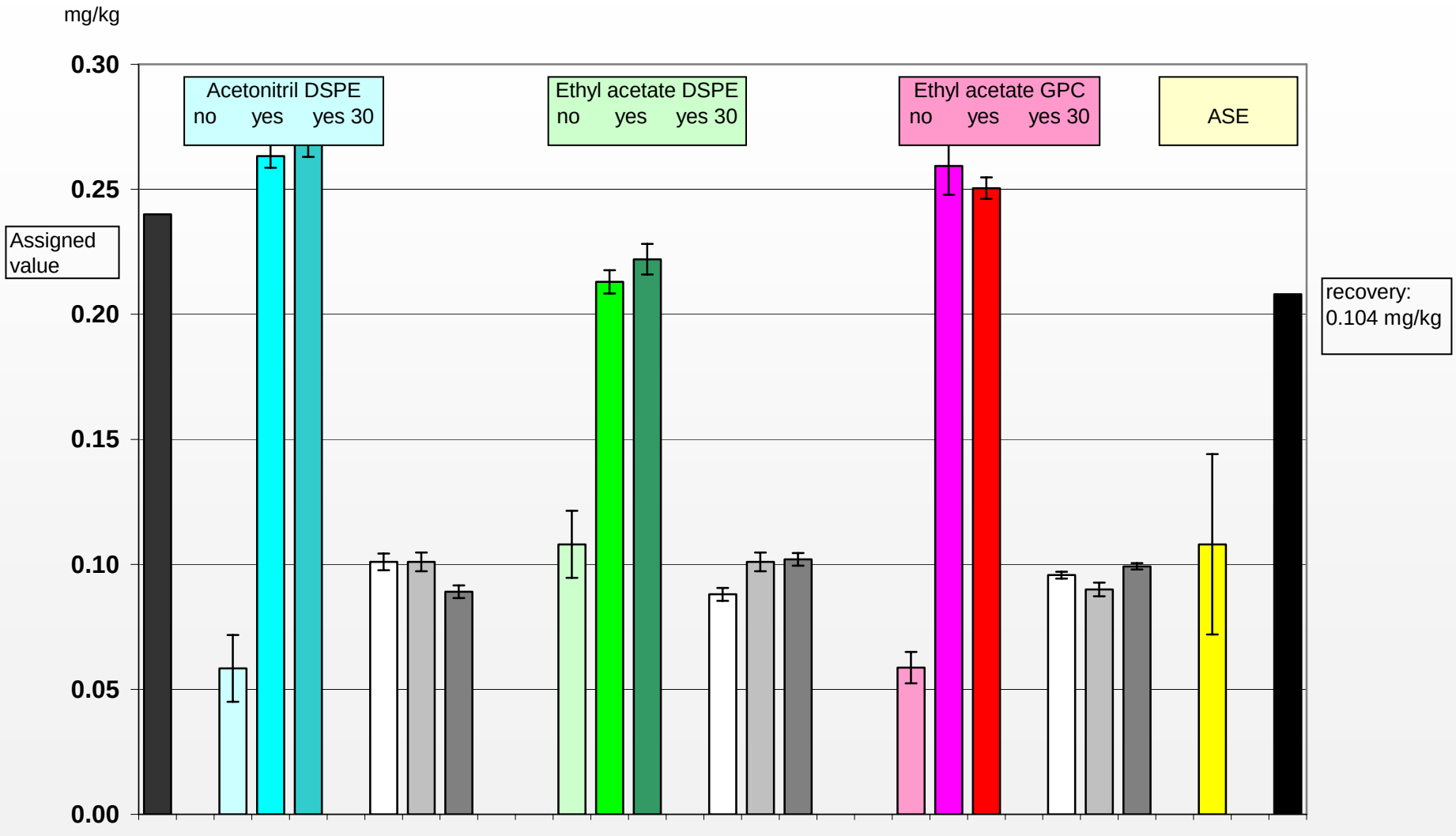
Azoxystrobin spiked

PTC2 - spiked



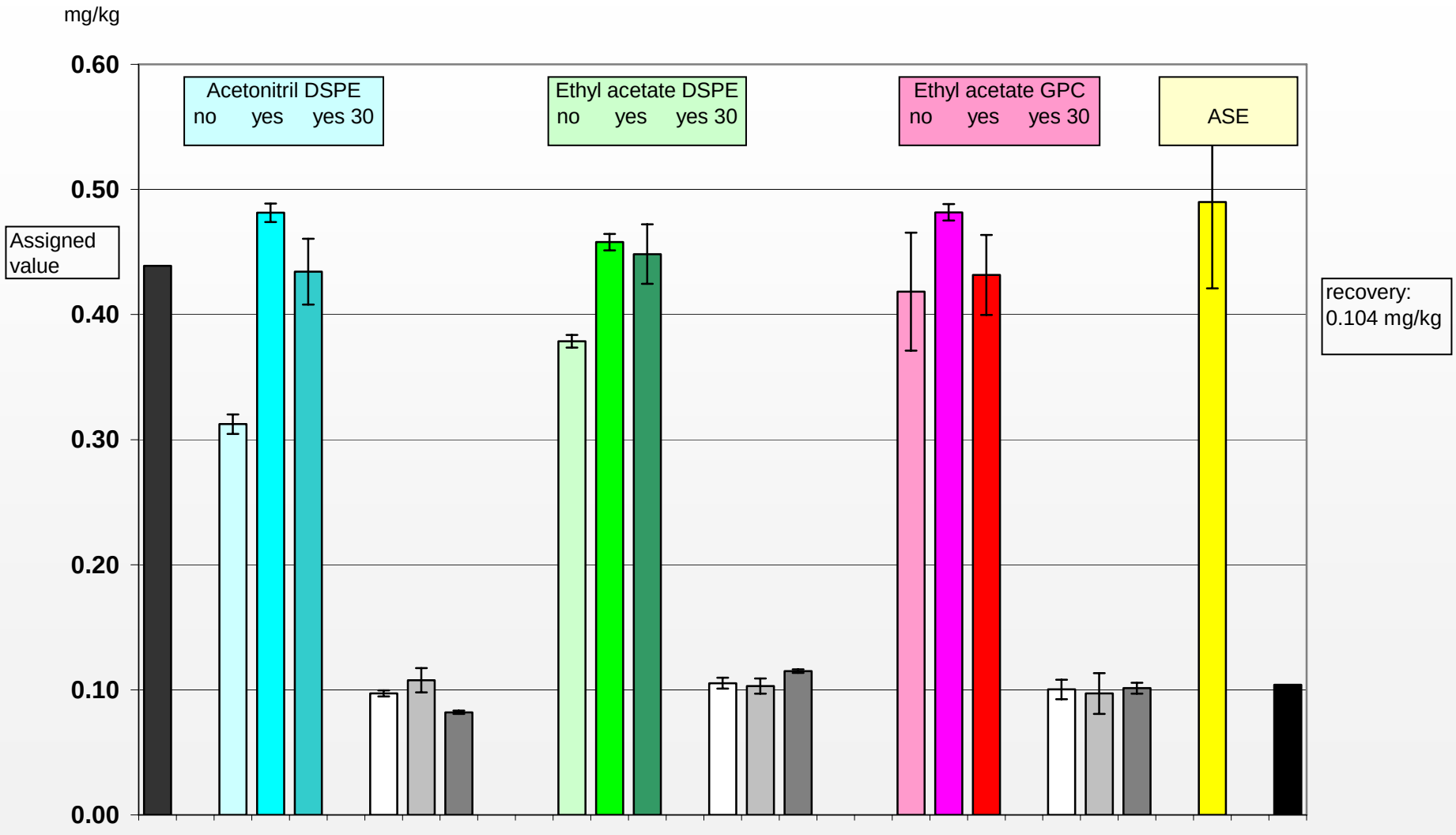
Azoxystrobin incurred

PTC1 - incurred



Trifloxystrobin

PTC2 - incurred



Conclusions

- Phosphor pesticides, azole pesticides, azoxystrobin and pirimicarb:
  - The extraction efficiency of incurred pesticides was increased when water was added to the samples before extraction
- Pyrethroids, organo chlorine pesticides (endosulfan):
  - no significant effect of water addition was seen
- The extraction efficiency was not increased if the samples were left for 30 min after water addition.
  - For some pesticides (malathion) the extraction efficiency droped, due to degradation
- Extraction efficiency was increased when ASE extraction were used without water addition