

ePrep Sample Preparation Workstation | Application Note 2020

Preparation of Glyphosate 360 Weed Killer Product for Ion Chromatography Analysis using the ePrep

INTRODUCTION

Bayer USA invited our USA Distributor (EST) to demonstrate if the ePrep (liquid dispense only) could match their current manual “gravimetric” chromatographic method for the sample preparation and subsequent analysis of their Glyphosate in a finished product. Their gravimetric methods have RSD less 0.3% with a reported instrument variability of 0% RSD.

The details of the Bayer chromatographic method are unknown but it has been inferred that an HPLC-RI detector is used.

Precision sample preparation studies were carried out at University Technology Sydney (UTS). As a RI detector was not available, an ion chromatography method was developed. This chromatographic technique was then used to analysis the ePrep sample preparation.



SUMMARY OF RESULTS

Glyphosate was analysed by Ion Chromatography using a fixed-loop injector with conductivity detection. The original formulation was diluted firstly from 360 g/l to 2400 ppm and then diluted again to a final concentration of 24 ppm. The contribution of the analytical method Relative Standard Deviation (RSD) was determined first and then the total RSD, which included the ePrep sample preparation and analytical method, was determined.

The RSD of the analytical method (based on peak area) was determined through repetitive injections of a single standard solution and an ePrep-prepared sample solution. The average of these two series of analysis shows the instrument RSD is 0.78%

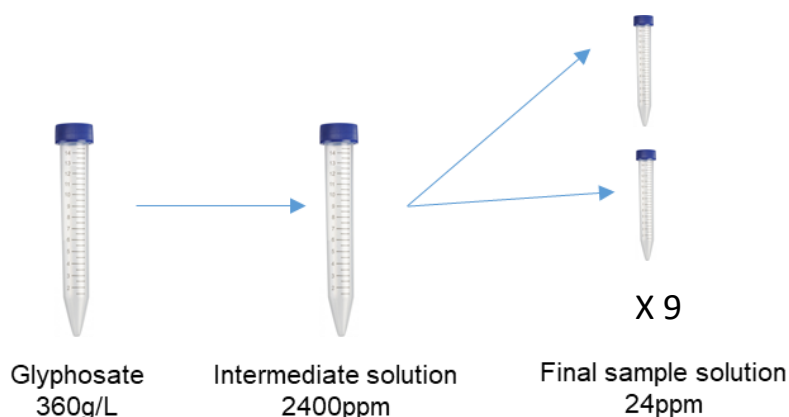
The RSD of the combined ePrep sample preparation and analytical method was determined by analyzing nine individually ePrep-prepared samples. Each sample was injected once and a blank was run between sample injections. The combined RSD was found to be 0.83%.

Therefore, the results show the contribution of the total RSD due to the ePrep sample preparation is minimal.

PROCEDURE

Sample Preparation:

The original formulation used was Weed Killer 360. This formulation was diluted twice (by the ePrep) to a final concentration of 24 ppm suitable for ion chromatography method. Two dilutions were performed to obtain the 24ppm solution.



Preparation of the intermediate 2400ppm solution

Tool Buffer (backlash)= 4%

Tool Wash = Auto

Prime Settings = Auto

- 1) Add Diluent 9000 μ L, Water (for needle dipping) [Asp: Auto@250uL/sec, Pause 2 sec | Disp: Auto Low@500uL/sec, Pause 2 sec]
- 2) Add Reagent 67 μ L, Nett Weed Killer 360 [Asp: Auto@3uL/sec, Pause 1 sec | Disp: Auto Low@100uL/sec, Pause 1 sec] *slow aspiration speed dur to viscosity and needle dipped on dispense to eliminate bubble formation*
- 3) Make up to Volume 10000 μ L, Water [Asp: Auto@250uL/sec, Pause 2 sec | Disp: Auto Low@150uL/sec, Pause 2 sec]

Preparation of final 24ppm solutions (x9)

Tool Buffer (backlash)= 4%

Tool Wash = Auto

Prime Settings = Auto

- 1) Add Diluent 9000 μ L, Water (for needle dipping) [Asp: Auto@250uL/sec, Pause 2 sec | Disp: Auto Low@500uL/sec, Pause 2 sec]
- 2) Add Reagent 100 μ L, 2400ppm Weed Killer Intermediate [Asp: Auto@3uL/sec, Pause 1 sec | Disp: Auto Low@100uL/sec, Pause 1 sec] *slow aspiration speed dur to viscosity and needle dipped on dispense to eliminate bubble formation*
- 3) Make up to Volume 10000 μ L, Water [Asp: Auto@250uL/sec, Pause 2 sec | Disp: Auto Low@150uL/sec, Pause 2 sec]

Preparation of the standard solution

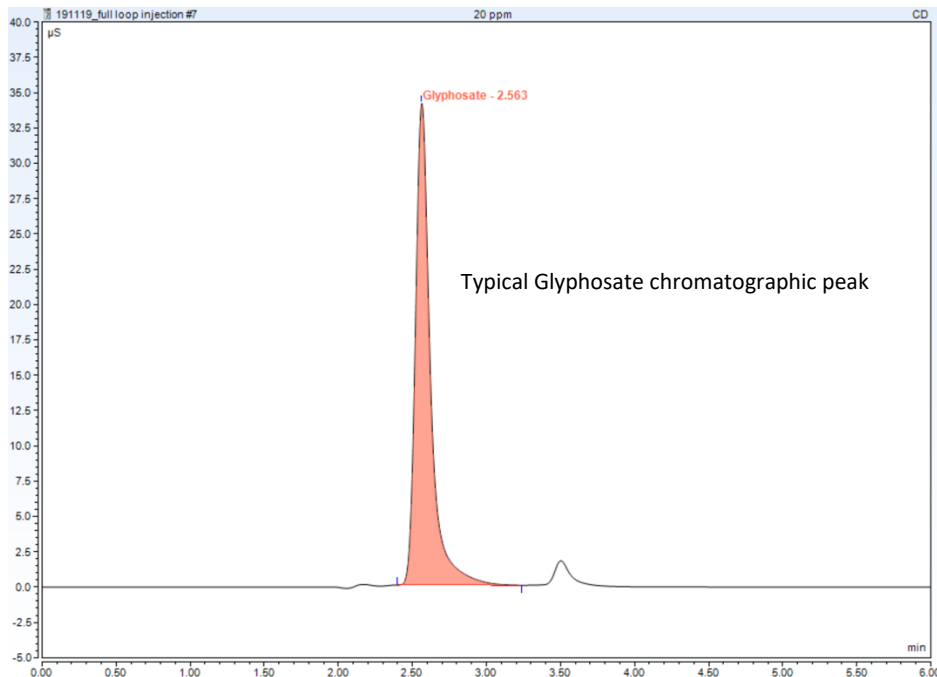
The glyphosate standard was manually prepared at a concentration of 20 ppm in deionised water.

Chromatographic Conditions:

Glyphosate was analysed by ion chromatography (Thermo Scientific) with conductivity detection. Eluent : 50mM KOH
Injection : 25uL, fixed loop push fill
Flow 0.38 mL/min
Isocratic – 6 min runtime

Column: Dionex IonPac AS11
Column Temperature: 30C

RESULTS



See Appendix #1 for Detailed Results

Calculated contribution of epPrep to repeatability is:

$$S\%(ePrep) = \left(\frac{\sqrt{S^2(\text{combined}) - S^2(\text{Chromatography})}}{PEAK AREA(Avg Total)} \right) \times 100$$

S = Standard Deviation

$$0.22\%(ePrep) = \left(\frac{\sqrt{0.031^2(\text{combined}) - 0.0299^2(Avg Chromatography)}}{3.752(Avg Combined Total)} \right) \times 100$$

Calculated reproducibility contribution of ePrep = 0.22% RSD

CONCLUSION

The RSD of the chromatographic instrument (ion chromatographic method) was measured and found to be 0.78% (avg Standard and Sample).

When sample preparation was added by using the ePrep, the total RSD rose only marginally to 0.83% demonstrating the excellent precision attainable with the ePrep.

Therefore, the uncertainty results show the contribution of the total RSD due to the ePrep sample preparation is minimal and mathematically calculated as 0.29%.

ACKNOWLEDGEMENTS

APPENDIX #1

CHROMATOGRAPHIC INSTRUMENT REPRODUCIBILITY

with glyphosate STANDARD repetitive injections, the same solution @ 20ppm.

Inj.	Ret.Time min	Area $\mu\text{S}^*\text{min}$
1	n.a.	n.a.
2	2.57	4.0959
3	2.563	4.021
4	2.567	4.0077
5	2.567	3.991
6	2.567	3.9985
7	2.563	3.9898
8	2.567	3.9973
9	2.563	3.9802
10	2.567	4.0001
11	2.567	4.0092
12	2.567	4.0034
Average	2.566	4.0085
SD	0.002	0.0309
%RSD	0.08%	0.77%

CHROMATOGRAPHIC INSTRUMENT REPRODUCIBILITY

with Weed Killer 360 SAMPLE repetitive injections, same solution approx 20ppm.

Inj.	Ret time (min)	Area
1	2.56	3.6266
2	2.56	3.6501
3	2.56	3.6214
4	2.56	3.6629
5	2.557	3.6324
6	2.56	3.6762
7	2.56	3.6956
8	2.56	3.6846
9	2.56	3.6939
Average	2.560	3.660
SD	0.001	0.029

RSD%	0.039	0.794
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ePrep and CHROMATOGRAPHIC INSTRUMENT (Combined) REPRODUCIBILITY
Weed Killer 360 x 9 samples one injection of each, prepared on ePrep per method workflow

Sample	Ret Time	Area
1	2.56	3.709
2	2.56	3.7286
3	2.56	3.7707
4	2.563	3.7427
5	2.56	3.718
6	2.56	3.7706
7	2.56	3.7898
8	2.56	3.7422
9	2.56	3.7951
Average	2.560	3.752
SD	0.001	0.031
RSD%	0.039	0.828



