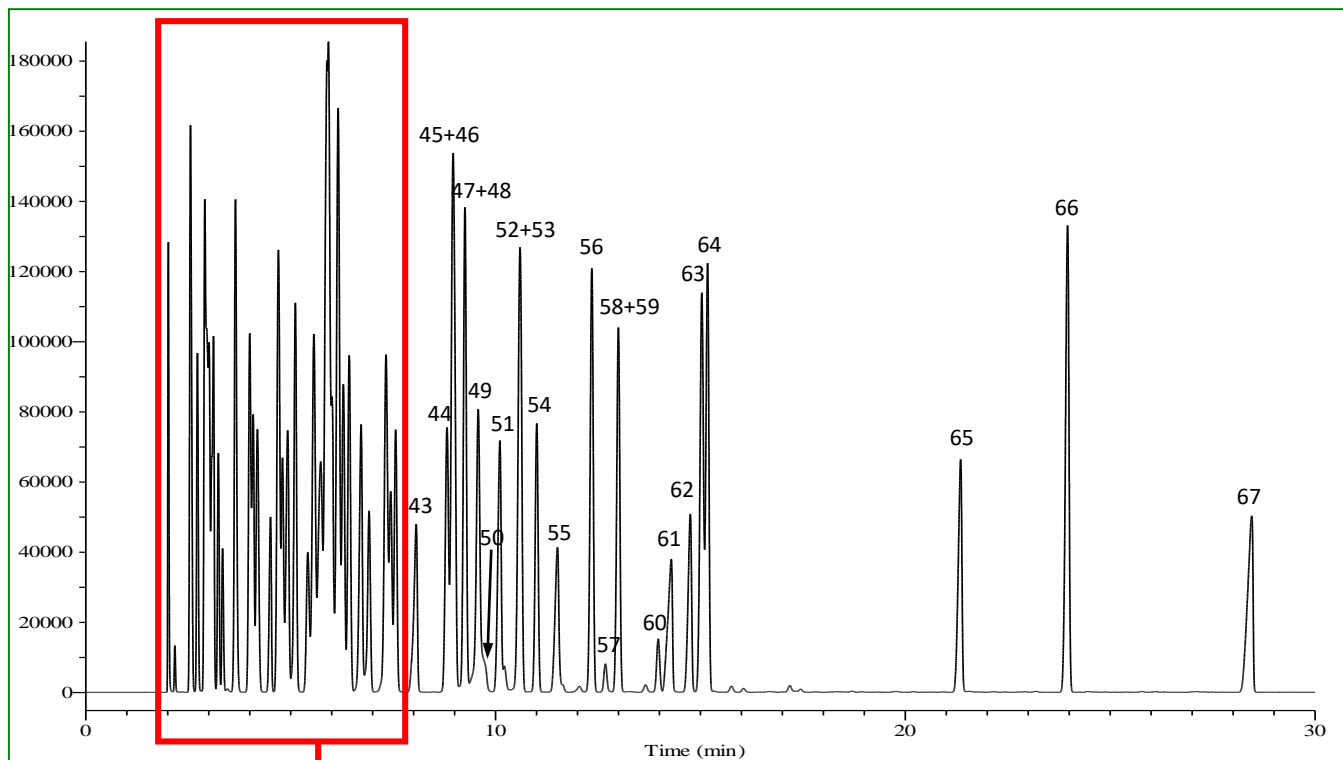


Analysis of Residual Solvent Components in Drugs - Using InertCap 624

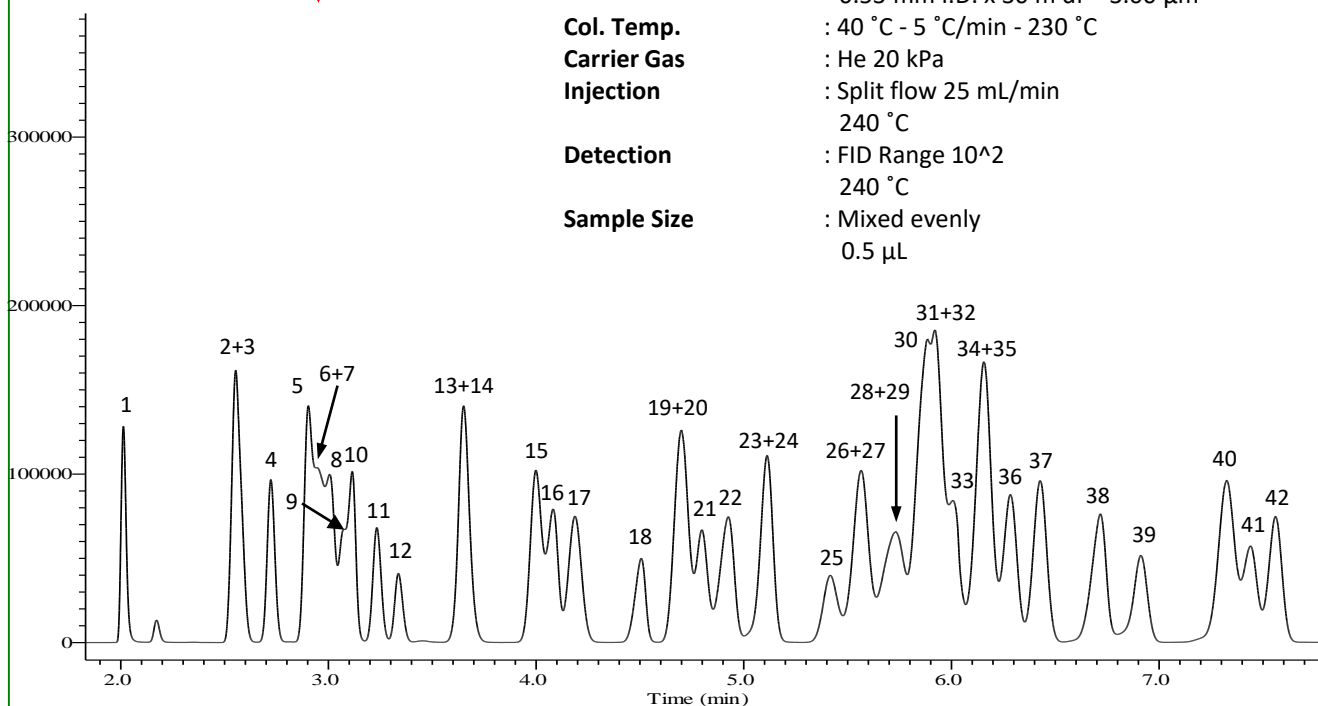
This application note is an example of the analysis of components subject to the Guideline for Residual Solvents in Pharmaceuticals.

According to the level of toxicity, this guideline classifies solvents as Class 1, Class 2, Class 3, and those for which no appropriate toxicity data is available. In this study, simultaneous analyses of components was made using GC/FID and performed with InertCap 624, a column for VOC analysis.

Example: Measurement of mixed sample



Enlarged
view



Conditions

| | |
|-------------|---|
| System | : GC - FID |
| Column | : InertCap 624 0.53 mm I.D. x 30 m df = 3.00 μ m |
| Col. Temp. | : 40 $^{\circ}$ C - 5 $^{\circ}$ C/min - 230 $^{\circ}$ C |
| Carrier Gas | : He 20 kPa |
| Injection | : Split flow 25 mL/min 240 $^{\circ}$ C |
| Detection | : FID Range 10^2 240 $^{\circ}$ C |
| Sample Size | : Mixed evenly 0.5 μ L |

Sample name and retention index

| Sample | RI | Sample | RI | Sample | RI |
|----------------------------------|-----|--------------------------------|-----|--------------------------------|------|
| 1. Methanol | 416 | 24. Chloroform | 646 | 46. 3-Methyl-1-butanol | 778 |
| 2. Ethanol | 500 | 25. 1,1,1-Trichloroethane | 659 | (Isoamyl alcohol) | |
| 3. n-Pentane | 500 | 26. Cyclohexane | 665 | 47. Toluene | 787 |
| 4. Diethyl ether | 510 | 27. 2,2-Dimethoxypropane | 665 | 48. Ethylene glycol | 787 |
| 5. Acetone | 523 | 28. Carbon tetrachloride | 671 | 49. Isobutyl acetate | 797 |
| 6. 1,1-Dichloroethylene | 528 | 29. 2-Methyl-1-propanol | 671 | 50. Formamide | 799 |
| 7. 1,1-Dimethoxymethane | 528 | (Isobutyl alcohol) | | 51. 1-Pentanol(Amyl alcohol) | 812 |
| 8. 2-Propanol(Isopropyl alcohol) | 530 | 30. 1,2-Dimethoxyethane | 677 | 52. Propionaldehyde diethyl ac | 826 |
| 9. Ethyl formate | 536 | 31. 1,2-Dichloroethane | 680 | 53. 2-Hexanone(MBK) | 826 |
| 10. Acetonitrile | 538 | 32. Benzene | 680 | 54. n-Butyl acetate | 837 |
| 11. Methyl acetate | 546 | 33. Isopropyl acetate | 683 | 55. N,N-Dimethylformamide | 850 |
| 12. Dichloromethane | 554 | 34. 2,2,4-Trimethylpentane | 689 | 56. Chlorobenzene | 874 |
| 13. trans-1,2-Dichloroethylene | 576 | 35. 2-Methyltetrahydrofuran | 689 | 57. Ethylbenzene | 883 |
| 14. tert-Butyl methyl ether | 576 | 36. Methyl isopropyl ketone | 694 | 58. p-Xylene | 891 |
| 15. n-Hexane | 600 | 37. n-Heptane | 700 | 59. m-Xylene | 891 |
| 16. 1-Propanol | 603 | 38. 1-Butanol | 709 | 60. o-Xylene | 918 |
| 17. Diisopropyl ether | 608 | 39. Trichloroethylene | 715 | 61. Dimethyl sulfoxide(DMSO) | 925 |
| 18. Nitromethane | 621 | 40. Methylcyclohexane | 728 | 62. N,N-Dimethylacetamide | 939 |
| 19. 2-Butanone(MEK) | 629 | 41. 1,4-Dioxane | 731 | 63. Cumene | 947 |
| 20. cis-1,2-Dichloroethylene | 629 | 42. n-Propyl acetate | 735 | 64. Anisole | 951 |
| 21. Ethyl acetate | 633 | 43. 2-Ethoxyethanol | 750 | 65. N-methyl-2-pyrrolidone | 1124 |
| 22. 2-Butanol | 638 | 44. 4-Methyl-2-pentanone(MIBK) | 774 | 66. 1,2,3,4-Tetrahydronaphthal | 1203 |
| 23. Tetrahydrofuran | 646 | 45. Pyridine | 778 | 67. Sulfolane | 1244 |

Xylene is a mixture of m Xylene, p Xylene, o Xylene, and Ethylbenzene.

Retention indices are...

This value is based on the number of carbons in the straight-chain alkane and is calculated using the retention time of each component and hydrocarbon.

In this application, a temperature-rise analysis was made. The formula is shown below.

$$\text{Retention index} = 100 \times \frac{\text{TR} - \text{tR}(Z)}{\text{TR}(Z+1) - \text{tR}(Z)} + 100 \times Z$$

TR = retention time of the target component

tR(Z) = retention time of straight-chain alkanes that precede the components of interest

tR(Z+1) = retention time of straight-chain alkanes emerging after the components of interest

Z = number of carbons in the straight-chain alkane with retention time tR(Z)

GL Sciences disclaims any and all responsibility for any injury or damage which may be caused by this data directly or indirectly. We reserve the right to amend this information or data at any time and without any prior announcement.

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