

เอกสารความคืบ

1. H.S.Bean; A.H.Beckett and J.E.Carless. Advance in Pharmaceutical Sciences. 2d ed. London. Academic Press Inc, 1976.
2. Julius Rath. "On the Hydrolytic Decomposition of Acetylated Hydroxycarboxylic Acids". Ann. 358, 98(1908); through Chemical Abstracts 2(April 1908):1002.
3. D.E.Tsakalotos, and S.Horsch. Bull. Soc. chim. 15, 745-747(1914). "Decomposition of Aspirin by Water". through Chemical Abstracts 9(March 1915):787.
4. D.E.Tsakalotos, and S.Horsch. Bull. Soc. chim. 17, 401-406(1915). "Anomalies of the Decomposition of Aspirin by Water". through Chemical Abstracts 10(February 1916):591.
5. Arvid Wolf. Svenk Kem. Tidskrift. 29, 109-112(1917). "Hydrolysis of Acetylsalicylic Acid". through Chemical Abstracts 11 (September 1917):2634.
6. C.Morton Quart.J. Pharm. Pharmacol., 6, 492(1933). "Determination of the Decomposition of Aspirin". through Journal of Pharmaceutical Sciences 59(August 1970):1054.
7. Victor K.La Mer, and Josept Greenspan. "Kinetics of the Saponification of Acetylated Hydroxy Acids". Journal of the American Chemical Society 56(July 1934):1492-1499.
8. L.J. Edwards. Trans. Faraday Soc., 46, 733(1950). "Determination of the Decomposition of Aspirin". through Journal of Pharmaceutical Sciences 59(August 1970):1055-1056.

9. L.J. Edwards. Trans. Faraday Soc., 48, 696(1952). "Determination of the Decomposition of Aspirin". through Journal of Pharmaceutical Sciences 59(August 1970):1055-1056.
10. David Davidson, and Leatrice Auerbach. "The Acid Anhydride Character of Aspirin". Journal of the American Chemical Society 75(December 1953):5984-5986.
11. E.A. Mario, and R.J. Gerraughty. Journal of Pharmaceutical Sciences 54(February 1965):321-323.
12. Edward R. Garrett. "The Kinetics of Saponification of Acyl Esters of Salicylic Acid". Journal of the American Chemical Society 79(July 1957):3401-3408.
13. Edward R. Garrett. "The Neutral Saponification of the Aspirin Anion in Aqueous and Mixed Solvent". The Journal of Organic Chemistry 26(October 1961):3660-3663.
14. The United States Pharmacopoeia. 19th rev. Easton Pa. Mack Publishing Co., 1975, p39.
15. The National Formulary. 14th ed. Easton Pa. Mack Publishing Co., 1975, p 55.
16. K.H. Lee; Leon Thompkins and Michael R. Spencer. "Separation of Acetylsalicylic Acid and Salicylic Acid by Sephadex Gel Filtration". Journal of Pharmaceutical Sciences 57(July 1968):1240-1242.
17. J.R. Watson; P. Crescuelo and Fumi Matsui. "Rapid Simultaneous Determination of Salicylic Acid and Aspirin by GC I:

- Analysis of Synthetic Aspirin-Salicylic Acid Mixtures and
of Single-Component Aspirin Tablets". Journal of
Pharmaceutical Sciences 60(March 1971):454-458.
18. S.Patel; J.H.Perrin and J.J.Windheuser. "GLC Analysis of
Aspirin from Solid Dosage Forms". Journal of Pharmaceutical
Sciences 61(November 1972):1794-1796.
19. Raymond C.Crippen, and Henry C.Freimuth. "Determination of
Aspirin by Gas Chromatography". Analytical Chemistry 36
(February 1964):273-275.
20. J.G.Kikelly. "Gas Chromatographic Determination of
Acetylsalicylic Acid". Analytical Chemistry 36(November
1964):2248-2250.
21. British Pharmacopoeia 1973. London. Her Majesty's Stationery
Office, 1973, p 37.
22. Pharmacopoeia of Japan. 2 vol. 8th ed. Part I. Japan. Society
of Japanese Pharmacopoeia, 1973. p75.
23. Glenn L.Jenkins; Adelbert M Knevel and Frank E.DiGangi.
Quantitative Pharmaceutical Chemistry. 7th ed. USA.
Mc Graw-Hill, Inc., 1977, p 111.
24. European Pharmacopoeia Council of Europe. 4 vols. no.I France.
Maisonneuve S.A., 1969, p 235.
25. British Pharmaceutical Codex 1973. Great Britain. Willium
Clowes & Sons, 1973, p 35.
26. Song-Ling Lin. "Differentiating Nonaqueous Titration of
Salicylic Acid and Acetylsalicylic Acid Combination".

- Journal of Pharmaceutical Sciences 56(July 1967):1130-1140.
27. Randall B.Tinker, and Arthur J.MacBay. "Spectrophotometric Determination of Acetylsalicylic Acid and Salicylic Acid". Journal of the American Pharmaceutical Association, Scientific Edition 43(May 1954):315-317.
28. A.Y.Gore et al. "Significance of Salicylic Acid Sublimation in Stability Testing of Aspirin-Containing Solids". Journal of Pharmaceutical Sciences 57(November 1968):1850-1854.
29. Edgar E.Theimer, and Emil W.Ciurezak. "Quick Specific Assay for Aspirin". Journal of Pharmaceutical Sciences 66 (January 1977):139-140.
30. George H.Schenk, Fred H.Boyer, Corbin I.Miles, and Donald R. Wirz. "Effect of Acids on Fluorescence of Acetysalicylic Acid and Salicylic Acid". Analytical Chemistry 44(August 1972):1593-1595.
31. Norman Shane, and Dennis Mielr. "Fluorometric Determination of Salicylic Acid in Aspirin Products Includin; Non-interfering, and Buffered Substances". Journal of Pharmaceutical Sciences 59 (March 1970): 397 - 400
32. Corbin I.Miles, and George H.Schenk."Fluorescence o: Acetylsalicylic Acid in Solution and Its Measurement in Presence of Salicylic Acid". Analytical Chemistry 42 (May 1970): 656 - 658.
33. Norman Shane, and Robert Stillman."Fluorometric Determination of Salicylic Acid of Salicylic Acid in Buffered Aspirin

- Product". Journal of Pharmaceutical sciences 60 (January 1971) : 114 - 116.
34. Maria Vietti -Michelina, Atti Sci.Torine, CI.Sci.Fis.Mat.Natur 89,383 (1954 - 1955) "Analytical Separation of Salicylic Acid from Their Mixture" through; Chemical Abstracts 50 (May 1956) : 6255h.
35. L.A.Korshunov; Z.B.Kuznetsova and M.K.Shchennikova. Zhur.fiz Khim 23,1292-8 (1949)."Reduction of Weak Acids at a Dropping - Mercury Cathode".through Chemical Abstracts 44 (April 1950): 2873 d.
36. F.Reimers. Arch.Pharm.Chami.74,531-548 (1967) "Simplified Thin-Layer Chromatography used for Identification of Compound Analgesic and Antipyretic Preparations". through Chemical Abstracts 67 (October 1967): 67624 h.
37. Edward R.Garrett, and James L.Johnson."Selection, Evaluation, and Control of The Assay of the Pharmaceutical Product III.Statistical and Economic Evaluation of Three-Component Infrared Spectrophotometric Assay of Aspirin Anhydried". Journal of Pharmaceutical Sciences 51 (August 1962): 767-770.
38. Robert M.Silverstein;G.Clayton Bassler and Terence C.Morrill. Spectrometric Identification of Organic Compounds. 3rd ed. New York.John Wiley & Sons, Inc.1974, p 96-10+

ภาคบันทึกตารางที่ 1

ตาราง Absorbance ของ Standard Aspirin Solution cm^{-1} Wavenumber 1750 cm⁻¹

Cell Pathlength 0.1 mm.

ใช้ Chloroform baseline, Baseline 1, Baseline 2.

Concentration % w/v	Chloroform	Absorbance	
		Baseline 1	Baseline 2
0.5	0.0788	0.0842	0.0730
1.0	0.1652	0.1656	0.1464
1.5	0.2416	0.2430	0.2186
2.0	0.3113	0.3137	0.2797
2.5	0.3933	0.3911	0.3564
3.0	0.4572	0.4507	0.4106
3.2	0.4847	0.4731	0.4328
3.4	0.5127	0.4986	0.4541
3.6	0.5427	0.5324	0.4827
3.8	0.5585	0.5463	0.4975
4.0	0.5816	0.5712	0.5307
4.2	0.6116	0.5975	0.5448
4.4	0.6677	0.6514	0.5871
4.6	0.6954	0.6628	0.6068
5.0	0.7227	0.7013	0.6435

ตารางที่ 2

ตาราง Absorbance ของ Standard Aspirin Solution cm^{-1} Wavenumber 1690 cm^{-1} .

Cell Pathlength 0.1 mm.

溶剂 Chloroform Baseline, Baseline 1 และ Baseline 2

Concentration % w/v	Absorbance cm^{-1} Baseline		
	Chloroform	1	2
0.5	0.0887	0.0969	0.0859
1.0	0.1825	0.1869	0.1608
1.5	0.2748	0.2777	0.2425
2.0	0.3643	0.3666	0.3249
2.5	0.4593	0.4611	0.4109
3.0	0.5466	0.5471	0.4862
3.2	0.5691	0.5687	0.5042
3.4	0.5998	0.5963	0.5311
3.6	0.6290	0.6263	0.5568
3.8	0.6522	0.6483	0.5759
4.0	0.6941	0.6684	0.5921
4.2	0.7143	0.7099	0.6281
4.4	0.7654	0.7592	0.6762
4.6	0.7786	0.7782	0.6908
5.0	0.8264	0.8223	0.7277

ການທີ 3

ມີ Absorbance ຂອງ Standard Salicylic Acid Solution ໃນ Wavenumber
 1657 cm^{-1} , Cell Pathlength 0.1 mm.

ໃຫ້ chloroform baseline, baseline 1, baseline 2.

Concentration % w/v	Absorbance ທີ Baseline		
	Chloroform	1	2
0.25	0.0423	0.0554	0.0342
0.50	0.1004	0.1127	0.0763
0.75	0.1641	0.1753	0.1252
1.00	0.2239	0.2345	0.1682
1.25	0.2827	0.2911	0.2113
1.50	0.3529	0.3598	0.2662
1.60	0.3723	0.3804	0.2806
1.70	0.3960	0.4058	0.3038
1.80	0.4291	0.4359	0.3253
1.90	0.4499	0.4567	0.3434
2.00	0.4718	0.4824	0.3619
2.10	0.5638	0.5844	0.4432
2.20	0.5670	0.5724	0.4459
2.30	0.5541	0.5807	0.4211
2.40	0.5735	0.5806	0.4334

ກາງທີ 4

Analysis of Aspirin in the Presence of Salicylic Acid

Aspirin in Sample, mg.	Added Salicylic Acid, mg.	Aspirin Found mg.	Aspirin Found %
625	0	625	100.00
625	125	629.19	100.67
625	250	629.94	100.79
625	375	623.00	99.68
625	500	616.00	98.56

Aspirin in Sample, mg.	Added Salicylic Acid, mg.	Aspirin Found mg.	Aspirin Found %
500	375	500	100.00
550	375	555	100.90
600	375	597.5	99.58
650	375	640	98.46
700	375	670	95.71
750	375	725	96.67

ตารางที่ 5

Effect of Time on Analysis of Aspirin and Salicylic Acid

เวลา (นาที)	Absorbance	
	Aspirin at 1750 cm^{-1}	Salicylic Acid at 1657 cm^{-1}
0	0.4177	0.3873
30	0.4088	0.3858
60	0.4051	0.3864
120	0.4015	0.3860
180	0.3999	0.3832
240	0.3975	0.3773

ตารางที่ ๖

Percentage recovery for Assay of Aspirin by Infrared

Spectrophotometry

Aspirin in Sample, mg.	Added Aspirin mg.	Aspirin Found mg.	% Recovery
500	175	172.8	98.74
500	166.8	167.8	100.60
500	170.6	179.9	100.73
500	176.1	177.5	100.30

% Recovery for the Assay Of Salicylic Acid by IR Method

Salicylic Acid in Sample,mg.	Added Salicylic Acid,mg.	Salicylic Acid Found,mg.	% Recovery
375	125.0	125.0	100.0
375	117.5	115.0	98.72
375	139.4	140.7	101.08
375	137.1	138.0	100.66

ตารางที่ 7

Percentage Recovery for the Assay of a Mixture of Aspirin and
Salicylic Acid by Infrared Spectrophotometry using Q - Analysis Method

Mixture No.	Added Aspirin mg.	Absorbance at		Q $\frac{1657}{1677}$	Aspirin Found, mg.	% Recovery
		1657 cm. ⁻¹	1677 cm. ⁻¹			
1.	125	0.3849	0.4024	0.9564	124.59	99.67
2.	125	0.3742	0.3913	0.9563	124.00	99.69
3.	125	0.4058	0.4242	0.9566	124.54	99.64
4.	125	0.4069	0.4254	0.9565	124.57	99.65

Mixture No.	Added Salicylic Acid, mg.	Absorbance at		Q $\frac{1657}{1677}$	Salicylic Acid Found, mg.	% Recovery
		1657 cm. ⁻¹	1677 cm. ⁻¹			
1.	125	0.3838	0.3838	1.00	125	100.85
2.	125	0.3849	0.3849	1.00	125	100.85
3.	125	0.4301	0.4325	0.9945	121	99.81
4.	125	0.4230	0.4242	0.9972	122.75	100.32

ตารางที่ 8

Comparison of Aspirin Tablet Assay by Infrared Spectrophotometry Method and Official Method

Sample No.	BP.1973 Method		Infrared Method	
	Aspirin % Labeled Amount	Salicylic Acid เทียบถูก Standard	Aspirin % Labeled Amount	Salicylic Acid Found (mg)
1	100.75	จางกว่า	101.23	0.1993
2	100.61	จางกว่า	101.61	-
3	102.62	จางกว่า	100.71	-
4	104.78	จางกว่า	100.38	-
5	103.31	จางกว่า	103.61	-
6	100.57	จางกว่า	96.40	1.7923
7	101.25	จางกว่า	97.05	1.9518
8	102.42	จางกว่า	100.06	-
9	100.17	จางกว่า	97.20	0.4759
10	99.44	จางกว่า	98.25	0.4818

ตารางที่ ๙

Percentage Recovery of Aspirin for Aspirin Tablet by
Infrared Spectrophotometry Method and Official Method

Sample No.	Added Aspirin, mg		Aspirin Found, mg		% Recovery	
	BP	IR	BP	IR	BP	IR
1	257.0	126.1	260.5	126.48	101.36	100.30
2	246.3	125.9	255.9	126.48	103.09	100.46
3	251.0	125.6	255.6	123.35	101.83	98.21
4	254.3	124.9	256.7	119.40	100.94	95.60

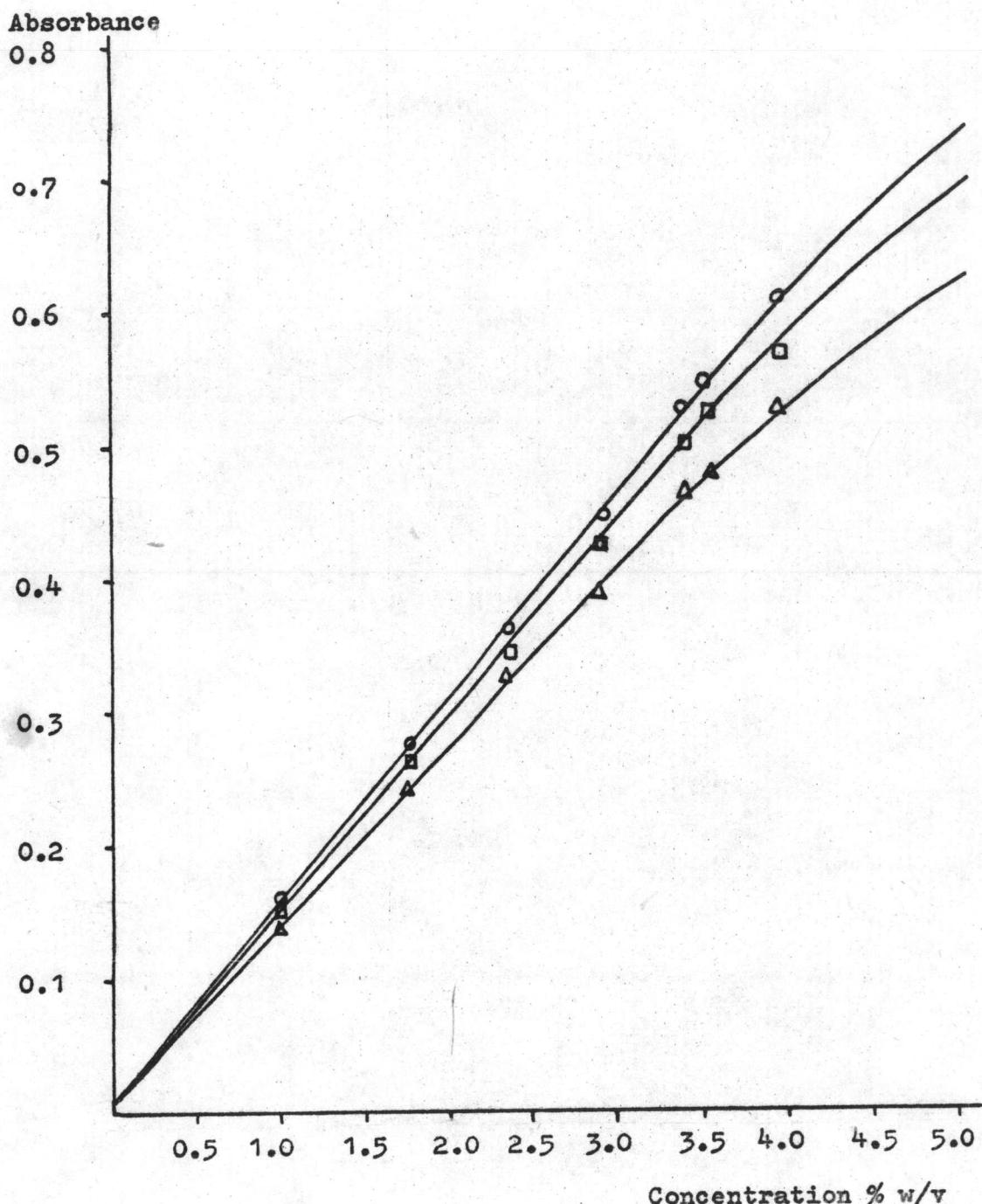
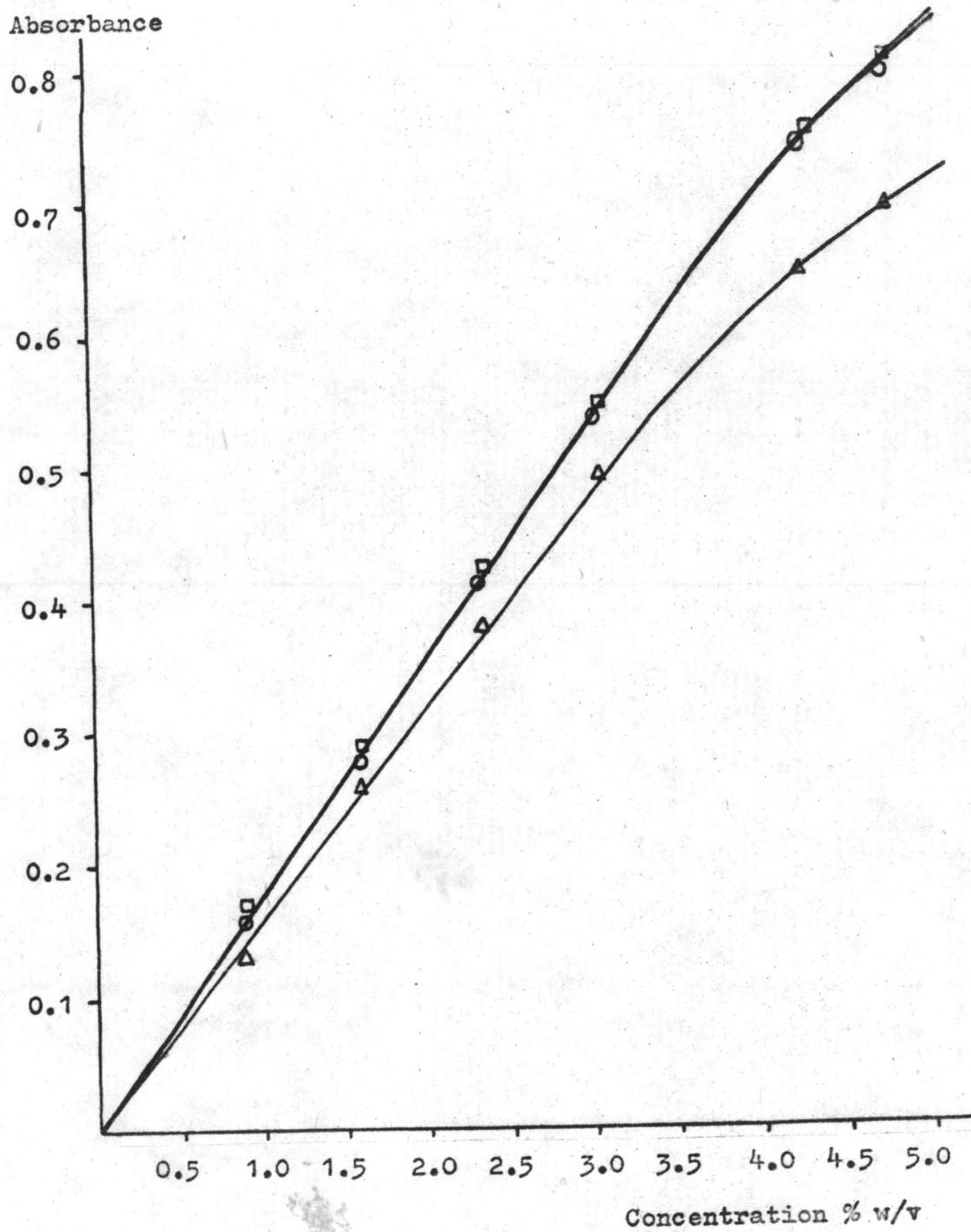


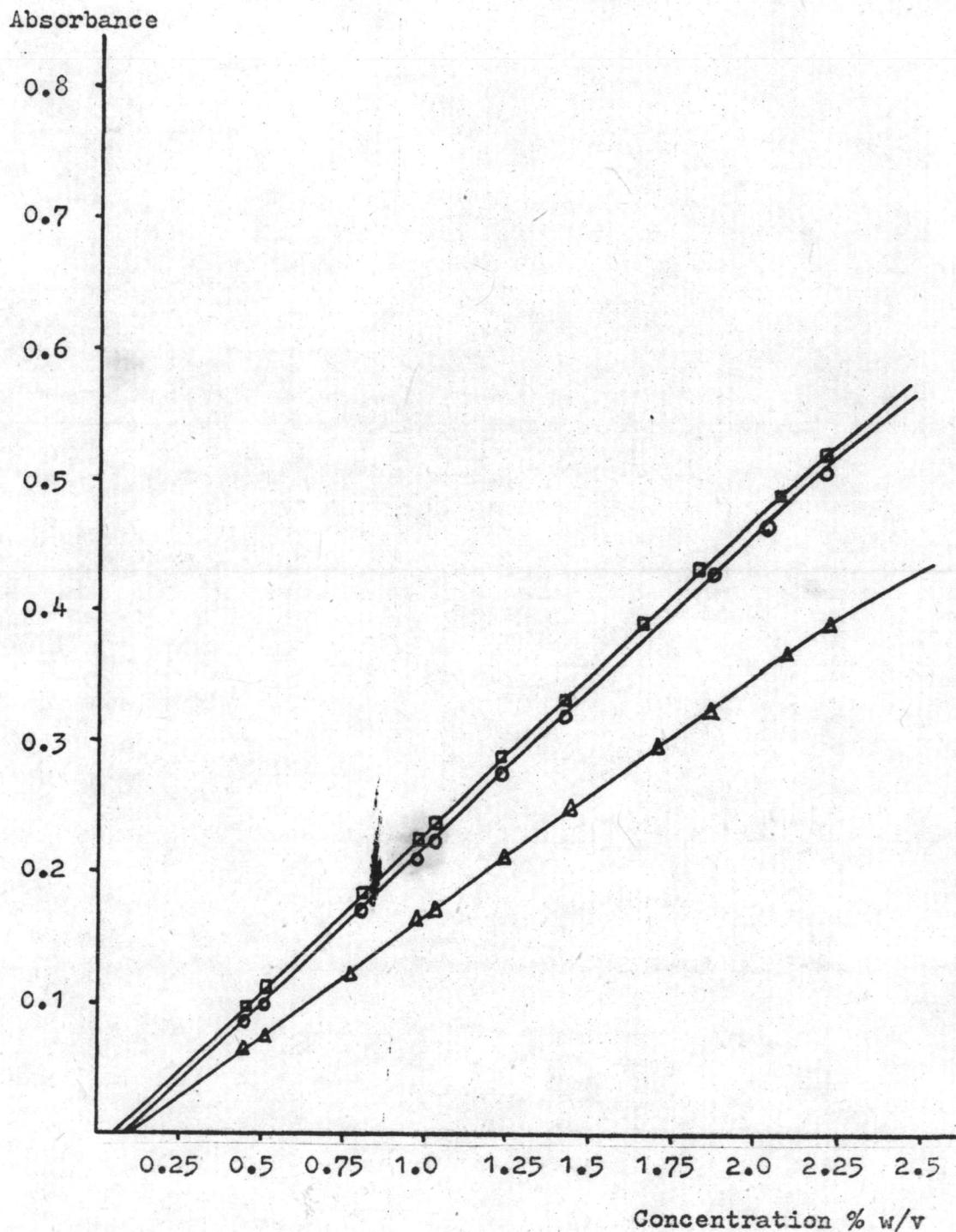
Fig 1 Calibration Curve of Standard Aspirin at 1750 cm^{-1}

Cell Pathlength 0.1 mm ○ = chloroform baseline

□ = baseline 1 △ = baseline 2



117 2 Calibration Curve of Standard Aspirin at 1690 cm^{-1}
 Cell Pathlength 0.1 mm ○ = chloroform baseline
 □ = baseline 1 △ = baseline 2



3 Calibration Curve of Standard Salicylic Acid at 1657 cm^{-1}

Cell Pathlength 0.1 mm

○ = chloroform baseline

□ = baseline 1

△ = baseline 2

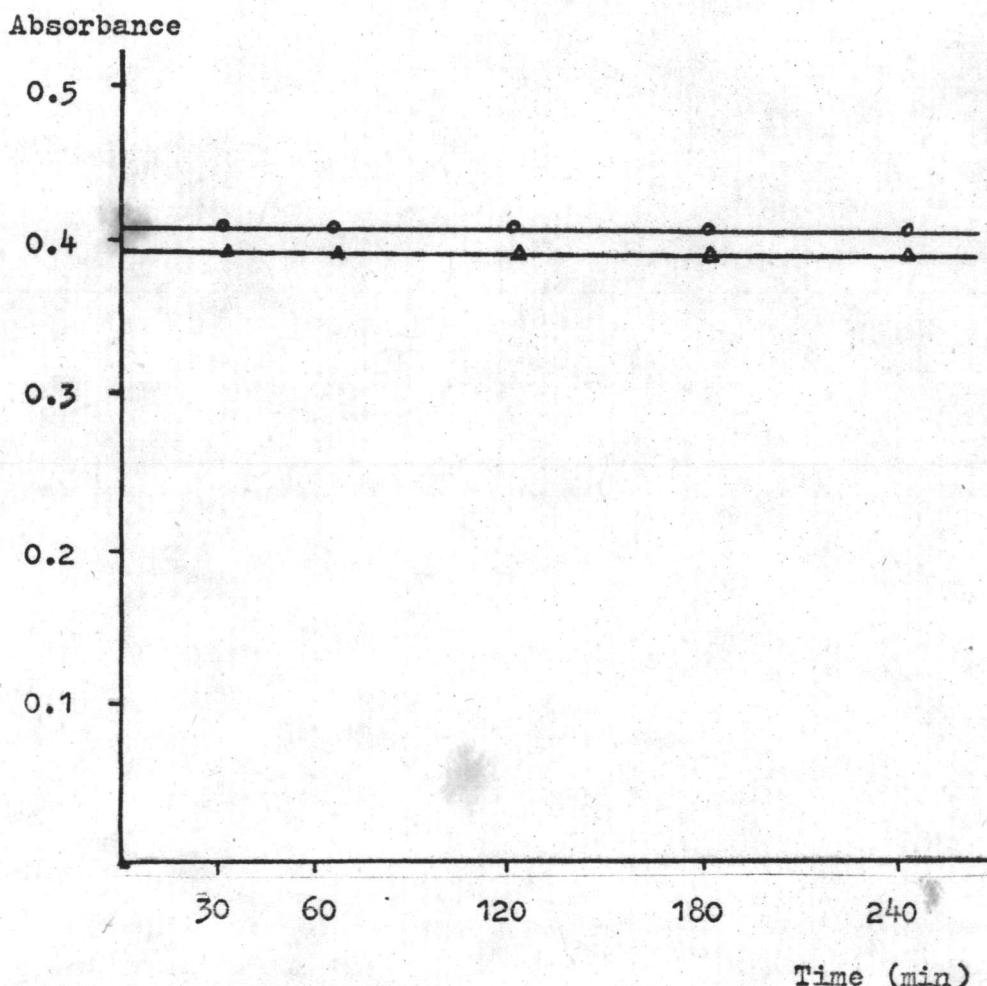


Fig 4 Effect of Time on Analysis of Aspirin and Salicylic Acid

\circ = Standard Aspirin 2.5 % Wavenumber 1750 cm^{-1}

Δ = Standard Salicylic Acid 1.5 % Wavenumber 1657 cm^{-1}

Chloroform baseline \approx cell pathlength 0.1 mm

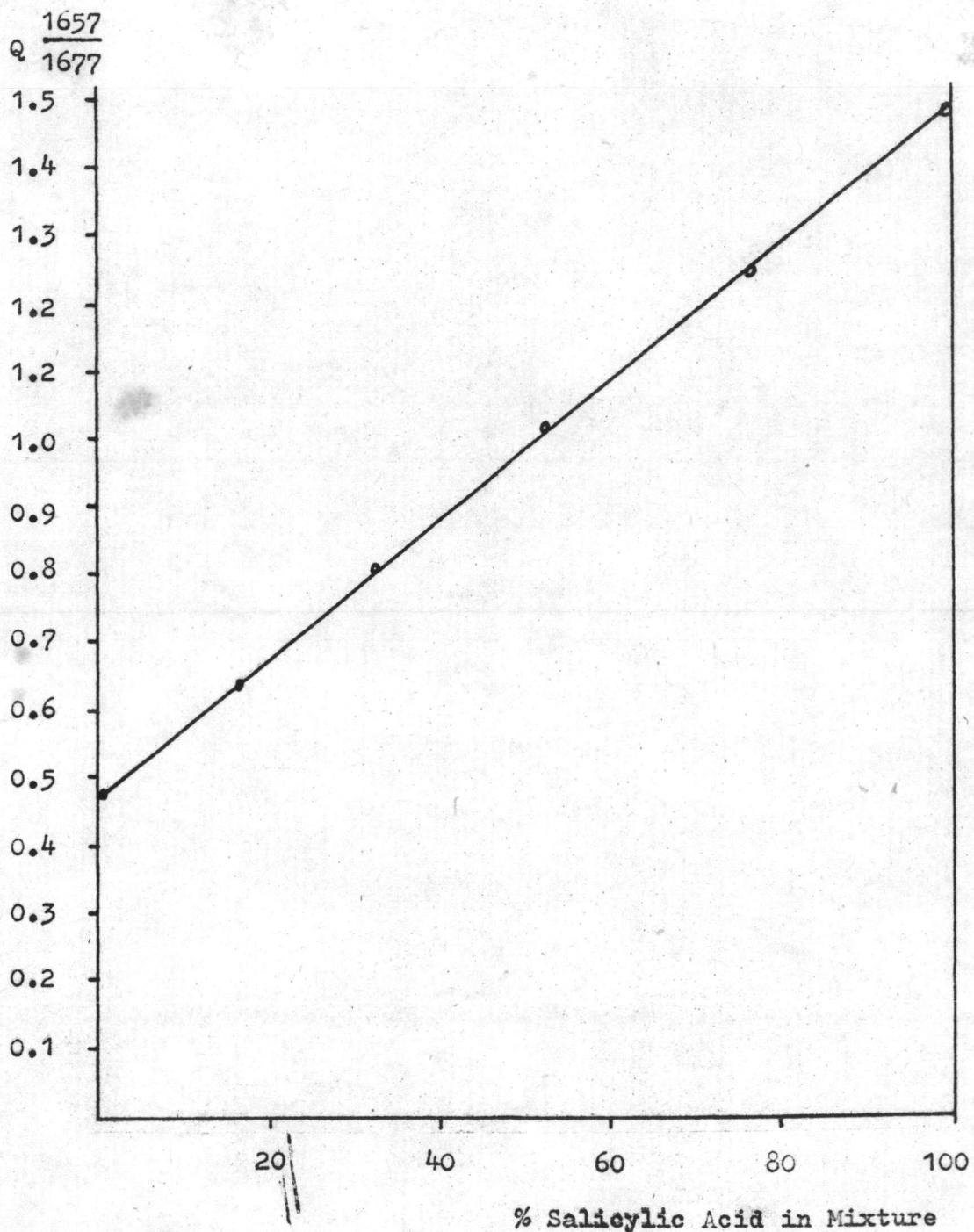


Fig 5 Q-Curve (Absorbancy Ratio Curve) of Aspirin and
Salicylic Acid in Binary Mixture.

ประวัติการศึกษา

ร.พ. นฤงค์ ศิริพงษ์ บุญธรรม
 วุฒิการศึกษา เกสต์ฟ้าสกอร์บันยินดี คณะ เกสต์ฟ้าสกอร์ มหาวิทยาลัยเชียงใหม่
 ปีการศึกษา 2515
 ตำแหน่งและสถานที่ทำงาน ประจำแผนก เกสต์ฟ้าสกอร์ กองวิจัยและគุฒนาครุภัณฑ์
 โรงงาน เกสต์ฟ้าสกอร์

