

L-column2 Application Data for LC

Application No.2074

LC/MS による塩基性医薬品の一斉分析

Analysis of Basic drugs by LC/MS

医薬品には塩基性化合物が多く、HPLC 分析を行う際には、充填剤の残存シラノール基と相互作用してテーリングが起こります。今回は、抗うつ剤、抗ヒスタミン剤など 49 種類を LC/MS で一斉分析を行いました。移動相にギ酸とアセトニトリルを用いたグラジエント分析で、カラムは **L-column2 ODS**, 3 μ m, 2.1 $\times$ 250 mm を使用しました。充填剤が高度エンドキャッピングされているため、テーリングしやすい塩基性医薬品がシャープなピークで分析することができました。このような一斉分析の場合、注入量が多いと溶出の早いピークのピーク形状が悪くなりやすいので注意が必要です。

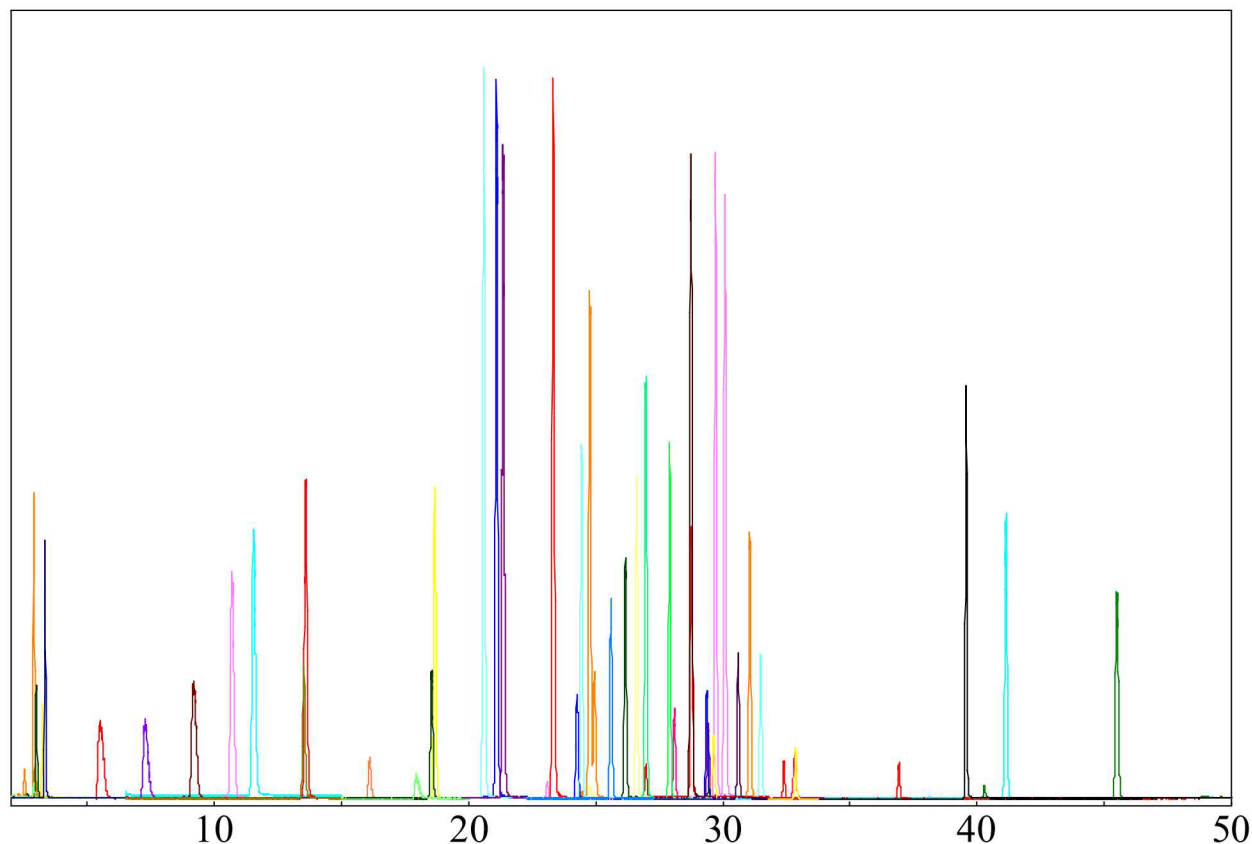


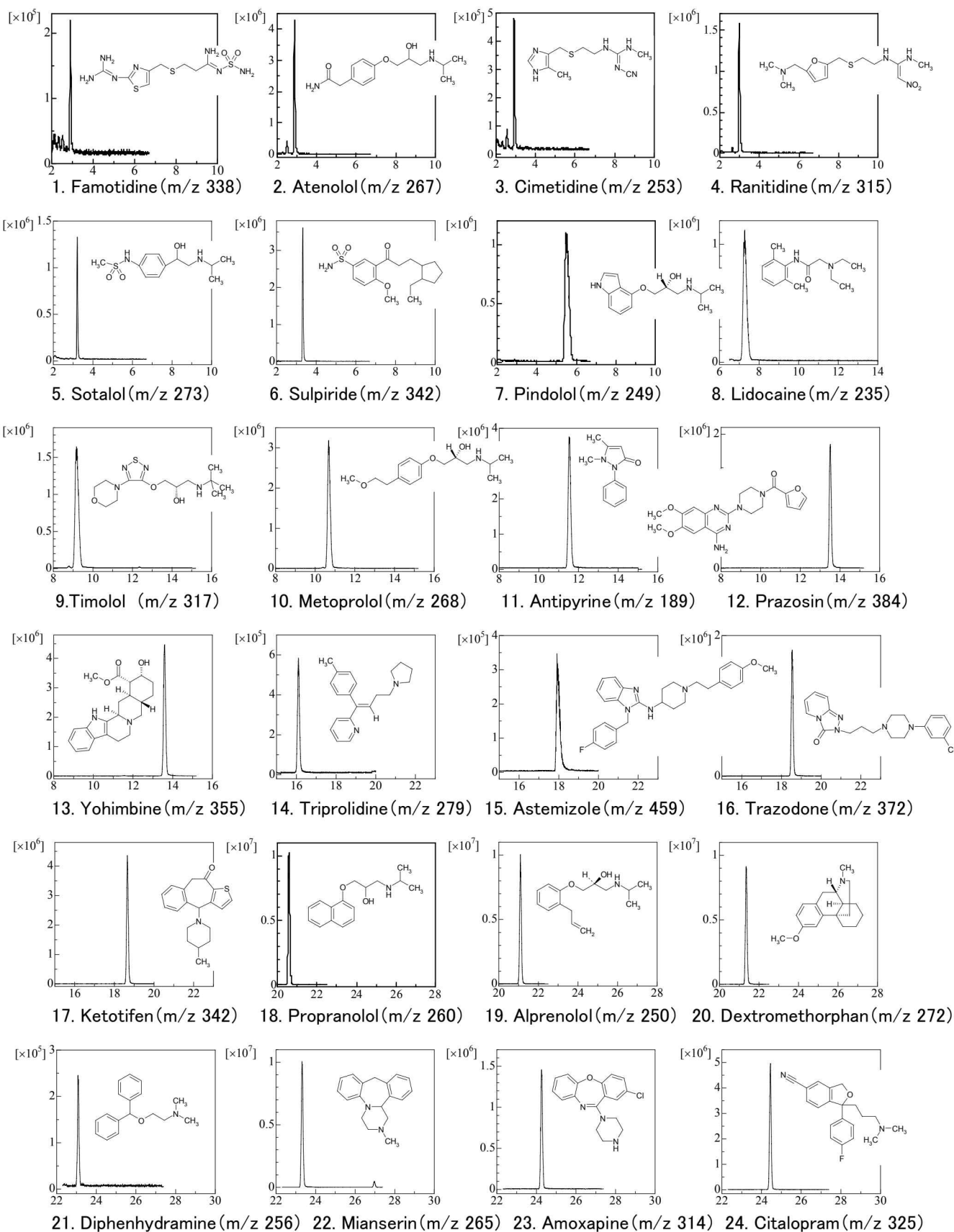
Fig. Chromatogram of Basic drugs.

【Analytical conditions】

Column:	L-column2 ODS (C18, 3 μ m, 12 nm) 2.1 mm I.D. $\times$ 250 mm L.
Mobile phase:	A: 0.1% HCOOH in CH_3CN ; B: 0.1% HCOOH in H_2O A/B, 20/80 $\rightarrow$ 40/60 $\rightarrow$ 100/0(0 $\rightarrow$ 30 $\rightarrow$ 50 min)
Flow rate:	0.2 mL/min
Temperature:	40°C
Detection:	ESI-MS(+)
Injection volume:	1 μ L
System:	LC: Alliance 2695 system(Waters Co.); MS: ZQ(Waters Co.)

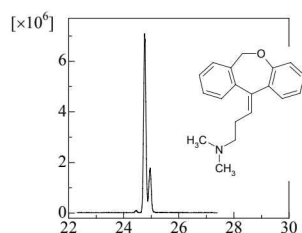
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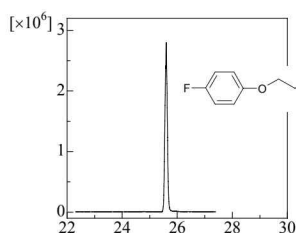


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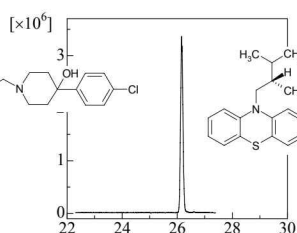
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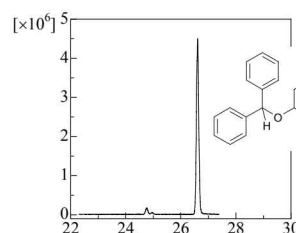
25. Doxepin (m/z 280)



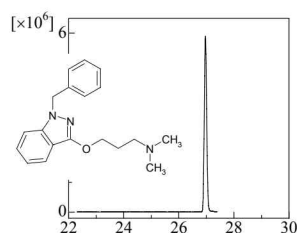
26. Haloperidol (m/z 376)



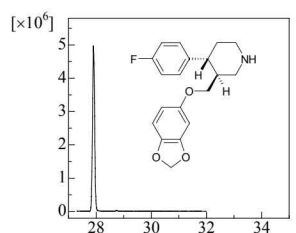
27. Promethazine (m/z 285)



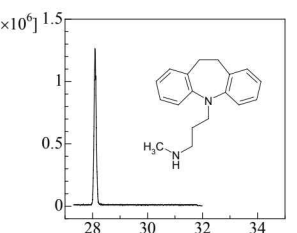
28. Diphenylpyraline (m/z 282)



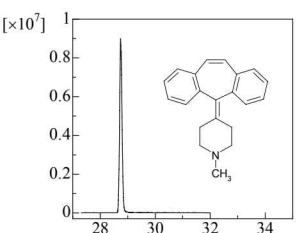
29. Benzydamine (m/z 310)



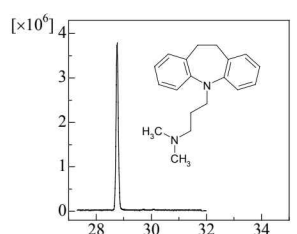
30. Paroxetine (m/z 330)



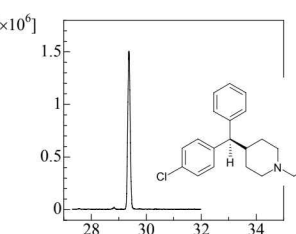
31. Desipramine (m/z 267)



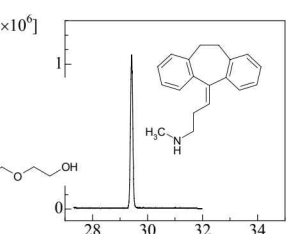
32. Cyproheptadine (m/z 288)



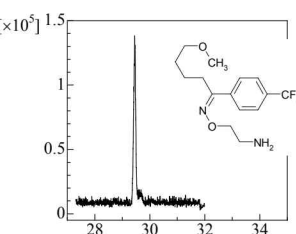
33. Imipramine (m/z 281)



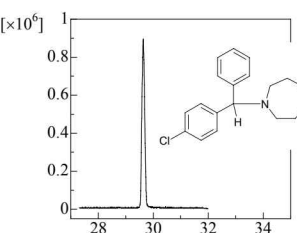
34. Hydroxyzine (m/z 375)



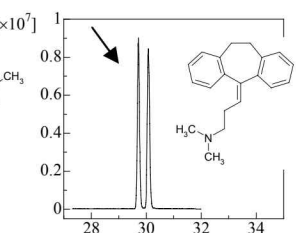
35. Nortriptyline (m/z 264)



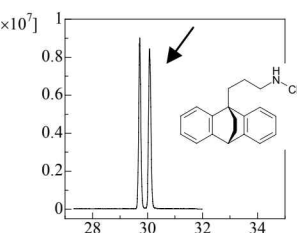
36. Fluvoxamine (m/z 319)



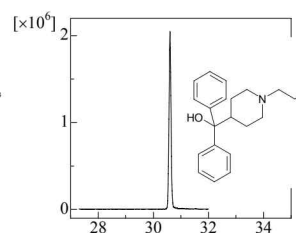
37. Homochlorcyclizine (m/z 315)



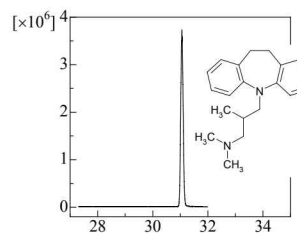
38. Amitriptyline (m/z 278)



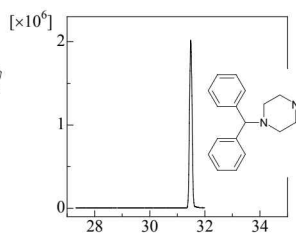
39. Maprotiline (m/z 278)



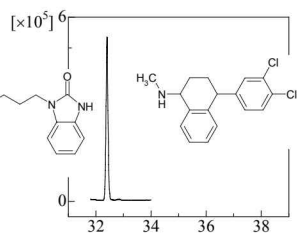
40. Fexofenadine (m/z 502)



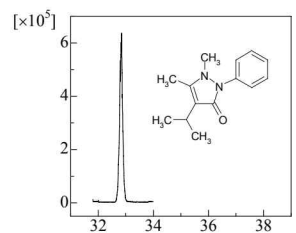
41. Trimipramine (m/z 295)



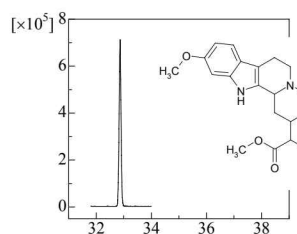
42. Oxatome (m/z 427)



43. Sertraline (m/z 275)



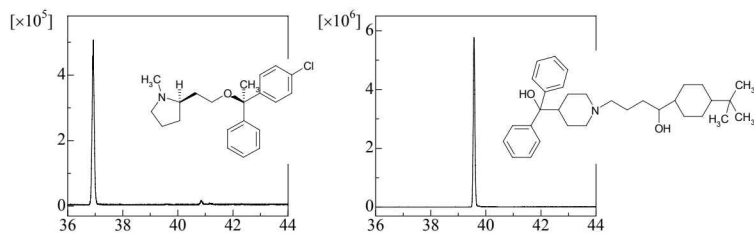
44. Isopropylantipyrene (m/z 231)



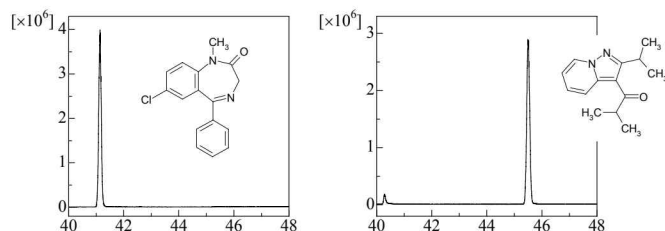
45. Reserpine (m/z 609)

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46. Clemastine (m/z 344) 47. Terfenadine (m/z 472)



48. Diazepam (m/z 285) 49. Ibudilast (m/z 231)

No.	Name	t _R (min)	m/z
1	Famotidine	2.89	338
2	Atenolol	2.89	267
3	Cimetidine	2.93	253
4	Ranitidine	2.97	315
5	Sotalol	3.19	273
6	Sulpiride	3.31	342
7	Pindolol	5.51	249
8	Lidocain	7.26	235
9	Timolol	9.14	317
10	Metoprolol	10.65	268
11	Antipyrine	11.52	189
12	Prazosin	13.49	384
13	Yohimbine	13.52	355
14	Triprolidine	16.02	279
15	Astemizole	17.82	459
16	Trazodone	18.48	372
17	Ketotifen	18.60	310
18	Propranolol	20.50	260
19	Alprenolol	21.02	250
20	Dextromethorphan	21.28	272
21	Diphenhydramin	23.01	256
22	Mianserin	23.24	265
23	Amoxapine	24.19	314
24	Citalopram	24.37	325
25	Doxepin	24.67	280

No.	Name	t _R (min)	m/z
26	Haloperidol	25.52	376
27	Promethazine	26.08	285
28	Diphenylpyraline	26.53	282
29	Benzydamine	26.87	310
30	Paroxetine	27.80	330
31	Desipramine	28.00	267
32	Cyproheptadine	28.63	288
33	Imipramine	28.65	281
34	Hydroxyzine	29.26	375
35	Nortriptyline	29.29	264
36	Fluvoxamine	29.31	319
37	Homochlorcyclizine	29.51	315
38	Amitriptyline	29.59	278
39	Maprotiline	29.96	278
40	Fexofenadine	30.51	502
41	Trimipramine	30.97	295
42	Oxatomide	31.41	427
43	Sertraline	32.30	275
44	Isopropylantipyrine	32.74	231
45	Reserpine	32.78	609
46	Clemastine	36.82	344
47	Terfenadine	39.46	472
48	Diazepam	41.01	285
49	Ibudilast	45.35	231