

lyser based on LEED optics configuration. In our Research Centre, an Auger spectrometer has been built, using the retarding grid analyser for the electron energy analysis. Details of construction of the spectrometer have been previously reported¹. In this communication, we report some of the results obtained for Mg-metal and MgO samples.

The magnesium metal sample, in the form of a strip, was mounted on a copper plate. The MgO sample was prepared in the form of a pellet. A primary electron beam, from an electron gun, having an energy 1 keV and a current of 2 micromperes was used to induce Auger electron emission from the metal and its oxide. A peak to peak modulation of 5 volts was employed. In Fig. 1, the low energy spectra obtained for both Mg-metal and MgO are shown. In both cases, there are three prominent peaks, marked 1, 2, 3 for metal and 4, 5, 6 for oxide. The peak energies and their transitions are given in Table I. In the case of metal, peak '2' (energy = 45 eV) corresponds to $L_{2,3}-VV$ transition. Out of the three peaks, this peak should have been most intense. The peak '3' (energy = 58 eV) appears most intense because of interference from Auger electrons with the same energy, resulting from the copper plate on which the metal was mounted. The peak at 34 eV (peak '1' in Fig. 1) arises due to a bulk plasmon loss as well as due to MgO layer formed on the metal. In the case of MgO (Fig. 1b) the most intense peak occurs at an energy of 34 eV (peak '4') and corresponds to $Mg^{2+} L_{2,3} O^{2-} L_{2,3} O^{2-} L_{2,3}$ transition.

The results (Table I) provide with one important piece of information, viz., that the low energy Auger spectra could also be used to know the chemical state of an element. The most intense peak, in the case of Mg-metal, occurs at 45 eV, involving a core level and two valence levels of Mg-atom in the transition. However, in the case of MgO, the most intense peak is seen at 34 eV, involving a core level of Mg-atom and two core levels of oxygen. Our results also demonstrate clearly

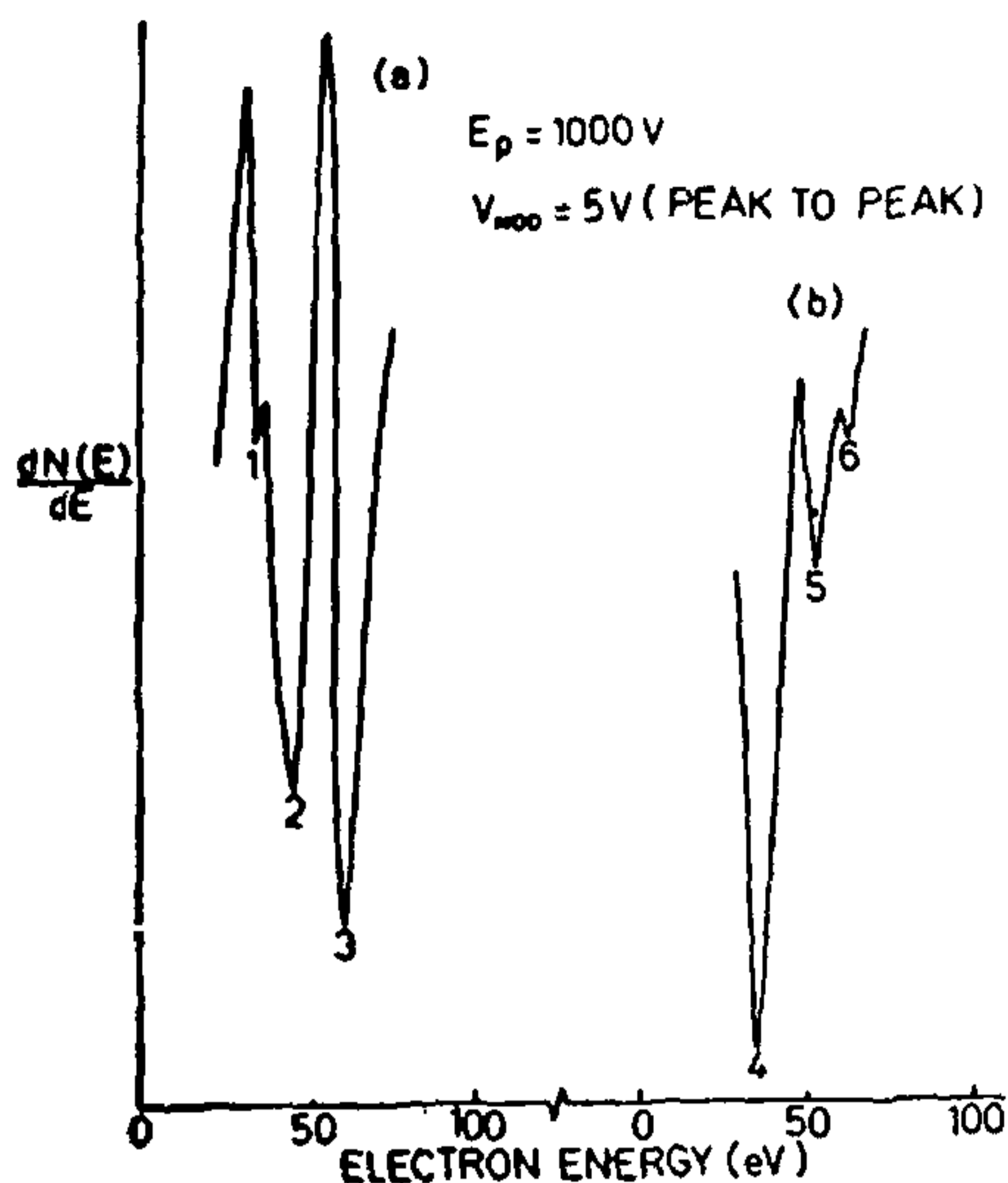


FIG. 1. Low energy Auger spectra of (a) Mg-metal and (b) MgO.

that the resolution of the indigenously built spectrometer is comparable to that obtained with some of the commercial Auger spectrometers.

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- Gopalaraman, C. P., Luktuke, R. D. and Kane, S. A., *Proceedings of the D.A.E. Symposium on Analytical Instruments and Instrumentation*, January 1979, p. 3.
- Janssen, A. P., Scheonmaker, R. C., Chambers, A. and Prutton, M., *Surf. Sci.*, 1974, 45, 45.

TABLE I
Observed Auger electron energies

Peak No.*	Energy (eV)	Transition ²
1	34	$Mg^{2+} L_{2,3} O^{2-} L_{2,3} O^{2-} L_{2,3} +$ bulk plasmon
2	45	$Mg L_{2,3} - VV$
3	58	$Mg L_{2,3} - VV$
4	34	$Mg^{2+} L_{2,3} O^{2-} L_{2,3} O^{2-} L_{2,3}$
5	52	$Mg^{2+} L_{2,3} O^{2-} L_{2,3} O^{2-} L_{2,3}$
6	60	$Mg^{2+} L_{1} O^{2-} L_{1} O^{2-} L_{2,3}$

* See Fig. 1 for Peak No.

SYNTHESIS WITH α -PHENYLCINNAMOYL CHLORIDE AND DIAZOALKANES

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1-DIAZO-1-alkyl-2,4-dihenyl-but-3-en-2-one (I) and 3-alkyldiazoacetyl-3,4-dihenyl-5-alkyl pyrazoline (II) were obtained by the action of 2 and 3 moles of diazoalkanes on 1 mole of α -phenylcinnamoyl chloride.

α -Phenylcinnamoyl chloride contains two sites of reactivity, an acid chloride group and a double bond, towards diazoalkanes. By adopting the method of Arndt, Eistert and Partale^{1,2} the action of 2 moles of diazomethane, diazethane, diazopropane, diazo-

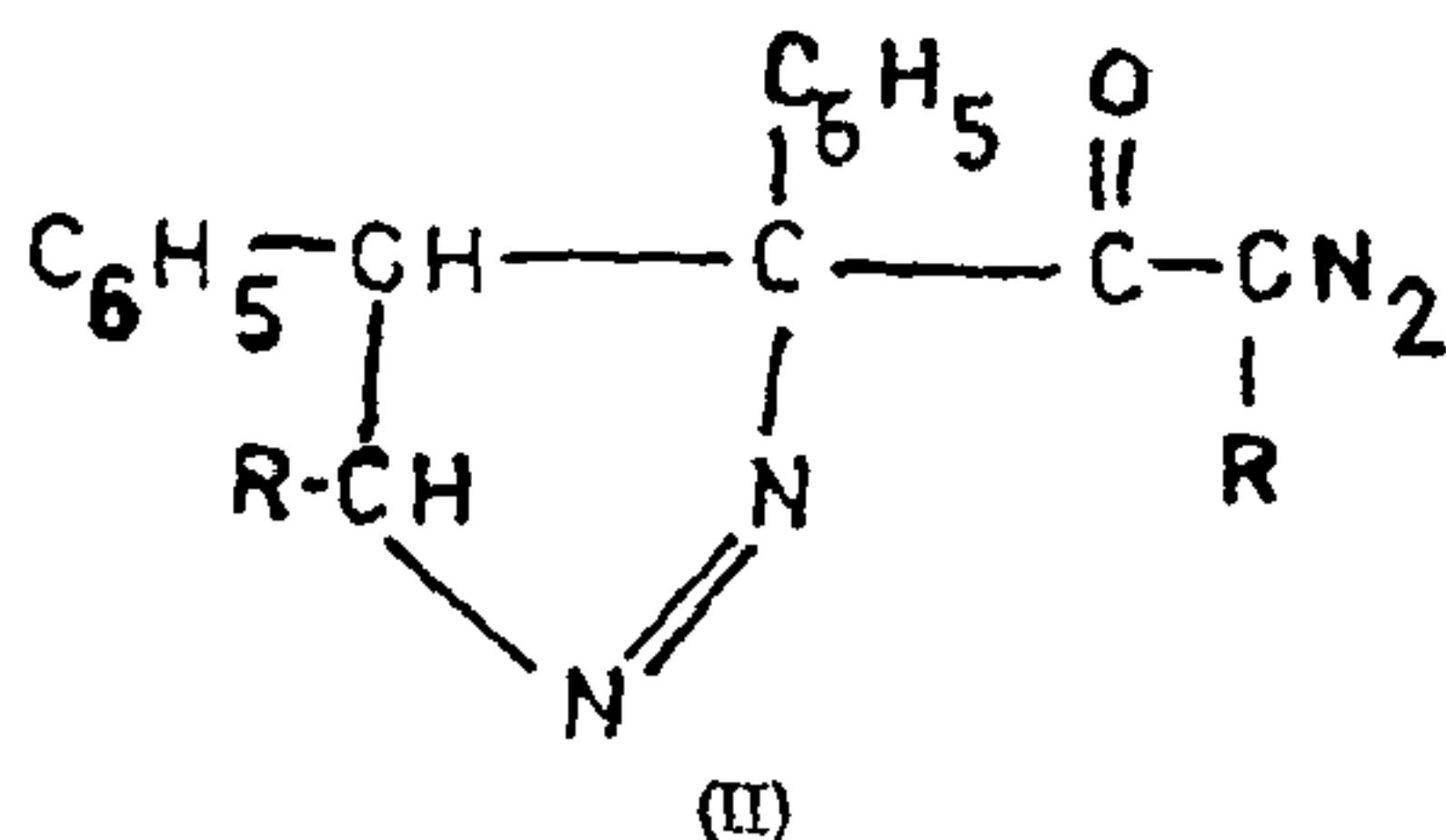
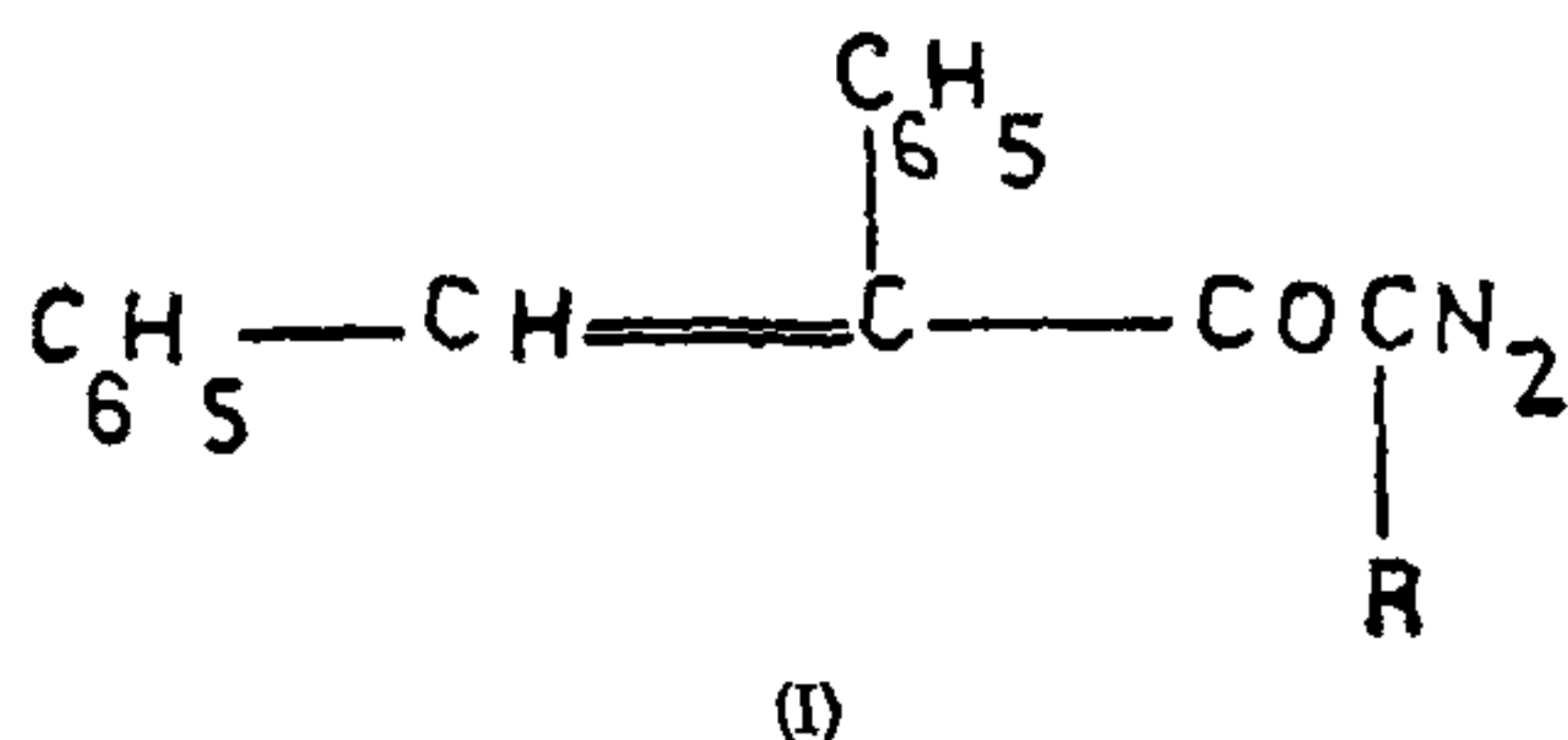
n-butane and phenyldiazomethane has been carried out with α -phenylcinnamoyl chloride (1 mole) in ether at 0°, α -diazoketones (I, R = -H, -CH₃, -C₂H₅, -*n*-C₃H₇, and -C₆H₅) were obtained. With 3 moles of diazoalkanes the double bond^{2,10} was also affected and pyrazolines (II, R = -H, -CH₃, -C₂H₅, -*n*-C₃H₇, and -C₆H₅) were formed.

All the diazoketones (I and II) were found to be orange-red viscous liquids. But these could not be distilled under reduced pressure on account of their decomposition; with 2,4-dinitrophenyl hydrazine^{3,8} all of these readily afforded the corresponding osazone derivatives.

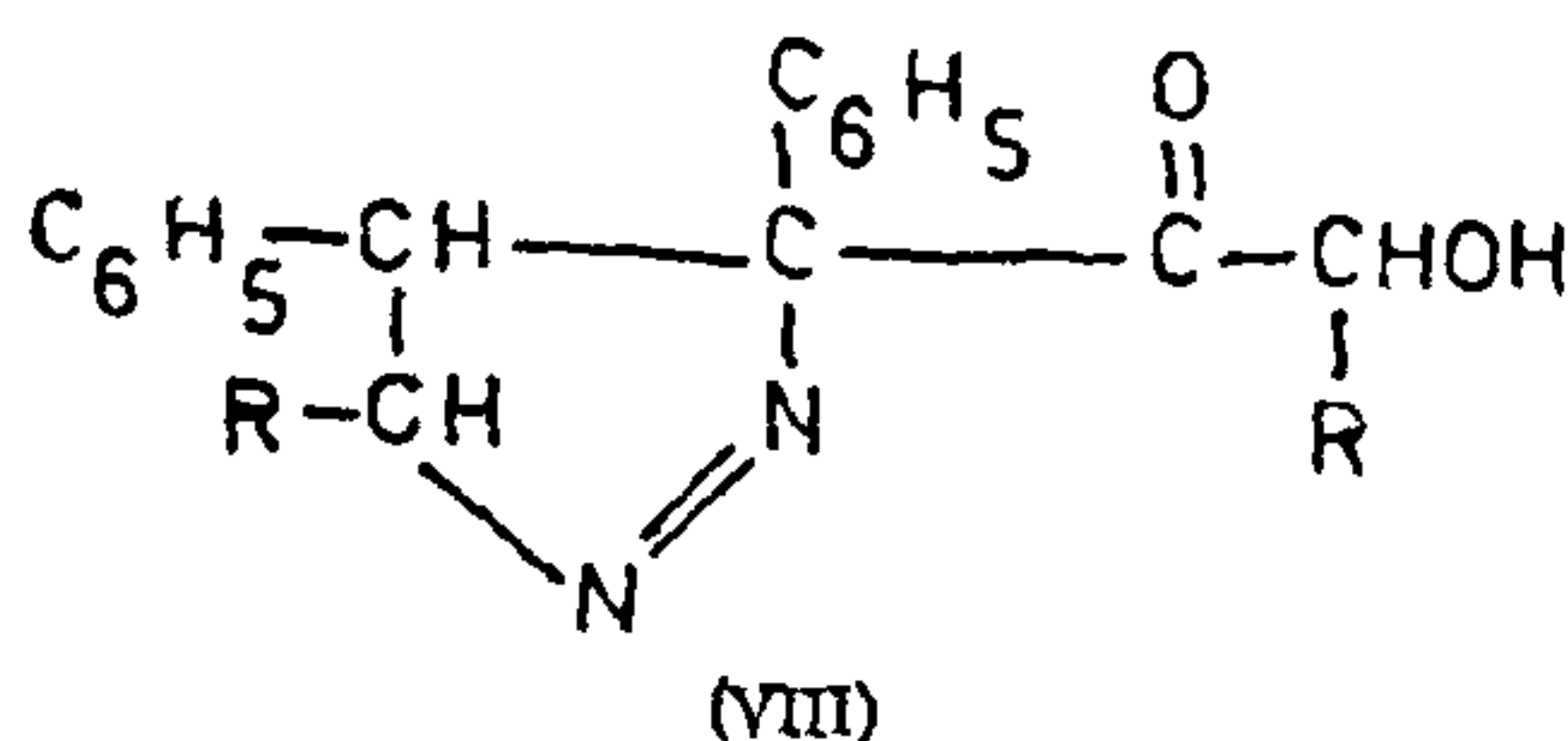
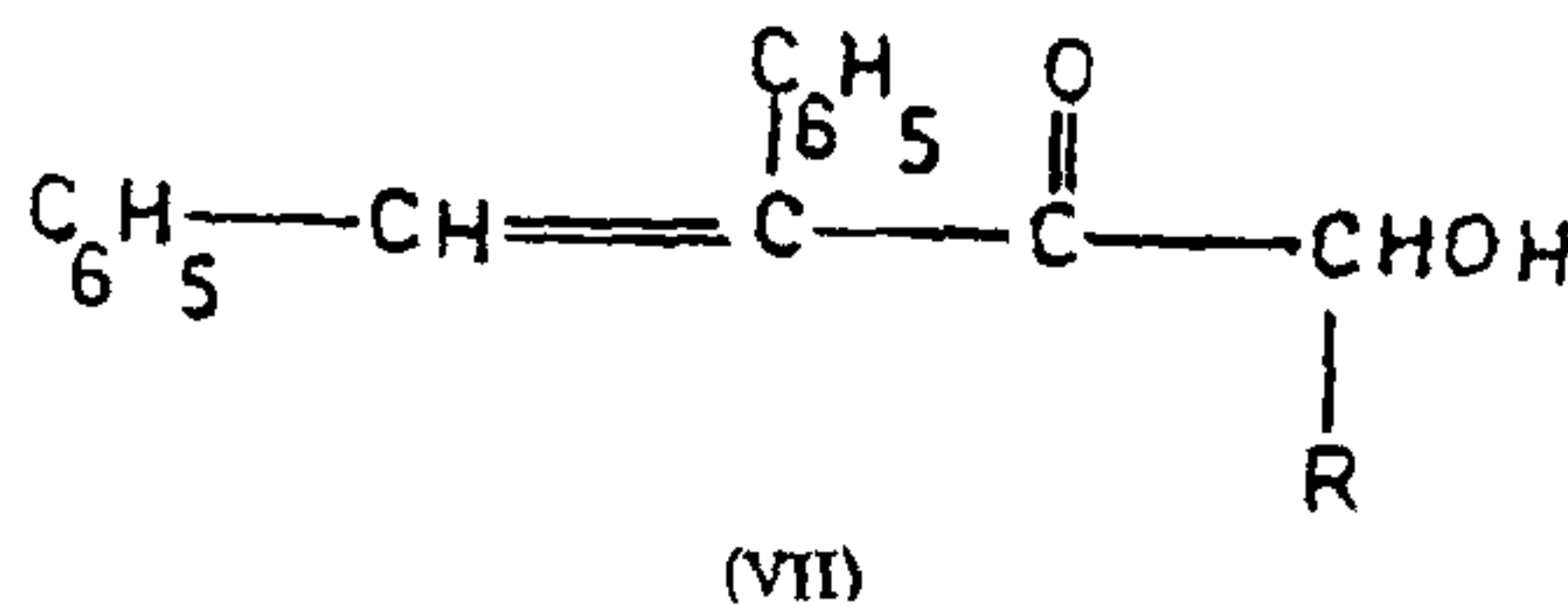
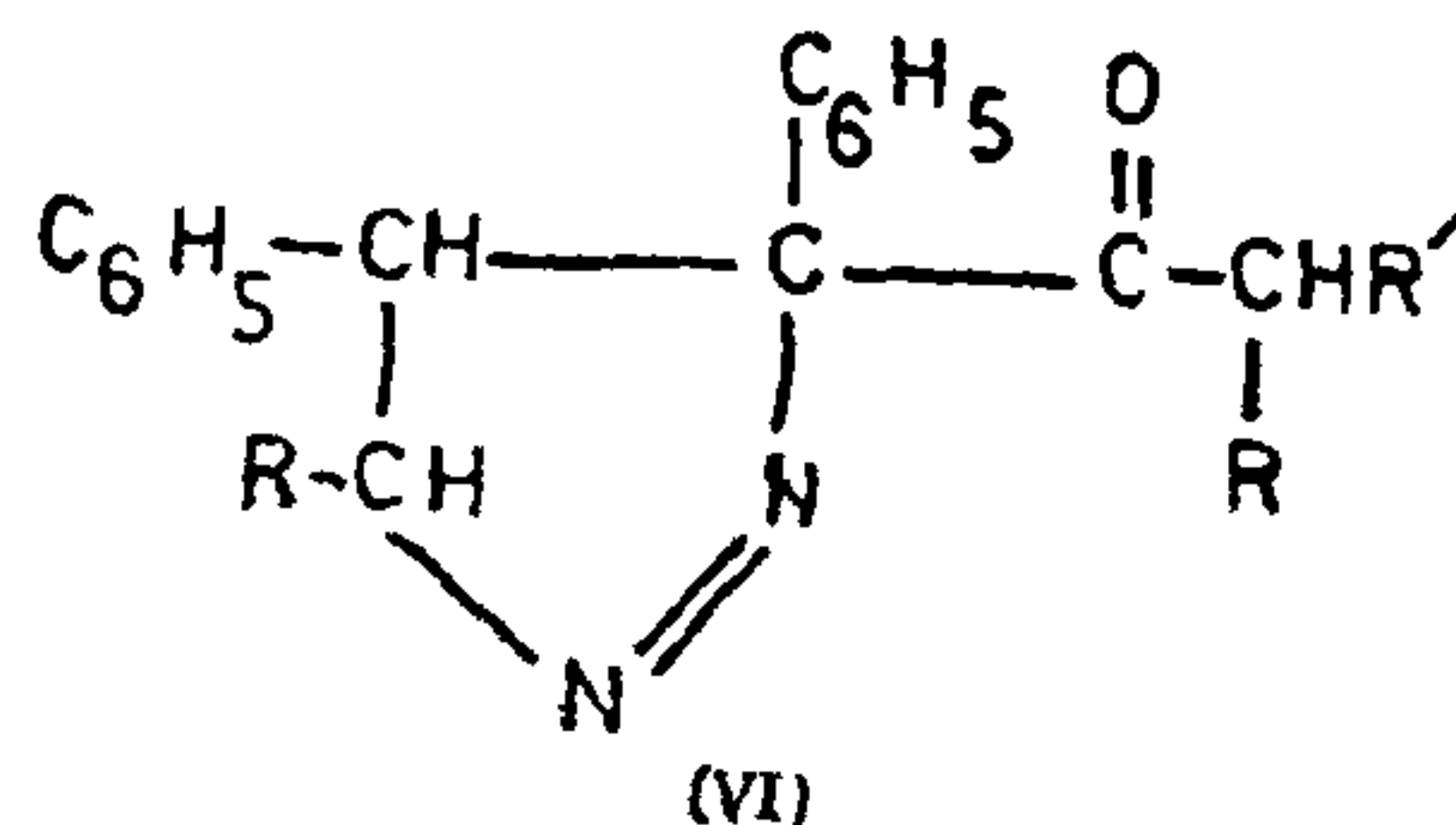
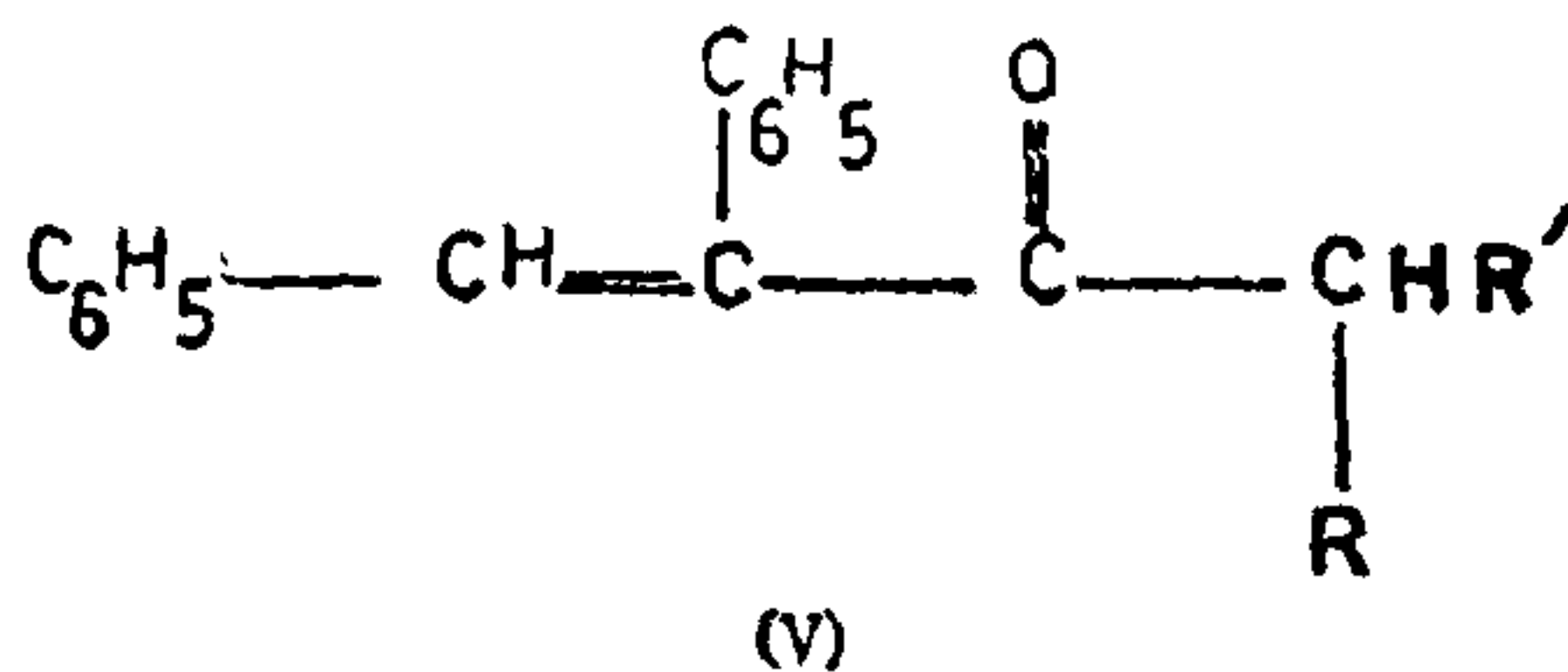
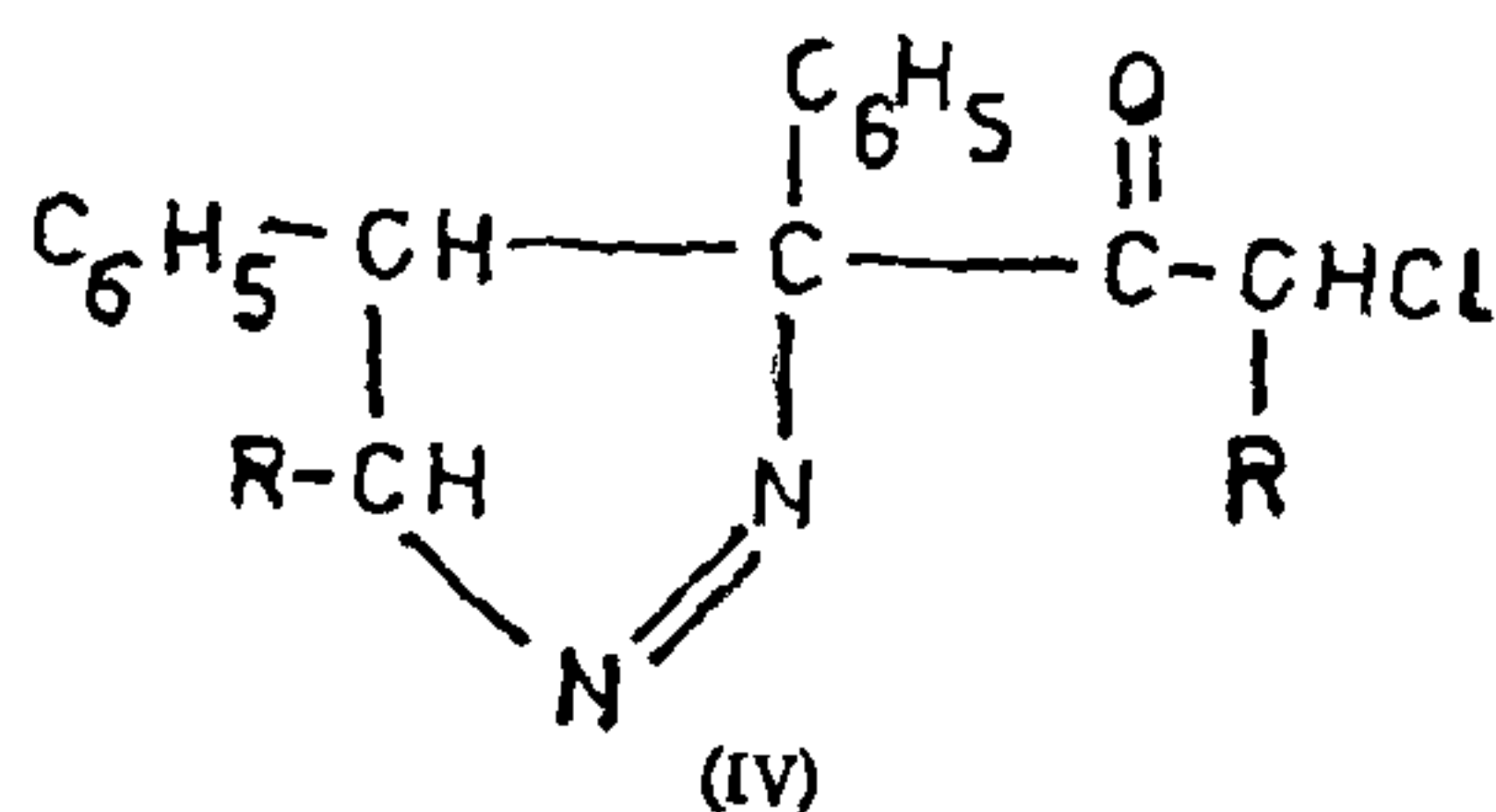
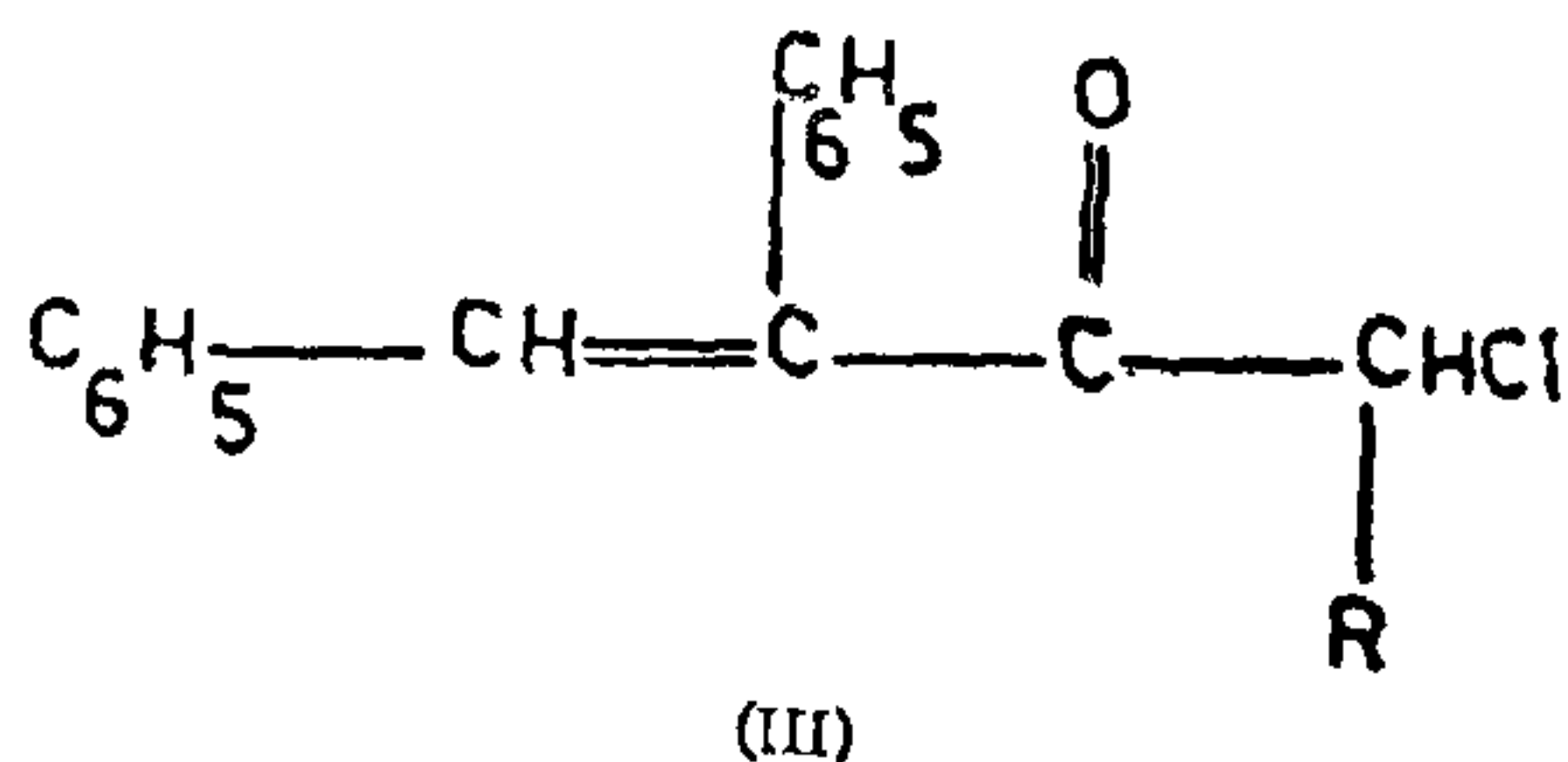
These diazoketones (I and II) on treatment with dry HCl⁵ in benzene evolved nitrogen and produced 1-chloro-1-alkyl-3,4-diphenyl-but-3-en-2-one (III) and 3-chloro-alkylacetyl-3,4-diphenyl-5-alkyl pyrazolines (IV), as red brown viscous liquids, those obtained with 2 and 3 moles with phenyldiazomethane were yellow solids, which on crystallisation from alcohol, melted sharply at 259° and 248° respectively.

All the diazoketones (I and II) on heating with molten phenol⁵ and benzoic acid⁵ and working up the reaction products afforded 1-benzyloxy-1-alkyl-3,4-diphenyl-but-3-en-2-one (V, R' = OC₆H₅), 3-benzyloxy-alkylacetyl-3,4-diphenyl-5-alkyl pyrazolines (VI, R' = OC₆H₅) and 1-benzoyloxy-1-alkyl-3,4-diphenyl-but-3-en-2-one (V, R' = OCOC₆H₅), 3-benzoyloxy-alkylacetyl-3,4-diphenyl-5-alkyl pyrazolines (VI, R' = OCOC₆H₅).

All the above compounds except (IV, VI, R = -*n*-C₃H₇) afforded their 2,4-dinitrophenylhydrazones, characterised by IR spectroscopy^{4,6} and elemental analysis.



(I & II, R = -H, -CH₃, -C₂H₅, -*n*-C₃H₇, and -C₆H₅)



All the diazoketones (I and II) on treatment with dilute hydrochloric acid¹¹ and extraction with ether gave 1-hydroxy-1-alkyl-3,4-diphenyl-but-3-en-2-one (VII) and 3-hydroxy alkylacetyl-3,4-diphenyl-5-alkyl pyrazolines (VIII) as orange red viscous liquid. These afforded the same 2,4-dinitrophenylosazones as of original diazoketones, further confirmed by determining their mixed melting points, which did not show any depression.

Experimental Procedure

Distilled α -phenylcinnamoyl chloride⁹ (1 mole) was dissolved in ether and added to pre-estimated ethereal solution of diazoalkanes (2 and 3 moles respectively) at 0° and kept overnight. Next day, solvent was stripped off and α -diazoketones (I and II) were obtained.

All these diazoketones (I and I') were subjected to the above reactions, i.e., with dry HCl, molten phenol and benzoic acid and different derivatives were formed. These were mobile or viscous liquids but could not be distilled under reduced pressure on account of their decomposition. With 2,4-dinitrophenylhydrazine most of them readily afforded the corresponding osazone derivative. Results are summarised as follows:

1-Diazo-3,4-diphenyl-but-3-en-2-one, $C_{16}H_{12}N_2O$

2,4-DNP-m.p. 142°; chloroketone, 2,4-DNP, m.p. 108°; phenoxy derivative 2,4-DNP, m.p. 96°; benzoyloxy derivative 2,4-DNP, m.p. 110°.

3-Diazoacetyl-3,4-diphenyl pyrazoline, $C_{17}H_{14}N_4O$

2,4-DNP, m.p. 120°; chloroketone 2,4-DNP, m.p. 125°; phenoxy derivative 2,4-DNP, m.p. 90°; benzoyloxy derivative 2,4-DNP, m.p. 60°.

1-Diazo-1-methyl-3,4-diphenyl-but-3-en-2-one, $C_{17}H_{14}N_2O$

2,4-DNP, m.p. 125°; chloroketone 2,4-DNP, m.p. 135°; phenoxy derivative 2,4-DNP, m.p. 117°; benzoyloxy derivative 2,4-DNP, m.p. 92°.

3-Methyl diazoacetyl-3,4-diphenyl-5-methyl pyrazoline, $C_{19}H_{18}N_4O$

2,4-DNP, m.p. 134°; chloroketone 2,4-DNP, m.p. 110°; phenoxy derivative 2,4-DNP, m.p. 156°; benzoyloxy derivative 2,4-DNP, m.p. 125°.

1-Diazo-1-propyl-3,4-diphenyl-but-3-en-2-one, $C_{18}H_{14}N_2O$

2,4-DNP, m.p. 130°; chloroketone 2,4-DNP, m.p. 104°; phenoxy derivative 2,4-DNP, m.p. 95°; benzoyloxy derivative 2,4-DNP, m.p. 77°.

3-Ethyl diazoacetyl-3,4-diphenyl-5-ethyl pyrazoline, $C_{21}H_{22}N_4O$

2,4-DNP, m.p. 78°; chloroketone 2,4-DNP, m.p. 134°; phenoxy derivative 2,4-DNP, m.p. 120°; benzoyloxy derivative 2,4-DNP, m.p. 118°.

1-Diazo-propyl-3,4-diphenyl-but-3-en-2-one, $C_{19}H_{18}N_2O$

2,4-DNP, m.p. 106°; chloroketone 2,4-DNP, m.p. 130°; phenoxy derivative 2,4-DNP, not melted upto 305°; benzoyloxy derivative 2,4-DNP, m.p. 230°.

3-Propyl-diazoacetyl-3,4-diphenyl-5-propyl pyrazoline, $C_{23}H_{26}N_4O$

2,4-DNP, m.p. 140°; chloroketone, phenoxy derivative and benzoyloxy derivative does not form 2,4-DNP.

1-Diazo-1-phenyl methyl-3,4-diphenyl-but-3-en-2-one, $C_{22}H_{16}N_2O$

2,4-DNP, m.p. 248°; chloroketone 2,4-DNP, m.p. 225°; phenoxy derivative 2,4-DNP, m.p. 227°; benzoyloxy derivative 2,4-DNP, m.p. 215°.

3-Phenyl diazoacetyl-3,4-diphenyl-5-phenyl pyrazoline $C_{29}H_{22}N_4O$

2,4-DNP, m.p. 240°; chloroketone 2,4-DNP, m.p. 236°; phenoxy derivative 2,4-DNP, m.p. 234°; benzoyloxy derivative 2,4-DNP, m.p. 225°.

Elemental analysis and IR data agreed with the literature values within the limit of experimental errors and also supported the structures.

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