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o FORMYLARYLAZOMETHYLENETRIPHENYLPHOSPHORANES A FACILE THERMALLY PROMOTED REARRANGEMENT TO 3-OXO-INDAZOLINE AND 4-OXO-DIHYDROQUINAZOLINE DERIVATIVES

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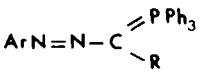
Abstract The o formylarylazomethylenetriphenylphosphoranes carrying an electron withdrawing group on the ylidic carbon undergo thermal intra molecular cyclization to 3-oxo-indazolin-2-yl-methylenetriphenylphosphorane derivatives The latter compounds, and their 1-alkyl derivatives, in turn, undergo thermal and/or acid catalyzed rearrangement to 4-oxo-1,4-dihydroquinazoline derivatives and PPh_3 Some possible reaction mechanisms are discussed, and some synthetic applications of the above reactions are shown

During the last years we have been exploring the reactivity of arylazomethylenetriphenylphosphoranes (1) towards C-C[1], C-N[2], and C-O[3,4] multiple bonds and electrophilic reagents[5], both inter



(1)

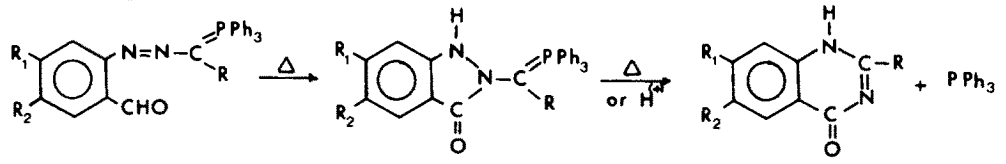
and intra molecularly, and we have already referred on some peculiar features of the behaviour of these compounds, whose reactivity seems strictly tied to their ability of delocalizing the negative charge from the ylidic carbon on the azo group In a preliminary note[3] we communicated that compound (1a), refluxed in toluene for a short time, gave the "intermediate" (2a), identified later on[4] as 3-oxo-indazolin-2-yl(methoxycarbonyl)methylenetriphenylphosphorane Compound (2a), on further heating, afforded the 4-oxo-1,4-dihydroquinazoline* derivative (3a) In the same note we reported also that compound (1b), heated at 70° in toluene for 2h, gave PPh_3 and the 1,3-dioxolo[4,5-g]quinazoline derivative (3b)

We wish now to communicate the results of our subsequent studies on the above thermally promoted intra molecular reactions

In order to gather some indication of their scope and mechanism , we tried them on other compounds, carrying different substituents on the ylidic carbon To this purpose we prepared the o formylphenylazomethylenetriphenylphosphoranes (1c-f) (1c-e) Were synthesized from the corresponding hydrazonoyl chlorides following a reported procedure[6] (1f) Was prepared from o aminobenzaldehyde by diazotization and coupling with (methylthio-thiocarbonyl)methylenetriphenylphospho

*The 4-oxo-1,4-dihydroquinazolines with unsubstituted nitrogen atoms are tautomeric with the corresponding 4-oxo-3,4-dihydroquinazolines

rane, following the method of Markl[7]

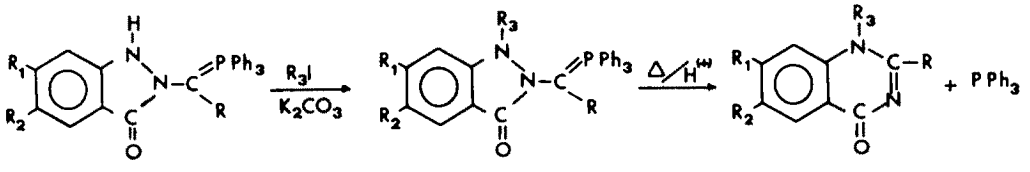


(1) (2) (3)

	a	b	c	d	e	f	g
R	$COOCH_3$	$COOCH_3$	$COCH_3$	SO_2Ph	SO_2-C$_6$H$_4$-CH_3	$C(=S)SCH_3$	$COOCH_3$
R_1	H	O, CH_2, O	H	H	H	H	H
R_2	H		H	H	H	H	Br

* (2g) Was prepared from 5-bromoisatine

A careful study of the thermal behaviour of compounds (1a-f) has shown only little differences in their reactivity and in that of the corresponding 2-oxo-indazolinyl derivatives (2a-f) (1a) Was shown to give (2a) on heating for a short time at 65-70° C in methanol, chloroform and benzene solution, the reaction is catalyzed by acids and made slower by the presence of bases Among the new compounds (1f) was the most reactive one and could not be isolated from its dichloromethane solutions, because on solvent evaporation at reduced pressure at room temperature it was transformed into (2f) (1c-e), instead, did not react at a reasonable rate until 70° C, in toluene solution, but, while (1c) gave (2c), (1d,e) were transformed straight into the corresponding 4-oxo-dihydroquinazoline derivatives (3d,e) and PPh_3, and it was not possible to isolate the "intermediates" (2d,e), nor to detect them To obtain (3c), (2c) was heated at 95° C for two hours Also (2g)[4] was transformed into (3g) by heating at 90° C in toluene solution for three hours (2f) Appeared as the most stable member of the series it remained practically unreacted until 110° C, at this temperature it gave $Ph_3P{=}S$ and an up to now intractable mixture of many products The (2a) to (3a) rearrangement, as well as that of (2g) to (3g), is catalyzed by the presence of acids, which allow the reaction to be carried out at room temperature The acid catalyzed 3-oxo-indazoline to 4-oxo-1,4-dihydroquinazoline rearrangement was successfully carried

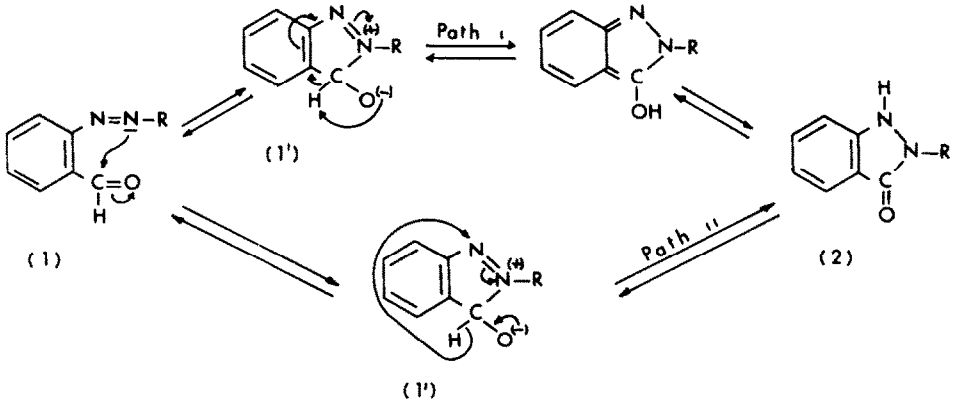


(2a,g) (2h,i) (3h,i)

	R	R_1	R_2	R_3
h	$COOCH_3$	H	H	CH_3
i	$COOCH_3$	H	Br	CH_3

out also on the 1-methyl derivatives of (2a,g), namely (2h,i), and led to the 1-methyl-2-methoxycarbonyl-4-oxo-1,4-dihydroquinazolines (3h,i), which cannot be obtained by direct alkylation of the corresponding 4-oxo-dihydroquinazolines (3a,g), that give the 3-alkyl derivatives[3]
In scheme 1 are indicated two mechanistic hypothesis which may account for the above experimental results at present we have no evidence to make a choice between them

Scheme 1



The azo group attacks, as a nucleophilic agent, the o formyl substituent, to give the cyclic dipolar species (1')* In path i, the formation of a stable compound by electron shift and charge neutralization should act as driving force for the following proton migration from a carbon to the adjacent O⁻** In path ii the same result should be achieved by hydride ion transfer*** In each case hydrogen migration seems a fundamental step in the whole reaction if in (1c) the formyl proton is exchanged with a methyl group, no reaction takes place under 110° C, and at this temperature thermal decomposition leads to an intractable mixture of tarry products
From the above results it comes out that the formation of 3-oxo-indazolines by intra molecular reaction of an aldehydic function with an ortho azo group is not much influcenced by the nature of the substituents on this group, and, in phosphoranes (1a-f), is preferred with respect to the intra molecular electrophilic attack on the ylidic carbon Actually, no phosphoranes of type (1), where R is a keto or an alkoxycarbonyl group, has ever given an inter molecular Wittig reaction, probably because of the presence of the additional electron withdrawing azo group**** This is also in accord with the fact that in these compounds the ^{1}H NMR signal of the methoxycarbonyl group is not split[10] Quite unexpectedly, also phosphoranes (2a,c,f), although the electron withdrawing N=N group is no longer present and their ^{1}H NMR spectra show the common features of phosphorus ylides stabilized only by an alkoxycarbonyl substituent, do not under

* The formation of 3-oxo-indazoline derivatives on thermal treatment of an o formylarylazo compound has been reported without mechanistic explanation[8]

** Analogous proton shifts are mechanistic steps generally accepted to explain other reactions (e g the benzoin condensation)

*** An analogous hydride ion shift is the key step in the Cannizzaro reaction

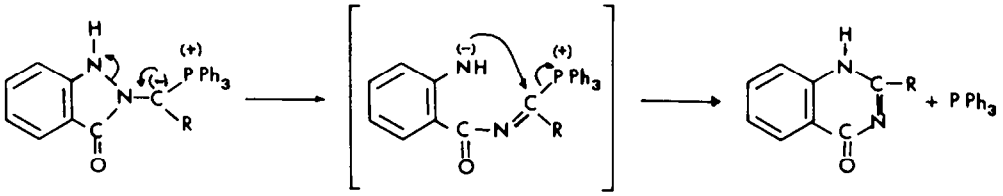
**** In accord with this behaviour, electrophiles like CS_2[5] or alkyl iodides[9] attacked phosphoranes (1) at the azo group

go the common reactions of these phosphorus ylides at moderate temperature they are not hydrolyzed in basic media, they are not oxidized by t butyl peroxide, they do not react with aldehydes, and methyliodide alkylates them at the indazolinonyl nitrogen At higher temperatures (2a,c) rearrange to (3a,c) before any other reaction and (2f) decomposes X ray analysis of (2i) has shown[4] that the ylidic carbon is rather shielded by the substituents and this might be the reason for the low reactivity of that carbon atom in compounds (2)

With regard to the rearrangement of (2) to (3) two reaction pathways are sketched in scheme 2, which can rationalize the experimental observations Following path I the (2) to (3) rearrangement

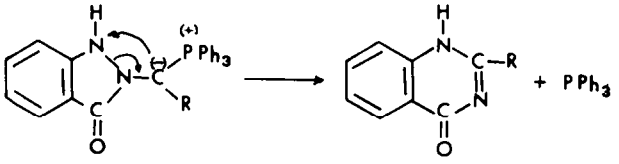
Scheme 2

Patn I



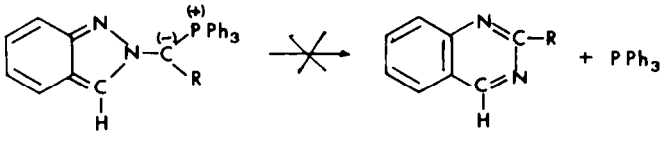
(2) (4) (3)

Path II



(2) (3)

should be a two-step reaction, with the intermediate formation of the open chain compound (4) Path II, instead, indicates a concerted one step reaction Analogous mechanisms can operate in the acid catalyzed reaction, where they would be favoured since the nitrogen in position 1 of compounds (2) would be protonated * Although we cannot rule out anyone of these pathways, we are rather inclined towards the two-step one, since the concerted one would imply the attack of a carbanion on an sp^3 hybridized nitrogen Moreover, the 3-oxo-indazolines C=O group seems to play an important role in the above rearrangement, possibly delocalizing the negative charge in the open chain compound (4) In fact, phosphoranes (5)[2] do not undergo a similar rearrangement to (6)

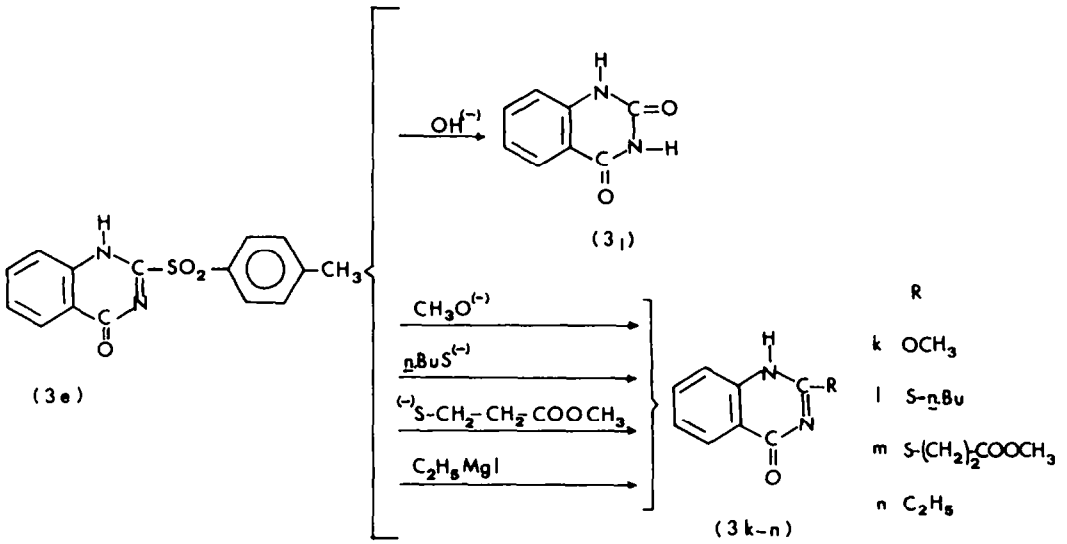


(5) (6)

* Such protonated form is likely to be in tautomeric equilibrium with compounds protonated on different positions, e g on the ylidic carbon

The 4-oxo-1,4-dihydroquinazoline (3e) has been differently functionalized in position 2

Scheme 3



(3e) Actually reacted very smoothly with a series of nucleophiles, namely water, alkoxides, thiolates, organometallics, as indicated in scheme 3, giving the 2,4-dioxo-1,2,3,4-tetrahydroquinazoline(3j) and the 4-oxo-1,4-dihydroquinazolines (3k,n) in good yields Therefore, by the reactions described in this paper, arylazomethylenetriphenylphosphoranes are shown to be useful starting materials also for the synthesis of a number of 4-oxo-dihydroquinazoline derivatives through the intermediacy of compounds (2), which undergo an unexpectedly facile rearrangement, with cleavage of an N-N bond and formation of a C-N one with PPh_3 elimination

EXPERIMENTAL

M p s were taken by means of a Buchi apparatus and are uncorrected IR spectra were recorded by a Perkin-Elmer X98 spectrophotometer ^1H NMR spectra were taken with a Varian EM 390 spectrometer Chemical shifts are expressed as δ values ($SiMe_4$ as internal standard) Silica gel 60 (Merck, 70-230 mesh) was used for column chromatography T l c s were performed on Merck precoated silica gel 60F-254 plates Crystallization solvents, analytical and chemico-physical data of 3-oxo-indazoline and 4-oxo-1,4-dihydroquinazoline derivatives are reported in Table 1 and 2, respectively

o Formylarylazomethylenetriphenylphosphoranes (1a-e)

Compounds (1a-e) were prepared from the corresponding hydrazonoyl chlorides by reaction with PPh_3 and Et_3N in CH_3CN at room temperature[6] (1a,c,e) Have been already described[2,6]

2-Formyl-4,5-methylenedioxy-phenylazo(methoxycarbonyl)methylenetriphenylphosphorane (1b) yellow solid, washed with Et_2O and then with water and acetone, at 160-170 °C becomes white, and melts at 200-210 °C ν_{max} (cm^{-1}, nujol) 1760, 1670, ^1H NMR ($CDCl_3$) 3,55(3H, s), 5 80(2H, s), 6 9-7 7(17H, m), 9 1(1H, s) Analysis found % C=68 29, H=4 58, N=5 43, for $C_{29}H_{23}N_2O_5P$ calctd % C=68 23, H 4 51, N 5 49

2-Formyl-phenylazo(phenylsulphonyl)methylenetriphenylphosphorane (1d) m p 157 °C (from C_6H_6)

ν_{max} (cm^{-1}, nujol) 1680, 1350, 1140 ^1H NMR ($CDCl_3$) 6 6-8(24H, m), 9 25, 10 8(1H, 2s) (mixture of cis-trans isomers) Analysis found % C=70 08, H=4 61, N=5 12, for $C_{32}H_{25}N_2O_3PS$ calctd % C= 70 07, H=4 56, N=5 11

(Methylthio-thiocarbonyl)methylenetriphenylphosphorane

This compound has been prepared by a modification of the method of Yoshida and coworkers[11] To a slurry of sodium hexamethyldisilazane (5g) in dry C_6H_6 (50 cm^3) methyltriphenylphosphonium iodide (10 25 g) was added at room temperature, while stirring under nitrogen After 1/2 h a solution of dimethyl trithiocarbonate (2 97 cm^3) in dry C_6H_6 (10 cm^3) was added The mixture was refluxed for 3 h and then filtered while hot The solvent was evaporated at reduced pressure and the residue, washed with EtOAc, gave 3 6 g of the title compound, m p 194 °C (lit [11] 184 °C)

3-Oxo-indazolin-2-yl(methylthio-thiocarbonyl)methylenetriphenylphosphorane (2f)

o Aminobenzaldehyde (0 215 g) dissolved in 18% HCl (1 2 cm^3) was diazotized with $NaNO_2$ (0 15 g) in H_2O (1 3 cm^3) This diazonium salt solution was slowly added to a slurry of (methylthio-thio carbonyl)methylenetriphenylphosphorane (0 65 g) in dioxane (7 75 cm^3), at a temperature of 7-8 °C 1N NaOH was added to pH 6-7, the mixture was extracted with CH_2Cl_2, the organic layer dried over Na_2SO_4 and the solvent evaporated at reduced pressure, at room temperature T l c (eluent $CHCl_3$-EtOAc 1 1) examination of the mixture before and after solvent evaporation showed that, during this operation, the main reaction product had undergone a transformation Treatment of the evaporation residue with Et_2O led to the precipitation of (2f) as an orange solid (0 36 g), which was purified by column chromatography (eluent $CHCl_3$-EtOAc 9 1) and crystallization

1-Methyl-3-oxo-indazolin-2-yl(methoxycarbonyl)methylenetriphenylphosphorane (2h)

To a slurry of (2a) (1 26 g) and K_2CO_3 (0 46 g) in MeOH (100 cm^3) CH_3I (10 cm^3) was added at room temperature The clear solution so obtained was stirred for 1h The solvent was evaporated at reduced pressure and the residue extracted several times with $CHCl_3$ By solvent evaporation a residue was obtained, which, on treatment with light petroleum, gave pure (2h) (1 12 g) For analytical purposes it was crystallized

Thermal treatment of (1a-e) general procedure

Solution of (1a-e) (2 10^{-3} moles) in toluene (25 cm^3) were heated under nitrogen at 70 °C (at lower temperature no reaction took place) Reaction progress was monitored by t l.c. (eluent C_6H_6-EtOAc 8 2) At reaction completion (2 h for (1a,b), 5 h for (1c,e), 3 h for (1d)) the mixtures were cooled at room temperature The products precipitated and were collected and crystallized from suitable solvents (1a,c) Gave (2a,c) in 81% yield, (1b,d,e) gave (3b,d,e) in 87%, 83% and 93% yield, respectively Mother liquors from (3d) and (3e), by evaporation, gave PPh_3 in nearly quantitative yield

2-Acetyl-4-oxo-dihydroquinazoline (3c)

(2c) (1 1 10^{-3} Moles) was heated at 95 °C in toluene (10 cm^3) under nitrogen for 2 h (reaction progress monitored by t l c , eluent C_6H_6-EtOAc 8 2) (3c) Precipitated on cooling the toluene solution at room temperature and was collected and crystallized Yield 98%

Acid catalyzed rearrangement of (2a,g) to (3a,g)

By addition of 5% HCl (0 5 cm^3) to a slurry of (2a,g) (4 4 10^{-4} moles) in MeOH (5 cm^3) clear solutions were obtained, but very soon white solids precipitated The mixtures were stirred

at room temperature for 1 h in the case of (2a) and 7 h in the case of (2g) The solids were collected, washed with water, dried at room temperature and washed with Et_2O, to give (3a) and (3g), practically pure, in 40% and 86% yield, respectively

Rearrangement of (2h,i) to (3h,i)

(2h,i) Refluxed in toluene or dioxane for 5 h, were recovered unchanged (2h) (1 10^{-3} Moles) and p toluensulphonic acid (PTSA) (1 5 10^{-3} moles) were stirred in toluene at 45 °C for 2 h The precipitate was collected, extracted with $CHCl_3$ and washed with water The organic layer, dried over Na_2SO_4 and evaporated, afforded (3h) in quantitative yield

(2i) (1.1 10^{-3} Moles) was refluxed 1 h in dioxane (25 cm^3) in the presence of a catalytic amount of PTSA The solvent was evaporated at reduced pressure and the residue was extracted with $CHCl_3$ and washed with water The organic layer, dried over Na_2SO_4 and evaporated, gave a residue, which, by crystallization, afforded (3i) in 35% yield

2,4-Dioxo-1,2,3,4-tetrahydroquinazoline (3j)

To a slurry of 2-p tosyl-4-oxo-dihydroquinazoline (3e) (1 g) in EtOH (10 cm^3) an EtOH solution of KOH was added to pH 8 The mixture was stirred 1 h at room temperature, the solvent evaporated at reduced pressure and the residue dissolved in water By making the solution acidic with 10% HCl a white solid precipitated, that was collected and crystallized to give 0 52 g of pure (3j), identified by comparison with an authentic sample

2-Methoxy-4-oxo-1,4-dihydroquinazoline (3k)

A mixture of (3d) (0,60 g), $NaOCH_3$ (0 50 g) and tetraoctylammonium bromide (TOAB) (0 30 g) was refluxed in C_6H_6 (20 cm^3) for 24 h After solvent evaporation at reduced pressure the residue was dissolved in water From this solution, brought to pH 4 with dil HCl, a white solid precipitated, which was collected and washed with hot acetone, to give the title compound (0 18 g)

2-n Butylthio-4-oxo-1,4-dihydroquinazoline (3l)

To the mixture obtained from n BuSH (0 15 g), NaOH (0 067 g) and TOAB (0 184 g) in $CHCl_3$ (15 cm^3), a solution of 2-p tosyl-4-oxo-dihydroquinazoline (3e) (0 5 g) in $CHCl_3$ (15 cm^3) was added After 24 h refluxing, the mixture was extracted with water The organic layer was dried over Na_2SO_4 and the solvent was evaporated at reduced pressure The residue was crystallized to give the title compound (0 30 g)

2-(2-Methoxycarbonyl)ethylthio-4-oxo-1,4-dihydroquinazoline (3m)

To the mixture obtained from 2-methoxycarbonyl-ethylthiol (1 6 g), solid ground NaOH (0 187 g) and TOAB (0 187 g) in C_6H_6 (15 cm^3) a solution of quinazoline (3e) (1 4 g) in C_6H_6 (50 cm^3) was added After 5 h refluxing the solvent was evaporated at reduced pressure and the residue was crystallyzed to give the title compound

2-Ethyl-3-oxo-1,4-dihydroquinazoline (3n)

To a Grignard solution, prepared from EtI (2 5 g) and Mg (0 36 g) in t BuOMe (40 cm^3), a solution of (3e) (0 5 g) in dry tetrahydrofuran (40 cm^3) was added After 3 h refluxing the mixture was cooled at room temperature, diluted with water, treated with conc HCl (3 cm^3) and extracted with Et_2O The water solution was brought to pH 8 with NaOH and extracted with $CHCl_3$ The organic layer, dried over Na_2SO_4, on solvent evaporation and crystallization of the residue, gave the title compound (0 2 g)

ACKNOWLEDGMENT The Italian Ministry of Education is acknowledged for financial support

Table 1

3-oxo-indazolin-2-ylmethylenetriphenylphosphoranes

Comp	M p °C (Cryst solvent)	Formula	Found % (Required) C	H	N	ν_{max} (cm^{-1}) (nujol)	NMR ($CDCl_3$ solution, if not otherwise stated)
(2a)	188-189 (CH_2Cl_2-light petr)	$C_{28}H_{23}N_2O_3P$	72 01 (71 75)	5 05 (5 13)	5 81 (5 70)	3100, 1637, 1615	3 25, 3 56 (3H, 2s), 6 8-8 (19H, m), 9 7 (1H, s)§
(2c)	194-195 ($CHCl_3$)	$C_{28}H_{23}N_2O_2P$	74 38 (74 67)	5 03 (5 11)	6 11 (6 22)	1650	1 8 (3H, s), 6 8-8 4 (19H, m), 10 3 (1H, s)
(2f)	220 (DMF-H_2O)	$C_{28}H_{23}N_2PS_2$	69 25 (69 71)	4 91 (4 77)	5 78 (5 81)	3210, 1654	2 4 (3H, s), 6 8-8 3 (19H, m), 10 4 (1H, s)*
(2g)	182-183 (EtOH)	$C_{28}H_{23}BrN_2O_3P$	61 47 (61 66)	3 91 (4 03)	5 06 (5 14)	1615	3 4, 3 6 (3H, 2s), 6 63-8 (18 5H, m), 10 (0 5H, s)§
(2h) H_2O	218 (i.PrOH)	$C_{29}H_{27}N_2O_4P$	69 67 (69 88)	5 40 (5 42)	5 66 (5 62)	1670, 1615	3 25, 3 30 (3H, 2s), 3 33, 3 57 (3H, 2s), 6 80-7 98 (19H, m)§
(2i)	214-215 (i PrOH)	$C_{29}H_{24}BrN_2O_3P$	62 10 (62 26)	4 33 (4 29)	5 08 (5 01)	1645	3 23, 3 27 (3H, 2s), 3 31, 3 55 (3H, 2s), 6 6-7 96 (18H, m)

§Cis-trans isomers[10]

*DMSO

Table 2

4-oxo-1,4-dihydroquinazolines

Comp	M p °C (Cryst solvent)	Formula	Found % (Required) C	H	N	ν_{max}(cm^{-1}) (nujol)	NMR ($CDCl_3$ solution, if not otherwise stated)
(3b)	225 (Pyridine)	$C_{11}H_8N_2O_5$	53 39 (53 22)	3 31 (3 22)	9 98 (9 86)	3190, 3160, 3110, 1765, 1680	3 9 (3H, s), 6 0 (2H, s), 7 3 (1H, s), 7 81 (1H, s)8 68 (H_2O+NH)*
(3c)	205 (EtOH)	$C_{10}H_8N_2O_2$	65 57 (63 83)	5 17 (5 32)	14 99 (14 89)	3200, 3070, 1710, 1670	2 65 (3H, s) 7 5-8 3 (4H, m), 12 2 (1H, s)**
(3d)	210 (MeOH)	$C_{14}H_{10}N_2O_3S$	58 44 (58 74)	3 68 (3 50)	9 51 (9 79)	1670	7 1-8 3(m)***
(3e)	198-200 (EtOH)	$C_{15}H_{12}N_2O_3S$	59 73 (60 00)	4 12 (4 00)	9 30 (9 33)	3170-3140, 1670, 1350, 1145	2 5 (3H, s), 7 2-8 35 (8H, m), 10 4 (1H, s)
(3g)	230-232 (AcOH)	$C_{10}H_7BrN_2O_3$	42 18 (42 42)	2 46 (2 47)	9 84 (9 89)	3170, 1735, 1670, 1610, 1600	3 91 (3H, s), 7 72 (1H, d), 8 00(1H, d), 8 21 (1H, s)**
(3h)	153-154 (i PrOH)	$C_{10}H_{10}N_2O_3$	60 64 (60 55)	4 63 (4 59)	12 84 (12 84)	1749, 1655	3 8 (3H, s), 4 06 (3H, s), 7 3-8 6 (4H, m)
(3i)	187 (EtOH)	$C_{10}H_9BeN_2O_3$	44 55 (44 46)	3 13 (3 03)	9 28 (9 43)	1740, 1650	3 8 (3H, s), 4 08 (3H, s), 7 33-8 48 (3H, m)
(3k)	232(dec)	$C_9H_8N_2O_2$	61 17 (61 36)	4 36 (4 54)	15 57 (15 60)	3260, 3160, 3050, 1720, 1705, 1680	4 (3H, s), 7 1-8 07 (4H, m), 12 (1H, s)**
(3l)	145 (i PrOH)	$C_{12}H_{14}NO_2OS$	61 28 (61 53)	6 21 (5 98)	11 74 (11 96)	3180, 3120, 3060, 1660	0 95 (3H, t), 1 2-2 (4H, m), 3 35 (2H, t), 7 1-8 38 (4H, m), 9 82 (1H, s)
(3m)	137-138 (C_6H_6-light petr)	$C_{12}H_{12}N_2O_3S$	54 79 (54 54)	4 74 (4 54)	10 48 (10 60)	3130, 1740, 1680	2 9 (2H, t), 3 6 (2H, t), 3 75 (2H, s), 7 2-8 2 (4H, m), 10 8 (1H, s)
(3n)	213 (i PrOH)	$C_{10}H_{10}N_2O$	68 67 (68 96)	5 76 (5 75)	15 98 (16 09)	3180, 1680	1 45 (3H, t), 2 85 (2H, q), 7 25-8 32 (4H, n)

* $^2H_5$ Pyridine

** DMSO

*** $CDCl_3$ + DMSO

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