Crystal structure of N-butyl-2,3-bis(dicyclohexylamino)cyclopropeniminium chloride benzene monosolvate

Gaby M. Muñoz Sánchez and Michael J. Zdilla*

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N-Butyl-2,3-bis(dicyclohexylamino)cyclopropenimine (1) crystallizes from benzene and hexanes in the presence of HCl as a monobenzene solvate of the hydrochloride salt, [1H]Cl-C$_6$H$_6$ or C$_{31}$H$_{54}$N$_3$+Cl$^-$C$_6$H$_6$, in the $P2_1/n$ space group. The protonation of 1 results in the generation of an aromatic structure based upon the delocalization of the cyclopropene double bond around the cyclopropene ring, giving three intermediate C—C bond lengths of ~1.41 Å, and the delocalization of the imine-type C—N double bond, giving three intermediate C—N bond lengths of ~1.32 Å. Ion–ion and ion–benzene packing interactions are described and illustrated.

1. Chemical context

Pentasubstituted diaminopropenimines are a relatively new class of superbases that operate via the establishment of a stable aromatic electronic delocalization upon protonation. Originally reported as four-electron Lewis donors (Bruns et al., 2010), a more recently exploited application for the use of pentasubstitution is that of a superbase, with one of the six nitrogen coordination sites available for protonation, making these molecules facile initiators of stereoselective Michael (Bandar & Lambert, 2012) and Mannich reactions (Bandar & Lambert, 2013), hydrosaminations (Mirabdolbaghi & Dudding, 2015), and ring-opening polymerization (Stukenbrocker et al., 2015; Xu et al., 2018). A number of examples of acid salts of these species have been structurally characterized, permitting direct observation of the aromatized cyclopropeniminium structures (Stukenbrocker et al., 2015; Bruns et al., 2010; Bandar et al., 2015; Belding & Dudding, 2014; Guest et al., 2020; Kozma et al., 2015; Belding et al., 2016; Bandar & Lambert, 2012, 2013; Mirabdolbaghi & Dudding, 2015). Examples of free-base pentasubstituted diaminopropenimines are uncommon, and these are typically only obtained with aromatic substituents at the imine position, which decreases the basicity of the imine by the delocalization of the nitrogen lone pair $p$-orbital into the aromatic group, facilitating isolation (Guest et al., 2020; Kozma et al., 2015; Bruns et al., 2010). Some of these (Guest et al., 2020; Kozma et al., 2015; Belding & Dudding, 2014) are bis(cyclopropenimine) variants of the famous ‘proton sponge’, 1,8-bis(dimethylamino)naphthalene and related classes of bifunctional Lewis superbases (Alder et al., 1968). The only other example, to our knowledge, is an N-aminosubstituted example, which also decreases the basicity of the nitrogen lone pair by induction, a minor resonance structure delocalizing the double bond into the N—N contact,
and, in the crystal structure, a nearby hydrogen bond with a water proton (Bruns et al., 2010).

N-Butyl-2,3-bis(dicyclohexylamino)cyclopropenimine (1) is a newer version of superbase with improved basicity, which has been explored as a catalyst for ring-opening polymerization. Cyclopropenimines have a conjugate acid $pK_a$ of about 27, an improvement over that of the superbase 2-tert-butyl-1,1,3,3-tetramethylguanidine (BTMG), which has a $pK_a$ of 23.56 (Bandar & Lambert, 2012). This allows 1 to deprotonate a lactide and initiate polymerization in the synthesis of poly-lactic acid, as shown in Fig. 1 (Stukenbroeker et al., 2015).

Compound 1 can mediate the polymerization of lactic acid to 99% completion in 10 minutes or less. However, no X-ray crystal structure of the free base, nor an acid salt of this superbase has been reported. In this report we provide the first X-ray crystallographic structure of a benzene solvate of the hydrochloride salt $[1\text{H}]Cl_{C_6H_6}$.

2. Structural commentary

$[1\text{H}]Cl$ crystallizes in the $P2_1/n$ space group on a general position as a closely associated ion pair, with the protonation site at the $n$-butyl imine as expected, and one formula unit in the asymmetric unit, as well as one benzene molecule, also on a general position (Fig. 2). The organic salt and the benzene molecule are generally well ordered, except for the $\delta$ methyl carbon of the $n$-butyl group, which shows a mild wagging disorder. This disorder was treated with a two-site disorder model.

Free-base 1 would be expected to have localized double bonds at the $n$-butylimine $C=N$ position, and at the opposing cyclopropene position (see scheme). In the isolated free base of 1-mesityl-2,3-bis(diisopropylamino)cyclopropenimine (Bruns et al., 2010), the unprotonated $C=N$ imine bond is 1.2951 (14) Å in length, while the $C-N$ bonds to the tertiary amines are longer, at an average of 1.3494 (10) Å. The localized cyclopropene double bond is shorter, at 1.3712 (14) Å, than the single bonded $C-C$ cyclopropane contacts at an average of 1.4155 (10) Å. Protonation of the $n$-butylimine position during crystal growth results in all nitrogen atoms being three-coordinate, leading to delocalization of the imine.
Table 1

Comparative bond lengths (Å) for 1-mesityl-2,3-bis(diisopropylamino)cyclopropenimine, 1-mesityl-2,3-bis(diisopropylamino)cyclopropeniminium (Bruns et al., 2010), and N-n-butyl-2,3-bis(dicyclohexyl)cyclopropeniminium.

Divided entries refer to separate, related pairs of atoms and their associated metrics, e.g., 1.3450 (14)/1.3539 (14) denotes two distances for the two C—N(amine) contacts.

<table>
<thead>
<tr>
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</thead>
<tbody>
<tr>
<td>C—N(amine)</td>
<td>1.2951 (14)</td>
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</tr>
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<td>1.3450 (14)/1.3539 (14)</td>
<td>1.3205 (15)/1.3286 (16)</td>
<td>1.3248 (17)/1.331 (2)</td>
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<tr>
<td>C—C(para)</td>
<td>1.3712 (14)</td>
<td>1.3984 (17)</td>
<td>1.388 (2)</td>
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<tr>
<td>C—C(meta)</td>
<td>1.4202 (14)/1.4108 (14)</td>
<td>1.3792 (16)/1.3827 (16)</td>
<td>1.377 (2)/1.3831 (19)</td>
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</table>

double-bond character across all three C—N contacts. Correspondingly, the cyclopropene double bond is delocalized around the ring, giving a three-membered aromatic system. In [IH]Cl, the central C3N3 triangle is thus highly planar, with the six atoms exhibiting an r.m.s. deviation of only 0.0052 Å from the best-fit plane of the six atoms. The three C—N bonds are approximately equal in length, with the two tertiary cyclohexylamine positions having C—N lengths of 1.3279 (13) Å on average. The C—N bond to the protonated butyl nitrogen is only slightly shorter at 1.319 (2) Å. The three cyclopropene C—C bonds exhibit lengths consistent with aromaticity; the unique C—C bond opposite the n-butyl group is 1.388 (2) Å, while the other two C—C bonds are similar or slightly shorter at 1.377 (2) and 1.383 (2) Å. Though these latter two bonds are equivalent under molecular point symmetry, their differences are attributed to the asymmetric crystal packing environment of the P21/n space group. The comparable nature of the bond metrics of the three C—N bonds and the three cyclopropenyl C—C bonds is consistent with aromatization, and an analogous aromatization of the C3N3 core of 1-mesityl-2,3-bis(diisopropylamino)cyclopropeniminium tetrafluoroborate was observed in the crystal structure of this salt (Bruns et al., 2010). See Table 1 for C3N3 bond metrics.

The comparison between free-base forms of cyclopropenimin (Bruns et al., 2010) and the protonated forms demonstrate aromatization upon protonation, and explain the behavior of I as a superbase. While alkylamines are typically weak bases (pKₐ of conjugate acid ranges from about 2–5 (Fraser et al., 1983), the pKₐ of IH⁺ is a staggering 27 (Bandar & Lambert, 2012), more on the scale of a C—H bond. The drastic difference in basicity between typical alkylamines and I can be explained by the observed aromatization upon protonation. As a result, the IH resonance of the N—H hydrogen in [IH]Cl is a sharp singlet at 7.4 ppm in deuterated chloroform, suggesting little to no exchange like that typically observed for broad N—H resonances. The stabilization of a molecule by aromatization is quantified by the Dewar Resonance Energy (DRE), which ranges from about 6–25 kJ mol⁻¹ per π electron (Slayden & Liebman, 2001). Thus in the case of I, aromatic stabilization between 12 and 50 kJ mol⁻¹ upon protonation explains the large reported basicity.

3. Supramolecular features

Interionic/molecular interactions were examined using packing diagrams, and by the determination of partial atomic charge from Hirshfeld analysis. In the following discussion Hirshfeld charges are presented in parenthesis. The proton of the butylimine group (+0.121) interacts strongly with the chloride ion (−0.666) at a short H···Cl distance of 2.26 (2) Å. The chloride is positioned in a pocket surrounded by hydrogen atoms. In addition to the strong interaction with the acidic N—H proton, the chloride resides 2.8152 (7) Å from a benzene proton, H6AA (+0.046), and 2.7169 (6) Å from an intramolecular axial cyclohexyl proton, H29A (+0.050). The crystal packing demonstrates that the C3N3 planes of all molecules pack parallel to each other (as required by the space-group symmetry), with a normal slightly oblique to the (101) plane (see Fig. 3). The molecular planes stack in a staggered fashion via intervening inversion centers at the origin (Fig. 3, red) and at the center of the a edge (Fig. 3, teal). One face of the benzene solvent molecule interacts distally with the cyclohexyl group of one IH⁺ ion [closest atomic C···C distance: 3.829 (3) Å, Fig. 3, green line], while the other face interacts distally with the disordered methyl group of the n-butyl chain [closest atomic C···C distance: 4.29 (3) Å, Fig. 3, orange line]. The benzene interacts weakly with two chloride ions approximately along its equatorial plane (Fig. 3, blue lines), one via H6S (+0.069) with H···Cl = 2.8152 (7) Å, also shown in Fig. 2, and the other via H3S (+0.062) with H···Cl = 2.8365 (7) Å. These benzene–chlorine interactions form a channel along the (101) plane, each channel situated 1/4 of the

![Figure 3](image-url) Partially packed thermal ellipsoid plot of [IH]Cl-C6H6 showing neighboring intermolecular/interionic nearest neighbor interactions.

**Table 1**

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<td>1.3792 (16)/1.3827 (16)</td>
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</tr>
</tbody>
</table>

**Figure 3**

Partially packed thermal ellipsoid plot of [IH]Cl-C6H6 showing neighboring intermolecular/interionic nearest neighbor interactions.
way along the \( b \) axis (Fig. 4, top). Viewed from 90° along the [101] direction, the benzene solvent molecules sit along a second channel, with the chloride ions sitting at the intersections of both channels, providing ionic bonds to the surrounding 1H\(^+\) cations (Fig. 4, bottom). In this latter view, it is apparent that along the [101] direction, the chloride ions are positioned between the axial protons H26 (+0.058) and H29B (+0.059) of the flanking cyclohexyl groups. In summary, the 1H\(^+\) cations interact with each other and through the benzene solvent molecule via their alkyl groups, and the chloride counter-ion is situated in a pocket of cyclohexyl and benzene C—H contacts, with the proximal N—H interaction on one side.

4. Database survey

In addition to the pentasubstituted examples discussed above, a survey of the Cambridge Structural Database (CSD, Version 4.34, November 2021; Groom et al., 2016) for cyclopropenimines reveals a number of other relevant structures. The parent (unsubstituted) diaminopropeniminium cation has been structurally characterized with chloride and iodide counter-cations (UJA VEI and UJA VIM; Mishiro et al., 2016). Aprotic hexasubstituted examples are reported, and represent planar polyatomic cations (AHUVEH, Holthoff et al., 2020; DOSRUB, Abdelbassit et al., 2019; FURCII, Clark et al., 1995; GAXYEJ, Radhakrishnan et al., 1987b; GERXUXO2, Butchard et al., 2012; GUNDUR, Curnow & Senthooran, 2020; IFAGUU, Curnow et al., 2018; LAYYOCC1, Jin et al., 2018; NUYBOB, Guest et al., 2020; SERVIW, Kniep et al., 2013; TUSDOD, Radhakrishnan et al., 1987a; UGITIQ, Barthes et al., 2020, XIKYAT01, ZABFUG, Wallace et al., 2015; XOSTIL, XOSTOR, XOSTUX, XOSVAF, XOSVEJ, Abdelbassit & Curnow, 2019, YUVRAK, YUWJOR, Jungbauer et al., 2015). Another class of variants includes cyclopropenamines tethered to ferrocene nuclei (TURNUQ, Bruns et al., 2010; BEBPIK, BEBRAE, BEBREI, BEBRIM, BEBROS, Jess et al., 2017). There are a few structural studies of Lewis complexes with metal ions (BEBRIM, Jess et al., 2017; UGITOW, UGITUC, Barthes et al., 2020; YOQPOM, Chen et al., 2019; TURNOK, Bruns et al., 2010) or other boron-based Lewis acids (NUYBOB, Guest et al., 2020; TURPOM, Bruns et al., 2010). One structural report of a trisubstituted cyclopropenimine is noted (XEXGEP, Xu et al., 2018), as well as several types of oligomeric versions (OGOLUT, OGORAF, OGOWUE, OGOXAL, OGOXEP, OGOXIT, OGOXOZ, OGOXUF, OGOYAM, Kozma et al., 2015; SUSWAG, SUSWOU, Nacsa & Lambert, 2015).

5. Synthesis and crystallization

Initially, crystals of [1H]Cl-C6H6 were obtained from the commercial sample of 1 via a double-vial apparatus by dissolution of N-butyl-2,3-bis(dicyclohexylamino)cyclopropenimine (I) in benzene in an inner vial, and charging the outer vial with hexanes. After diffusion for a few days at room temperature, powdery solid and a few colorless crystals of [1H]Cl-C6H6 were observed inside. The yield of crystalline [1H]Cl-C6H6 was significantly improved by the addition of HCl. To a glass shell vial containing 7.2 mg of N-butyl-2,3-bis(dicyclohexylamino)cyclopropenimine, 2 mL of benzene were added. A drop of dilute HCl (0.730 M) was added. This was diffused with 3 mL of hexanes in the outer vial for 2–3 days. Crystallization works best when the drop is not in contact with the walls of the vial where the crystals grow. Crystals were isolated by decanting the liquid from the inner vial using a disposable pipette, and taking care to remove the visible aqueous HCl droplet with the first pipette draw. After removing the mother liquor, the crystals were rinsed with hexanes. Yield 6.3 mg (70%). Yields in this small-scale preparation ranged from 22% to 70% across multiple attempts. \(^1\)H NMR (ppm) 400 MHz, CDCl3: δ(ppm): 0.97 (t, 3H, Me), 1.62–1.82 (m, 14H, Cy and Bu), 1.62–1.76 (m, 14H, Cy and Bu), 1.80 (d, 8H, Cy-β-H), 1.96 (d, 8H, Cy-β-H), 3.34
Table 2
Experimental details.

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<th>Crystal data</th>
<th>Chemical formula</th>
<th>M</th>
<th>Crystal system, space group</th>
<th>Temperature (K)</th>
<th>a, b, c (Å)</th>
<th>β (°)</th>
<th>V (Å³)</th>
<th>Z</th>
<th>Radiation type</th>
<th>μ (mm⁻¹)</th>
<th>Crystal size (mm)</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>C₁₁H₂₃N₃⁺Cl⁻·C₆H₆</td>
<td>582.33</td>
<td>Monoclinic, P2₁/n</td>
<td>100</td>
<td>12.253 (3), 22.699 (7), 12.884 (3)</td>
<td>104.164 (7)</td>
<td>3474.6 (16)</td>
<td>4</td>
<td>Mo Kα</td>
<td>0.14</td>
<td>0.55 × 0.53 × 0.16</td>
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Data collection
Diffractometer Bruker D8 Quest Photon 100
Absorption correction Multi-scan (SADABS; Krause et al., 2015)

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<th>Tmin</th>
<th>Tmax</th>
<th>No. of measured, independent and observed [I &gt; 2σ(I)] reflections</th>
<th>Rint</th>
<th>(sinθ/λ)max (Å⁻¹)</th>
<th>Refinement</th>
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<tr>
<td>0.662</td>
<td>0.746</td>
<td>48756, 8072, 6516</td>
<td>0.044</td>
<td>0.658</td>
<td></td>
</tr>
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</table>

H-atom treatment H atoms treated by a mixture of independent and constrained refinement

Δρmax, Δρmin (e Å⁻³)

0.55, -0.40

Computer programs: COSMO, XPREP, and SAINT (Bruker, 2008), SHELXTL (Sheldrick, 2015a), SHELX11 (Sheldrick, 2015b), and OLEX2 (Dolomanov et al., 2009).

(π, 4H, Cy-α-H), 3.56 (t, 2H, Bu-α-H), 7.4 (s, 1H, NH). ¹³C NMR (ppm) (400 MHz, CDCl₃): δ(ppm): 13.97, 19.94, 24.58, 25.84, 32.34, 33.79, 46.15, 59.55, 114.01, 128.35. FTIR (cm⁻¹): 2926 (m), 2851 (m), 1503 (s), 1445 (m), 1383 (w), 1374 (w), 1345 (w), 1324 (w), 1253 (w), 1188 (w), 1180 (w), 1102 (w), 1092 (w), 1004 (w), 895 (w), 696 (m). Analysis calculated for C₁₁H₂₃N₃⁺·0.5 C₆H₆ (%): C 76.31; H, 10.38; N, 7.22. Found: C 75.873; H, 10.83; N, 7.24. M.p. 353–356 K (decomposes).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. A disordered methyl group was treated with a two-site disorder model, with atom positions freely refined, and relative occupancies refined using Free Variable 2 with a final ratio of 0.71 (3): 0.29 (3). RIGU/SIMU restraints were applied to the wagging methyl group. C—H hydrogen atoms were treated using a standard riding model. The imine proton was located as a peak in the Fourier difference map and was freely refined.

Hirshfeld charge was determined at the 3-21G/B3LYP level of theory by iterative computation of electronic structure of [1H]Cl-C₆H₆ using ORCA (Neese, 2018) followed by refinement of the structure using non-spherical form factors computed using NoSpherA2 (Kleemiss et al., 2021), and repeating the process until the structure converged. Hirshfeld charges resulting from this approach are given in Table 3.

Acknowledgements
The National Science Foundation under award 1800105, is gratefully acknowledged for support of this work. GMS acknowledges the Temple University Minority Access to Research Careers (MARC) program and its support from the National Institutes of Health under NIH/NIGMS award 5 T34 GM136494 for an undergraduate research fellowship.

Funding information
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References


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\(a = 12.253\) (3) Å

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\(\beta = 104.164\) (7)°

\(V = 3474.6\) (16) Å³

\(Z = 4\)

\(F(000) = 1280\)

Data collection

Bruker D8 Quest Photon 100
diffractometer

Radiation source: sealed tube

Detector resolution: 10.417 pixels mm⁻¹

\(\varphi\) and \(\omega\) scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

\(T_{\text{min}} = 0.662, T_{\text{max}} = 0.746\)

Refinement

Refinement on \(F^2\)

Least-squares matrix: full

\(R[F^2 > 2\sigma(F^2)] = 0.055\)

\(wR(F^2) = 0.136\)

\(S = 1.02\)

8072 reflections

385 parameters

6 restraints

Primary atom site location: dual

Secondary atom site location: difference Fourier map

Hydrogen site location: mixed

\(H\) atoms treated by a mixture of independent and constrained refinement

\(w = 1/[\sigma(F_o^2) + (0.0605P)^2 + 2.3777P]\)

where \(P = (F_o^2 + 2F_c^2)/3\)

\((\Delta\sigma)_{\text{max}} = 0.001\)

\(\Delta\rho_{\text{max}} = 0.55\) e Å⁻³

Computing details

Data collection: \(COSMO\) and \(XPREP\) (Bruker, 2008); cell refinement: \(SAINT\) (Bruker, 2008); data reduction: \(SAINT\) (Bruker, 2008); program(s) used to solve structure: \(SHELXT\) (Sheldrick, 2015a); program(s) used to refine structure: \(SHELXL\) (Sheldrick, 2015b); molecular graphics: \(OLEX2\) (Dolomanov et al., 2009); software used to prepare material for publication: \(OLEX2\) (Dolomanov et al., 2009).

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where \(P = (F_o^2 + 2F_c^2)/3\)

\((\Delta\sigma)_{\text{max}} = 0.001\)

\(\Delta\rho_{\text{max}} = 0.55\) e Å⁻³
Δρ_{\text{min}} = -0.40 \text{ e Å}^{-3}

Extinction correction: SHELXL2018/3
(Sheldrick 2015b),

\text{Fc}^\ast = k\text{Fc}[1+0.001\text{Fc}^2λ^2/\sin(2θ)]^{-1/4}

Extinction coefficient: 0.0081 (7)

**Special details**

**Experimental.** Single-crystal X-ray crystallographic data were obtained on a Bruker D8 Quest PHOTON 100 diffractometer with an Oxford Cryosystem 700 low-temperature device. The radiation was from a sealed-tube molybdenum Ka source with a TRIUMPH monochromator. Crystals were typically multiple, and a single piece was cut away with a razor blade, mounted on a MiTeGen loop with paratone-N oil, and collected at 100K in \(ω/φ\) scansets.

Integration was performed using SAINT, and data were reduced and absorption-corrected using SADABS (Bruker, 2008). Space group determination was performed using XPREP (Sheldrick, 2008), and the structure was solved using intrinsic phasing using SHELXT (Sheldrick, The structural model of [1H]Cl·C₆H₆ was refined using the least-squares approach with the ShelX package (Sheldrick, 2015a) with Olex2 as a GUI (Dolomanov et al., 2009). 2015b).

**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)**

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