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# Crystal structures of new psychoactive substances 4-HO-DET and 4-AcO-DET

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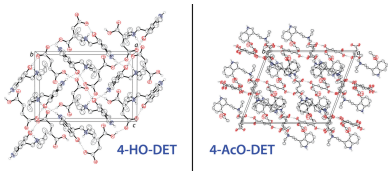
The solid-state structures of 4-hydroxy-*N*,*N*-diethyltryptammonium (4-HO-DET) hydrofumarate {systematic name: diethyl[2-(4-hydroxy-1*H*-indol-3-yl) ethyl]azanium 3-carboxyprop-2-enoate}, $C_{14}H_{21}N_2O^+\cdot C_4H_3O_4^-$, and 4-acetoxy-*N*,*N*-diethyltryptammonium (4-AcO-DET) fumarate/fumaric acid {systematic name: bis{[2-(4-acetoxy-1*H*-indol-3-yl)ethyl]diethylazanium} but-2-enedioate–(*E*)-butenedioic acid (1/1)}, $2C_{16}H_{23}N_2O^{2+}\cdot C_4H_2O_4^{2-}\cdot C_4H_4O_4$, were determined by single-crystal X-ray diffraction. The 4-HO-DET structure exhibits a single tryptammonium cation and a single hydrofumarate anion in the asymmetric unit. The ions are joined together by N—H···O and O—H···O hydrogen bonds in two-dimensional sheets lying in the (001) plane. The 4-AcO-DET structure exhibits a single tryptammonium cation, half of a fumarate dianion and half of a fumaric acid molecule in the asymmetric unit. The ions and molecules are joined together by N—H···O and O—H···O hydrogen bonds in an infinite three-dimensional network.

## 1. Chemical context

Psychedelics, entactogens and psychoplastogens have gained a great deal of interest recently in the treatment of major depressive disorder, generalised anxiety disorders, and treatment-resistant depression (Hoyer, 2025). Psilocybin (4-phosphoryloxy-*N*,*N*-dimethyltryptamine) is a natural product found in 'magic mushrooms', and is one traditional psychedelic that is being explored to treat mental health disorders. Psilocybin enzymatically hydrolyses to its active metabolite psilocin (4-hydroxy-*N*,*N*-dimethyltryptamine), which is the primary psychoactive compound found in 'magic mushrooms' due to its action as a potent agonist of the serotonin 2A receptor (Glatfelter, Pottie *et al.*, 2022). In 1959, Albert Hofmann reported the synthesis of the diethyl derivative of psilocybin, ethocybin (4-phosphoryloxy-*N*,*N*-diethyltryptamine), and the diethyl derivative of psilocin, ethocin (4-hydroxy-*N*,*N*-diethyltryptamine; 4-HO-DET) (Troxler *et al.*, 1959). In 1989, Jochem Gartz showed that both of these diethyl derivatives can be produced in mushrooms by introducing the unsubstituted tryptamine, *N*,*N*-diethyltryptamine, to the mycelium of *Psilocybe* mushrooms (Gartz, 1989). 4-HO-DET shows strong binding at the serotonin 2A receptor and is known to be psychoactive. There is also a synthetic prodrug of 4-HO-DET, 4-acetoxy-*N*,*N*-diethyltryptamine (4-AcO-DET), which was first synthesized by Albert Hofmann (Hofmann & Troxler, 1963). As a prodrug, 4-AcO-DET does not show binding at the serotonin 2A receptor (Glatfelter, Naeem *et al.*, 2023), but it demonstrates similar molar efficacy to 4-HO-DET in animal studies as it quickly hydrolyses to produce

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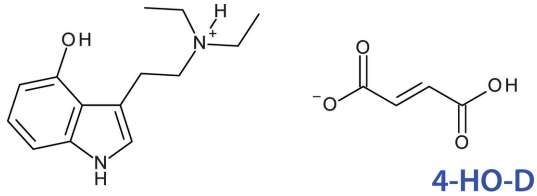
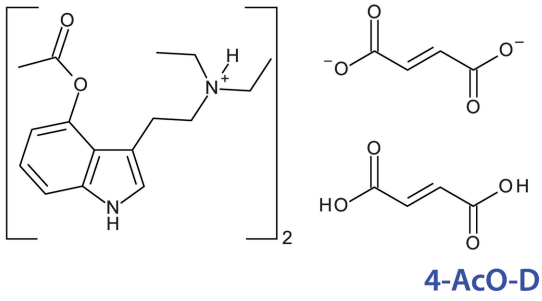
4-HO-DET (Klein *et al.*, 2021). Recently, 4-AcO-DET and 4-HO-DET have appeared in products for sale in US retail stores and online. Products marketed as psilocybin-containing 'magic mushroom' edibles frequently contain no psilocybin. In a recent analysis of twelve gummy and chocolate products sold as 'magic mushroom' edibles, psilocybin was not detected in any sample; the active constituents identified instead included synthetic tryptamines and other undisclosed compounds (van Breeman *et al.*, 2025).




Tryptomics, LLC analyzes commercial samples with a tryptamine assay that provides testing for an array of compounds often found in psychoactive substances. Across anonymously analyzed samples using HPLC-DAD technology following solvent and sonication extraction protocols 4-AcO-DET and/or 4-HO-DET were found in 87 samples. Twenty-five samples that were extracts and isolates showed the widest and highest range of 4-AcO-DET content (2.1–733.5 mg $g^{-1}$), with 4-HO-DET content reaching a maximum value of 981.8 mg $g^{-1}$. Sixty samples of infused edibles had maximum concentrations of 16.42 and 37.02 mg $g^{-1}$ for 4-AcO-DET and 4-HO-DET, respectively; these concentrations corresponded to edible package contents of 58.8 mg/package for 4-AcO-DET and 72.9 mg/package for 4-HH-DET. One inhalable sample contained 14.6 mg $g^{-1}$ of 4-AcO-DET. Reported orally actived doses of 4-AcO-DET are on the order of tens of milligrams (Shulgin & Shulgin, 1997). The per-package contents measured here therefore correspond to several such doses in a single retail unit. As these new psychoactive substances become more prevalent in the marketplace, the study of these materials and their properties becomes more important. As such the crystal structures of 4-hydroxy-*N*,*N*-diethyltryptamine as its hydrofumarate salt and 4-acetoxy-*N*,*N*-diethyltryptamine as a fumarate salt with a fumaric acid adduct are reported.

## 2. Structural commentary

The molecular structure of 4-HO-DET hydrofumarate is shown in Fig. 1. The asymmetric unit contains one 4-hydroxy-*N*,*N*-diethyltryptammonium ($C_{14}H_{21}N_2O^+$) cation and one hydrofumarate ($C_4H_3O_4^-$) anion. The indole ring of the cation is near planar with a r.m.s. deviation from planarity of 0.007 Å for the non-hydrogen atoms. The hydrofumarate anion is slightly less planar with a r.m.s. deviation from

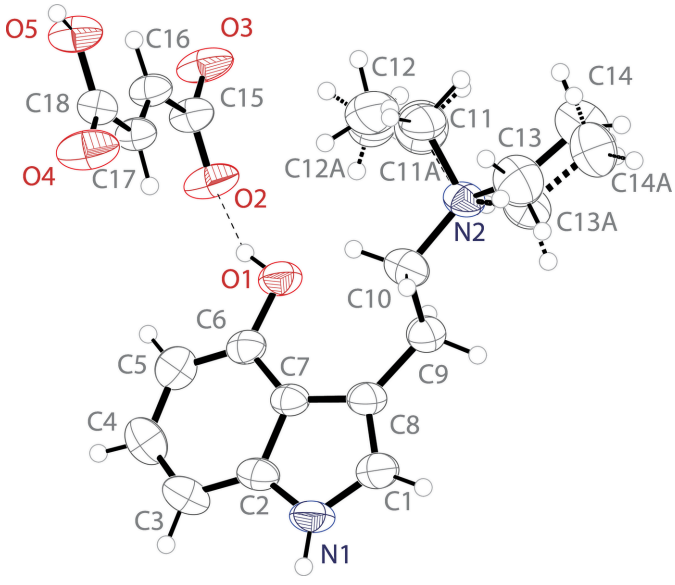



**Figure 1**
The molecular structure of 4-HO-DET hydrofumarate showing the atomic labeling. Displacement ellipsoids are drawn at the 50% probability level. Dashed bonds indicate a disordered component in the structure. Hydrogen bonds are shown as dashed lines.

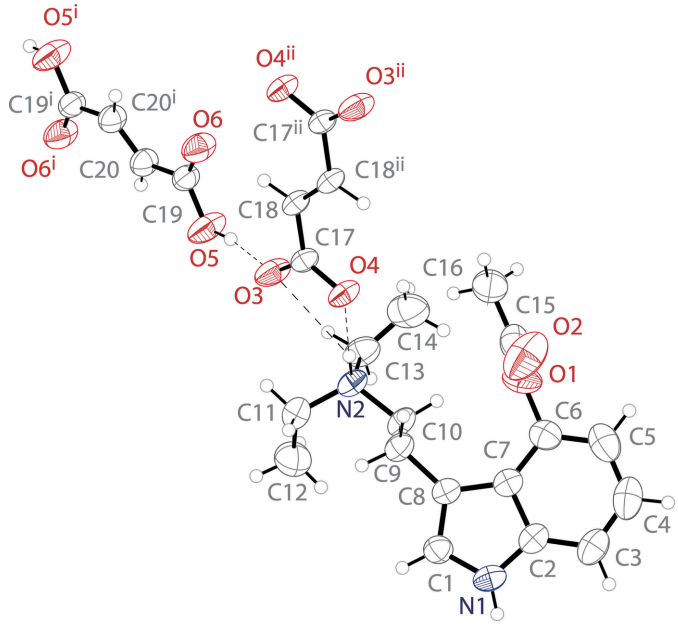



**Figure 2**
The molecular structure of 4-AcO-DET fumarate/fumaric acid showing the atomic labeling. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dashed lines. Symmetry codes: (i) $\frac{1}{2}-x, \frac{3}{2}-y, 1-z$; (ii) $1-x, 2-y, 1-z$.

**Table 1**
Hydrogen-bond geometry (Å, °) for 4-HO-DET.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O5—H5$A$···O3$^{i}$ | 0.90 (1) | 1.70 (2) | 2.559 (2) | 157 (4) |
| O1—H1···O2 | 0.87 (1) | 1.79 (2) | 2.607 (3) | 155 (4) |
| N1—H1$A$···O1$^{ii}$ | 0.87 (1) | 2.15 (1) | 3.011 (3) | 169 (3) |
| N2—H2···O3$^{iii}$ | 0.85 (1) | 1.96 (1) | 2.783 (3) | 165 (3) |

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (iii) $-x+1, -y+1, -z+1$.

**Table 2**
Hydrogen-bond geometry (Å, °) for 4-AcO-DET.

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O4$^{i}$ | 0.87 (1) | 2.12 (2) | 2.876 (3) | 145 (3) |
| N2—H2···O3 | 0.91 (1) | 2.65 (2) | 3.241 (2) | 124 (2) |
| N2—H2···O4 | 0.91 (1) | 1.91 (1) | 2.818 (2) | 177 (3) |
| O5—H5$A$···O3 | 0.95 (1) | 1.62 (1) | 2.558 (2) | 167 (3) |

Symmetry code: (i) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2}$.

planarity of 0.053 Å for non-hydrogen atoms. The ethylamino arm of the tryptamine is turned away from the indole plane with a C7—C8—C9—C10 torsion angle of 76.0 (3)° and the C8—C9—C10—N2 grouping has an *anti* conformation [torsion angle = 177.2 (2)°]. Each ethyl group is disordered over two orientations with C11 and C12 showing a 0.80 (3):0.20 (3) ratio and C13 and C14 showing a 0.82 (2):0.18 (2) ratio.

The molecular structure of 4-AcO-DET fumarate/fumaric acid is shown in Fig. 2. The asymmetric unit contains one 4-acetoxy-*N*,*N*-diethyltryptammonium ($C_{16}H_{23}N_2O_2^+$) cation, one half of a fumarate ($C_4H_2O_4^{2-}$) dianion, and one half of a fumaric acid ($C_4H_4O_4$) molecule. The indole ring system of the cation is near planar with a r.m.s. deviation from planarity of 0.013 Å. The ethylamino arm is turned away from the indole plane with a C7—C8—C9—C10 torsion angle of 73.5 (3)° and the C8—C9—C10—N2 grouping has an *anti* conformation [torsion angle = 174.0 (2)°]. The complete fumarate dianion is generated through inversion, and is near planar with a r.m.s. deviation from planarity of 0.025 Å. The complete fumaric acid dianion is generated similarly and only slightly less planar with a r.m.s. deviation of 0.044 Å.

## 3. Supramolecular features

In 4-HO-DET hydrofumarate, the hydroxy groups of the tryptammonium cations are connected to the hydrofumarate anion in the asymmetric unit via O1—H1···O2 hydrogen bonds. The carboxylic acid groups of the hydrofumarate anions are connected to another hydrofumarate anion via O5—H5$A$··O3 hydrogen bonds. The indole nitrogen atoms are connected with the hydroxy group of another tryptammonium cation through N1—H1···O1 hydrogen bonds. Finally, the ethylammonium nitrogen hydrogen bonds through N2—H2···O3 interactions with hydrofumarate anions. These hydrogen bonds are summarized in Table 1, and join the ions in the solid together into two-dimensional sheets lying in the (100) plane. The packing of these ions is shown in Fig. 3 (left).

In 4-AcO-DET fumarate/fumaric acid, the ethylammonium nitrogen has a bifurcated hydrogen bond with the two oxygen atoms of the fumaric acid as N2—H2···(O3,O4) interactions. The indole N atom hydrogen bonds to one of the O atoms of the fumaric acid via N1—H1···O4 interactions. The fumaric acid also hydrogen bonds to the fumarate dianion through the O5—H5$A$···O3 interaction. These hydrogen bonds are

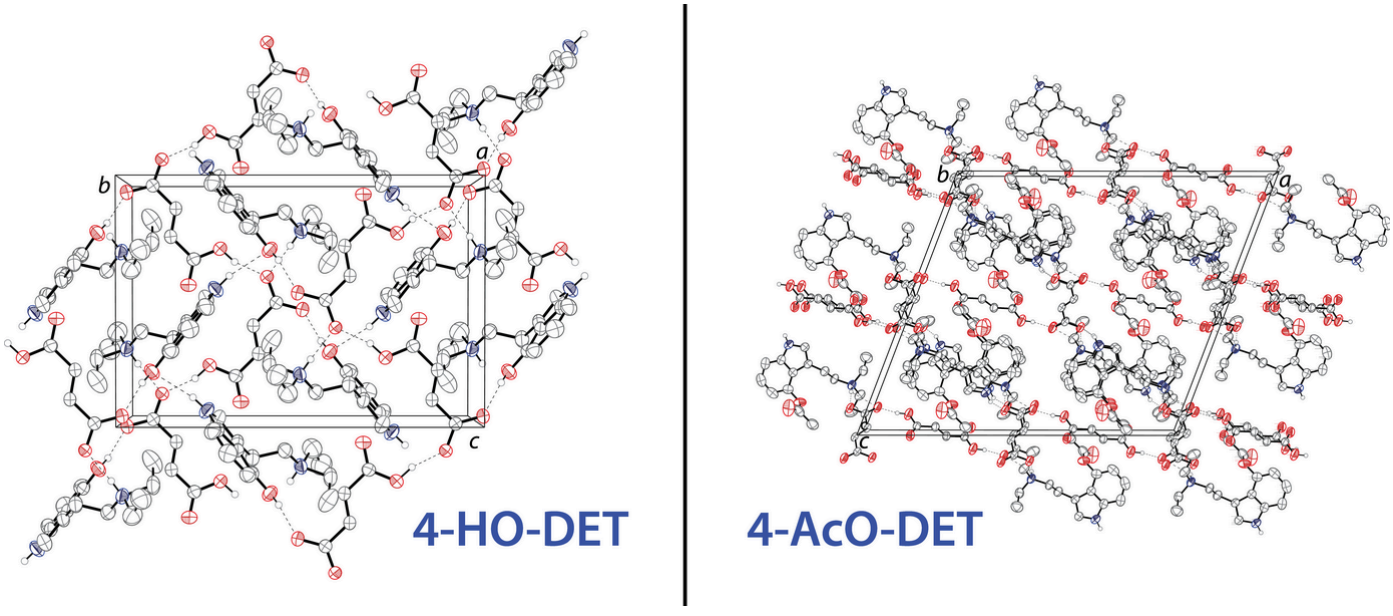



**Figure 3**
The crystal packing of 4-HO-DET hydrofumarate (left) viewed along the *a*-axis direction, and the crystal packing of 4-AcO-DET fumarate/fumaric acid (right) views along the *a*-axis direction. The hydrogen bonds (Tables 1 and 2) are shown as dashed lines. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen atoms not involved in hydrogen bonding are omitted for clarity. Only the major components of disordered residues are shown.

**Table 3**
Experimental details.

| | 4-HO-DET | 4-AcO-DET |
|---|---|---|
| Crystal data | | |
| Chemical formula | $C_{14}H_{21}N_2O^+\cdot C_4H_3O_4^-$ | $2(C_{16}H_{23}N_2O_2^+)\cdot C_4H_2O_4^{2-}\cdot C_4H_4O_4$ |
| $M_r$ | 348.39 | 390.43 |
| Crystal system, space group | Monoclinic, $P2_1/c$ | Monoclinic, $C2/c$ |
| Temperature (K) | 300 | 300 |
| $a, b, c$ (Å) | 10.9690 (9), 15.6452 (12), 10.6839 (7) | 24.410 (2), 8.4508 (7), 21.445 (2) |
| $\beta$ (°) | 92.084 (2) | 110.963 (3) |
| $V$ (Å$^3$) | 1832.3 (2) | 4131.1 (7) |
| $Z$ | 4 | 8 |
| Radiation type | Mo $K\alpha$ | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.09 | 0.09 |
| Crystal size (mm) | 0.42 × 0.3 × 0.12 | 0.18 × 0.15 × 0.12 |
| Data collection | | |
| Diffractometer | Bruker D8 Venture CMOS | Bruker D8 Venture CMOS |
| Absorption correction | Multi-scan (*SADABS*; Krause *et al.*, 2015) | Multi-scan (*SADABS*; Krause *et al.*, 2015) |
| $T_{min}$, $T_{max}$ | 0.580, 0.745 | 0.697, 0.745 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 46191, 3499, 2735 | 47661, 3525, 2420 |
| $R_{int}$ | 0.062 | 0.113 |
| (sin $\theta/\lambda)_{max}$ (Å$^{-1}$) | 0.612 | 0.588 |
| Refinement | | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.056, 0.164, 1.10 | 0.048, 0.122, 1.04 |
| No. of reflections | 3499 | 3525 |
| No. of parameters | 284 | 268 |
| No. of restraints | 67 | 9 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.34, −0.19 | 0.33, −0.17 |

Computer programs: *APEX4* and *SAINT* (Bruker, 2025), SHELXT2014 (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *OLEX2* (Dolomanov *et al.*, 2009) and *publCIF* (Westrip, 2010).

summarized in Table 2, and join the ions in the solid together into an infinite three-dimensional network. The packing of these ions is shown in Fig. 3 (right).

## 4. Database survey

There are 14 crystal structures of 4-hydroxytryptamines reported in the literature. These are the natural products psilocin, 4-hydroxy-*N*,*N*-dimethyltryptamine, as its freebase (Petcher & Weber, 1974; CCDC refcode PSILIN) and norpsilocin, 4-hydroxy-*N*-methyltryptamine, as its freebase and fumarate salt (Chadeayne *et al.*, 2020*c*; MULXAV, MULXEZ). Three synthetic variants of norpsilocin have been reported, 4-hydroxy-*N*-isopropyltryptamine (Laban *et al.*, 2023; YEYZOV), 4-hydroxy-*N*-*n*-propyltryptamine and 4-hydroxy-*N*-ethyltryptamine (Naeem *et al.*, 2026). The synthetic variants of psilocin that have been reported are 4-hydroxy-*N*,*N*-diisopropyltryptamine as its hydrofumarate salt (Naeem *et al.*, 2025; BUWSOF), 4-hydroxy-*N*,*N*-di-*n*-propyltryptamine as its chloride (Sammeta *et al.*, 2020; WAMGEA) and fumarate (Chadeayne, Pham *et al.*, 2019*b*; WUCGAF) salts, and 4-hydroxy-*N*-methyl-*N*-isopropyltryptamine as its hydrofumarate (Chadeayne, Pham *et al.*, 2019*a*; RONSUL) and fumarate (Chadeayne, 2020*b*; TUFQAP) salts. There are also four quaternary 4-hydroxytryptamines that are reported as their iodide salts in 4-hydroxy-*N*,*N*,*N*-trimethyltryptammonium (Chadeayne *et al.*, 2020*a*; XUXFAA), 4-hydroxy-*N*,*N*-dimethyl-*N*-ethyltryptammonium, 4-hydroxy-*N*,*N*-dimethyl-*N*-*n*-propyltryptammonium and 4-hydroxy-*N*,*N*-dimethyl-*N*-isopropyltryptammonium (Glatfelter, Pham *et al.*, 2022; EDOYIJ, EDOYUV, EDOZIK).

There are 11 crystal structures of 4-acetoxytryptamines reported in the literature. There is one monoalkyltryptamine with 4-acetoxy-*N*-methyltryptamine (Glatfelter, Pottie *et al.*, 2022). There are four quaternary 4-acetoxytryptamines reported as their iodide salts in 4-acetoxy-*N*,*N*,*N*-trimethyltryptammonium (Chadeayne *et al.*, 2020*a*; XUXDUS), 4-acetoxy-*N*,*N*-dimethyl-*N*-ethyltryptammonium, 4-acetoxy-*N*,*N*-dimethyl-*N*-*n*-propyltryptammonium and 4-acetoxy-*N*,*N*-dimethyl-*N*-isopropyltryptammonium (Glatfelter, Pham *et al.*, 2022; EDOZEG, EDOYOP, EDOZAC). There are six dialkyltryptamines with 4-acetoxy-*N*-ethyl-*N*-*n*-propyltryptamine as a fumarate/fumaric acid compound (Pham *et al.*, 2023; BIYKED), 4-acetoxy-*N*-methyl-*N*-ethyltryptamine as its hydrofumarate salt, 4-acetoxy-*N*-methyl-*N*-allyltryptamine as its hydrofumarate salt, 4-acetoxy-*N*,*N*-diallyltryptamine as a fumarate/fumaric acid compound (Pham *et al.*, 2021; OJIQIK, OJIQOQ, OJIQUW), and 4-acetoxy-*N*,*N*-dimethyltryptamine as both fumarate (Chadeayne, Golen *et al.*, 2019*b*; XOFDOO) and hydrofumarate (Chadeayne, Golen *et al.*, 2019*a*; HOCJUH) salts. The structure of 4-acetoxy-*N*,*N*-diallyltryptammonium fumarate/fumaric acid is the most similar to the reported structure of 4-acetoxy-*N*,*N*-diethyltryptammonium fumarate/fumaric acid, demonstrating the same space group, general packing, and similar unit-cell parameters.

## 5. Synthesis and crystallization

Single crystals of 4-hydroxy-*N*,*N*-diethyltryptammonium hydrofumarate suitable for X-ray analysis were obtained from the slow evaporation of an ethanolic solution of a commercial sample (ChemLogix). Single crystals of 4-acetoxy-*N*,*N*-diethyltryptammonium fumarate/fumaric acid suitable for X-ray analysis were grown from the slow evaporation of a methanol/acetone solution of a commercial sample (The Indole Shop).

## 6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. The H atoms bonded to C atoms were included in the refinement using riding models. The H atoms bonded to heteroatoms (N and O) were located in difference-Fourier maps and were refined isotropically with the use of DFIX commands. The ethyl groups in 4-HO-DET are disordered over two orientations and refined with the use of DFIX, SADI and DELU commands resulting in occupancies for C11 and C12 of 0.80 (3):0.20 (3), and for C13 and C14 of 0.82 (2):0.18 (2).

## Acknowledgements

Financial statements and conflicts of interest: The study was funded by CaaMTech, Inc. ARC reports an ownership interest in CaaMTech, Inc., which owns US and worldwide patent applications covering new tryptamine compounds, compositions, formulations, novel crystalline forms, and methods of making and using the same.

## Funding information

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# supporting information

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## Crystal structures of new psychoactive substances 4-HO-DET and 4-AcO-DET

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### Computing details

**Diethyl[2-(4-hydroxy-1*H*-indol-3-yl)ethyl]azanium 3-carboxyprop-2-enoate (4-HO-DET)**

*Crystal data*

$C_{14}H_{21}N_2O^+·C_4H_3O_4^-$
$M_r$ = 348.39
Monoclinic, $P2_1/c$
$a$ = 10.9690 (9) Å
$b$ = 15.6452 (12) Å
$c$ = 10.6839 (7) Å
$\beta$ = 92.084 (2)°
$V$ = 1832.3 (2) Å$^3$
$Z$ = 4
$F(000)$ = 744
$D_x$ = 1.263 Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda$ = 0.71073 Å
Cell parameters from 9923 reflections
$\theta$ = 2.6–25.7°
$\mu$ = 0.09 mm$^{-1}$
$T$ = 300 K
BLOCK, colourless
0.42 × 0.3 × 0.12 mm

*Data collection*

Bruker D8 Venture CMOS diffractometer
$\varphi$ and $\omega$ scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.580, $T_{max}$ = 0.745
46191 measured reflections
3499 independent reflections
2735 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.062
$\theta_{max}$ = 25.8°, $\theta_{min}$ = 2.6°
$h$ = −13→13
$k$ = −19→19
$l$ = −13→12

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.164
$S$ = 1.10
3499 reflections
284 parameters
67 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 1.6679P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.34 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.19 e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ | Occ. (<1) |
|---|---|---|---|---|---|
| O1 | 0.34358 (19) | 0.41479 (12) | 0.71108 (17) | 0.0522 (5) | |
| O2 | 0.2169 (2) | 0.49336 (11) | 0.53482 (17) | 0.0599 (6) | |
| O3 | 0.2145 (2) | 0.59686 (11) | 0.39464 (16) | 0.0629 (6) | |
| O4 | 0.1874 (2) | 0.67378 (13) | 0.93001 (17) | 0.0713 (7) | |
| O5 | 0.1643 (2) | 0.76553 (11) | 0.77401 (17) | 0.0558 (5) | |
| N1 | 0.4000 (2) | 0.23712 (15) | 1.0502 (2) | 0.0520 (6) | |
| N2 | 0.6971 (2) | 0.50168 (13) | 0.7990 (2) | 0.0460 (5) | |
| C1 | 0.5095 (3) | 0.26678 (17) | 1.0111 (3) | 0.0541 (7) | |
| H1B | 0.584988 | 0.252123 | 1.047254 | 0.065* | |
| C2 | 0.3081 (2) | 0.27094 (15) | 0.9750 (2) | 0.0435 (6) | |
| C3 | 0.1832 (3) | 0.2587 (2) | 0.9772 (3) | 0.0592 (8) | |
| H3 | 0.148887 | 0.222588 | 1.035325 | 0.071* | |
| C4 | 0.1123 (3) | 0.3021 (2) | 0.8902 (3) | 0.0630 (8) | |
| H4 | 0.027991 | 0.295863 | 0.890565 | 0.076* | |
| C5 | 0.1633 (3) | 0.35544 (18) | 0.8008 (3) | 0.0520 (7) | |
| H5 | 0.112732 | 0.383251 | 0.742156 | 0.062* | |
| C6 | 0.2868 (2) | 0.36705 (15) | 0.7990 (2) | 0.0410 (6) | |
| C7 | 0.3631 (2) | 0.32505 (14) | 0.8882 (2) | 0.0392 (5) | |
| C8 | 0.4928 (2) | 0.32060 (15) | 0.9123 (2) | 0.0448 (6) | |
| C9 | 0.5922 (2) | 0.36298 (16) | 0.8423 (3) | 0.0489 (6) | |
| H9A | 0.669315 | 0.335311 | 0.863418 | 0.059* | |
| H9B | 0.575828 | 0.356207 | 0.752999 | 0.059* | |
| C10 | 0.6016 (3) | 0.45621 (17) | 0.8730 (3) | 0.0530 (7) | |
| H10A | 0.621549 | 0.462623 | 0.961710 | 0.064* | |
| H10B | 0.523007 | 0.483028 | 0.855991 | 0.064* | |
| C11 | 0.6538 (5) | 0.5871 (3) | 0.7564 (8) | 0.0597 (17) | 0.80 (3) |
| H11A | 0.628697 | 0.619802 | 0.828136 | 0.072* | 0.80 (3) |
| H11B | 0.720520 | 0.617411 | 0.719005 | 0.072* | 0.80 (3) |
| C12 | 0.5519 (7) | 0.5809 (6) | 0.6654 (11) | 0.091 (2) | 0.80 (3) |
| H12A | 0.542057 | 0.634329 | 0.622087 | 0.136* | 0.80 (3) |
| H12B | 0.478657 | 0.567686 | 0.707897 | 0.136* | 0.80 (3) |
| H12C | 0.567983 | 0.536465 | 0.606158 | 0.136* | 0.80 (3) |
| C11A | 0.638 (3) | 0.569 (2) | 0.717 (4) | 0.090 (9) | 0.20 (3) |
| H11C | 0.614850 | 0.614272 | 0.773652 | 0.108* | 0.20 (3) |
| H11D | 0.703801 | 0.592688 | 0.668931 | 0.108* | 0.20 (3) |
| C12A | 0.537 (3) | 0.561 (3) | 0.629 (3) | 0.082 (8) | 0.20 (3) |
| H12D | 0.482185 | 0.608488 | 0.638077 | 0.124* | 0.20 (3) |
| H12E | 0.494331 | 0.508715 | 0.643874 | 0.124* | 0.20 (3) |
| H12F | 0.566036 | 0.560781 | 0.545039 | 0.124* | 0.20 (3) |
| C13 | 0.8072 (16) | 0.5348 (14) | 0.869 (2) | 0.078 (9) | 0.18 (2) |
| H13A | 0.782496 | 0.582344 | 0.920944 | 0.093* | 0.18 (2) |
| H13B | 0.837871 | 0.490228 | 0.925030 | 0.093* | 0.18 (2) |
| C14 | 0.906 (4) | 0.563 (3) | 0.793 (4) | 0.088 (10) | 0.18 (2) |
| H14A | 0.977771 | 0.571574 | 0.845499 | 0.133* | 0.18 (2) |
| H14B | 0.883418 | 0.615919 | 0.752859 | 0.133* | 0.18 (2) |

| | | | | | |
|---|---|---|---|---|---|
| H14C | 0.921327 | 0.520605 | 0.731116 | 0.133* | 0.18 (2) |
| C13A | 0.8131 (4) | 0.5074 (5) | 0.8827 (5) | 0.0603 (16) | 0.82 (2) |
| H13C | 0.797340 | 0.544268 | 0.953288 | 0.072* | 0.82 (2) |
| H13D | 0.832192 | 0.450964 | 0.915395 | 0.072* | 0.82 (2) |
| C14A | 0.9196 (6) | 0.5403 (6) | 0.8187 (8) | 0.081 (2) | 0.82 (2) |
| H14D | 0.990161 | 0.537559 | 0.874354 | 0.121* | 0.82 (2) |
| H14E | 0.905105 | 0.598504 | 0.794044 | 0.121* | 0.82 (2) |
| H14F | 0.933153 | 0.506213 | 0.745725 | 0.121* | 0.82 (2) |
| C15 | 0.2073 (3) | 0.56983 (15) | 0.5051 (2) | 0.0451 (6) | |
| C16 | 0.1899 (2) | 0.63673 (15) | 0.6032 (2) | 0.0433 (6) | |
| H16 | 0.178566 | 0.692838 | 0.576447 | 0.052* | |
| C17 | 0.1895 (2) | 0.62200 (15) | 0.7229 (2) | 0.0416 (6) | |
| H17 | 0.195706 | 0.565513 | 0.749521 | 0.050* | |
| C18 | 0.1799 (2) | 0.68909 (15) | 0.8200 (2) | 0.0404 (5) | |
| H5A | 0.169 (4) | 0.8081 (18) | 0.831 (3) | 0.099 (13)* | |
| H1 | 0.285 (3) | 0.440 (2) | 0.668 (3) | 0.097 (13)* | |
| H1A | 0.383 (3) | 0.1985 (18) | 1.105 (3) | 0.087 (12)* | |
| H2 | 0.711 (2) | 0.4712 (15) | 0.7356 (17) | 0.043 (7)* | |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0642 (12) | 0.0451 (10) | 0.0478 (11) | 0.0062 (9) | 0.0079 (9) | 0.0135 (8) |
| O2 | 0.1038 (17) | 0.0329 (9) | 0.0428 (10) | 0.0092 (10) | −0.0004 (10) | 0.0024 (8) |
| O3 | 0.1209 (18) | 0.0350 (9) | 0.0334 (10) | 0.0147 (10) | 0.0104 (10) | 0.0021 (7) |
| O4 | 0.129 (2) | 0.0509 (12) | 0.0333 (10) | 0.0010 (12) | −0.0030 (11) | 0.0006 (9) |
| O5 | 0.0945 (15) | 0.0362 (10) | 0.0365 (10) | −0.0031 (9) | −0.0003 (9) | −0.0035 (8) |
| N1 | 0.0622 (15) | 0.0440 (12) | 0.0499 (13) | −0.0045 (11) | 0.0044 (11) | 0.0164 (10) |
| N2 | 0.0553 (13) | 0.0334 (11) | 0.0499 (13) | −0.0082 (9) | 0.0105 (10) | −0.0050 (9) |
| C1 | 0.0541 (16) | 0.0452 (15) | 0.0628 (17) | −0.0008 (12) | −0.0002 (13) | 0.0129 (13) |
| C2 | 0.0564 (15) | 0.0324 (12) | 0.0421 (13) | −0.0079 (11) | 0.0062 (11) | 0.0001 (10) |
| C3 | 0.0611 (18) | 0.0577 (17) | 0.0592 (18) | −0.0188 (14) | 0.0079 (14) | 0.0085 (14) |
| C4 | 0.0487 (16) | 0.072 (2) | 0.0685 (19) | −0.0131 (15) | 0.0061 (14) | −0.0015 (16) |
| C5 | 0.0536 (16) | 0.0541 (16) | 0.0479 (15) | 0.0024 (13) | −0.0039 (12) | −0.0036 (12) |
| C6 | 0.0554 (15) | 0.0317 (12) | 0.0363 (12) | 0.0039 (10) | 0.0080 (10) | −0.0021 (9) |
| C7 | 0.0507 (14) | 0.0288 (11) | 0.0385 (12) | −0.0011 (10) | 0.0070 (10) | −0.0010 (9) |
| C8 | 0.0505 (15) | 0.0332 (12) | 0.0511 (14) | −0.0023 (10) | 0.0064 (11) | 0.0038 (10) |
| C9 | 0.0517 (15) | 0.0384 (13) | 0.0573 (16) | −0.0002 (11) | 0.0105 (12) | 0.0008 (11) |
| C10 | 0.0661 (18) | 0.0445 (15) | 0.0493 (15) | −0.0094 (13) | 0.0152 (13) | −0.0037 (12) |
| C11 | 0.070 (3) | 0.035 (2) | 0.074 (3) | −0.0067 (16) | 0.009 (2) | 0.0007 (18) |
| C12 | 0.080 (4) | 0.082 (5) | 0.110 (5) | −0.002 (3) | −0.010 (3) | 0.039 (4) |
| C11A | 0.089 (9) | 0.091 (9) | 0.090 (9) | 0.001 (4) | 0.005 (4) | 0.003 (4) |
| C12A | 0.079 (10) | 0.084 (11) | 0.084 (10) | 0.005 (7) | 0.003 (6) | 0.002 (7) |
| C13 | 0.077 (9) | 0.078 (9) | 0.079 (9) | −0.006 (4) | 0.007 (4) | −0.007 (4) |
| C14 | 0.086 (12) | 0.090 (13) | 0.090 (12) | −0.014 (7) | 0.014 (7) | 0.008 (8) |
| C13A | 0.065 (2) | 0.062 (3) | 0.054 (2) | −0.0123 (18) | 0.0010 (16) | −0.003 (2) |
| C14A | 0.061 (3) | 0.092 (5) | 0.089 (4) | −0.010 (3) | 0.003 (3) | −0.004 (3) |
| C15 | 0.0656 (17) | 0.0348 (12) | 0.0349 (13) | 0.0034 (11) | 0.0009 (11) | 0.0005 (10) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C16 | 0.0611 (16) | 0.0299 (12) | 0.0390 (13) | 0.0014 (11) | 0.0032 (11) | 0.0002 (10) |
| C17 | 0.0541 (15) | 0.0314 (12) | 0.0394 (13) | −0.0010 (10) | 0.0017 (10) | 0.0009 (10) |
| C18 | 0.0478 (14) | 0.0387 (13) | 0.0347 (13) | −0.0052 (10) | 0.0001 (10) | −0.0008 (10) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C6 | 1.368 (3) | C10—H10A | 0.9700 |
| O1—H1 | 0.872 (10) | C10—H10B | 0.9700 |
| O2—C15 | 1.241 (3) | C11—H11A | 0.9700 |
| O3—C15 | 1.259 (3) | C11—H11B | 0.9700 |
| O4—C18 | 1.200 (3) | C11—C12 | 1.458 (7) |
| O5—C18 | 1.302 (3) | C12—H12A | 0.9600 |
| O5—H5A | 0.901 (10) | C12—H12B | 0.9600 |
| N1—C1 | 1.367 (4) | C12—H12C | 0.9600 |
| N1—C2 | 1.372 (3) | C11A—H11C | 0.9700 |
| N1—H1A | 0.870 (10) | C11A—H11D | 0.9700 |
| N2—C10 | 1.512 (3) | C11A—C12A | 1.436 (16) |
| N2—C11 | 1.484 (4) | C12A—H12D | 0.9600 |
| N2—C11A | 1.506 (10) | C12A—H12E | 0.9600 |
| N2—C13 | 1.492 (10) | C12A—H12F | 0.9600 |
| N2—C13A | 1.531 (5) | C13—H13A | 0.9700 |
| N2—H2 | 0.847 (10) | C13—H13B | 0.9700 |
| C1—H1B | 0.9300 | C13—C14 | 1.444 (16) |
| C1—C8 | 1.358 (4) | C14—H14A | 0.9600 |
| C2—C3 | 1.384 (4) | C14—H14B | 0.9600 |
| C2—C7 | 1.407 (3) | C14—H14C | 0.9600 |
| C3—H3 | 0.9300 | C13A—H13C | 0.9700 |
| C3—C4 | 1.369 (4) | C13A—H13D | 0.9700 |
| C4—H4 | 0.9300 | C13A—C14A | 1.468 (6) |
| C4—C5 | 1.401 (4) | C14A—H14D | 0.9600 |
| C5—H5 | 0.9300 | C14A—H14E | 0.9600 |
| C5—C6 | 1.367 (4) | C14A—H14F | 0.9600 |
| C6—C7 | 1.409 (3) | C15—C16 | 1.498 (3) |
| C7—C8 | 1.438 (4) | C16—H16 | 0.9300 |
| C8—C9 | 1.499 (4) | C16—C17 | 1.301 (3) |
| C9—H9A | 0.9700 | C17—H17 | 0.9300 |
| C9—H9B | 0.9700 | C17—C18 | 1.482 (3) |
| C9—C10 | 1.498 (4) | | |

| | | | |
|---|---|---|---|
| C6—O1—H1 | 105 (3) | C12—C11—H11A | 109.2 |
| C18—O5—H5A | 115 (3) | C12—C11—H11B | 109.2 |
| C1—N1—C2 | 109.0 (2) | C11—C12—H12A | 109.5 |
| C1—N1—H1A | 131 (3) | C11—C12—H12B | 109.5 |
| C2—N1—H1A | 119 (3) | C11—C12—H12C | 109.5 |
| C10—N2—C13A | 107.4 (3) | H12A—C12—H12B | 109.5 |
| C10—N2—H2 | 107.6 (19) | H12A—C12—H12C | 109.5 |
| C11—N2—C10 | 111.4 (3) | H12B—C12—H12C | 109.5 |
| C11—N2—C13 | 94.9 (7) | N2—C11A—H11C | 105.1 |

| | | | |
|---|---|---|---|
| C11—N2—H2 | 109.0 (19) | N2—C11A—H11D | 105.1 |
| C11A—N2—C10 | 110.2 (12) | H11C—C11A—H11D | 105.9 |
| C11A—N2—C13A | 129.0 (16) | C12A—C11A—N2 | 129 (3) |
| C11A—N2—H2 | 91 (3) | C12A—C11A—H11C | 105.1 |
| C13—N2—C10 | 117.6 (11) | C12A—C11A—H11D | 105.1 |
| C13—N2—H2 | 116 (2) | C11A—C12A—H12D | 109.5 |
| C13A—N2—H2 | 109.3 (19) | C11A—C12A—H12E | 109.5 |
| N1—C1—H1B | 124.7 | C11A—C12A—H12F | 109.5 |
| C8—C1—N1 | 110.7 (2) | H12D—C12A—H12E | 109.5 |
| C8—C1—H1B | 124.7 | H12D—C12A—H12F | 109.5 |
| N1—C2—C3 | 130.0 (2) | H12E—C12A—H12F | 109.5 |
| N1—C2—C7 | 107.1 (2) | N2—C13—H13A | 108.4 |
| C3—C2—C7 | 122.8 (3) | N2—C13—H13B | 108.4 |
| C2—C3—H3 | 121.4 | H13A—C13—H13B | 107.4 |
| C4—C3—C2 | 117.3 (3) | C14—C13—N2 | 116 (3) |
| C4—C3—H3 | 121.4 | C14—C13—H13A | 108.4 |
| C3—C4—H4 | 119.1 | C14—C13—H13B | 108.4 |
| C3—C4—C5 | 121.8 (3) | C13—C14—H14A | 109.5 |
| C5—C4—H4 | 119.1 | C13—C14—H14B | 109.5 |
| C4—C5—H5 | 119.7 | C13—C14—H14C | 109.5 |
| C6—C5—C4 | 120.6 (3) | H14A—C14—H14B | 109.5 |
| C6—C5—H5 | 119.7 | H14A—C14—H14C | 109.5 |
| O1—C6—C7 | 116.4 (2) | H14B—C14—H14C | 109.5 |
| C5—C6—O1 | 124.0 (2) | N2—C13A—H13C | 108.7 |
| C5—C6—C7 | 119.5 (2) | N2—C13A—H13D | 108.7 |
| C2—C7—C6 | 117.9 (2) | H13C—C13A—H13D | 107.6 |
| C2—C7—C8 | 107.3 (2) | C14A—C13A—N2 | 114.0 (4) |
| C6—C7—C8 | 134.7 (2) | C14A—C13A—H13C | 108.7 |
| C1—C8—C7 | 105.9 (2) | C14A—C13A—H13D | 108.7 |
| C1—C8—C9 | 125.6 (3) | C13A—C14A—H14D | 109.5 |
| C7—C8—C9 | 128.5 (2) | C13A—C14A—H14E | 109.5 |
| C8—C9—H9A | 109.3 | C13A—C14A—H14F | 109.5 |
| C8—C9—H9B | 109.3 | H14D—C14A—H14E | 109.5 |
| H9A—C9—H9B | 108.0 | H14D—C14A—H14F | 109.5 |
| C10—C9—C8 | 111.5 (2) | H14E—C14A—H14F | 109.5 |
| C10—C9—H9A | 109.3 | O2—C15—O3 | 123.7 (2) |
| C10—C9—H9B | 109.3 | O2—C15—C16 | 120.5 (2) |
| N2—C10—H10A | 109.0 | O3—C15—C16 | 115.8 (2) |
| N2—C10—H10B | 109.0 | C15—C16—H16 | 117.6 |
| C9—C10—N2 | 112.8 (2) | C17—C16—C15 | 124.7 (2) |
| C9—C10—H10A | 109.0 | C17—C16—H16 | 117.6 |
| C9—C10—H10B | 109.0 | C16—C17—H17 | 117.8 |
| H10A—C10—H10B | 107.8 | C16—C17—C18 | 124.5 (2) |
| N2—C11—H11A | 109.2 | C18—C17—H17 | 117.8 |
| N2—C11—H11B | 109.2 | O4—C18—O5 | 123.8 (2) |
| H11A—C11—H11B | 107.9 | O4—C18—C17 | 122.7 (2) |
| C12—C11—N2 | 111.9 (4) | O5—C18—C17 | 113.5 (2) |

| | | | |
|---|---|---|---|
| O1—C6—C7—C2 | −175.9 (2) | C5—C6—C7—C8 | 178.3 (3) |
| O1—C6—C7—C8 | 1.1 (4) | C6—C7—C8—C1 | −178.4 (3) |
| O2—C15—C16—C17 | 3.5 (4) | C6—C7—C8—C9 | −0.4 (5) |
| O3—C15—C16—C17 | −174.7 (3) | C7—C2—C3—C4 | −0.1 (4) |
| N1—C1—C8—C7 | 0.2 (3) | C7—C8—C9—C10 | 76.0 (3) |
| N1—C1—C8—C9 | −177.8 (2) | C8—C9—C10—N2 | −177.2 (2) |
| N1—C2—C3—C4 | 179.4 (3) | C10—N2—C11—C12 | −66.3 (5) |
| N1—C2—C7—C6 | 179.3 (2) | C10—N2—C11A—C12A | −52 (4) |
| N1—C2—C7—C8 | 1.5 (3) | C10—N2—C13—C14 | 169 (2) |
| C1—N1—C2—C3 | 179.1 (3) | C10—N2—C13A—C14A | 172.6 (5) |
| C1—N1—C2—C7 | −1.4 (3) | C11—N2—C10—C9 | 139.2 (4) |
| C1—C8—C9—C10 | −106.4 (3) | C11—N2—C13—C14 | −74 (2) |
| C2—N1—C1—C8 | 0.7 (3) | C11A—N2—C10—C9 | 117 (2) |
| C2—C3—C4—C5 | 1.2 (5) | C11A—N2—C13A—C14A | −51 (2) |
| C2—C7—C8—C1 | −1.1 (3) | C13—N2—C10—C9 | −112.9 (10) |
| C2—C7—C8—C9 | 176.9 (2) | C13—N2—C11—C12 | 171.5 (13) |
| C3—C2—C7—C6 | −1.1 (4) | C13A—N2—C10—C9 | −97.8 (4) |
| C3—C2—C7—C8 | −178.9 (2) | C13A—N2—C11A—C12A | 173 (3) |
| C3—C4—C5—C6 | −1.1 (5) | C15—C16—C17—C18 | 176.1 (2) |
| C4—C5—C6—O1 | 176.7 (3) | C16—C17—C18—O4 | −175.2 (3) |
| C4—C5—C6—C7 | −0.2 (4) | C16—C17—C18—O5 | 3.9 (4) |
| C5—C6—C7—C2 | 1.2 (3) | | |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O5—H5*A*···O3$^{i}$ | 0.90 (1) | 1.70 (2) | 2.559 (2) | 157 (4) |
| O1—H1···O2 | 0.87 (1) | 1.79 (2) | 2.607 (3) | 155 (4) |
| N1—H1*A*···O1$^{ii}$ | 0.87 (1) | 2.15 (1) | 3.011 (3) | 169 (3) |
| N2—H2···O3$^{iii}$ | 0.85 (1) | 1.96 (1) | 2.783 (3) | 165 (3) |

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) *x*, −*y*+1/2, *z*+1/2; (iii) −*x*+1, −*y*+1, −*z*+1.

**Bis{[2-(4-acetoxy-1*H*-indol-3-yl)ethyl]diethylazanium} but-2-enedioate–(*E*)-butenedioic acid (1/1) (4-AcO-DET)**

*Crystal data*

$2(C_{16}H_{23}N_2O_2^+)\cdot C_4H_2O_4^{2-}\cdot C_4H_4O_4$
$M_r$ = 390.43
Monoclinic, *C*2/*c*
*a* = 24.410 (2) Å
*b* = 8.4508 (7) Å
*c* = 21.445 (2) Å
*β* = 110.963 (3)°
*V* = 4131.1 (7) Å$^3$
*Z* = 8
*F*(000) = 1664
$D_x$ = 1.256 Mg m$^{-3}$
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 5098 reflections
*θ* = 2.6–25.3°
*μ* = 0.09 mm$^{-1}$
*T* = 300 K
Block, colourless
0.18 × 0.15 × 0.12 mm

*Data collection*

Bruker D8 Venture CMOS diffractometer
*φ* and *ω* scans
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)
$T_{min}$ = 0.697, $T_{max}$ = 0.745
47661 measured reflections

3525 independent reflections
2420 reflections with $I > 2\sigma(I)$
$R_{int} = 0.113$
$\theta_{max} = 24.7°$, $\theta_{min} = 2.6°$

$h = -28 \rightarrow 28$
$k = -9 \rightarrow 9$
$l = -25 \rightarrow 25$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.122$
$S = 1.04$
3525 reflections
268 parameters
9 restraints

Primary atom site location: structure-invariant direct methods
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0421P)^2 + 4.0733P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å$^{-3}$
$\Delta\rho_{min} = -0.17$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.74350 (9) | 0.6039 (2) | 0.61017 (9) | 0.0633 (5) |
| O2 | 0.76587 (11) | 0.8498 (3) | 0.59230 (16) | 0.1071 (9) |
| N1 | 0.85177 (9) | 0.5727 (3) | 0.83707 (11) | 0.0508 (5) |
| H1 | 0.8769 (10) | 0.528 (3) | 0.8721 (9) | 0.072 (9)* |
| N2 | 0.59712 (8) | 0.5647 (2) | 0.67889 (10) | 0.0430 (5) |
| H2 | 0.5887 (11) | 0.651 (2) | 0.6524 (10) | 0.057 (8)* |
| C1 | 0.79489 (10) | 0.6118 (3) | 0.82720 (12) | 0.0477 (6) |
| H1A | 0.779946 | 0.624586 | 0.861145 | 0.057* |
| C2 | 0.85802 (10) | 0.5604 (3) | 0.77654 (13) | 0.0444 (6) |
| C3 | 0.90724 (12) | 0.5215 (3) | 0.76154 (16) | 0.0589 (7) |
| H3 | 0.943156 | 0.500534 | 0.795016 | 0.071* |
| C4 | 0.90072 (15) | 0.5153 (3) | 0.69572 (19) | 0.0721 (9) |
| H4 | 0.932990 | 0.490469 | 0.684192 | 0.087* |
| C5 | 0.84718 (15) | 0.5451 (3) | 0.64554 (16) | 0.0678 (8) |
| H5 | 0.843846 | 0.537899 | 0.601061 | 0.081* |
| C6 | 0.79898 (12) | 0.5854 (3) | 0.66107 (13) | 0.0515 (7) |
| C7 | 0.80299 (10) | 0.5954 (2) | 0.72714 (12) | 0.0406 (6) |
| C8 | 0.76320 (10) | 0.6296 (3) | 0.76112 (11) | 0.0398 (5) |
| C9 | 0.69942 (10) | 0.6730 (3) | 0.73296 (13) | 0.0464 (6) |
| H9A | 0.693496 | 0.755282 | 0.699628 | 0.056* |
| H9B | 0.687531 | 0.714640 | 0.768327 | 0.056* |
| C10 | 0.66171 (9) | 0.5311 (3) | 0.70143 (13) | 0.0449 (6) |
| H10A | 0.671155 | 0.496520 | 0.663330 | 0.054* |
| H10B | 0.671103 | 0.445124 | 0.733515 | 0.054* |
| C11 | 0.57684 (11) | 0.6015 (3) | 0.73551 (13) | 0.0527 (7) |

| | | | | |
|---|---|---|---|---|
| H11A | 0.535407 | 0.627464 | 0.717297 | 0.063* |
| H11B | 0.597624 | 0.694451 | 0.758699 | 0.063* |
| C12 | 0.58588 (15) | 0.4689 (4) | 0.78553 (16) | 0.0786 (9) |
| H12A | 0.571742 | 0.501205 | 0.819968 | 0.118* |
| H12B | 0.626920 | 0.444336 | 0.804997 | 0.118* |
| H12C | 0.564717 | 0.376855 | 0.763378 | 0.118* |
| C13 | 0.56146 (12) | 0.4341 (3) | 0.63634 (13) | 0.0574 (7) |
| H13A | 0.572445 | 0.334770 | 0.660275 | 0.069* |
| H13B | 0.520334 | 0.452764 | 0.628564 | 0.069* |
| C14 | 0.56919 (15) | 0.4199 (4) | 0.57135 (16) | 0.0833 (10) |
| H14A | 0.541126 | 0.345901 | 0.543643 | 0.125* |
| H14B | 0.608145 | 0.383101 | 0.578315 | 0.125* |
| H14C | 0.563306 | 0.521349 | 0.549841 | 0.125* |
| C15 | 0.73154 (15) | 0.7429 (4) | 0.57790 (16) | 0.0655 (8) |
| C16 | 0.67192 (15) | 0.7417 (4) | 0.52599 (16) | 0.0836 (10) |
| H16A | 0.669563 | 0.660156 | 0.493983 | 0.125* |
| H16B | 0.664099 | 0.842500 | 0.503877 | 0.125* |
| H16C | 0.643501 | 0.721812 | 0.546504 | 0.125* |
| O3 | 0.48170 (7) | 0.7667 (2) | 0.59379 (9) | 0.0618 (5) |
| O4 | 0.56861 (6) | 0.82722 (19) | 0.59214 (8) | 0.0473 (4) |
| O5 | 0.37498 (8) | 0.6824 (2) | 0.56621 (10) | 0.0696 (6) |
| H5A | 0.4125 (8) | 0.724 (4) | 0.5712 (17) | 0.109 (12)* |
| O6 | 0.34179 (7) | 0.8613 (2) | 0.48661 (9) | 0.0620 (5) |
| C17 | 0.51391 (9) | 0.8438 (3) | 0.57089 (11) | 0.0376 (5) |
| C18 | 0.48603 (9) | 0.9610 (2) | 0.51631 (11) | 0.0387 (5) |
| H18 | 0.446073 | 0.979934 | 0.504643 | 0.046* |
| C19 | 0.33287 (10) | 0.7592 (3) | 0.52073 (12) | 0.0418 (6) |
| C20 | 0.27361 (9) | 0.7103 (3) | 0.51714 (12) | 0.0450 (6) |
| H20 | 0.269777 | 0.619973 | 0.540034 | 0.054* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0847 (14) | 0.0524 (11) | 0.0450 (10) | −0.0030 (10) | 0.0135 (10) | 0.0114 (9) |
| O2 | 0.0861 (17) | 0.0642 (15) | 0.176 (3) | 0.0127 (13) | 0.0530 (18) | 0.0485 (16) |
| N1 | 0.0401 (13) | 0.0544 (13) | 0.0471 (14) | −0.0021 (10) | 0.0024 (10) | 0.0022 (11) |
| N2 | 0.0359 (11) | 0.0347 (11) | 0.0512 (12) | 0.0040 (9) | 0.0070 (9) | 0.0036 (10) |
| C1 | 0.0422 (15) | 0.0537 (15) | 0.0448 (15) | −0.0044 (11) | 0.0124 (12) | −0.0024 (12) |
| C2 | 0.0412 (14) | 0.0329 (12) | 0.0565 (16) | −0.0031 (10) | 0.0142 (12) | 0.0018 (11) |
| C3 | 0.0479 (16) | 0.0450 (15) | 0.085 (2) | 0.0015 (12) | 0.0248 (15) | 0.0063 (14) |
| C4 | 0.072 (2) | 0.0570 (18) | 0.106 (3) | 0.0061 (15) | 0.054 (2) | 0.0070 (18) |
| C5 | 0.095 (3) | 0.0536 (17) | 0.070 (2) | 0.0008 (16) | 0.049 (2) | 0.0010 (15) |
| C6 | 0.0646 (18) | 0.0391 (14) | 0.0497 (16) | −0.0002 (12) | 0.0193 (14) | 0.0042 (11) |
| C7 | 0.0439 (14) | 0.0281 (11) | 0.0475 (14) | −0.0028 (10) | 0.0137 (11) | 0.0028 (10) |
| C8 | 0.0381 (13) | 0.0338 (12) | 0.0426 (14) | −0.0017 (10) | 0.0085 (11) | −0.0007 (10) |
| C9 | 0.0413 (14) | 0.0396 (13) | 0.0533 (15) | 0.0046 (11) | 0.0108 (12) | −0.0008 (11) |
| C10 | 0.0340 (13) | 0.0383 (13) | 0.0566 (15) | 0.0076 (10) | 0.0092 (11) | 0.0005 (11) |
| C11 | 0.0453 (15) | 0.0516 (15) | 0.0619 (17) | 0.0053 (12) | 0.0199 (13) | −0.0004 (13) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C12 | 0.091 (2) | 0.080 (2) | 0.069 (2) | 0.0118 (18) | 0.0329 (18) | 0.0145 (17) |
| C13 | 0.0501 (16) | 0.0460 (14) | 0.0623 (18) | −0.0023 (12) | 0.0032 (13) | −0.0032 (13) |
| C14 | 0.088 (2) | 0.071 (2) | 0.070 (2) | 0.0002 (18) | 0.0037 (18) | −0.0170 (17) |
| C15 | 0.079 (2) | 0.0580 (19) | 0.076 (2) | 0.0190 (17) | 0.0485 (18) | 0.0212 (16) |
| C16 | 0.092 (3) | 0.097 (2) | 0.070 (2) | 0.030 (2) | 0.0385 (19) | 0.0336 (19) |
| O3 | 0.0325 (9) | 0.0784 (13) | 0.0661 (12) | −0.0012 (9) | 0.0076 (9) | 0.0319 (10) |
| O4 | 0.0281 (9) | 0.0491 (10) | 0.0560 (10) | 0.0027 (7) | 0.0043 (8) | 0.0108 (8) |
| O5 | 0.0346 (10) | 0.0803 (14) | 0.0847 (14) | 0.0017 (10) | 0.0101 (10) | 0.0404 (11) |
| O6 | 0.0397 (10) | 0.0781 (13) | 0.0660 (12) | −0.0010 (9) | 0.0162 (9) | 0.0282 (10) |
| C17 | 0.0280 (13) | 0.0395 (12) | 0.0386 (13) | −0.0021 (10) | 0.0036 (10) | −0.0011 (10) |
| C18 | 0.0254 (12) | 0.0387 (13) | 0.0445 (14) | 0.0012 (9) | 0.0033 (9) | 0.0008 (10) |
| C19 | 0.0341 (13) | 0.0468 (14) | 0.0425 (14) | 0.0025 (11) | 0.0110 (11) | 0.0052 (12) |
| C20 | 0.0362 (13) | 0.0505 (15) | 0.0482 (15) | −0.0020 (10) | 0.0148 (11) | 0.0068 (11) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| O1—C6 | 1.412 (3) | C11—H11A | 0.9700 |
| O1—C15 | 1.342 (3) | C11—H11B | 0.9700 |
| O2—C15 | 1.195 (4) | C11—C12 | 1.512 (4) |
| N1—H1 | 0.868 (10) | C12—H12A | 0.9600 |
| N1—C1 | 1.368 (3) | C12—H12B | 0.9600 |
| N1—C2 | 1.364 (3) | C12—H12C | 0.9600 |
| N2—H2 | 0.906 (10) | C13—H13A | 0.9700 |
| N2—C10 | 1.502 (3) | C13—H13B | 0.9700 |
| N2—C11 | 1.499 (3) | C13—C14 | 1.477 (4) |
| N2—C13 | 1.497 (3) | C14—H14A | 0.9600 |
| C1—H1A | 0.9300 | C14—H14B | 0.9600 |
| C1—C8 | 1.358 (3) | C14—H14C | 0.9600 |
| C2—C3 | 1.390 (4) | C15—C16 | 1.483 (4) |
| C2—C7 | 1.413 (3) | C16—H16A | 0.9600 |
| C3—H3 | 0.9300 | C16—H16B | 0.9600 |
| C3—C4 | 1.364 (4) | C16—H16C | 0.9600 |
| C4—H4 | 0.9300 | O3—C17 | 1.249 (3) |
| C4—C5 | 1.387 (4) | O4—C17 | 1.255 (3) |
| C5—H5 | 0.9300 | O5—H5A | 0.952 (10) |
| C5—C6 | 1.375 (4) | O5—C19 | 1.308 (3) |
| C6—C7 | 1.387 (3) | O6—C19 | 1.201 (3) |
| C7—C8 | 1.437 (3) | C17—C18 | 1.498 (3) |
| C8—C9 | 1.500 (3) | C18—C18$^{i}$ | 1.316 (4) |
| C9—H9A | 0.9700 | C18—H18 | 0.9300 |
| C9—H9B | 0.9700 | C19—C20 | 1.480 (3) |
| C9—C10 | 1.516 (3) | C20—C20$^{ii}$ | 1.309 (4) |
| C10—H10A | 0.9700 | C20—H20 | 0.9300 |
| C10—H10B | 0.9700 | | |
| | | | |
| C15—O1—C6 | 117.9 (2) | N2—C11—H11A | 108.6 |
| C1—N1—H1 | 127 (2) | N2—C11—H11B | 108.6 |
| C2—N1—H1 | 120 (2) | N2—C11—C12 | 114.5 (2) |

| | | | |
|---|---|---|---|
| C2—N1—C1 | 108.9 (2) | H11A—C11—H11B | 107.6 |
| C10—N2—H2 | 109.3 (16) | C12—C11—H11A | 108.6 |
| C11—N2—H2 | 106.0 (16) | C12—C11—H11B | 108.6 |
| C11—N2—C10 | 113.05 (19) | C11—C12—H12A | 109.5 |
| C13—N2—H2 | 105.2 (16) | C11—C12—H12B | 109.5 |
| C13—N2—C10 | 111.80 (18) | C11—C12—H12C | 109.5 |
| C13—N2—C11 | 111.0 (2) | H12A—C12—H12B | 109.5 |
| N1—C1—H1A | 124.6 | H12A—C12—H12C | 109.5 |
| C8—C1—N1 | 110.9 (2) | H12B—C12—H12C | 109.5 |
| C8—C1—H1A | 124.6 | N2—C13—H13A | 109.0 |
| N1—C2—C3 | 129.7 (2) | N2—C13—H13B | 109.0 |
| N1—C2—C7 | 107.3 (2) | H13A—C13—H13B | 107.8 |
| C3—C2—C7 | 123.0 (3) | C14—C13—N2 | 113.1 (2) |
| C2—C3—H3 | 121.4 | C14—C13—H13A | 109.0 |
| C4—C3—C2 | 117.3 (3) | C14—C13—H13B | 109.0 |
| C4—C3—H3 | 121.4 | C13—C14—H14A | 109.5 |
| C3—C4—H4 | 119.2 | C13—C14—H14B | 109.5 |
| C3—C4—C5 | 121.7 (3) | C13—C14—H14C | 109.5 |
| C5—C4—H4 | 119.2 | H14A—C14—H14B | 109.5 |
| C4—C5—H5 | 119.8 | H14A—C14—H14C | 109.5 |
| C6—C5—C4 | 120.5 (3) | H14B—C14—H14C | 109.5 |
| C6—C5—H5 | 119.8 | O1—C15—C16 | 110.8 (3) |
| C5—C6—O1 | 120.5 (3) | O2—C15—O1 | 121.7 (3) |
| C5—C6—C7 | 120.6 (3) | O2—C15—C16 | 127.5 (3) |
| C7—C6—O1 | 118.7 (2) | C15—C16—H16A | 109.5 |
| C2—C7—C8 | 107.2 (2) | C15—C16—H16B | 109.5 |
| C6—C7—C2 | 117.0 (2) | C15—C16—H16C | 109.5 |
| C6—C7—C8 | 135.8 (2) | H16A—C16—H16B | 109.5 |
| C1—C8—C7 | 105.7 (2) | H16A—C16—H16C | 109.5 |
| C1—C8—C9 | 124.7 (2) | H16B—C16—H16C | 109.5 |
| C7—C8—C9 | 129.6 (2) | C19—O5—H5A | 111 (2) |
| C8—C9—H9A | 109.4 | O3—C17—O4 | 122.5 (2) |
| C8—C9—H9B | 109.4 | O3—C17—C18 | 118.56 (19) |
| C8—C9—C10 | 111.29 (19) | O4—C17—C18 | 119.0 (2) |
| H9A—C9—H9B | 108.0 | C17—C18—H18 | 117.6 |
| C10—C9—H9A | 109.4 | C18$^{i}$—C18—C17 | 124.7 (3) |
| C10—C9—H9B | 109.4 | C18$^{i}$—C18—H18 | 117.6 |
| N2—C10—C9 | 113.21 (18) | O5—C19—C20 | 113.1 (2) |
| N2—C10—H10A | 108.9 | O6—C19—O5 | 123.1 (2) |
| N2—C10—H10B | 108.9 | O6—C19—C20 | 123.8 (2) |
| C9—C10—H10A | 108.9 | C19—C20—H20 | 118.9 |
| C9—C10—H10B | 108.9 | C20$^{ii}$—C20—C19 | 122.3 (3) |
| H10A—C10—H10B | 107.7 | C20$^{ii}$—C20—H20 | 118.9 |

| | | | |
|---|---|---|---|
| O1—C6—C7—C2 | 173.7 (2) | C5—C6—C7—C8 | −178.5 (3) |
| O1—C6—C7—C8 | −3.8 (4) | C6—O1—C15—O2 | −1.6 (4) |
| N1—C1—C8—C7 | 1.4 (3) | C6—O1—C15—C16 | 179.6 (2) |
| N1—C1—C8—C9 | −179.8 (2) | C6—C7—C8—C1 | 176.9 (3) |

| | | | |
|---|---|---|---|
| N1—C2—C3—C4 | 178.9 (3) | C6—C7—C8—C9 | −1.9 (4) |
| N1—C2—C7—C6 | −178.2 (2) | C7—C2—C3—C4 | −1.0 (4) |
| N1—C2—C7—C8 | 0.0 (2) | C7—C8—C9—C10 | 73.5 (3) |
| C1—N1—C2—C3 | −179.1 (2) | C8—C9—C10—N2 | 174.0 (2) |
| C1—N1—C2—C7 | 0.8 (3) | C10—N2—C11—C12 | −61.4 (3) |
| C1—C8—C9—C10 | −105.1 (3) | C10—N2—C13—C14 | −67.2 (3) |
| C2—N1—C1—C8 | −1.4 (3) | C11—N2—C10—C9 | −65.1 (3) |
| C2—C3—C4—C5 | −0.5 (4) | C11—N2—C13—C14 | 165.6 (2) |
| C2—C7—C8—C1 | −0.8 (2) | C13—N2—C10—C9 | 168.8 (2) |
| C2—C7—C8—C9 | −179.6 (2) | C13—N2—C11—C12 | 65.1 (3) |
| C3—C2—C7—C6 | 1.8 (3) | C15—O1—C6—C5 | −82.6 (3) |
| C3—C2—C7—C8 | 180.0 (2) | C15—O1—C6—C7 | 102.8 (3) |
| C3—C4—C5—C6 | 1.3 (4) | O3—C17—C18—C18$^{i}$ | −173.5 (3) |
| C4—C5—C6—O1 | −175.1 (2) | O4—C17—C18—C18$^{i}$ | 6.9 (4) |
| C4—C5—C6—C7 | −0.6 (4) | O5—C19—C20—C20$^{ii}$ | 168.7 (3) |
| C5—C6—C7—C2 | −0.9 (3) | O6—C19—C20—C20$^{ii}$ | −9.7 (5) |

Symmetry codes: (i) −*x*+1, −*y*+2, −*z*+1; (ii) −*x*+1/2, −*y*+3/2, −*z*+1.

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1···O4$^{iii}$ | 0.87 (1) | 2.12 (2) | 2.876 (3) | 145 (3) |
| N2—H2···O3 | 0.91 (1) | 2.65 (2) | 3.241 (2) | 124 (2) |
| N2—H2···O4 | 0.91 (1) | 1.91 (1) | 2.818 (2) | 177 (3) |
| O5—H5*A*···O3 | 0.95 (1) | 1.62 (1) | 2.558 (2) | 167 (3) |

Symmetry code: (iii) −*x*+3/2, *y*−1/2, −*z*+3/2.