ThP 095

Water quality determination: a myriad of analysis simplified

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Overview

- To enable analysis of different classes of compounds required by water control regulations a versatile and sensitive LC-MS/MS equipment is required.
- By applying Shimadzu LCMS-8050 (**Fig. 1**), with a variety of column it was possible to achieve enough sensitivity for the analysis of low volatility compounds required by Brazilian regulation, Portaria 888/21 MS.

Shimadzu LCMS-8050 was used for a variety of residue determination.

1. Introduction

- On 2021, Brazilian regulation for tap water quality determination was updated with the addition of compounds and reducing some maximum residue levels (MRL), which required development of methodologies that are both versatile and sensitive. Different classes of compounds are included in the regulation, within a wide range of polarities and requiring no pre-concentration in the final methods, following tendencies of other countries.
- The broad range of compounds are analyzed between GCMS, LCMS and other techniques, but even within compounds that are suitable for LCMS, polarities vary greatly. In that sense Nexera UHPLC system coupled to LCMS-80XX equipment can be used as a single platform for the analysis of medium to low volatility compounds, from phenols to glyphosate, reaching the MRLs required by the regulation.

2. Methods

- A LCMS-8050 coupled to a Nexera XS was used for most analysis in this work. Electrospray ionization was used as default, with APCI being required for some regulated compounds as NDMA and Phenols (data not showed here). Mobile phases consisted in methanol or acetonitrile, along with water in binary gradient, with acid and ammonium salts.
- Shim-pack GIST C18-AQ was used for the analysis of all compounds, but glyphosate and AMPA which was analyzed under Restek Raptor Polar X columns
- Labsolutions Connect (**Fig. 2**) was used for MRM and source optimization; data was processed using Labsolutions Insight.

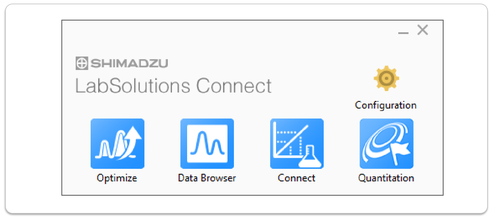
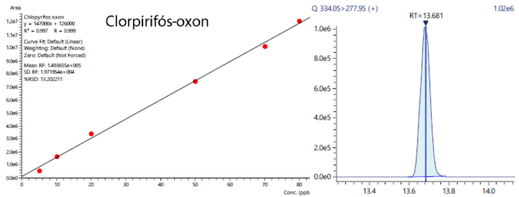


Fig. 2 Labsolutions Connect interface, comprising the entire workflow from optimization, results review, method development, batch processing and data analysis

3. Result

- Below are found the results for a variety of compounds determined, showing sensitivity below MRLs



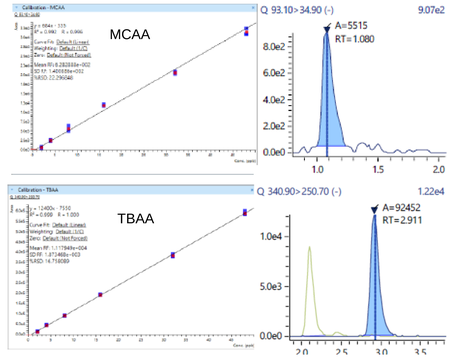


Fig. 3 Chlorpyrifos-oxon (5-100µg/L), monochloroacetic acid (2-48µg/L) and tribromoacetic acid (2-48µg/L) with chromatogram for the lowest point

Compound	MRLs (µg/L)	LOQ (µg/L)	Compound	MRLs (µg/L)	LOQ (µg/L)
2,4-D	30,0	5,0	Hydroxy-atrazine	120,0	20,0
Acephate	7,0	1,0	Imidazoliditione (ETU)	8,0	1,0
Acrilamide	0,5	0,1	Malathion	60,0	20,0
Aldicarb	10,0	2,0	Metamidophos	7,0	1,0
Aldicarb sulfoxide	10,0	2,0	Metholachlor	10,0	1,0
Aldicarb sulfone	10,0	1,0	Metribuzim	25,0	5,0
Ametryn	60,0	10,0	Molinate	6,0	1,0
Atrazine	2,0	0,05	Omethoate	1,2	0,05
Deaminochlortriazine	2,0	0,05	Picloram	60,0	10,00
Carbendazim	120,0	20,0	Propargite	30,0	5,0
Carbofuran	7,0	1,0	Prothioconazole	3,0	0,1
Chlorpyrifos-oxon	30,0	5,0	Prothioconazole-desthio	3,0	0,05
Chlorpyrifos	30,0	5,0	Simazine	2,0	0,05
Cyproconazole	30,0	5,0	Tebuconazole	180,0	10,0
Desethyl-atrazine	2,0	0,1	Terbufos	1,2	0,1
Desisopropyl-atrazine	2,0	0,05	Tiamethoxam	36,0	10,0
Difenoconazole	30,0	5,0	Tiodicarb	90,0	20,0
Dimethoate	1,2	0,05	Microcystin (each)	0,3	0,15
Diuron	20,0	5,0	Haloacetic acids	80,0 (sum)	2,0
Epoxyconazole	60,0	10,0	Glyphosate	500,0 (sum)	50,0
Fipronil	1,2	0,05	AMPA		
Flutriafol	30,0	5,0			

Table 1 Compounds analyzed from Portaria 888/21, MRLs and lowest point from the callibration curve

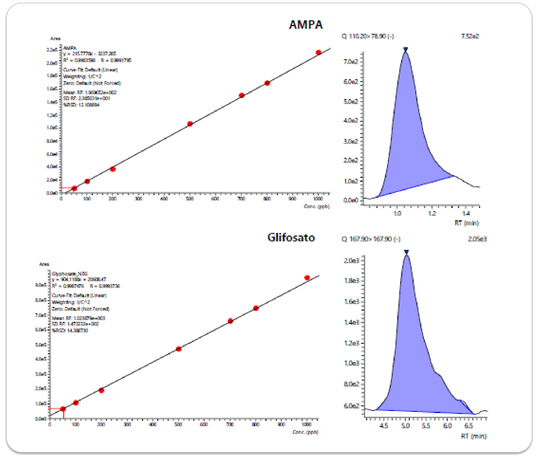


Fig. 4 Glyphosate and AMPA determination from 50-1000 µg/L, with chromatogram for the 50µg/L concentration

#	Name	Conc.	Unit	m/z
1	DCAA	4.4211	ppb	127.00>82.90
2	MCAA	4.5236	ppb	93.10>34.90
3	BCAA	4.3479	ppb	218.80>173.00
4	DBAA	4.3502	ppb	262.80>216.90
5	MBAA	4.3075	ppb	182.90>137.00
6	TCAA	4.5052	ppb	207.00>117.00
7	BDCAA	4.3374	ppb	252.80>163.00
8	CDBAA	4.3939	ppb	297.10>207.00
9	TBAA	4.3023	ppb	340.90>250.70
10	acidos haloacéticos totais	39.4892	ppb	----

Table 2 Concentration sums used on Labsolutions Insight for the haloacetic acids

4. Conclusion

- ✓ Shimadzu LCMS system was able to detect a wide range of compounds from different polarities below the regulated MRLs for tap water quality Brazilian regulation.
- ✓ Column and mobile phase switching can be used in Nexera UHPLC, enhancing operability and throughput for the sample analysis