

The Analysis of Acid Herbicides by RLVI/GC/MS

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Introduction

Acid herbicides were first introduced as weedkillers in the 1940s. They are applied as esters or salts, as they are readily metabolised in this form, to the top of the soil or grass, regulating the growth of mainly broadleaf weeds. They readily degrade in the environment, however the esters are oil soluble and form emulsions in water, whereas the salts are highly soluble in water, therefore the leaching of the acid herbicides into the groundwater causing contamination is of major concern.

The analysis of acid herbicides using large volume injection enables detection limits to be more easily reached, and therefore sample preparation to be reduced. A method for the large volume injection using the Optic is presented, along with some performance data.

Instrumentation and Conditions

- ATAS Optic 2-200 programmable injector
- Varian Star GC/MS

Optic Conditions

Injection volume: 100 μ L
 Solvent type: iso-Octane
 Liner: ATAS 'A' Type
 Mode: Large Volume
 Gas Flows: Split: 50 ml/min
 Vent: 150 ml/min

Initial temperature: 55 $^{\circ}$ C
 Vent time: 0:35 m:s
 Ramp rate: 4 $^{\circ}$ C/s
 Final temperature: 280 $^{\circ}$ C
 End time: 16 mins
 Split open time: 3 mins
 Purge pressure: 7.5 psi
 Transfer pressure: 10 psi
 Transfer time: 0 mins
 Initial pressure: 10 psi
 Final pressure: 19.7 psi

GC conditions:

Column: HP5-MS 30m x 0.25 mm i.d. x 0.25 μ m film
 Initial Temperature: 90 $^{\circ}$ C
 Ramp Rate: 18 $^{\circ}$ C/min
 Final Temperature: 260 $^{\circ}$ C (8 mins)

MS conditions:

Mode: EI Scan
 Mass range: 50-560 m/z
 Scan time: 0.500
 Segment length: 16 mins
 Fil./Mul. Delay: 7.50 mins

Chromatogram

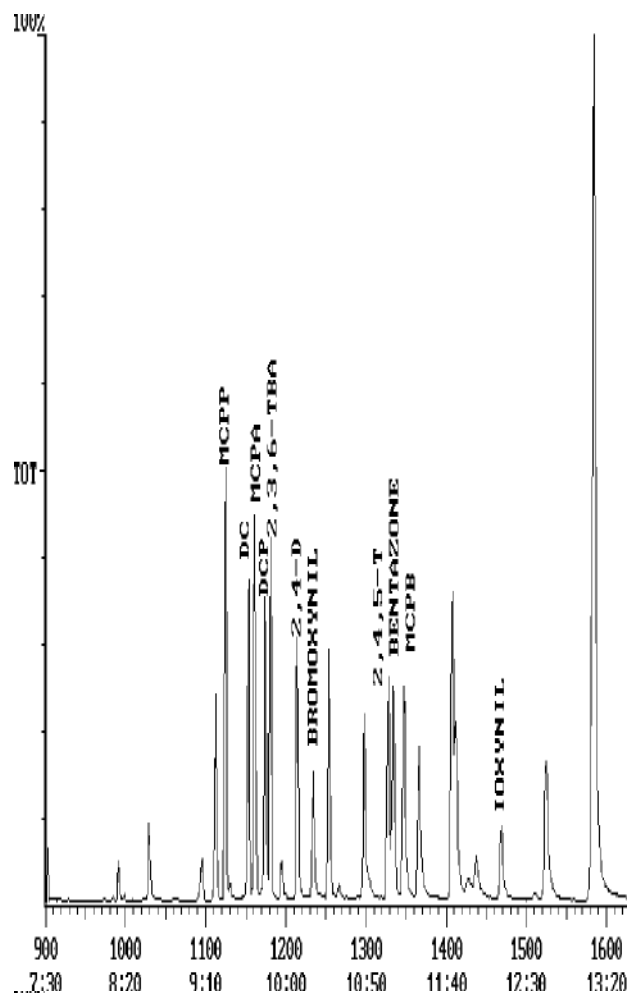


Figure: TIC trace of 100 μ L injection of pentafluorobenzylated acid herbicides

	MCPP	Dicamba	MCPA	DCP	2,3,6-	2,4-D
	18378519	6272885	17622206	16036002	17731526	15483243
	15973951	6042029	16453422	15766101	16549488	14595024
	17077417	5556178	16233722	15884024	16093709	13681623
	16065303	5545997	17128006	16991573	17685038	14560007
	18215936	5884711	17407745	15526930	16213725	15539061
	17477382	6367606	17507105	15938242	17724500	14951204
	16693034	6554292	17778900	14621522	16422293	14561732
	18247275	5849119	16572871	17160051	17669785	14937468
	18321583	5659629	16622656	17244105	18224494	14125622
Mean	17383378	5970271.8	17036293	16129839	17146062	14714998
Std Dev	975502.11	364975.73	573642.46	860397.84	810986.77	597044.89
%RSD	5.61	6.11	3.37	5.33	4.73	4.06

	Bromoxynil	Bentazone	2,4,5-T	MCPB	loxynil
	2021145	3859888	16370278	12752681	5928920
	1747445	4077172	15055415	13057591	5431112
	1669279	3826801	15739879	13260490	5441033
	1808800	3882137	16059119	13242397	5795764
	1710949	3593640	15703900	12637239	5940109
	1570204	4092116	15924068	13039286	5598643
	1851130	3863778	15263766	12983837	5675318
	1432978	4017227	15614099	12898000	5730178
	1580814	4079584	16337942	13608795	5616698
Mean	1710304.89	3921371.4	15785385	13053368	5684197.2
Std Dev	174113.416	163031.84	445052.58	291525.42	185430.89
%RSD	10.18	4.16	2.82	2.23	3.26

Tables: RSDs by peak area of 9 repeated injections of 100 μ L pentafluorobenzylated acid herbicides

Conclusions

The 'A' Type liner is suitable for the large volume analysis of acid herbicides, as a means of increasing the injection volume thereby reducing detection limits and decreasing the sample size and preparation.

Acknowledgements

We would like to thank Surinder Verik from Anglian Water for providing us with the data for this application note.