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# CERTIFICATE OF ANALYSIS

## REFERENCE MATERIAL No. IPO 692

# SIMAZINE

*6-chloro-$N^2$,$N^4$-diethyl-1,3,5-triazine-2,4-diamine*

$C_7H_{12}ClN_5$ (201.7) CAS REG. No. [*122-34-9*]

Series No. 3D/14 Valid to: December 2020

**Purity: 99.5 ± 0.3% (*m/m*)**

Unit: 0.25 g of crystalline solid in a brown glass vial.
Storage: The material should be stored in the original closed vial in a refrigerator at 5 °C ± 4 °C until it is required to use . Allow to equilibrate to ambient temperature before opening. It is intended for use as a reference material for the calibration of measuring equipment, for the evaluation of analytical procedures.

CONFIRMATION OF THE IDENTITY

The identity of the product was established by infrared spectroscopy and mass spectrometry.
The IR spectrum (KBr disc technique, scanning from 4000 to 400 $cm^{-1}$) of sample was compared with simazine literature spectrum [1,2]. Significant differences were not observed.
The mass spectrum (EI, 70 eV, temperature of ion source 250°C) was also recorded and no significant differences were observed in comparison with the literature spectrum [2,3].

DETERMINATION OF THE PURITY

Representative samples were drawn from the bulk material. The purity value was based on determinations made on these representative samples using the following methods:

- gas chromatography (GC)
- high performance liquid chromatography (HPLC)
- determination of sulphated ash
- determination of water content

The uncertainties quoted below are the half-width of a 95% confidence interval based on the standard deviation of the results obtained. The certified uncertainty is the combined uncertainty calculated according to the methodology described in[4] with a coverage factor k = 2. It corresponds to a confidence level of 95 %.

Determination by GC

Column I: fused-silica DB-1 (30 m x 0.53 mm i.d.), film thickness 3.0 µm.
Temperature conditions:

- Column: 40°C (5 min) to 180°C (5°/min), 180°C (10 min) to 240°C (5°/min)
- Injector: 230°C
- Detector (flame ionisation): 270°C

Carrier gas: 5 ml/min.
Injection volume: 1.0 µl of sample solutions in chloroform (0.06 – 0.10 % *m/V*).
The purity was calculated by peak area normalisation of the chromatograms. Five impurities were detected with a total concentration of 0.51 ± 0.05 % (*m/m*) (n = 4).

Column II: fused-silica DB-210 (30 m x 0.53 mm i.d.), film thickness 1.5 µm.
Temperature conditions:

- Column: 40 °C (3 min) to 230 °C (5°/min)
- Injector: 230°C
- Detector (flame ionisation): 270°C

Carrier gas: 6 ml/min.
Injection volume: 1.0 µl of sample solutions in chloroform (0.06 – 0.10% *m/V*).
The purity was calculated by peak area normalisation of the chromatograms. Four impurities were detected with a total concentration of 0.51 ± 0.01 % (*m/m*) (n = 4).

Determination by HPLC

- Column: Luna $C_{18}$, 5 µm, 250 x 4.6 mm i.d.
- Mobile phase: methanol + water (60:40, *V/V*)
- Flow rate: 0.8 ml/min
- UV detection at 215 nm.
- Injection volume: 20 µl of sample solutions in methanol (0.02 – 0.03% *m/V*).

The purity was determined assuming equal detector response factors for all constituents.
Two impurities were detected with a total concentration of 0.14 ± 0.01 % (*m/m*) (n = 4).

Determination of sulphated ash

Result: not more than 0.02% (*m/m*)

Determination of water content

By the Karl Fischer method. Result: 0.07% (*m/m*)

CONCLUSIONS

Based on the results of GC, HPLC method and other determinations the purity of this batch of simazine was assessed as 99.5 ± 0.3 % (*m/m*).

**The analytical measurements were coordinated by:**

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January 2014

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