

BATCH DRYING OF ACETIC ACID BY AZEOTROPIC DISTILLATION (BATCH 3)

Water and acetic acid are difficult to separate. Production of dry acetic as a bottoms product while taking water overhead with a low acetic acid content is not easy in conventional distillation; many trays are required, and the column must be operated at a high reflux.

In this example, acetic acid containing 15 wt % water is dehydrated using ethyl acetate as an entrainer. Ethyl acetate and water are only partially miscible, thus they form a heterogeneous azeotrope. Ethyl acetate and water are easy to separate. The procedure is as follows: Bring the column to total reflux. Decant the water layer, while refluxing the ethyl acetate layer. The refluxing ethyl acetate effectively washes down to any acetic acid, so that the overhead product contains little acetic acid. As water is removed, the rate at which water is decanted drops off. At the point of diminishing returns, the decanter is taken out of service and ethyl acetate (containing some water and acetic acid) is taken as distillate to a different accumulator. When the ethyl acetate content of material in the pot has dropped to a sufficiently low concentration, the distillation is discontinued, and the pot is dumped out. Wet acetic acid for the next run is charged to the pot, and the ethyl acetate (containing some acetic acid and a small amount of water) is recycled.

In batch distillation, it is often difficult to decide on the details of a run at the start. Accordingly, the distillation is carried out in four steps. After each step, the user can stop to decide what to do in the next step; indeed, even the number of steps does not have to be fixed in advance. This strategy is used rather than planning details of the procedure at the start.

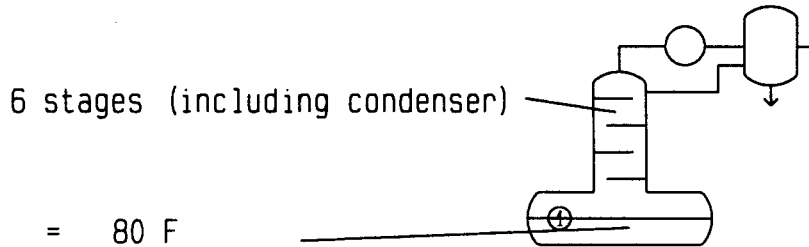
Step 1 - Decant water until the water rate from the decanter drops to 35% of the original rate. This is best done by plotting the water rate (and contained ethyl acetate) as a function of time, then pressing ESC.

Step 2 - The decanter is not used; instead the overhead is run in a conventional manner to remove part of the ethyl acetate and a large part of the remaining water. A 1/1 reflux ratio is used. The mass fraction water and acetic acid in the distillate are plotted. When the acetic acid content of the distillate reaches 5.0 wt%, this step is discontinued. (An alternate strategy would be to use the Stop Option to discontinue the operation when the acetic acid content of the distillate reaches 5.00 wt%).

Step 3 - The acetic acid content of the overhead product is set at 5.30 wt% and the required reflux ratio plotted. When the reflux ratio reaches 5/1, this step is continued. Note that the purity is 5.0 wt% at the start of this step; thus, stability is no problem. If step 2 were eliminated (go directly from Step 1 to Step 3), stability and convergence problems would be likely. The required reflux ratio is followed on the screen and this step is discontinued when it reaches 5/1.

Step 4 - Fix reflux ratio at 5/1 and plot the ethyl acetate content of the pot residue. When it reaches 0.5 wt%, stop the distillation.

Azeotropic Distillation
Separation of Acetic Acid and Water
using Ethyl Acetate as an entrainer



T = 80 F
P = 14.7 psia
Water = 208 lb-moles
Acetic Acid = 417 lb-moles

Batch Charge:

Temperature= 80 F
Pressure= 14.7 psia
Ethyl Acetate = 284 lbmoles
Water = 208 lbmoles
Acetic Acid = 417 lbmoles

Column:

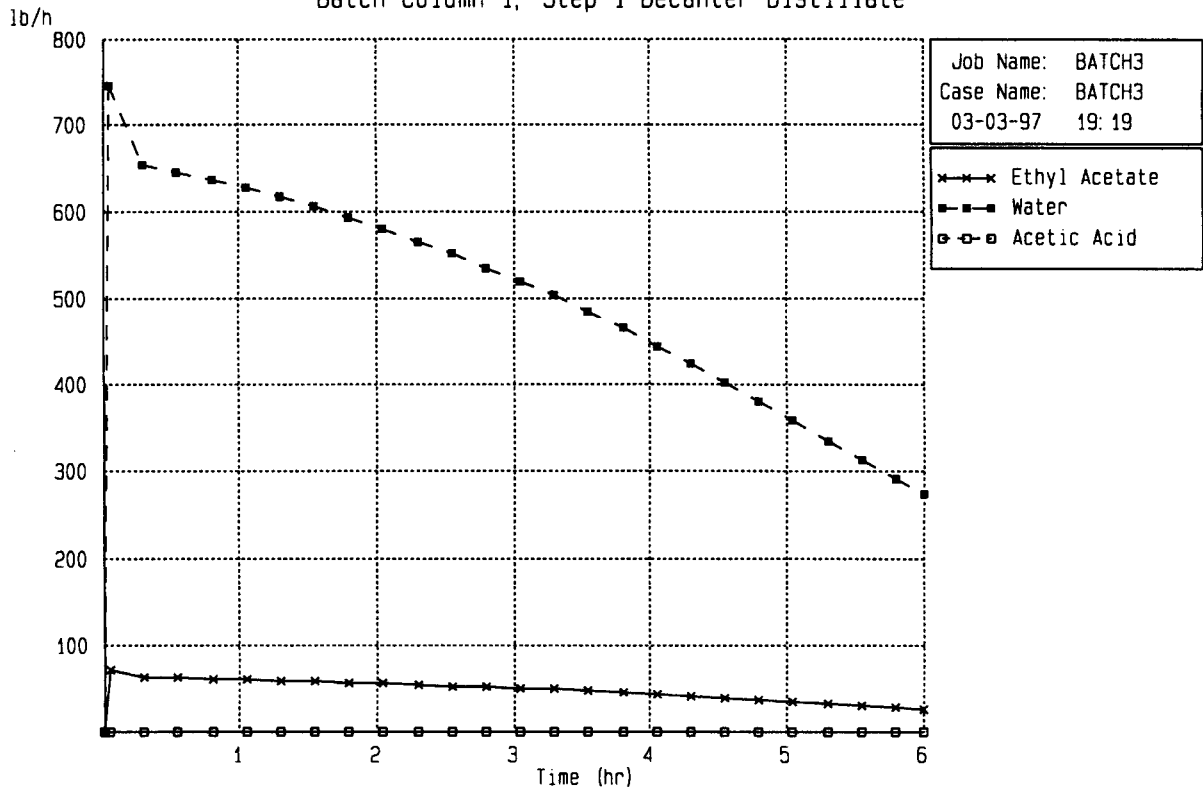
6 stages
4 Operating Steps
Total condenser with decant
Cond P = 14.7
Volume holdup units
Condenser holdUp = 1.35
Stage holdup = 0.94

Operating Steps:

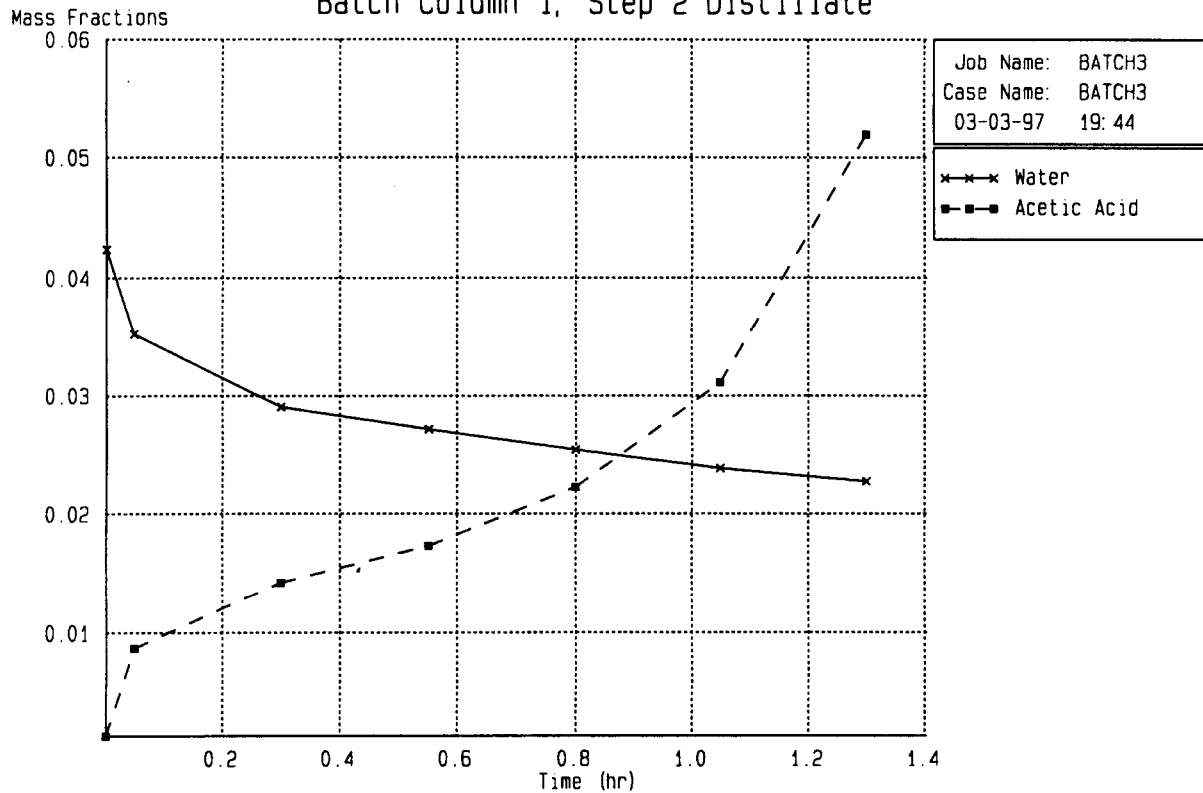
	1	2	3	4
Overhead Tank	1	2	3	4
Decant Tank	11	12	13	
Mode #1	Cond. Q	RR	Dist. Moss.Fr.	RR
Value #1	-5	1	.05	5
Comp #1			3	
Mode #2	Dist.Mol.Flow	Reb. Q	Reb. Q	Reb. Q
Value #2	.00303	5	5	5
Comp #2				
Sub. Cond. T.	100	100	100	100
Stop Mode	Time	Time	Time	Time
Stop Value	6	1.3	1	2.3
Decant	on	off	off	off

Chemcad Training Manual

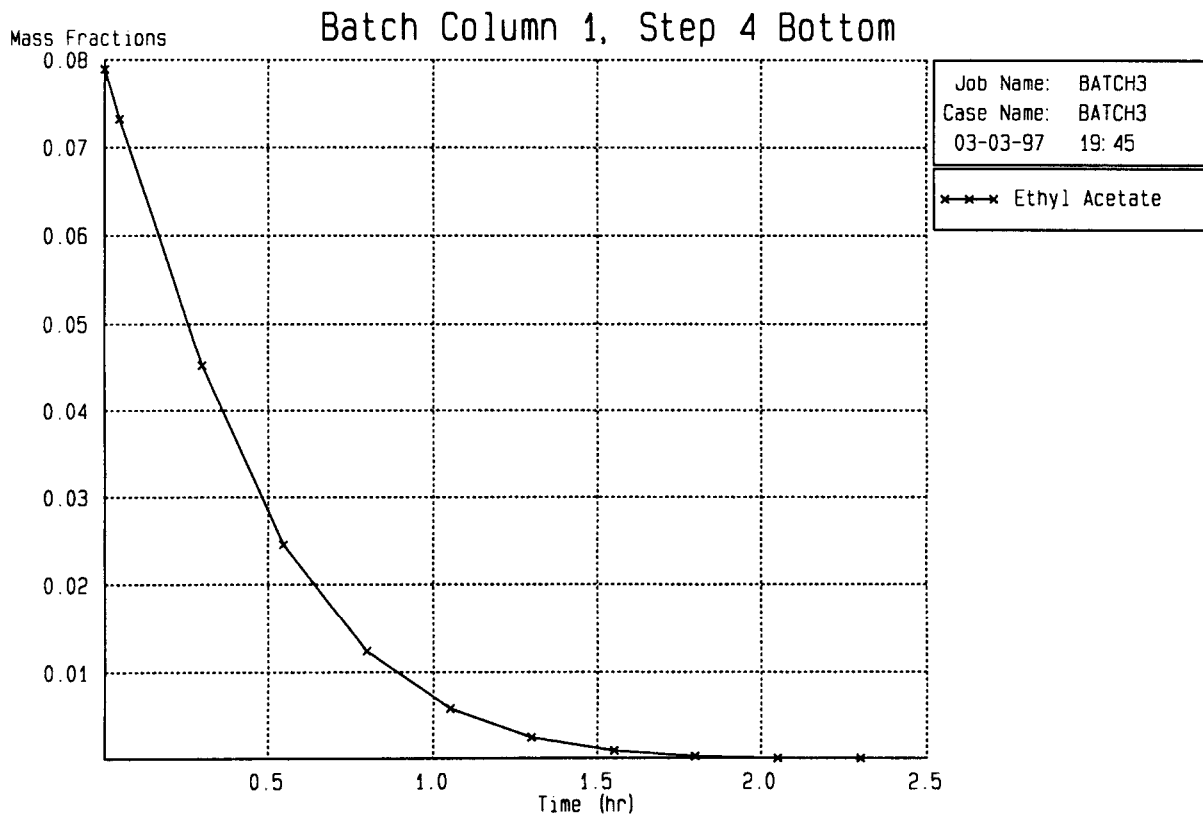
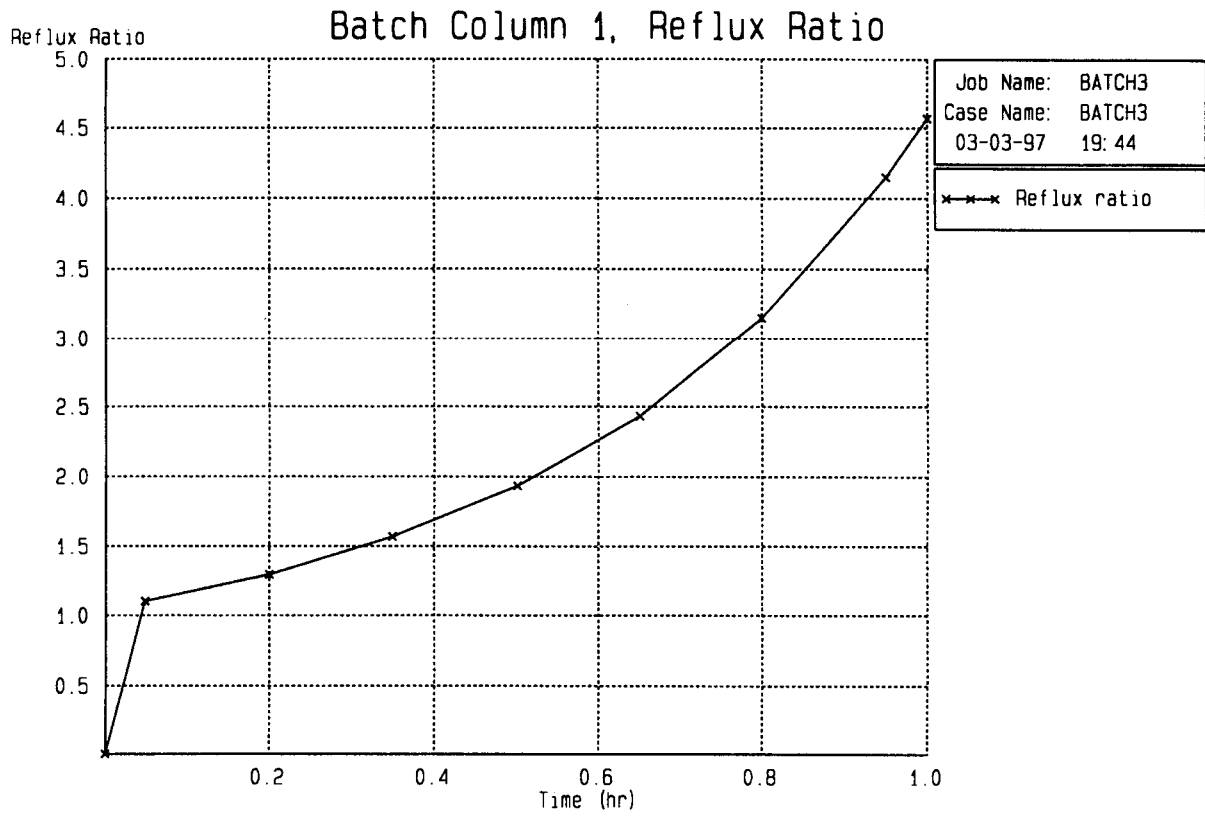
Batch Column 1, Step 1 Decanter Distillate



Batch Column 1, Step 2 Distillate



Chemcad Training Manual



(1) WATER

(2) ACETIC ACID

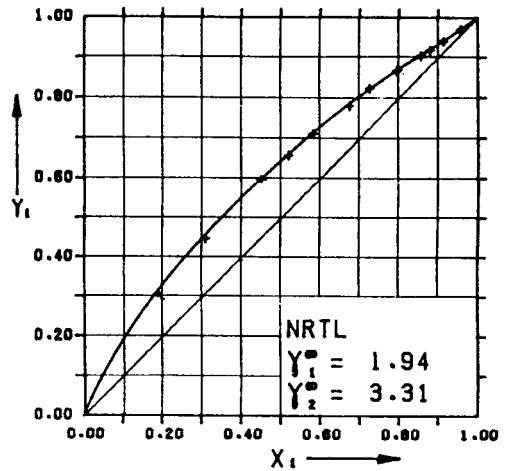
H2O
C2H4O2

++++ ANTOINE CONSTANTS REGION +++++ CONSISTENCY
(1) P. 07131 1730.630 233.426 1- 100 C
(2) P. 02100 1936.010 258.451 18- 119 C

PRESSURE= 760.00 MM HG (1.013 BAR)

LIT: BRUSSET H., KAISER L., HOCQUEL J., CHIM. IND., GENIE CHIM. 99, 237 (1969).

CONSTANTS: A12 A21 ALPHA12
MARGULES C.4178 0.9533
VAN LAAR 0.4973 1.0623
WILSON 795.5876 111.6579
NRTL 712.1791 370.1059 1.4032
UNIQUAC -55.4002 116.7514



EXPERIMENTAL DATA			MARGULES		VAN LAAR		WILSON		NRTL		UNIQUAC	
T DEG C	X1	Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1
118.30	0.0	0.0	0.11	0.0	0.11	0.0	0.11	0.0	0.11	0.0	0.11	0.0
110.60	0.1531	0.3063	-0.76	-0.0105	-0.61	-0.0073	-0.71	-0.0048	0.10	-0.0077	-0.51	-0.0080
107.80	0.3084	0.4467	-0.22	-0.0316	-0.30	-0.0224	-0.38	-0.0200	0.02	-0.0089	-0.22	-0.0220
105.20	0.4498	0.5573	-0.03	-0.0239	-0.18	-0.0181	-0.24	-0.0154	-0.20	-0.0000	-0.14	-0.0159
104.30	0.5135	0.6580	0.07	-0.0190	-0.04	-0.0172	-0.08	-0.0147	-0.13	-0.0021	-0.02	-0.0146
103.50	0.5324	0.7112	0.01	-0.0194	-0.03	-0.0124	-0.07	-0.0102	-0.14	-0.0018	-0.04	-0.0096
102.80	0.6750	0.7797	0.19	-0.0019	0.25	-0.0077	0.23	-0.0063	0.19	-0.0050	0.22	-0.0052
102.10	0.7261	0.8239	-0.10	0.0105	0.00	0.0039	-0.01	0.0048	-0.00	0.0028	-0.03	0.0059
101.50	0.7951	0.8671	-0.20	0.0093	-0.06	0.0042	-0.05	0.0044	0.01	0.0002	-0.09	0.0053
100.90	0.8556	0.9042	-0.39	0.0050	-0.23	0.0029	-0.22	0.0029	-0.13	-0.0007	-0.27	0.0032
100.80	0.8797	0.9186	-0.32	0.0027	-0.18	0.0020	-0.17	0.0020	-0.06	-0.0007	-0.21	0.0020
100.50	0.9134	0.9400	-0.36	-0.0005	-0.24	0.0006	-0.23	0.0006	-0.12	-0.0002	-0.26	0.0002
100.20	0.9578	0.9708	-0.28	-0.0029	-0.21	-0.0011	-0.20	-0.0009	-0.13	0.0002	-0.22	-0.0015
100.00	1.0000	1.0000	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0	0.00	0.0
MEAN DEVIATION:			0.24	0.0107	0.19	0.0083	0.22	0.0073	0.10	0.0025	0.19	0.0078
MAX. DEVIATION:			0.76	0.0316	0.61	0.0224	0.71	0.0200	0.20	0.0089	0.51	0.0220

(1) ETHYL ACETATE

(2) WATER

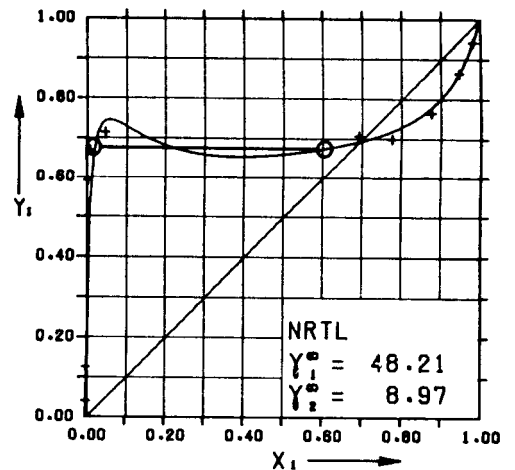
C4H8O2
H2O

++++ ANTOINE CONSTANTS REGION +++++ CONSISTENCY
(1) 7.10179 1244.951 217.881 16- 76 C METHCD 1 -
(2) 8.07131 1730.630 233.426 1- 100 C METHCD 2 +

PRESSURE= 760.00 MM HG (1.013 BAR)

LIT: ELLIS S.R.M., GARBETT R.D., IND. ENG. CHEM. 52, 385 (1960).

CONSTANTS: A12 A21 ALPHA12
MARGULES 3.7016 1.8656
VAN LAAR 3.8245 1.9685
WILSON 1147.4297 2084.0256
NRTL 935.6880 2316.3631 0.4104
UNIQUAC 757.5853 151.6420



EXPERIMENTAL DATA			MARGULES		VAN LAAR		WILSON		NRTL		UNIQUAC	
T DEG C	X1	Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1
98.85	0.0006	0.0405	0.17	-0.0064	0.34	-0.0121	0.46	-0.0164	0.43	-0.0153	0.33	-0.0118
96.20	0.0011	0.1256	-1.43	0.0426	-1.14	0.0330	-0.95	0.0267	-0.96	0.0270	-1.15	0.0333
86.50	0.0049	0.5910	-4.33	0.3009	-3.44	0.2774	-3.48	0.2785	-2.44	0.2516	-3.27	0.2728
76.20	0.0086	0.6680	-9.54	0.2495	-8.44	0.2249	-9.39	0.2468	-6.79	0.1899	-8.00	0.2155
70.55	0.0459	0.7140	5.38	-0.0466	5.05	-0.0423	-4.33	0.0841	3.94	-0.0277	5.74	-0.0490
70.45	0.6900	0.7070	0.40	0.1913	-0.34	0.0676	-1.26	-0.0013	-1.13	0.0160	-0.33	0.0557
70.50	0.7750	0.6570	0.11	0.0027	-0.66	0.0210	-1.26	-0.0305	-1.19	-0.0191	-0.58	0.0146
71.45	0.8737	0.7650	-0.74	0.0170	-1.02	0.0021	-0.90	-0.0129	-0.96	-0.0101	-0.81	0.0037
73.35	0.9444	0.8650	-1.18	-0.0039	-1.09	-0.0033	-0.50	0.0088	-0.66	0.0055	-0.89	0.0015
75.55	0.9807	0.9411	-0.57	-0.0095	-0.46	-0.0063	-0.04	0.0060	-0.15	0.0028	-0.36	-0.0034
MEAN DEVIATION:			2.41	0.0827	2.20	0.0690	2.25	0.0712	1.98	0.0565	2.15	0.0661
MAX. DEVIATION:			9.54	0.3009	8.44	0.2774	9.39	0.2785	6.79	0.2516	8.00	0.2728

(1) ETHYL ACETATE C4H8O2

(2) ACETIC ACID C2H4O2

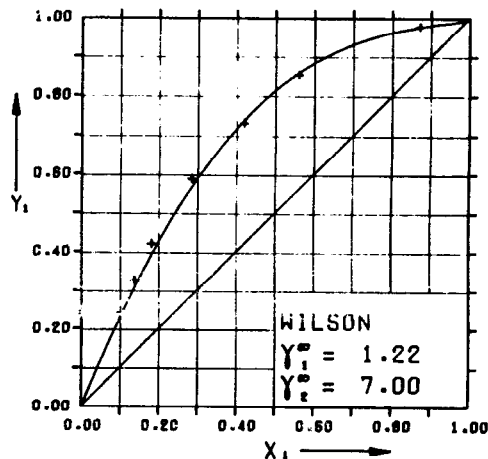
++++ ANTOINE CONSTANTS REGION +++++
 (1) 7.18179 1244.950 217.881 16- 76 C METHOD 1 +
 (2) 7.55960 1644.048 233.524 17- 118 C METHOD 2 -

PRESSURE= 760.00 MM HG (1.013 BAR)

LIT: GARNER F.H., ELLIS S.R.M., PEARCE C.J.
 CHEM. ENG. SCI. 3, 48 (1954).

CONSTANTS: A12 A21 ALPHA12

MARGULES 0.2051 0.3976
 VAN LAAR 0.2313 0.4282
 WILSON -997.8935 2738.1344
 NRTL 844.2996 -436.9443 0.3138
 UNIQUAC 677.2519 -376.3130



EXPERIMENTAL DATA			MARGULES		VAN LAAR		WILSON		NRTL		UNIQUAC	
T DEG C	X1	Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1	DIFF T	DIFF Y1
108.10	0.1380	0.3270	0.08	0.0125	0.18	0.0121	-0.09	0.0136	0.01	0.0142	-0.02	0.0137
105.10	0.1820	0.4220	-0.21	0.0232	-0.15	0.0240	-0.39	0.0228	-0.31	0.0253	-0.33	0.0240
99.70	0.2860	0.5890	0.02	0.0229	-0.01	0.0255	0.02	0.0149	-0.11	0.0247	-0.06	0.0216
93.60	0.4200	0.7320	-0.19	0.0084	-0.26	0.0103	0.40	-0.0117	-0.25	0.0078	-0.16	0.0045
88.30	0.5610	0.8540	-0.56	0.0170	-0.57	0.0169	0.90	-0.0087	-0.49	0.0148	-0.40	0.0126
79.50	0.8720	0.9780	-0.89	0.0020	-0.84	0.0027	0.85	0.0003	-0.01	0.0029	-0.79	0.0029
MEAN DEVIATION:			0.33	0.0144	0.34	0.0152	0.44	0.0120	0.33	0.0149	0.29	0.0132
MAX. DEVIATION:			0.89	0.0232	0.84	0.0255	0.90	0.0228	0.81	0.0253	0.79	0.0240