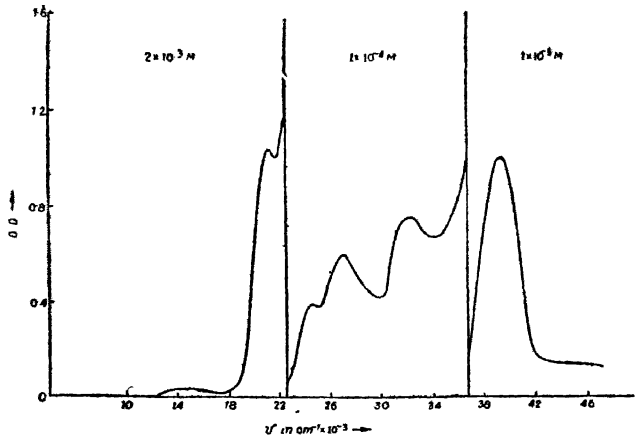

Fig. 1. Electronic spectra of (Oxin H_2) [$MoOBr_5$] in 9M HBr.

line with stirring. This was cooled in freezing mixture when greenish yellow precipitate of the compound appeared. This was filtered, recrystallised from hydrobromic acid and dried in vaccum over solid KOH to constant weight, yield 3g.

Found : Mo, 11.48% ; Br, 49.97% ; N, 3.49%

Calculated for $C_{18}H_{16}N_2O_2$ [$MoOBr_5$], Mo, 11.93% ; Br, 49.73% ; N, 3.48% Oxidation state of the metal determined by modified ceric sulphate method was +5.

Oxo-μ-dioxoaquo ,(8-hydroxyquinolinato) molydenum (V), [Mo_2O_4 $(Oxine)_2(H_2O)_2$] : 2g of (Oxine $H_2)_2$ [$MoOBr_5$] was slowly boiled with 100 ml water when there was immediate precipitation of a pink solid. This was filtered, washed with water and dried as before, yield 0.5g.

Found : Mo, 32.90% ; N, 4.59% ; oxidation state of Mo, +5.

Calculated for [Mo_2O_4 $(oxine)_2$ $(H_2O)_2$], Mo, 32.98% ; N, 4.81%.

Properties : Conductivity measurements of the salt $(OxineH_2)_2$ [$MoOBr_5$] in aqueous medium show that it is extensively hydrolysed and ionised. But the molar conductance of the derivative in acetonitrile is much less than that of a binary electrolyte Δ=8.5 ohm^{-1} cm^2 for $10^{-3}M$ solution, which indicates the nonelectrolytic nature of the compound. The salt (μ eff = 1.55 B. M. at 33°) contains molybdenum in quinquevalent state. The nonelectrolytic compound is diamagnetic due to the presence of oxobridged species $Mo_2O_4^{2+}$. Infrared spectra of these compounds clearly show the ν(M=O) frequency at 991 cm^{-1} and 961 cm^{-1}. The broad bands in the region 700-800 cm^{-1} may be traced as metal-oxygen chain (-Mo-O-Mo-O) vibrations[2] in the nonelectrolytic species.

Electronic spectra : The spectra of the salt $(OxineH_2)_2$ [$MoOBr_5$] was taken in 9 M HBr and the nonelectrolytic compound [$Mo_2O_4(Oxine)_2$ $(H_2O)_2$] in acetonitrile. The results are tabulated in Table 1.

TABLE 1

Compound	λ_{max} in mu	$\nu \times 10^{-3}$ in cm^{-1}	ϵ_{molar} extinction
	695	14.3	14
(Oxine H)[$MoOBr_5$]	469	21.3	519
	408	24.5	3800
	370	27.0	5950
	308	32.4	7510
	254	39 3	1007
	538	18.5	168
[Mo_2O_4(Oxine)(HO)]	384	26.0	660
	312	32.0	840
	246	40 6	4620

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References

1. R. G. JAMES and W. WARDLAW., *J. Chem. Soc.*, 1927, 2145.
2. P. C. H. MITCHELL., *J. Inorg. Nuclear Chem.*, 1963, 25, 965.
3. W. WARDLAW., *J. Chem. Soc.*, 1924, 125, 1941.
4. W. M. CHARMICHAEL and D. A. EDWARDS., *J. Inorg. Nuclear Chem.* 1968, 2641.
5. C.G. BARRACLOUGH, H. LEWIS and R. S. NYHOLM., *J. Chem. Soc.* 1957, 8552.
6. H. L. KRAUSS and W. HUBES., *Chem. Ber.* 1961, 94,2864.
7. H. B. GRAY and C. R. HARE., *Inorg. Chem.* 1962, 1, 363.

Chalcones XIII : Potential Germicides derived from Substituted Acetophenones

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THE antimicrobial and toxic action of amino, hydroxy, methoxy, and halo Chalcones have been reported by a few workers[1-12]. The antibacterial and germicidal activity of these compounds is due to conjugated systems[2,8-10]. These results have prompted us to synthesise several chalcones with a view to study their and other biological activity. The various subtituent, present in the nucleus, by changing their position from ortho to meta and then to para extends the length of the conjugated system. This may reinforce the germicidal activity of compounds.

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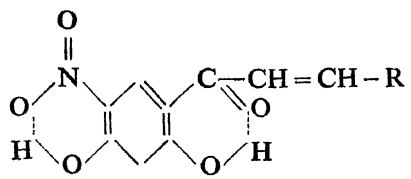
In all twenty five substituted chalcone compouds have been prepared by Claisen-Schmidt condensation of 2-methyl-4-methoxy ; 3-methyl-4-methoxy-; 3-methyl-6-methoxy-; 2,5-dimethoxy- and 5-nitro-2, 4-dihydroxy acetophenones, with different aryl aldehydes at room temperature.

Russel[3,13] has observed, during the preparation of polyhydroxy chalcones, that dark coloured tarry masses were obtained instead of desired compounds. Similar types of difficulties have been encountered in the syntheses of nitro chalcones[14-17]. Therefore, the presence of both nitro and hydroxy groups in the reacting molecules will be more detrimental to chalcone syntheses. But contrary to the general experience we have obtained 2', 4'-dihydroxy-5'-nitrochalcones in good yields. To us, one feasible reason seems to be the hydrogen bonding between the pairs of nitro hydroxy and carbonyl hydroxy groups, which are situated ortho to each other, The formation of hydrogen bonding leaves no hydroxyl group free. which in earlier syntheses were reported to responsible for poor or no yields of chalcones.



The identity of the chalcones was established by elemental analyses, halochromism with Conc. H_2SO_4 (produced orange to violet shades) and in a few cases by preparing 2,4-DNP's which analysed satisfactorily for nitrogen.

Determination of Germicidal Activity

The compounds were assayed against *E. Coli*, *S. Aureus* and *A. Niger* for germicidal and fungicidal activity at a concentration of $150 \pm 10 \mu g$/ml. in 50% aqueous ethanol by following Agar-Cup method. The inocula were prepared from stationary culture and inhibition of growth was checked at an incubation temperature of ($32° \pm 2°$) after 24 hr. The strength is reported by measuring the diameter of zone of inhibition of a particular microorganism (Table 1) and the results were compared against benjoic acid.

Experimental

Preparation of 5-nitro resacetophenone[10] : It was prepared by nitration of resacetophenone[10] in glacial acetic acid.

Preparation of Chalcone : Chalcones reported here have been prepared by the methods reported[3,4,7,12] earlier. One typical example is given below.

An equimolar alcoholic mixture of 2,5-dimethoxy acetophenone and 2-chloro benzaldehyde was treated with saturated solution of sodium in methanol (5 ml) at 25° with constant stirring. After keeping the reaction mixture for an overnight yellow crystalline compound was obtained, which was recrystallised from alcohol (Table 1).

TABLE 1—PHYSICAL CHARACTERISTICS AND ANALYTICAL DATA OF CHALCONES*
Ar—CH=CH—CO—Ar'

No.	Ar	Ar'	M. P. °C Chal.	M. P. °C 2,4-DNP	% Yield	M. F.	Found C	Found H	Calc C	Calc H	Dia. of zon. of inh.in mm.
1.	2-Cl	2',5'-$(OMe)_2$	98	—	81	$C_{17}H_{15}O_3Cl$	67.38	4.83	67.44	4.96	10
2.	3-Cl	2',5'-$(OMe)_2$	167	—	78	$C_{17}H_{15}O_3Cl$	67.41	4.66	67.44	4.96	9
3.	4-Cl	2',5'-$(OMe)_2$	224	—	79	$C_{17}H_{15}O_3Cl$	67.40	4.80	67.44	4.96	12
4.	3-Me	2',5'-$(OMe)_2$	151	—	80	$C_{18}H_{18}O_3$	76.50	6.33	76.59	6.38	—
5.	4-Me	2',5'-$(OMe)_2$	78	215	85	$C_{18}H_{18}O_3$	76.55	6.34	76.59	6.38	—
6.	4-Me-6-OMe	2'-Me-4'OMe	103	—	49	$C_{19}H_{20}O_3$	77.14	6.72	77.02	6.76	—
7.	2-Cl-6-OMe	2'-Me-4'-OMe	91	237	48	$C_{18}H_{17}O_3Cl$	68.21	5.25	68.25	5.37	10
8.	3-Cl-6-OMe	2'-Me-4'-OMe	115	—	50	$C_{18}H_{17}O_3Cl$	68.13	5.18	68.25	5.37	9
9.	4-NMe_2	2'-Me-4'-OMe	108	—	68	$C_{19}H_{21}ON$	81.56	7.50	81.72	7.53	—
10.	2-Cl	2'-Me-4'-OMe	226	—	38	$C_{17}H_{15}O_2Cl$	71.18	5.16	71.19	5.24	9
11.	4-Cl	2'-Me-4'-OMe	233	—	36	$C_{17}H_{15}O_2Cl$	71.03	5.10	71.19	5.24	10
12.	4-Cl	3'-Me-4'-OMe	92	220	43	$C_{17}H_{15}O_2Cl$	71.06	5.02	71.16	5.24	9
13.	4-NMe_2	3'-Me-6'-OMe	141	—	64	$C_{19}H_{21}O_2N$	77.06	7.06	77.29	7.12	—
14.	2-Cl	2',4'-$(OH)_2$-5'-NO_2	205	—	80	$C_{15}H_{10}O_5NCl$	56.50	3.00	56.34	3.13	9
15.	3-Cl	2',4'-$(OH)_2$-5'-NO_2	203	—	76	$C_{15}H_{10}O_5NCl$	56.15	2.90	56.34	3.13	8
16.	4-Cl	2',4'-$(OH)_2$-5'-NO_2	216	254	86	$C_{15}H_{10}O_5NCl$	56.20	3.30	56.34	3.13	11
17.	4-NMe_2	2',4'-$(OH)_2$-5'-NO_2	141	—	60	$C_{17}H_{16}O_5N_2$	62.00	4.95	62.20	4.87	—
18.	2-Me	2',4'-$(OH)_2$-5'-NO_2	197	246	75	$C_{16}H_{13}O_5N$	64.10	4.45	64.21	4.35	—
19.	3-Me	2',4'-$(OH)_2$-5'-NO_2	185	—	83	$C_{16}H_{13}O_5N$	64.00	4.50	64.21	4.35	—
20.	4-Me	2',4'-$(OH)_2$-5'-NO_2	189	—	80	$C_{16}H_{13}O_5N$	64.45	4.20	64.21	4.35	—
21.	3-OMe	2',4'-$(OH)_2$-5'-NO_2	172	—	76	$C_{16}H_{13}O_6N$	60.70	4.00	60.95	4.12	—
22.	2-OH-3-OMe	2',4'-$(OH)_2$-5'-NO_2	224	—	62	$C_{16}H_{13}O_7N$	57.80	4.20	58.00	3.93	—
23.	2,3-OMe_2	2',4'-$(OH)_2$-5'-NO_2	190	—	84	$C_{17}H_{15}O_7N$	59.00	4.20	58.13	4.34	—
24.	3-OH	2',4'-$(OH)_2$-5'-NO_2	238	—	50	$C_{15}H_{17}O_6N$	59.55	3.85	59.80	3.65	—
25.	4-OH	2',4'-$(OH)_2$-5'-NO_2	137	275	37	$C_{15}H_{11}O_6N$	60.00	3.43	59.80	3.65	—
Benzoic Acid											*13

* All melting points are uncorrected

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References

1. E. SCHRAUFSTATTER and S. DEUTSCH., *Z. Naturforsch.*, 1948, **4b**, 430.
2. J. K. EATON and R. G. DAVIES,, *Ann. Applied Bio.*, 1950, **37**, 471.
3. S. S. MISRA and R. S. TEWARI., *Proc. Nat. Acad. Sci.* (India), 1970, **40**(A), 468.
4. S. S. MISRA, S. C. KUSHWAHA and J. B. LAL., *Proc. Nat. Acad. Sci.* (India), 1970, **40**(A)/301,
5. R. SCHRAUFSTATTER and S. DEUTSCH., *Z. Naturforsch*, 1948, **3b**, 168.
6. S. S. MISRA, R. S. TEWARI and J. B. LAL., *Proc. Nat. Acad. Sci.* (India), 1970, **40**(A), 296.
7. S. S. MISRA and R. S. TEWARI., *Labdev J. Sci. Tech.* (India), 1971, **8**, 152.
8. W. B. GEIGER and J. E. CONN., *J. Amer. Chem. Soc.*, 1945, **67**, 112.
9. S. S. MISRA, Ph. D. Thesis, 1970, Kanpur University, Kanpur.
10. S. S. MISRA and R. S. TEWARI., *Indian J. Appl. Chem.*, 1971, **34**, 260.
11. M. KAMODA., *J. Agr. Chem. Soc., Japan*, 1954, **38**, 791.
12. S. S. MISRA and DINKAR., *J. Indian Chem. Soc.*, 1972, **49**, 637.
13. J. RUSSEL and H. CLARKE., *J. Amer. Chem. Soc*, 1939, **61**, 3651.
14. T. SZELL., *Chem. Ber.*, 1959, **92**, 1672.
15. G.SIPOS and T.SZELL., *Act. Phys. et Chem.*, 1959, **5**, 70.
16. T. SZELL., *Chem. Ber.*, 1960, **93**, 1928.
17. D. N. DHAR and J. B. LAL., *J. Org. Chem.*, 1958, **23**, 115.
18. J. W. COOPER., *Org. Synth.*, 1941, **21**, 102.
19. R. E. OMER and N. HAMILTON., *J. Amer. Chem. Soc.*, 1937, **59**, 642.
20. T. A. GEISSMAN and R. O. CLINTON., *J. Amer. Chem. Soc.*, 1946, **68**, 692.
21. S. S. MISRA., *J. Indian Chem. Soc.*, 1973, **50**, 68.
22. S. S. MISRA., *J. Indian Chem. Soc.*, 1973, **50**, 335.