Toxins 2020, 12, 723 5 of 12

and 2) were smaller and so deteriorated that separated analyses of muscle and viscera were not possible. For mullets 3, 4, 5 and 6, a separate analysis of the muscle and digestive tract was conducted. GTX3 was quantified in the digestive tract of mullets 3 and 6. However PSTs were not detected in the corresponding muscle samples. Two mackerels (immediately caught by fishermen) were obtained from the contaminated area and separate analyses were conducted for the muscle and digestive tract in both of them. PSTs were only detected and quantified in mackerel 2 (Table 2), with results showing higher levels in the digestive tract. Five fresh squid samples were analyzed and only one of them showed low GTX3 levels. Surprisingly, two starfish, separately analyzed, presented low GTX2 and GTX3 concentrations. All these samples were well below the legal limit regulated in the EU legislation for PSTs (800 µg equivalents STX-diHCl/kilogram) [10]. PSTs were not detected in any of the six dolphin samples tested (either stomach or stomach contents) or in the homogenate of a triggerfish found dead. It is worth mentioning the results from one ascidians sample, showing levels around half the EU legal limit for PSTs and the same toxic profile (GTX1, 2, 3 and 4) as the cultures established from cells isolated from the bloom.

**Table 2.** Results from fish and invertebrate samples in which PSTs were quantified after analyses by HPLC-FLD-PCOX. (a) Next to 6: Náutico, (b) next to 3: Beiramar.

Samples and Their Codes	Sampling Point and Date	Tissues Employed for Analysis	Total Toxicity μg eq STX·diHCl/kg	Toxic Profile
Grey mullet-1	V: (-)	Marala di catina		
25 cm, 380 g	Vigo (a)	Muscle, digestive	132	GTX3
Liza ramada	10/07/18	tract, gonads		
Grey mullet-2				
25 cm, 420 g	Vigo (a)	Muscle, digestive	21.7	GTX3
Liza ramada	10/07/18	tract, gonads		37.0
Grey mullet-3				
40 cm, 750 g	Vigo (b)	Digestive tract	151	GTX3
Liza ramada	11/07/18	Digestive tract	131	GIAS
Grey mullet-6				
	Vigo (a)	D: .:	<b>67.0</b>	CTV2
37 cm, 550 g	20/07/18	Digestive tract	67.8	GTX3
Liza ramada	, ,			
Mackerel-2	Vigo (b)			
28 cm, 200 g	10/07/18	Muscle	27.6	GTX2, GTX3
Scomber scombrus	10/07/10			
Mackerel-2	Vice (b)			
28 cm, 200g	Vigo (b)	Digestive tract	292	GTX1, GTX2, GTX3, GTX4
Scomber scombrus	10/07/18	0		
Mussels-1				
65 g homogenate	Vigo (a)	Whole body	1877	GTX1, GTX2, GTX3, GTX4, STX
Mytilus galloprovincialis	20/07/18	vinole body	10,,	017(1) 017(2) 017(0) 017(1) 017(
Ascidians-1				
15 g homogenate	Vigo (a)	Whole body	363	GTX1, GTX2, GTX3, GTX4
Ciona intestinalis	20/07/18	whole body	303	G1X1, G1X2, G1X3, G1X4
Scallops-1	Vigo (a)	**** 1 1 1	2.427	CTV4 CTV4 CTV4 CTV4 CTV4 CTV4 CTV
37 g homogenate	20/07/18	Whole body	3437	GTX1, GTX2, GTX3, GTX4, STX, NE
Chlamys varia	,,			
Scallops-2	Vigo (a)			
33 g homogenate	20/07/18	Whole body	2670	GTX1, GTX2, GTX3, GTX4, STX, NE
Chlamys varia	20/07/10			
Oysters-1	Vice (a)			
24 g homogenate	Vigo (a)	Whole body	485	GTX1, GTX2, GTX3, GTX4
Östrea edulis	20/07/18	-		
Oysters-2	*** ( )			
5 g homogenate	Vigo (a)	Whole body	805	GTX1, GTX2, GTX3, GTX4
Ostrea edulis	20/07/18			,
Squids-5				
17 g homogenate	Vigo (a)	Whole body	16.3	GTX3
Loligo vulgaris	18/07/18	Wiloic Dody	10.5	01/0
Starfish-1				
	Vigo (a)	Amalauda anal ana	20.4	CTV2 CTV2
Marthasterias glacialis	20/07/18	Ambulacral groove	38.4	GTX2, GTX3
18 cm				
Starfish-2	Vigo (a)			OTT 14 OTT 14
Marthasterias glacialis	20/07/18	Ambulacral groove	57.8	GTX2, GTX3
16 cm	20,0.,10			

The five bivalve mollusks samples evaluated had the highest PSTs levels (Table 2). With the exception of one oyster sample, all had toxicity levels above the maximum total toxicity PSTs levels regulated in the EU. Mussels and scallops samples showed the presence of the four GTXs together with