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Double Conjugate Addition of Dithiols to Propargylic Carbonyl Systems To Generate Protected 1,3-Dicarbonyl Compounds

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'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
'H' 'H' 0.0000 0.0000
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'x, -y-1/2, z-1/2'

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multi-scan from symmetry-related measurements
Sortav (Blessing 1995)
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Poor crystals - diffraction pattern contained spots from more
than one crystal. Some spots overlapped ... (JED).
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HKL Denzo and Scalepack (Otwinowski & Minor 1997)
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Refinement of F2 against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F2, conventional R-factors R are based
on F, with F set to zero for negative F2. The threshold expression of
F2 > 2sigma(F2) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F2 are statistically about twice as large as those based on F, and R-
factors based on ALL data will be even larger.
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O1 O 0.1091(3) 0.0958(3) -0.0682(2) 0.0497(8) Uani 1 1 d . . .
O2 O 0.1587(4) -0.1120(3) 0.0088(3) 0.0613(10) Uani 1 1 d . . .
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C1 C 0.2206(6) 0.0160(5) 0.0013(3) 0.0522(12) Uani 1 1 d . . .
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C2 C 0.2666(6) 0.0908(5) 0.0956(3) 0.0504(11) Uani 1 1 d . . .
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H5B H 0.3492 0.4513 -0.0390 0.059 Uiso 1 1 calc R . .
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H6B H 0.1682 0.6176 -0.1129 0.079 Uiso 1 1 calc R . .
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H7B H 0.0066 0.4858 -0.2358 0.077 Uiso 1 1 calc R . .
C8 C 0.0452(6) 0.2963(5) -0.1662(4) 0.0569(13) Uani 1 1 d . . .
H8A H -0.0457 0.3014 -0.1460 0.068 Uiso 1 1 calc R . .
H8B H 0.0232 0.2403 -0.2262 0.068 Uiso 1 1 calc R . .
C9 C 0.1668(5) 0.2275(5) -0.0868(3) 0.0480(11) Uani 1 1 d . . .
H9 H 0.2545 0.2121 -0.1104 0.058 Uiso 1 1 calc R . .
C10 C 0.6282(5) 0.1544(5) 0.1690(3) 0.0494(11) Uani 1 1 d . . .
H10A H 0.5948 0.0588 0.1736 0.059 Uiso 1 1 calc R . .
H10B H 0.7291 0.1499 0.1625 0.059 Uiso 1 1 calc R . .
C11 C 0.6348(5) 0.2300(5) 0.2630(3) 0.0492(11) Uani 1 1 d . . .

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S2 0.0584(7) 0.0454(7) 0.0429(7) -0.0020(5) 0.0148(5) -0.0003(5)
O1 0.0575(19) 0.0325(15) 0.0478(17) -0.0002(13) -0.0016(14) -0.0056(14)
O2 0.070(2) 0.0307(17) 0.075(2) 0.0028(16) 0.0085(18) -0.0069(15)
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C2 0.067(3) 0.037(2) 0.043(2) 0.0043(19) 0.011(2) -0.003(2)
C3 0.058(3) 0.032(2) 0.033(2) -0.0034(17) 0.0050(19) -0.0017(19)
C4 0.054(3) 0.038(2) 0.041(2) 0.0008(19) 0.008(2) 0.001(2)
C5 0.065(3) 0.030(2) 0.044(2) 0.0022(18) 0.004(2) 0.000(2)
C6 0.085(4) 0.045(3) 0.063(3) 0.016(2) 0.016(3) 0.009(3)
C7 0.078(4) 0.050(3) 0.052(3) 0.014(2) 0.001(3) 0.007(3)
C8 0.060(3) 0.048(3) 0.048(3) 0.004(2) -0.007(2) 0.000(2)
C9 0.057(3) 0.039(2) 0.042(2) 0.0022(19) 0.006(2) 0.000(2)
C10 0.056(3) 0.045(3) 0.045(2) 0.005(2) 0.013(2) 0.007(2)
C11 0.050(3) 0.056(3) 0.036(2) 0.002(2) 0.0050(19) -0.004(2)
C12 0.055(3) 0.057(3) 0.032(2) 0.001(2) 0.0083(19) -0.005(2)

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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.

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O1 C9 1.449(5) . ?
O2 C1 1.394(6) . ?
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C1 C2 1.489(6) . ?
C1 H1 1.0000 . ?
C2 C3 1.537(6) . ?
C2 H2B 0.9900 . ?
C2 H2C 0.9900 . ?
C3 C4 1.564(6) . ?
C4 C9 1.507(6) . ?
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C4 H4 1.0000 . ?
C5 C6 1.534(7) . ?
C5 H5A 0.9900 . ?

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C5 H5B 0.9900 . ?
 C6 C7 1.486(8) . ?
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 C6 H6B 0.9900 . ?
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 C7 H7A 0.9900 . ?
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 O2 C1 O1 107.1(4) . . ?
 O2 C1 C2 112.3(4) . . ?
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 C2 C3 C4 107.5(4) . . ?
 C2 C3 S2 109.1(3) . . ?
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 C2 C3 S1 114.2(3) . . ?
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 C7 C6 H6B 109.3 . . ?

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 C6 C7 H7B 109.4 . . ?
 C8 C7 H7B 109.3 . . ?
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 O1 C9 C8 106.6(4) . . ?
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 C6 C7 C8 C9 -55.1(6) ?
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 C1 O1 C9 C8 175.3(4) ?
 C5 C4 C9 O1 -174.6(4) ?
 C3 C4 C9 O1 57.3(5) ?
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