SUPPLEMENTARY MATERIAL FOR

Two Phases of $C_9H_{12}O_4$: Why Is the Structure at 295 K So Complicated?

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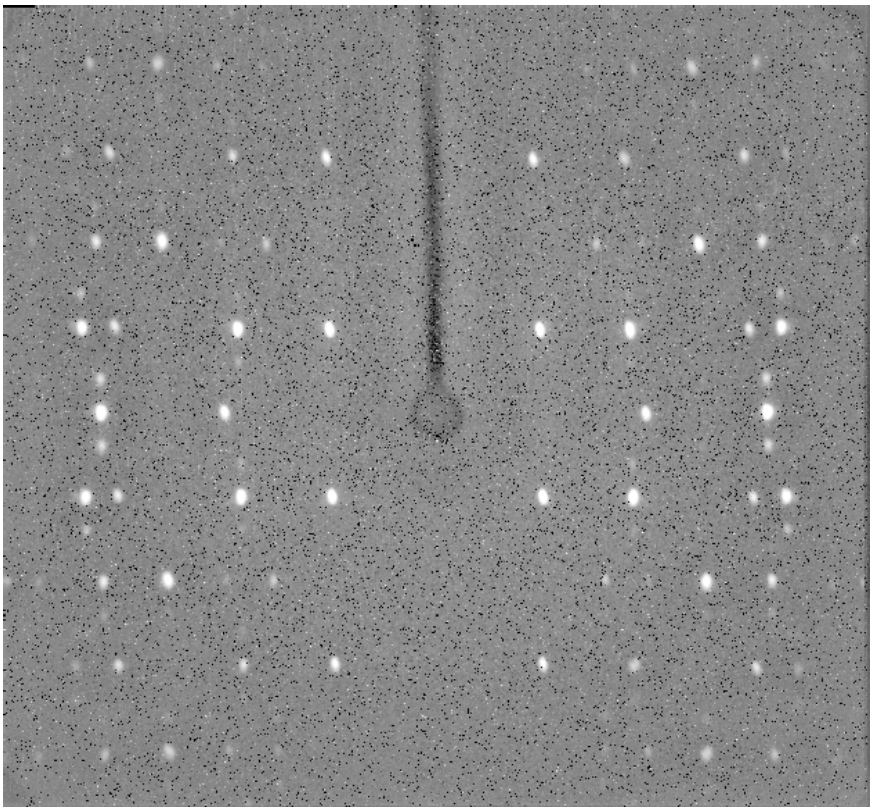
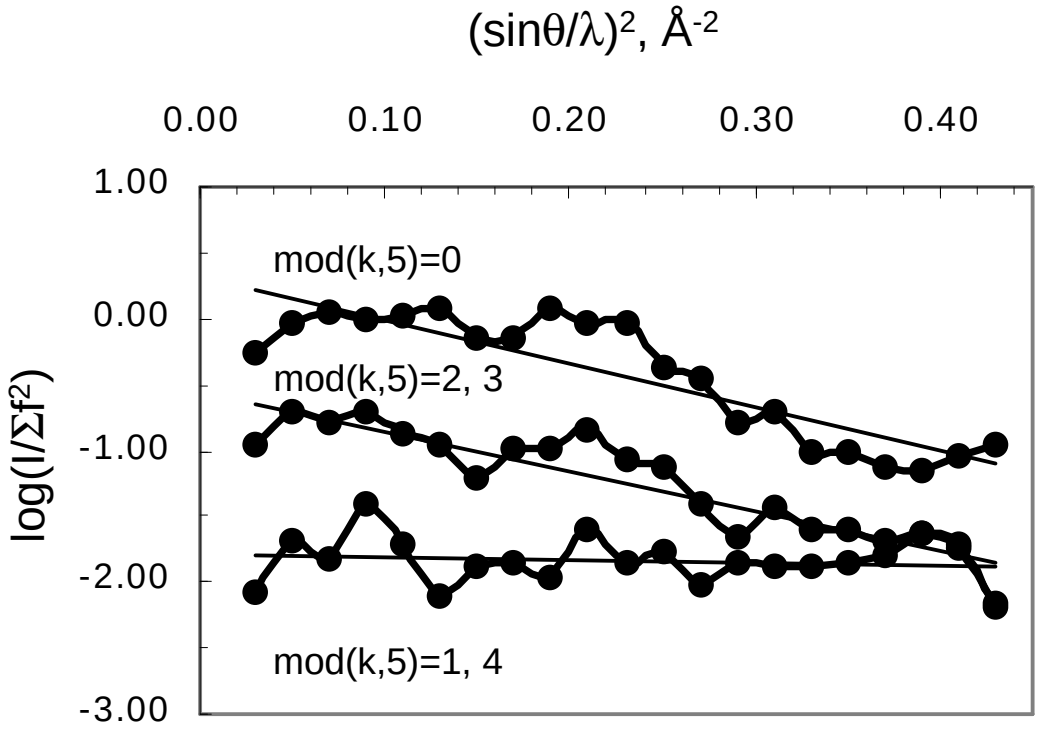
Synopsis: The structures of two modulated phases of a small organic molecule have been determined. The structure at 295 K has five mirror-symmetric molecules in the asymmetric unit; four of the molecules are disordered. At 130 K the unit cell and asymmetric unit are smaller and the structure is ordered.

Contents:

K98055 (C9O4/Stiles)
Rotation Photograph around **b**
(Dx = 120 mm; 170° oscillation;
180 s; 2 iterations)



k = -20
k = -15
k = -10
k = -5
k = 0
k = 5
k = 10
k = 15
k = 20



mod(k,5)=	Slope	Intercept
0	-3.4(4)	+0.35(11)
2, 3	-2.7(3)	-0.59(7)
1, 4	+0.1(3)	-1.84(8)

(First and last points omitted)

```
TITL ME2C7O4 at RT in Cmc21
CELL 0.71073  10.474  66.245   6.662  90.00  90.00  90.00
ZERR   20.00   0.005   0.035   0.003   0.00   0.00   0.00
LATT -7
SYMM -X, -Y, .5+Z
SYMM  X, -Y, .5+Z
SYMM -X,  Y,    Z
rem
EQIV  $1   -X,  Y,  Z
EQIV  $2  1-X,  Y,  Z
rem
SFAC C H O
UNIT 180 240 80
OMIT -2 55
MERG 3
L.S. 6
MORE  3
ACTA
BOND $H
FMAP 2
PLAN 20
rem
rem  Constraints and restraints:
rem
rem  For main molecules B, E, A, D:
rem   (atom names end in placeholder "x')
rem
rem  Take out the following, which may not be needed and may even
rem    be counterproductive.
rem  FLAT  0.1   O1Ax O1Ax_$2 O3Ax O3Ax_$2 C7Ax C1Ax C2Ax C2Ax_$2
rem  FLAT  0.1   O1Bx O1Bx_$1 O3Bx O3Bx_$1 C7Bx C1Bx C2Bx C2Bx_$1
rem  FLAT  0.1   O1Cx O1Cx_$1 O3Cx O3Cx_$1 C7Cx C1Cx C2Cx C2Cx_$1
rem  FLAT  0.1   O1Dx O1Dx_$2 O3Dx O3Dx_$2 C7Dx C1Dx C2Dx C2Dx_$2
rem  FLAT  0.1   O1Ex O1Ex_$2 O3Ex O3Ex_$2 C7Ex C1Ex C2Ex C2Ex_$2
rem
rem  For minor disordered components of molecules B, E, A, D:
rem   (atom names end in "'")
rem
rem  Occupancy of C4B', C4E' not large enough to allow independent
rem   refinement:
EXYZ  C4Bx  C4B'
EXYZ  C4Ex  C4E'
rem
rem  Hook together the two sections of the molecules
SADI  0.01  C2Cx C3Cx  C2Bx C3Bx  C2Ex C3Ex  C2Ax C3Ax  C2Dx C3Dx =
                       C2BX C3B'  C2EX C3E'  C2AX C3A'  C2DX C3D'
rem
rem  Restrain Uij values to rigid-body motion
rem   for the "front" parts of the molecules:
DELU  0.01  0.01   O1Cx > C2Cx
DELU  0.01  0.01   O1Bx > C2Bx
DELU  0.01  0.01   O1Ex > C2Ex
DELU  0.01  0.01   O1Ax > C2Ax
DELU  0.01  0.01   O1Dx > C2Dx
rem  and for the "back" ends of the molecules:
DELU  0.01  0.01   C3Cx > C9Cx
DELU  0.01  0.01   C3Bx > C9Bx
DELU  0.01  0.01   C3Ex > C9Ex
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```
DELU  0.01  0.01   C3Ax > C9Ax
DELU  0.01  0.01   C3Dx > C9Dx
DELU  0.01  0.01   C3A' > C9A'
DELU  0.01  0.01   C3D' > C9D'
rem
rem  Uij values for C3', C4' cannot be refined independently:
EADP  C3Bx  C3B'
EADP  C4Bx  C4B'
EADP  C3Ex  C3E'
EADP  C4Ex  C4E'
EADP  C3Ax  C3A'
EADP  C4Ax  C4A'
EADP  C3Dx  C3D'
EADP  C4Dx  C4D'
rem
rem  NB/  C8B', C9B', C8E', and C9E' are isotropic.
rem
rem  One Uiso value for all H1n atoms
EADP  H1Ax H1Bx H1Cx H1Dx H1Ex
rem
rem  (Save the idealized rigid-body fragment that was used for
rem   idealization)
rem  FRAG  17
rem  O1   3    1.09    2.10    0.00
rem  O2   3   -1.09    2.10    0.00
rem  O3   3    2.33   -0.06    0.00
rem  O4   3   -2.33   -0.06    0.00
rem  C1   1    0.00    0.00    0.00
rem  C2   1    1.21   -0.70    0.00
rem  C3   1    1.30   -2.20    0.00
rem  C4   1    0.00   -2.90    0.45
rem  C5   1   -1.30   -2.20    0.00
rem  C6   1   -1.21   -0.70    0.00
rem  C7   1    0.00    1.47    0.00
rem  C8   1    0.00   -2.85    1.92
rem  C9   1    0.00   -4.22   -0.19
rem  FEND
rem
rem
WGHT    0.060000
FVAR       1.37893   0.59077   0.53458   0.76465   0.86985
O1BX  3   -0.106081    0.114393    0.031690    11.00000    0.10789    0.06483 =
         0.09714   -0.00674   -0.00086    0.02395
O3BX  3   -0.223540    0.081786   -0.011233    11.00000    0.04747    0.08995 =
         0.12121   -0.00023   -0.00530    0.02003
AFIX   3
H1BX  2   -0.188365    0.099496    0.015450    11.00000    0.15539
AFIX   0
C7BX  1   10.000000    0.104927    0.020674    10.50000    0.08288    0.05858 =
         0.04815   -0.00099    0.00000    0.00000
C1BX  1   10.000000    0.083123   -0.012035    10.50000    0.05143    0.05246 =
         0.04761    0.00783    0.00000    0.00000
C2BX  1   -0.116058    0.072618   -0.024257    11.00000    0.04679    0.07116 =
         0.07705    0.00155   -0.00759    0.00467
PART  1
C3BX  1   -0.117995    0.050648   -0.081868    41.00000    0.04147    0.06359 =
         0.07557   -0.00892   -0.00645   -0.00586
AFIX   3
```

```
H3B1  2   -0.127203    0.049209   -0.224874    41.00000   -1.20000
H3B2  2   -0.191173    0.044479   -0.020054    41.00000   -1.20000
AFIX   0
C4BX  1   10.000000    0.039378   -0.011638    40.50000    0.04919    0.05143 =
         0.08234    0.00399    0.00000    0.00000
C8BX  1   10.000000    0.018097   -0.099356    40.50000    0.07013    0.06492 =
         0.11034   -0.02096    0.00000    0.00000
AFIX   3
H8B1  2    0.000000    0.019334   -0.243278    40.50000   -1.50000
H8B2  2    0.074910    0.010864   -0.057206    41.00000   -1.50000
AFIX   0
C9BX  1   10.000000    0.037714    0.220359    40.50000    0.08049    0.05702 =
         0.07239    0.00275    0.00000    0.00000
AFIX   3
H9B1  2    0.000000    0.051044    0.277406    40.50000   -1.50000
H9B2  2    0.074910    0.030564    0.263906    41.00000   -1.50000
AFIX   0
rem
PART  2
C3B'  1   -0.114958    0.051042    0.065158   -41.00000    0.04147    0.06359 =
         0.07557   -0.00892   -0.00645   -0.00586
AFIX   3
H3B3  2   -0.192482    0.044109    0.030346   -41.00000   -1.20000
H3B4  2   -0.110272    0.051869    0.209186   -41.00000   -1.20000
AFIX   0
C4B'  1   10.000000    0.039378   -0.011638   -40.50000    0.04919    0.05143 =
         0.08234    0.00399    0.00000    0.00000
C8B'  1   10.000000    0.017734    0.065829   -40.50000    0.08945
AFIX   3
H8B3  2    0.000000    0.016686    0.209902   -40.50000   -1.50000
H8B4  2    0.074910    0.011256    0.012892   -41.00000   -1.50000
AFIX   0
C9B'  1   10.000000    0.039456   -0.245905   -40.50000    0.05304
AFIX   3
H9B3  2    0.000000    0.053086   -0.295532   -40.50000   -1.50000
H9B4  2    0.074910    0.032566   -0.293312   -41.00000   -1.50000
AFIX   0
PART  0
rem
O1EX  3    0.605211    0.185560    0.522458    11.00000    0.10431    0.06650 =
         0.09673    0.00227    0.00496    0.01963
O3EX  3    0.722503    0.218369    0.552742    11.00000    0.05331    0.08828 =
         0.14063   -0.00098   -0.00064    0.01511
AFIX   3
H1EX  2    0.688487    0.201276    0.524598    11.00000    0.15539
AFIX   0
C7EX  1   10.500000    0.194920    0.533726    10.50000    0.08554    0.06175 =
         0.05910   -0.00048    0.00000    0.00000
C1EX  1   10.500000    0.216873    0.550715    10.50000    0.05260    0.05789 =
         0.07029   -0.00968    0.00000    0.00000
C2EX  1    0.616173    0.227302    0.568451    11.00000    0.04895    0.06513 =
         0.10658    0.00793    0.00143    0.01403
PART  1
C3EX  1    0.619262    0.249422    0.627798    51.00000    0.05720    0.06295 =
         0.11734   -0.00381   -0.01698   -0.00632
AFIX   3
H3E1  2    0.694038    0.255822    0.573515    51.00000   -1.20000
H3E2  2    0.624459    0.250143    0.771833    51.00000   -1.20000
```

```
AFIX   0
C4EX  1   10.500000    0.260638    0.560713    50.50000    0.05642    0.05932 =
         0.08622   -0.00763    0.00000    0.00000
C8EX  1   10.500000    0.282165    0.647611    50.50000    0.09482    0.05493 =
         0.17440   -0.00954    0.00000    0.00000
AFIX   3
H8E1  2    0.500000    0.281379    0.791840    50.50000   -1.50000
H8E2  2    0.574910    0.289259    0.603231    51.00000   -1.50000
AFIX   0
C9EX  1   10.500000    0.262218    0.326967    50.50000    0.11926    0.09141 =
         0.11603    0.04295    0.00000    0.00000
AFIX   3
H9E1  2    0.500000    0.248833    0.271231    50.50000   -1.50000
H9E2  2    0.574910    0.269323    0.282721    51.00000   -1.50000
AFIX   0
rem
PART  2
C3E'  1    0.616365    0.249056    0.487674   -51.00000    0.05720    0.06295 =
         0.11734   -0.00381   -0.01698   -0.00632
AFIX   3
H3E3  2    0.615213    0.248523    0.343339   -51.00000   -1.50000
H3E4  2    0.692653    0.255995    0.528469   -51.00000   -1.50000
AFIX   0
C4E'  1   10.500000    0.260638    0.560713   -50.50000    0.05642    0.05932 =
         0.08622   -0.00763    0.00000    0.00000
C8E'  1   10.500000    0.282332    0.484819   -50.50000    0.08452
AFIX   3
H8E3  2    0.500000    0.282691    0.340418   -50.50000   -1.50000
H8E4  2    0.574790    0.289061    0.534948   -51.00000   -1.50000
AFIX   0
C9E'  1   10.500000    0.260926    0.795319   -50.50000    0.07419
AFIX   3
H9E3  2    0.500000    0.247166    0.841051   -50.50000   -1.50000
H9E4  2    0.573110    0.268156    0.844751   -51.00000   -1.50000
AFIX   0
PART  0
rem
O1AX  3    0.394745    0.014732   -0.003048    11.00000    0.09624    0.06801 =
         0.10216   -0.00498   -0.00427    0.02138
O3AX  3    0.276856   -0.018208   -0.006429    11.00000    0.04157    0.09332 =
         0.13203   -0.00774   -0.00842    0.01790
AFIX   3
H1AX  2    0.314484   -0.001129    0.001371    11.00000    0.15539
AFIX   0
C7AX  1   10.500000    0.005070   -0.000736    10.50000    0.07473    0.06985 =
         0.04730   -0.00117    0.00000    0.00000
C1AX  1   10.500000   -0.016795   -0.010026    10.50000    0.04642    0.06180 =
         0.05882    0.00991    0.00000    0.00000
C2AX  1    0.384000   -0.027416   -0.006868    11.00000    0.04020    0.06754 =
         0.13479   -0.00149   -0.00675    0.00298
rem
PART  1
C3AX  1    0.380431   -0.049662    0.056523    21.00000    0.03958    0.07698 =
         0.09096    0.00318    0.01288   -0.00722
AFIX   3
H3A1  2    0.306369   -0.056223    0.001748    21.00000   -1.20000
H3A2  2    0.374389   -0.050053    0.200608    21.00000   -1.20000
AFIX   0
```

```
C4AX  1   10.500000   -0.060764   -0.009231    20.50000    0.04896    0.05801 =
         0.07782    0.00994    0.00000    0.00000
C8AX  1   10.500000   -0.082498    0.065241    20.50000    0.09821    0.06031 =
         0.13471    0.02172    0.00000    0.00000
AFIX   3
H8A1  2    0.500000   -0.082503    0.209616    20.50000   -1.50000
H8A2  2    0.573360   -0.089473    0.013266    21.00000   -1.50000
AFIX   0
C9AX  1   10.500000   -0.060996   -0.244141    20.50000    0.13113    0.06760 =
         0.08540   -0.02176    0.00000    0.00000
AFIX   3
H9A1  2    0.500000   -0.047525   -0.297894    20.50000   -1.50000
H9A2  2    0.574410   -0.068165   -0.289374    21.00000   -1.50000
AFIX   0
rem
PART  2
C3A'  1    0.382223   -0.049031   -0.089222   -21.00000    0.03958    0.07698 =
         0.09096    0.00318    0.01288   -0.00722
AFIX   3
H3A3  2    0.379266   -0.048749   -0.233563   -21.00000   -1.20000
H3A4  2    0.307127   -0.055849   -0.041434   -21.00000   -1.20000
AFIX   0
C4A'  1   10.500000   -0.060287   -0.017612   -20.50000    0.04896    0.05801 =
         0.07782    0.00994    0.00000    0.00000
C8A'  1   10.500000   -0.081525   -0.105755   -20.50000    0.06330    0.08798 =
         0.13114   -0.03424    0.00000    0.00000
AFIX   3
H8A4  2    0.500000   -0.079563   -0.248852   -20.50000   -1.50000
H8A5  2    0.575750   -0.088853   -0.069333   -21.00000   -1.50000
AFIX   0
C9A'  1   10.500000   -0.061643    0.215169   -20.50000    0.05930    0.05454 =
         0.08816   -0.00392    0.00000    0.00000
AFIX   3
H9A3  2    0.500000   -0.048186    0.269242   -20.50000   -1.50000
H9A4  2    0.575070   -0.068646    0.260312   -21.00000   -1.50000
AFIX   0
PART  0
rem
rem
O1DX  3    0.393691    0.214459    0.049659    11.00000    0.10966    0.06570 =
         0.10792    0.00687    0.00835    0.02227
O3DX  3    0.277336    0.181903    0.026725    11.00000    0.05155    0.08935 =
         0.14267    0.00597   -0.00119    0.01911
AFIX   3
H1DX  2    0.312137    0.198968    0.032865    11.00000    0.15539
AFIX   0
C7DX  1   10.500000    0.205246    0.047058    10.50000    0.08525    0.06258 =
         0.05215   -0.00273    0.00000    0.00000
C1DX  1   10.500000    0.183302    0.027887    10.50000    0.05563    0.05589 =
         0.06386    0.00810    0.00000    0.00000
C2DX  1    0.383957    0.172769    0.025130    11.00000    0.05113    0.06958 =
         0.10771    0.00202   -0.00633    0.01074
rem
PART  1
C3DX  1    0.383200    0.150960   -0.048509    31.00000    0.04447    0.07435 =
         0.08442    0.00541    0.00945   -0.00735
AFIX   3
H3D1  2    0.385918    0.150647   -0.192857    31.00000   -1.20000
```

```
H3D2  2    0.306287    0.144327   -0.005587    31.00000   -1.20000
AFIX   0
C4DX  1   10.500000    0.139373    0.026647    30.50000    0.04454    0.06323 =
         0.07800    0.00857    0.00000    0.00000
C8DX  1   10.500000    0.118481   -0.067076    30.50000    0.07801    0.06483 =
         0.10361   -0.00243    0.00000    0.00000
AFIX   3
H8D1  2    0.500000    0.119548   -0.210846    30.50000   -1.50000
H8D2  2    0.571850    0.110989   -0.017056    31.00000   -1.50000
AFIX   0
C9DX  1   10.500000    0.138311    0.258359    30.50000    0.06388    0.04239 =
         0.09386   -0.00004    0.00000    0.00000
AFIX   3
H9D1  2    0.500000    0.151716    0.313696    30.50000   -1.50000
H9D2  2    0.574890    0.131216    0.302807    31.00000   -1.50000
AFIX   0
rem
PART  2
C3D'  1    0.381905    0.150679    0.102453   -31.00000    0.04447    0.07435 =
         0.08442    0.00541    0.00945   -0.00735
AFIX   3
H3D3  2    0.307845    0.144149    0.047275   -31.00000   -1.20000
H3D4  2    0.375025    0.150349    0.246455   -31.00000   -1.20000
AFIX   0
C4D'  1   10.500000    0.139305    0.030436   -30.50000    0.04454    0.06323 =
         0.07800    0.00857    0.00000    0.00000
C8D'  1   10.500000    0.117648    0.104755   -30.50000    0.09433    0.05324 =
         0.13636    0.00095    0.00000    0.00000
AFIX   3
H8D3  2    0.500000    0.117471    0.249068   -30.50000   -1.50000
H8D4  2    0.572940    0.110701    0.050988   -31.00000   -1.50000
AFIX   0
C9D'  1   10.500000    0.138872   -0.205390   -30.50000    0.11603    0.07846 =
         0.06924   -0.03230    0.00000    0.00000
AFIX   3
H9D3  2    0.500000    0.152571   -0.253049   -30.50000   -1.50000
H9D4  2    0.575670    0.132261   -0.253779   -31.00000   -1.50000
AFIX   0
rem
SAME  0.01  0.02  O1Bx > C9Bx
SAME  0.01  0.02  O1Ex > C9Ex
SAME  0.01  0.02  O1Ax > C9Ax
SAME  0.01  0.02  O1Dx > C9Dx
O1CX  3    0.104845    0.085250    0.489340    11.00000    0.10227    0.06785 =
         0.07912    0.00298    0.00217    0.02115
O3CX  3    0.223103    0.117973    0.532334    11.00000    0.04687    0.09268 =
         0.13706    0.00726    0.00757    0.01619
AFIX   3
H1CX  2    0.192835    0.101171    0.504908    11.00000    0.15539
AFIX   0
C7CX  1   10.000000    0.094614    0.504728    10.50000    0.08183    0.06385 =
         0.05068    0.02046    0.00000    0.00000
C1CX  1   10.000000    0.116539    0.529808    10.50000    0.05228    0.06174 =
         0.05855    0.00304    0.00000    0.00000
C2CX  1    0.116550    0.126888    0.550119    11.00000    0.05170    0.07102 =
         0.09389    0.01158    0.00312    0.00813
SAME  0.01  0.02  C3B' > C9B'
SAME  0.01  0.02  C3E' > C9E'
```

```
SAME  0.01  0.02  C3A' > C9A'
SAME  0.01  0.02  C3D' > C9D'
C3CX  1    0.117132    0.149049    0.597853    11.00000    0.05257    0.07145 =
         0.13418   -0.00311   -0.01674   -0.00813
AFIX   3
H3C1  2    0.125625    0.150348    0.741100    11.00000   -1.20000
H3C2  2    0.191254    0.155098    0.537638    11.00000   -1.20000
AFIX   0
C4CX  1   10.000000    0.160411    0.532407    10.50000    0.05036    0.04881 =
         0.09875   -0.00221    0.00000    0.00000
C8CX  1   10.000000    0.181854    0.616996    10.50000    0.10925    0.05686 =
         0.20882   -0.01758    0.00000    0.00000
AFIX   3
H8C1  2    0.000000    0.180675    0.760965    10.50000   -1.50000
H8C2  2    0.074910    0.189065    0.574565    11.00000   -1.50000
AFIX   0
C9CX  1   10.000000    0.161990    0.298199    10.50000    0.11574    0.09949 =
         0.11497    0.03878    0.00000    0.00000
AFIX   3
H9C1  2    0.000000    0.148652    0.241282    10.50000   -1.50000
H9C2  2    0.074910    0.169132    0.254593    11.00000   -1.50000
PART  0
rem
HKLF  4

REM  ME2C7O4 at RT in Cmc21
REM R1 =  0.0697 for   1388 Fo > 4sig(Fo)  and  0.1449 for all   2987 data
REM    388 parameters refined using    430 restraints

END

WGHT      0.1057      1.2603
REM Highest difference peak  0.168,  deepest hole -0.152,  1-sigma level  0.027
Q1    1   0.5000 -0.0117 -0.0330  10.50000  0.05    0.17
Q2    1   0.5000  0.2126  0.5731  10.50000  0.05    0.16
Q3    1   0.5000  0.2610  0.5041  10.50000  0.05    0.14
Q4    1   0.0484  0.0794 -0.0227  11.00000  0.05    0.12
Q5    1   0.4517  0.2210  0.5777  11.00000  0.05    0.11
Q6    1   0.0000  0.0182  0.1563  10.50000  0.05    0.11
Q7    1   0.0000  0.1009  0.4800  10.50000  0.05    0.11
Q8    1   0.0511  0.1205  0.5564  11.00000  0.05    0.11
Q9    1   0.0000  0.0667  0.1616  10.50000  0.05    0.10
Q10   1   0.5000  0.2027  0.0065  10.50000  0.05    0.10
Q11   1   0.4540  0.1794 -0.0054  11.00000  0.05    0.10
Q12   1   0.7177  0.2168  0.5059  11.00000  0.05    0.10
Q13   1   0.5000 -0.0810  0.1311  10.50000  0.05    0.10
Q14   1   0.5561 -0.0224 -0.0583  11.00000  0.05    0.10
Q15   1   0.5000  0.2180 -0.1289  10.50000  0.05    0.09
Q16   1  -0.1915  0.0481 -0.1181  11.00000  0.05    0.09
Q17   1   0.1143  0.0226  0.1985  11.00000  0.05    0.09
Q18   1   0.5000  0.2809  0.3972  10.50000  0.05    0.09
Q19   1   0.2261  0.1491  0.4154  11.00000  0.05    0.09
Q20   1   0.5000 -0.0956  0.0080  10.50000  0.05    0.08
```

```
TITL ME2C7O4 at 130 K (n=2) in Pmnb cell
CELL 0.71073  10.471  26.240   6.434  90.00  90.00  90.00
ZERR    8.00   0.003   0.009   0.002   0.00   0.00   0.00
LATT 1
SYMM 0.5-X, 0.5+Y, 0.5-Z
SYMM     X, 0.5+Y, 0.5-Z
SYMM 0.5-X,     Y,     Z
rem
rem
EQIV  $1   0.5-X,    Y,  Z
EQIV  $2   1. -X, 1.+Y,  Z
rem
SFAC C H O
UNIT  72  96 32
OMIT -2 55
MERG 3
L.S. 12
rem
MORE  3
ACTA
MPLA  O1A O1A_$1 O1A_$2
MPLA  8  O1A O1A_$1 O3A O3A_$1 C7A C1A C2A C2A_$1 C3A C4A C8A C9A
MPLA  3  C3A C4A C3A_$1

MPLA  O1A O1A_$1 O1A_$2
MPLA  8  O1B O1B_$1 O3B O3B_$1 C7B C1B C2B C2B_$1 C3B C4B C8B C9B
MPLA  3  C3B C4B C3B_$1
BOND $H
FMAP 2
PLAN 20
rem   ADP restraints:
EADP  C7A  C7B
EADP  O1A  O1B
EADP  O3A  O3B
EADP  C1A  C1B
EADP  C2A  C2B
EADP  C3A  C3B
EADP  C4A  C4B
EADP  C8A  C8B
EADP  C9A  C9B
rem
rem  Tie the two mirror-related halves of the molecule together:
DFIX  2.20   0.0001  O1A  O1A_$1  O1B  O1B_$1
DFIX  4.67   0.0001  O3A  O3A_$1  O3B  O3B_$1
DFIX  2.44   0.0001  C2A  C2A_$1  C2B  C2B_$1
DFIX  2.45   0.0001  C3A  C3A_$1  C3B  C3B_$1
rem
rem  Molecule 1
WGHT    0.060000
FVAR       0.06083
AFIX   6
C7A   1    0.250000    0.389060    0.254180    10.50000    0.01363
O1A   3    0.145030    0.412508    0.234630    11.00000    0.02578
O3A   3    0.026950    0.330056    0.285167    11.00000    0.02244
H1A   2    0.057148    0.372333    0.253264    11.00000   -1.20000
C1A   1    0.250000    0.333867    0.285263    10.50000    0.01687
C2A   1    0.133552    0.307757    0.307116    11.00000    0.02549
C3A   1    0.133009    0.252098    0.361697    11.00000    0.02053
```

```
H3AA  2    0.123763    0.249637    0.510166    11.00000   -1.20000
H3AB  2    0.059272    0.236415    0.299772    11.00000   -1.20000
C4A   1    0.249997    0.223153    0.298206    10.50000    0.01203
C8A   1    0.249999    0.169520    0.390779    10.50000    0.02353
H8AA  2    0.250004    0.173324    0.539531    10.50000   -1.50000
H8AB  2    0.175900    0.151005    0.347522    11.00000   -1.50000
C9A   1    0.249989    0.217818    0.056138    10.50000    0.02558
H9AA  2    0.249988    0.251154   -0.005895    10.50000   -1.50000
H9AB  2    0.177354    0.199471    0.011649    11.00000   -1.50000
rem  Molecule 2
AFIX   6
C7B   1    0.250000    0.361053    0.768742    10.50000    0.01363
O1B   3    0.145080    0.337488    0.785893    11.00000    0.02578
O3B   3    0.026947    0.420123    0.740992    11.00000    0.02244
H1B   2    0.057159    0.377717    0.769830    11.00000   -1.20000
C1B   1    0.250000    0.416378    0.741886    10.50000    0.01687
C2B   1    0.133553    0.442553    0.721364    11.00000    0.02549
C3B   1    0.133018    0.498451    0.671045    11.00000    0.02053
H3BA  2    0.124088    0.501593    0.522740    11.00000   -1.20000
H3BB  2    0.059119    0.513824    0.733740    11.00000   -1.20000
C4B   1    0.249999    0.527134    0.737415    10.50000    0.01203
C8B   1    0.250000    0.581184    0.648959    10.50000    0.02353
H8BA  2    0.250002    0.578063    0.499943    10.50000   -1.50000
H8BB  2    0.175682    0.599473    0.693198    11.00000   -1.50000
C9B   1    0.249996    0.531354    0.979848    10.50000    0.02558
H9BA  2    0.249995    0.497739    1.039322    10.50000   -1.50000
H9BB  2    0.176503    0.549471    1.025310    11.00000   -1.50000
HKLF  4

REM  ME2C7O4 at 130 K in Pmnb cell
REM R1 =  0.0807 for    250 Fo > 4sig(Fo)  and  0.1371 for all    465 data
REM     20 parameters refined using      8 restraints

END

WGHT      0.1068      4.6007
REM Highest difference peak  0.421,  deepest hole -0.348,  1-sigma level  0.075
Q1    1   0.0193  0.4174  0.6148  11.00000  0.05    0.42
Q2    1   0.2500  0.5274  0.8223  10.50000  0.05    0.34
Q3    1   0.0268  0.3322  0.4269  11.00000  0.05    0.30
Q4    1   0.2500  0.4162  0.6395  10.50000  0.05    0.27
Q5    1   0.0269  0.3352  0.1224  11.00000  0.05    0.24
Q6    1   0.2500  0.2152  0.1881  10.50000  0.05    0.24
Q7    1   0.1066  0.4110  0.1491  11.00000  0.05    0.24
Q8    1   0.1126  0.3420  0.8910  11.00000  0.05    0.21
Q9    1   0.2500  0.3330  0.3728  10.50000  0.05    0.19
Q10   1   0.0383  0.4232  0.9387  11.00000  0.05    0.16
Q11   1   0.1253  0.5910  0.5141  11.00000  0.05    0.15
Q12   1   0.2500  0.5899  0.8288  10.50000  0.05    0.14
Q13   1   0.1168  0.4090  0.3960  11.00000  0.05    0.14
Q14   1   0.1426  0.2787  0.4660  11.00000  0.05    0.14
Q15   1   0.1772  0.5178  0.3435  11.00000  0.05    0.14
Q16   1   0.2500  0.1898 -0.2123  10.50000  0.05    0.14
Q17   1   0.0788  0.5919  0.8293  11.00000  0.05    0.13
Q18   1   0.1071  0.3432  0.6377  11.00000  0.05    0.13
Q19   1   0.0828  0.2617  0.1928  11.00000  0.05    0.13
Q20   1   0.1830  0.3214  0.1233  11.00000  0.05    0.13
```