

An Executive Summary of the final report of the work done on the Minor Research Project of **Dr Prakash Kamath K** entitled **“Synthesis and crystal Structure determination of Chalcone Derivatives”** Sanctioned by UGC, Vide sanction letter no. **MRP(S)-0141/12-13/KAMA002/UGC-SWRO** Dated: **23rd Sept 2013.**

Synthesis of two chalcone compounds: Chemically chalcones consist of open-chain flavonoids in which the two aromatic rings are joined by a three-carbon, α -unsaturated carbonyl system. A vast number of naturally occurring chalcones are polyhydroxylated in the aryl rings. The radical quenching properties of the phenolic groups present in many chalcones have raised interest in using the compounds for many useful applications. Hence in continuation with the synthesis and crystal structure determination and also owing to the importance of chalcones, a bromochalcone is synthesized and its crystal structure is studied.

The bromochalcone, (2E)-1-(4-Bromophenyl)-3-(2-methoxyphenyl)prop-2-en-1-one is a chalcone derivative with 4-bromophenyl and 2-methoxy rings bonded at the opposite ends of a propenone group. It is synthesized as follows:

To a mixture of 4-bromoacetophenone (0.01 mol, 1.99 g) and 2-methoxybenzaldehyde (0.01 mol, 1.36 g) in 30 ml of ethanol, 7 ml of 30% KOH solution was added. The mixture was stirred for 6 hours at room temperature and the precipitate was collected by filtration and purified by recrystallization from ethanol. Single crystals were grown from a acetone-toluene (1:1 v/v) mixture by the slow evaporation method.

Chalcone Derivative 1:

(2E)-3-(3-Bromophenyl)-1-(4-chlorophenyl)prop-2-en-1-one

The crystal structure of the compound (2E)-3-(3-Bromophenyl)-1-(4-chlorophenyl)prop-2-en-1-one has been determined at room temperature. Data were collected using a Bruker SMART diffractometer system and data reduction is done using SAINT. The structure was solved by using SHELXS97 program. Programs SHELXL97 and PLATON are used to refine the structure. For molecular graphics, ORTEP-3 is used. The crystal data, data collection and refinement are summarized.

Chalcone Derivative 2:

(2E)-1-(1,3-Benzodioxol-5-yl)-3-(2-bromophenyl)prop-2-en-1-one

The molecule of the title compound, $C_{16}H_{11}BrO_3$, is shown in figure. It is essentially planar with a maximum deviation of $0.178(4)\text{\AA}$ and the configuration of the ketogroup with respect to the olefinic double bond is typically *cis*. In the crystal structure, intermolecular Br...O interactions [$3.187(3)\text{\AA}$] give rise to chains parallel to the *b*-axis. Adjacent chains are further linked along the *a*-axis by C...H...r interactions. The crystal studied was a racemic twin with a $0.595(13):0.405(13)$ ratio. The crystal data, data collection and refinement done for the above compound.

Conclusion:In the compound, (2E)-3-(3-Bromophenyl)-1-(4-chlorophenyl) prop-2-en-1-one: a non-merohedral twin, the molecule adopts an E configuration with respect to the C = C double bond and the dihedral angle between the aromatic ring planes is $3.98 (16)^\circ$. In the crystal, inversion dimers linked by pairs of C \cdots H \cdots O bonds are seen and weak $\pi - \pi$ stacking [centroid–centroid separation = $3.8776 (19) \text{ \AA}$] may further consolidate the structure. The crystal studied was a non-merohedral twin with a ratio of the twin components of 0.9093 (13):0.0907 (13). The twin operation is a twofold rotation around c.

In the compound, (2E)-1-(1,3-Benzodioxol-5-yl)-3-(2-bromophenyl) prop-2-en-1-one has, the dihedral angle between the mean planes of the methoxy- and bromosubstituted benzene rings is $24.6 (1)^\circ$. The angles between the mean plane of the prop-2-en-1-one group and the 4-bromophenyl and 2-methoxyphenyl ring planes are $18.8 (1)$ and $6.0 (1)^\circ$, respectively.

The molecule of the compound, (2E)-1-(1,3-Benzodioxol-5-yl)-3-(2-bromophenyl) prop-2-en-1-one, is essentially planar with a maximum deviation of $0.178 (4) \text{ \AA}$ and the configuration of the keto group with respect to the olefinic double bond is typically s-cis. In the crystal structure, intermolecular Br \cdots O interactions [$3.187 (3) \text{ \AA}$] give rise to chains parallel to the b axis. Adjacent chains are further linked along the a axis by C \cdots H \cdots π interactions. The crystal studied was a racemic twin with a 0.595 (13):0.405 (13) ratio.