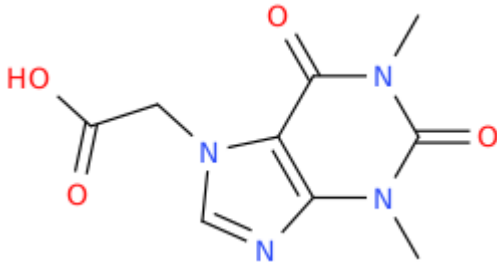


ANALYTICAL REPORT¹Acefylline (C₉H₁₀N₄O₄)

2-(1,3-dimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-7-yl)acetic acid

Remark – other active cpd. detected

Sample ID:	3029-21
Sample description:	powder - white
Sample type:	RM-reference material
Comments:	CAY Lot#0625215-2,
Date of entry (DD/MM/YYYY):	06/01/2022
web link	http://www.policija.si/apps/nfl_response_web/seznam.php

Substance identified-structure ² (base form)	
Systematic name:	2-(1,3-dimethyl-2,6-dioxo-2,3,6,7-tetrahydro-1H-purin-7-yl)acetic acid
Other names:	1,2,3,6-tetrahydro-1,3-dimethyl-2,6-dioxo-7H-purine-7-acetic acid; Acephylline; Acetyloxytheophylline; Carboxymethyltheophylline; NSC 52996; Theophylline-7-acetic Acid
Formula (per base form)	C ₉ H ₁₀ N ₄ O ₄
M _w (g/mol)	238,2
Salt form:	base
StdInChIKey (per base form)	HCYFGRCYSCXKNQ-UHFFFAOYSA-N
Other active cpd. detected	
Add.info (purity..)	≥98%

¹ Approved by: Dr. Sonja Klemenc² Created by OPSIN free tool: <http://opsin.ch.cam.ac.uk/> DOI: 10.1021/ci100384d

Report updates

date	comments (explanation)

Supporting information

Analytical technique:	applied	remarks
GC-MS (EI ionization)	+	NFL GC-RT (min): 8,31 BP(1): 194; BP(2): 109,BP(3) :193,
FTIR-ATR	+	direct measurement
GC-IR (condensed phase)	+	always as base form
HPLC-TOF	+	exact mass theoretical: 238,0702 / measured Δ ppm: 0,51

1. GC-MS (Agilent): GC-method is RT locked to tetracosane (9.258 min). Injection volume 1 μ l and split mode (1:50). Injector temperature: 280 °C. Chromatographic separation: on column HP1-MS (100% dimethylpolysiloxane), length 30 m, internal diameter 0.25 mm, film thickness 0.25 μ m. Carrier gas He: flow-rate 1.2 ml/min. GC oven program: 170 °C for 1 min, followed by heating up to 190 °C at rate 8 °C/min, then heating up to 293 °C at a rate of 18 °C/min, hold for 6.1 min, then heating at 50 °C/min up to 325 °C and finally 9.1 min isothermal. MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300 until 6 min) amu.

2. FTIR-ATR (Perkin Elmer): scan range 4000-400 cm^{-1} ; resolution 4 cm^{-1}

3. GC- (MS)-IR condensed phase (GC-MS (Agilent) & IR (Spectra analyses-Danny)

GC-method: Injection volume 1 μ l and split mode (1:5). Injector temperature 280 °C. Chromatographic separation as above (1). Split MS: IR = 1 : 9.

MSD source EI = 70 eV. GC-MS transfer line T= 235°C, source and quadropole temperatures 280°C and 180°C, respectively. Scan range m/z scan range: from 50 (30 until 6 min.) to 550 (300) amu.

IR (condensed (solid) phase): IR scan range 4000 to 650, resolution 4 cm^{-1} .

4. HPLC-TOF (Agilent): 6230B TOF with Agilent 1260 Infinity HPLC with binary pump, column: Zorbax Eclipse XDB-C18, 50 x 4.6 mm, 1.8 micron. Mobile phases (A) 0.1% formic acid and 1mM ammonium formate in water; (B) 0.1% formic acid in methanol (B). Gradient: starting at 5% B, changing to 40% B over 4 min, then to 70% over 2 min and in 5 min to 100%, hold 1 min and back to 5%, equilibration for 1.7 min. The flow rate: 1.0 ml/min; Injection volume 1 μ l. MS parameters: 2GHz, Extended Dynamic range mode to a maximum of 1700 amu, acquisition rate 1.30 spectra/sec. Sample ionisation: by Agilent Jet Stream technology (Dual AJS ESI). Ion source: positive ion scan mode with mass scanning from 82 to 1000 amu. Other TOF parameters: drying gas (N₂) and sheath temperature 325 °C; drying gas flow rate 6 l/min; sheath gas flow rate 8 l/min; nebulizer 25 psig; Vcap. 4000 V; nozzle 2000 V; skimmer 65 V; fragmentor 175 V and Octopole RF 750 V.

ANALYTICAL RESULTS

MS (EI)

Abundance

