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## Committee D22 on Air Quality
## Subcommittee D22.04 on Workplace Air Quality

## Research Report D22-1030

# Interlaboratory Study to Establish Precision Statements for ASTM D5836, Standard Test Method for Determination of 2,4-Toluene Diisocyanate (2,4-TDI) and 2,6-Toluene Diisocyanate (2,6-TDI) in Workplace Atmospheres (1-2 PP Method)

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## 1. Introduction

This research report presents precision and accuracy data obtained in an interlaboratory test study involving analysis of spiked samples by 13 different participating laboratories.

## 2. Test Method

The test method uses glass-fiber filters impregnated with 1-(2-pyridyl)piperazine (1-2PP) to sample air suspected to be contaminated with 2,4-TDI and/or 2,6-TDI. 2,4-TDI and/or 2,6-TDI are converted to stable derivatives by 1-2PP that are analyzed by high-performance liquid chromatography (HPLC) with fluorescence or ultraviolet detection.

## 3. List of Participating Laboratories

A list of participating laboratories is shown in Table 3.1.

Table 3.1
Participating Laboratories

| Laboratory | Address | Telephone | Contact |
|---|---|---|---|
| The Travelers Industrial Hygiene Laboratory | 90 Lamerton Rd.<br>Windsor, CT | 800.842.0355 | George Johnson |
| AFIERA/SDC | 2350 Gillingham<br>Brooks City Base, TX 78235 | 210.536.6165 | Gloria Gover |
| Kemper/NATLSCO | 95 Oakwood Road<br>Zurich, IL 60047 | 847.320.7188 | Bill Walsh |
| Johns Manville IH Lab | 10100 W. Ute Ave.<br>Littleton, CO 80127 | 303.978.2584 | Scott Stiener |
| OSHA SLTC | 1781 South 300 West<br>Salt Lake City, UT 84115-1802 | 801.524.7900 | Wayne Potter |
| DataChem | 960 W. Levoy Dr.<br>Salt Lake City, UT 84123 | 801.266.7700 | Jim Perkins |
| WOHL | 2601 Agriculture Dr.<br>Madison, WI 53718 | 608.224.6210 | Derek Popp |
| BASF Corp. | 1609 Biddle Ave.<br>Wyandotte, MI 48192 | 734.324.6320 | Rob Laney |
| Liberty Mutual Insurance Lab | 71 Frankland Rd.<br>Hopkinton, MA 01748 | 800.230.6263<br>X 252 | Ethyl Patricio |
| Bayer Polymer Organization | 100 Bayer Rd.<br>Pittsburgh, PA 15205 | 412.777.2931 | V. Dharmarajan |
| WISHA Laboratory | 805 Plum St.<br>First Floor MS-4613<br>Olympia, WA 98504-4613 | 360.902.5171 | Philip Peters |
| Czartech Analytical Inc. | 42910 W. Ten Mile Rd.<br>Complex A-6<br>Novi, MI 48375-5419 | 248.348.2300 | Bruce Czarnecki |
| NEPMU | 1887 Powhatan St.<br>Norfolk, VA 23511-3319 | 757-444-7671<br>X 3038 | George Lindsay |

## 4. Interlaboratory Test Program Instructions

A cover letter summarizing analytical instructions discussed in telephone conversations with each participating laboratory was included with the spiked samples and it is presented in Appendix 1. A copy of ASTM D5836-95 was included with the spiked samples and a copy is included in Appendix 3. ASTM Standard E691-99 Standard

Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method was used as a guide for this study and to perform statistical calculations.

## 5. Statistical Data Summary

Thirteen sets of glass-fiber filters were spiked with 4 levels of 1-2PP derivatives of TDI (TDIP) and sent to 13 participating laboratories by overnight shipping. Each filter set consisted of 4 filters that were first coated with 1-2PP as described in the method, and then spiked with 2,4-TDIP and 2,6-TDIP from the same solution, plus a blank coated filter. The filters were shipped in separate 4-mL glass vials. A sample of the diluted TDIP mixture used to spike the coated filters was included with each filter set for use as an analytical standard. The amounts of TDIP (expressed respectively as mg/mL of free 2,4- and 2,6-TDI) present in the mixture solution were made known to the participating laboratories. The amounts of 2,4- and 2,6-TDIP (expressed as free 2,4- and 2,6-TDI) spiked on the filters are shown in Table 5.1.

Table 5.1
Amounts (Φg) of 2,4- and 2,6-TDIP (as Free TDI) Spiked on the Filters

| Filter 1 | | Filter 2 | | Filter 3 | | Filter 4 | |
|---|---|---|---|---|---|---|---|
| 2,4-TDI | 2,6-TDI | 2,4-TDI | 2,6-TDI | 2,4-TDI | 2,6-TDI | 2,4-TDI | 2,6-TDI |
| 5.37694 | 5.32496 | 3.97426 | 3.93584 | 2.80536 | 2.77824 | 1.40268 | 1.38912 |

Results from the analysis of the spiked filters were reported by the participating labs in terms of g per sample. The raw data is presented in Appendix 2. The results are expressed here as percent of the amounts spiked on each filter, and are shown in Tables 5.2 and 5.3.

Table 5.2
2,4-TDI

| | Recovery (percent of theoretical) | | | | Statistical Calculations | | | | |
|---|---|---|---|---|---|---|---|---|---|
| level | 1 | 2 | 3 | 4 | 0 | s | d | h | k |
| Lab ID | | | | | | | | | |
| 1 | 100.15 | 99.11 | 100.84 | 101.81 | 100.4775 | 1.1379 | -5.4086 | -0.91 | 0.17 |
| 2 | 100.24 | 107.44 | 102.66 | 105.51 | 103.9625 | 3.1645 | -1.9236 | -0.32 | 0.48 |
| 3 | 106.01 | 106.43 | 108.36 | 115.49 | 109.0725 | 4.3990 | 3.1864 | 0.54 | 0.67 |
| 4 | 85.55 | 103.16 | 99.81 | 99.81 | 97.0825 | 7.8488 | -8.8036 | -1.48 | 1.20 |
| 5 | 102.75 | 103.67 | 105.16 | 109.43 | 105.2525 | 2.9567 | -0.6336 | -0.11 | 0.45 |
| 6 | 99.50 | 93.60 | 102.30 | 106.23 | 100.4075 | 5.3119 | -5.4786 | -0.92 | 0.81 |
| 7 | 107.87 | 101.65 | 102.30 | 121.20 | 108.2550 | 9.0703 | 2.3689 | 0.40 | 1.38 |
| 8 | 106.01 | 103.16 | 106.94 | 106.94 | 105.7625 | 1.7895 | -0.1236 | -0.02 | 0.27 |
| 9 | 92.43 | 99.39 | 98.38 | 103.37 | 98.3925 | 4.5211 | -7.4936 | -1.26 | 0.69 |
| 10 | 98.59 | 118.46 | 118.81 | 129.61 | 116.3673 | 12.9321 | 10.4811 | 1.76 | 1.97 |
| 11 | 111.59 | 110.71 | 117.63 | 121.20 | 115.2825 | 5.0023 | 9.3964 | 1.58 | 0.76 |
| 12 | 104.52 | 108.70 | 107.29 | 117.63 | 109.5350 | 5.6691 | 3.6489 | 0.61 | 0.86 |
| 13 | 97.55 | 101.53 | 107.12 | 120.48 | 106.6700 | 10.0085 | 0.7839 | 0.13 | 1.53 |

Average of cell averages 105.89
Standard deviation of cell averages 5.95 (RSD 5.62)
Repeatability standard deviation 6.56 (RSD 6.20)
Reproducibility standard deviation 8.23 (RSD 7.77)