

表2 (つづき)

No.	分析対象化合物	0.1 ppm						0.01 ppm					
		牛肉			牛乳			牛肉			牛乳		
		真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)
51	Pyrimethamine	86	2	7	102	1	3	91	3	3	100	1	3
52	Robenidine	76	3	7	85	3	5	76	1	17	84	11	12
53	Sarafloxacin	52	8	17	73	5	10	55	12	30	59	10	12
54	Sulfabenzamide	66	6	9	92	2	3	62	6	12	87	2	7
55	Sulfabromomethazine	78	6	6	95	3	6	67	9	10	84	3	7
56	Sulfacetamide	25	14	25	82	4	5	- ^a	- ^a	- ^a	- ^a	- ^a	- ^a
57	Sulfachlorpyridazine	75	5	8	87	4	6	74	12	14	82	7	7
58	Sulfadiazine	61	5	8	41	4	16	38	18	24	38	6	66
59	Sulfadimethoxine	76	7	7	95	3	6	67	7	14	87	3	7
60	Sulfadimidine	72	3	6	88	3	7	70	6	7	78	5	9
61	Sulfadoxine	76	7	7	95	3	6	67	7	14	87	3	7
62	Sulfaethoxypyridazine	76	5	7	84	5	7	76	8	13	77	5	9
63	Sulfamerazine	81	4	7	90	4	11	75	7	8	79	5	9
64	Sulfamethoxazole	77	6	7	93	3	6	71	6	8	88	3	7
65	Sulfamethoxypyridazine	74	4	7	90	4	7	58	6	22	75	5	10
66	Sulfamonomethoxine	84	4	6	91	4	6	76	6	8	84	5	7
67	Sulfapyridine	76	2	5	157	4	13	64	6	7	92	5	17
68	Sulfaquinoxaline	77	5	7	80	9	10	72	7	9	77	5	6
69	Sulfathiazole	80	2	9	84	4	7	80	38	40	74	6	10
70	Sulfatroxazole	80	5	7	96	2	4	86	6	10	87	4	6
71	Temephos	71	7	13	99	3	14	57	4	8	94	6	11
72	Thiabendazole	91	1	8	106	2	3	83	4	4	104	9	9
73	Tiamulin	96	2	7	107	1	11	98	2	3	99	1	4
74	Tolfenamic acid	75	3	8	91	3	7	66	5	6	89	3	7
75	β-Trenbolone	89	1	7	102	2	6	73	2	3	96	2	5
76	Trichlorfon	71	2	10	82	1	5	60	7	7	78	5	17
77	Trimethoprim	83	1	6	121	2	6	80	3	4	94	2	12
78	Tripelethamine	98	3	14	92	2	8	104	5	6	93	4	6
79	Tylosin	90	5	8	89	10	17	89	9	17	83	16	17
80	Valnemulin	89	2	11	103	1	2	86	7	8	101	3	3
81	Warfarin	91	2	6	106	1	2	81	4	12	106	2	4
82	Xylazine	81	2	6	95	3	4	81	3	5	86	8	8

a) 感度不足

表3 真度・精度(マトリックス検量線法)

No.	分析対象化合物	0.1 ppm						0.01 ppm					
		牛肉			牛乳			牛肉			牛乳		
		真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)
1	2-Acetylamino-5-nitrothiazole	101	5	8	102	4	6	105	6	11	99	12	12
2	Albendazole	97	1	6	107	1	3	95	2	4	103	1	4
3	Altrenogest	85	3	8	87	3	4	95	2	5	86	4	6
4	Azaperone	110	2	19	101	2	8	114	2	3	108	4	8
5	Benzocaine	79	2	5	72	9	9	72	4	8	61	6	12
6	Bromacil	101	2	6	109	3	6	108	4	6	104	6	9
7	Brotizolam	101	1	6	105	2	4	107	1	4	104	2	6
8	Cefoperazone	77	7	11	102	6	6	- ^a	- ^a	- ^a	- ^a	- ^a	- ^a
9	Chlormadinone	110	3	11	109	2	3	117	7	8	107	4	7
10	Clostebol	100	2	7	108	1	3	106	4	6	105	2	7
11	Danofloxacin	82	6	19	71	6	16	62	9	11	52	22	25
12	Dexamethasone	103	2	7	108	1	6	112	8	8	104	6	9
13	Diaveridine	93	3	8	109	2	3	92	3	5	96	2	17
14	Dicyclanil	107	2	4	104	1	2	105	3	5	100	2	6
15	Difloxacin	101	1	14	94	2	7	86	3	7	75	14	14
16	Diflubenzuron	106	8	12	106	2	2	- ^a	- ^a	- ^a	- ^a	- ^a	- ^a
17	Emamectin B1a	98	2	6	100	1	4	94	7	13	90	9	13
18	Enrofloxacin	96	2	19	88	3	9	74	6	8	65	18	18
19	Ethopabate	104	2	5	111	2	3	110	3	3	104	2	5
20	Famphur	103	2	5	110	2	3	111	2	7	108	3	8
21	Flenobucarb	69	8	10	64	6	6	81	9	9	63	10	15
22	Flubendazole	103	1	5	112	1	2	109	1	5	107	1	5
23	Flumequine	100	4	8	103	1	2	95	6	8	94	9	9
24	Flunixin	95	2	7	107	1	2	99	3	6	104	2	6
25	Halofuginone	78	5	8	100	2	4	67	7	7	79	8	18
26	Hydrocortisone	100	2	4	111	2	2	60	18	25	107	3	5
27	5-Hydroxythiabendazole	82	6	7	106	2	5	49	20	38	103	4	4
28	Ketoprofen	98	2	7	107	1	2	107	2	8	107	4	9
29	Levamisole	99	2	6	106	1	3	100	4	4	99	2	5
30	Lincomycin	88	5	15	107	1	2	78	5	7	96	6	10
31	Mafofrazine	107	1	8	111	1	3	106	2	5	107	1	6
32	Marbofloxacin	82	3	19	79	8	15	70	7	10	58	20	29
33	Mebendazole	105	1	7	111	1	3	109	1	6	108	1	8
34	Meloxicam	90	2	6	90	3	3	90	3	8	91	2	11
35	Menbutone	100	2	9	107	1	3	106	8	8	107	2	12
36	Methylprednisolone	101	3	7	106	3	4	115	6	7	109	4	6
37	Miloxacin	92	11	17	86	6	16	85	5	14	90	15	18
38	Morantel	72	2	8	74	1	3	76	3	3	74	4	9
39	Nalidixic acid	99	2	7	102	2	4	94	7	8	89	10	10
40	Ofloxacin	85	4	19	79	7	14	65	5	8	59	21	27
41	Olaquinox	38	8	22	45	9	10	37	26	30	41	14	24
42	Orbifloxacin	92	2	10	100	2	4	77	3	7	78	8	9
43	Ormetoprim	98	2	10	107	1	5	96	2	4	102	2	4
44	Oxibendazole	104	1	7	109	1	2	104	1	3	106	1	6
45	Oxolinic acid	98	7	13	91	4	7	91	7	11	80	8	19
46	Phenoxymethylpenicillin	92	3	10	82	10	10	- ^a	- ^a	- ^a	- ^a	- ^a	- ^a
47	Praziquantel	103	2	6	109	1	2	109	2	5	107	2	9
48	Prednisolone	102	4	9	110	3	4	110	4	6	107	3	6
49	Prifinium bromide	95	2	5	94	1	3	96	2	5	92	1	8
50	Pyrantel	85	1	7	88	1	3	86	2	2	87	2	7

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No.	分析対象化合物	0.1 ppm						0.01 ppm					
		牛肉			牛乳			牛肉			牛乳		
		真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)	真度 (%)	併行精度 (RSD%)	室内精度 (RSD%)
51	Pyrimethamine	97	2	11	107	1	2	97	1	2	104	1	5
52	Robenidine	79	7	9	89	4	7	70	3	12	84	12	12
53	Sarafloxacin	80	6	10	85	4	8	70	5	20	72	13	13
54	Sulfabenzamide	81	5	12	95	3	4	88	3	12	87	1	11
55	Sulfabromomethazine	86	5	7	95	3	5	84	7	12	87	4	11
56	Sulfacetamide	87	6	14	97	5	7	.. ^a	.. ^a	.. ^a	.. ^a	.. ^a	.. ^a
57	Sulfachlorpyridazine	83	4	10	91	4	9	87	3	7	77	6	18
58	Sulfadiazine	86	3	6	90	3	15	88	3	9	72	2	33
59	Sulfadimethoxine	88	4	6	97	2	5	87	4	8	86	5	10
60	Sulfadimidine	88	3	7	89	4	8	88	4	4	75	7	12
61	Sulfadoxine	88	4	6	97	2	5	87	4	8	86	5	10
62	Sulfaethoxypyridazine	85	2	7	85	5	8	84	4	8	73	7	12
63	Sulfamerazine	88	3	6	92	5	9	89	3	6	77	5	12
64	Sulfamethoxazole	89	4	7	98	2	5	87	3	8	85	4	6
65	Sulfamethoxypyridazine	89	2	6	83	4	9	88	4	6	73	7	9
66	Sulfamonomethoxine	93	4	6	95	3	8	89	5	8	81	6	6
67	Sulfapyridine	91	3	5	87	4	8	87	5	5	75	9	19
68	Sulfaquinoxaline	86	4	6	85	7	9	85	4	12	75	7	7
69	Sulfathiazole	80	4	11	88	4	7	78	16	36	72	6	9
70	Sulfatroxazole	88	4	7	97	3	5	91	4	10	85	4	8
71	Temephos	77	5	11	67	3	11	68	4	18	72	7	25
72	Thiabendazole	99	1	6	105	2	2	103	1	3	98	1	7
73	Tiamulin	100	1	5	104	1	3	106	1	6	100	1	4
74	Tolfenamic acid	91	6	11	97	2	5	91	3	9	94	1	5
75	β-Trenbolone	94	1	5	98	3	3	103	3	5	96	3	4
76	Trichlorfon	102	2	5	105	2	3	105	3	3	95	4	13
77	Trimethoprim	96	2	7	106	1	4	96	2	4	100	2	6
78	Tripeleminamine	100	2	12	89	1	6	101	3	3	94	5	8
79	Tylosin	83	5	11	85	5	7	88	8	12	83	13	16
80	Valnemulin	90	3	7	103	2	2	86	3	5	99	4	4
81	Warfarin	100	2	7	109	1	3	111	3	7	104	2	4
82	Xylazine	90	2	5	93	3	3	86	6	6	86	9	9

a) 感度不足

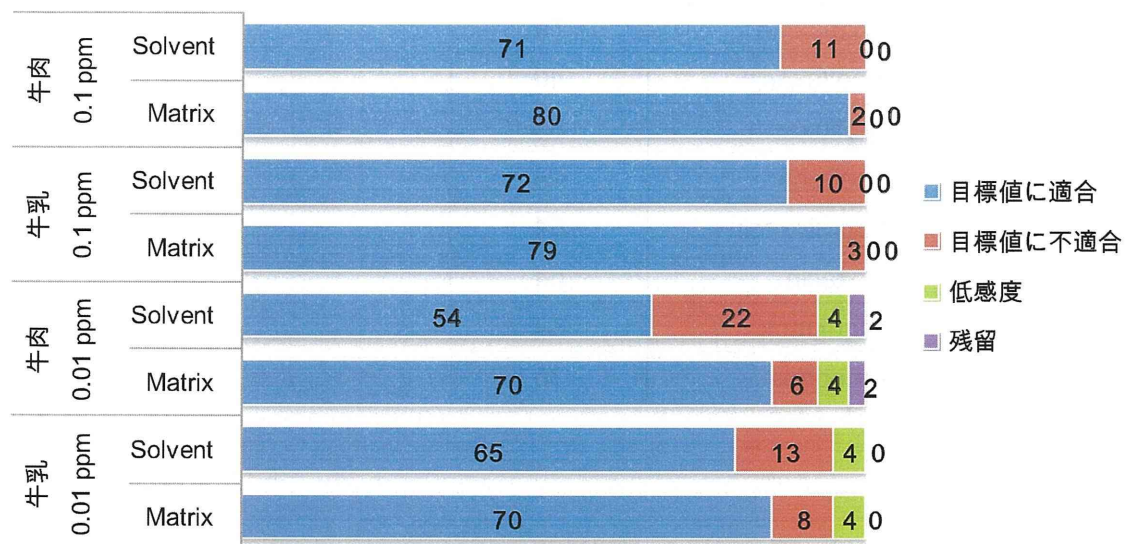


図2 妥当性評価試験結果の概要

(数字は化合物数)

Solvent: 溶媒検量線法により定量、Matrix:マトリックス検量線法により定量

目標値(真度・精度): 添加濃度 0.1 ppm: 真度 70~120%、併行精度 RSD 25%未満、室内精度 RSD 30%未満、添加濃度 0.01 ppm: 真度 70~120%、併行精度 RSD 15%未満、室内精度 RSD 20%未満

表 4 牛乳マトリックス存在下での ODS ミニカラムからの回収率

化合物	回収率 (%)
Benzocaine	70
Danofloxacin	40
Enrofloxacin	60
Fenobucarb	46
Marbofloxacin	64
Ofloxacin	64
Olaquinox	54
Temephos	36

図 3-1 検量線(高濃度)

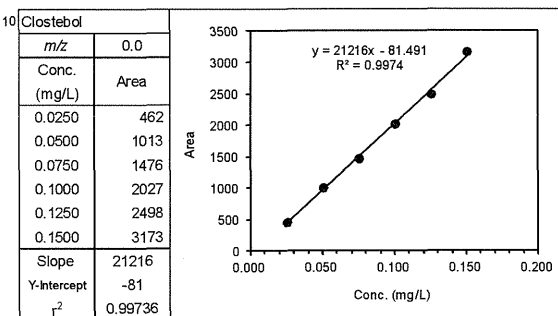
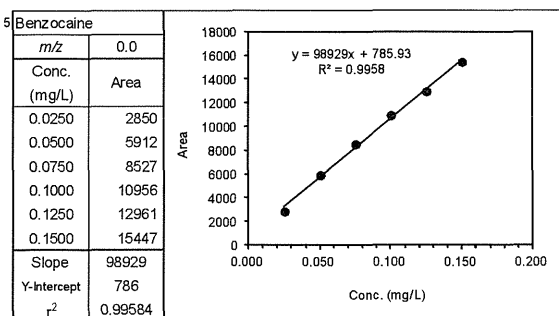
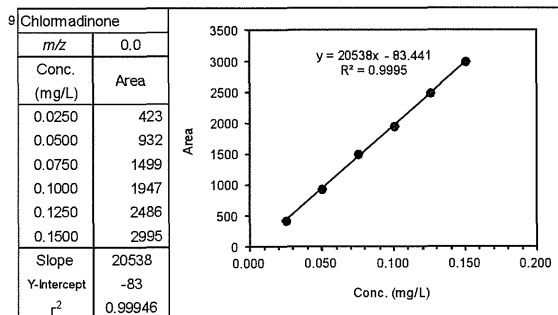
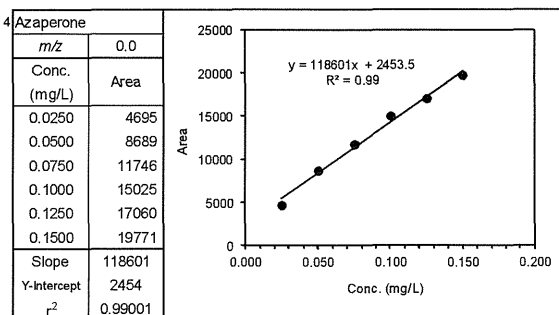
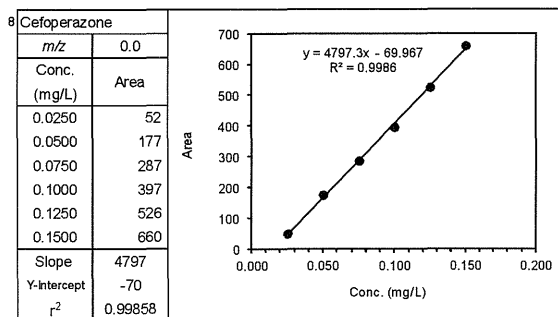
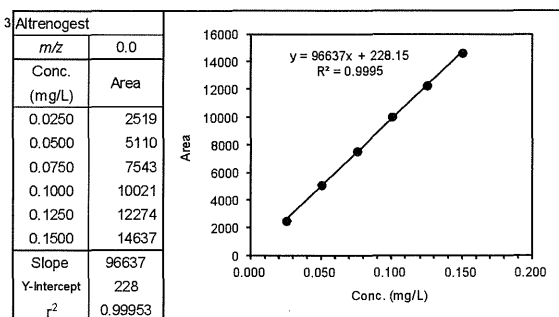
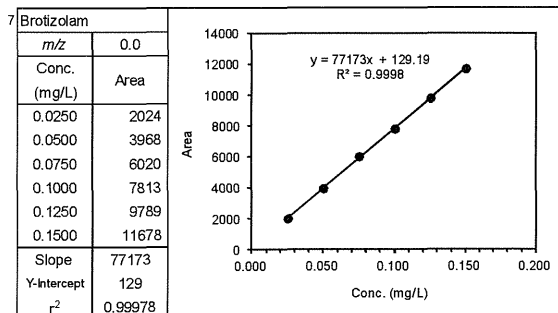
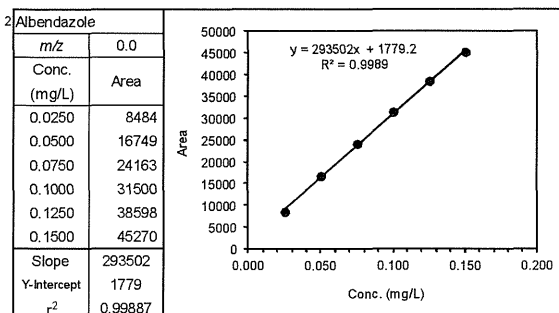
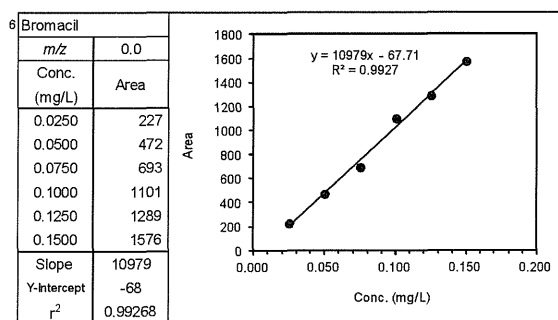
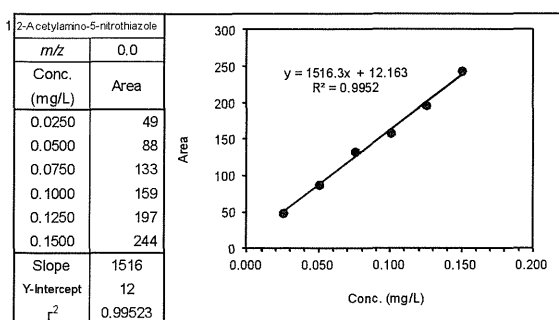
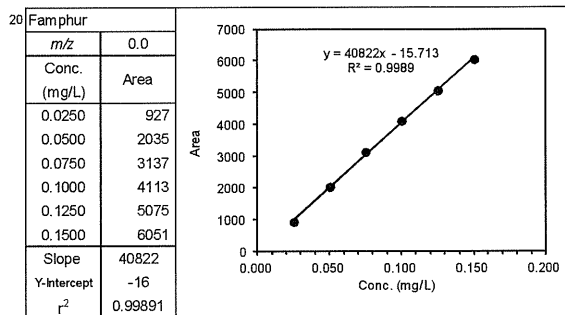
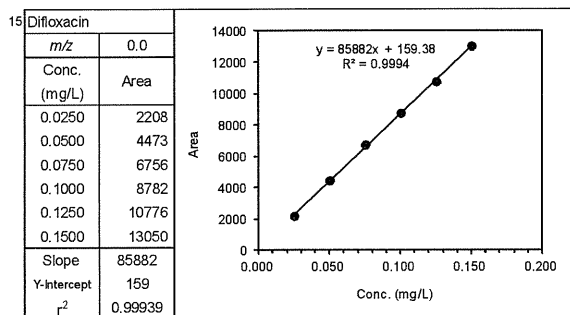
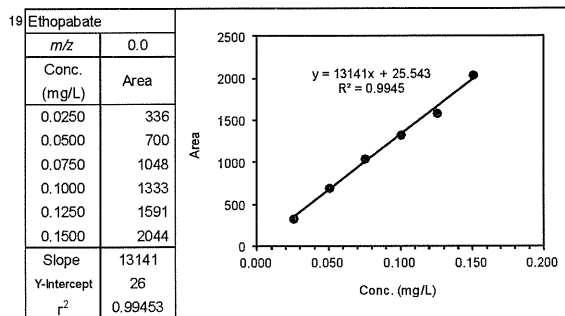
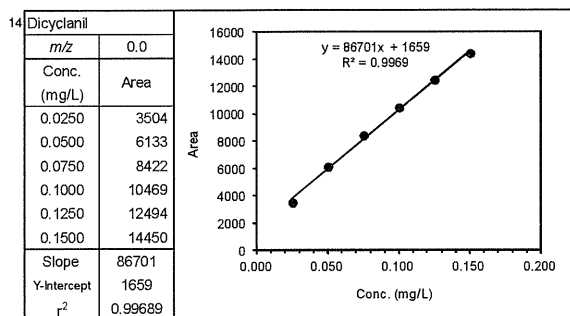
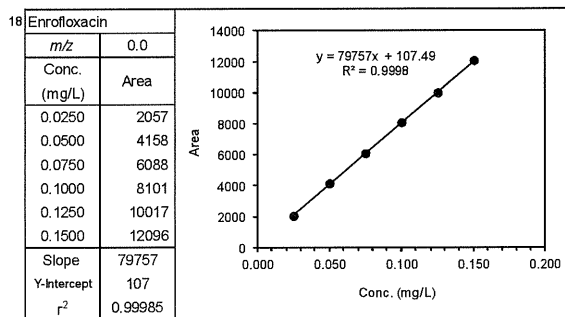
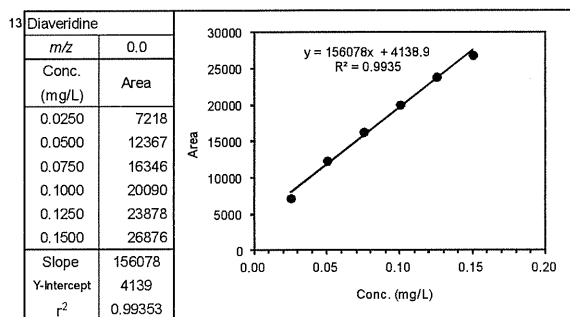
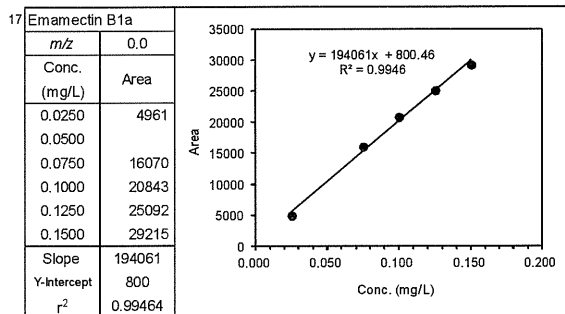
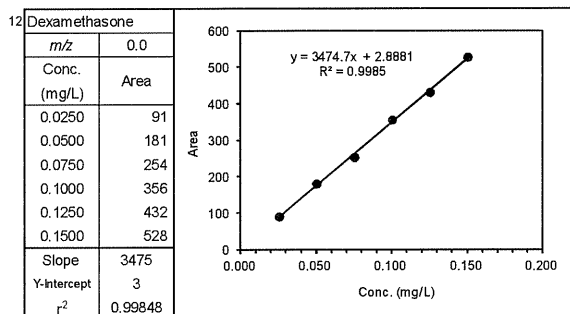
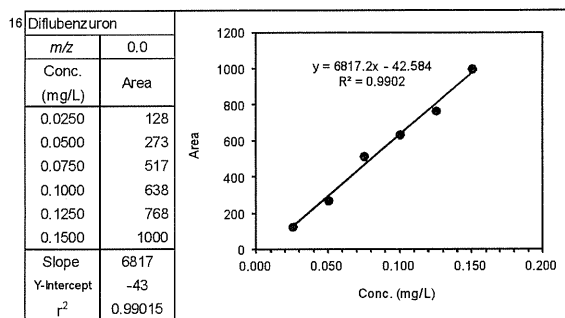
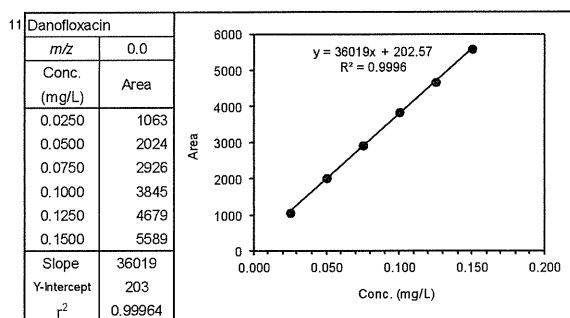


図 3-1 (つづき)



3-1 (つづき)

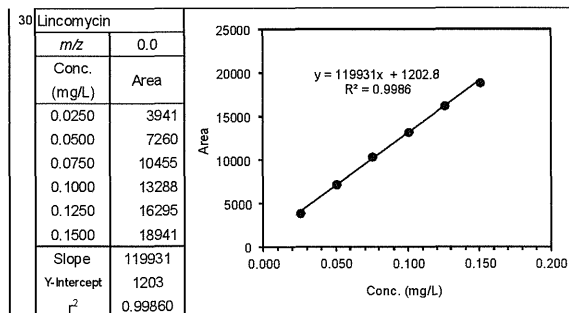
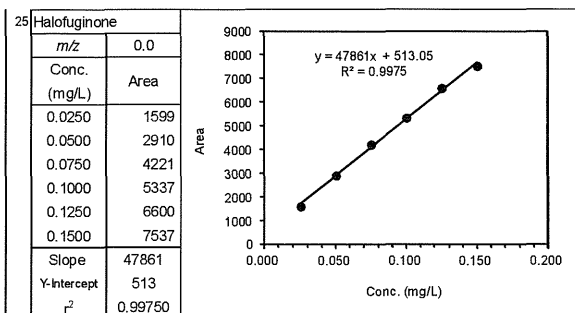
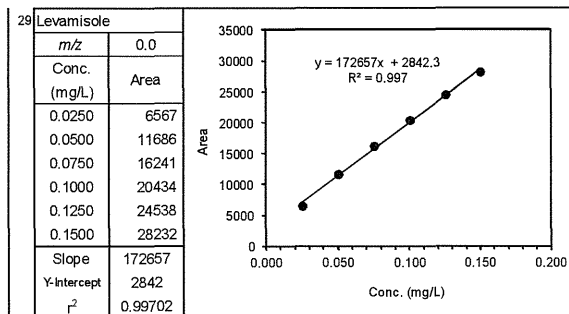
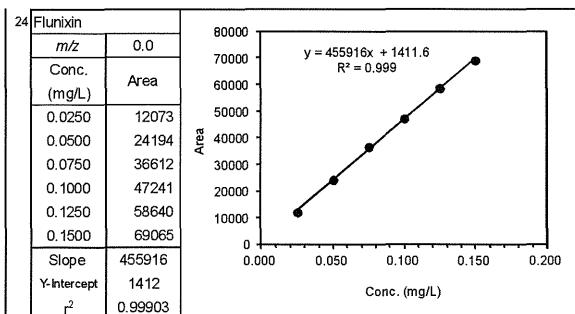
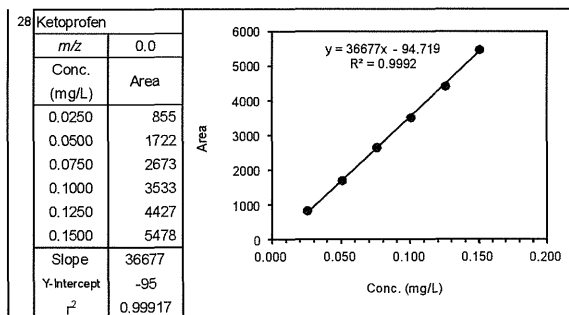
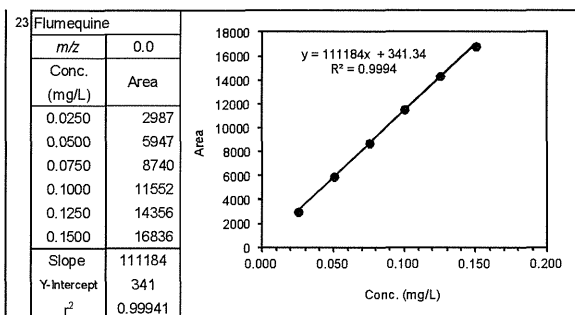
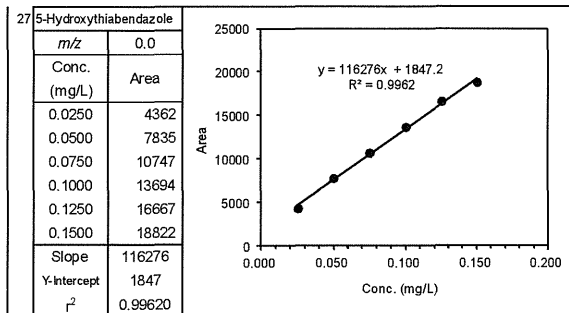
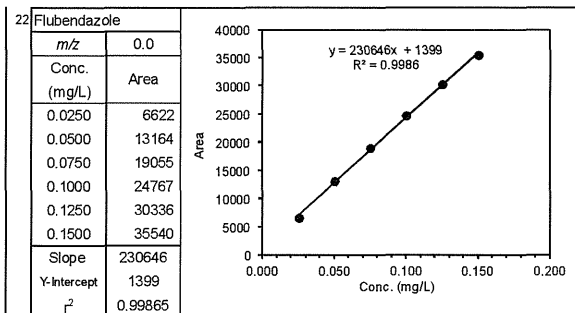
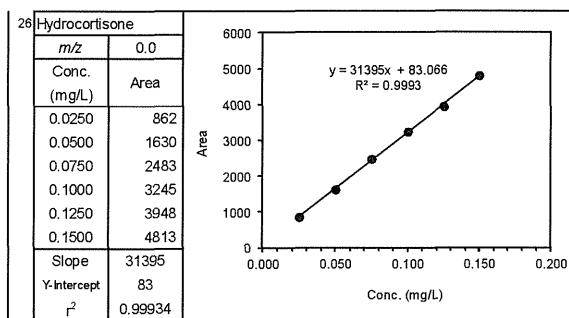
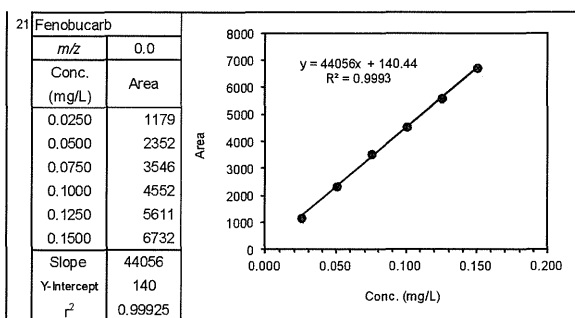
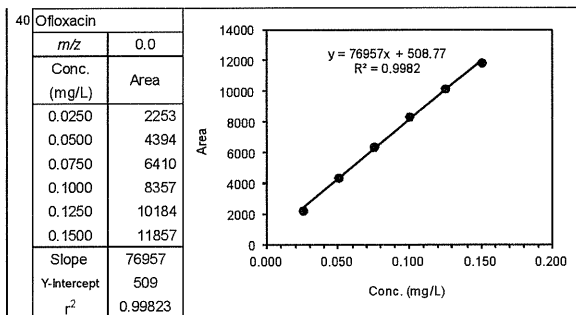
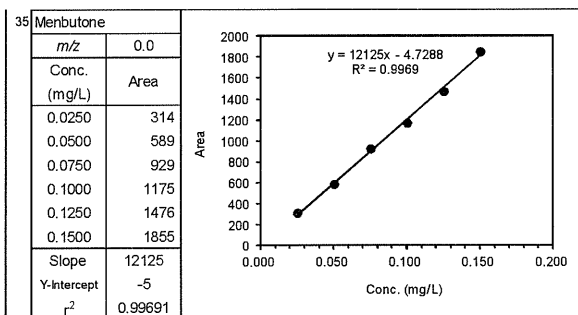
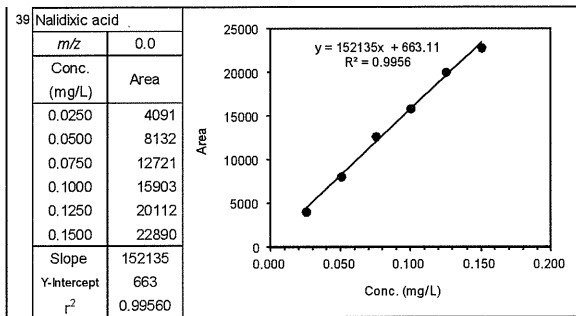
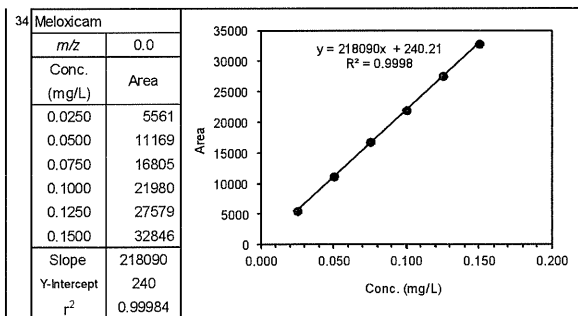
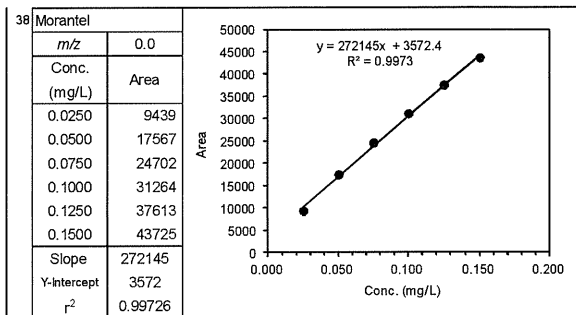
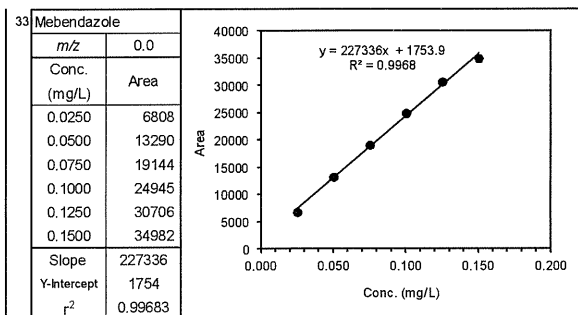
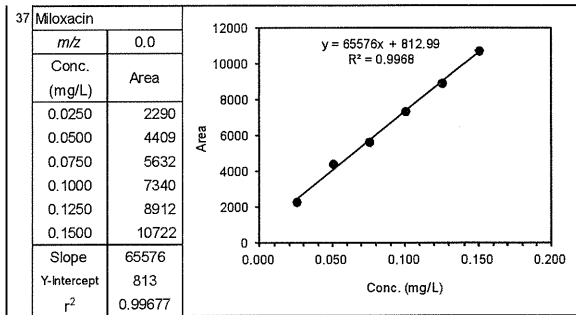
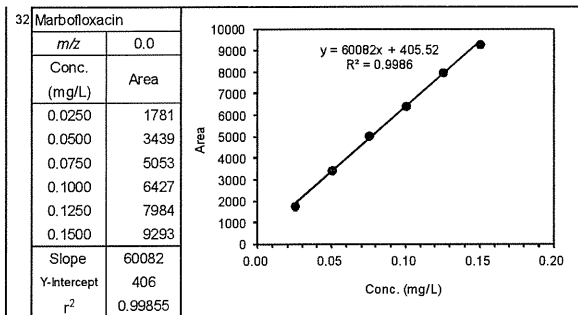
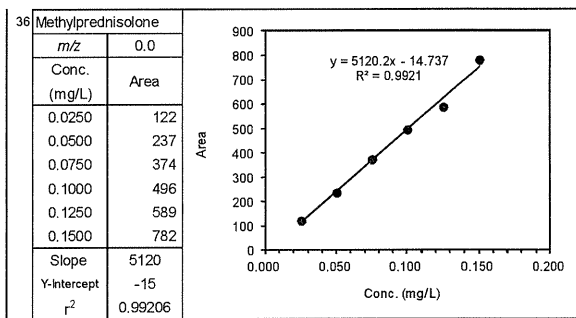
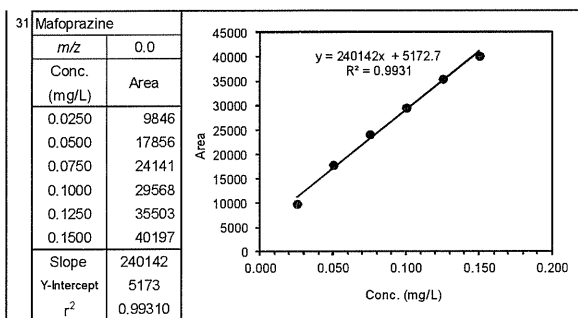
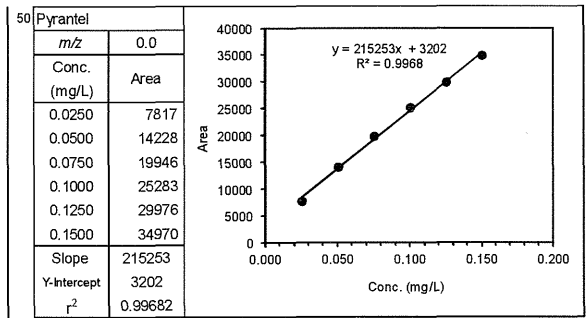
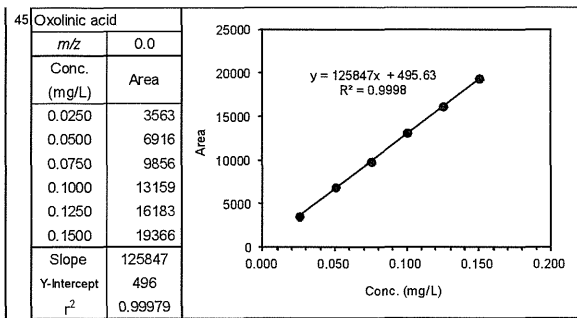
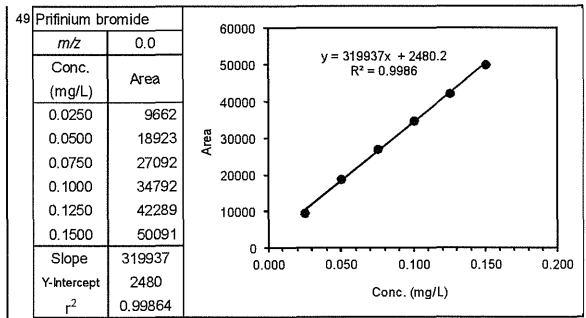
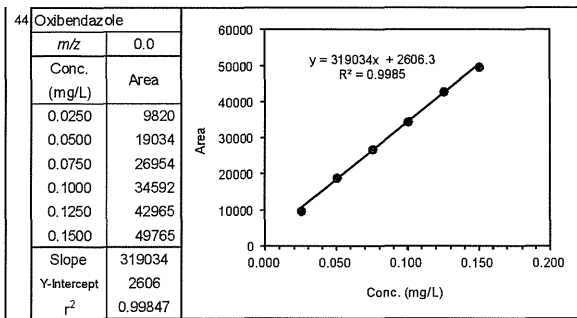
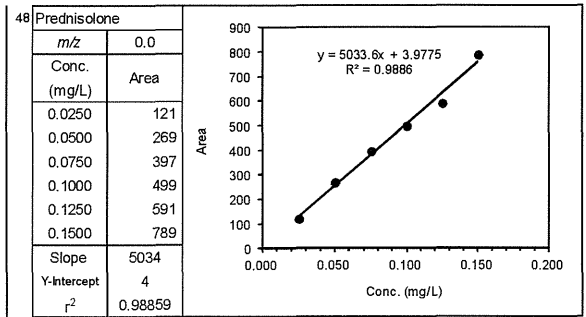
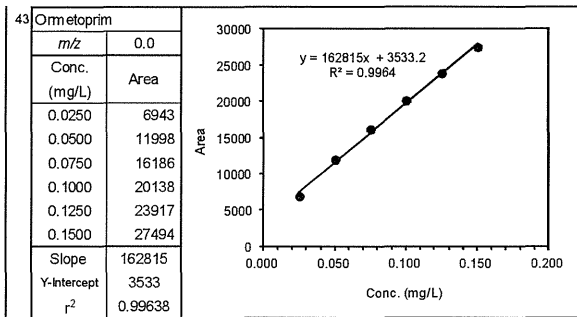
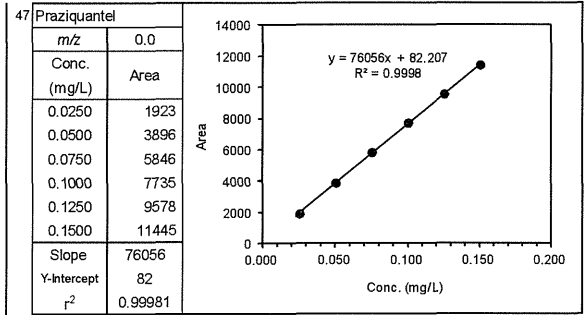
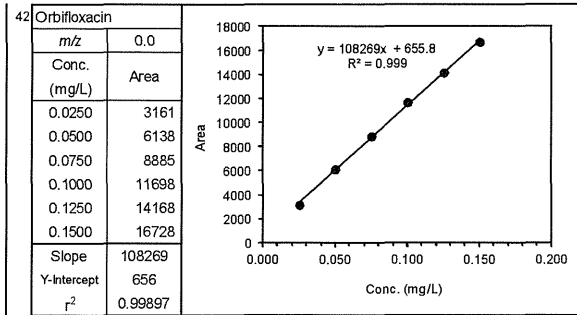
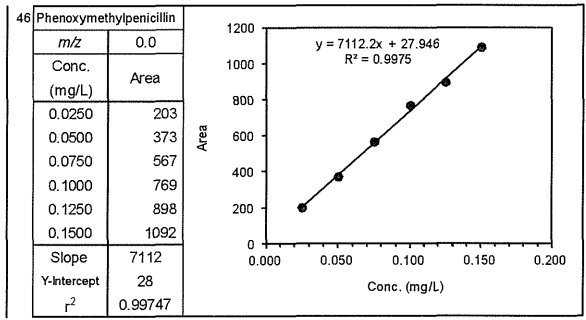
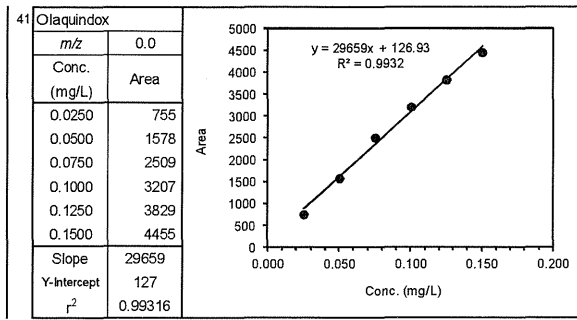


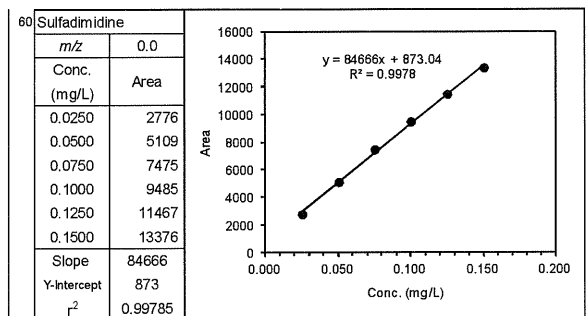
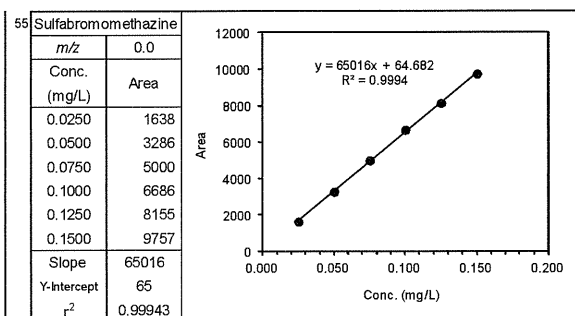
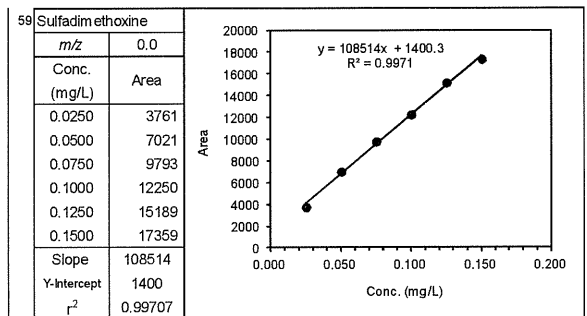
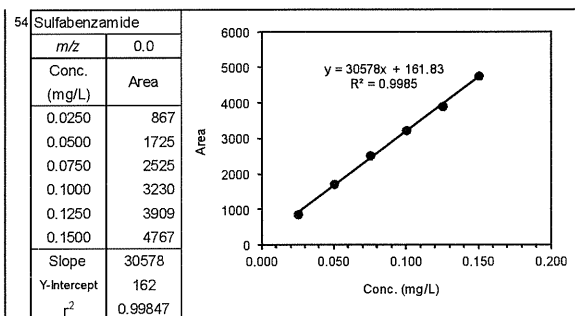
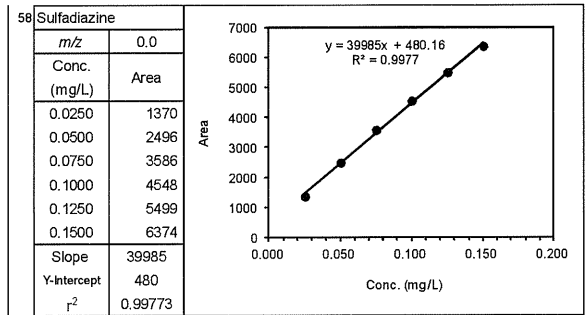
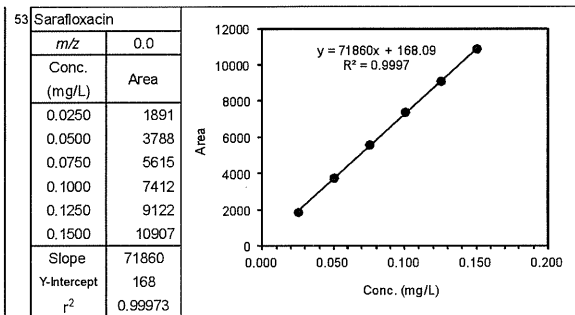
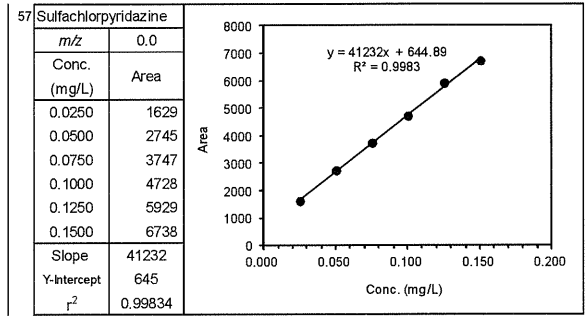
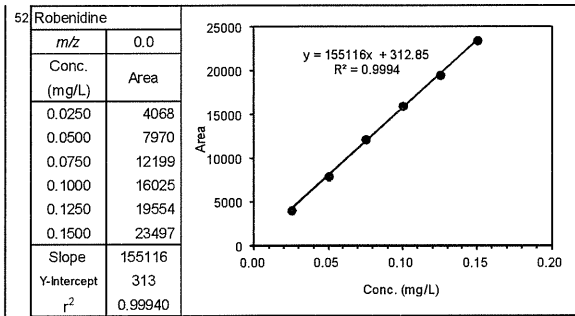
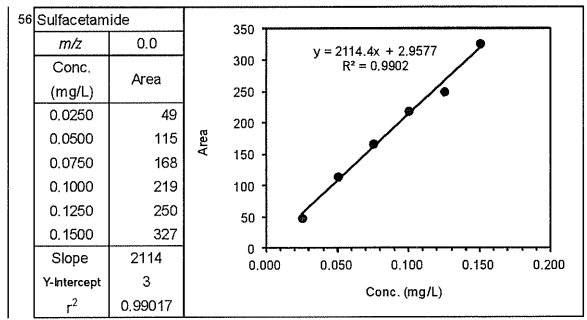
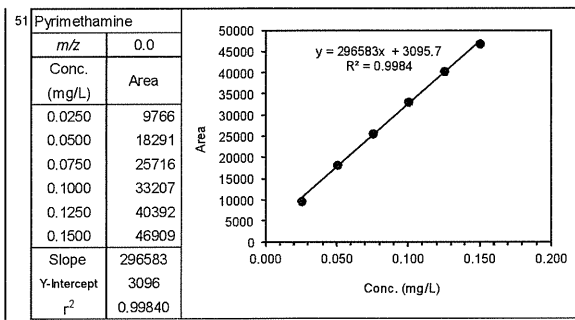
図 3-1 (つづき)



☒ 3-1 (つづき)



☒ 3-1 (つづき)



☒ 3-1 (つづき)

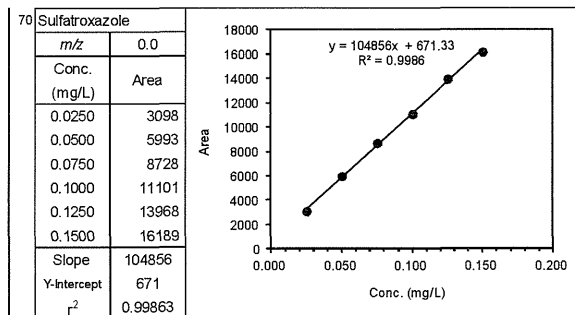
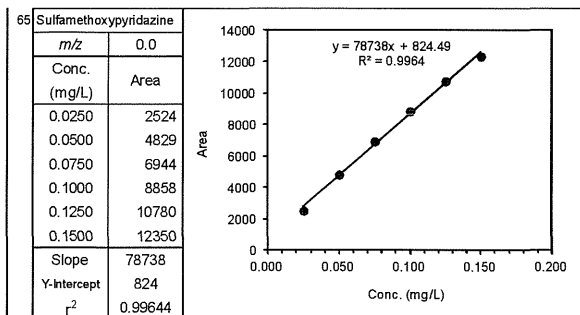
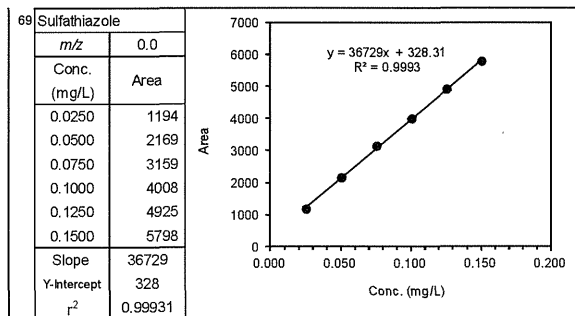
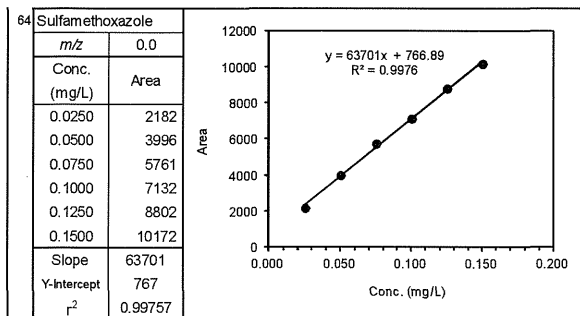
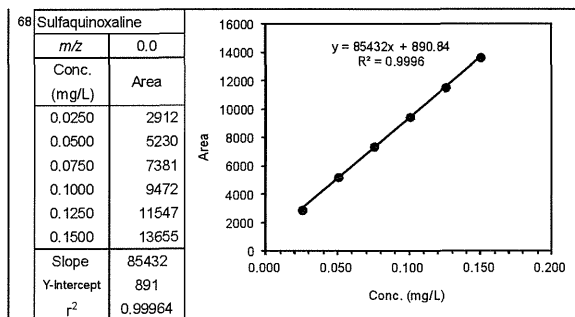
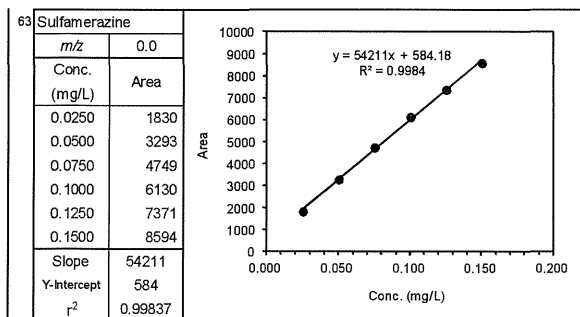
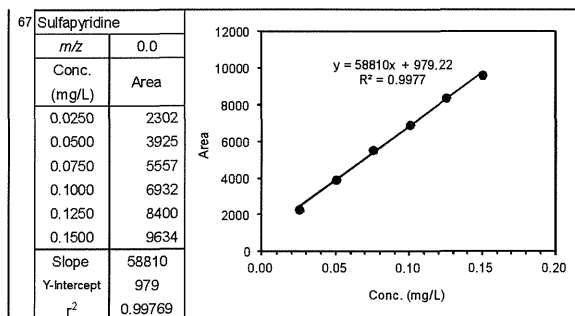
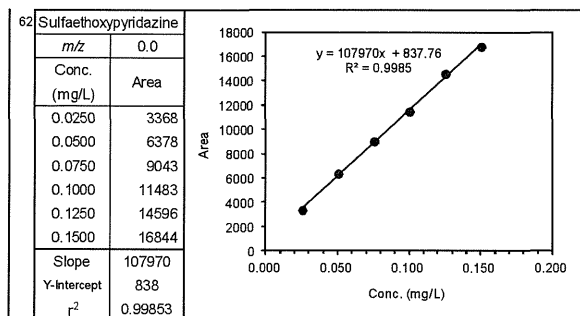
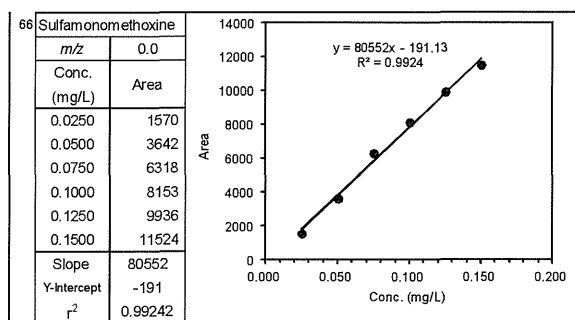
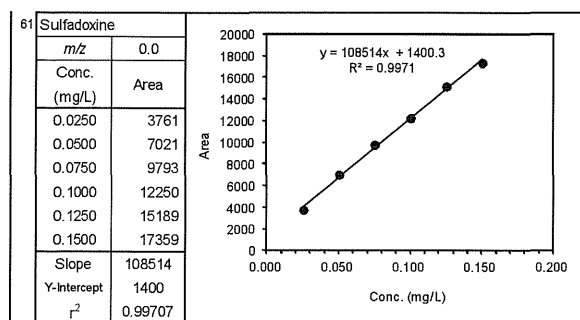


図 3-1 (つづき)

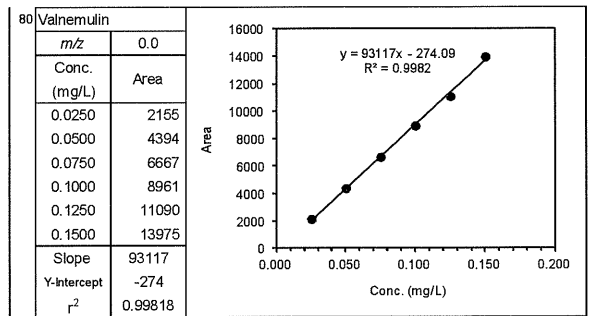
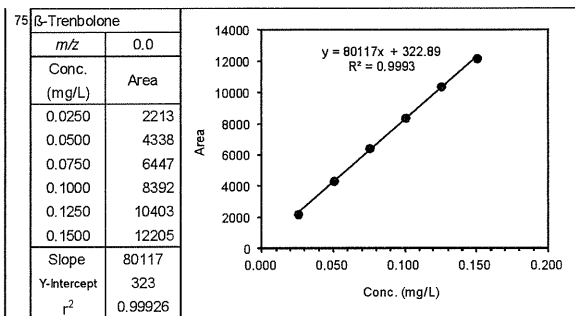
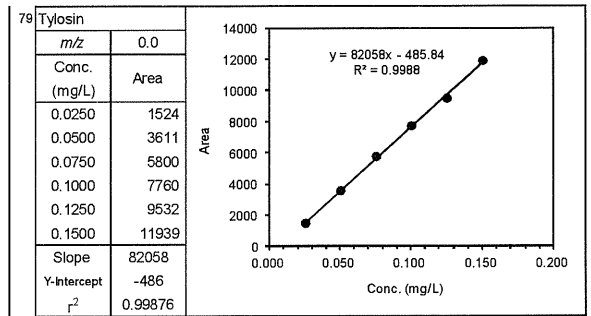
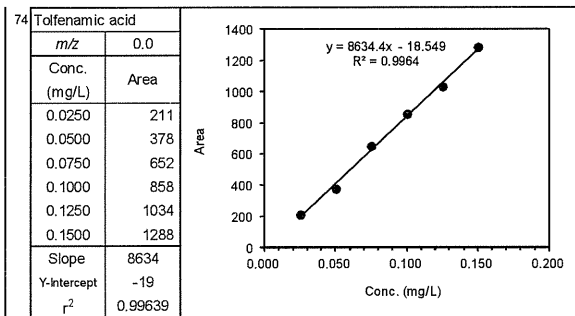
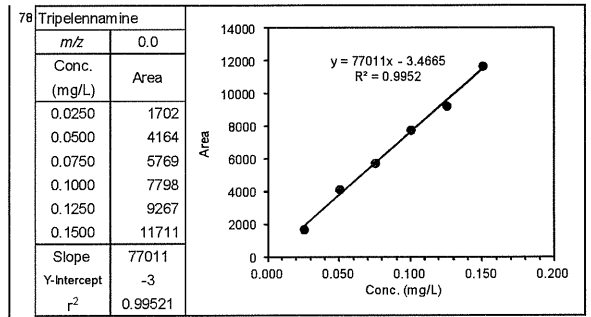
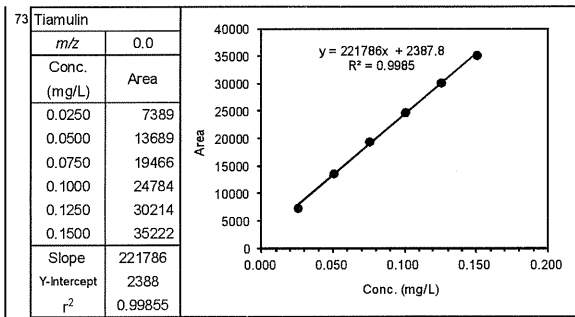
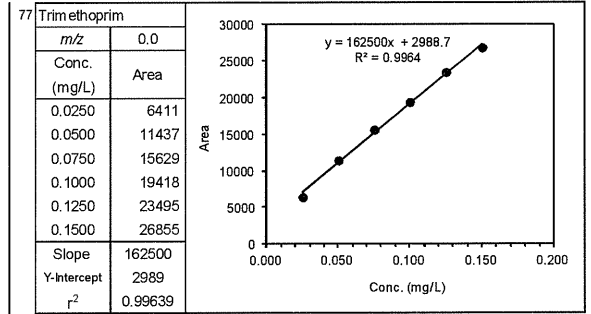
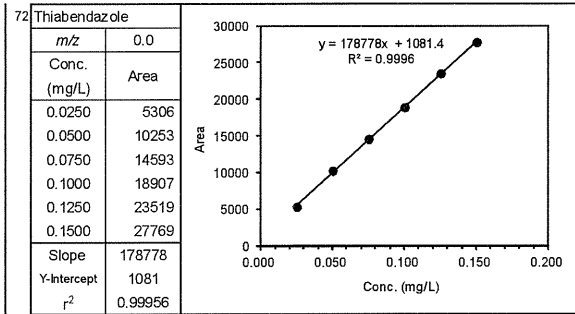
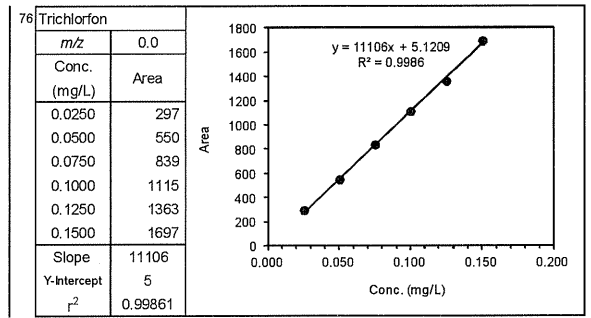
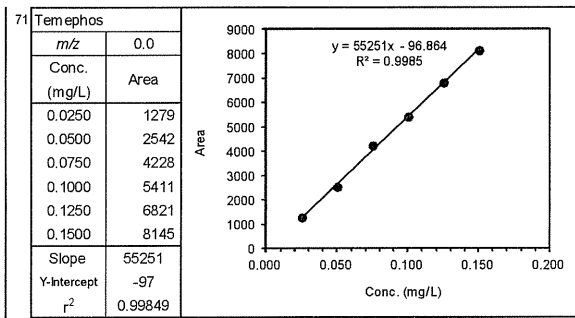


図 3-1 (つづき)

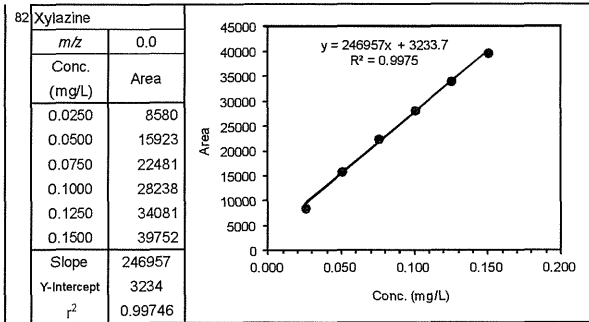
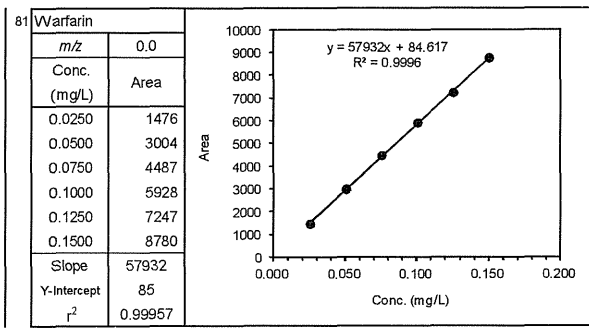


図 3-2 検量線(低濃度)

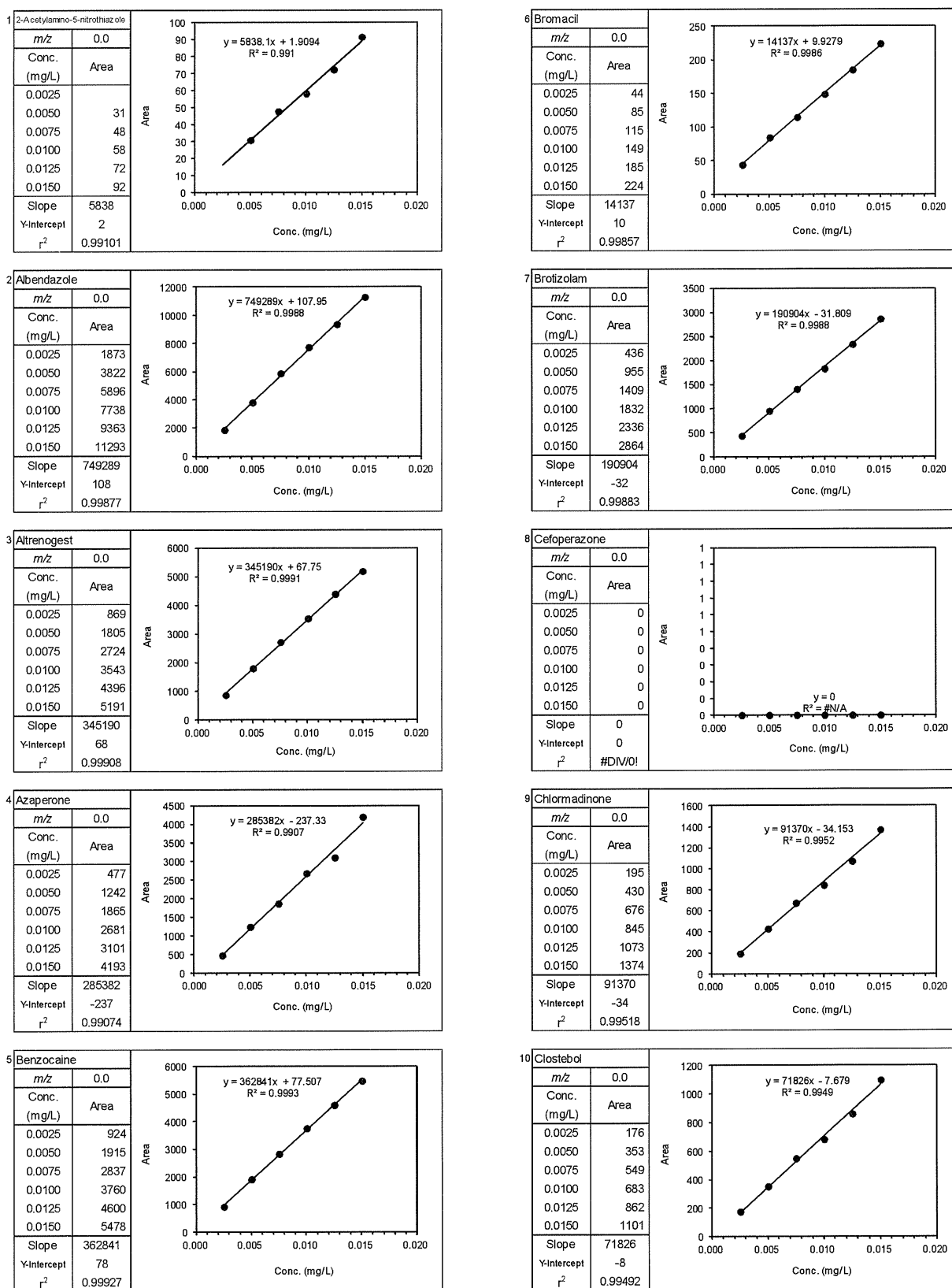


図 3-2 (つづき)

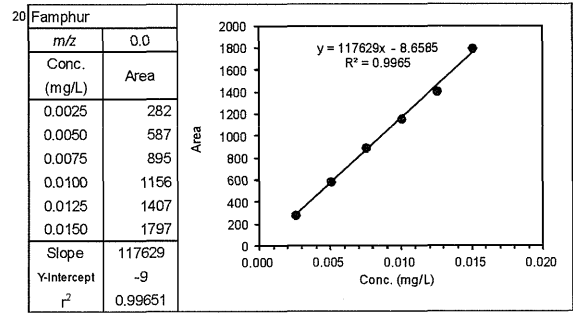
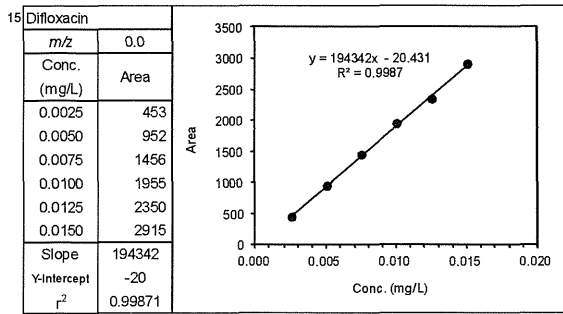
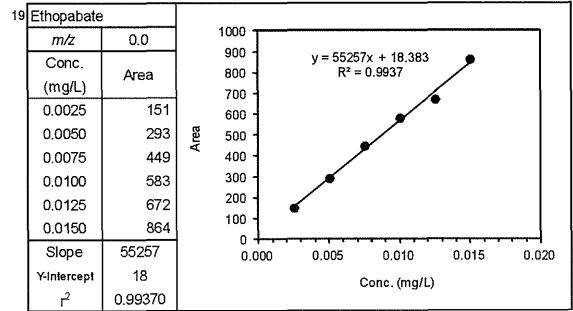
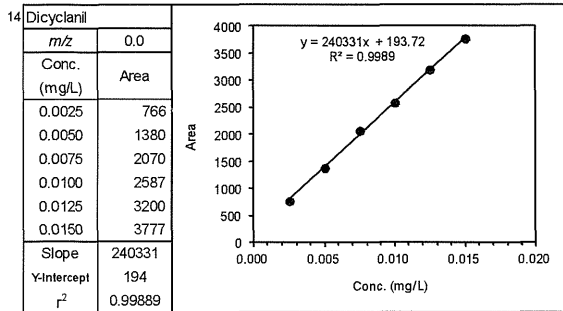
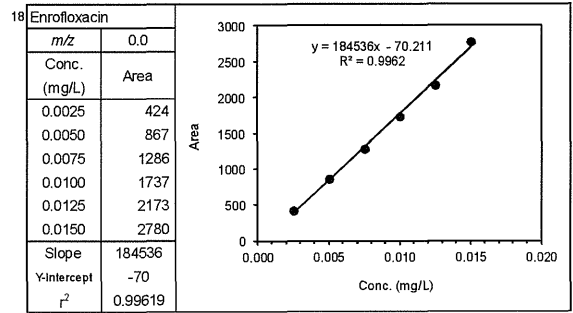
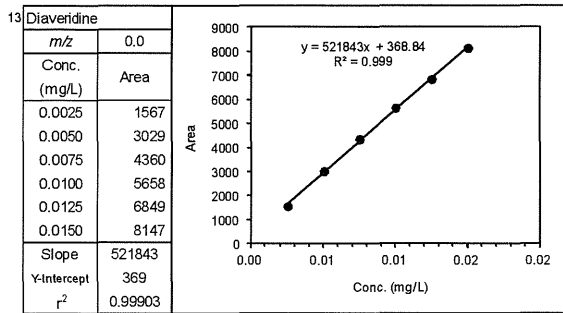
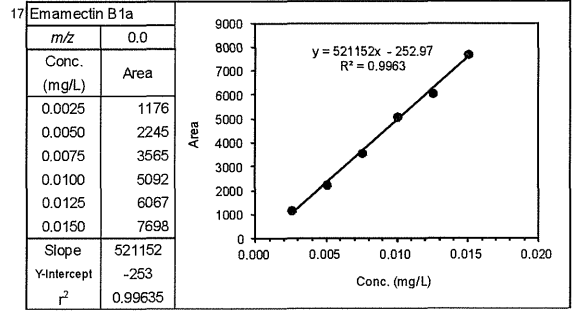
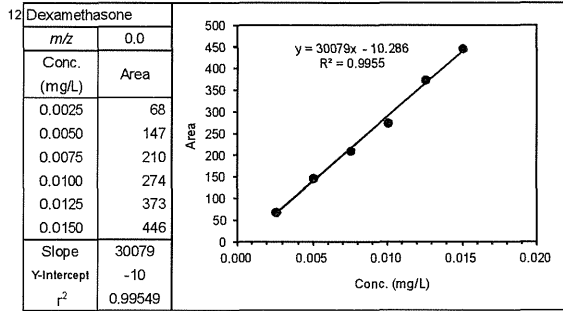
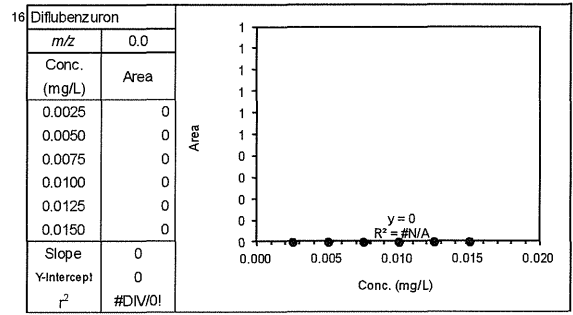
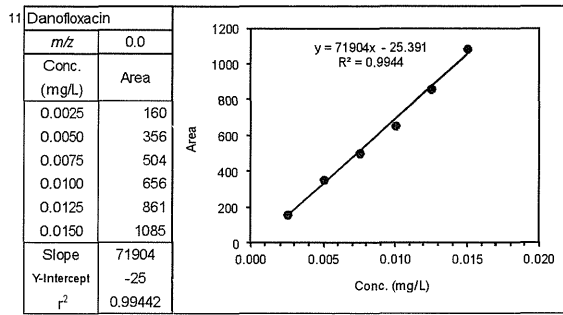
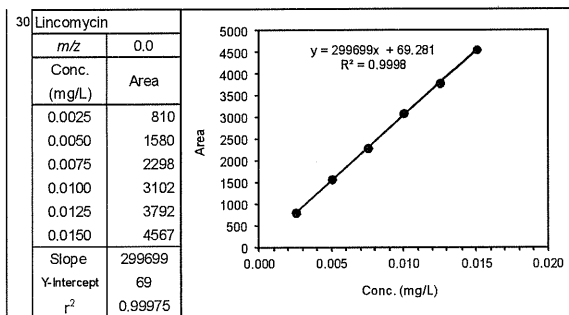
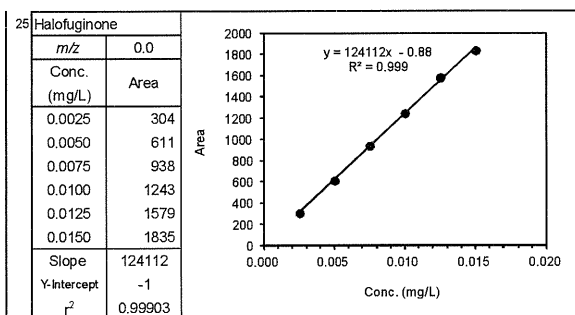
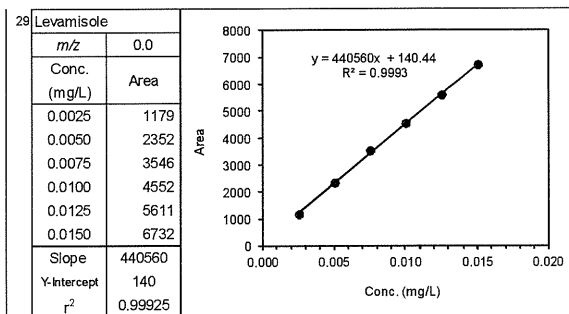
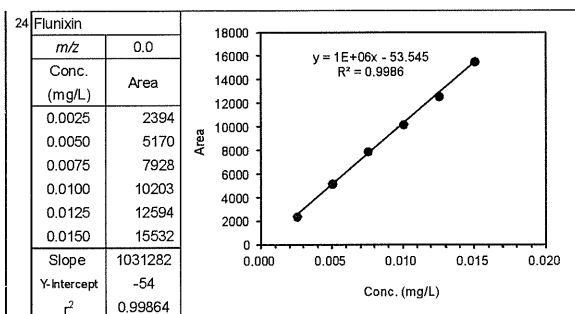
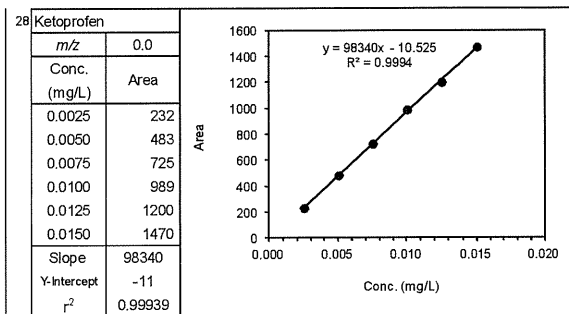
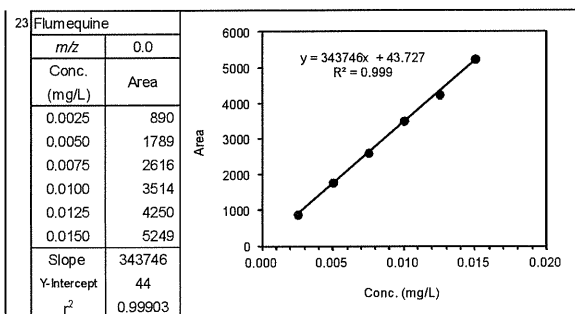
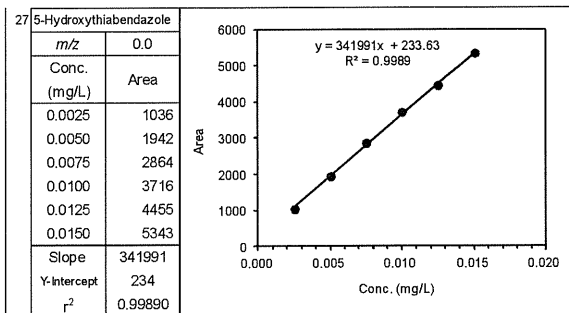
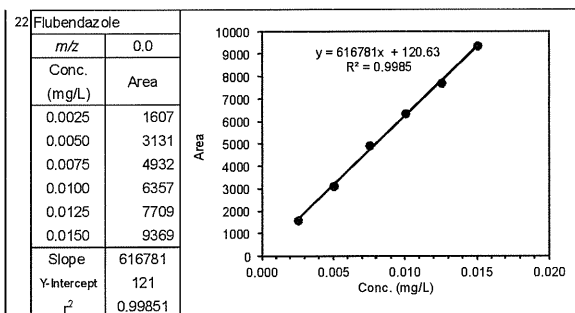
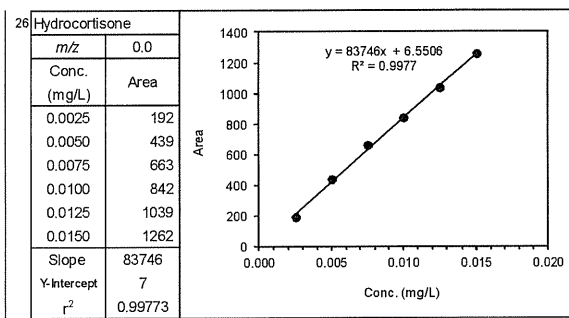
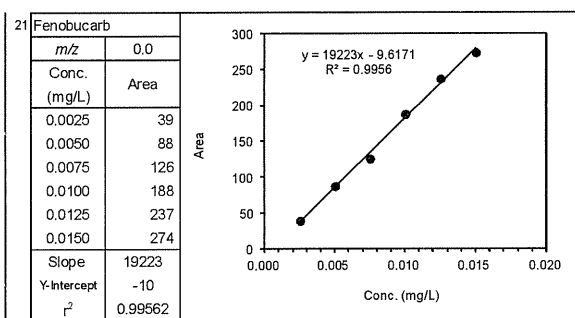


図 3-2 (つづき)



☒ 3-2 (つづき)

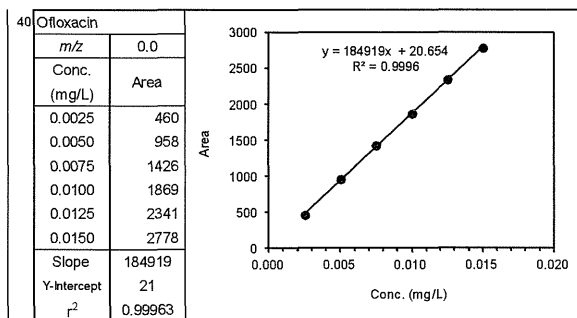
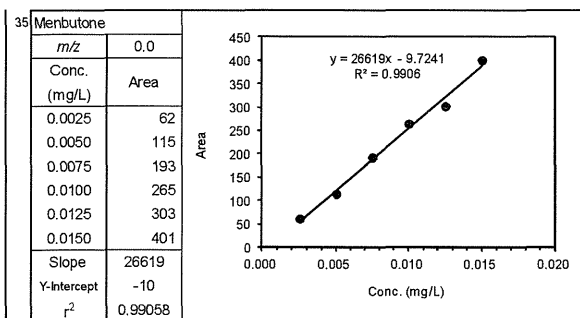
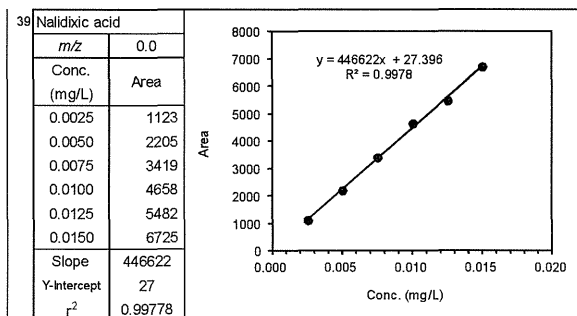
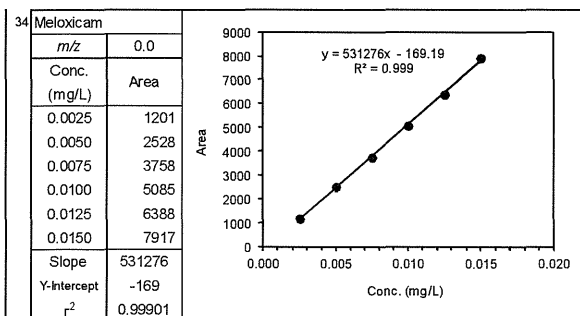
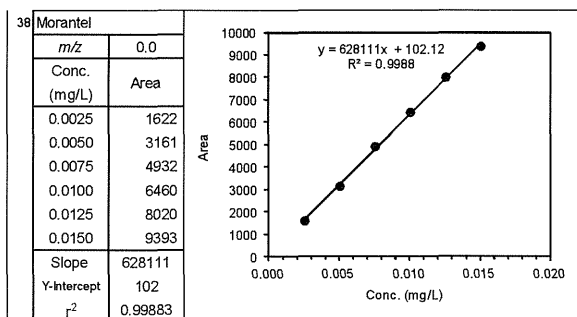
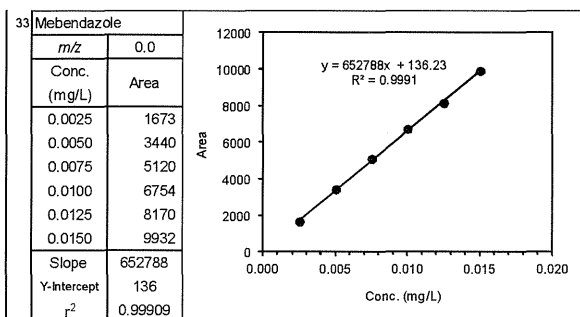
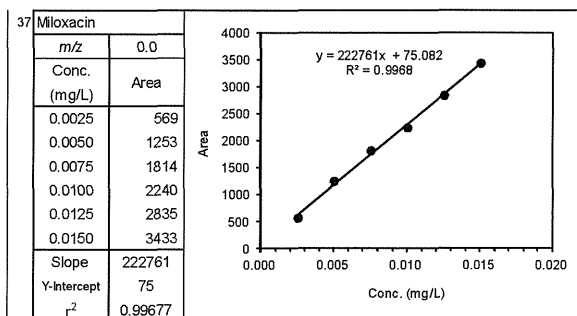
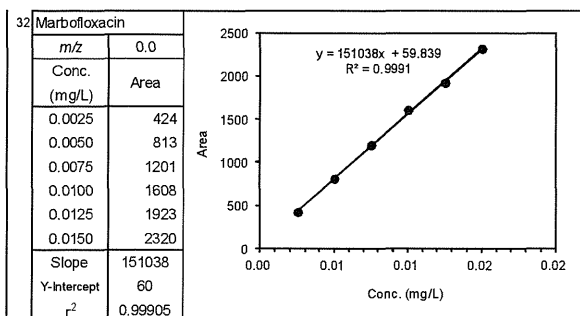
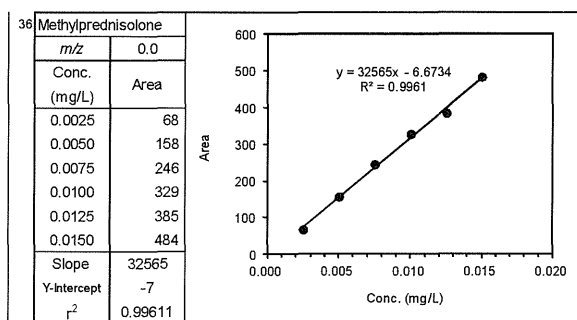
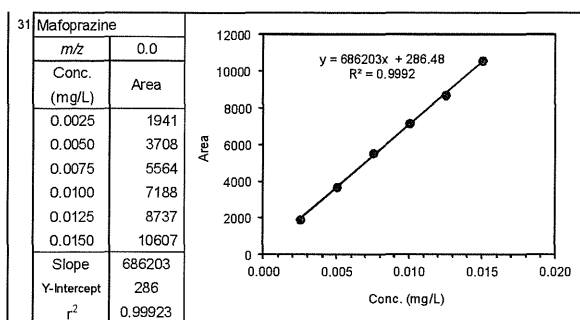
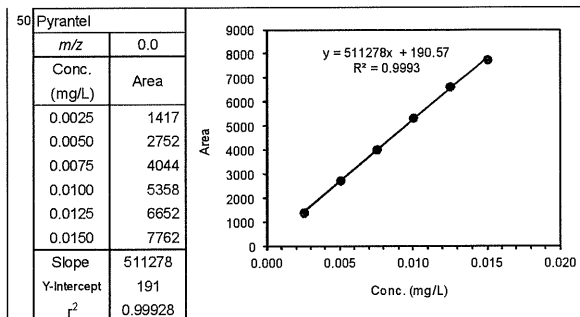
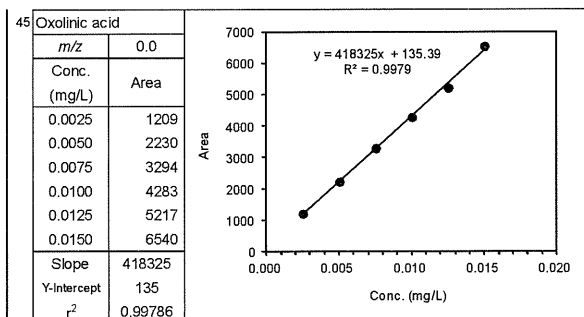
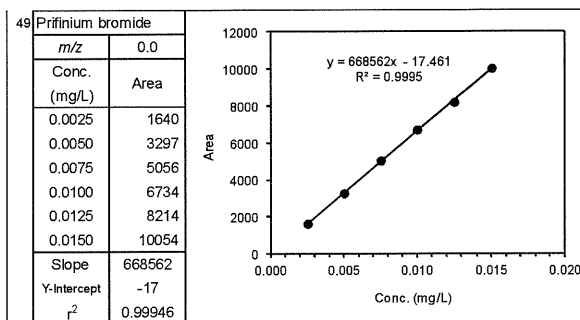
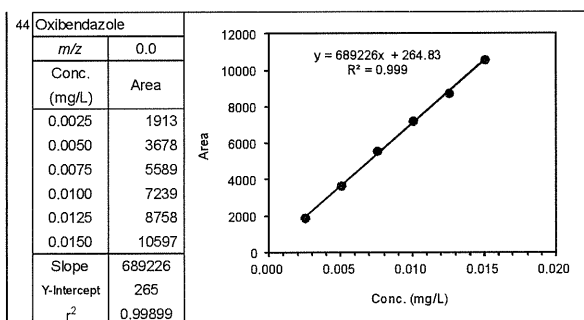
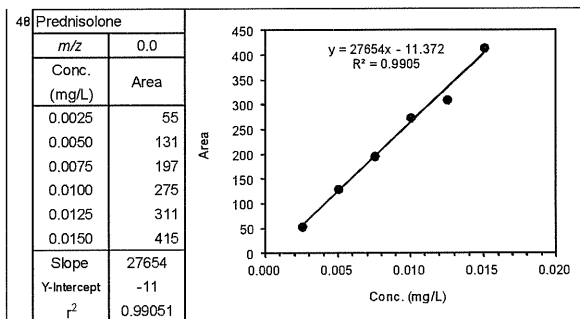
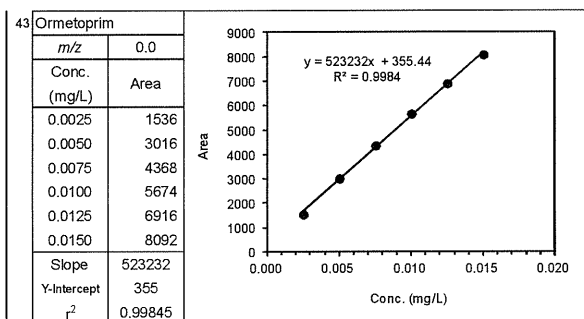
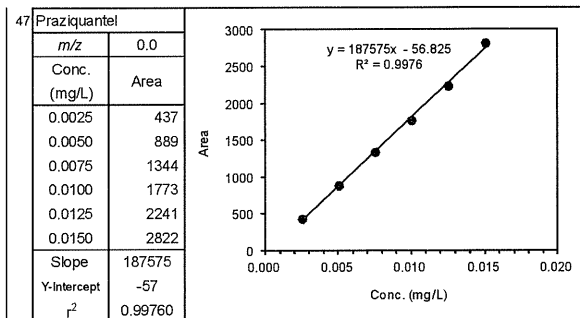
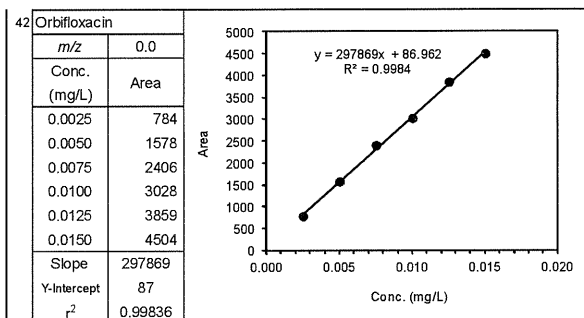
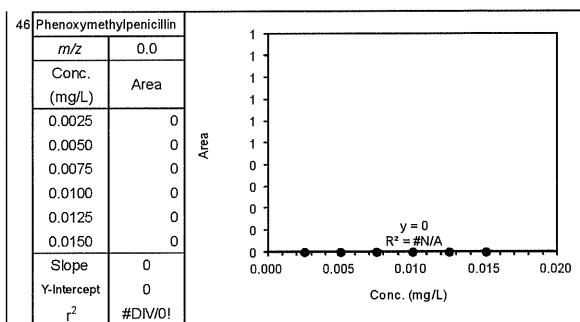
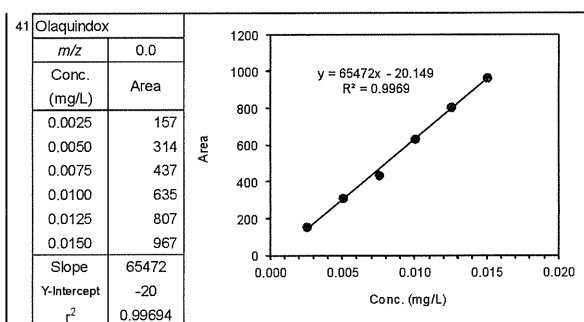


図 3-2 (つづき)



3-2 (つづき)

