# DETERMINATION OF PHENOLIC COMPOUNDS (HJ 703-2014)

Technology Advantage:
Agilent Intuvo 9000 GC with FID


## Introduction

HJ 703-2014 is a method approved by the Chinese Ministry of Environmental Protection for the determination of 21 phenolic compounds in soils and sediments by gas chromatography with flame ionization detection (FID). The method specifies procedures for sample extraction, analysis, identification, and quantitation [1].

This application brief demonstrates that the Agilent Intuvo 9000 can easily achieve the performance specification for the analysis of phenols outlined in method HJ 703-2014.

## Instrumentation

- Agilent Intuvo 9000 GC with FID
- Agilent J&W DB-8270D Ultra Inert Intuvo, 30 m × 0.25 mm, 0.25 µm column (p/n 122-9732-INT)
- All instrumental settings were the same as those listed in the method with the exception of the flow rate, which was set to 2 mL/min.
- The guard chip was programmed to follow the column temperature program.

## Sample preparation

- A stock mixture of 21 phenols at a concentration of 1,000 µg/mL was used to prepare standards for the study (AccuStandard, New Haven, CT).
- Standards were prepared in a 4:1 mixture of dichloromethane and ethyl acetate (v/v) at the concentrations listed in the method 1, 5, 20, 50, and 100 µg/mL.

For more information, visit:
**www.agilent.com**

Agilent Technologies

## Results and Discussion

Figure 1 shows an example chromatogram of the 21 phenols at a concentration of 20 µg/mL. Table 1 lists the peak number identifications. Response and peak shape for all the target compounds closely match the reference chromatogram shown in HJ 703-2014. In particular, note the response and peak shape for 2,4-dinitrophenol, 4-nitrophenol, and pentachlorophenol. These compounds are among the most acidic, and are the most difficult to analyze within the group of phenols. The Intuvo 9000 demonstrates good performance for the analysis of these compounds, indicating a highly inert flowpath from inlet to detector.

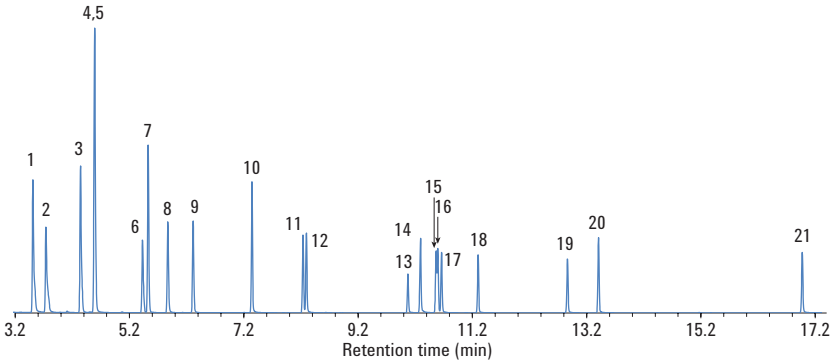


**Figure 1.** Chromatogram of the 21 phenols in HJ 703-2014.

**Table 1.** Retention time windows for target identification.

| Compound number | Target | Average RT (min) | Standard deviation (min) | RT Window (min) | Range (min) |
|---|---|---|---|---|---|
| **1** | Phenol | 3.511 | 0.004 | 3.500–3.523 | 0.023 |
| **2** | 2-Chlorophenol | 3.738 | 0.002 | 3.732 -3.744 | 0.012 |
| **3** | 2-Methylphenol | 4.344 | 0.003 | 4.334–4.354 | 0.020 |
| **4,5** | 4-Methylphenol and 3-Methylphenol | 4.595 | 0.012 | 4.559–4.631 | 0.072 |
| **6** | 2-Nitrophenol | 5.429 | 0.003 | 5.419–5.439 | 0.020 |
| **7** | 2,4-Dimethylphenol | 5.528 | 0.006 | 5.510–5.546 | 0.036 |
| **8** | 2,4-Dichlorophenol | 5.871 | 0.003 | 5.862–5.881 | 0.019 |
| **9** | 2,6-Dichlorophenol | 6.314 | 0.003 | 6.304–6.323 | 0.019 |
| **10** | 4-Chloro-3-methylphenol | 7.345 | 0.005 | 7.330–7.360 | 0.030 |
| **11** | 2,4,6-Trichlorophenol | 8.237 | 0.004 | 8.226–8.249 | 0.022 |
| **12** | 2,4,5-Trichlorophenol | 8.297 | 0.005 | 8.282–8.312 | 0.030 |
| **13** | 2,4-Dinitrophenol | 10.077 | 0.007 | 10.057–10.096 | 0.039 |
| **14** | 4-Nitrophenol | 10.301 | 0.020 | 10.241–10.361 | 0.120 |
| **15** | 2,3,4,5-Tetrachlorophenol | 10.570 | 0.010 | 10.540–10.599 | 0.059 |
| **16** | 2,3,5,6-Tetrachlorophenol | 10.599 | 0.009 | 10.571–10.626 | 0.055 |
| **17** | 2,3,4,6-Tetrachlorophenol | 10.599 | 0.008 | 10.641–10.687 | 0.045 |
| **18** | Methyl-4,6-dinitrophenol | 10.664 | 0.010 | 11.275–11.335 | 0.061 |
| **19** | Pentachlorophenol | 11.305 | 0.006 | 12.849–12.887 | 0.038 |
| **20** | 2-*sec*-Butyl-4,6-dinitrophenol | 12.868 | 0.009 | 13.386–13.438 | 0.052 |
| **21** | 2-Cyclohexyl-4,6-dinitrophenol | 13.412 | 0.008 | 16.954–17.001 | 0.047 |

Compound identification is based upon the retention time of a target eluting within a range of retention times. The time range is defined as six standard deviations of the average retention time for the calibration standards. Table 1 lists the average retention time, standard deviation, retention time window, and retention time range. Retention time precision is quite good. For two closely eluting isomers, 2,4,6-trichlorophenol and 2,4,5-trichlorophenol, the retention time windows do not overlap. This allows separate identification and quantitation for these two compounds.

Quantitation is based upon instrument response calibration. Either the peak area or height may be used to construct a calibration curve fitted with a straight line. For a calibration to be valid, the correlation coefficient must be greater than or equal to 0.995, and the calculated concentration of the middle calibration standard must be within ±30 %. Table 2 lists the correlation coefficients and the error in calculated concentration for the 20 µg/mL standard. All of them exceed the method calibration requirements.

**Table 2.** Calibration metrics for target compounds.

| Compound number | Target | Correlation coefficient | Calculated of 20 µg/mL standard | Percent error of calculated 20 µg/mL standard |
|---|---|---|---|---|
| 1 | Phenol | 1.000 | 18.4 | -7.8 |
| 2 | 2-Chlorophenol | 1.000 | 18.5 | -7.5 |
| 3 | 2-Methylphenol | 0.999 | 18.0 | -10.1 |
| 4,5 | 4-Methylphenol and 3-Methylphenol | 0.999 | 18.4 | -7.8 |
| 6 | 2-Nitrophenol | 0.998 | 17.2 | -13.9 |
| 7 | 2,4-Dimethylphenol | 1.000 | 18.5 | -7.6 |
| 8 | 2,4-Dichlorophenol | 1.000 | 18.6 | -7.0 |
| 9 | 2,6-Dichlorophenol | 1.000 | 18.6 | -7.1 |
| 10 | 4-Chloro-3-methylphenol | 1.000 | 18.6 | -6.8 |
| 11 | 2,4,6-Trichlorophenol | 1.000 | 18.4 | -7.9 |
| 12 | 2,4,5-Trichlorophenol | 1.000 | 18.9 | -5.4 |
| 13 | 2,4-Dinitrophenol | 0.996 | 16.4 | -17.8 |
| 14 | 4-Nitrophenol | 0.999 | 17.9 | -10.4 |
| 15 | 2,3,4,5-Tetrachlorophenol | 0.997 | 17.7 | -11.3 |
| 16 | 2,3,5,6-Tetrachlorophenol | 0.999 | 20.2 | 1.1 |
| 17 | 2,3,4,6-Tetrachlorophenol | 1.000 | 19.6 | -2.2 |
| 18 | Methyl-4,6-dinitrophenol | 0.998 | 17.4 | -13.2 |
| 19 | Pentachlorophenol | 0.999 | 18.3 | -8.3 |
| 20 | 2-*sec*-Butyl-4,6-dinitrophenol | 0.999 | 17.7 | -11.5 |
| 21 | 2-Cyclohexyl-4,6-dinitrophenol | 0.997 | 17.0 | -14.8 |

## Conclusion

The Agilent Intuvo 9000 demonstrates outstanding performance for the analysis of phenols, and meets the requirements specified in HJ 703-2014.

## Reference

1. Soil and Sediment – Determination of phenolic compounds by Gas Chromatography. National Environmental Protection Standard of the People's Republic of China. HJ 703-2014.

www.agilent.com
Information, descriptions and specifications in this publication are subject to change without notice.


Published in USA, July 13, 2017
5991-8087EN

Agilent Technologies