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CERTIFICATE OF ANALYSIS

REFERENCE MATERIAL No. 923

2-CHLOROACETOPHENONE

C_8H_7ClO (154.59) CAS REG. No. [*532-27-4*]

Series No. 2A/18 Valid to: December 2022

Purity: 99.8 ± 0.1 % (*m/m*)

Unit: 0.10 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 ± 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 2-chloroacetophenone literature spectra [1]. Differences were not observed.
The mass spectrum of sample was also recorded (EI, 70 eV, temperature of ion source 250 °C) and no differences were observed in comparison with the literature spectrum [1,2].

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 techniques:

- gas chromatography (GC)
- high performance liquid chromatography (HPLC)

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 [3] 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 240 °C (5 °C/min)
- Injector: 200 °C
- Detector (flame ionisation): 270 °C

Carrier gas: 4.9 ml/min.
Injection volume: 0.2 µL of sample solutions in acetone (0.5-1.4 % *m/V*).
The purity was calculated by peak area normalisation method. Three impurities were detected with a total concentration of 0.08 ± 0.01 % (*m/m*) (n = 4).

Determination by HPLC

- Column: Kinetex Biphenyl 100 A, 5 µm, 250 x 4.6 mm i.d
- Mobile phase: methanol + 0.1 % aqueous solution of phosphoric acid (62 + 38, V/V)
- Flow rate: 1.0 mL/min
- UV detection at 206 and 245 nm.
- Injection volume: 10 µl of sample solutions in methanol (0.03-0.07 % *m/V*).

UV detection (206 nm) result: One impurity was detected with a total concentration of 0.11 ± 0.02 % (*m/m*) (n = 4).
UV detection (245 nm) result: One impurity was detected with a total concentration of 0.03 ± 0.01 % (*m/m*) (n = 4).

CONCLUSIONS

On the basis of the results of two independent quantitative techniques (GC and HPLC), the purity of this batch of 2-chloroacetophenone was assessed as 99.8 ± 0.1 % (*m/m*).

The analytical measurements were coordinated by:

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1. Integrated Spectral Data Base System for Organic Compounds, SDBS No.6099, National Institute of Advanced Industrial Science and Technology, Tsukuba, Ibaraki, Japan, http://sdbs.riodb.aist.go.jp
2. Database NIST 05, 2006 ID
3. NIST Special Publication 1012. An approach to the metrologically sound traceable assessment of the chemicals purity of organic reference materials David L. Duever and others, US Departament of Commerce USA, September 2004 , p.22