

Validated Reference Material

Glyphosate, N-acetyl glyphosate, AMPA, N-acetyl AMPA, and glufosinate in soy bean cake

P1601-RMSoy

- Summary -



Please note: Reference material P1601-RMSoy is a validated control material and not a certified reference material. The reference material consists of 200 g of homogenised soy bean cake (powder), which is spiked with glyphosate, N-acetyl glyphosate, AMPA, N-acetyl AMPA and glufosinate. The reference material is validated in ring test P1601-RT with 8 laboratories. The spiked levels and the assigned values, calculated of the results of the participants of the ring test P1601-RT, are summarised in table 1.

The corresponding untreated soy bean cake (200 g, powder) is available as blank material (P1601-BLSoy).

Table 1. Descriptive statistics

| Parameter | Spiked level [mg/kg] | Assigned value [mg/kg] | Total No. of results |
|---------------------|-------------------------|---------------------------|----------------------|
| Glyphosate | 0.12 | 0.112 | 8 |
| AMPA | 0.13 | 0.115 | 8 |
| Glufosinate | 0.31 | 0.346 | 8 |
| N-acetyl-glyphosate | 0.098 | - | 1 |
| N-acetyl-AMPA | 0.086 | - | 2 |

Homogeneity testing

Seven samples of the test material are used for homogeneity testing. Each subsample is analysed in duplicate (table 2). The results confirm the homogeneous distribution of the analytes in the test material and the spiked levels.

Table 2. Results of the homogeneity testing

| Subsample No. | Extraction No. | Glyphosate [mg/kg] | AMPA [mg/kg] | Glufosinate [mg/kg] | N-acetyl-glyphosate [mg/kg] | N-acetyl-AMPA [mg/kg] |
|----------------------------------|----------------|--------------------|--------------|---------------------|-----------------------------|-----------------------|
| 1 | 1 | 0.16 | 0.18 | 0.45 | 0.11 | 0.11 |
| | 2 | 0.15 | 0.18 | 0.46 | 0.10 | 0.11 |
| 2 | 1 | 0.16 | 0.17 | 0.42 | 0.10 | 0.10 |
| | 2 | 0.14 | 0.18 | 0.44 | 0.10 | 0.10 |
| 3 | 1 | 0.15 | 0.17 | 0.42 | 0.10 | 0.10 |
| | 2 | 0.15 | 0.18 | 0.42 | 0.10 | 0.10 |
| 4 | 1 | 0.14 | 0.17 | 0.47 | 0.095 | 0.11 |
| | 2 | 0.14 | 0.18 | 0.43 | 0.092 | 0.10 |
| 5 | 1 | 0.15 | 0.17 | 0.45 | 0.095 | 0.10 |
| | 2 | 0.15 | 0.17 | 0.35 | 0.082 | 0.10 |
| 6 | 1 | 0.15 | 0.16 | 0.39 | 0.087 | 0.10 |
| | 2 | 0.13 | 0.16 | 0.36 | 0.085 | 0.10 |
| 7 | 1 | 0.13 | 0.15 | 0.35 | 0.11 | 0.086 |
| | 2 | 0.15 | 0.17 | 0.44 | 0.086 | 0.10 |
| Mean [mg/kg] | | 0.15 | 0.17 | 0.42 | 0.095 | 0.10 |
| Standard deviation [mg/kg] | | 0.0085 | 0.010 | 0.037 | 0.0074 | 0.0051 |
| Coefficient of variation [%] | | 5.8 | 6.0 | 8.9 | 7.8 | 5.1 |
| Spiked level [mg/kg] | | 0.12 | 0.13 | 0.31 | 0.098 | 0.086 |
| Recovery of the spiked level [%] | | 121 | 132 | 134 | 97 | 117 |

Stability testing

One subsample of test material was stored for stability testing at -18 °C in the dark. After the deadline of results reporting, the test sample is analysed in duplicate for all parameters (table 3). Recoveries of $\geq 83\%$ compared to the mean value of the homogeneity testing confirm the stability of all parameters throughout the whole testing period.

Table 3. Results of the stability testing

| Parameter | Spiked level [mg/kg] | Mean level at homogeneity testing [mg/kg] | Result of stability testing sample 1 [mg/kg] | Result of stability testing sample 2 [mg/kg] | Mean value of the stability testing [mg/kg] | Recovery compared to mean of the homogeneity testing [%] |
|---------------------|----------------------|---|--|--|---|--|
| Glyphosate | 0.12 | 0.146 | 0.127 | 0.124 | 0.126 | 86 |
| AMPA | 0.13 | 0.171 | 0.134 | 0.149 | 0.142 | 83 |
| Glufosinate | 0.31 | 0.417 | 0.325 | 0.37 | 0.348 | 83 |
| N-Acetyl glyphosate | 0.098 | 0.095 | 0.105 | 0.102 | 0.1035 | 109 |
| N-Acetyl AMPA | 0.086 | 0.101 | 0.084 | 0.087 | 0.0855 | 85 |