



## SWITCHING FROM SOLVENT EXTRACTION TO SOLID PHASE EXTRACTION

WORK IN PROGRESS

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## WHY SWITCH ?

- Possible discontinued use of DCM
- Health Hazards of DCM
- Faster?
- Cheaper ?
- Improved Recoveries?

## WANTED:

- **A method that will perform equal to or better than the present solvent extraction method.**

# EQUIPMENT NEEDED

- **Vacuum Manifold**
- **SPE tubes**
  - ◆ **Envi-Carb - Supelco**
  - ◆ **Isolute Env+ - Chromatographic Specialties**



# ENVI-CARB TUBES

- **Carbon Based Packing**

- **250 mg**



## APPLICATION NOTES AVAILABLE BUT:

- **HPLC methods not GC/MS**
- **No Imidazolinones**
- **High spike levels (5 ppb)**

# PROCEDURE

- **No sample prep necessary**
- **Condition tubes**
  - ◆ **DCM / MeOH 4:1**
  - ◆ **MeOH**
  - ◆ **2% Acetic Acid**



# ADSORPTION

- **500 mL sample**
- **Wash with 10 mL water**
- **Air Dry 5 – 10 minutes**

# DESORPTION SOLVENTS INVESTIGATED

1. MeOH / DCM 1:4  
Followed by MeOH / DCM 1:4 + KOH
2. MeOH / DCM 1:4  
Followed by MeOH / DCM 1:4 + Acid
3. Acetone / DCM 1:4  
Followed by Acetone / DCM 1:4 + KOH
4. Acetone / DCM 1:4  
Followed by Acetone / DCM 1:4 + Acid

# RESULTS – ACID HERBICIDES

Pesticide	MeOH/ DCM	M/D- basic	MeOH/ DCM	M/D- acid	Acetone/ DCM	ACE/D- basic	Acetone/ DCM	ACE/D- acid	solvent extraction
	%	%	%	%	%	%	%	%	%
2,4-D	0	5	0	100	0	0	0	21	102
2,4-DP	0	11	0	24	0	0	0	25	84
Picloram	0	0	0	0	0	0	0	0	54
Bromoxynil	0	0	0	11	0	0	0	4	90
Dicamba	0	13	0	32	0	0	0	12	50
Quinclorac	38	0	20	0	23	0	60	0	40

# QUINCLORAC - METHYLATED



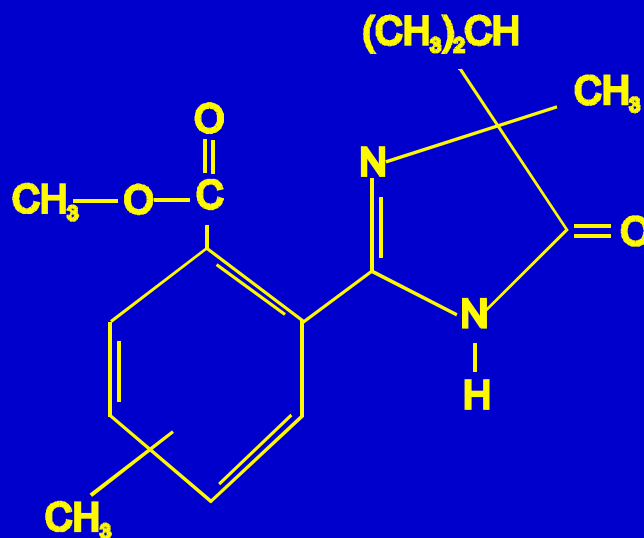
## RESULTS – NEUTRAL HERBICIDES

Pesticide	MeOH/ DCM	M/D- basic	MeOH/ DCM	M/D- acid	Acetone/ DCM	ACE/D- basic	Acetone/ DCM	ACE/D- acid	solvent extraction
	%	%	%	%	%	%	%	%	%
Ethal	20	0	48	0	35	0	35	6	58
Trial	17	0	63	0	32	0	36	9	73
Lindane	24	0	65	0	56	0	63	0	76
Bromacil	0	0	0	0	0	0	10	0	33
Atrazine	74	0	32	0	25	0	75	0	62

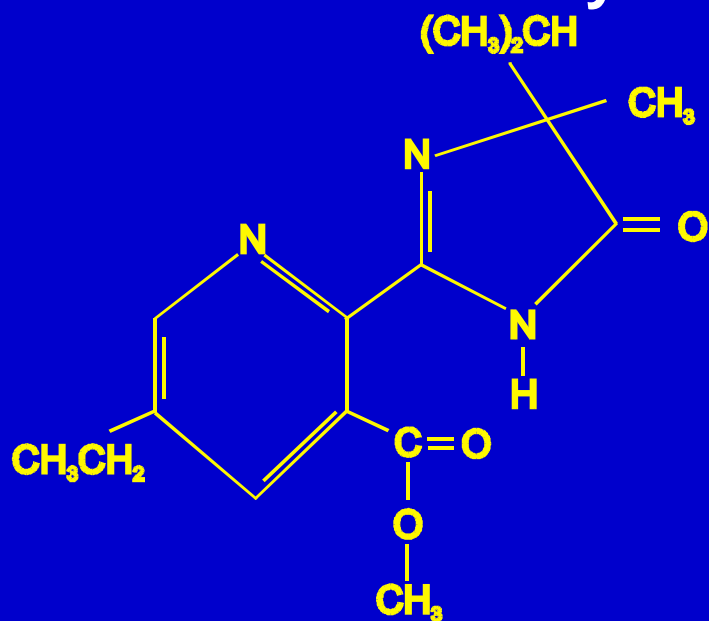
# RESULTS - IMIDAZOLINONES

Pesticide	MeOH/ DCM	M/D- basic	MeOH/ DCM	M/D- acid	Acetone/ DCM	ACE/D- basic	Acetone/ DCM	ACE/D- acid	solvent extraction
	%	%	%	%	%	%	%	%	%
Assert Mono	ND	ND	ND	ND	ND	2	7	4	82
Assert Di	6000	10	800	ND	1200	300	2600	450	
Imazethapyr	0	0	0	67	0	0	0	22	63
Assert	100% Di	100% Di	100% Di		100% Di	100% Di	100% Di	100% Di	80%M
Imazethapyr	-	-	-	100% M	-	-	-	100% M	90%M

# IMAZAMETHABENZ – METHYL Methylated

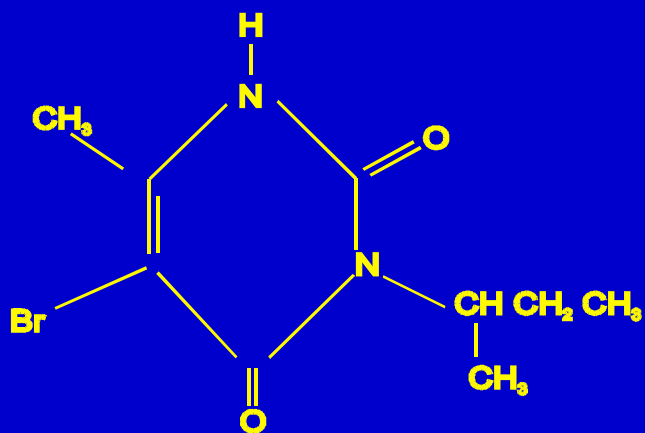


# IMAZETHAPYR - Methylated

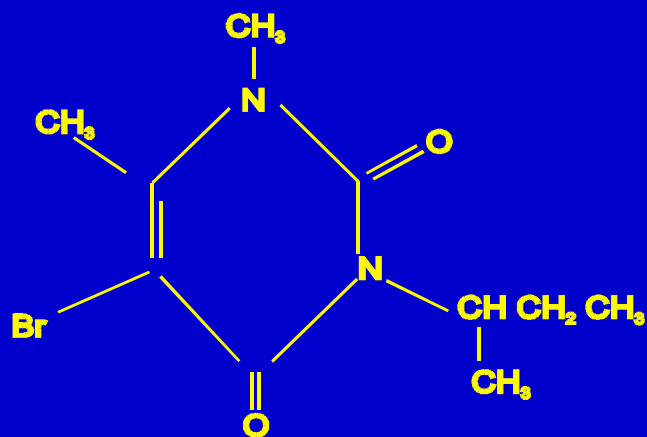




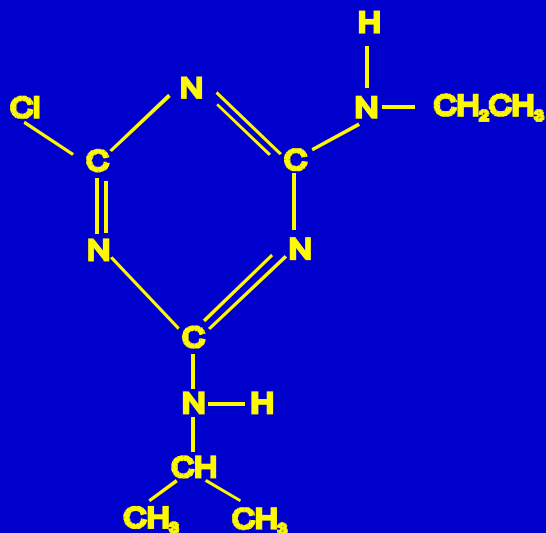
# BROMACIL



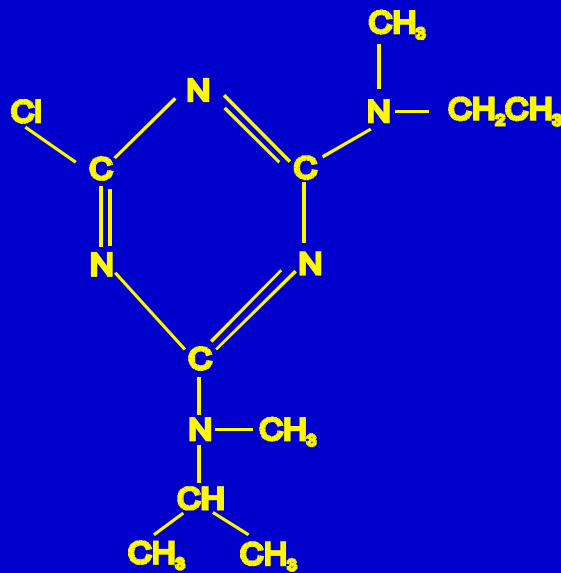
# BROMACIL - METHYLATED



# ATRAZINE



# ATRAZINE – DI-METHYLATED



## Results – General Comments

- **Acetic Acid hard to evaporate**
  - ◆ **Use TFA next time**
- **Some extracts still had water in them**
  - ◆ **Air dry longer**
- **KOH not useful**

## Results – General Comments

- Di-methylation may be a problem
- Larger desorption volumes may improve recoveries
- Isolute Env+ had better acid/neutral recoveries but no imidazolinones
- More development work required but looks promising