## Section A7.1.3 Adsorption / Desorption screening test of

**Annex Point IIA7.7** **DIMETHYLAMINOSULFANILID (DMSA)**

| | | | Official use only |
|---|---|---|---|
| | | **1 REFERENCE** | |
| **1.1** | **Reference** | H. Sommer, 2001, Estimation of the Adsorption Coefficient ($K_{OC}$) of DMSA on Soil using High Performance Liquid Chromatography (HPLC), Bayer AG, Institute for Metabolism Research and Residue Analysis, Report No. MR-011/01 (unpublished), 2001-05-31 | |
| **1.2** | **Data protection** | Yes | |
| 1.2.1 | Data owner | Bayer Chemicals AG | |
| 1.2.2 | Companies with letter of access | - | |
| 1.2.3 | Criteria for data protection | Data submitted to the MS after 13 May 2000 on existing a.s. for the purpose of its entry into Annex I/IA / authorisation | |
| | | **2 GUIDELINES AND QUALITY ASSURANCE** | |
| **2.1** | **Guideline study** | Yes,<br>OECD Guideline for the Testing of Chemicals, Proposal for a new Guideline 121, "Estimation of the Adsorption Coefficient ($K_{OC}$) on Soil and on Sewage Sludge using High Performance Liquid Chromatography (HPLC)"(2001) | |
| **2.2** | **GLP** | Yes | |
| **2.3** | **Deviations** | No | |
| | | **3 MATERIALS AND METHODS** | |
| **3.1** | **Test material** | Dimethylaminosulfanilid (DMSA) | |
| 3.1.1 | Lot/Batch number | [redacted] | |
| 3.1.2 | Specification | | |
| 3.1.3 | Purity | [redacted] | |
| 3.1.4 | Further relevant properties | - | X |
| 3.1.5 | Method of analysis | HPLC, fitted with a pulse-free pump and a suitable detection device | |
| **3.2** | **Degradation products** | DMSA is the metabolite of the active substance dichlofluanid. | |
| **3.3** | **Reference substance** | Yes,<br>thirteen reference substances were used to determine an average capacity factor k`: Acetanilide, Aniline, Atrazine, Cyfluthrin, N,N-dimethylbenz-amide, DMST, Fenthion, Isoproturon, Linuron, Methiocarb, Phenan-trene, Pyrazophos and Triadimenol.<br>Sodium nitrate was used to determine the HPLC dead time ($t_0$). | X |
| 3.3.1 | Method of analysis for reference | HPLC | |

| | | | |
|---|---|---|---|
| | substance | | |
| **3.4** | **Testing procedure** | | X |
| 3.4.1 | Test system | HPLC (HP 1090) is performed on analytical columns packed with a commercially available cyanopropyl solid phase containing lipophilic and polar moieties (Zorbax CN, 5 µm, length = 250 mm, i.d. = 4.6 mm). As mobile phase methanol/0.01 M citrate-buffer pH 6.0 (55/45, v/v) was used.<br><br>As a result of partitioning between mobile and stationary phases the test substance is retarded. The dual composition of the stationary phase having polar and non-polar sites allows for interaction of polar and non-polar groups of a molecule in a similar way as is the case for organic matter in soil. This enables the relationship between the retention time on the column and the adsorption coefficient on organic matter to be established. | |
| 3.4.2 | Test solution and Test conditions | According to the guideline, the maximum concentration of the test substance should not exceed ½ the solubility in the solvent. Therefore the measurements were carried out at concentrations of approx. 5 mg/l.<br><br>Stock solution: 9.62 mg DMSA were weighed into a 10 ml volumetric flask and diluted to volume with methanol.<br><br>Standard solution: 0.1 ml of the stock solution was transferred into a 20 ml volumetric flask and diluted to volume with the mobile phase methanol/citrate buffer pH 6.0. The flask was shaken and ultrasonicated for one minute to dissolve the substance.<br><br>HPLC parameters:<br>Oven temperature: 40 °C, Injection volume: 250 µl, Flow rate: 1.5 ml/min and Run time: 30 min. | |
| **3.5** | **Calculations** | **$K_d$:** Distribution coefficient is defined as the ratio of equilibrium concentrations C of a dissolved test substance in a two phase system consisting of a sorbent (soil or sewage sludge) and an aqueous phase. It can be dimensionless or have the dimension mg/l.<br><br>**$K_{OC}$:** Distribution coefficient ($K_d$) or Freundlich adsorption coefficient ($K_f$) normalised to the organic carbon content ($f_{OC}$) of a sorbent. Depending on the dimensions of $K_d$ and $K_f$, $K_{OC}$ can be dimensionless or have the dimensions ml/g or µg/g organic matter. Using the HPLC estimation method the adsorption coefficient ($K_{OC}$) is deduced from the capacity factor (k`) using a calibration plot of log k` vs. log $K_{OC}$ of the selected reference substances.<br>$K_{OC}$ is an approximate indicator for the extent of adsorption between a substance and the sorbent and allows comparison to be made between different chemicals.<br><br>**k`:** Capacity factor = $\frac{t_R - t_0}{t_0}$<br><br>$t_R$ = HPLC retention time of test and reference substance (min)<br>$t_0$ = HPLC dead time (min) | X |

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| | | | |
|---|---|---|---|
| | | **log $K_{OC}$**: log $K_{OC}$ = Slope • log k` + intercept; Slope and intercept derived from the linear regression of the reference standards using $K_{OC}$. | |
| | | **4 RESULTS** | X |
| **4.1** | **Measurements** | HPLC retention time data for the reference substances and the test substance dimethylaminosulfanilid (DMSA) are given in table A7.1.3.1_1. The dead time ($t_0$) was determined to be 1.536 minutes using sodium nitrate. Variability of the retention times from repetitive injections was low, confirming HPLC system stability throughout the analysis period. | X |
| **4.2** | **Calculations** | Calculated adsorption parameter for the reference substances and the test substance dimethylaminosulfanilid (DMSA) are given in table A7.1.3.1_1. | |
| **4.3** | **Degradation product(s)** | DMSA is the metabolite of the active substance dichlofluanid. | |
| | | **5 APPLICANT'S SUMMARY AND CONCLUSION** | |
| **5.1** | **Materials and methods** | The adsorption coefficient $K_{OC}$ of dimethylaminosulfanilid (DMSA) on soil was estimated using High Performance Liquid Chromatography (HPLC). The test was performed according to the OECD Guideline for the testing of chemicals, Proposal for a new Guideline 121, "Estimation of the Adsorption Coefficient ($K_{OC}$) on Soil and on Sewage Sludge using High Performance Liquid Chromatography (HPLC)" (2001) in order to determine the mobility of DMSA in soil.<br><br>Thirteen reference standards of known $K_{OC}$ were analysed on a HPLC system to determine an average capacity factor k`. Sodium nitrate was used to determine the HPLC system dead time ($t_0$). A regression line was plotted with the determined k` values and the known $K_{OC}$ values (log k` vs. log $K_{OC}$).<br><br>The study shows no significant deviations from the test guideline. | |
| **5.2** | **Results and discussion** | Dimethylaminosulfanilid (DMSA) was analysed on the same HPLC system during the same sample sequence as the reference substances, and average k` values were determined. The $K_{OC}$ value for DMSA was estimated by interpolation from the reference substance regression line.<br><br>The linear regression of measured k` values against literature $K_{OC}$ values yielded a line with a slope of 4.41, an intercept of 2.46 and a correlation coefficient $R^2$ of 0.893. The estimated $K_{OC}$ value for DMSA is 53. | |
| **5.3** | **Conclusion** | Based on classification of Briggs (Proc. 7th British Insecticide and Fungicide Conference, Nottingham/UK, 83-86, 1973) and Verdam et al. (RIVM, Rapport No. 728473001, NL, 1988) for the estimation of the mobility of plant protectants in soil based on $K_d$ and/or $K_{OC}$-values, dimethylaminosulfanilid (DMSA) is to be classified as an intermediate mobile substance. | |
| 5.3.1 | Reliability | 1 | |
| 5.3.2 | Deficiencies | No | |

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| Evaluation by Competent Authorities | |
|---|---|
| | Use separate "evaluation boxes" to provide transparency as to the comments and views submitted |
| | **EVALUATION BY RAPPORTEUR MEMBER STATE (*)** |
| **Date** | 2/12/2004 |
| **Materials and Methods** | The applicant's version is acceptable with the following comments:<br>**3.1.4** The solubility of DMSA in the HPLC solvents (methanol and citrate buffer) appears to have been considered, but not presented here.<br>**3.3** The name of the reference substance DMST should be given in full.<br>**3.4** and **3.5** The numbering and headings within these sections are incorrect. According to the Technical Notes for Guidance Section 3.4 should have been 'Soil Types' (this section has been omitted, but does not affect the study report conclusions as the study does not include soil), Section 3.5 should have been 'Testing Procedure' (and the required information has been given as Section 3.4) and Section 3.6 should have been 'Test Performance' (this section has been omitted, but the necessary information is given in Section 5.1). |
| **Results and discussion** | The applicant's version is acceptable with the following comments:<br>**4.** The numbering and headings within this section do not follow the Technical Notes for Guidance, although the information required is given. Section 4.1 should be named 'Preliminary Test' but does include the necessary results, Sections 4.2 & 4.3 on screening tests are omitted and do not affect the study conclusions, and Sections 4.2 and 4.3 should be numbered 4.4 and 4.5 respectively.<br>**4.1** The order in which the samples were analysed, and whether blank runs were included, would have been useful to rule out any likelihood that the aniline chromatogram contained carryover from acetanilide (identical $t_r$). The consistency in rounding, to two decimal points, k' and mean log k' values seems to be ignored for DMSA compared to the reference compounds. |
| **Conclusion** | The applicant's version is acceptable. |
| **Reliability** | 1 |
| **Acceptability** | Acceptable |
| **Remarks** | All endpoints and data presented in the summary and tables have been checked against the original study and are correct. |
| | **COMMENTS FROM ...** |
| **Date** | *Give date of comments submitted* |
| **Materials and Methods** | *Discuss additional relevant discrepancies referring to the (sub)heading numbers and to applicant's summary and conclusion.*<br>*Discuss if deviating from view of rapporteur member state* |
| **Results and discussion** | *Discuss if deviating from view of rapporteur member state* |

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|---|---|
| **Conclusion** | *Discuss if deviating from view of rapporteur member state* |
| **Reliability** | *Discuss if deviating from view of rapporteur member state* |
| **Acceptability** | *Discuss if deviating from view of rapporteur member state* |
| **Remarks** | |

**Table A7.1.3.1_1 HPLC Retention Time Data and $K_{OC}$ Calculations**

| Substance | Mean Retention Time [min] | Mean Dead Time [min] | Mean k´ | Mean log k´ | Mean $K_{OC}$ | Mean log $K_{OC}$ |
|---|---|---|---|---|---|---|
| Sodium nitrate | - | 1.536 | - | - | - | - |
| Acetanilide | 2.485 | 1.536 | 0.62 | -0.21 | 17.8 | 1.25 |
| N,N-dimethyl-benzamide | 2.643 | 1.536 | 0.72 | -0.14 | 33.1 | 1.52 |
| Atrazine | 2.746 | 1.536 | 0.79 | -0.10 | 64.6 | 1.81 |
| Isoproturon | 2.966 | 1.536 | 0.93 | -0.03 | 72.4 | 1.86 |
| Aniline | 2.485 | 1.536 | 0.62 | -0.21 | 117 | 2.07 |
| Triadimenol | 3.044 | 1.536 | 0.98 | -0.01 | 251 | 2.40 |
| Linuron | 3.323 | 1.536 | 1.16 | 0.07 | 389 | 2.59 |
| Methiocarb | 3.012 | 1.536 | 0.96 | -0.02 | 1,259 | 3.10 |
| Fenthion | 4.013 | 1.536 | 1.61 | 0.21 | 2,042 | 3.31 |
| Pyrazophos | 4.010 | 1.536 | 1.61 | 0.21 | 4,467 | 3.65 |
| Phenantrene | 4.600 | 1.536 | 2.00 | 0.30 | 12,303 | 4.09 |
| Cyfluthrin | 7.496 | 1.536 | 3.88 | 0.59 | 64,300 | 4.81 |
| DMST | 2.693 | 1.536 | 0.76 | -0.12 | 76.25 | 1.88 |
| **DMSA** | **2.580** | **1.536** | **0.679** | **-0.168** | **52.95** | **1.724** |