

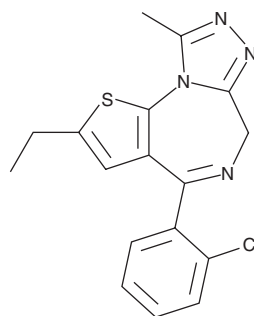
# CERTIFICATE of ANALYSIS



ACCREDITED  
ISO 17034 #AR-1774

## Etizolam (CRM)

Certified Reference Material



|                       |                                                                                                                                                                                                        |
|-----------------------|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Item No.:             | 20991                                                                                                                                                                                                  |
| Batch No.:            | 0676891                                                                                                                                                                                                |
| CAS Registry No.:     | 40054-69-1                                                                                                                                                                                             |
| Molecular Formula:    | C <sub>17</sub> H <sub>15</sub> ClN <sub>4</sub> S                                                                                                                                                     |
| Formula Weight:       | 342.80 amu                                                                                                                                                                                             |
| Expiry Date:          | 28APR2027 (valid from date of certification)                                                                                                                                                           |
| Supplied as:          | A 1 mg/ml (nominal) solution in methanol                                                                                                                                                               |
| Volume per Ampule:    | Not less than 1 ml. Ampules are overfilled.                                                                                                                                                            |
| Storage:              | Unopened at -20°C.                                                                                                                                                                                     |
| Safety:               | Refer to Safety Data Sheet                                                                                                                                                                             |
| Intended Use:         | For analytical testing purposes only, not intended for human or animal use.                                                                                                                            |
| Instructions for Use: | This product is designated for one-time use and should be used immediately after opening. It is advised that laboratories warm the vial to room temperature prior to opening and use measured volumes. |

### Certified Concentration · 1.000 mg/ml ± 0.007 mg/ml

Concentration is calculated based on product mass, solution mass, corrected purity, and density at 20°C. It is traceable to SI units through an unbroken chain of measurements. Uncertainty of concentration is expressed as an expanded uncertainty in accordance with ISO standards for Testing Laboratories and Reference Material Producers at the approximate 95% confidence interval using a coverage factor of k=2 and incorporates uncertainties from the corrected purity, solution preparation, homogeneity, and long- and short-term stability. Concentration was verified by comparison to an independently prepared calibration standard.

### Corrected Purity · 99.70% ± 0.52%

Corrected purity is determined as follows: Corrected Purity = [(100 - % LOD - % ROI)\*Chromatographic Purity/100] or [(100 - % KF - % RS - % ROI)\*Chromatographic Purity/100]. All measurement uncertainties are expressed as expanded uncertainties in accordance with ISO standards for Testing Laboratories and Reference Material Producers at the approximate 95% confidence interval using an appropriate coverage factor. Where applicable, optical rotation, chiral purity, and/or isotopic purity testing are performed to support the identification of the reference material, therefore the uncertainty is considered null.

Approval:

Title: ISO Quality Manager

Certification Date: 28APR2023

Cayman Chemical certifies that this standard meets the specifications stated in this certificate and warrants this product to meet the stated acceptance criteria through the expiration date when stored unopened as recommended.



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## CRM Assay

| Method Parameters |                                                                                                        |
|-------------------|--------------------------------------------------------------------------------------------------------|
| Cayman Method     | TST SD142                                                                                              |
| Column            | 4.6 x150 mm, 5 µm Gemini C18                                                                           |
| Mobile Phase      | A: 10:90:0.1% Acetonitrile:Water:Ammonium Formate<br>B: 90:10:0.1% Acetonitrile:Water:Ammonium Formate |
| Gradient          | <i>Time (min)</i> <i>%B</i><br>0-10            31.2-100%<br>10-13          100%<br>13.1-20       31.2% |
| Flow Rate         | 1 ml/min                                                                                               |
| Column Temp       | 30°C                                                                                                   |
| Wavelength        | UV monitored at 242 nm                                                                                 |

## Homogeneity

A minimum sample size of 1.0 µg was used to determine homogeneity. Homogeneity was determined by HPLC using ampules selected from a random sampling plan from early, middle, and late fill positions.

| %RSD  | Acceptance Criteria |
|-------|---------------------|
| 0.58% | ≤3%                 |

The recommended minimum quantity for use is 1.0 µg. Quantities below this have not been evaluated.

## Neat Material Quality Information (Item No.: 12063, Batch No.: 0612698)

| Qualifier                    | Method                       | Result          |
|------------------------------|------------------------------|-----------------|
| Chromatographic Purity, HPLC | Cayman Method TST SD142      | >99.90% ± 0.18% |
| Identity, LC-MS              | Cayman Method TST SD13, +ESI | 343.0 amu       |
| Identity, GC-MS              | Cayman Method TST SD12       | Conforms        |
| Identity, FTIR               | USP<854> (diamond ATR)       | Conforms        |
| % LOD                        | Cayman Method TST SD24       | <0.10% ± 0.45%  |
| % ROI                        | Cayman Method TST SD06       | <0.10% ± 0.19%  |
| Identity, NMR                | <sup>1</sup> H NMR           | Conforms        |

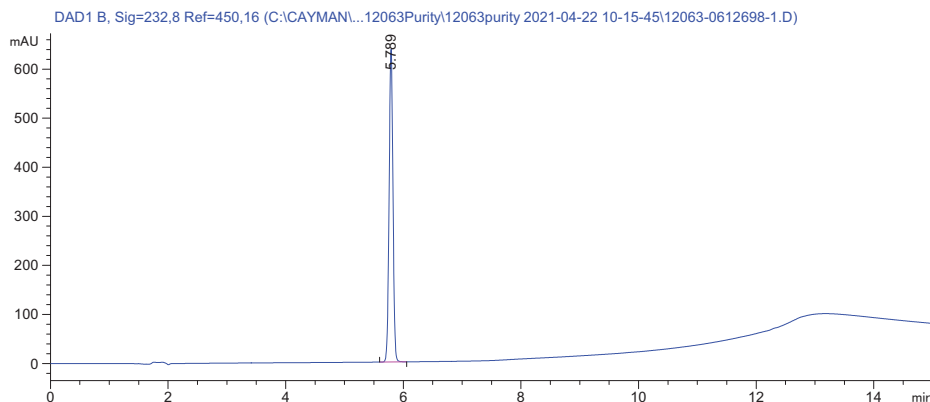
NMR and optical rotation (if applicable) are provided as supplemental information but are not within scope of ISO accreditation.  
Property values are traceable to SI units through an unbroken chain of measurements.

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## Supplemental Data (Neat Material)

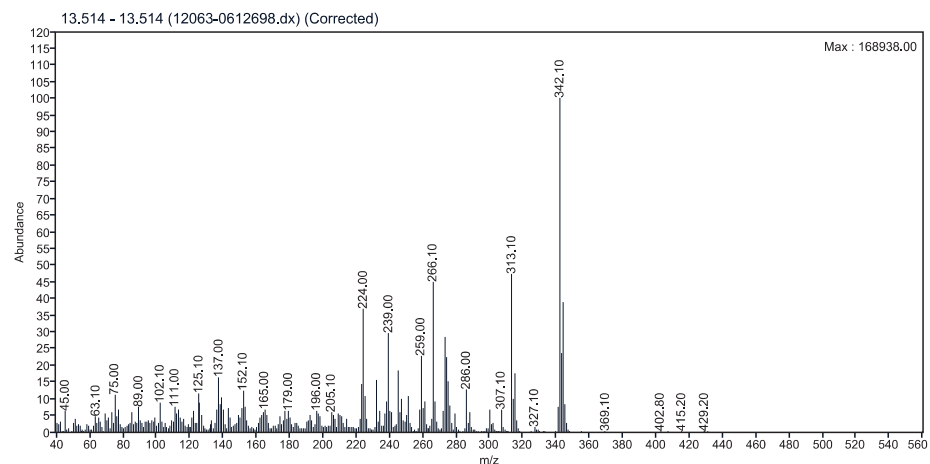
### HPLC-UV



### Conditions

| Instrument   | Agilent 1100/1200 Series                                                                                                                                                                                                                |            |    |      |           |       |      |         |       |
|--------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------|----|------|-----------|-------|------|---------|-------|
| Column       | 4.6 x 150 mm, 5 µm Gemini C18                                                                                                                                                                                                           |            |    |      |           |       |      |         |       |
| Mobile Phase | A: 10:90:0.1% Acetonitrile: Water:Ammonium Formate<br>B: 90:10:0.1% Acetonitrile: Water:Ammonium Formate                                                                                                                                |            |    |      |           |       |      |         |       |
| Gradient     | <table border="1"> <thead> <tr> <th>Time (min)</th> <th>%B</th> </tr> </thead> <tbody> <tr> <td>0-10</td> <td>31.2-100%</td> </tr> <tr> <td>10-13</td> <td>100%</td> </tr> <tr> <td>13.1-20</td> <td>31.2%</td> </tr> </tbody> </table> | Time (min) | %B | 0-10 | 31.2-100% | 10-13 | 100% | 13.1-20 | 31.2% |
| Time (min)   | %B                                                                                                                                                                                                                                      |            |    |      |           |       |      |         |       |
| 0-10         | 31.2-100%                                                                                                                                                                                                                               |            |    |      |           |       |      |         |       |
| 10-13        | 100%                                                                                                                                                                                                                                    |            |    |      |           |       |      |         |       |
| 13.1-20      | 31.2%                                                                                                                                                                                                                                   |            |    |      |           |       |      |         |       |
| Flow Rate    | 1 ml/min                                                                                                                                                                                                                                |            |    |      |           |       |      |         |       |
| Column Temp  | 30°C                                                                                                                                                                                                                                    |            |    |      |           |       |      |         |       |
| Wavelength   | UV monitored at 232 nm                                                                                                                                                                                                                  |            |    |      |           |       |      |         |       |

### GC-MS



### Conditions

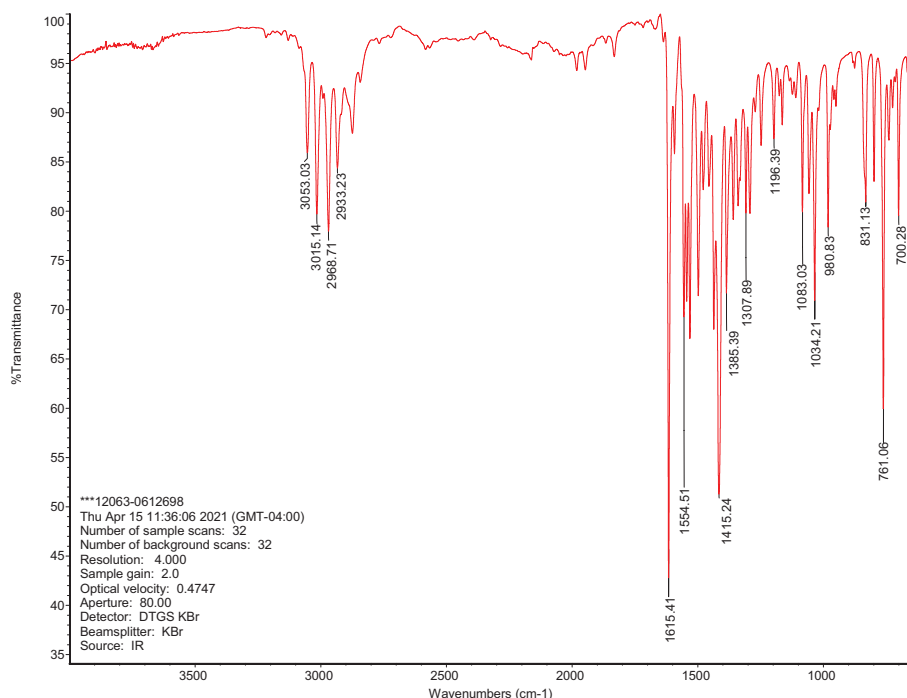
|                    |                                                                                 |
|--------------------|---------------------------------------------------------------------------------|
| Instrument         | Agilent GC MSD                                                                  |
| Column             | 30 m x 0.32 mm, 0.5 µm Rtx-5MS                                                  |
| Carrier Gas        | He                                                                              |
| Flow Rate          | 2 ml/min                                                                        |
| Inlet Temp         | 300°C                                                                           |
| Split Ratio        | 15:1                                                                            |
| Oven Program       | 50°C hold for 1 min, ramp to 300°C at 30°C per min, hold at 300°C to 15 minutes |
| Transfer Line Temp | 300°C                                                                           |
| Voltage            | 70ev EI MS                                                                      |
| Scan Range         | 40-650 m/z                                                                      |
| Tune File          | atune (custom)                                                                  |

Apex spectrum – background (1 min window in front of peak)

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## FTIR

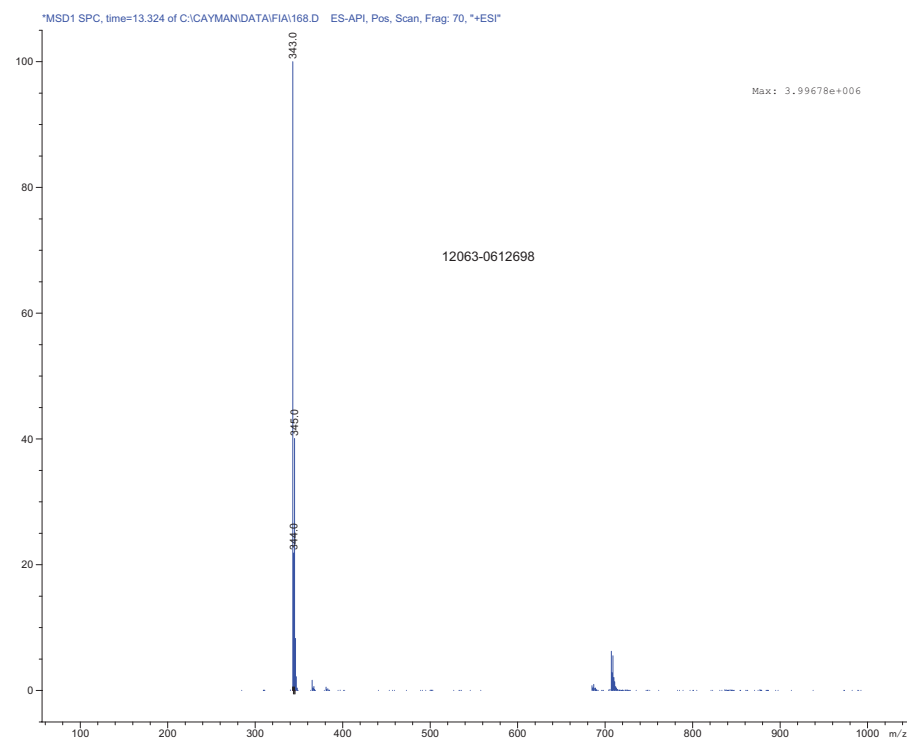


## Conditions

|            |                                                             |
|------------|-------------------------------------------------------------|
| Instrument | Thermo Nicolet iS10 FTIR / Diamond SmartATR (single bounce) |
| Scans      | 32 scans / 32 background scans                              |
| Range      | 650-4,000 cm <sup>-1</sup>                                  |
| Resolution | 4.000                                                       |

ATR and background corrected

## ESI-MS



## Conditions

|                      |                                      |
|----------------------|--------------------------------------|
| Instrument           | Agilent HPLC MSD                     |
| Mobile Phase         | 50:50:0.1 methanol/water/acetic acid |
| Flow Rate            | 0.5 ml/min                           |
| Ionization Mode      | +ESI                                 |
| Mass Range           | 100-1,000 m/z                        |
| Nebulizer            | 60 psi                               |
| Desolvation Gas      | 13 L/min                             |
| Desolvation Temp     | 350°C                                |
| Electrospray Voltage | 4kV                                  |

MS collected across peak width at half height

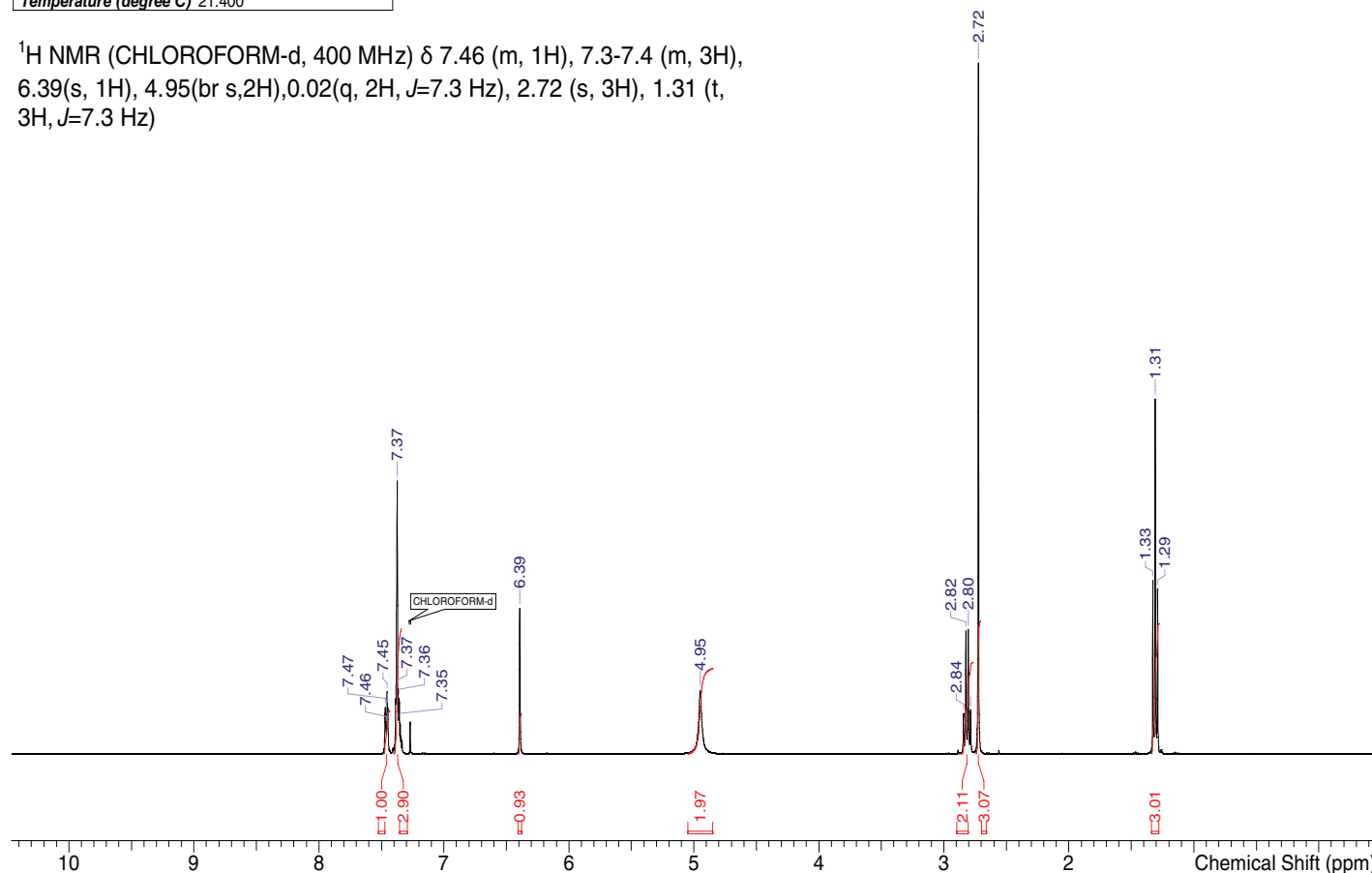
# CERTIFICATE of ANALYSIS



## NMR (not within scope of ISO accreditation)

|                        |                                                                                           |                      |    |                 |                      |
|------------------------|-------------------------------------------------------------------------------------------|----------------------|----|-----------------|----------------------|
| File Name              | \\sulfur\private\nmrdata\JEOL_2021\12063-0612698\12063-0612698_PROTON_02-Apr-2021-1-1.jdf |                      |    |                 |                      |
| Date                   | 02 Apr 2021 15:09:40                                                                      | Nucleus              | 1H | Frequency (MHz) | 399.7822             |
| Solvent                | CHLOROFORM-d                                                                              | Number of Transients | 16 | Origin          | JEOL ECZ400S Ca v600 |
| Temperature (degree C) | 21.400                                                                                    |                      |    |                 |                      |

$^1\text{H}$  NMR (CHLOROFORM-d, 400 MHz)  $\delta$  7.46 (m, 1H), 7.3-7.4 (m, 3H), 6.39(s, 1H), 4.95(br s, 2H), 0.02(q, 2H,  $J=7.3$  Hz), 2.72 (s, 3H), 1.31 (t, 3H,  $J=7.3$  Hz)



### Conditions

|                   |               |
|-------------------|---------------|
| <b>Instrument</b> | JEOL ECZ 400S |
| <b>Scans</b>      | 16 scans      |

### Stability

The effect of the components of stability on the combined standard uncertainty of the CRM property value are considered negligible unless indicated in stability studies.

### Short-Term Stability

No decrease in the property value was observed at ambient or 60°C after two weeks. This data supports shipping of this product at ambient temperature.

### Long-Term Stability

Long-term stability data confirmed four years stability at the -20°C storage temperature.

# CERTIFICATE of ANALYSIS



## Revision History

| Revision No. | Date      | Reason for Revision |
|--------------|-----------|---------------------|
| 01           | 28APR2023 | Initial version     |

## Disclaimers

### Material Safety Data

This material should be considered hazardous until information to the contrary becomes available. Do not ingest, swallow, or inhale. Do not get in eyes, on skin, or on clothing. Wash thoroughly after handling. This information contains some but not all of the information required for the safe and proper use of this material. Before use, review the complete Safety Data Sheet, which has been sent *via* email to your institution.

### Warranty and Limitation of Remedy

Cayman Chemical Company makes no warranty or guarantee of any kind, whether written or oral, expressed or implied, including without limitation, any warranty of fitness for a particular purpose, suitability and merchantability, which extends beyond the description of the chemicals hereof. Cayman warrants only to the original customer that the material will meet our specifications at the time of delivery.

Cayman will carry out its delivery obligations with due care and skill. Thus, in no event will Cayman have any obligation or liability, whether in tort (including negligence) or in contract, for any direct, indirect, incidental or consequential damages, even if Cayman is informed about their possible existence.

This limitation of liability does not apply in the case of intentional acts or negligence of Cayman, its directors or its employees.

Buyer's exclusive remedy and Cayman's sole liability hereunder shall be limited to a refund of the purchase price, or at Cayman's option, the replacement, at no cost to Buyer, of all material that does not meet our specification.

Said refund or replacement is conditioned on Buyer giving written notice to Cayman within thirty (30) days after arrival of the material at its destination. Failure of Buyer to give said notice within thirty (30) days shall constitute a waiver of Buyer of all claims hereunder with respect to said material.

For further details, please refer to our Warranty and Limitations of Remedy located on our website and in our catalog.

This Certificate shall not be reproduced except in full, without written approval from the Cayman Chemical ISO Quality Manager.

ISO CRT SD02 v 4.2

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