# MS51. Methods and results in chemical crystallography

## MS51-P1 Crystal structure and synthesis of 2,2-dimethyl-5-(prop-2-yn-1-yloxy) -4H-benzo[d][1,3]dioxin-4-one

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The title compound, 2,2-dimethyl-5-(prop-2-yn-1-yloxy) -4H-benzo[d][1,3]dioxin-4-one, ($C_{13}H_{12}O_4$), has been synthesized and characterized by spectroscopic and single crystal X-ray diffraction techniques. The compound (I) is in triclinic form and the unit-cell dimensions for (I) at 296(2)K are *a*=6.7498(1)Å, *b*=7.8207(2)Å, *c*=11.6311(2)Å, α=87.1780(10)°, β=83.1690(10)°, γ=68.2250(10)°, *V*=566.119(19)Å$^3$, $D_x$=1.362 g/cm$^3$, and *Z*=2. The *R* and GOF values for the compound are 0.0388 and 1.078, respectively, for the number of 2311 reflections. The whole molecule is not planar and all the bond lengths and angles are within normal ranges. In the crystal there is a number of strong C-H…O inter-molecular hydrogen bonds present. In addition, three C-H…*Cg* (min H…*Cg*=3.031Å and C-H…*Cg*=141.00°) and an aromatic *Cg*2…*Cg*2 [1-*x*, 1-*y*, 1-*z*; *Cg*2 is the centroid of the (C4-C9) ring] stacking interactions [centroid–centroid distance =5.8212(7)Å ] involving neighboring molecules are also observed.

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**Keywords:** X-ray Crystallography, NMR, IR.

## MS51-P2 The synthesis, characterization and crystal structure of 4-((e)-4-bromophenyl)diazenyl) -2-((e)-(phenylimino)methyl)phenol

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Apart from their purely chemical interest, azo-azomethine dyes are being increasingly used in the textile, leather and plastic industries [1]. Regarding the industrial importance of metallized azo dyes relative to their structures, they can be classified into two main types: those in which the azo group participates in the coordination to the metal ion with formation of the chelate ring and those in which it is not [2].

In this study, the synthesis and characterization of 4-((E)-4-bromophenyl)diazenyl) -2-((E)-(phenylimino)methyl)phenol which sythesiszed from the reaction of (E)-1-(5-((4-bromophenyl)diazenyl) -2-hydroxyphenyl)ethanone with aniline is reported. The structure of the compound, [$C_{19}H_{14}BrN_30$], is characterized by single-crystal X-ray diffraction analysis. Its crystallizes in a monoclinic system C1/c space group, with a= 37.4840 , b= 7.0935 , c= 6.2089 , b= 97.497°, V= 1636.79 $^3$, Z= 4, m(Mo$K_a$) = 2.522 mm$^{-1}$. The structure was solved by direct methods SHELXS-97 [3], and refined by full matrix least squares techniques on $F^2$ using SHELXL-97[3] with refinement of $F^2$ against all reflections.There is an intermoleculer O-H…N hydrogen bond and molecules are linked by C-H…pi and hydrogen bonds and weak pi-pi stacking interactions in the crystal structure. All Bond lenghts, bond angles, torsion angles and dihedral angles are determined. ORTEP III [4] drawing of completed molecule and atom labelling-scheme are drawn.

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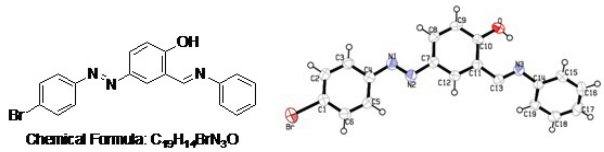


**Figure 1.** Crystal form of 4-((E)-4-bromophenyl)diazenyl) -2-((E)-(phenylimino)methyl)phenol

**Keywords:** Azogroup, schiffbase, X-Ray study