**PROPICONAZOLE**

**Collaborative Study**

Full Scale Collaborative Study
for the
Determination of Propiconazole

in Technical Concentrate and Formulations
by Gas Chromatography and

Flame Ionisation Detection

Report to CIPAC

by

Syngenta Crop Protection AG
in collaboration with DAPA

Dr. Christian Mink

Breitenloh 5

CH-4333 Münchwilen

Switzerland

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# Participants

|  |  |
| --- | --- |
| Benke, Lajos | National Food Chain Safety OfficeDirectorate of Plant Protection, Soil Conservation and Agri-environmentPesticide Analytical Laboratory, VelenceH-2481 Velence, Ország út 23 |
| Carr, DenisGarvey, Jim | The Pesticide Control Laboratory, Backweston Laboratory Complex,Backweston,Celbridge,Co. Kildare,Ireland |
| Checa, Brenda | Ministerio de Desarrollo AgropecuarioDirección Nacional de Sanidad VegetalLaboratorio de Control de Calidad de PlaguicidasRío Tapia Vía Tocumen, Entrando por Harinas del Istmo 1.5 kmPanamá, City |
| Ciotea, Florentina | National Phytosanitary Authority 11, Voluntari Blv77910 VoluntariRomania |
| Förster, Rolf | BASF SEAPR/DP - Li721Speyerer Strasse 267117 LimburgerhofGermany |
| Grecu, Cornel | Alchimex 63 Alexandru Constantinescu Street. 011472 Bucharest-1, ROMANIA. |
| Haustein, Michael | Currenta GmbH & Co. OHGCHEMPARK Dormagen, A559 41538 DormagenGermany |
| Jacobsen, Eva  | Danish Technological InstituteLaboratory for Chemistry and MicrobiologyKongsvang Allé 298000 Aarhus CDenmark |
| Jiang, Qibai | Yangnong GLP Laboratory 39 Wenfeng Road, Yangzhou, Jiangsu, China |
| Karasali, Elen Balayiannis, George | Benaki Phytopathological Institute7 Stefanou Delta street14561 Kifissia Athens Greece |
| Krautstrunk, GerhardWagener, Peter | Bayer CropScience AGBCS AG-R&D-SMR-RT-AF- PC3Industriepark Hoechst65926 FrankfurtGermanyGermany |
| Kuranami, Yumiko | Agricultural Chemicals Inspection Station (ACIS), Food and Agricultural Materials Inspection Center (FAMIC) 2-772, Suzuki-Cho, Kodaira-Shi, Tokyo, Japan |
| Manso, Luis | Laboratorio Arbitral AgroalimentarioMinisterio de Agricultura y Pesca, Alimentación y Medio AmbienteAguarón, 13. Aravaca28023 Madrid. Spain |
| Mink, ChristianGähler, Bruno | Syngenta Crop Protection AGBreitenloh 54333 MünchwilenSwitzerland |
| Monisse, Isabelle | Agence Fédérale pour la Sécurité AlimentaireRue de Visé, 4954020 Wandre04/252.01.58Belgium |
| Novakova, Olga | Central Institute for Supervising and Testing in Agriculture, National ReferenceLaboratory, Department of Testing Plant Protection ProductsZemědělská 1a613 00 BrnoCzech Republic |
| Lecocq, Vanessa | Walloon Agricultural Research Centre (CRA-W)Agriculture and Natural Environment Department (D3)Plant Protection Products and Biocides Physico-chemistry and Residues Unit (U10)Carson BuildingRue du Bordia, 115030 GemblouxBelgium |
| Rezvani, Ahmad | Pesticide Formulations & Residues, State Chemist SectionMaryland Department of Agriculture50 Harry S. Truman ParkwayAnnapolis, MD 21401  |
| Schaller, Ulrich | Agroscope Institute for Plant Production Sciences IPS Plant Protection ChemistrySchloss 18820 WädenswilSwitzerland |
| Vinke, Claudia | Federal Office of Consumer Protection and Food Safety- Dept. Plant Protection Products -Messeweg 11/12D-38104 Braunschweig |
| Wang, Yue | Nutrichem Laboratory Co LtdChaoyang DistrictNo. 22 Maizidian StreetBeijingPR China |

Participants are listed in alphabetical sequence, lab numbers in the result tables were assigned in sequence of result receipt.

# Propiconazole, General Information

Chemical name: 1-[2-(2,4-dichlorophenyl)-4-propyl-1,3-dioxolan-2-yl-methyl]-1H-1,2,4-triazole

ISO common name: propiconazole

CAS-Nr.: 60207-90-1

Structure:



\* stereocenter

Molecular mass: 342.2

Empirical formula: C15H17Cl2N3O2

# Samples

In October 2017, Information Sheet No. 312 was sent out by the CIPAC Secretary inviting members to participate in a collaborative study on the determination of propiconazole as technical concentrate and in formulations by gas chromatography and flame Ionisation detection.

The participants who completed the study are listed in section 1.

Five test samples, the analytical standard and the internal standard were sent to the participants:

1. EC formulation (Sample A)

1. EC formulation (Sample B)
2. EC formulation (Sample C)
3. TC (Sample D)
4. TC (Sample E)

Propiconazole analytical standard, 96.3 % purity and docosan internal standard.

21 participants sent back their results in time.

# Method

## Scope

The content of Propiconazole as sum of its diastereomers is determined in technical concentrate and organic solvent based liquid formulated products.

## Principle

The Propiconazole content of the samples is determined by capillary gas chromatography (column with low-medium polarity stationary phase: (5% phenyl)methylpolysiloxane, e.g. DB-5, or equivalent, 15 m x 0.32 mm (i.d.), 1.0 μm film thickness), hydrogen carrier gas and flame ionisation detection. Quantification is done by internal standardization

## Procedure

The samples were analyzed twice at two different days. All test solutions were prepared freshly on day 2.

# Remarks of the Participants

Several participants made comments about the performance of the method and noted deviations from the method:

Laboratory 1 Hydrogen, DB-5 (15 m x 0.32 i.d., 1.0 µm), Solvent MIBK

Laboratory 2 Helium, DB-5 (15 m x 0.53 i.d., 1.0 µm), Solvent MIBK

Laboratory 3 Hydrogen, DB-5 (30 m x 0.32 i.d., 1.0 µm), Solvent MIBK

Laboratory 4 Hydrogen, TG-5 (30 m x 0.32 i.d., 1.0 µm), Solvent MIBK

Laboratory 5 Helium, DB-5 (30 m x 0.25 i.d., 1.0 µm), Solvent MIBK

Laboratory 6 Helium, DB-5 (15 m x 0.32 i.d., 1.0 µm), Solvent MIBK

,

Laboratory 7 Hydrogen, HP-5 (30 m x 0.32 i.d., 0.25 µm), Solvent MIBK

Laboratory 8 Helium, DB-5 (15 m x 0.32 i.d., 1.0 µm), Solvent MIBK

Laboratory 9 Hydrogen, CP-Sil 13CB (14% phenyl/86% dimethylpolysiloxane; 25 m x 0.32 i.d., 0.2 µm), Solvent MIBK, Flow 1,2 mL/min

Laboratory 10 Nitrogen, FactorFour, VF-1ms (100% dimethylpolysiloxane phase, 15 m x 0.25 i.d., 0.25 µm), Solvent MIBK, Flow 1 ml/min, Split 50:1

Laboratory 11 Helium, ZB-5 (30 m x 0.32 i.d., 0.25 µm), Solvent MIBK

Laboratory 12 Helium, DB-5 (15 m x 0.32 i.d., 1.0 µm), Solvent MIBK

Laboratory 13 Helium, CP-Sil 5CB (100% dimethylpolysiloxane, 15 m x 0.25 i.d., 0.25 µm), Flow 1 mL/min

Laboratory 14 Hydrogen, DB-5 (15 m x 0.32 i.d., 1.0 µm), Solvent MIBK

Laboratory 15 Helium, DB-5 (15 m x 0.32 i.d., 0.5 µm), Solvent MIBK

Laboratory 16 Helium, HP-5 (30 m x 0.32 i.d., 0.25 µm), Solvent MIBK

Laboratory 17 Nitrogen, Ultra 2 ((5%-Phenyl)-methylpolysiloxane, 25 m x 0.32 i.d., 0.52 µm), Solvent acetone for sample and reference solutions, isooctane for docosane)

Laboratory 18 Helium, HP-5 (30 m x 0.32 i.d., 0.25 µm), Solvent MIBK

Laboratory 19 Helium, DB-1 (30 m x 0.53 i.d., 1.5 µm), Solvent MIBK, Flow: 6.24 mL/min, Split 10:1 Gradient:

temp 1: 200 ºC, hold 0 min, ramp rate 10 ºC/min

temp 2: 230 ºC, hold 7 min, ramp rate 20 ºC/min

temp 3 280 °C, hold 5 min.

Laboratory 20 Hydrogen, DB-5 (15 m x 0.32 i.d., 1.0 µm), Solvent Acetone

Laboratory 21 Nitrogen, HP-5 (30 m x 0.32 i.d., 0.25 µm),

 Gradient (for TC):

temp 1 180ºC, hold 1 min, ramp rate 10 ºC/min

temp 2 230ºC, hold 4 min."

Gradient (for EC):

temp 1 180ºC, hold 1 min, ramp rate 10 ºC/min

temp 2 230ºC, hold 7 min, ramp rate 20 ºC/min

temp 3 280ºC, hold 0 min.

# Evaluation and Discussion

## Evaluation of the Quality of Data and Chromatograms

The data obtained from each of the laboratories were reviewed to determine if there were any significant deviations regarding the chromatography which might affect the analysis results.

Visual examination of the chromatograms showed no evidence for invalid data.

All other changes and observations noted by the participants were not expected to affect the analysis results significantly.

## Determination of Propiconazole

Results reported by the laboratories and the statistical evaluation are listed in tables 1-6 and displayed in figures 1-5.

The statistical evaluation of the data was done following the “Guidelines for CIPAC Collaborative Study Procedures for Assessment of Performance of Analytical Methods”, according to DIN ISO 5725. The data were examined for outliers and stragglers using Mandel’s k-statistics on the within-lab variance, followed by Mandel’s h-statistics on the lab means, and iterating where necessary. The tests were performed at an alpha level of 0.01 for outlier, and 0.05 for straggler.

Mandel’s k-statistics observed stragglers (marked with \* in Table 1) and outliers (marked with \*\* in Table 1) according to Mandel’s k-statistics were observed for the EC formulations as well as for the technical concentrate.

The Mandel’s h-statistic test identified outliers for the EC formulations as well as for the technical concentrate (marked with +/++ in Table 2). The isomeric ratio for technical concentrate samples is given in Table 3.

A comparison of the RSDR of this collaborative Study with the unmodified Horwitz equation showed that the relative reproducibility standard deviation (RSDR) is below the Horwitz value in all five samples even without elimination of stragglers and outliers (see Table 4). The RSDR s further improved if stragglers and outliers are improved (see Table 5 and 6). No more than 3 values have been removed per sample (Table 6) The validity of the results and the suitability of the analytical method is shown. This collaborative trial is acceptable.

**Table 1: Propiconazole assay in TC and Formulation (g/kg); results for each laboratory on day 1 and day 2**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|   | Propiconazole SAMPLE A | Propiconazole SAMPLE B | Propiconazole SAMPLE C | Propiconazole SAMPLE D | Propiconazole SAMPLE E |
|   | Day1 | Day2 | Day1 | Day2 | Day1 | Day2 | Day1 | Day2 | Day1 | Day2 |
| Laboratory 1 | 252.9 | 253.3 | 259.8 | 260.6 | 259.9 | 260.9 | 969.0 | 972.8 | 974.6 | 973.4 |
| Laboratory 2 | 245.6 | 250.6 | 251.1 | 255.4 | 250.6 | 254.6 | 955.9 | 961.2 | 958.0 | 954.4 |
| Laboratory 3 | 255.0 | 256.0 | 261.8 | 263.1 | 261.6 | 263.5 | 979.1 | 986.7 | 981.4 | 984.1 |
| Laboratory 4 | 255.7 | 253.9 | 262.2 | 261.9 | 263.6 | 261.4 | 970.4 | 962.3 | 977.2 | 967.8 |
| Laboratory 5 | 249.7 | 252.1 | 256.3 | 259.4 | 257.6 | 260.5 | 960.4 | 971.8 | 966.2 | 972.6 |
| Laboratory 6 | 253.9 | 254.7 | 259.9 | 261.6 | 261.2 | 262.6 | 973.8 | 977.6 | 975.5 | 983.2 |
| Laboratory 7 | 252.1 | 262.7\*\* | 254.7 | 264.8\*\* | 259.5 | 264.3 | 964.5 | 968.5 | 989.5 | 988.8 |
| Laboratory 8 | 253.9 | 254.0 | 261.0 | 260.1 | 260.9 | 262.4 | 958.7 | 973.2 | 989.9 | 979.4 |
| Laboratory 9 | 252.7 | 252.2 | 260.0 | 259.2 | 260.3 | 259.7 | 974.3 | 970.4 | 978.1 | 973.9 |
| Laboratory 10 | 257.8 | 260.4 | 262.2 | 261.1 | 262.0 | 263.6 | 979.4 | 979.5 | 979.5 | 978.4 |
| Laboratory 11 | 255.1 | 252.6 | 262.7 | 260.0 | 262.4 | 261.2 | 987.5 | 965.8\* | 982.7 | 970.7 |
| Laboratory 12 | 256.1 | 253.3 | 262.5 | 260.9 | 262.8 | 261.0 | 981.9 | 977.6 | 986.4 | 980.8 |
| Laboratory 13 | 257.0 | 255.8 | 259.6 | 253.7 | 263.2 | 258.3 | 995.5 | 992.3 | 1000.3 | 993.8 |
| Laboratory 14 | 253.0 | 253.9 | 260.4 | 261.9 | 261.0 | 262.2 | 969.3 | 977.4 | 979.0 | 977.9 |
| Laboratory 15 | 270.6 | 267.2 | 277.3 | 273.9 | 276.9 | 273.7 | 979.2 | 978.4 | 984.7 | 983.7 |
| Laboratory 16 | 255.3 | 254.5 | 262.2 | 257.3 | 262.1 | 257.6 | 975.1 | 974.4 | 981.5 | 984.5 |
| Laboratory 17 | 256.5 | 254.7 | 261.7 | 263.2 | 261.8 | 268.1\* | 985.1 | 986.8 | 979.3 | 978.1 |
| Laboratory 18 | 255.0 | 256.7 | 261.6 | 263.1 | 262.6 | 264.4 | 1020.4 | 985.7\*\* | 1021.3 | 992.3\*\* |
| Laboratory 19 | 250.8 | 254.7 | 261.8 | 264.7 | 263.3 | 265.5 | 989.2 | 988.9 |   |   |
| Laboratory 20 | 253.3 | 259.2 | 264.6 | 262.7 | 264.9 | 267.6 | 977.2 | 961.2 | 966.4 | 988.6\* |
| Laboratory 21 | 250.7 | 250.2 | 259.5 | 258.0 | 260.2 | 258.8 | 963.9 | 966.1 | 976.7 | 977.2 |

**\*** Mandel’s k-statistic straggler

\*\* Mandel’s k-statistic outlier

° Laboratory 19 did not receive Sample E

Table 2: Mean values

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|   | Propiconazole SAMPLE A | Propiconazole SAMPLE B | Propiconazole SAMPLE C | Propiconazole SAMPLE D | Propiconazole SAMPLE E |
|   |   |   |   |   |   |
| Laboratory 1 | 253.1 | 260.2 | 260.4 | 970.9 | 974.0 |
| Laboratory 2 | 248.1 | 253.3+ | 252.6 | 958.6+ | 956.2 |
| Laboratory 3 | 255.5 | 262.5 | 262.6 | 982.9 | 982.8 |
| Laboratory 4 | 254.8 | 262.0 | 262.5 | 966.4 | 972.5 |
| Laboratory 5 | 250.9 | 257.8 | 259.1 | 966.1 | 969.4 |
| Laboratory 6 | 254.3 | 260.7 | 261.9 | 975.7 | 979.3 |
| Laboratory 7 | 257.4 | 259.8 | 261.9 | 966.5 | 989.2 |
| Laboratory 8 | 253.9 | 260.6 | 261.7 | 965.9 | 984.6 |
| Laboratory 9 | 252.4 | 259.6 | 260.0 | 972.4 | 976.0 |
| Laboratory 10 | 259.1 | 261.6 | 262.8 | 979.5 | 979.0 |
| Laboratory 11 | 253.9 | 261.3 | 261.8 | 976.7 | 976.7 |
| Laboratory 12 | 254.7 | 261.7 | 261.9 | 979.7 | 983.6 |
| Laboratory 13 | 256.4 | 256.6 | 260.7 | 993.9 | 997.1 |
| Laboratory 14 | 253.4 | 261.1 | 261.6 | 973.4 | 978.4 |
| Laboratory 15 | 268.9++ | 275.6++ | 275.3++ | 978.8 | 984.2 |
| Laboratory 16 | 254.9 | 259.7 | 259.8 | 974.7 | 983.0 |
| Laboratory 17 | 255.6 | 262.5 | 265.0 | 985.9 | 978.7 |
| Laboratory 18 | 255.8 | 262.3 | 263.5 | 1003.0++ | 1006.8++ |
| Laboratory 19 | 252.8 | 263.3 | 264.4 | 989.0 | ° |
| Laboratory 20 | 256.3 | 263.6 | 266.2 | 969.2 | 977.5 |
| Laboratory 21 | 255.1 | 261.3 | 262.3 | 976.5 | 980.5 |

+ Mandel’s h-statistic straggler

++ Mandel’s h-statistic outlier

° Laboratory 19 did not receive Sample E

**Table 3: Isomeric ratio of TC samples**

|  |  |  |
| --- | --- | --- |
| Laboratory | SAMPLE D | SAMPLE E |
|  |  |  |
|  | Day1 | Day2 | Day1 | Day2 |
| 1 | 1.32 | 1.32 | 1.31 | 1.31 |
| 2 | 1.32 | 1.32 | 1.31 | 1.31 |
| 3 | 1.32 | 1.32 | 1.31 | 1.31 |
| 4 | 1.32 | 1.32 | 1.31 | 1.31 |
| 5 | 1.34 | 1.32 | 1.33 | 1.31 |
| 6 | 1.32 | 1.32 | 1.31 | 1.32 |
| 7 | 1.31 | 1.32 | 1.29 | 1.31 |
| 8 | 1.32 | 1.32 | 1.31 | 1.32 |
| 9 | 1.32 | 1.32 | 1.31 | 1.31 |
| 10 | 1.32 | 1.33 | 1.31 | 1.34 |
| 11 | 1.32 | 1.32 | 1.31 | 1.31 |
| 12 | 1.32 | 1.32 | 1.31 | 1.31 |
| 13 | 1.32 | 1.27 | 1.31 | 1.32 |
| 14 | 1.32 | 1.32 | 1.31 | 1.31 |
| 15 | 1.32 | 1.32 | 1.31 | 1.31 |
| 16 | 1.32 | 1.32 | 1.31 | 1.32 |
| 17 | 1.32 | 1.33 | 1.33 | 1.29 |
| 18 | 1.32 | 1.32 | 1.31 | 1.31 |
| 19 | 1.32 | 1.35 | ° |  |
| 20 | 1.32 | 1.30 | 1.30 | 1.28 |
| 21 | 1.31 | 1.31 | 1.31 | 1.31 |
|  |  |  |  |  |
| mean | 1.32 | 1.32 | 1.31 | 1.31 |
| min | 1.31 | 1.27 | 1.29 | 1.28 |
| max | 1.34 | 1.35 | 1.33 | 1.34 |

° Laboratory 19 did not receive Sample E

**Table 4: Summary of the statistical evaluation - no elimination of any
 outliers /stragglers**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | sample A | sample B | sample C | sample D | sample E |
| Xm | 254.9 | 261.2 | 262.1 | 975.9 | 980.3 |
| L | 21 | 21 | 21 | 21 | 20° |
| Sr | 2.39 | 2.38 | 0.88 | 7.85 | 6.91 |
| SL | 3.66 | 3.72 | 3.97 | 9.19 | 8.97 |
| SR | 4.37 | 4.42 | 4.06 | 12.08 | 11.32 |
| r | 6.69 | 6.67 | 2.45 | 21.99 | 19.34 |
| R | 12.24 | 12.36 | 11.37 | 33.84 | 31.69 |
| RSDr | 0.94 | 0.91 | 0.33 | 0.80 | 0.70 |
| RSDR | 1.72 | 1.69 | 1.55 | 1.24 | 1.15 |
| RSDR(Hor) | 2.46 | 2.45 | 2.45 | 2.01 | 2.01 |

° Laboratory 19 did not receive Sample E

**Table 5: Summary of the statistical evaluation - with elimination of Mandel’s
 k-statistic straggler/outlier**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | sample A | sample B | sample C | sample D | sample E |
| Xm | 254.8 | 261.2 | 262.0 | 974.4 | 979.0 |
| L | 20 | 20 | 20 | 19 | 18 |
| Sr | 1.79 | 1.84 | 0.88 | 4.91 | 3.98 |
| SL | 3.89 | 3.97 | 4.02 | 8.53 | 8.06 |
| SR | 4.28 | 4.37 | 4.12 | 9.84 | 8.99 |
| r | 5.00 | 5.16 | 2.45 | 13.74 | 11.15 |
| R | 12.00 | 12.25 | 11.53 | 27.56 | 25.16 |
| RSDr | 0.70 | 0.71 | 0.33 | 0.50 | 0.41 |
| RSDR | 1.68 | 1.67 | 1.57 | 1.01 | 0.92 |
| RSDR(Hor) | 2.46 | 2.45 | 2.45 | 2.01 | 2.01 |

Sample A: Results of Lab 7 eliminated; Sample B: Results of Lab 7 eliminated, Sample C: Results of Lab 17 eliminated, Sample D results of Lab 11 and 18 eliminated, Sample E, Results 18 and 20 removed

**Table 6: Summary of the statistical evaluation - with elimination of Mandel’s h and k
 statistic outliers / stragglers**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | sample A | sample B | sample C | sample D | sample E |
| Xm | 254.0 | 260.9 | 261.8 | 974.4 | 979.0 |
| L | 19 | 18 | 18 | 19 | 18 |
| Sr | 1.75 | 1.72 | 0.88 | 4.91 | 3.98 |
| SL | 2.13 | 1.40 | 1.58 | 8.53 | 8.06 |
| SR | 2.75 | 2.22 | 1.81 | 9.84 | 8.99 |
| r | 4.89 | 4.81 | 2.45 | 13.74 | 11.15 |
| R | 7.71 | 6.21 | 5.06 | 27.56 | 25.16 |
| RSDr | 0.69 | 0.66 | 0.33 | 0.50 | 0.41 |
| RSDR | 1.08 | 0.85 | 0.69 | 1.01 | 0.92 |
| RSDR(Hor) | 2.46 | 2.45 | 2.45 | 2.01 | 2.01 |

Sample A: Results of Lab 7 and 15 eliminated, Sample B Results of Lab 2, 7 and 15 eliminated, Sample C Results of 2, 15 and 17 eliminated, Sample D Samples 11 and 18 eliminated, Sample E Results of Lab 2, 18, and 20 eliminated

xm = overall sample mean

L = number of laboratories

sr  = repeatability standard deviation

RSDr = relative repeatability standard deviation

r = repeatability limit

sR = reproducibility standard deviation

RSDR = relative reproducibility standard deviation

R = reproducibility limit

sL = “pure” between laboratory standard deviation

RSDR(Hor) = relative reproducibility standard deviation (Horwitz equation)

**Figures 1 – 5 (all results)**

**Fig. 1:**

**Fig. 2:**

**Fig. 3:**

**Fig. 4:**

**Fig. 5:**

 Laboratory 19 did not receive Sample E

# Conclusions

21 different laboratories participated in this collaborative study. The results of the labs are given in Table 1-3, the statistical summary given in Table 4-6. The results are illustrated in figures 1 – 5.

Without elimination of any outliers or stragglers the between lab experimental Relative Reproducibility Standard Deviation (% RSDR ) is below the calculated acceptable value based on the Horwitz curve calculation (% RSDR (Hor)) in all samples.

The minimum number of considered results after elimination of stragglers and outliers was 18).

The mean isomeric ratio for sample D was 1.32 (min 1.27, max 1.35), for sample E it was 1.31 (min 1.28, max 1.34).

Two labs (17 and 20) have chosen Acetone as solvent for HSE or the lack of availability of Isobutylmethylketone, which seems to have no negative effect on the results. The solubility of Propiconazole in Acetone is good, the miscibility with the Product formulated in organic solvents is given.

Taking into account the relatively high number of participating laboratories a broad basis was given even after elimination of the outliers. Therefore, we consider this method to be suitable without further changes and recommend accepting it as a provisional CIPAC MT-method for the determination of propiconazole as sum of its diastereomers in technical concentrate and organic solvent based liquid formulated products.