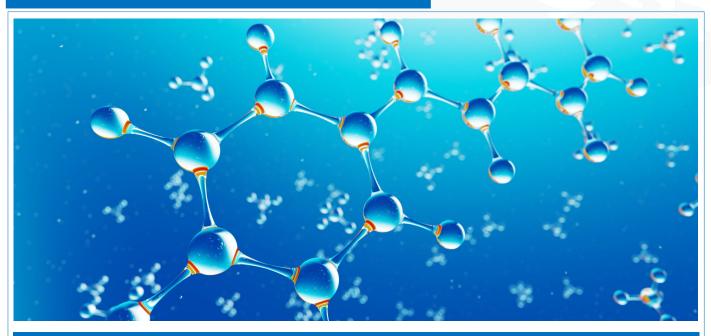
APPLICATION NOTE AN158





UOP555, Trace impurities in Benzene by Gas chromatography.

KEY WORDS: Impurities, Benzene, UOP

INTRODUCTION

SCION Instruments offers the solution for trace impurities in high-purity benzene by gas chromatography (GC).

After manufacturing products might contain trace impurities. The UOP555 describes the determination of individual and total trace hydrocarbon impurities in olefin-free, high purity benzene. It is possible to determine C_8 , lower boiling paraffines, naphthenes, toluene and C_8 aromatics.

This application is applicable on the SCION Instruments 4X6 and the new 8X00 GC-platform. Figure 1 shows the new SCION Instruments 8X00 GC platform with 8400PRO autosampler.



Figure 1. SCION Instruments 8300 and 8500 GC platform equipped with the 8400PRO autosampler.



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RESULTS AND DISCUSSION

UOP555 describes the determination of individual and total trace hydrocarbon impurities in olefin-free, high purity benzene. It is possible to determine C_8 , lower boiling paraffines, naphthene, toluene and C_8 aromatics.

After manufacturing the product might contain trace impurities, this analysis can determine the type and amount of these hydrocarbon impurities. The lower limit of quantitation (LOQ) for any single component is 1 mg/kg.

When looking at interreferences it shows that the complete separation of m-xylene and p-xylene can be problematic when these components are analysed. The separation can be considered adequate when the distance to from the baseline to the valley between two peaks is not greater than 50% of the peak height, this is determined by the lowest of the two peaks.

It is important to check the separation of these components with a check sample before implementing the method. Figure 2 shows an example chromatogram of standard ST00033, and figure 3 shows the m/p-xylene separation of this standard. In addition, the check sample can be used to determine the retentions times of the components.

This method uses the effective carbon number (ECN) since the FID does not response equally on a mass basis to all the components. In addition to this theoretical factor the absolute response factor for n -octane is calculated using the calibration blend.

Table 1. Analytical conditions

| Injector | Split 50:1, 250 ℃ |
|--------------|--|
| Column | SCION-DHA |
| Oven Program | 32°C (6 min), 5°C/min to 52°C (14 min), 20°C/min to 250°C 0 min) |
| Carrier | Hydrogen |
| Flow | 1.2 ml/min (constant flow) |
| Detector | FID with ceramic jet, 250°C |
| | Air: 300 ml/min, Fuel gas (H ₂): 30 ml/ min, Make up (N ₂): 28 ml/min |
| Inj. Volume | 0.5 μΙ |
| Autosampler | 8400PRO |
| Software | Compass CDS |

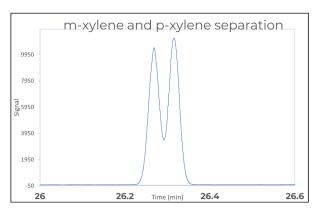


Figure 3. Separation example of m-xylene and p-xylene using standard ST000033.

Using both the ECN and calculated response factor the mass of the components can be calculated in mg/kg.

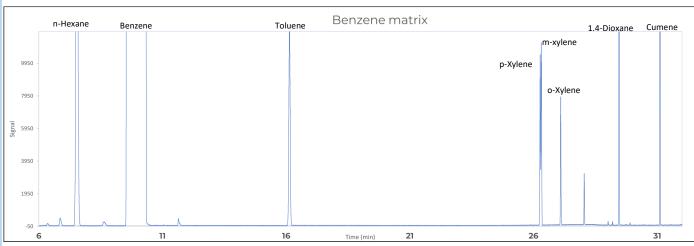


Figure 2. Example chromatogram of standard ST000033.



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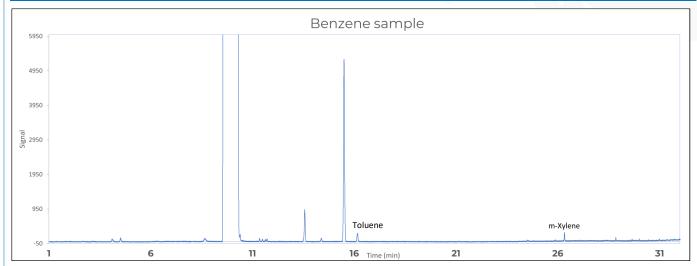


Figure 4. Example chromatogram of a Benzene sample.

The repeatability of the method was determined by performing seven consecutive injections of standard ST000033. This mixture is a certified standard with low concentrations. The results in table 2 show good results for these low concentrations.

Figure 4 shows an example chromatogram from a actual benzene sample. In this chromatogram 0.80 mg/kg Toluene and 0.26 mg/kg m-Xylene was identified and determined (table 3). In addition, there is also 25.2 mg/kg unknow impurities present in the sample.

CONCLUSION

The SCION 8X00-GC analyser is perfectly capable of individual and total trace hydrocarbon impurities in olefin-free, high purity benzene with good repeatability and in compliance with UOP555.

The equipment of the 8X00-GC analyser is pre determined. Ordering information can be found on the next page. For customisation please contact your local sales representative.

Although the 4X6-GC series is not shown in this application note, it is possible to perform this analysis on the SCION Instruments 4X6 GC series.

Table 2. Repeatability of ST00033

| | Rsd % |
|-------------|-------|
| n-Hexane | 2.77 |
| Toluene | 2.30 |
| m-Xylene | 2.65 |
| p-Xylene | 2.94 |
| o-Xylene | 2.68 |
| 1.4-Dioxane | 2.74 |
| Cumene | 2.73 |

Table 3. Benzene sample results in mg/kg

| | mg/kg |
|----------|-------|
| Toluene | 0.80 |
| m-Xylene | 0.26 |
| Misc. | 25.2 |



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ORDER INFORMATION

| ORDER INFORMATION | | |
|---|--|--|
| Available standards | | |
| D7504 AROMATIC MULTI-COMPONENT CHECK STANDARD IN BENZENE - 10X2 ML | | |
| D7504 AROMATIC MULTI-COMPONENT CHECK STANDARD IN P-XYLENE - 10X2 ML | | |
| D7504 AROMATIC MULTI-COMPONENT CHECK STANDARD IN TOLUENE - 10X2 ML | | |
| Available systems UOP555 | | |
| UOP 555 analyzer based on 8300-GC 120V and 8400Pro Auto Sampler | | |
| UOP 555 analyzer based on 8300-GC 120V and 8410Pro Auto Injector | | |
| UOP 555 analyzer based on 8300-GC 230V and 8400Pro Auto Sampler | | |
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SCION Instruments

UK

4 Michaelson Square Livingston EH54 7DP, Scotland, UK Phone +44 1506 300 200 sales-eu@scioninstruments.com The Netherlands

Amundsenweg 22-24 4462 GP Goes, The Netherlands Phone +31 (0) 113 287 600 sales-eu@scioninstruments.com