

US EPA Method 549.2 — Determination of Diquat (1,1'-ethylene-2,2'-bipyridilium dibromide salt) in Drinking Water Sources and Finished Drinking Water

INTRODUCTION

This is a high performance liquid chromatography (HPLC) method.

METHOD SUMMARY

A measured volume of liquid sample, approximately 250 mL, is extracted using a C₈ solid sorbent cartridge or a C₈ disk which has been specially prepared for the reversed-phase, ion-pair mode. The disk or cartridge is eluted with 4.5 mL of an acidic aqueous solvent. After the ion-pair reagent is added to the eluate, the final volume is adjusted to 10.0 mL. Liquid chromatographic conditions are described which permit the separation and measurement of diquat in the extract by absorbance detection at 308 nm. A photodiode array detector is utilized to provide simultaneous detection and confirmation of the method analytes.

INSTRUMENTATION USED FOR SAMPLE PREPARATION

Dionex AutoTrace[®] instrument

EQUIPMENT SPECIFICATIONS AND OPERATING CONDITIONS

Adjust the pH of this sample to between 8-10.5 by adding 10% HCl(aq) or 10% NaOH(aq).

Loading Procedure

(20 mL/min). Use C8 Cartridge:

1. Condition column with 5.0 mL DH₂O into aqueous waste.
2. Condition column with 5.0 mL CH₃OH into aqueous waste.
3. Condition column with 5.0 mL DH₂O into

aqueous waste.

4. Condition column with 5.0 mL conditioning Solution A into aqueous waste.
5. Condition column with 5.0 mL DH₂O into aqueous waste.
6. Column with 5.0 mL CH₃OH into aqueous waste.
7. Condition column with 5.0 mL DH₂O into aqueous waste.
8. Condition column with 5.0 mL Conditioning Solution B.
9. Condition column with 5.0 mL DH₂O into aqueous waste.
10. Load 255.0 mL of sample onto column (10.0 mL/min).
11. Pause and alert operator. Place vials into the AutoTrace rack for elution.
12. Manually rinse sample container with 5 mL of CH₃OH into aqueous waste.

Eluting Procedure

(1.0 mL/min)

1. Soak and collect 3.0 mL fraction using DES.
2. Collect 1.5 mL fraction into sample tube using DES.
3. Add 200 µL of Ion pair concentrate into each vial.
4. Transfer extract into 10 mL volumetric flask, rinse vials 3X with DES and bring to final 10 mL volume.
5. Store away from light in refrigerator until analysis.

RESULTS

A chart showing target list of compounds and recoveries; could be mean of 5-10 samples i.e., PCB recoveries on soil samples.

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Compound	Mean	Std. Deviation	Number Data Points
Diquat	89.8	11.9	20

CONCLUSION

The automation provided by the AutoTrace instrument allows for fast throughput of samples with less hands on time than manifold. It uses column versus disk technology, which has shown to improve the overall recovery of Diquat.

ACKNOWLEDGEMENTS

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