XLR-11

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

1. GENERAL INFORMATION

**IUPAC Name:** [1-(5-fluoropentyl)-1H-indol-3-yl](2,2,3,3-tetramethylcyclopropyl)methanone

**CAS #:** 1364933-54-9

**Synonyms:** 5-Fluoro-UR-144

**Source:** DEA Reference Material Collection

**Appearance:** White powder

**UV\textsubscript{max}:** 218.8, 246.9, 303.4

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\textsubscript{21}H\textsubscript{29}FNO</td>
<td>329</td>
<td>73.0</td>
</tr>
</tbody>
</table>

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3. QUALITATIVE DATA

3.1 NUCLEAR MAGNETIC RESONANCE

Method NMR CDCl₃

Sample Preparation: Sample is dissolved at ~20 mg/mL in CDCl₃ containing TMS for 0 ppm reference and dimethylfumarate as quantitative ISTD.

Instrument: Varian Mercury 400 MHz NMR spectrometer with proton detection probe

Parameters:
- Spectral width: at least containing -3 ppm through 13 ppm
- Pulse angle: 90°
- Delay between pulses: 45 seconds
- Number of scans (NT): 8
- Number of steady state scans: 0
- Oversampling: 4 or more
- Shimming: automatic gradient shimming of Z1-4 shims
- Phasing, Drift Correction: automatic or manual

1H NMR: XLR-11 Lot # N1-P12; CDCl₃; 400 MHz
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1H NMR: XLR-11 Lot # N1-P12; CDCl₃; 400 MHz

Dimethylfumarate

CHCl₃

1H NMR: XLR-11 Lot # N1-P12; CDCl₃; 400 MHz

0 0.05 0.10 0.15 0.20 0.25 0.30 0.35

0 8.5 8.0 7.5 7.0

Chemical Shift (ppm)

1 1 3

1 1 2

Chemical Shift (ppm)
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1H NMR: XLR-11 Lot # N1-P12; CDCl₃; 400 MHz
3.2 GAS CHROMATOGRAPHY/MASS SPECTROMETRY

Sample Preparation: Dilute analyte to ~1 mg/ml in MeOH.

Instrument: Gas chromatograph operated in split mode with MS detector

Column: DB-1 MS or equivalent; 30m x 0.25mm x 0.25μm

Carrier Gas: Helium at 1 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 300°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: 34-550 amu
- Threshold: 100
- Tune file: stune.u
- Acquisition mode: scan

Retention Time:
- XLR-11: 16.452 min; Rearrangement product: 16.641 min

GC/MS TIC: XLR-11 Lot # N1-P12
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**GC/MS Analytical Observation:**

The GC/MS TIC of XLR-11 shows two peaks with similar mass spectra (shown above). The major peak, having a retention time of 16.452 minutes, is XLR-11 while the other peak, with a retention time of 16.641 minutes, is a thermally induced rearrangement product of XLR-11. This rearrangement product is an artifact induced by the high temperatures of the GC injection port.

Several ion fragments have been observed to vary in abundance including 210, 212, 294, 309, and 329; however, there may be other variations that have not yet been observed. See the spectra below for illustrations of the phenomenon.
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3.3 INFRARED SPECTROSCOPY (FTIR)

**Instrument:** FTIR with diamond ATR attachment (3 bounce)

**Scan Parameters:**
- Number of scans: 32
- Number of background scans: 32
- Resolution: 4cm⁻¹
- Sample gain: 8
- Aperture: 150
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**FTIR Analytical Observation:** Polymorphism is suspected in this compound. See additional spectra below.
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4. ADDITIONAL RESOURCES

Forendex

Wikipedia