



**Queensland University of Technology**  
Brisbane Australia

This is the author's version of a work that was submitted/accepted for publication in the following source:

Smith, Graham, Wermuth, Urs D., & Williams, Michael L. (2012) Resolution of the chiral (1R,2S) enantiomer of cis-cyclohexane-1,2-dicarboxylic acid in the brucinium salt 2,3-dimethoxy-10-oxostrychnidinium (1R,2S)-2-carboxycyclohexane-1-carboxylate dihydrate. *Journal of Chemical Crystallography*, 42(6), pp. 555-559.

This file was downloaded from: <http://eprints.qut.edu.au/51201/>

© Copyright 2012 Springer

<http://www.springerlink.com>

**Notice:** *Changes introduced as a result of publishing processes such as copy-editing and formatting may not be reflected in this document. For a definitive version of this work, please refer to the published source:*

<http://dx.doi.org/10.1007/s10870-012-0278-9>

# Journal of Chemical Crystallography

## Resolution of the (1R,2S) Enantiomer of cis-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-Dimethoxy-10-oxostrychnidinium (1R,2S)-2-carboxycyclohexane-1-carboxylate Dihydrate.

--Manuscript Draft--

<b>Manuscript Number:</b>	JOCC2357R1
<b>Full Title:</b>	Resolution of the (1R,2S) Enantiomer of cis-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-Dimethoxy-10-oxostrychnidinium (1R,2S)-2-carboxycyclohexane-1-carboxylate Dihydrate.
<b>Article Type:</b>	Original Research
<b>Keywords:</b>	brucine; cis-cyclohexane-1,2-dicarboxylic acid; proton-transfer compound; hydrogen bonding
<b>Corresponding Author:</b>	Graham Smith, PhD Queensland University of Technology Brisbane, Queensland AUSTRALIA
<b>Corresponding Author Secondary Information:</b>	
<b>Corresponding Author's Institution:</b>	Queensland University of Technology
<b>Corresponding Author's Secondary Institution:</b>	
<b>First Author:</b>	Graham Smith, PhD
<b>First Author Secondary Information:</b>	
<b>Order of Authors:</b>	Graham Smith, PhD Urs D. Wermuth, PhD Michael L. Williams, PhD
<b>Order of Authors Secondary Information:</b>	
<b>Abstract:</b>	<p>The structure of the 1:1 brucinium salt of cis-cyclohexane-1,2-dicarboxylic acid, 2,3-dimethoxy-10-oxostrychnidinium (1R,2S)-2-carboxycyclohexane-1-carboxylate dehydrate, has revealed the resolved (1R,2S) enantiomer of the acid. Crystals of the compound are orthorhombic, space group P212121, with unit cell dimensions <math>a = 8.1955(3)</math>, <math>b = 12.4034(3)</math>, <math>c = 29.9073(9)</math> Å, and <math>Z = 4</math>. The asymmetric unit comprises the brucinium cation, the hydrogen cis-cyclohexane-1,2-dicarboxylate cation, in which the carboxylate group is disordered over two sites (58, 42%), and two water molecules of solvation, one of which occupies two 50% sites. The classic undulating brucinium cation substructures are present, with the anion and the water molecules occupying the interstitial cavities and are hydrogen-bonded to them in a two-dimensional network structure.</p>
<b>Response to Reviewers:</b>	<p>Article JOCCxxxx</p> <p>Replies and Actions taken post-Review for the Article.</p> <p>Minor corrections have also been made where indicated by the Reviewers. Also, I have completed a re-refinement of the structure paying attention to the handling of the disorder in the carboxylate group of the cyclohexane-1,2-carboxylate (CHDC) monoanion and in so doing I have rectified the problem of the high difference residual in the original structure. There has been little change to the original Discussion except in those points pertaining to the disorder and the numerical values contained in the text and tables. All figures except Fig. 2 remain unchanged from the original submission.</p> <p>(1) The revised manuscript has been uploaded. (2) The revised Fig. 2 as been uploaded together with the Index Abstract figure. (3) The revised CIF has been submitted to the CSD replacing the original deposited</p>

CIF (CCDC 838579)

Responses are as below:

Reviewer #1.

Points (1)-(4).

The structure shows no signs of major structural problems, the only oddity is the rotationally disordered carboxyl group of the CHDC monoanion. On the matter of space group ambiguity the above is not going to be the reason. The disorder affects only two carboxyl O atoms in a total formula of 43 non-H atoms. Furthermore the space group options are limited with brucine structures with the requirement of chirality, the known space groups being limited to four in total: P1, P21, C2 and P212121, the latter providing probably 60% of the total.

On these counts I therefore reject a mis-assigned space group or crystal class as being the causative factor in the resultant higher-than-expected R factors.

However, in the structure, although the brucinium cation is thermally stable with small, almost isotropic displacement parameters ( $U_{eq}$  ca. 0.03-0.04), the values for the atoms of the CHDC anion are invariably larger and irregularly anisotropic (0.04-0.10). This I would suggest is the major contributing factor to the problem and the higher-than-expected refinement residual.

Supporting this contention is the observation that the CHDC anions in a number of structures I have determined recently (there are very few examples of cis-CHDC salts in the crystallographic literature) (three examples are in fact contained in a JOCC paper which I am working on at the moment, two of which have significant conformational disorder in the CHDC ring).

Reviewer #2.

The modelling of the disordered carboxylate group.

I have addressed this matter applying DANG constraints to both the major and minor components of the disordered carboxyl group. In addition I have refined the O-atoms with the incorporation of a split carboxyl O (O15A) with an S.O.F. of 0.12 which now absorbs the electron density which contributed to the abnormally large residual electron density ( $1.07 \text{ e}\text{\AA}^{-3}$ ). This residual now is more satisfactory at  $0.54 \text{ e}\text{\AA}^{-3}$ . The Refinement residual is not changed significantly ( $R = 0.068$ )

.

**(a) Cover Page**

**Resolution of the Chiral (1*R*,2*S*) Enantiomer of *cis*-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-Dimethoxy-10-oxostrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate Dihydrate.**

**Graham Smith\***

Science and Engineering Faculty, Queensland University of  
Technology, G.P.O. Box 2434, Brisbane, Qld. 4001, Australia.

Telephone: (07) 3864 2293; Fax. (07) 3864 1804; e-mail: [g.smith@qut.edu.au](mailto:g.smith@qut.edu.au).

**Urs D. Wermuth**

Science and Engineering Faculty, Queensland University of  
Technology, G.P.O. Box 2434, Brisbane, Qld. 4001, Australia.

**Michael L. Williams**

School of Biomolecular and Physical Sciences, Griffith University  
Nathan, Qld. 4111, Australia.

**(b) Index Abstract**

Resolution of the (1*R*,2*S*) Enantiomer of *cis*-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-dimethoxy-10-oxostrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate Dihydrate.

by *Graham Smith, Urs D. Wermuth and Michael L. Williams*

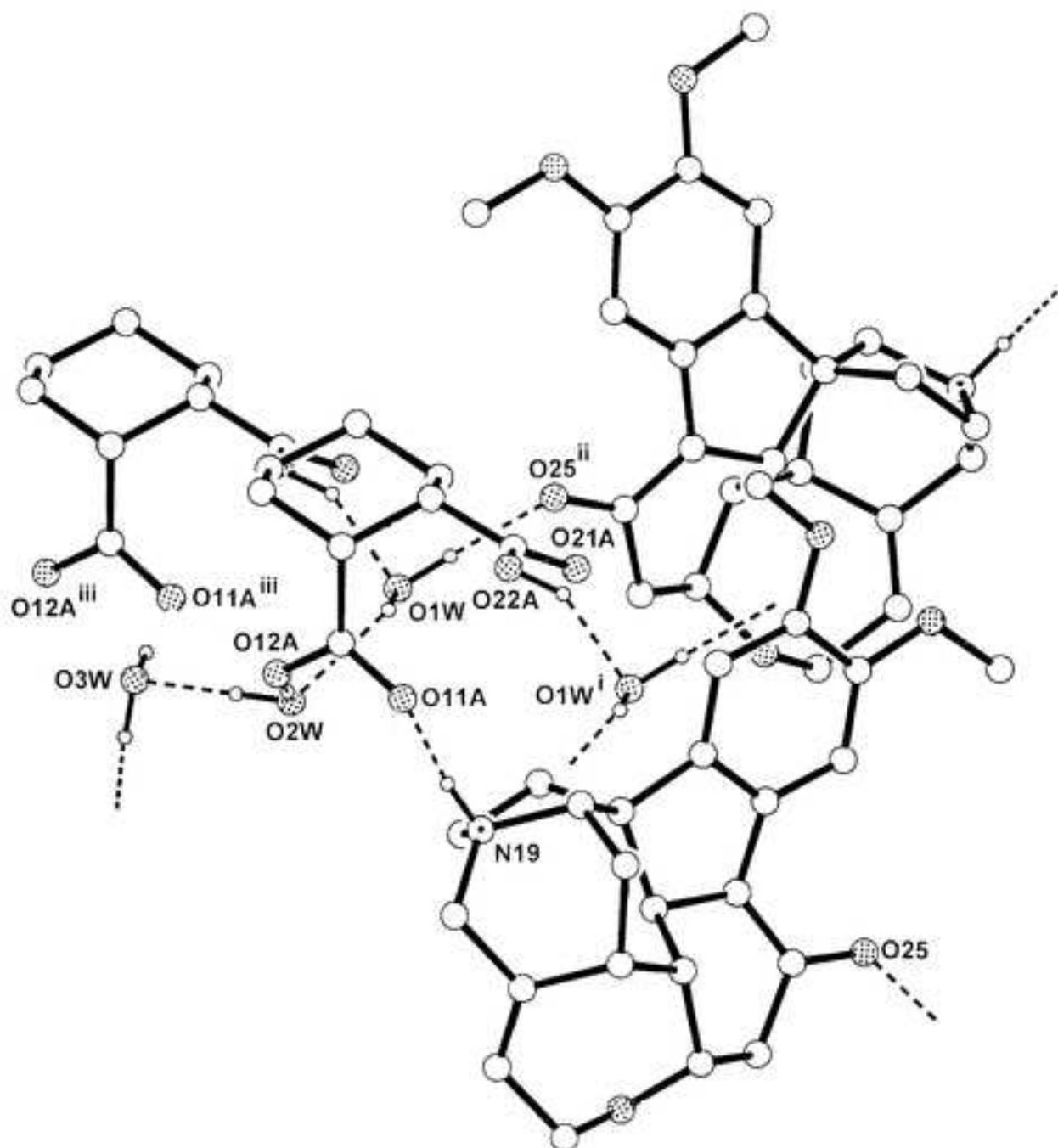
---

The structure determination of the 1:1 proton-transfer hydrate salt of *cis*-cyclohexane-1,2-dicarboxylic acid with brucine has revealed the presence of the (1*R*,2*S*) enantiomeric form of the acid in a hydrogen-bonded structure.

---

**Figure for insertion in Index Abstract: (CHDCBRUAds.TIF)**

---



**(a) Cover Page**

**Resolution of the Chiral (1*R*,2*S*) Enantiomer of *cis*-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-Dimethoxy-10-oxostyrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate Dihydrate.**

**Graham Smith\***

Science and Engineering Faculty, Queensland University of  
Technology, G.P.O. Box 2434, Brisbane, Qld. 4001, Australia.  
Telephone: (07) 3864 2293; Fax. (07) 3864 1804; e-mail: [g.smith@qut.edu.au](mailto:g.smith@qut.edu.au).

**Urs D. Wermuth**

Science and Engineering Faculty, Queensland University of  
Technology, G.P.O. Box 2434, Brisbane, Qld. 4001, Australia.

**Michael L. Williams**

School of Biomolecular and Physical Sciences, Griffith University  
Nathan, Qld. 4111, Australia.

**(b) Index Abstract**

Resolution of the (1*R*,2*S*) Enantiomer of *cis*-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-dimethoxy-10-oxostyrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate Dihydrate.

by *Graham Smith, Urs D. Wermuth and Michael L. Williams*

---

The structure determination of the 1:1 proton-transfer hydrate salt of *cis*-cyclohexane-1,2-dicarboxylic acid with brucine has revealed the presence of the (1*R*,2*S*) enantiomeric form of the acid in a hydrogen-bonded structure.

---

**Figure for insertion in Index Abstract: (CHDCBRUAbs.TIF)**

---

**(c,d). Title: Authors and Affiliations**

**Title**

**Resolution of the (1*R*,2*S*) Enantiomer of *cis*-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-Dimethoxy-10-oxostrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate Dihydrate.**

**Authors and Affiliations**

Graham Smith\*

Science and Engineering Faculty, Queensland University of  
Technology, G.P.O. Box 2434, Brisbane, Qld. 4001, Australia.  
Telephone: (07) 3864 2293; Fax. (07) 3864 1804; e-mail: [g.smith@qut.edu.au](mailto:g.smith@qut.edu.au).

Urs D. Wermuth

Science and Engineering Faculty, Queensland University of  
Technology, G.P.O. Box 2434, Brisbane, Qld. 4001, Australia.

Michael L. Williams

School of Biomolecular and Physical Sciences, Griffith University  
Nathan, Qld. 4111, Australia.

**Abstract**

The structure of the 1:1 brucinium salt of *cis*-cyclohexane-1,2-dicarboxylic acid, 2,3-dimethoxy-10-oxostrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate dihydrate, has revealed the resolved (1*R*,2*S*) enantiomer of the acid. Crystals of the compound are orthorhombic, space group  $P2_12_12_1$ , with unit cell dimensions  $a = 8.1955(3)$ ,  $b = 12.4034(3)$ ,  $c = 29.9073(9)$  Å, and  $Z = 4$ . The asymmetric unit comprises the brucinium cation, the hydrogen *cis*-cyclohexane-1,2-dicarboxylate cation, in which the carboxylate group is disordered over two sites (58, 42%), and two water molecules of solvation, one of which occupies two 50% occupancy sites. The

classic undulating brucinium cation substructures are present with the anion and the water molecules occupying the interstitial cavities and are hydrogen-bonded to them in a two-dimensional network structure.

**Key Words:** brucine; *cis*-cyclohexane-1,2-dicarboxylic acid; proton-transfer compound; hydrogen bonding.

**Running Title:**

brucinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate dihydrate



**Resolution of the Chiral (1*R*,2*S*) Enantiomer of *cis*-Cyclohexane-1,2-dicarboxylic Acid in the Brucinium Salt 2,3-Dimethoxy-10-oxostrychnidinium (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate Dihydrate.**

by

Graham Smith\* , Urs D. Wermuth and Michael L. Williams

---

**Introduction**

Cyclohexane-1,2-dicarboxylic acid may exist in two diastereoisomeric forms, one *trans* [(1*R*,2*R*) or (1*S*,2*S*)], the other *cis* [(1*R*,2*S*) or (1*S*,2*R*)]. Whereas the *trans*-isomer forms separable *dl* pairs, with the *cis*-isomer, the *dl* pairs are inseparable because the two enantiomeric forms have a low interconversion potential, hence forming racemic pairs [1]. Quinine and cinchonidine have been used for the resolution of the *trans*-form of the acid [2] but brucine has not, although it has been used on a hit-or-miss basis for the resolution of racemic carboxylic acids, first by Emil Fischer in 1899 [3] with the *N*-benzoyl derivatives of the amino acids alanine, glutamic acid and aspartic acid. The crystal structures of a number of these chiral salts have been determined, e.g. *N*-benzoyl-D- $\alpha$ -alanine [4]; (*S*)-3-[2,4--dimethyl-5-(ethoxycarbonyl)-1*H*-pyrrol-3-yl]butanoic acid [5];  $\alpha$ -D-glucuronic and  $\beta$ -D-galacturonic acids [6]; *N*-phthaloyl-L- $\alpha$ -alanine [7]; L-glyceric acid [8] and (*S*)-malic acid and (2*R*,3*R*)-tartaric acid [9].

The crystalline product from our 1:1 stoichiometric reaction of cyclohexane-1,2-dicarboxylic anhydride with brucine in aqueous ethanol was the hydrated title salt  $C_{23}H_{27}N_2O_4^+ C_8H_{11}O_4^- \cdot 2H_2O$  (**1**) (scheme 1) and the structure is reported here.

**INSERT 1 Schematic of 1 is given here (CHDCBRU.eps)**

The molecular structure of **1**

### **Experimental**

*Preparation.* The title compound **1** was synthesized by heating together under reflux for 10 min., 1 mmol quantities of brucine tetrahydrate and cyclohexane-1,2-dicarboxylic anhydride in 50 mL of 50% ethanol-water. After concentration to ca. 30 mL, partial room temperature evaporation of the hot-filtered solution gave colourless plates of **1** (m.p. 101 °C) from which a specimen was cleaved for the X-ray analysis.

*Crystallography.*

X-ray diffraction data were acquired for **1** at 180(2) K on an Oxford Diffraction Gemini-S Ultra CCD-detector diffractometer employing graphite crystal monochromatized Mo  $K\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ). Data collection and reduction was completed using CrysAlis PRO [10] with data corrected for absorption (multi-scan, CrysAlis PRO [10]). The structure was solved using direct methods (SIR92 [11]) and refined with SHELXL97 [12] operating within WinGX [13]. The carboxylate group at C1 of the anion was found to be rotationally disordered over three sites with the occupancy of the four oxygen atoms being determined as O11A (0.58), O12A (0.38), O15A (0.12) and O13A, and O14A both 0.42), all components subsequently being refined isotropically.

Water molecules O3W and O4W were found to have 50(1)% occupancy. Hydrogen atoms potentially involved in hydrogen-bonding interactions were located by difference methods but their positional and isotropic thermal parameters were constrained in the final refinement cycles. Other H atoms were included in the refinement at calculated positions and treated as riding with C-H = 0.93-0.98Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . In the absence of a heavy atom and because the known absolute structure for the strychnidin-10-one cage was invoked [14] [giving the overall absolute structure classification as C7(*S*), C8(*S*), C12(*S*), C13(*R*), C14(*R*), C16(*S*), N19(*S*) for the brucinium cation], the Friedel pairs (2868) being merged in the final cycles of refinement. General crystallographic details are given in Table 1. The atom numbering scheme employed for the brucinium cation species (Fig. 1) [15] follows the original Robinson convention for strychnine [16].

## Results and Discussion

In the structure of compound **1**, protonation has occurred as expected at N19 of the brucine cage (Fig. 1), the invoked Peerdeman absolute configuration [14] for the seven chiral centres in the brucinium cation allowing the assignment of the configuration of the *cis*-anion component as (1*R*,2*S*)-2-carboxycyclohexane-1-carboxylate. In this anion, the carboxylate group has an atypical (for this anionic species) rotational disorder giving four oxygen atoms sites for the two groups [O11A, O12A (S.O.F = 0.58(1)) and O13A, O14A (S.O.F. = 0.42(1))].

**INSERTS 2, 3 :**

**Figure 1 (Atom numbering scheme for the cation, anion and water species in the asymmetric unit of 1) CHDCBRU1.TIF**

**Figure 2 (The cation-anion-water hydrogen-bonding interactions)**

**CHDCBRU2.TIF**

The brucinium cations form the previously described head-to-tail overlapping, undulating sheet host substructures [4,6-9] with a dimeric repeat period in **1** along the direction of propagation (a  $2_1$  screw axis: the *b* cell dimension) of 12.4034(3)Å [16] (Figs. 2, 3). This value is consistent with those for similarly structured brucine compounds (range 12.366-12.660 Å ) [4, 17]. The monoanion and the three water molecules [two of which (O2W and O3W) have a site occupancy of 0.50(1)], occupy the interstitial spaces between the brucine substructures and are hydrogen-bonded to them (Table 1, Fig. 2), giving a two-dimensional structure (Fig. 4). Interactions include brucine aminium N-H...O(carboxyl), water O-H...O(brucine carbonyl), and strong carboxylic acid O-H...O(water) hydrogen bonds. In addition, the water molecules are inter-associated, with all giving hydrogen-bonding interactions, with the exception of one of the H atoms of water molecule O3W (H32W) which has no reasonable acceptor in the structure. The water stabilization of brucinium carboxylates is quite common, e. g. the *N*-benzoylalaninate [4] (a 4.5-hydrate); the glycerate [8] (a 4.75--hydrate); the dihydrogen citrate [18] (a trihydrate), and the (*S*)-malate [9] (a pentahydrate).

**INSERTS 4 and 5:**

**Figure 3 (the undulating brucine substructure) (CHDCBRU3.TIF)**

**Figure 4 (packing of **1** in the unit cell) CHDCBRU4.TIF)**

## Conclusion

This work provides another example of the molecular selectivity of brucine in forming stable salts having the classic brucine host substructures, and also represents the first reported structure of a resolved enantiomer of *cis*-cyclohexane-1,2-dicarboxylic acid or its salt as a guest molecule.

## Supplementary material

CCDC 838579 contains the supplementary crystallographic data for compound **1** from this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* [http://www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif) or by e-mailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

## Acknowledgements.

The author acknowledges financial support from the Australian Research Council, the Science and Engineering Faculty (Queensland University of Technology) and Griffith University.

## References

1. Eliel EL (1962) Stereochemistry of Carbon Compounds. McGraw-Hill, New York, pp. 211-215
2. Newman P (1982) Optical Resolution Procedures for Chemical Compounds Vol. 2 Acids, Part 1. Optical Resolution Centre, New York, pp. 332-333
3. Fischer E (1899) Ber 32:2451
4. Gould RO, Walkinshaw MD (1984) J Am Chem Soc 106:7840
5. Boiadjiev SE, Person RV, Puzicha G, Knobler C, Maverick E, Trueblood KN, Lightner DA (1992) J Am Chem Soc 114:10123
6. Dijkstra FJJ, Gould, RO, Parsons S, Taylor P, Walkinshaw MD (1998) Chem Commun:745
7. Bialońska A, Ciunik Z (2004) CrystEngComm 6:276

8. Bialońska A, Ciunik Z, Popek T, Lis T (2005) *Acta Crystallogr C*61: o88
9. Smith G, Wermuth UD, White JM (2006) *Acta Crystallogr C*62:o353
10. CrysAlis PRO (2010) (version 1.171.55). Agilent Technologies Ltd., Yarnton, Oxfordshire, England
11. Altomare A, Cascarno C, Giacovazzo A, Guagliardi A, Burla MC, Polidori G, Camalli M (1994). *J Appl Crystallogr* 27:435
12. Sheldrick GM (2008) *Acta Crystallogr A*64:112
13. Farrugia LJ (1999) *J Appl Crystallogr* 32:837
14. Peerdeman AF (1956). *Acta Crystallogr.* 9:824.
15. Spek AL (2009) *Acta Crystallogr D*65:48
16. Holmes HL (1952) In: Manske RFH, Holmes HL (eds). *The Alkaloids Vol II*. Academic, New York, p514
17. Smith G, Wermuth UD, Healy PC, White JM. (2006) *Aust J Chem* 59:320
18. Smith G, Wermuth UD, White JM. (2005) *Acta Crystallogr C*61:o621

**Table 1.** Crystal data for compound **1**.

Compound	<b>1</b>
CCDC reference	838579
Molecular formula	C <sub>31</sub> H <sub>42</sub> N <sub>2</sub> O <sub>10</sub>
<i>M<sub>r</sub></i>	603.67
Temperature (K)	180(2)
Wavelength (λ)	0.71073
Crystal system	orthorhombic
Space group	<i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
<i>a</i> (Å)	8.1955(3)
<i>b</i> (Å)	12.4034(3)
<i>c</i> (Å)	29.9073(9)
<i>V</i> (Å <sup>3</sup> )	3040.14(16)
<i>Z</i>	4
<i>D<sub>c</sub></i> (g cm <sup>-3</sup> )	1.317
μ (mm <sup>-1</sup> )	0.098
<i>F</i> (000)	1288
Instrument	Oxford Diffraction CCD
Reflections total, θ <sub>max</sub> (°)	11479, 28.7
Crystal size (mm)	0.40 x 0.30 x 0.28
Collection range:	
<i>h</i>	-10 to 7
<i>k</i>	-9 to 15
<i>l</i>	-24 to 39
Reflections (independent)	3953
Reflections [ <i>F</i> <sup>2</sup> > 2 σ( <i>F</i> <sup>2</sup> )]	3378
<i>R</i> <sub>int</sub>	0.0287
<i>R</i> 1 <sup>a</sup> [ <i>F</i> <sup>2</sup> > 2σ( <i>F</i> <sup>2</sup> )]	0.0684
<i>wR</i> 2 <sup>a</sup> (all data)	0.2007
<i>S</i> <sup>a</sup>	1.04
<i>n<sub>p</sub></i>	399
Transmission factors (max/min)	0.990/0.908
Residuals (max/min) (eÅ <sup>-3</sup> )	0.544/-0.385

$$^a R1 = (\sum |F_o| - |F_c|) / \sum |F_o|; wR2 = \{\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]\}^{1/2}; S = \{\sum [w(F_o^2 - F_c^2)^2] / (n-p)\}^{1/2}.$$

**Table 2.** Hydrogen-bonding interactions ( $\text{\AA}/^\circ$ ) for **1**

D-H...A	D-H	H...A	D...A	$\angle$ DH..A
N19-H19...O11A	0.91	1.69	2.583(6)	168
N19-H19...O13A	0.91	1.78	2.657(10)	162
O22A-H22A...O1W <sup>i</sup>	0.91	1.77	2.601(7)	151
O1W-H11W...O25 <sup>ii</sup>	0.92	1.83	2.753(6)	173
O1W-H12W...O2W	0.80	2.05	2.631(10)	130
O2W-H21W...O12A	0.88	1.78	2.686(15)	178
O2W-H22W...O3W	0.90	1.68	2.582(13)	180
O3W-H31W...O14A <sup>iii</sup>	0.90	2.41	3.015(18)	127

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



## Figures

**Figure 1.** Atom numbering scheme for the cation, anion and the two water molecules of solvation, two of which (O2W,O3W) have 50% occupancy. The carboxylate group is also disordered over two sites. Inter-species hydrogen bonds are shown as dashed lines and non-hydrogen atoms are shown as 30% probability ellipsoids [26].

**Figure 2.** The cation-anion-water hydrogen-bonding environment in **1** showing the head-to-tail overlap of the brucinium cations in the substructure extending along *b*. For symmetry codes, see Table 2.

**Figure 3.** The convoluted brucinium cation host substructures with interstitial guest anions and water molecules removed.

**Figure 4.** A perspective view of **1** in the unit cell viewed down the approximate *a* axial direction. The minor disordered component of the carboxylate group is omitted for clarity.

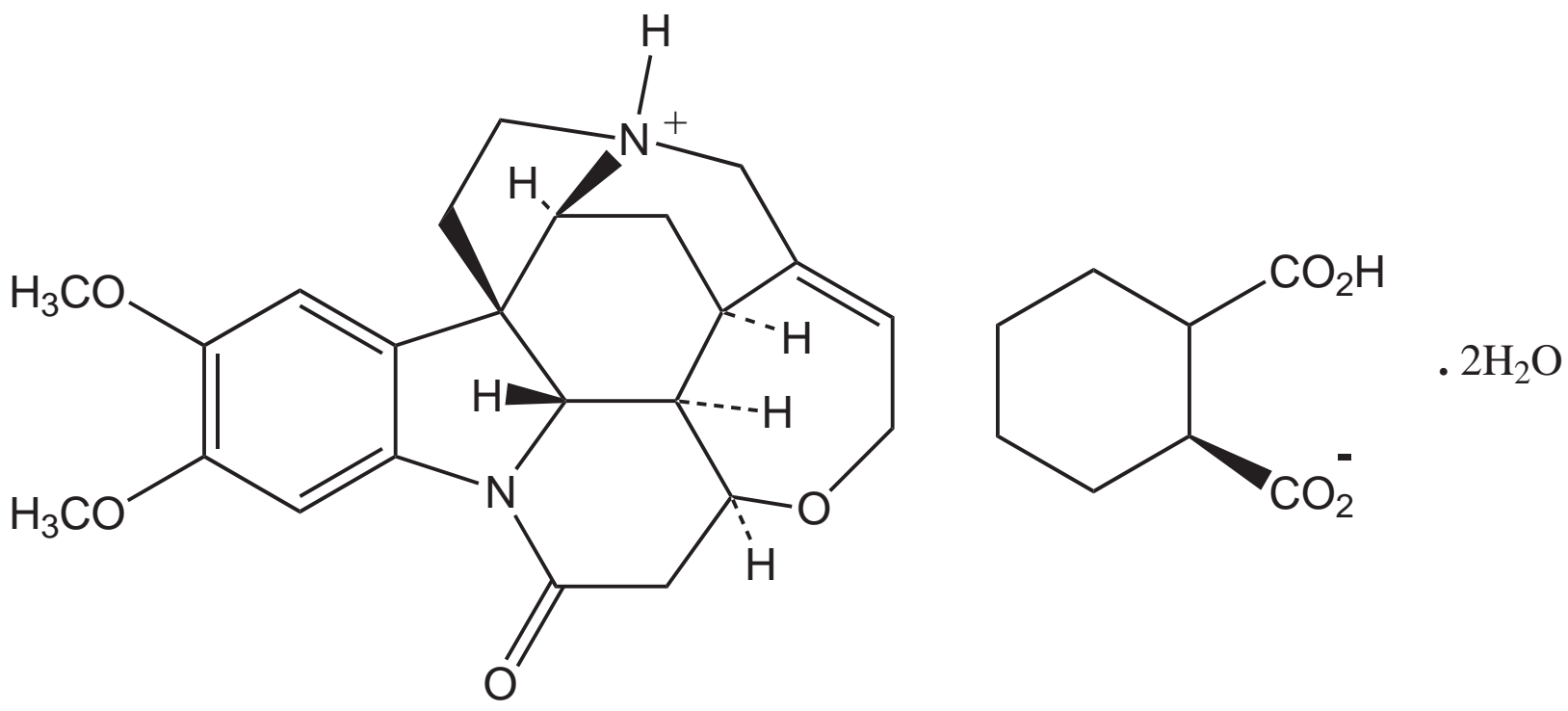


Figure 1  
[Click here to download high resolution image](#)

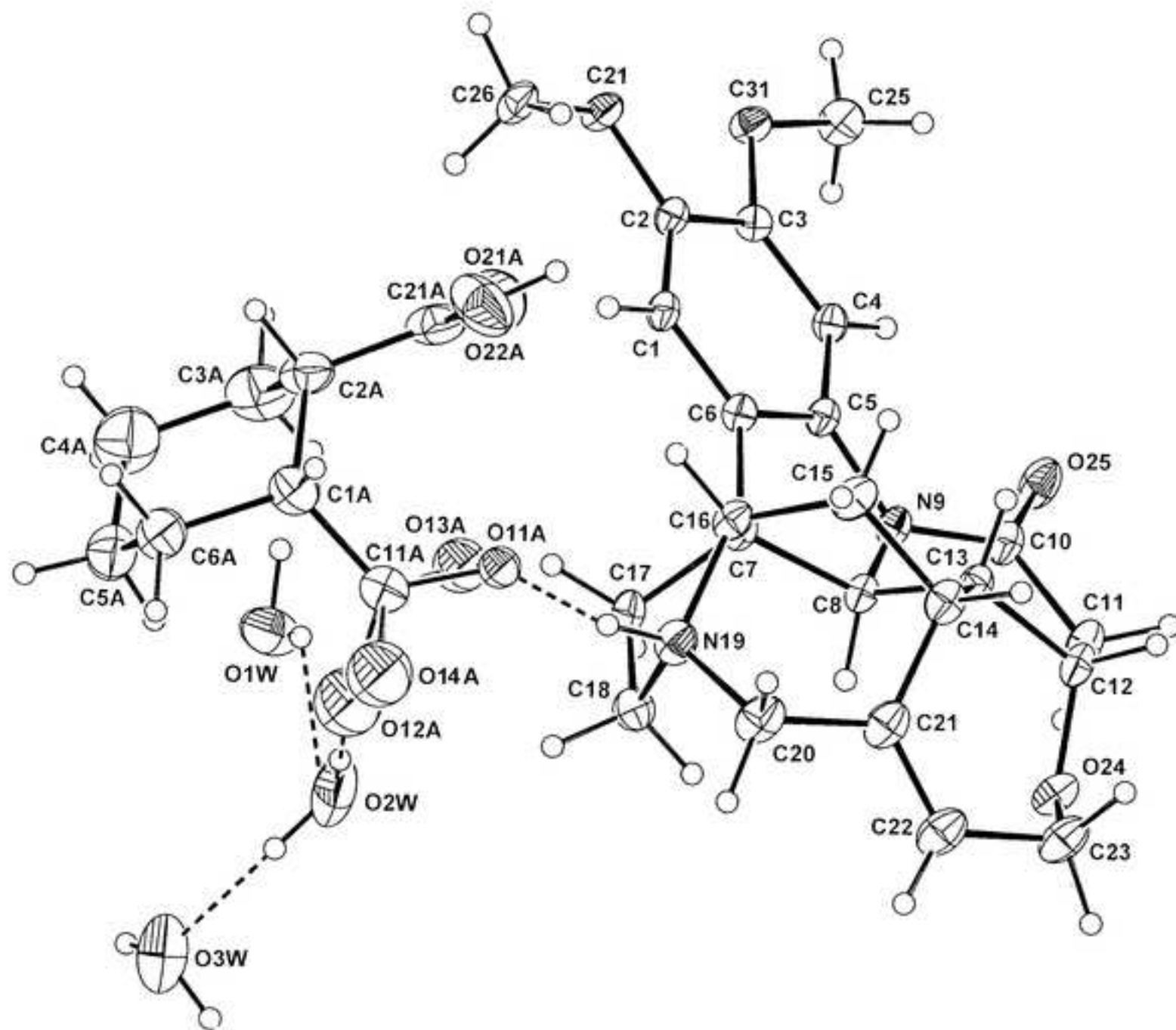


Figure 2  
[Click here to download high resolution image](#)

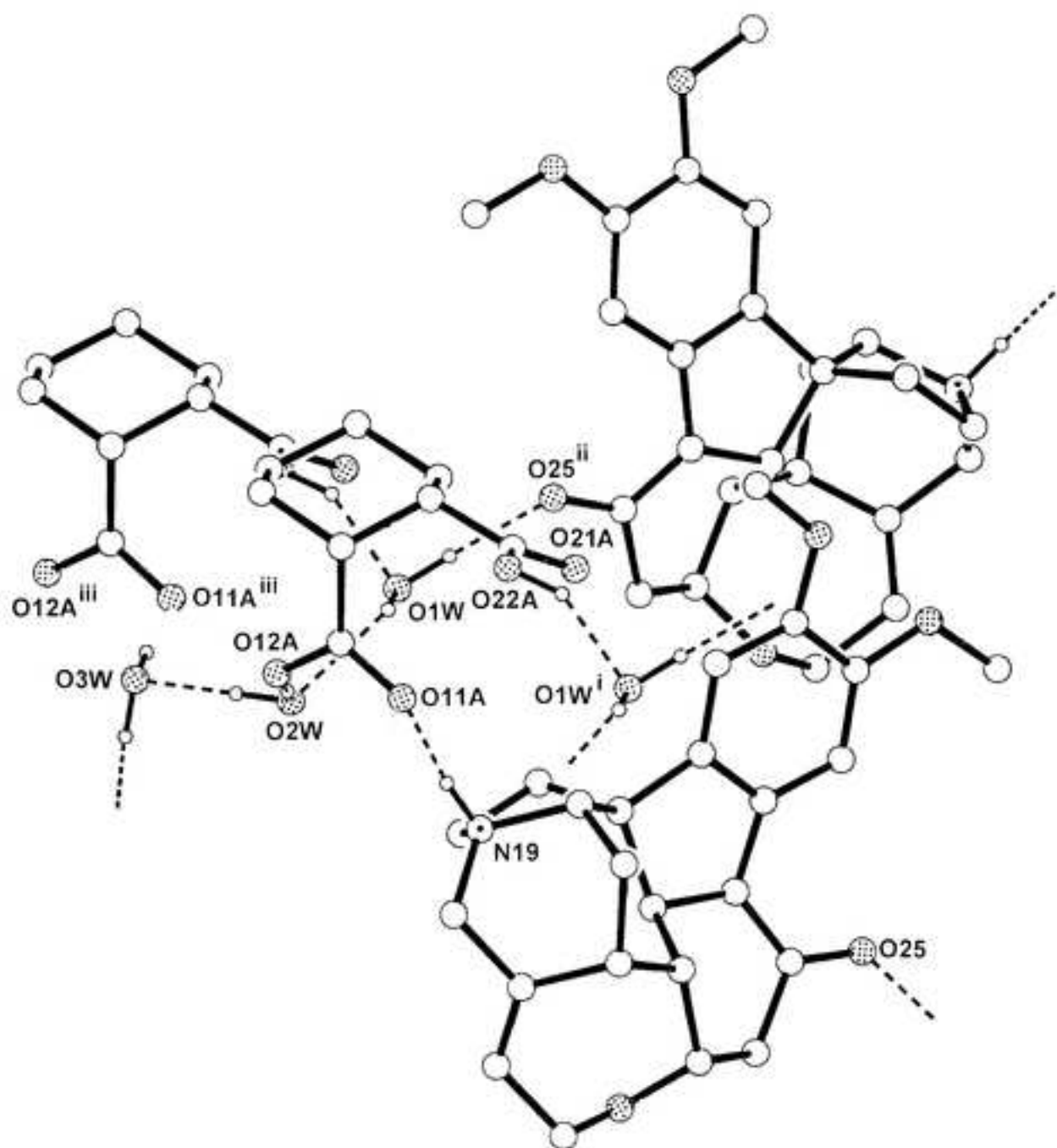


Figure 4  
[Click here to download high resolution image](#)

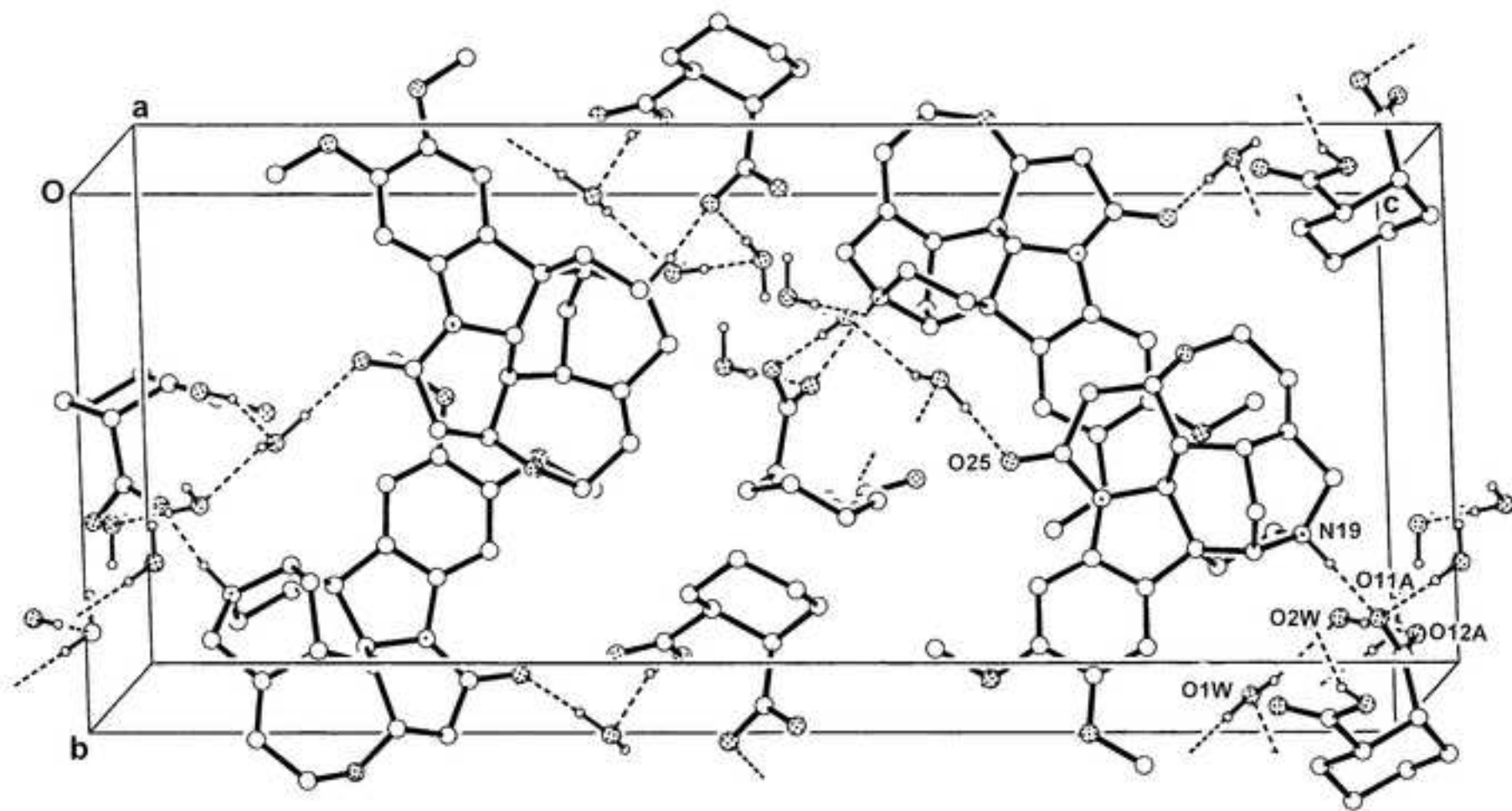
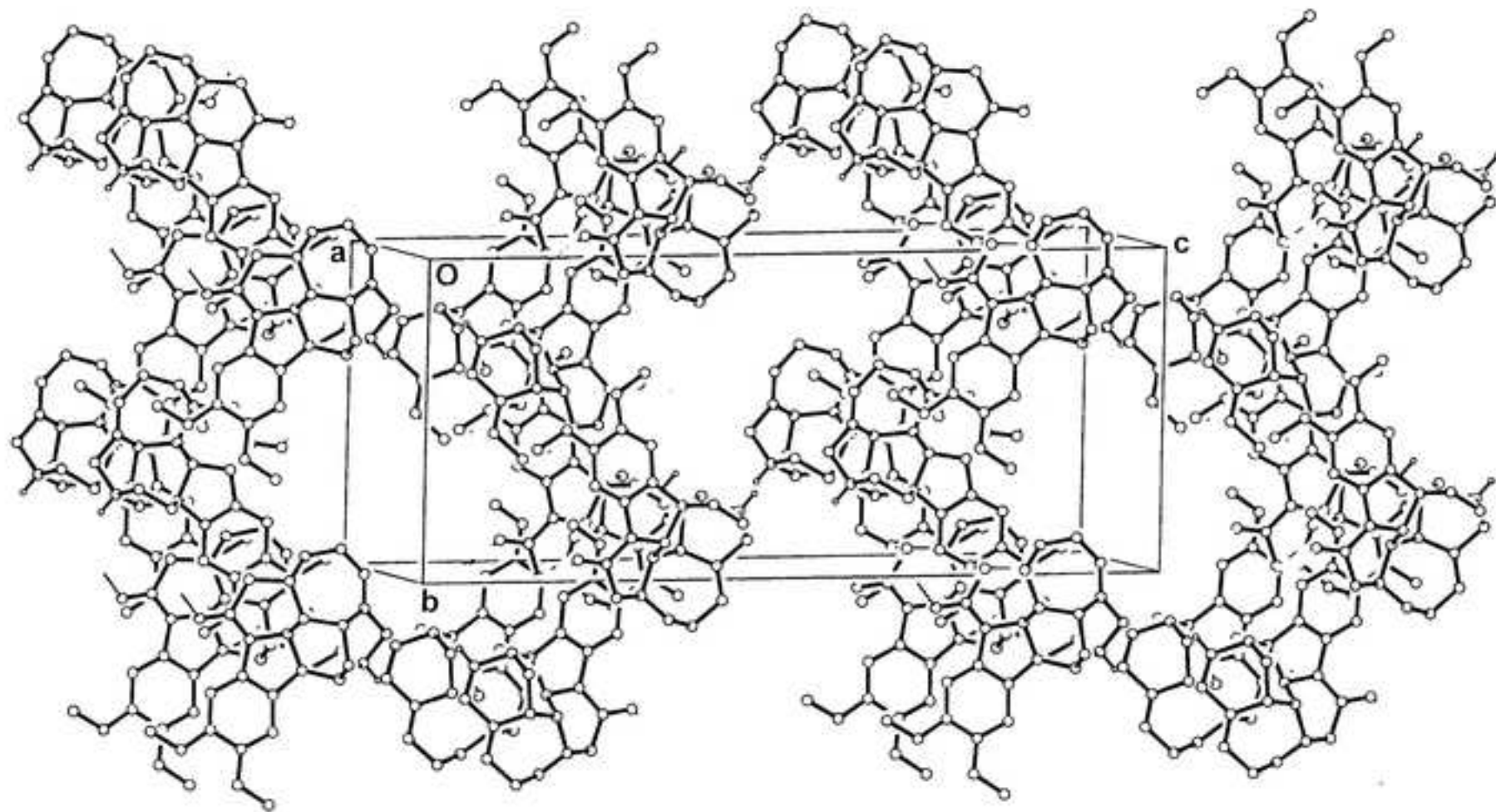


Figure 3  
[Click here to download high resolution image](#)



## TRANSFER OF COPYRIGHT

To be signed by the author or, in the case of multiple authorship, by at least one of the authors who agrees to inform the others, or, in the case of a work made for hire, by the employer. The signed statement must be received by the Editor before the manuscript can be accepted for publication. **The author's signature on this form certifies that the form has not been altered in any way.**

Copyright to the article by G. SMITH, U. D. WIERNITZ & M. L. WILLIAMS

submitted under the title RESOLUTION OF THE (1R, 2S) GRANITUMITE OF KAPSCYONIAZANKA - 2011

is hereby transferred to Plenum Publishing Corporation effective if and when the article is accepted for publication in

## JOURNAL OF CHEMICAL CRYSTALLOGRAPHY

For authors who are employees of the United States Government, the transfer of copyright is understood to be effective only to the extent that such copyright is transferable.

The authors explicitly reserve the following rights:

All proprietary rights other than copyright, such as patent rights.

The right to use all or part of this article in future works of their own, such as lectures, reviews, textbooks, or reprint books.

The right to make copies for the authors' own teaching use.

The right to use figures and tables in future publications, provided explicit acknowledgment is made of their initial appearance in this journal.

  
\_\_\_\_\_  
Signature

\_\_\_\_\_  
Signature

GRAHAM SMITH  
\_\_\_\_\_  
Name (print)

\_\_\_\_\_  
Name (print)

\_\_\_\_\_  
Title, if not Author

\_\_\_\_\_  
Title, if not Author

10/8/2011  
\_\_\_\_\_  
Date

\_\_\_\_\_  
Date