Zolazepam

The Drug Enforcement Administration's Special Testing and Research Laboratory generated this monograph using structurally confirmed reference material.

1. GENERAL INFORMATION

**IUPAC Name:** 4-(2-fluorophenyl)-1,3,8-trimethyl-6,8-dihydropyrazolo[3,4-e][1,4]diazepin-7(1H)-one

**CAS#:** 31352-82-6 (base), 33754-49-3 (HCl)

**Synonyms:** 4-(2-fluorophenyl)-1,3,8-trimethyl-6H-pyrazolo[3,4-e][1,4]diazepin-7-one, Flupyrazazon

**Source:** DEA Reference Material Collection

**Appearance:** White powder

**UV\text{max}(nm):** Not determined

2. CHEMICAL AND PHYSICAL DATA

2.1 CHEMICAL DATA

<table>
<thead>
<tr>
<th>Form</th>
<th>Chemical Formula</th>
<th>Molecular Weight</th>
<th>Melting Point (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>C_{15}H_{15}N_{4}OF</td>
<td>286.31</td>
<td>Not Determined</td>
</tr>
<tr>
<td>HCl</td>
<td>C_{15}H_{15}N_{4}OF HCl</td>
<td>322.77</td>
<td>Not Determined</td>
</tr>
</tbody>
</table>
3. QUALITATIVE DATA

3.1 NUCLEAR MAGNETIC RESONANCE

Sample Preparation: Dilute analyte to ~12 mg/mL in D₂O containing TSP for 0 ppm reference and maleic acid as quantitative internal standard.

Instrument: 400 MHz NMR spectrometer
Parameters: Spectral width: at least containing -3 ppm through 13 ppm
Pulse angle: 90°
Delay between pulses: 45 seconds

¹HNMR: Zolazepam HCl; Lot# R067K0; D₂O; 400MHz
3.2 GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Sample Preparation: Dilute analyte ~4 mg/mL in MeOH

Instrument: Agilent gas chromatograph operated in split mode with MS detector
Column: HP-5 MS (or equivalent); 30m x 0.25 mm x 0.25 μm
Carrier Gas: Helium at 1.5 mL/min
Temperatures: Injector: 280°C MSD transfer line: 280°C
MS Source: 230°C MS Quad: 150°C
Oven program:
1) 100°C initial temperature for 1.0 min
2) Ramp to 280°C at 12 °C/min
3) Hold final temperature for 9.0 min

Injection Parameters: Split Ratio = 25:1, 1 μL injected
MS Parameters: Mass scan range: 30-550 amu Threshold: 250
Tune file: stune.u Acquisition mode: scan
Retention Time: 14.35 min

EI Mass Spectrum: Zolazepam HCl; Lot# R067K0
3.3 INFRARED SPECTROSCOPY (FTIR)

Instrument: FTIR with diamond ATR attachment (1 bounce)

Scan Parameters: Number of scans: 32
Number of background scans: 32
Resolution: 4 cm\(^{-1}\)
Sample gain: 1
Aperture: 150

FTIR ATR (Diamond 1 Bounce): Zolazepam HCl; Lot# R067K0