An Acta E from Hell

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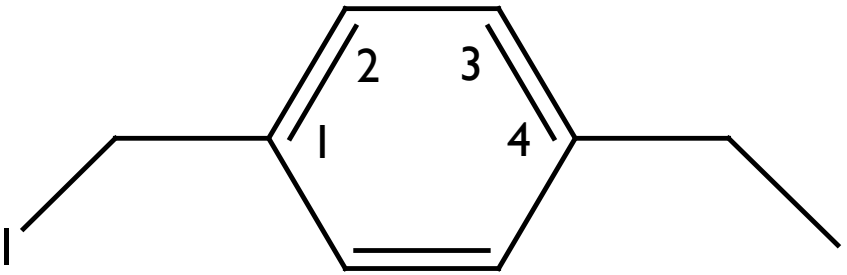
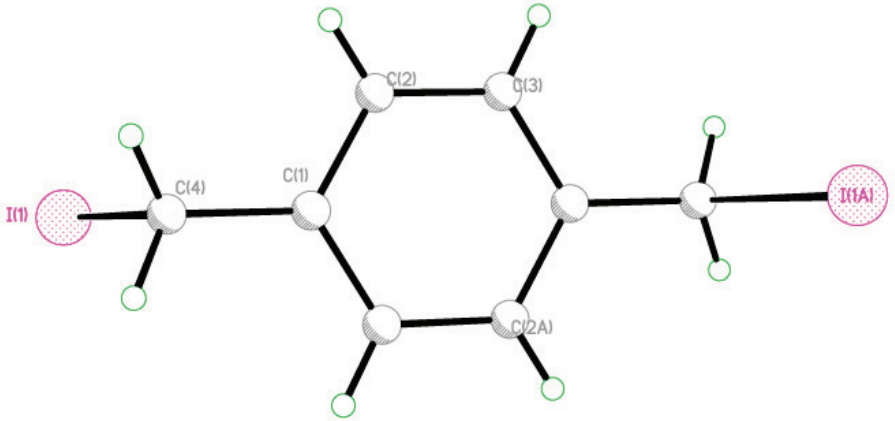
Title: 1,3-bis(iodomethyl) benzene a re-determination.

- Seemingly no very obvious problems here – the fact that the paper reports a re-determination and/or other unusual features e.g. powder or synchrotron data should be indicated in the Title
- Despite this however, first impressions can be deceptive!

Title: 1,3-bis(iodomethyl) benzene a re-determination.

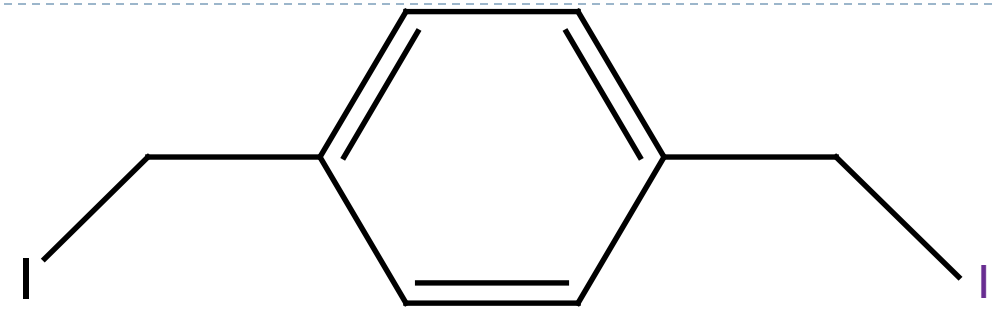
- However, always check the title against the Scheme and the molecular plot



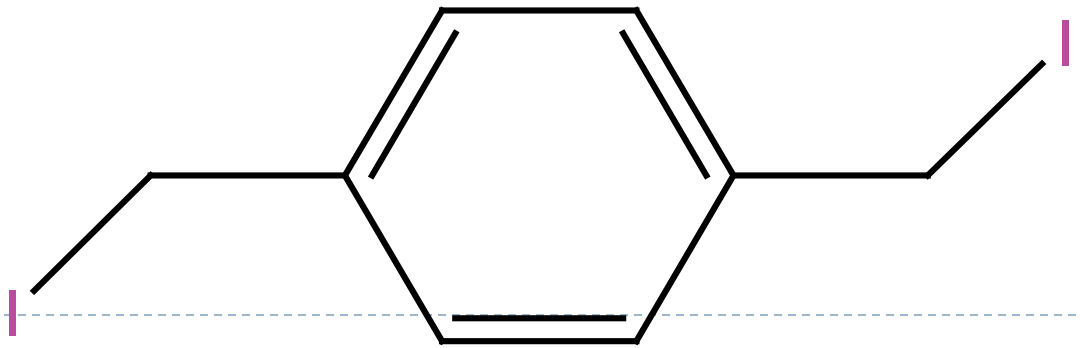


- Only the latter provides a correct picture of the structure this instance! This is clearly a 1,4 derivative not 1,3; also one I atom is missing from the Scheme

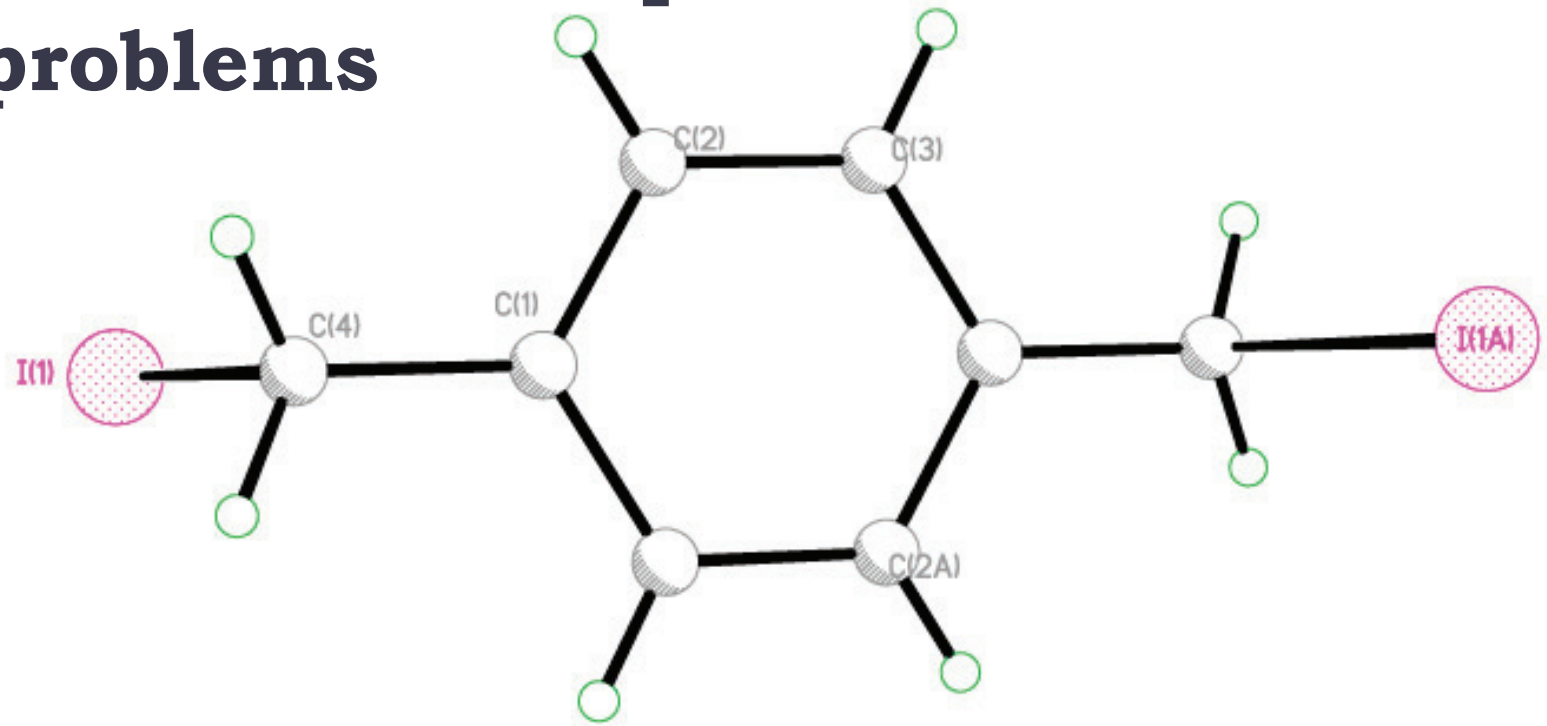
Title & Scheme



- **Corrected title is 1,4-bis(iodomethyl) benzene**
- **The compound lies about an inversion centre – this too should be indicated in the Scheme**

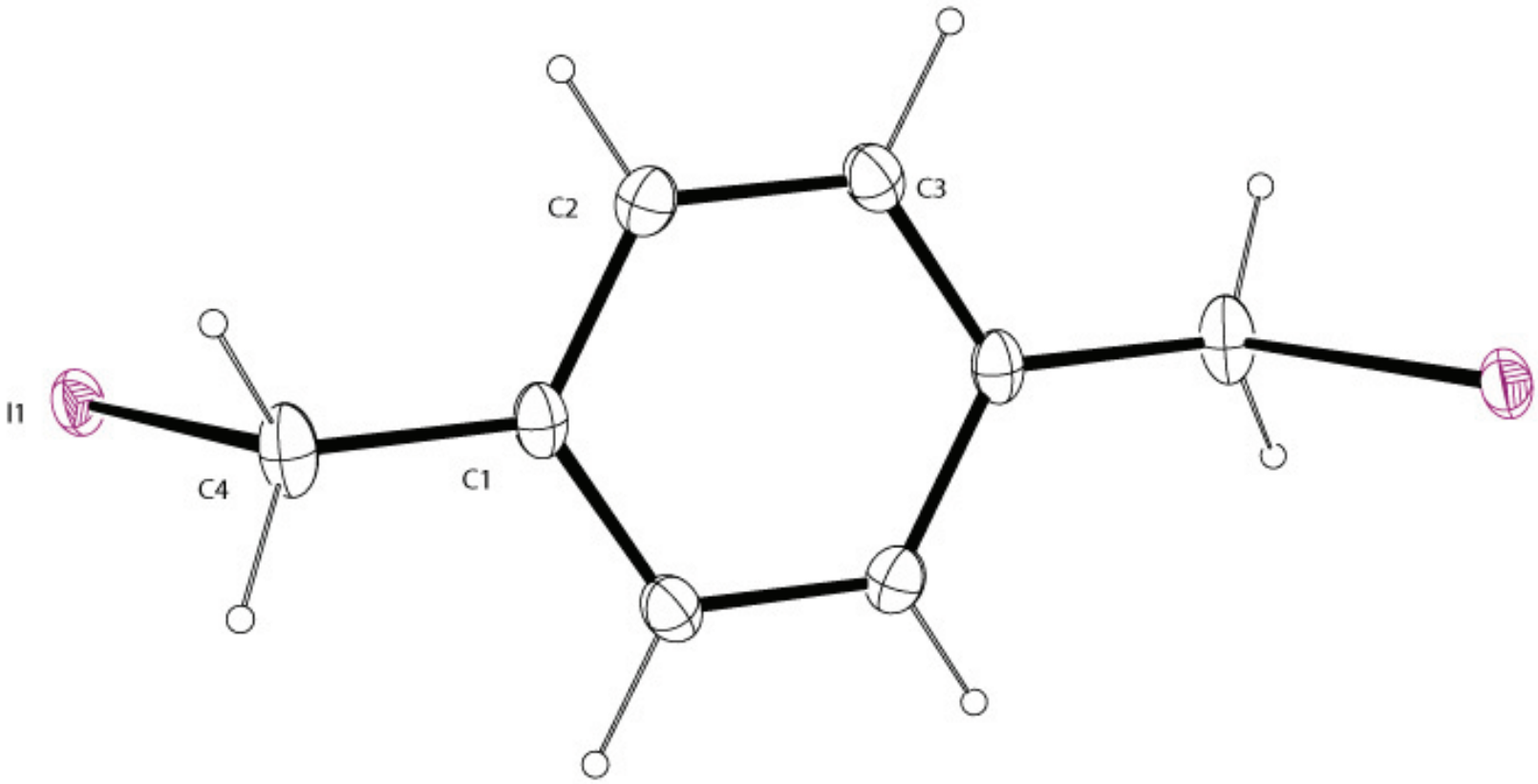


The molecular plot is not without problems



- Atoms should be shown as ellipsoids not spheres
- Atom numbers should not be in parenthesis
- Atom labels should not intersect with bonds or ellipsoids
- Display labels either for all or preferably none of the symmetry related atoms

Corrected molecular plot



Redeterminations

- Notes for authors are very specific about criteria for re-determinations.
- "If a structure has been redetermined correctly and the result adds significantly to the information already in the public domain then the article can be considered for publication. Redeterminations that report a small improvement in precision or are merely carried out at a different temperature to previous studies will not normally be considered for publication"

Is this a publishable redetermination?

- In this Abstract: 'but in that report the compound was recrystallised from acetone whereas in the present report, crystals were obtained from methanol.'
- **This does not qualify under any circumstances. Should be withdrawn.**

Redeterminations

- The most common classes of non-compliant redeterminations involve structures that :
  - Have slightly improved R factors
  - Have had data recorded at a lower temperature.
- Neither would qualify for acceptance
- If the original publication reported a structure refined from film data, no coordinates were given or H atoms were not assigned in the previous determination and there was no discussion of H-bonding, then publication can be considered.

Other Title Matters

- Check any implication of the absolute structure in the title. This should only be present if the absolute structure is known
- e.g if anomalous scattering is sufficient to permit the reliable determination of the absolute structure in which case a credible Flack parameter must be reported.
- or by comparison with a structure of known absolute configuration
- or from the synthesis

Abstract – missing items

- The four unique C atoms in a molecule lie in the plane.
- **If there are only 4 unique C atoms in this structure there must also be crystallographically imposed symmetry – an inversion centre in this case. This must be detailed in the Abstract.**
- In the crystal structure, weak C---H...I interactions link the molecules into stacks.
- **There is no Table 1 with details of these contacts. Also the direction in which the stacks form should also be mentioned.**

Abstract missing items

- A structure is further stabilised by short \pi...\pi contacts
- **Centroid ...centroid distance (su) Å? Also \pi...\pi = πi...πi**
- A structure is further stabilised by short I...I contacts, forming undulating sheets.
- **I...I distance (su) Å? Sheets in which plane?**

Abstract – English – the definite (the) and indefinite (a) articles

- but in that report a compound *(were)* ***was*** recrystallised from acetone whereas (here they were) **in the present report, crystals were** obtained from methanol.
- **The** structure has been reported previously
- but in that report (a) **the** compound was recrystallised from acetone
- The four unique C atoms in (a) **the** molecule lie in (the) **a** plane.
- (A) **The** structure is further stabilised by short I...I contacts, forming undulating sheets.

Related literature

- For related literature, **(on what subject)** see: Samakande et al., (2007); Asandei et al. (2008). **Specific details should always be given. Probably the most common problem with accepted papers.**
- For related **(crystal)** structures, **- delete crystal.**
- and for bond lengths **(and angles)**, see: Allen et al. (1987). This paper **only** discusses bond distances.
- For other structures of organic compounds published recently by the author, see: Osman et al. (2009), Hanton et al. (2010); Saeed et al., (2010a,b,c,d,e,f,g), Saeed et al.,(2009a,b,c,d); Li et al. (2009), Shafiq et al. (2009); Haider et al. (2010). **Self citation – not totally discouraged but this is over the top!!! Reduce to 3 or 4 references bearing some relationship to the material reported – preferably also in Acta Journals .**

Computer programs section

- Check that the programs cited fit with the diffractometer used.
- Claimed here to use a Bruker APEXII but with Rigaku software – surprising how often this problem occurs
- Check also that the software release date is reasonable – e.g. APEX 2 (Bruker, 1996) should be questioned!
- NB SHELXL97 is not a suitable entry for _computing_molecular_graphics
- Note also that there must be entries for ALL of the _Computing sections in the CIF

References

- Are all of the references cited? **PublCIF** is invaluable for checking this.

- **The following references are not cited in the preprint:**

Hunter, K. A. & Simpson, J. (1999). *TITAN2000*. University of Otago, New Zealand. – **just not there!**

Khalaji, A. D. & Simpson, J. (2009). *Acta Cryst.* **E**65 o553. - **cited ambiguously**

Li, J. S. & Simpson, J. (2009). *Acta Cryst.* **E**65, o2814. - **cited ambiguously**

Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan. – **incorrect format**

Spek, A. L. (2009). *Acta Cryst.* D**65**, 148--155. – **wrong date given**

Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920--925. – **wrong citation**

References

- _computing_data_collection 'Rigaku CrystalClear‘ – should be 'CrystalClear (Rigaku, 2005) '
- _computing_cell_refinement 'Rigaku CrystalClear' should be 'CrystalClear (Rigaku, 2005) '
- _computing_data_reduction 'Rigaku CrystalClear' should be 'CrystalClear (Rigaku, 2005) '
- _computing_publication_material

;

SHELXL-97, enCIFer (Allen et al., 2004), PLATON (Spek, 2003) & publCIF (Westrip, 2010)

;

Should be PLATON (Spek, 2009) – Westrip 2010 OK but reference to it isn’t!

References

- Warning: *et al.* used incorrectly: Jones *et al.*, 2007
- Jones, P. G. & Kus, P. (2007). Z. Naturforsch., B: Chem.Sci. 62, 725--731.
- Should normally be cited as Jones & Kus (2007) in Rel Lit or (Jones & Kus, 2007) in the Comment

- Warning: ambiguous? Khalaji *et al.* (2009
- Khalaji, A.D. and Simpson, J. (2009). Acta Cryst. E65 o553.
- Khalaji, A.D. and Simpson, J. (2009). Acta Cryst. E65 o362.
- Should be cited as Khalaji & Simpson (2009a, b) in Rel Lit or (Khalaji & Simpson, 2009a, b) in the Comment with an appropriate variation in the reference list

- Several corresponding warnings indicate similar problems.

References

- **The following references are not cited in the Acta E preprint (though cited elsewhere in the CIF):**

- Au, R. H.W., Fraser, C. S.A., Eisler, D. J., Jennings, M. C. & Puddephat, R. J. (2009). *Organometallics*, **28**, 1719--1729.
  Hochberg, G. C. & Schulz, R. C. (1993). *Polym. Int.* **32**, 309--317.
  Khalaji, A. D. & Simpson, J. (2009). *Acta Cryst.* **E**65 o362.
  Leir, C. M. & Stark, J. E. (1989). *J. Appl. Polym. Sci.* **38**, 1535--1547.
  Le Baccon, M., Chuburu, F., Toupet, L., Handel, H., Soibinet, M., Dechamps-Olivier, I., Barbier, J.-P. & Aplincourt, M. (2001). *New J. Chem.* **25**, 1168--1174.
  Sobransingh, D. & Kaifer, A. E. (2006). *Org. Lett.* **8**, 3247--3250.
  Song, Z., Weng, X., Weng, L., Huang, J., Wang, X., Bai, M., Zhou, Y., Yang, G. & Zhou, X. (2008). *Chem. Eur. J.* **14**, 5751--5754.
- These references appear only in the Comment but are not cited in the Related Literature or Computer Programs sections of the paper. It is now required that **ALL** references cited in the Comment are mentioned in the Related Literature section.

References

- 2 date(s) found in _computing_publication_material that could be part of a citation but not found in reference list
  Spek, A. L. (2009). Acta Cryst. D65, 148--155.
  Westrip, S. P. (2009). publCIF. In preparation.

But

_computing_publication_material

;

SHELXL-97, enCIFer (Allen et al., 2004), PLATON (Spek, 2003) & publCIF (Westrip, 2010)

;

- References should be alphabetical – currently arranged alphabetically but in order of date.

References

- And an odd – but by no means unprecedented problem to finish the references:
- John McAdam, C., Lyall, R.H., Stephen, C.M. and Jim, S.
- (2009). Acta Cryst. E65, o1573--o1574
- Given names and surnames Spoonerised! Occurs surprisingly often.

Comment

- Is nominally optional but we encourage authors to include one, particularly to provide a background to the investigation.
- Should be checked for glaring errors in English
- Normally 3 or 4 references are sufficient in the introductory section.
- Two page introductions should be actively discouraged.
- Word by word repetition of what appears in the Abstract, as found here, should be avoided
- The Comment does give authors the opportunity to be more specific by the use of atom designators not allowed in the Abstract.

Experimental

- check that starting materials would give the reported product – wrong isomer in this case!
- check that preparation is novel
- recrystallisation solvent should be stated
- if a solvent mixture, state ratios
- check consistency of crystal colour, shape and size (experimental, block; table, rectangular plate)
- consign large quantities of spectral data to _exptl_special_details

Refinement

- give number of Friedel pairs if Flack reported
- indicate if Friedel equivalents have been merged; but merging no longer necessary
- totally unnecessary to suggest that Friedel opposites were merged in this instance

Refinement

- check that the number of independent reflections and the number of reflections used in the refinement are the same – if not, as here, this should either be corrected or the difference explained
- H atom treatment must be mentioned – check that details correspond with the procedures actually used and the H atoms actually included in the structure. Here: All H-atoms were placed in calculated positions and refined using a riding model with U~eq~(H) = 1.2U~eq~(C,N).
- In fact – refall and no N atoms in the molecule.
- don't need to revisit software used or give details of heavy atom refinement here
- Details of the treatment of disorder, if present, should be mentioned.

Conclusion

- This paper represents what is hopefully a "worst case" scenario.
- However authors will never cease to surprise us!
- Up to us as co-editors to maintain the high quality of the Journal and thanks to your great efforts this is done most effectively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THANKS FOR
YOUR ATTENTION