REFERENCES

1. Phelps, P. D.; Caringi, J. J.; Flowers, L. I.; Boden, E. P.; and Ramsey, D. L. Adsorption Process for Organic Base Recovery from Aqueouse Brine Solutions. US. Patent. 5,759,406, 1998.
2. Mark, Meckes. Extraction Process Separation Organics from Sludges, Solids and Sediment. Tech Trends, 1993.
3. DSM Andeno, Co,. Ltd. An Overview. pp 33. Netherlands, 1994.
4. ยูไนเต็ดฟาร์มาแอนด์ไบโอติคส์, บริษัท. แบบบันทึกกระบวนการผลิตยาแอมมอกซิซิลลิน. ปทุมธานี, 2541.
5. Merk, Co,. Ltd. Safety Data Sheet. CD ROM. Germany, 2000/1.
6. G. Robertson.; and Thomas L. Jacobs. Laboratory Practice of Organic Chemistry. 4th ed. pp116-117. New York: Macmillan, 1965.
7. Robert, T. M.; and Robret, N. B. Organic Chemistry. 6th ed. pp 246-271. New Jersey: Prentice-Hall, 1992.
8. Wade L.G, JR. Organic Chemistry. 2 nd ed. pp 823-878. Singapore: Prentice-Hall, 1991.
9. Leonard, D.; Lygo, B.; and Proter, G. Advanced Pratical Organic Chemistry. 2 nded. pp 58-59. Chapman & Hall, 1995.
10. Brian, S.F. Antony,; J. H. Peter, W.G. S.; and Austin R.T. Textbook of Practical Organic Chemistry. 5th ed., pp 167. John Wiley & Sons, 1989.
11. Cherry, Chan.; Nicole, Chau,; Keena, Chiu,; and Eva, Yip. Absorption and Adsorption. Chemistry Project. Chapter 7, 1999.
12. Timothy Oppelt, E. Soluvent Extraction Technology. Resources Conservation Company B. E. S. T.® Solvent Extraction Technology. USA, 1993.
13. Bohrer, M. P. Chandross, E. A. Mixon, D.A. Psota-Kelty, L.A.; and Trevor, D.J. Process for Treating By-Product of Optical Fiber Fabrication. US. Patent. 6,007,727, 1999.

14. Holtzapple, M.T.; Davison, R.R.; and Lucttich, T. Recovery of Fermentation Salts from Dilute Aqueous Solution. US. Patent. 5,986,133, 1999.

15. Martin, P.H.; and Michacels, S.L. Use of Aqueous Triethylamine/Phosphoric Acid Salt Solution to Extract Water and Triethylamine from Solution Thereof in Organic Solvents. US. Patent. 4,326,082, 1982.

16. Daniel, J. P.; Carl, R.J.; and Marvin, J. M. Experiments and Techniques in Organic Chemistry. pp 26-29. Singapore: Prentice-Hall, 1992.

17. Andrew D. E.; Lenore S. C.; and Arnold, E. G. Standard Methods for the Examination of Water and Wastewater. 19th ed. Chapter 5. pp13-14. Washington: APHA, 1995.

18. Burlington, Ontario.Treatment. Great Lakes, 1994.

19. Salzman, W. R. Vapor Pressure Diagrams and Boiling Diagrams. Arizona University, 2001.

APPENDICES

APPENDIX A

1. Determination of Percentage of Solvents Recovery

$$\%\text{Recovery} = \frac{\text{volume of recovered solvents}}{\text{volume of starting solvents}} \times 100$$

Volume of starting solvents = 18% of solvents in mother liquor 2,000 ml, or 270 ml of solvents mother liquor 2,000 ml.

Volume of starting solvents = 17% of solvents in mother liquor 2,000 ml, or 255 ml of solvents mother liquor 2,000 ml.

Volume of starting solvents = 16% in of solvents mother liquor 2,000 ml, or 240 ml mother liquor 2,000 ml.

Volume of recovered solvents = 190 ml in of solvents mother liquor 2,000 ml.

An example of calculation

$$\%\text{Recovery of solvents} = \frac{190}{270} \times 100$$

$$= 65.18$$

Table A-1 Distillation of fresh mother liquor by control temperature-top column for methylene chloride and acetone separation.

Experiment	Temp.- Top. Column (°C)	Time (min)	Solvents in Mother Liquor (ml)	(%)	Recovery Sovents (ml)	(%)
1	42-55	0-25	270	18	20	7.41
	55-59	26-215	270	18	176	65.18
Total	42-59	215	270	18	196	72.59
2	43-55	0-15	255	17	20	7.84
	55-59	15-200	255	17	166	65.10
Total	42-59	200	255	17	186	72.94
3	43-55	0-20	255	17	20	7.84
	55-59	20-130	255	17	166	65.00
Total	42-59	130	255	17	186	72.94
4	42-55	0-20	240	16	16	6.67
	55-59	20-120	240	16	156	65.00
Total	42-59	120	240	16	172	71.67
5	43-55	0-20	255	17	17	6.67
	55-59	20-190	255	17	170	66.66
Total	42-59	190	255	17	187	73.33
Average	55-59	-	255	17	-	65.39

APPENDIX B

1. Determination of Percentage of Triethylamine Recovery

$$\%\text{Recovery} = \frac{\text{volume of recovered triethylamine}}{\text{volume of starting triethylamine}} \times 100$$

Volume of starting triethylamine = 185 L of triethylamine in mother liquor 3,200 L.

Volume of starting triethylamine = 115.6 ml of triethylamine in mother liquor 2,000 ml.

Volume of starting triethylamine = 4.8 ml of triethylamine in residual mother liquor 75 ml, volume decrease 10% of mother liquor.

Volume of starting triethylamine = 5.4 ml of triethylamine in residual mother liquor 75 ml, volume decrease 20% of mother liquor

Volume of starting triethylamine = 6.2 ml of triethylamine in residual mother liquor 75 ml, volume decrease 30% of mother liquor.

Volume of starting triethylamine = 7.2 ml of triethylamine in residual mother liquor 75 ml, volume decrease 40% of mother liquor.

Volume of starting triethylamine = 8.7 ml of triethylamine in residual mother liquor 75 ml, volume decrease 50% of mother liquor.

Volume of starting triethylamine = 10.8 ml of triethylamine in residual mother liquor 75 ml, volume decrease 60% of mother liquor.

Volume of starting triethylamine = 14.4 ml of triethylamine in residual mother liquor 75 ml, volume decrease 70% of mother liquor.

Volume of recovered triethylamine = 3.1 ml of triethylamine in residual of mother liquor 75 ml, volume decrease 10% of mother liquor.

An example of calculation

$$\%\text{Recovery of triethylamine} = \frac{3.1}{4.8} \times 100$$

$$= 64.58$$

Table B-1 Effect of mother liquor quantity on triethylamine separation from residual mother liquor.

Volume	Mother Liquor		Starting	TEA Recovery						
decrease	Orginal	Residual	TEA	Experiment 1.		Experiment 2.		Experiment 3.		Average
(%)	(ml)	(ml)	(ml)	(ml)	(%)	(ml)	(%)	(ml)	(%)	(%)
10	2,000	1,800	4.8	3.1	64.58	3.3	68.75	3.5	72.92	68.75
20	1,725	1,533	5.4	3.5	64.81	3.8	70.37	3.8	70.37	68.52
30	1,458	1,276	6.2	4.3	69.35	4.4	69.35	4.8	77.42	72.04
40	1,201	1,025	7.2	5.6	77.78	5.7	79.17	5.6	77.78	78.24
50	954	795	8.7	6.8	78.16	7.1	81.61	7.3	83.91	81.23
60	720	576	10.8	8.5	78.79	8.8	81.48	8.8	81.48	80.55
70	501	376	14.4	11.9	82.64	11.9	82.64	12.1	84.02	83.10

APPENDIX C

1. Determination of Percentage of Recovery Triethylamine

$$\%\text{Recovery} = \frac{\text{volume of recovered triethylamine}}{\text{volume of starting triethylamine}} \times 100$$

Volume of starting triethylamine = 185 L of triethylamine in mother liquor 3,200 L or 185 L of triethylamine in residual mother liquor 1,296 L, volume decrease 55% of residual mother liquor after solvents separation.

Volume of starting triethylamine = 28.5 ml of triethylamine in residual mother liquor 200 ml

Volume of recovered triethylamine =1.9 ml of triethylamine in residual mother liquor 75 ml, volume decrease 55% of residual mother liquor after solvents separation.

An example of calculation

$$\%\text{Recovery of triethylamine} = \frac{1.9}{28.5} \times 100$$

$$= 6.67$$

Table C-1 Effect of pH on triethylamine separation from residual of mother liquor.

NaOH	NaOH	Experiment 1.			Experiment 2.			Experiment 3.			Average
(w/v)	(50%w/v)	pH	TEA Recovery		pH	TEA Recovery		pH	TEA Recovery		TEA Recovery
(%)	(ml)		(ml)	(%)		(ml)	(%)		(ml)	(%)	(%)
0.00	0	3.23	-	-	3.17	-	-	3.25	-	-	-
0.25	1	8.05	-	-	8.16	-	-	7.65	-	-	-
0.50	2	9.33	-	-	9.26	-	-	9.23	-	-	-
0.64	3	9.63	-	-	9.50	-	-	9.80	-	-	-
0.98	4	9.69	-	-	9.59	2.40	8.24	10.04	-	-	2.81
1.22	5	9.77	1.90	6.67	9.77	4.30	15.09	10.12	1.90	6.67	9.48
1.46	6	9.83	3.80	13.33	9.83	6.20	21.75	10.14	3.80	13.33	16.14
1.69	7	9.93	4.70	16.49	10.00	7.60	26.67	10.31	4.30	15.09	19.42
1.92	8	10.02	8.10	28.42	10.38	11.50	40.35	10.43	6.20	21.75	30.17
2.15	9	10.15	9.10	31.93	10.96	14.80	51.93	10.54	7.20	25.26	36.37
2.38	10	10.31	11.90	41.75	11.89	17.70	62.11	10.75	7.60	26.67	43.51
2.61	11	10.54	16.70	58.60	12.50	21.00	73.68	11.13	12.40	43.51	58.60
2.83	12	10.97	20.50	71.93	12.73	22.00	77.19	11.96	13.40	47.02	65.38
3.05	13	12.05	23.40	82.11	12.93	23.00	80.70	12.64	16.70	58.60	73.80
3.27	14	12.53	23.80	83.51	13.06	23.50	82.46	12.91	17.70	62.11	76.13
3.49	15	12.76	23.80	83.51	13.12	23.60	82.81	13.08	20.50	71.93	79.42
3.70	16	12.92	24.40	85.61	13.22	23.80	83.51	13.24	22.00	77.19	82.10
3.92	17	13.01	24.40	85.61	13.27	23.80	83.51	13.32	22.50	78.93	82.69
4.13	18	13.06	24.40	85.61	13.33	23.80	83.51	13.38	22.50	78.93	82.69

APPENDIX D

1. Determination of Percentage of Recovery Triethylamine

$$\%\text{Recovery} = \frac{\text{volume of recovered triethylamine}}{\text{volume of starting triethylamine}} \times 100$$

Volume of starting triethylamine = 185 L of triethylamine in mother liquor 3,200 L or 185 L of triethylamine in residual mother liquor 1,296 L, volume decrease 55% of residual mother liquor after solvents separation.

Volume of starting triethylamine = 10.7 ml of triethylamine in residual mother liquor 75 ml, volume decrease 55% of residual mother liquor after solvents separation.

Volume of recovered triethylamine = 8.8 ml of triethylamine in residual mother liquor 75 ml, volume decrease 55% of residual mother liquor after solvents separation.

An example of calculation

$$\%\text{Recovery of triethylamine} = \frac{8.8}{10.7} \times 100$$

$$= 82.24$$

Table D-1 Effect of reaction temperature on triethylamine separation from residual of mother liquor.

Temp.	TEA Recovery						
	Experiment 1.		Experiment 2.		Experiment 3.		Average
(°C)	(ml)	(%)	(ml)	(%)	(ml)	(%)	(%)
25	8.4	78.50	8.1	75.70	8	74.77	76.32
35	8.2	76.64	8.2	76.64	7.8	72.90	75.39
45	8.6	80.37	8.5	79.44	8.2	76.64	78.82
55	8.6	80.37	8.5	79.44	8.2	76.64	78.82
65	8.8	82.24	8.8	82.24	8.4	78.50	80.99
75	8.8	82.24	8.8	82.24	8.6	80.37	81.61

Table D-2 Effect of reaction time on triethylamine separation from residual of mother liquor

Time	Recovery TEA						
	Experiment 1.		Experiment 2.		Experiment 3.		Average
(min)	(ml)	(%)	(ml)	(%)	(ml)	(%)	(%)
5	8.4	72.9	7.8	78.5	8.4	78.66	76.63
10	8.8	75.7	8.1	82.24	8.8	82.23	80.06
15	8.9	77.57	8.3	82.24	8.8	82.23	81.00
20	8.9	78.5	8.4	82.24	8.8	82.23	81.31
25	8.9	79.44	8.5	82.24	8.8	82.23	81.62
30	8.9	79.44	8.5	83.18	8.9	83.23	81.93

APPENDIX E

1. Determination of COD

$$\text{COD as mg } O_2/l = \frac{(A - B) \times M \times 8000}{\text{ml of sample}}$$

Where

A = ml of standard ferrous ammonium sulfate used for blank,

B = ml of standard ferrous ammonium sulfate used for sample, and

M = molarity of standard ferrous ammonium sulfate.

When

Volume of sample = 0.05 ml

Standard ferrous ammonium sulfate used for blank = 23.4 ml

Standard ferrous ammonium sulfate used for sample = 13.6 ml

Molarity of standard ferrous ammonium sulfate = 0.0985 M

An example of calculation

$$\text{COD as mg } O_2/l = \frac{(23.4\text{-}13.6) \times 0.0985 \times 8{,}000}{0.05}$$

$$= 153{,}280$$

APPENDIX F

1. Determination of Yield of Amoxicillin Trihydrate

$$\text{Yield of Amoxicillin trihydrate} = \frac{\text{weight of Amoxicillin trihydrate}}{\text{weight of 6-APA}}$$

When

Weight of Amoxicillin trihydrate = 70.11 g

Weight of 6-APA = 40.04 g

An example of calculation

$$\text{Yield of Amoxicillin trihydrate} = \frac{70.11}{40.04}$$

$$= 1.751$$

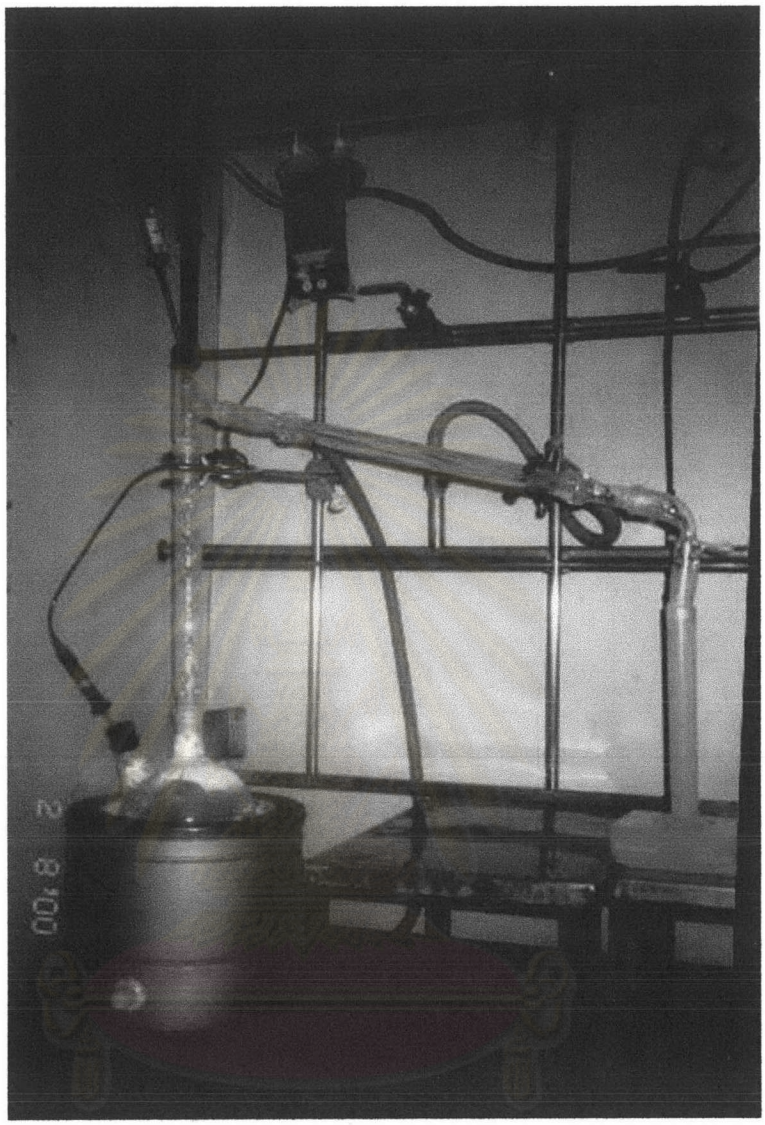

Figure G-1 Distillation of mother liquor from amoxicillin trihydrate process.

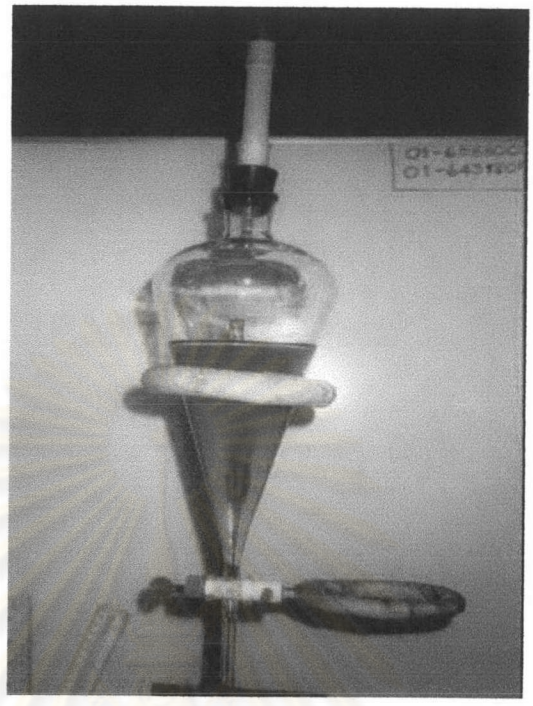

Figure G-2 Triethylamine was separated from residual mother liquor.

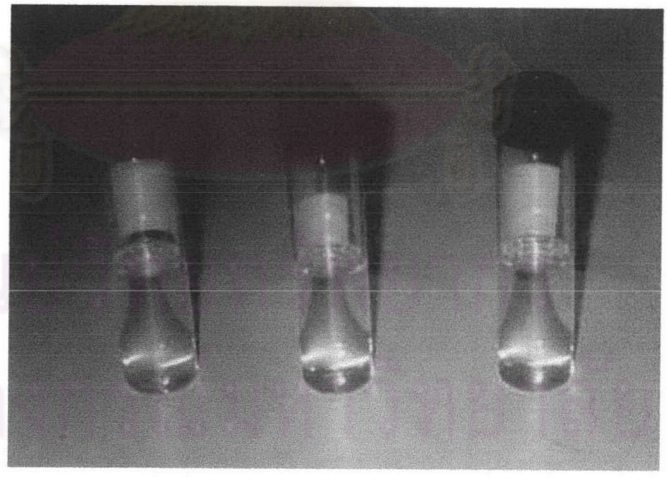

Figure G-3 Recovered triethylamine product.

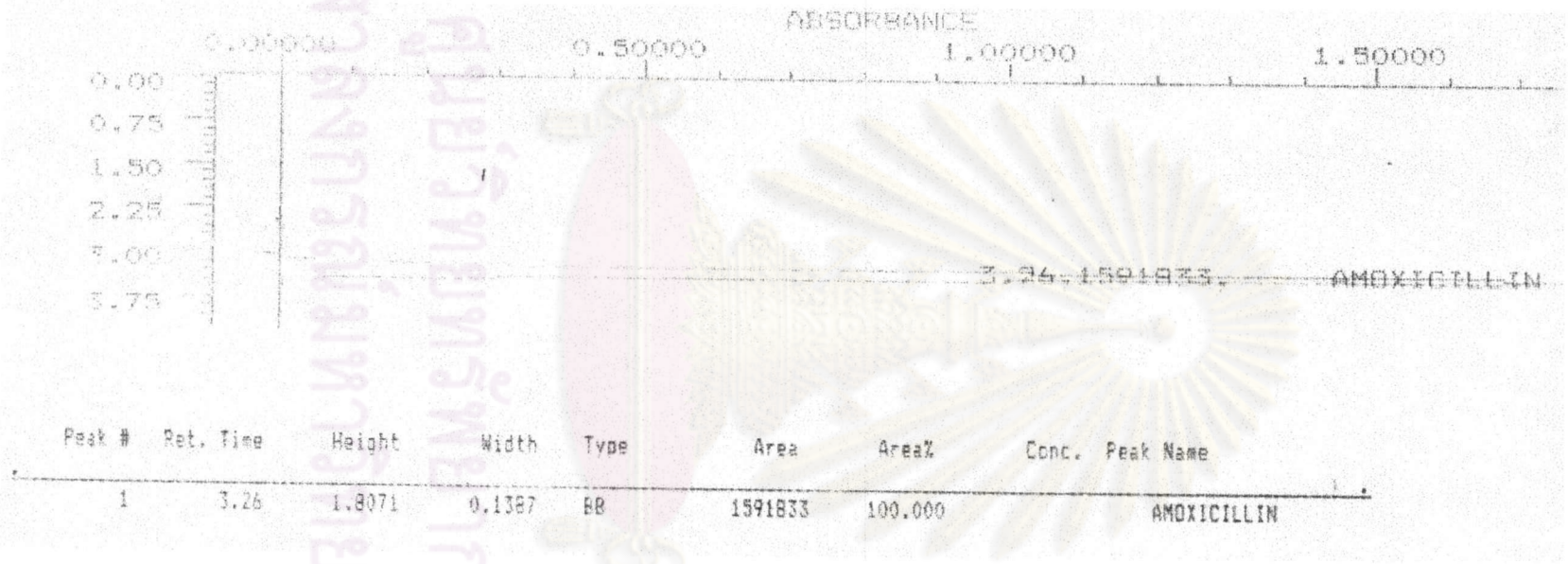

Figure G-4 The peak of amoxicillin trihydrate from HPLC analysis (standard).

Figure G-5 The peak of amoxicillin trihydrate from HPLC analysis (fresh triethylamine).

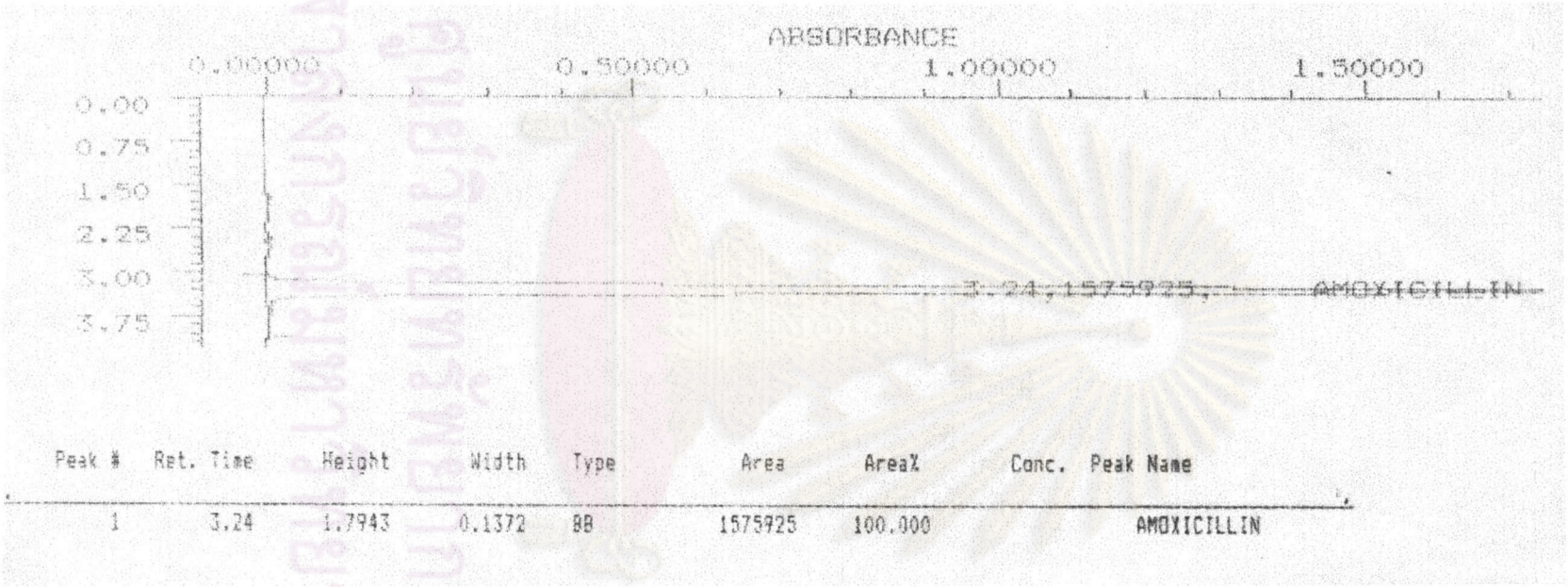

Figure G-6 The peak of amoxicillin trihydrate from HPLC analysis (recovered triethylamine).

VITA

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