

Synthesis of Aurone Dibromide from Aurone

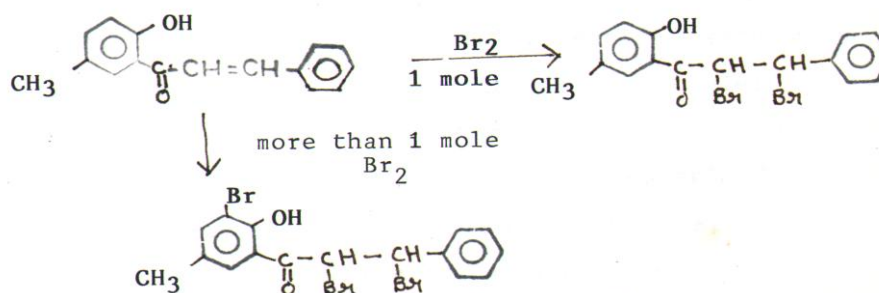
Dr. Shamal K. Doifode
 HOD Chemistry G.C.O.E., AMRAVATI

Bromination of olefinic = bond or α, β unsaturated ketone can be carried out by molecular bromine in solvent like acetic acid, CS_2 , CCl_4 , 1,2-dioxane, DMSO, ether etc. Chalcone (IUPAC Name- 1,3- diaryl pro-2-en-one) is usually brominated by bromine in acetic acid to obtain chalcone dibromide (2,3-dibromo-1, 3-diphenyl propane-1-one)¹. There are number of brominating reagent for addition of bromine^{3,4,5}.

Dioxane dibromide has also been reported as a versatile brominating reagent. Bromination with this reagent gives good yield⁶. Buffer bromination using sodium acetate, acetic acid & bromine has been used⁷.

Aurone has the exo C=C(bond length 1.33 Å) & therefore it is expected to undergo addition reaction. No such work is referred in literature for the preparation aurone dibromide from aurone. So we can prepared aurone dibromide from aurone.

For Ex: Preparation of Aurone dibromide from Aurone. The Aurone (0.01 Mol) was dissolved in Glacial acetic acid then Bromine in acetic acid solution, (25% W/V, 6.4 ml) was added with stirring. After 15 minutes, solid separated was filtered and washed with petroleum ether.



Using the same procedure, the other Aurone dibromide were prepared.

Table 1

Sr.no.	Aurone (4a-h)	M.P.	Aurone dibromide	M.P.
1	2-(4'-methoxy benzilidene)-5-methyl coumaran-3-one	154	2,α-dibromo-2-(4'-methoxy benzyl)coumaran-3-one	136
2	2-(4'-methoxy benzilidene)-coumaran-3-one	140	2,α-dibromo-2-(4'-methoxy benzyl)-5-methyl coumaran-3-one	158
3	2-(benzilidene)-5-methyl coumaran-3-one	112	2,α-dibromo-2-(benzyl)-5-methyl coumaran-3-one	107
4	2-(4'-methoxy benzilidene)3-nitro-5-methylcoumaran-3-one	230	2,α-dibromo-2-(4'-methoxy benzyl)-3-nitro-5-methyl coumaran-3-one	228
5	2-(benzilidene)3-nitro-5-methyl coumaran-3-one	226	2,α-dibromo-2-(benzyl)-3-nitro-5-methyl coumaran-3-one	217
6	2-(4'-methoxy benzilidene)3-bromo-5-methyl	176	2,α-dibromo-2-(4'-methoxy benzyl)-3-bromo-5-methyl	182

	coumaran-3-one		coumaran-3-one	
7	2-(4'-methoxy benzilidene)3-bromo-5-methylcoumaran-3-one	160	2,α-dibromo-2-(benzyl)-3-bromo-5-methyl coumaran-3-one	145
8	2-(4'-methoxy benzilidene)-4-methoxy coumaran-3-one	180	2,α-dibromo-2-(4'-methoxy benzyl)-4-methoxy coumaran-3-one	138

PROPERTIES OF PRODUCT

It is white powdery solid compound having melting point 158⁰C. It does not give any colouration with neutral ferric chloride solution. From the analytical data, the molecular formula was found to be C₁₇H₁₄Br₂O₃. The molecular wt =426.

TLC. RF value was found to be 0.36 for Benzene as a solvent on silica gel G plate1 with a layer thickness of 0.3 mm.

Elemental analysis:

C: found 47.62% C: calculated 47.88%
H: found 3.10% H: calculated 3.28%
Br: found 38.15% Br: calculated 37.55%

UV SPECTRUM

UV spectrum was recorded in methanol & is reproduced on plate no.2
λ_{max} value are recorded 203.8 nm, 254.2 nm, 343.2nm & 395.4 nm corresponding.
π- π* and n- π*in Aurone dibromide.

IR SPECTRUM

IR spectrum was recorded in nujol and reproduced on plate no.3.

Region	frequency	Co-relation
1750-1725	1730(s)	C=O stretching in 5 member ring
1300-1200	1280(s)	Ar-O stretching in Aromatic ether
1350-1100	1180(s)	C-O stretching
1050-1010	1010(s)	-OCH ₃ stretching in aromatic ether
750-500	750(s)	C-Br stretching

NMR SPECTRUM

PMR spectrum was recorded in CDCl₃ with TMS as an internal standard and is reproduced on plate no4.

The observed chemical shift can be CO- related as follows

1.6δ	s	1H	C-H
2.44 δ	s	3H	ArCH ₃
3.94 δ	s	3H	-OCH ₃
6.92-8.1 δ	m	7H	Ar-H

The spectra are in harmony with the proposed structure for Aurone dibromide.

REFERENCES

- [1] Ghiya, B.J. and Marathe, M.G., J. Sci. Industrial research, 20B(1961) 41.
- [2] Ghiya, B.J. and Marathe, M.G., J. Sci. Industrial research, 21B(1962) 28.
- [3] Kohr and mulley monatsch, 30 (1909) 407
- [4] E. Schulrk and k. brger, Acla,China, Acad. Sci.Hung., 17(1958) 211 (chem.. Abst.m 55 (1960) 5391)
- [5] Varlkr Migradisichian 'organic synthesis' Vol. II, Reinhold publication Corp. New York, USA 1951.
- [6] Tarent ev. A.P., chem.. Abstr., 47(1953) 8032h
- [7] Doifode K.B., " Chemistry of diketones", Ph.D. thesis, Nagpur university,1965.