Polymorphs in PDF-4 ORGANICS 2010

International Centre for Diffraction Data®

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ICDD Website - www.icdd.com

Common Polymorphs in PDF

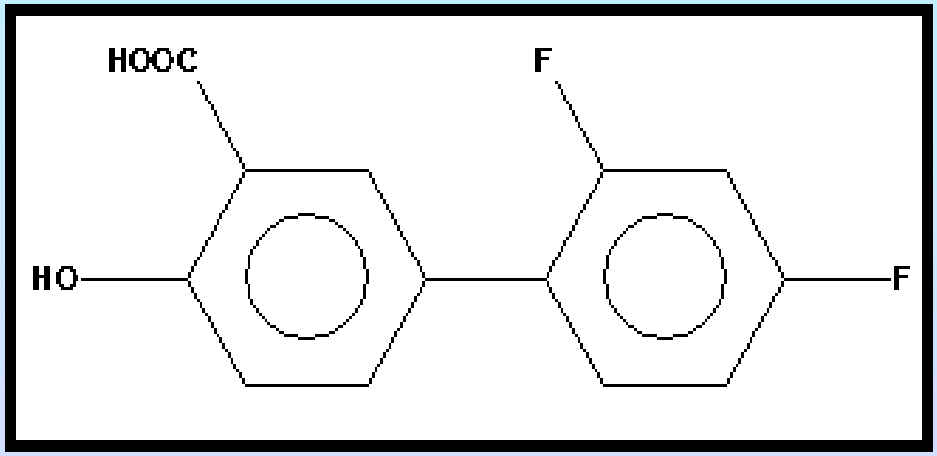
Name		Sp Group	Name		Sp Group
Cellulose I	Excip-	A P1	Torasemide	Blood-	M P21/c
Cellulose II	ients	M P21	Torasemide	Pressure	M P2/n
Cimetidine	Stomach	M P21/n	meso-Tartaric acid	Food	A P-1
Cimetidine		M P21/c	meso Tartaric acid	Additive	O Pbca
Retinoic acid	VitaminA	A P-1	Tartaric acid		M P21
Retinoic acid		M P21/n	DL-Malic acid		M Cc
Tegafur	Colon-	A P-1	DL-Malic acid		M P21/c
Tegafur	Cancer	M P21/n	Di-butyl-methylphenol		O P212121
DL-Valine	Amino-	M P21/a	Di-butyl-methylphenol		M I2/a
DL-Valine	Acid	A P-1	Di-butyl-methylphenol		M C2/c
Tyrosine		O Pnam	Cortisone acetate	Anti-	O P212121
Tyrosine		O Pnc2	Cortisone acetate	Inflam.	M P21
Sulfanilamide	Anti-	O Pbca	Piroxicam		M P21/c
Sulfanilamide	Bactrial	M P21/c	Piroxicam		O Pca21
Sulfapyridine		M C2/c	Estrone	Steroid	O P212121
Sulfapyridine		M P21/c	Estrone		M P21
Sulfamethazine		M P21/a	p-Cresol	Cleaning	M P21/c
Sulfamethazine		O Pca21	p-Cresol	Agent	M C2/c

Polymorphs

- Kinetic/Thermodynamic
- $G_0 – G_1$, $G_0 – G_2$; G_1 - G_2
- Kinetic Process/Rate of Nucleoation
  - Degree of Supersaturation
  - Surface free energy
  - Temperature
  - Solubility
- Hydrogen Bonding
  - Atoms
  - Energy
- Detection/Characterization

  IR/DSC/PXRD

Diflunisal – Properties



C13 H8 F2 O3

History/Source

Patent : **Dolobid- Merck & Co.**

Present: Patent expired

Sample from ICN Biomedicals Inc., Aurora, Ohio, USA.

2',4'-Difluoro-4-hydroxy-3-biphenylcarboxylic acid

Pharmaceutical Application

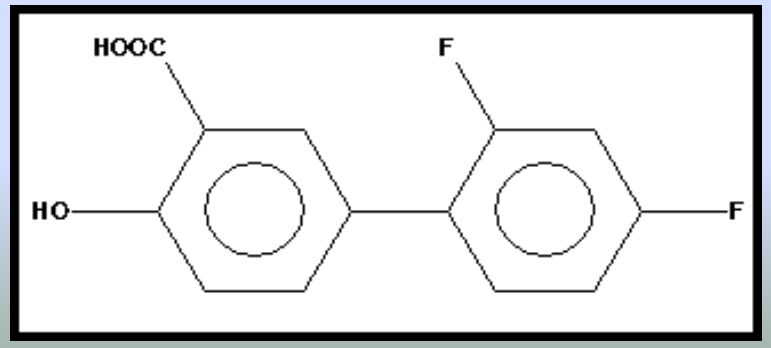
non-steroidal anti-inflammatory drug.

Colorless/White Prostaglandin production inhibitor.

Arthritis, post – wisdom tooth extraction.

Diflunisal
Preparations
Experimental Patterns

ID	Density	Volume	Sys
FormA	1.643	1011.51	a
FormB	1.429	2326.18	t
FormC	1.387	2395.76	a
FormD	1.084	766.44	a
Triclinic I	1.526	544.44	a
Ortho-rhombic III	1.545	2151.51	o
Monoclinc V	1.319	2519.18	m



Form A (000581379):
Solvates crystallized from benzene, 'C H Cl3', 'H2 O‘, and hexane - were heated at 423 K; Vapor diffusion of benzene into a saturated solution in diethyl ether or of ethanol into an acetone solution.

Form B (000581380) :
Water was added to a saturated solution of diflunisal in ethanol and the crystals were dried under reduced pressure at 298 K.

Form C (000581381) : slow solvent evaporation from ethanol at 298 K.

Form D (000581382) :
crystallization from a saturated solution in tetrahydrofuran by vapor diffusion of ''C H Cl3''.

Diflunisal - Simulated Patterns

ID	a	b	c	Alpha	Beta	Gamma
FormA	7.066	21.836	7.062	98.33	109.24	93.27
FormB	17.9	17.9	7.26	90	90	90
FormC	14.674	20.126	8.67	102.21	106.8	86.07
FormD	9.102	11.252	8.814	96.29	107.14	113.17
Triclinic I	6.77	21.586	3.8	94.07	98.02	96.31
Ortho-rhombic III	14.129	39.7	3.836	90	90	90
Monoclinc V	33.565	3.743	20.737	90	104.77	90

triclinic I (020793519)

Color: Colorless
Habit: needles
Crystallized from: toluene.
Structures: An ortho fluorine atom is equally disordered over two sites.

monoclinic V (020815120)
Color: colorless. Habit: prism.
Crystallized from: acetone/water.
Structures: The molecule is partially disordered by rotation about the C1-C7 bond; F2 and F3 have 0.5 occupancies.

orthorhombic III (020793520)

Color: white.
Habit: acicular.

Diflunisal

Polymorphs - Indexed Lines

Form A

4.1743	19	1	0	0
13.3841	100m	2	0	-1
13.3841	m	0	1	0
13.4865	100m	1	-1	0
13.4865	m	1	0	-1
14.4849	45m	3	0	-1
14.4849	m	1	1	0
14.8766	15m	2	-1	0
14.8766	m	0	0	1
15.4241	6	1	1	-1
15.9267	4	-2	-1	1
16.6502	21	2	1	0
17.0372	14	3	1	-1
17.1703	41m	3	-1	0
17.1703	m	1	0	1
26.1103	8	2	2	-1
26.6675	16m	1	-2	0
26.6675	m	1	1	-2
28.2171	10m	2	-2	1
28.2171	m	0	1	-2
29.2570	4	2	2	0

Form B

4.9327	43	1	0	0
12.1474	32	0	0	1
13.1837	26	0	1	1
14.0683	39	1	1	1
14.7520	100	3	0	0
16.4324	60	1	2	1
18.5469	22	2	2	1
19.9358	14	3	1	1
21.0850	16	3	3	0
24.9208	18	0	1	2
26.5058	11	0	2	2

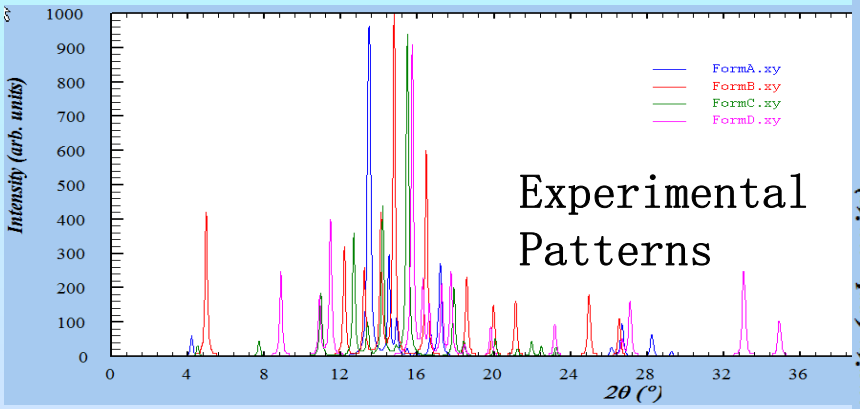
Form C

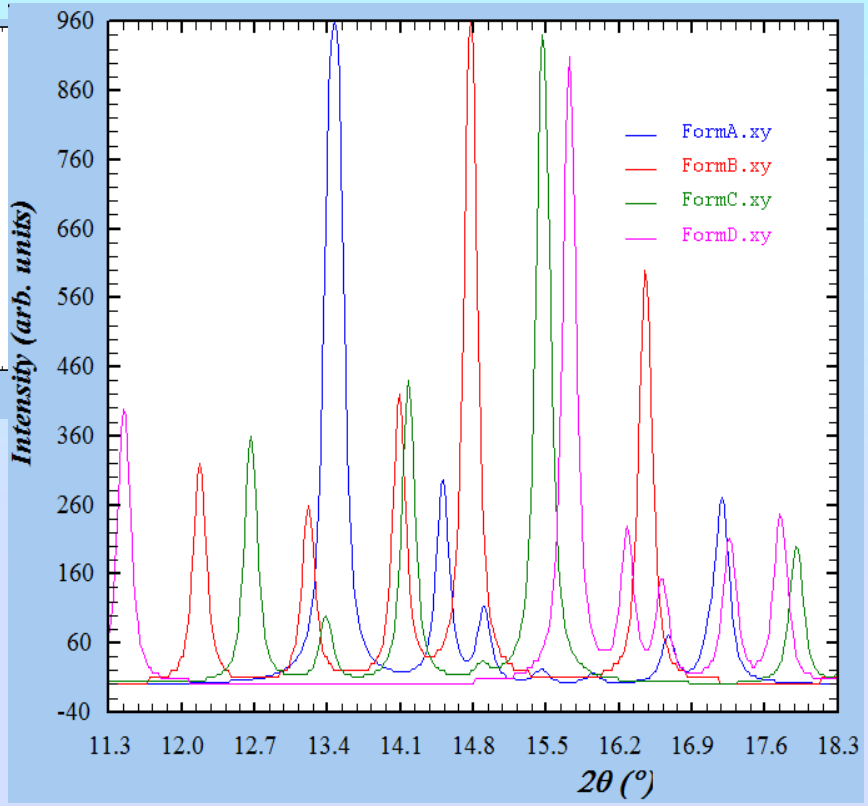
4.4840	6	1	0	0
7.7013	9	1	0	-1
10.9273	18m	0	1	0
10.9273	m	0	0	1
12.6534	36m	1	1	0
12.6534	36	2	-1	0
13.3637	19	3	0	-2
14.1588	42m	1	1	-1
14.1588	m	2	1	-1
14.8515	4	1	0	1
15.4512	100m	0	1	1
15.4512	m	0	1	-1
17.8682	38	1	-1	1
18.3917	8	1	0	-2
20.0268	9	2	1	-2
21.1868	4	-5	-1	1
21.9281	4m	0	2	0
21.9281	m	0	0	2
22.4328	5	-5	2	1
23.2046	5	3	0	-3

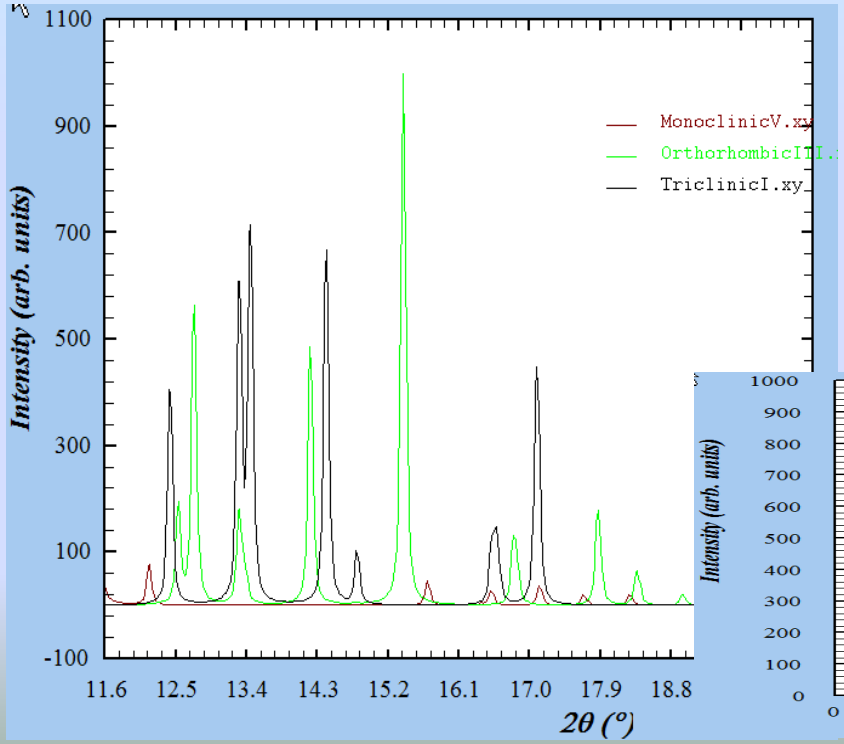
Form D

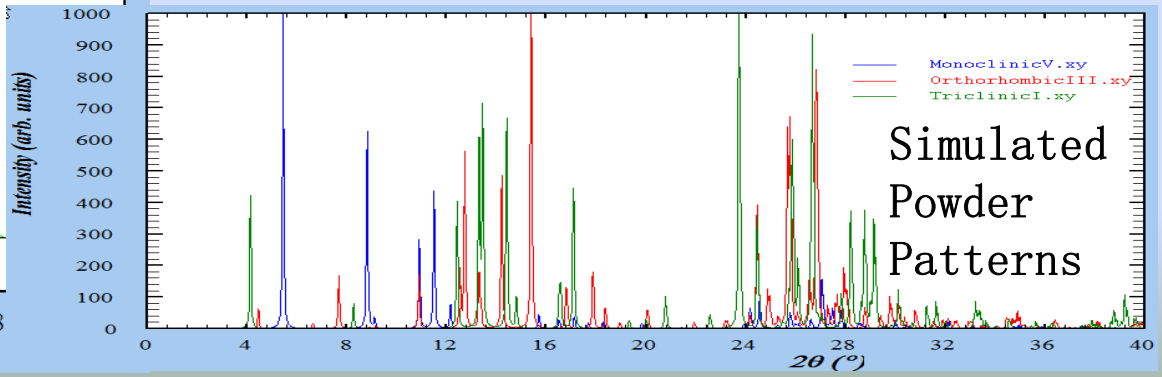
8.8355	28	1	0	0
10.8465	10m	0	1	0
10.8465	m	1	0	-1
11.4230	49	1	-1	0
15.6993	100	0	0	1
16.2503	24	1	1	0
16.5874	15	-3	1	1
17.2376	23	2	-1	0
17.7241	30	2	0	0
18.4302	4	0	1	-1
19.8010	10	0	1	1
23.1434	11	1	0	1
26.6675	6	3	0	0
27.0804	19	3	1	-2
33.0264	15m	0	3	0
33.0264	m	-3	3	2
34.8816	13	1	2	1

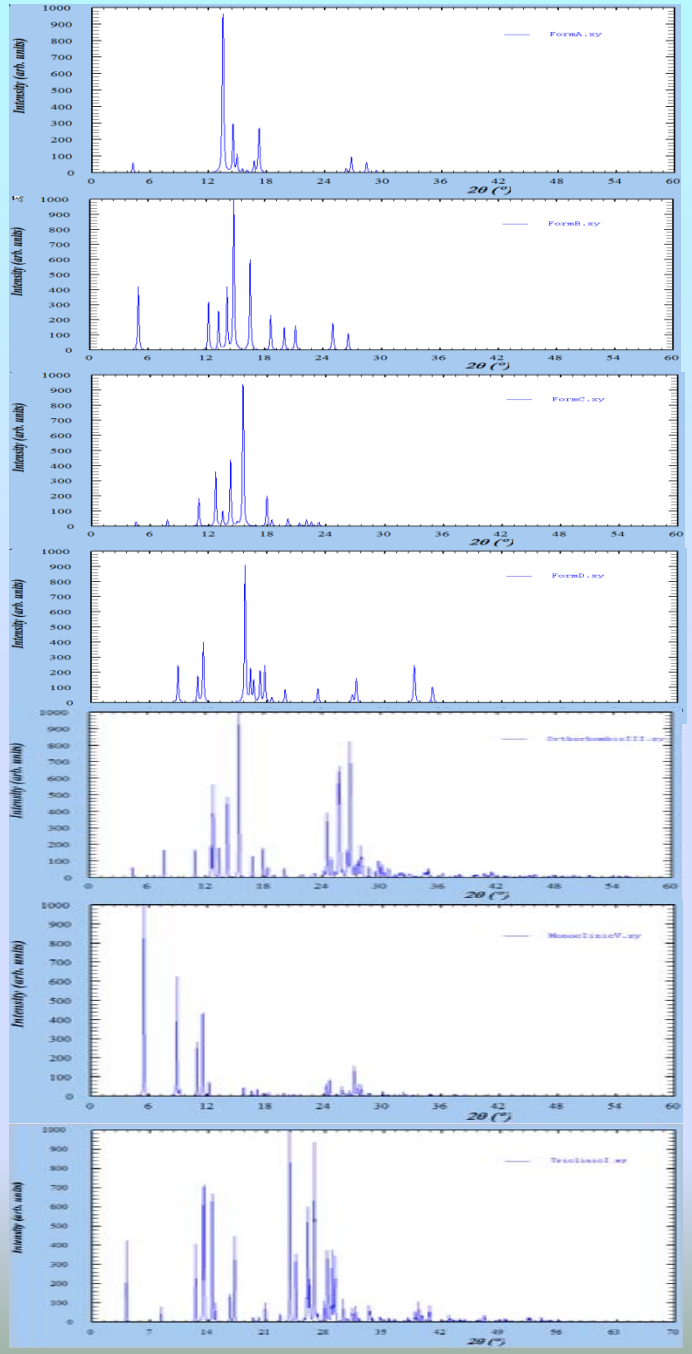
Diflunisal – Polymorphs (PDF Patterns)











Diflunisal - Polymorphs

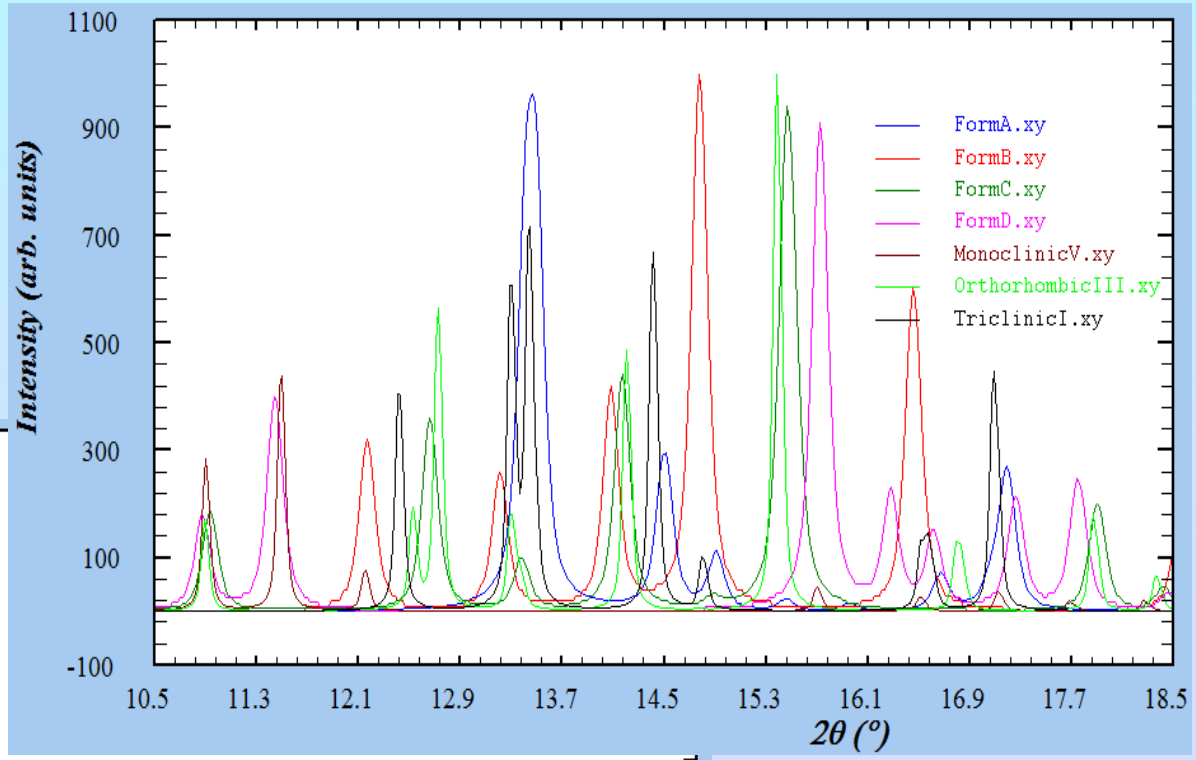
C13 H8 F2 O3

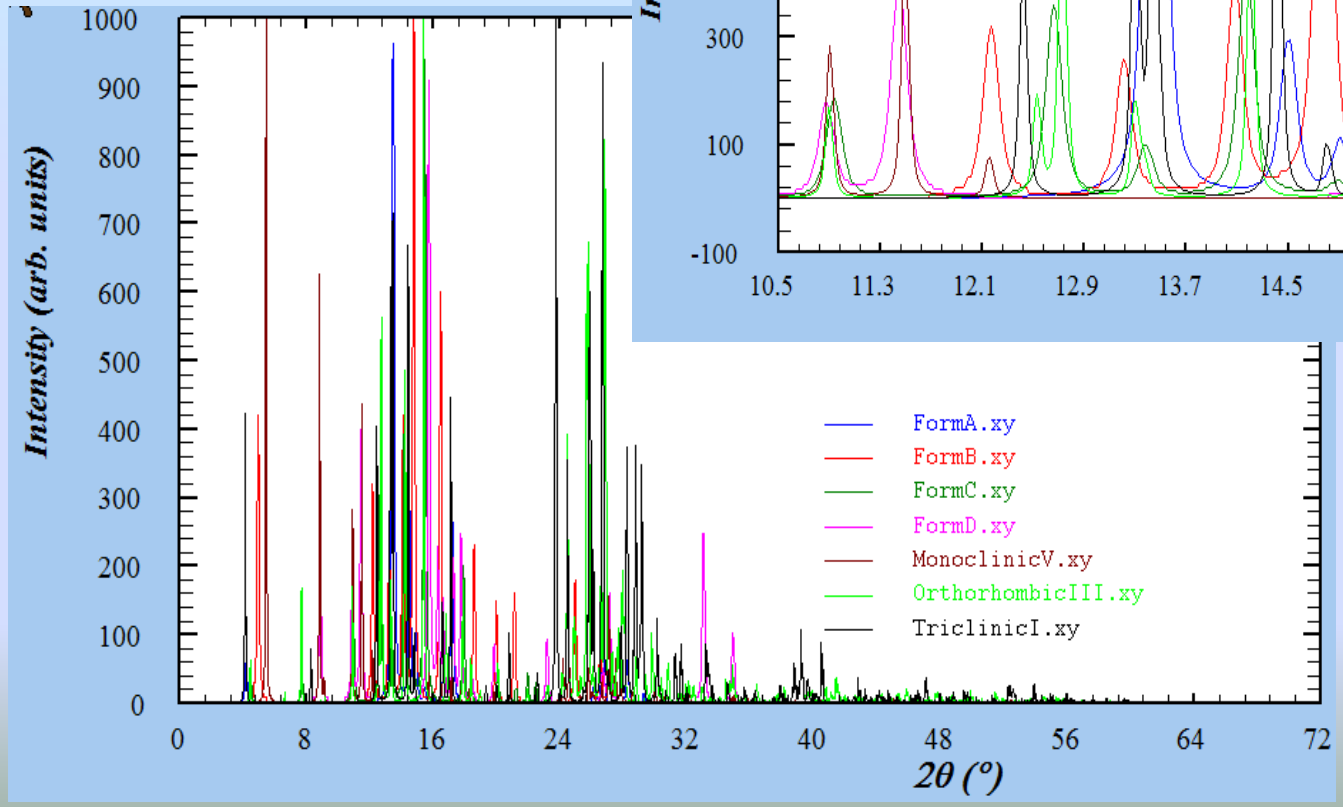
1. Form A. (000581379)
2. Form B. (000581380)
3. Form C. (000581381)
4. Form D. (000581382)
5. triclinic I (020793519)
6. orthorhombic III (020793520)
7. monoclinic V (020815120)

ID	a	b	c	Alpha	Beta	Gamma
1	7.066	21.836	7.062	98.33	109.24	93.27
2	17.9	17.9	7.26	90	90	90
3	14.674	20.126	8.67	102.21	106.8	86.07
4	9.102	11.252	8.814	96.29	107.14	113.17
5	6.77	21.586	3.8	94.07	98.02	96.31
6	14.129	39.7	3.836	90	90	90
7	33.565	3.743	20.737	90	104.77	90

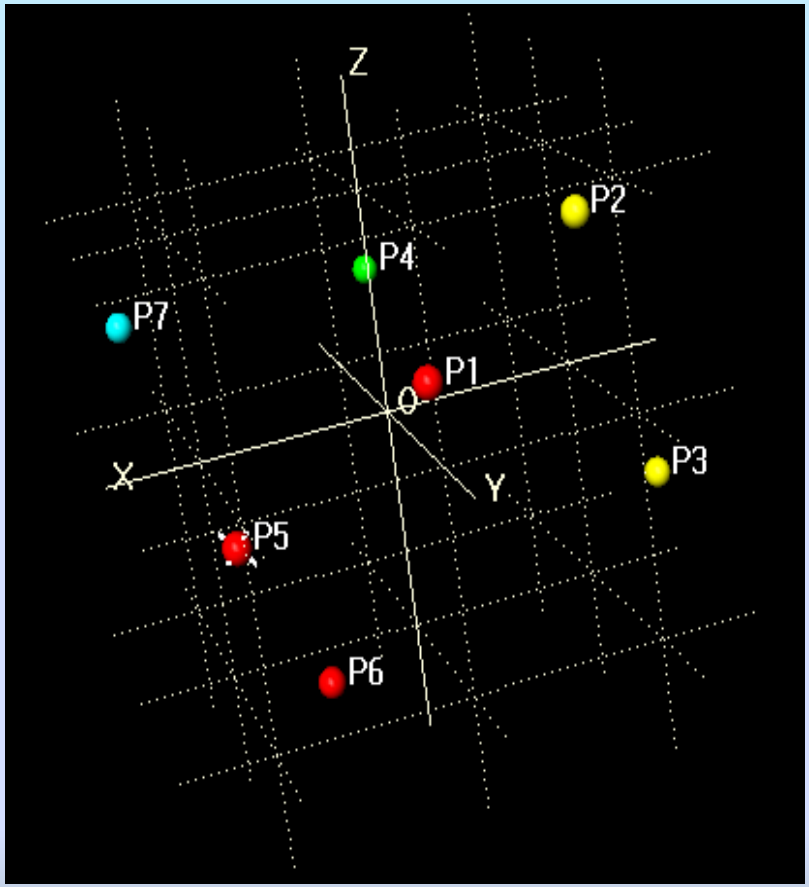
Diflunisal Polymorphs C13 H8 F2 O3

Seven Overlapping Powder Patterns

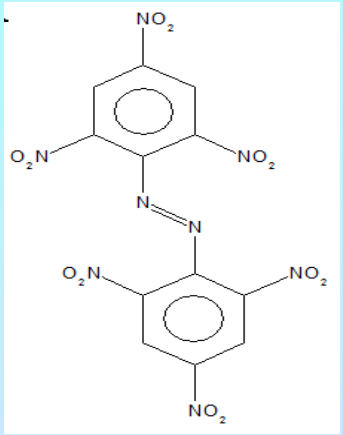




Diflunisal - Cluster Analysis

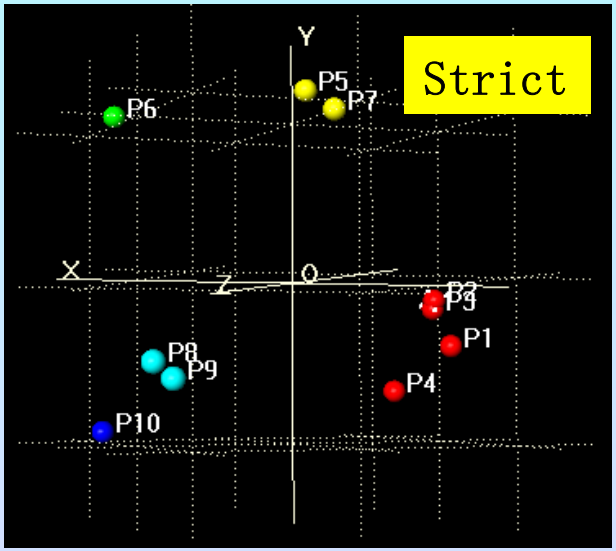


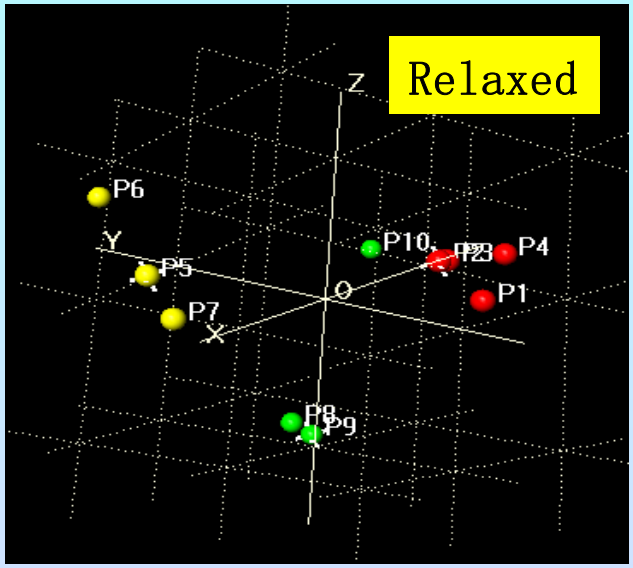
#	ID	Density	Volume	Sys
1	FormA	1.643	1011.51	a
2	FormB	1.429	2326.18	t
3	FormC	1.387	2395.76	a
4	FormD	1.084	766.44	a
5	Triclinic I	1.526	544.44	a
6	Orthorhombic III	1.545	2151.51	o
7	Monoclinc V	1.319	2519.18	m



C12H4N8O12
2,2`,4,4`,6,6`-Hexanitroazo-benzene

Polymorphs





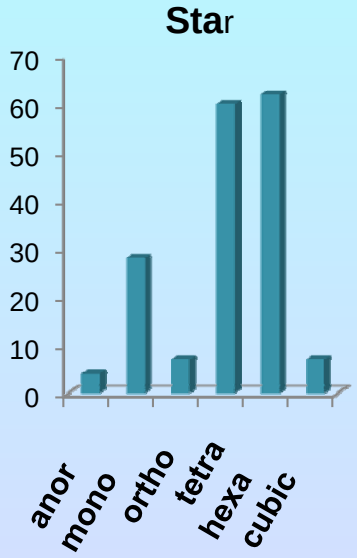
Cluster Analysis

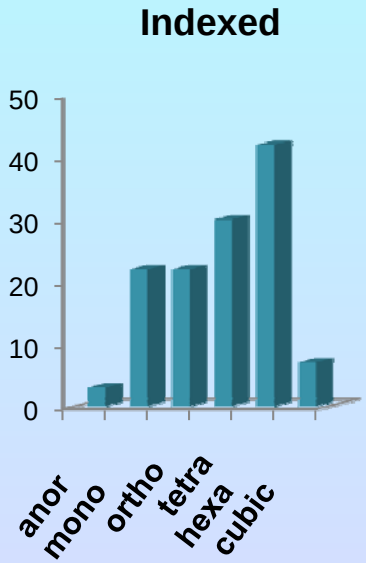
	ID	Common Name	Spgr	Sp #	Sys	a(red)	b(red)	c(red)	alpha	beta	gamma	volume	Density
1	000401743	HNAB-III	C222	21	O	5.498	15.359	22.065	110.37	90.00	90.00	1746.72	1.722
2	000441616	HNAB-III	C2221	20	O	5.502	15.361	22.061	110.37	90.00	90.00	1747.84	1.718
3	000441617	HNAB-III	C2221	20	O	5.502	15.350	22.057	110.36	90.00	90.00	1746.42	1.718
4	020871485	diazene	P21	4	M	5.524	15.401	22.118	110.37	90.00	90.00	1764.13	
5	000421767	HNAB-II	P21/a	14	M	7.571	10.635	21.859	90.00	90.00	102.58	1717.78	
6	000401742	HNAB-II	P21/a	14	M	7.579	10.680	21.903	90.00	90.00	102.38	1731.69	1.735
7	000441615	HNAB-II	P21/a	14	M	7.585	10.632	21.632	90.00	90.00	102.56	1702.74	1.744
8	000441613	HNAB-I	P21/c	14	M	8.263	10.055	10.149	97.29	90.00	90.00	836.41	1.795
9	000441614	HNAB-I	P21/c	14	M	8.281	10.042	10.090	97.25	90.00	90.00	832.35	1.795
10	000401741	HNAB-I	P21/c	14	M	8.285	10.013	10.220	97.34	90.00	90.00	840.88	1.786

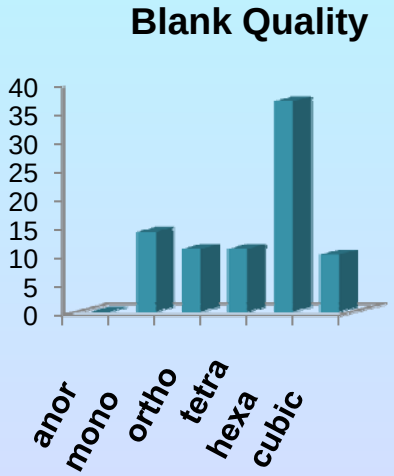
SiO_2

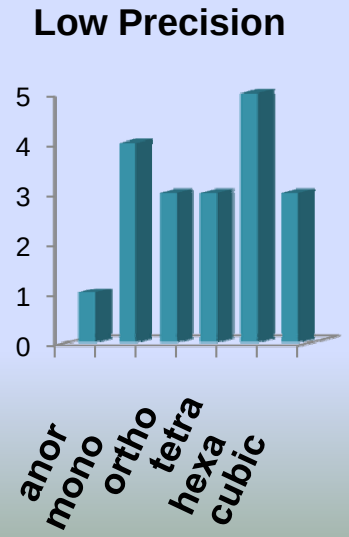
Quality

S-Star
I-Indexed
O-Lo Quality
B-not S/I/O
C-Calculated
R-Rietveld
P-Prototype
H-Hypothetical





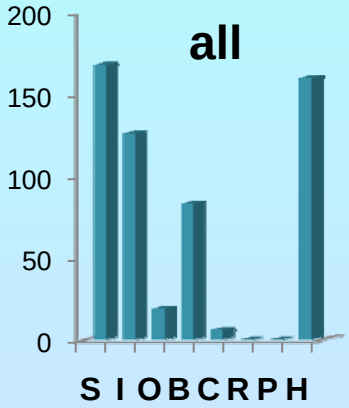


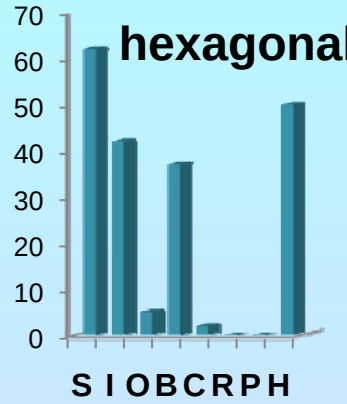


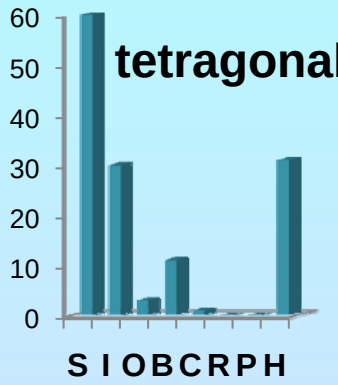
Classes	S	I	O	B	C	R	P	H
anorthic	4	3	1	0	0	0	0	12
monoclinic	28	22	4	14	1	0	0	19
orthorhombic	7	22	3	11	2	0	0	24
tetragonal	60	30	3	11	1	0	0	31
hexagonal	62	42	5	37	2	0	0	50
cubic	7	7	3	10	0	0	0	24
all	168	126	19	83	6	0	0	160

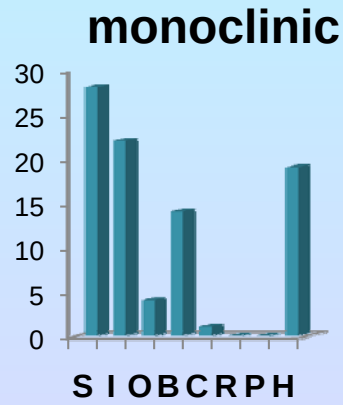
Quality

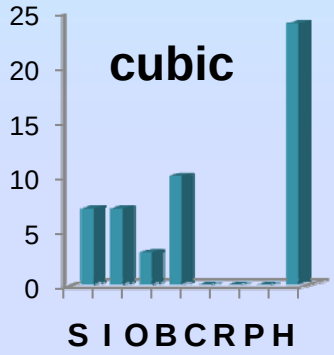
SiO_2



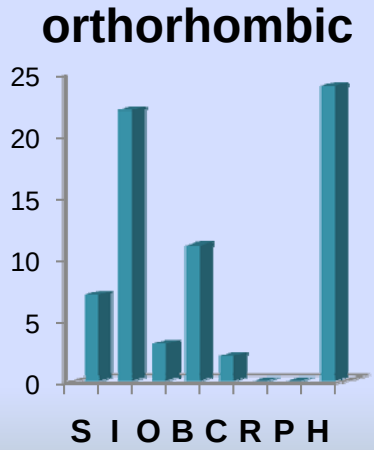


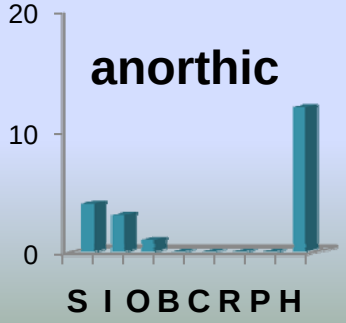






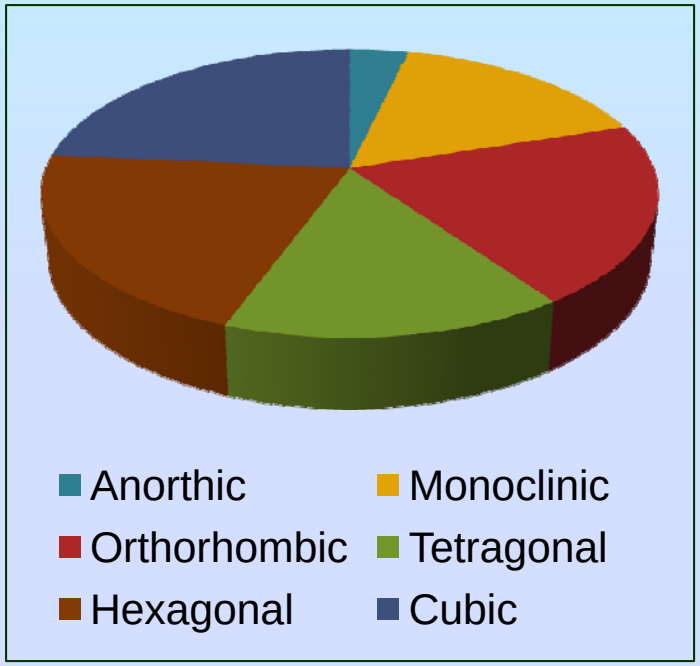
Class	S	I	O	B	C	R	P	H
anorthic	4	3	1	0	0	0	0	12
monoclinic	28	22	4	14	1	0	0	19
orthorhombic	7	22	3	11	2	0	0	24
tetragonal	60	30	3	11	1	0	0	31
hexagonal	62	42	5	37	2	0	0	50
cubic	7	7	3	10	0	0	0	24
all	168	126	19	83	6	0	0	160





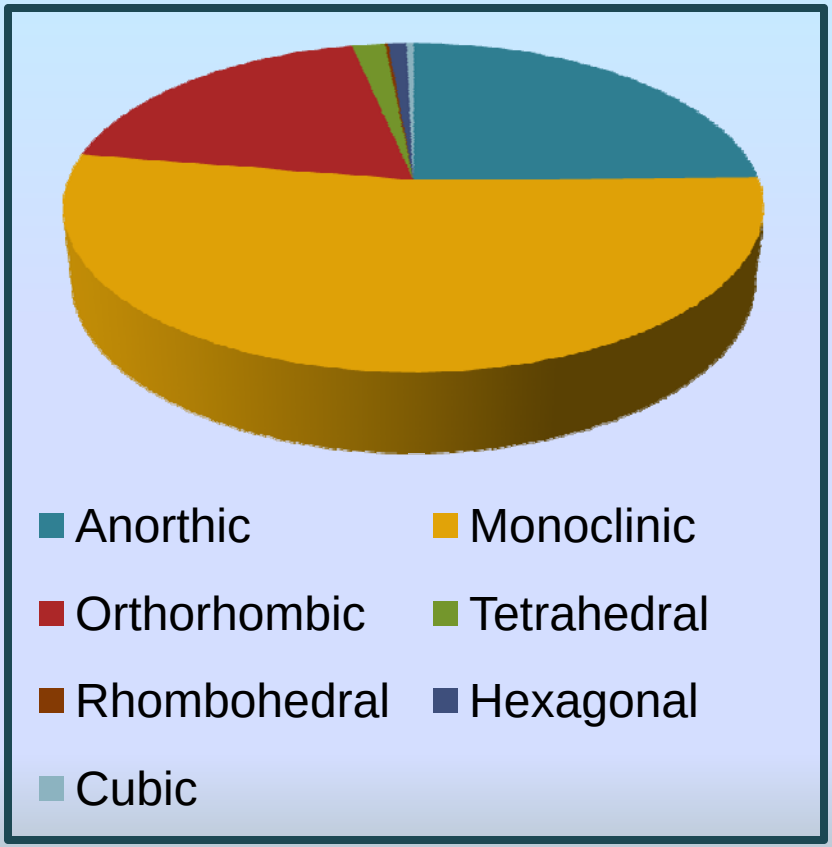
Crystal System Distribution Inorganic

System	Count	%
Anorthic	9461	3.63
Monoclinic	42609	16.37
Orthorhombic	52689	20.24
Tetragonal	39917	15.33
Hexagonal	54496	20.93
Cubic	61172	23.50



Crystal System Distribution
Organic

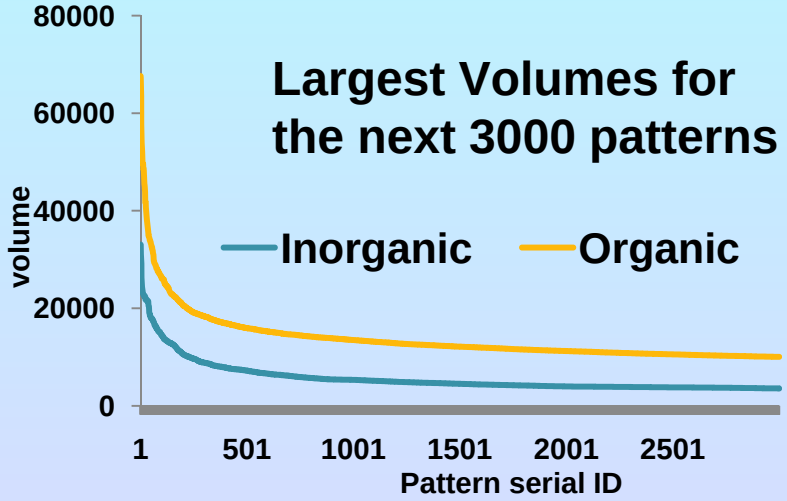
Classes	Counts	Percent
Anorthic	95754	24.76222
Monoclinic	204236	52.81592
Orthorhombic	73830	19.09262
Tetrahedral	6913	1.787718
Rhombohedral	722	0.186711
Hexagonal	3787	0.979327
Cubic	1452	0.375491

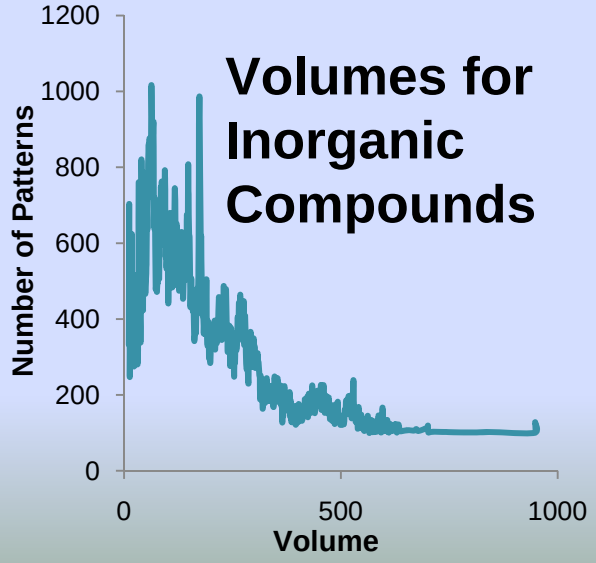


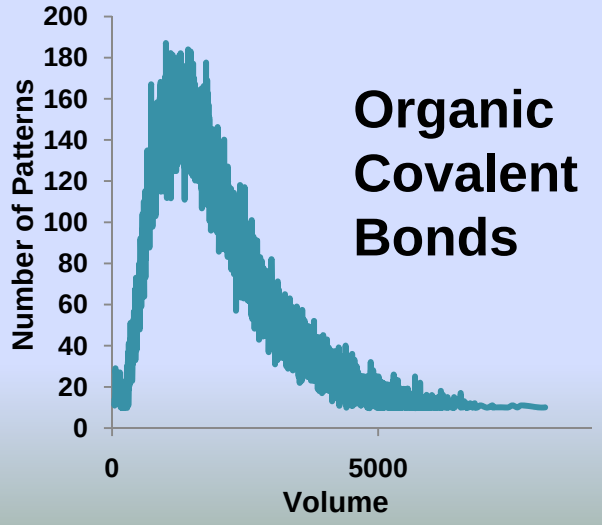
Inorganic/Organic – Reduced Cell Volume

First 20 Patterns with Largest Volumes

Inorganic	Organic
513622	30724756
266607	20674974
265721	17760888
96062	15672629
58184	5687412
55489	4486489
51558	3741766
50798	1442897
50726	1274448
49877	1033899
47452	991027
42524	960248
42136	592704
41893	496793
37134	333814
36616	318612
36616	194690
34571	125023
34571	68770







Thank You for Your Attention