

**Di-*p*-bromophenyl ether, a redetermined crystal structure derived from low quality diffraction data.**

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**SUPPLEMENTARY INFORMATION**

**Contents**

Appendix 1: Details of MOPAC calculation.

Appendix 2: Details of the scaling and merging process

References

Table S1.

Table S2.

Figure S1.

Figure S2.

## **Appendix 1: Details of MOPAC calculation.**

The map in Fig. S3 was calculated on a 10-degree grid with values for each torsion angle ranging from  $-180^\circ$  to  $180^\circ$ , a total of 37 steps in each direction. This sums up to a total of 1369 energy optimizations where only the two torsion angles are locked at each grid point. The molecular conformation at each grid point could thus be said to have been relaxed. The end values ( $-180^\circ$  and  $180^\circ$ ) designate the same conformation and were included only to check the periodicity of the calculations. As each of the rings have C2 symmetry along the O-C1...C4-Br direction the conformational map will have translation symmetry along both torsion angles with a translation period of  $180^\circ$ . The total computation time on a standard 1 GHz Pentium III processor was of the order of 10 hours.

## Appendix 2: Details of the scaling and merging process

Details of the merging and scaling procedure are given below.

All data sets were collected and integrated with the diffractometer software using the same unit cell setting. A check for having the correct absolute configuration was done on all individual datasets. None of the refinements on the individual datasets gave indications of having the opposite absolute configuration. One of the six component datasets gave a Flack parameter in the order of 0.5, however with a large s.u.

After that the complete structure model was derived from the individual data sets and checked that it fitted with acceptable residual R-factors we determined individual batch scale factor using the BASF and MERG 0 instructions in SHELXL97. One data set together with an additional BASF was added at a time. Approximately 30 least square cycles were necessary in order for the refinements to be completely convergent. When the refinements had converged for all added individual data sets, totally six individual data sets, the refined BASF were used to scale each of the individual intensity data sets. The scale factor applied to each, except the first data set, was computed as  $1.0/\text{BASF}$  for each of the individual data sets. The scaling was applied both to  $F^2$  and  $\text{sig}(F^2)$  using a locally written computer program REFLEX available from one of the authors. A final check that the computed scale factors were correct was done such as setting all BASF parameters in the instruction file for the SHELXL97 runs to unity and doing refinements with the scaled data set. All BASF parameters refined to unity within the estimated standard uncertainty derived by SHELXL97. In all these calculation no merging i.e. MERG 0 was used in SHELXL97. Thereafter the MERG 2 instruction was used in SHELXL97 to perform the merging of the whole data set producing the final dataset of 1166 unique reflections.

## References

Smith, G. S. & Snyder, R. L. (1979). *J. Appl. Cryst.* 12, 60-65.

deWolff, P.M. de (1968). *J. Appl. Cryst.* 1, 108-113.

**Table S1.**

Unitcell data for the six component data sets and their corresponding average values.

<b>crystal</b>	<b>a (Å)</b>	<b>b (Å)</b>	<b>c (Å)</b>	<b>volume (Å<sup>3</sup>)</b>
I	26.98(5)	7.692(3)	5.725(5)	1188(4)
II	26.74(8)	7.729(10)	5.753(8)	1189(7)
III	26.75(3)	7.759(6)	5.731(4)	1189(3)
IV	26.65(1)	7.577(2)	5.715(1)	1154(1)
V	26.14(6)	7.696(10)	5.807(15)	1168(7)
VI	26.76(3)	7.741(8)	5.699(5)	1180(4)
Average	26.67	7.699	5.738	1178

**Table S2**

Indexed powder diffraction data for (I). Data recorded with CuK $\alpha$ 1-radiation

( $\lambda \approx 1.5405981 \text{ \AA}$ )

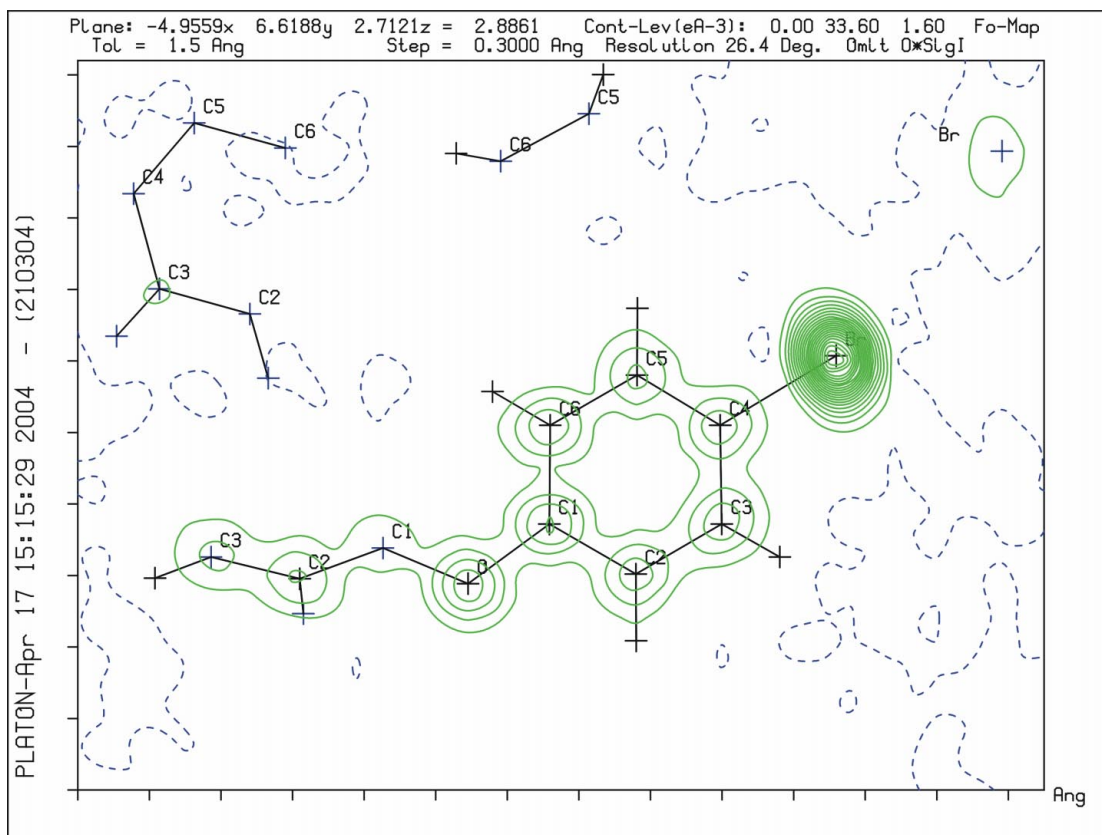
The refined cell parameters are: a=26.645(6)  $\text{\AA}$ , b=7.6902(14)  $\text{\AA}$ , c=5.7223(8)  $\text{\AA}$

M(19) = 48.2 (deWolff, 1968) and F(19) = 83.2 (0.006522, 35) (Smith & Snyder, 1979)

<b>N</b>	<b>H</b>	<b>K</b>	<b>L</b>	<b>2<math>\theta</math><sub>obs</sub></b>	<b>2<math>\theta</math><sub>calc</sub></b>	<b><math>\Delta</math>2<math>\theta</math></b>
1	2	0	0	6.6294	6.6294	0.0000
2	1	1	0	11.9728	11.9685	0.0044
3	1	1	1	19.6052	19.6086	-0.0034
4	3	1	1	21.7783	21.7771	0.0012
5	0	2	0	23.1220	23.1129	0.0092
6	2	2	0	24.0766	24.0701	0.0065
7	5	1	1	25.5964	25.5925	0.0038
8	8	0	0	26.7568	26.7450	0.0118
	4	2	0		26.7488	0.0080
10	2	2	1	28.7437	28.7422	0.0015
11	7	1	1	30.4893	30.4840	0.0053
12	0	0	2	31.2270	31.2423	-0.0153
13	2	0	2	31.9604	31.9733	-0.0129
14	1	1	2	33.5806	33.5667	0.0139
	10	0	0		33.6083	-0.0277
15	4	0	2	34.0872	34.0820	0.0051
16	3	1	2	34.9463	34.9282	0.0182
17	1	3	0	35.1385	35.1419	-0.0034
18	8	2	0	35.6228	35.6343	-0.0115
19	9	1	1	36.0745	36.0718	0.0027

**Figure S1**

Electron density map calculated from the combined data set, clearly showing the bromine and the six resolved carbon atoms of the benzene ring. Figure drawn with PLATON (Spek, 2003).



**Figure S2**

Heat of formation map for different values of the two torsion angles ( $\phi_1$ ,  $\phi_2$ ) describing the molecular conformation. The  $\Delta H_f^\circ$  was calculated at each grid point from  $-180^\circ$  to  $180^\circ$  in  $10^\circ$  steps. The contours are drawn at 1 kcal/mole. All calculations done with MOPAC6 (Stewart, 1990) and figure drawn with Easyplot (Karon, 1999). The conformation derived from the crystal structure ( $\phi_1 \approx 150^\circ$ ,  $\phi_2 \approx -34^\circ$ ), marked with a dot, resides in an energy minimum.

