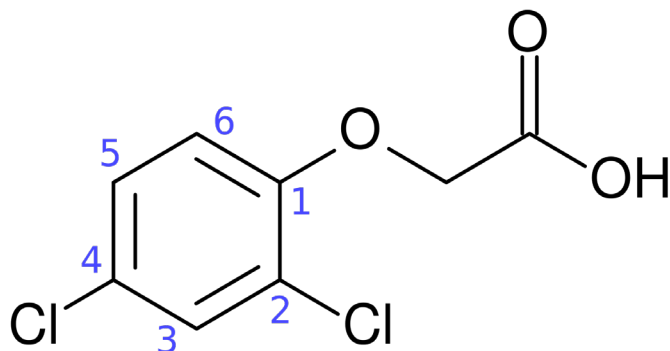


QuEChERS Determination of 2, 4-Dichlorophenoxyacetic acid (2, 4-D) and other Acidic Herbicides by LC-MS/MS



UCT Part Numbers

ECQUEU750CT-MP

4000 mg MgSO₄, 1000 mg NaCl,
500 mg Na Citrate
dibasic sesquihydrate, 1000 mg
Na Citrate tribasic dihydrate

ECMSC1850CT

500 mg C18 and 1500 mg
MgSO₄

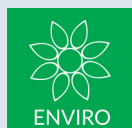
Summary:

The pesticide 2,4-dichlorophenoxyacetic acid (2,4-D; Agent Orange; CASRN 94-75-7; pKa 2.73) along with other acidic pesticides can be extracted and analyzed using this QuEChERS approach.

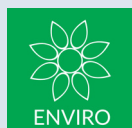
Procedure:

1. Sample Preparation

- Weigh 10 g of homogenized, hydrated sample into a 50 mL centrifuge tube
- Add ISTD
- Add NH₄OH dropwise with shaking until pH 12
- Cap and let sit for 30 min at room temperature
- Neutralize sample by evaporation of NH₄. Dry N₂ may be used
- Add 10 mL of acetonitrile
- Shake
- Add contents of **ECQUEU750CT-MP** pouch to centrifuge tube
- Immediately shake or vortex for 2 min
- Centrifuge for 5 min at >1500 rcf
- Remove 5 - 10 mL aliquot and add to **ECMSC1850CT** tube
- Shake vigorously or vortex for 2 min
- Centrifuge for 5 min at > 1500 rcf
- Pass extract through a 0.45 µm syringe filter into sample vial
- Analyze by LC-MS/MS



Instruments	
MS	AB Sciex 5500 QTrap
HPLC	Shimadzu LC-20AD pump, Sil-20AC autosampler & CTO-20AC column oven
LC Parameters	
Guard Column	Agilent Eclipse Plus C18, 5 µm, 4.6 x 12.5 mm
Column	Agilent Eclipse Plus C18, 1.8 µm, 4.6 x 75 mm
Flow	0.5 ml/min
Injection Vol	10 µl
Oven	40 °C
Equilibration Time	0 min
Rinsing Vol	200 µl
Rinsing Speed	35 µl/sec
Sampling Speed	15 µl/sec
Cooler Temperature	15 °C
Mobile Phase	
Aqueous A	0.1 % formic acid in water
Organic B	0.1 % formic acid in methanol
Time	%B
0	10
3.0	70
8.0	95
12.0	95
12.5	10
14.5	10
MS Instrument Settings	
MRM Window	40 sec
Target Scan	0.25 sec
Cycle	0.5 sec
Resolution	Unit
PosNeg Switch	50 mS
Pause	5 mS
Curtin Gas	50 ml/min
Collision	High
Ion Spray Voltage	4500 volts
Turbospray Temperature	450 °C
Ion Source Gas 1	50 ml/min
Ion Source Gas 2	80 ml/min
Interface Heater	On



Analyte	ESI	RT	Q1	Q2	DP	EP	CE	CXP
2,3,6-TBA	-	5.9	224.6	180.8	-30	-10	-12	-18
2,3,6-TBA	-	5.9	222.6	178.9	-30	-10	-12	-18
2,4,5-T	-	7.7	252.6	194.9	-35	-10	-20	-18
2,4,5-T	-	7.7	254.8	196.9	-60	-10	-18	-18
2,4,5-TB	-	8.7	282.9	196.8	-35	-10	-20	-18
2,4,5-TB	-	8.7	280.8	194.8	-35	-10	-16	-18
2,4-D	-	6.8	218.7	160.9	-20	-10	-20	-18
2,4-D	-	6.8	220.6	162.9	-35	-10	-20	-18
2,4-DB	-	7.7	246.6	160.9	-20	-10	-18	-18
2,4-DB	-	7.7	248.6	162.9	-35	-10	-18	-18
4-CPA	-	6	184.7	126.9	-15	-10	-20	-18
4-CPA	-	6	186.7	128.9	-40	-10	-20	-18
Acifluorfen	-	8.2	359.8	316	-25	-10	-14	-18
Acifluorfen	-	8.2	359.8	194.9	-25	-10	-38	-18
Bromoxynil	-	6.4	273.4	78.8	-25	-10	-62	-18
Bromoxynil	-	6.4	275.4	80.8	-20	-10	-60	-18
Chloramben	-	5.2	203.6	159.9	-35	-10	-10	-18
Chloramben	-	5.2	205.6	161.9	-35	-10	-12	-18
Dalapon	-	4.4	140.6	96.9	-50	-10	-12	-18
Dalapon	-	4.4	142.6	98.9	-40	-10	-12	-18
Dicamba	-	6	218.7	175	-45	-10	-10	-18
Dicamba	-	6	220.4	176.9	-30	-10	-10	-18
Dicamba	-	6	218.7	35	-45	-10	-40	-18
Dichlorprop	-	7.4	232.6	160.9	-20	-10	-18	-18
Dichlorprop	-	7.4	234.5	162.9	-20	-10	-18	-18
Diclofop	-	9	324.9	253	-25	-10	-22	-18
Diclofop	-	9	326.9	255	-25	-10	-22	-18
Fenac	-	7.2	192.7	35	-55	-10	-35	-18
Fenac	-	7.2	194.7	35	-55	-10	-35	-18
Haloxyfop	-	8.4	359.8	288.1	-40	-10	-20	-18
Haloxyfop	-	8.4	361.9	290	-35	-10	-20	-18
MCPA	-	6.8	198.6	140.9	-20	-10	-20	-18
MCPA	-	6.8	200.7	142.9	-30	-10	-20	-18
MCPB	-	7.8	226.8	141	-30	-10	-18	-18
MCPB	-	7.8	228.5	143	-20	-10	-18	-18
Mecoprop	-	7.4	212.7	141	-20	-10	-22	-18
Mecoprop	-	7.4	214.6	143	-30	-10	-24	-18
PCP	-	10	262.4	35	-50	-10	-66	-18
PCP	-	10	262.4	35	-60	-10	-68	-18
Quizalofop	-	8.4	343	271	-20	-10	-22	-18
Silvex	-	8.2	266.6	194.8	-30	-10	-18	-18
Silvex	-	8.2	266.6	158.9	-30	-10	-40	-18
Triclopyr	-	7.2	255.4	197.9	-35	-10	-18	-18
Triclopyr	-	7.2	253.3	195.8	-35	-10	-18	-18
Diflufenzopyr	-	6.5	333	128	-50	-10	-34	-18

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