



iMethods for
Environmental Analysis

A Rapid iMethod™ Test for Analysis of Acid Herbicides in Water

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The following description outlines the instrument requirements and expected results obtainable from the Spark Holland iMethod™ Test for the quantification of twenty two acid herbicides in water when using a Spark Holland Symbiosis™ PICO integrated online SPE and HPLC system with a Mistral™ column oven and AB SCIEX 4000 series (API 4000™ or 4000 QTRAP®) LC/MS/MS instrument.



Sample preparation is based upon filtration and PH adjustment followed by on-line SPE sample clean up and concentration using the capabilities of the Spark PICO system in conjunction with Spark HySphere™ SPE C18HD sorbent cartridges. More in depth sample preparation, and instrument parameter information is included as part of the standard operating procedure provided with the method. Solvents, standards and any supplies required for sample preparation are not.

The mobile phase consists of the use of an acetonitrile/ methanol / water/ ammonium acetate mixture with separation on a Waters Atlantis dC18 3.5 µm 2.1 x 50 mm HPLC column. An example chromatogram of the separation achieved is shown below in figure 1.

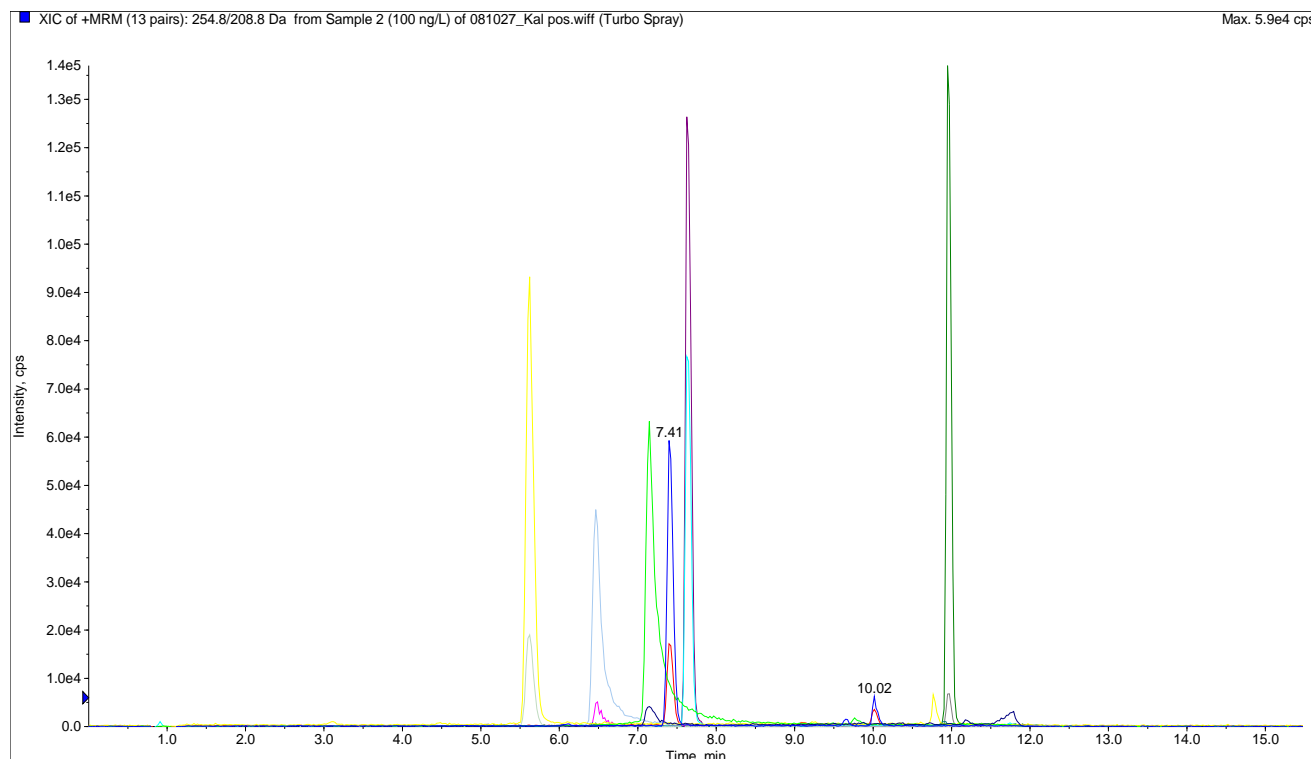


Figure1: Chromatogram of a 100 ng/L spiked water sample and run on an API 4000™ LC/MS/MS System in + ESI mode according to the compound list shown in table 1.

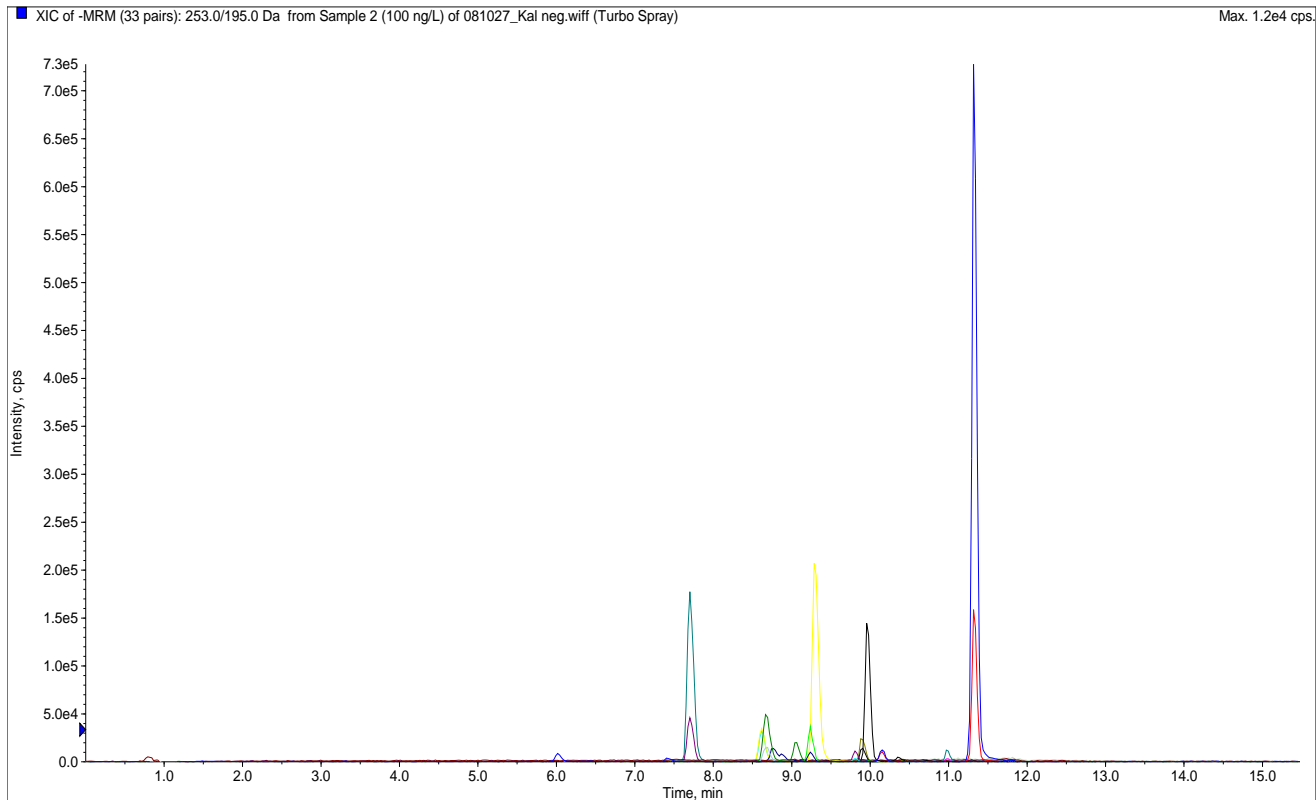


Figure 2: Chromatogram of a 100 ng/L spiked water sample and run on an API 4000TM LC/MS/MS System in - ESI mode according to the compound list shown in table 1.

Compound	Mode	Retention Time (Min)	Compound	Mode	Retention Time (Min)
Haloxypop	ESI +	11.00	Dichlorprop	ESI -	17.41
Nicosulfuron	ESI +	7.68	Fluazifop	ESI -	17.24
Fluroxypyr	ESI +	7.46	loxynil	ESI -	16.85
Sulcotrione	ESI +	7.17	MCPA	ESI -	16.79
Mesotrion	ESI +	6.47	2,4-D	ESI -	16.66
Quinmerac	ESI +	5.61	DNOC	ESI -	16.34
Dinoterb	ESI -	18.49	Bromoxynil	ESI -	16.27
Fenoxaprop-P	ESI -	18.48	Bentazon	ESI -	15.46
MCPB	ESI -	17.89	Dicamba	ESI -	14.66
2,4-DB	ESI -	17.86	Clopyralid	ESI -	4.89
2,4,5-T	ESI -	17.67	Mecoprop	ESI -	17.46

Table 1: Compound name, retention time, and ESI mode (pos/neg).

Results

The following calibration curves are representative of the performance obtained on the instrument using the method described here, and may not be representative of performance on any other instrument.

Substance	Mode	LOQ [ng/L]	LOD [ng/L]	Correlation coefficient
2,4,5-T	ESI -	11.84	3.19	0.9995
2,4-D	ESI -	9.83	2.65	0.9996
2,4-DB	ESI -	30.29	8.27	0.9966
Bentazon	ESI -	3.34	0.90	0.9999
Bromoxynil	ESI -	4.47	1.19	0.9999
Clopyralid	ESI -	11.91	3.15	0.9996
Dicamba	ESI -	28.95	7.52	0.9993
Dichlorprop	ESI -	10.29	2.75	0.9995
Dinoterb	ESI -	7.19	1.81	0.9999
DNOC	ESI -	5.02	1.35	0.9997
Fenoxypop-P	ESI -	9.69	2.63	0.9990
Fluazifop	ESI -	5.13	1.39	0.9998
Ioxynil	ESI -	5.98	1.62	0.9998
MCPA	ESI -	3.64	0.98	0.9999
MCPB	ESI -	14.10	3.76	0.9995
Mecoprop	ESI -	10.17	2.74	0.9995
Fluroxypyr	ESI +	2.29	0.60	1.0000
Haloxypop	ESI +	5.00	1.36	0.9998
Mesotrion	ESI +	8.09	2.19	0.9997
Nicosulfuron	ESI +	1.68	0.45	0.9999
Quinmerac	ESI +	4.15	1.11	0.9999
Sulcotrione	ESI +	4.72	1.28	0.9998

Table 2: Representative limits of quantitation (LOQ), limits of detection (LOD) and correlation coefficients obtained.

Please note that the results presented above were obtained using a single instrument and single set of standards and samples. Prior to production use, the method should be fully validated with real samples, and the results here may not be typical for all instruments. Variations in LC column properties, chemicals, environment, instrument performance and sample preparation procedures will impact performance, thus these results should be considered as informative rather than representative.

System Requirements

In order to run this method as outlined above, the following equipment and reagents are required:

- An AB SCIEX 4000 Series LC/MS/MS System
- Spark Holland Symbiosis™ PICO Online SPE HPLC system with a Mistral™ column oven
- Acid herbicide standards (www.sigmaaldrich.com)
- LC/MS grade acetonitrile, methanol, water, ammonium acetate
- A Waters Atlantis dC18 3.5 µm 2.1 x 50 mm HPLC column
- Spark HySphere™ C18HD SPE cartridges
- Pipettes and standard laboratory glassware

Please note that the Waters Atlantis HPLC column is required but not included with this iMethod Test. Also, that this method can also be run on other HPLC systems, given that they are supported for use by Cliques® Software and the retention times are updated to reflect the configuration used.

Important Note

The purchase and use of certain chemicals listed above may require the end user to possess any necessary licenses, permits or approvals, if such are required in accordance with local laws and regulations. It is the responsibility of the end user to purchase these chemicals from a licensed supplier, if required in accordance with local laws and regulations. The suppliers and part numbers listed below are for illustrative purposes only and may or may not meet the aforementioned local requirements.

Product Name

Part Number

iMethod™ Test for the Analysis of Herbicides in Water

free download at www.absciex.com

Legal Acknowledgements / Disclaimers

The iMethod™ Test described above has been developed by AB SCIEX to provide all the sample prep and instrument parameters required to accelerate the adoption of this method for routine testing. The performance of this method will need to be verified in a given lab due to potential variations in instrument performance, maintenance, chemicals and procedures used, technical experience, sample matrices and environmental conditions. It is the responsibility of the end user to make adjustments to this method to account for slight differences in equipment and/or materials from lab to lab as well as to determine and validate the performance of this method for a given instrument and sample type. Please note that a working knowledge of Analyst® Software may be required to do so.

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Publication number: 1430110-01

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