Brandt, SD, Kavanagh, PV, Westphal, F, Elliott, SP, Wallach, J, Colestock, T, Burrow, TE, Chapman, SJ, Stratford, A, Nichols, DE and Halberstadt, AL

Return of the lysergamides. Part II: Analytical and behavioural characterization of N6-allyl-6-norlysergic acid diethylamide (AL-LAD) and (2'S,4'S)-lysergic acid 2,4-dimethylazetidide (LSZ)

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Brandt, SD, Kavanagh, PV, Westphal, F, Elliott, SP, Wallach, J, Colestock, T, Burrow, TE, Chapman, SJ, Stratford, A, Nichols, DE and Halberstadt, AL (2016) Return of the lysergamides. Part II: Analytical and behavioural characterization of N6-allyl-6-norlysergic acid diethylamide (AL-LAD) and

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Return of the lysergamides. Part II: Analytical and behavioural characterization of N⁶-allyl-6-norlysergic acid diethylamide (AL-LAD) and (2’S,4’S)-lysergic acid 2,4-dimethylazetidide (LSZ)


ᵃ School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool L3 3AF, UK
ᵇ Department of Pharmacology and Therapeutics, School of Medicine, Trinity Centre for Health Sciences, St. James Hospital, Dublin 8, Ireland
ᶜ State Bureau of Criminal Investigation Schleswig-Holstein, Section Narcotics/Toxicology, Mühlenweg 166, D-24116 Kiel, Germany
ᵈ ROAR Forensics, Malvern Hills Science Park, Geraldine Road, WR14 3SZ, UK
ᵉ Department of Chemistry and Biochemistry, University of the Sciences, 600 South 43rd Street, Philadelphia, PA 19104, USA
ᶠ Department of Chemistry, University of Toronto, St. George Street, Toronto, ON M5S 3H6, Canada
ᵍ Isomer Design, 4103-210 Victoria Street, Toronto, ON, M5B 2R3, Canada
ʰ Synex Ltd, 67-68 Hatton Garden, N13 4BS, London, UK
ᵣ Division of Chemical Biology and Medicinal Chemistry, University of North Carolina, Genetic Medicine Building, 120 Mason Farm Road, Chapel Hill, NC 27599, USA
ᵢ Department of Psychiatry, University of California San Diego, 9500 Gilman Drive, La Jolla, CA 92093-0804, USA

* Correspondence to: Simon D. Brandt, School of Pharmacy and Biomolecular Sciences, Liverpool John Moores University, Byrom Street, Liverpool, L3 3AF, UK. E-Mail: s.brandt@ljmu.ac.uk
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LSZ Isomers (minor)
(spectra background subtracted)
Chemical Formula: $C_{22}H_{28}N_3O^+$
Exact Mass: 350.22269

Chemical Formula: $C_{18}H_{21}N_2O^+$
Exact Mass: 281.16484

Loss of ethyne

Chemical Formula: $C_{22}H_{28}N_3O^+$
Exact Mass: 350.22269

Loss of $N$

Chemical Formula: $C_{14}H_{12}N_2$•
Exact Mass: 208.09950

Chemical Formula: $C_{17}H_{17}N_2$•
Exact Mass: 249.13862

Chemical Formula: $C_{15}H_{15}N_2$•
Exact Mass: 223.12297

Chemical Formula: $C_{19}H_{23}N_3O$•
Exact Mass: 309.18356

Chemical Formula: $C_{18}H_{17}N_2O$•
Exact Mass: 277.13354

Chemical Formula: $C_{10}H_{17}N_2$•
Exact Mass: 249.13862
AL-LAD
Powder
HPLC-DAD
220 nm

LSZ
Powder
HPLC-DAD
220 nm
**Chiral HPLC analysis**

Chiral HPLC analyses were performed using the equipment described in the manuscript. Separation was obtained on a CHIRALPAK® AD-H column (250 × 4.6 mm, 5 µm) from Daicel Chemical Industries, Ltd. (Cedex, France). An isocratic mobile phase consisting of 9:1, 2-propanol: n-hexane was used. Powdered AL-LAD and LSZ, and LSZ reference material was dissolved individually in the isocratic mobile phase and diluted to a final concentration of 0.1 mg/mL. LSZ blotter was dissolved and extracted into the isocratic mobile phase following the procedure described in the manuscript. Injection volume was 10 µL, flow rate was 0.3 mL/min, and the column temperature was set at 25 °C. The total run time was 30 minutes.
AL-LAD hemitartrate

$^1$H-NMR (700 MHz)
d$_6$-DMSO
AL-LAD hemitartrate
$^1$H-NMR (700 MHz)
d$_6$-DMSO
AL-LAD hemitartrate
$^1$H-NMR (700 MHz)
d$_6$-DMSO

TA = Tartraric acid

$^{19}$ppm
AL-LAD hemitartrate
$^1$H-NMR (700 MHz)
d$_6$-DMSO
AL-LAD hemitartrate

\(^1\)H-NMR (700 MHz)
d\(\text{s}\)-DMSO

4\(\alpha\) + solvent
AL-LAD hemitartrate
$^{13}$C-NMR (176 MHz)
d$_6$-DMSO

TA = Tartaric acid
AL-LAD hemitartrate

HSQC (700 / 176 MHz)

$d_6$-DMSO

TA = Tartaric acid
LSZ tartrate

$^1$H-NMR (700 MHz)
d$_6$-DMSO
LSZ tartrate

$^1$H-NMR (700 MHz)
d$_6$-DMSO
LSZ tartrate

$^1$H-NMR (700 MHz)
d$_6$-DMSO

TA = Tartaric acid
LSZ tartrate
$^1$H-NMR (700 MHz)
d$_6$-DMSO
LSZ tartrate

$^1$H-NMR (700 MHz)

d$_6$-DMSO
LSZ tartrate

$^{13}$C-NMR (176 MHz)

d$_6$-DMSO