

# Regio- and Stereoselective Addition of Imides to Ethyl 3-Phenyl-2-propynoate in the Presence of Triphenylphosphine. Single Crystal X-Ray Structure of Ethyl (*Z*)-2-(1,3-Dioxo-1,3,3a,4,7,7a-hexahydro-2*H*-isoindol-2-yl)-3-phenyl-2-propenoate

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A one-pot synthesis of sterically congested *N*-vinyl imides in fairly high yields by the reaction of ethyl 3-phenyl-2-propynoate, imides and triphenylphosphine is reported. The structures of these compounds were confirmed by IR, <sup>1</sup>H, and <sup>13</sup>C NMR spectroscopy, and by a single crystal X-ray structure determination. The structural analysis of the products indicated that the reaction is regio- and stereoselective.

**Key words:** Phosphorus Ylide, Ethyl 3-Phenyl-2-propynoate, Vinyltriphenylphosphonium Salts, Crystal Structure, *N*-Vinyl Imide

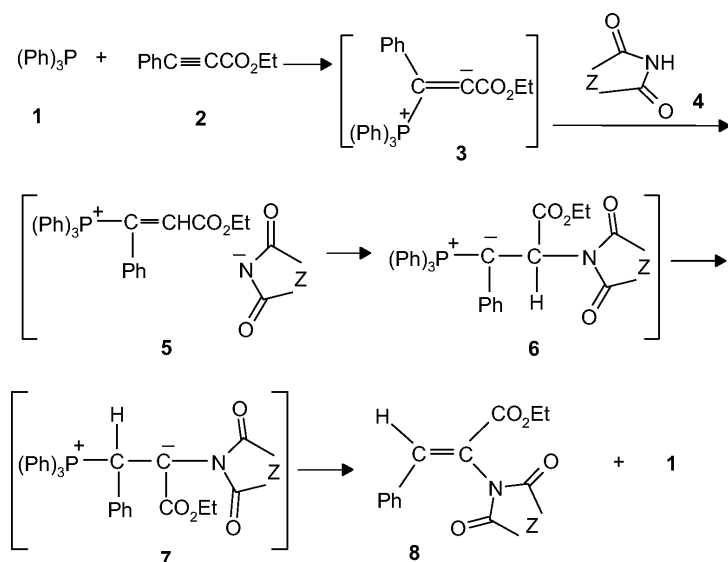
## Introduction

$\beta$ -Additions of nucleophiles to the vinyl group of vinylic phosphonium salts leading to the formation of new alkylidenephosphoranes has attracted much attention as a very convenient and synthetically useful method in organic synthesis [1]. Organophosphorus compounds have been extensively used in organic synthesis as useful reagents as well as important ligands in a number of transition metal catalysts [2]. Phosphorus ylides are a special type of zwitterions, which bear a strongly nucleophilic, electron-rich carbanion-like function adjacent to a phosphonium center. The electron distribution around the  $P^+-C^-$  bond and its consequent chemical implications have been probed and assessed through theoretical, spectroscopic and crystallographic investigations [1]. The nucleophilicity at the ylidic carbon is a factor of essential mechanistic importance in the use of phosphorus ylides as Wittig reagents. These are important reagents in synthetic organic chemistry, especially in the synthesis of naturally occurring products, compounds with biological and pharmacological activity [3]. These ylides are usually prepared by treatment of a phosphonium salt with a base, and phosphonium salts are usually obtained

from a phosphine and an alkyl halide. Phosphonium salts are also prepared by Michael addition of phosphorus nucleophiles to activated olefins and in other ways [4]. The phosphonium salts are most often converted to the ylide by treatment with a strong base, though weaker bases can be used if the salt is acidic enough. In recent years, we have established a one-pot method for the synthesis of stabilized ylides [5–8]. In this paper, we wish to describe the preparation and – for one example – the single crystal X-ray structure of sterically congested *N*-vinyl imides from the ethyl 3-phenyl-2-propynoate and imides in the presence of triphenylphosphine in fairly high yields.

## Results and Discussion

Compound **5** may result from initial addition of triphenylphosphine (**1**) to ethyl 3-phenyl-2-propynoate (**2**) and concomitant protonation of the 1 : 1 adduct **3**, followed by the attack of the NH-acidic anion on the vinyltriphenylphosphonium cation to form the phosphorane **6** (Scheme 1) which undergoes intramolecular proton transfer leading to the formation of sterically congested *N*-vinyl imides **8** via the zwitterionic intermediate **7**. In this reaction triphenylphosphine acts as



**8a:** Z = CH<sub>2</sub>CH<sub>2</sub>; **8b:** Z = *o*-phenylene (C<sub>6</sub>H<sub>4</sub>); **8c:** Z = *cis*-4,5-cyclohexene (C<sub>6</sub>H<sub>8</sub>)

Scheme 1.

a catalyst. We reduced the amount of triphenylphosphine to 50 % molar ratio. In all cases where we have used triphenylphosphine as a catalyst in the range of 50 % to 100 % molar ratio, the reaction time amounted to 48 h. In all cases where we have used triphenylphosphine as a catalyst in a molar ratio below 50 %, the reaction time was longer than 48 h. In the absence of triphenylphosphine no product formation was observed. The NMR spectra indicated that solutions of compounds **8a–b** (CDCl<sub>3</sub> as solvent) contained only the *Z* isomer. However, in the case of **8c** the NMR spectra indicated that solutions of the compound (CDCl<sub>3</sub> as solvent) contained two isomers (*E* and *Z*). The relative percentages of rotamers of **8c** in CDCl<sub>3</sub> were determined from the <sup>1</sup>H NMR spectra. The structures **8a–c** were deduced from their IR, <sup>1</sup>H and <sup>13</sup>C NMR spectra.

#### Description of the crystal and molecular structure of **8c**

Crystals of **8c** are built up from molecules the structure of which is shown in Fig. 1. Table 1 contains a summary of the crystallographic data. Two well defined planes (with the atom C(4) being common) may be distinguished in the molecule of **8c**. These are ethyl (2*Z*)-2-amino-3-phenylacrylate [forming plane 1 with a r.m.s. deviation of fitted atoms of 0.085 Å] and the five-membered pyrrolidine-2,5-dione part of the tetrahydrophthalimide moiety [along with atom C(4); plane

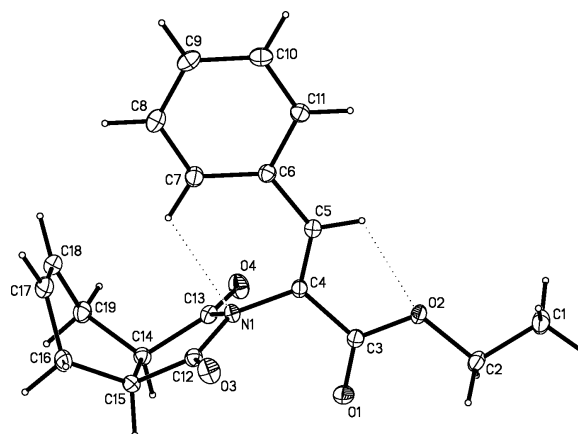


Fig. 1. The molecular structure of compound **8c** showing the atom numbering scheme and the intramolecular C(5)–H(5)···O(2) and C(7)–H(7)···N(1) contacts forming *S*(5) and *S*(6) motifs, respectively (dotted lines). Displacement ellipsoids are shown at the 50 % probability level.

2 with r.m.s. = 0.060 Å]. The atoms which deviate the most from these planes are C(7) [being displaced by 0.148(1) Å from plane 1] and N(1) [deviating by 0.138(1) Å from plane 2]. The dihedral angle between the least-squares plane 1 and plane 2 is 83.8(1)°, so the two planes are almost perpendicular to each other.

The molecule adopts a *Z* geometry with respect to the C(4)–C(5) double bond, which is reflected in the value of the torsion angle N(1)–C(4)–C(5)–C(6) of –0.2(2)° (Table 2). The carbonyl atom O(1) of the

Table 1. Crystal data and structure refinement details for **8c**.

<i>Crystal data:</i>	
Empirical formula	C <sub>19</sub> H <sub>19</sub> NO <sub>4</sub>
Formula weight [g mol <sup>-1</sup> ]	325.35
Crystal system, space group	monoclinic, <i>P</i> <sub>2</sub> <sub>1</sub> / <i>c</i>
<i>a</i> [Å]	10.004(3)
<i>b</i> [Å]	11.786(3)
<i>c</i> [Å]	14.197(3)
$\beta$ [deg]	106.57(3)
<i>V</i> [Å <sup>3</sup> ]	1604.4(7)
<i>Z</i>	4
<i>D</i> <sub>calc</sub> [g cm <sup>-3</sup> ]	1.347
$\mu$ [mm <sup>-1</sup> ]	0.095
<i>F</i> (000) [e]	688
Crystal size [mm <sup>3</sup> ]	0.46 × 0.40 × 0.28
Crystal color and form	colorless block
<i>Data collection:</i>	
Diffractometer	Kuma KM4CCD
Data collection method	$\omega$ scans
Monochromator	graphite
Radiation type, $\lambda$ [Å]	MoK $\alpha$ , 0.71073
<i>T</i> [K]	100(2)
$\theta$ Range [deg]	3.40–36.61
<i>h</i> , <i>k</i> , <i>l</i> Ranges	–16 ≤ <i>h</i> ≤ 13, –19 ≤ <i>k</i> ≤ 19, –23 ≤ <i>l</i> ≤ 23
Measured reflections	25999
Independent reflections	7391
Observed refl. [ <i>I</i> ≥ 2 $\sigma$ ( <i>I</i> )]	5775
Completeness to $\theta$ = 30.00°	0.991
<i>Refinement:</i>	
Refinement on	<i>F</i> <sup>2</sup>
Data/restraints/parameters	7391/0/293
<i>R</i> [ <i>F</i> <sub>o</sub> <sup>2</sup> ≤ 2 $\sigma$ ( <i>F</i> <sub>o</sub> <sup>2</sup> )]	<i>R</i> 1 = 0.0420, <i>wR</i> 2 = 0.1126
<i>R</i> (all data)	<i>R</i> 1 = 0.0576, <i>wR</i> 2 = 0.1179
Goof = <i>S</i>	1.070
Weighting parameter <i>a/b</i>	0.0715/0.0362
$\Delta\rho_{\max}/\Delta\rho_{\min}$ [e Å <sup>-3</sup> ]	0.56/–0.23

*R*1 =  $\Sigma||F_o| - |F_c||/\Sigma|F_o|$ ; *wR*2 =  $\sqrt{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]}$ ; Weighting scheme:  $w = 1/[\sigma^2(F_o^2) + (aP)^2 + bP]$  where  $P = (F_o^2 + 2F_c^2)/3$ .

ester group is in *antiperiplanar* conformation in relation to the vinyl atom C(5). The short intramolecular contacts C(5)⋯O(2) and C(7)⋯N(1) may indicate the presence of weak intramolecular hydrogen interactions C(5)–H(5)⋯O(2) and C(7)–H(7)⋯N(1), giving rise to five-membered *S*(5) and six-membered *S*(6) motifs, respectively. Both of them are formed in plane 1 and stabilize the molecular structure of **8c**. It is to note here, that a search of the Cambridge Structural Database [9] revealed fourteen hits for compounds of related structures, *i. e.* those bearing different substituents at nitrogen and different ester groups (usually –C(O)OMe and –C(O)OEt), with most of them existing in the *Z* rather than *E* isomeric form (only two hits for the latter). Most of the *Z* geometrical isomers also show some deviations from the planarity of the frag-

Table 2. Selected interatomic distances (Å), valence angles (deg) and torsion angles (deg) in **8c**.

O(1)–C(3)	1.2146(10)	N(1)–C(12)	1.3980(10)
O(2)–C(2)	1.4554(9)	N(1)–C(13)	1.4026(10)
O(2)–C(3)	1.3336(9)	C(3)–C(4)	1.4911(10)
O(3)–C(12)	1.2133(9)	C(4)–C(5)	1.3461(11)
O(4)–C(13)	1.2113(9)	C(5)–C(6)	1.4675(10)
N(1)–C(4)	1.4278(9)	C(17)–C(18)	1.3363(12)
C(12)–N(1)–C(4)	122.48(6)	C(19)–C(14)–C(15)	115.04(6)
C(13)–N(1)–C(4)	122.43(6)	C(12)–C(15)–C(14)	104.97(6)
C(12)–N(1)–C(13)	112.13(6)	C(12)–C(15)–C(16)	110.30(6)
C(4)–C(5)–C(6)	131.77(7)	C(14)–C(15)–C(16)	115.21(6)
C(13)–C(14)–C(15)	104.86(6)	C(18)–C(17)–C(16)	120.08(7)
C(13)–C(14)–C(19)	110.70(6)	C(17)–C(18)–C(19)	120.44(7)
C(3)–O(2)–C(2)–C(1)	179.8(1)		
C(2)–O(2)–C(3)–C(4)	176.1(1)		
C(12)–N(1)–C(4)–C(5)	–93.2(1)		
C(13)–N(1)–C(4)–C(5)	107.8(1)		
O(2)–C(3)–C(4)–C(5)	–4.5(1)		
O(2)–C(3)–C(4)–N(1)	177.3(1)		
N(1)–C(4)–C(5)–C(6)	–0.2(2)		
C(4)–C(5)–C(6)–C(7)	9.2(2)		
N(1)–C(12)–C(15)–C(16)	–118.4(1)		
N(1)–C(13)–C(14)–C(19)	121.2(1)		
C(19)–C(14)–C(15)–C(12)	–123.6(1)		
C(13)–C(14)–C(15)–C(16)	119.8(1)		
C(19)–C(14)–C(15)–C(16)	–2.0(1)		
C(16)–C(17)–C(18)–C(19)	–0.8(1)		

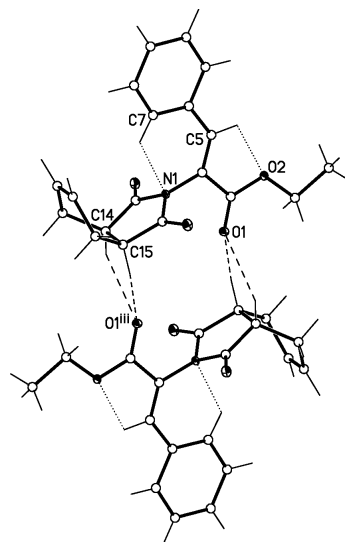


Fig. 2. The centrosymmetric molecular dimer formed by the adjacent molecules joined by C(14)–H(14)⋯O(1)<sup>iii</sup> and C(15)–H(15)⋯O(1)<sup>iii</sup> hydrogen bonds (dashed lines). Intramolecular C(5)–H(5)⋯O(2) and C(7)–H(7)⋯N(1) close contacts are shown with dotted lines. Symmetry codes are as given in Table 3.

ment defined here as plane 1, usually the phenyl group being slightly twisted. Most of the compounds, except

D–H...A	D–H (Å)	H...A (Å)	D...A (Å)	D–H...A (deg)	Offset (Å)
C(1)–H(1C)...O(3) <sup>i</sup>	0.99(2)	2.56(2)	3.424(2)	147(1)	–
C(5)–H(5)...O(2)	0.96(1)	2.28(1)	2.720(1)	107(1)	–
C(5)–H(5)...O(4) <sup>ii</sup>	0.96(1)	2.52(1)	3.398(1)	151(1)	–
C(7)–H(7)...N(1)	1.00(2)	2.52(2)	3.138(2)	120(1)	–
C(11)–H(11)...O(4) <sup>ii</sup>	0.97(1)	2.64(1)	3.514(2)	150(1)	–
C(14)–H(14)...O(1) <sup>iii</sup>	1.00(1)	2.51(1)	3.233(2)	130(1)	–
C(15)–H(15)...O(1) <sup>iii</sup>	0.99(1)	2.42(1)	3.202(2)	135(1)	–
C(19)–H(19A)...O(3) <sup>iv</sup>	0.98(1)	2.62(1)	3.574(2)	166(1)	–
C(2)–H(2A)...Cg(1) <sup>ii</sup>	0.99(1)	2.73(1)	3.541(2)	140(1)	0.21
C(2)–H(2B)...Cg(1) <sup>i</sup>	0.99(1)	2.83(2)	3.627(2)	139(1)	0.13

Table 3. Geometry of proposed hydrogen bonds and C–H...O/N/ $\pi$  close contacts for **8c** (Å, deg).

Symmetry codes: <sup>i</sup>  $-x+1, y-1/2, -z+3/2$ ; <sup>ii</sup>  $-x+1, -y+1, -z+1$ ; <sup>iii</sup>  $-x, -y+1, -z+1$ ; <sup>iv</sup>  $x, -y+3/2, z-1/2$ . Cg(1) is the centroid of the phenyl ring [C(6)–C(7)–C(8)–C(9)–C(10)–C(11)].

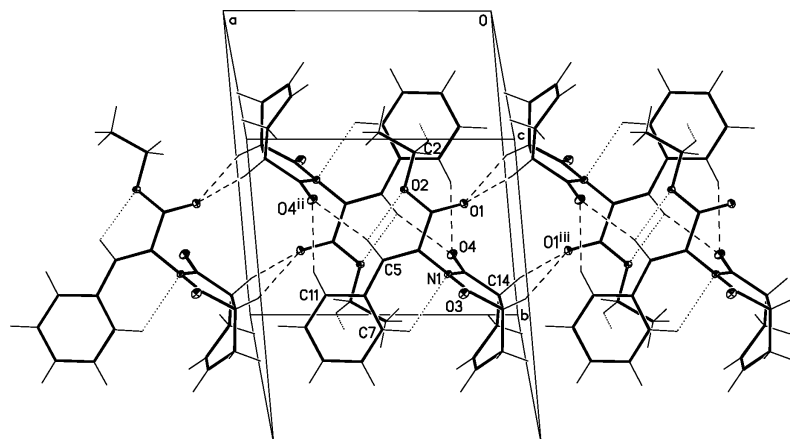


Fig. 3. The arrangement of the molecules within the ribbons parallel to *a* axis. Intermolecular C–H...O contacts are shown with dashed lines, and intramolecular C–H...O/N and intermolecular C–H... $\pi$  contacts – with dotted lines. Symmetry codes are given in Table 3.

for two metal complexes, also have the carbonyl O atom oriented *antiperiplanar*ly in relation to the vinylic C–H bond. Short C...O and C...N intramolecular contacts, similar to that observed in **8c**, may also be found in the compounds described so far, usually in those of them, which were shown to have the fragment defined as plane 1 close to planar.

The overall geometry of the tetrahydrophthalimide moiety in **8c** is identical to that observed in free tetrahydrophthalimide [10]. Atoms H(14) and H(15) at the fused five-membered ring and the partially hydrogenated six-membered ring junction have a mutual *cis* orientation. Cremer & Pople [11] puckering parameters for the cyclohexene ring of  $Q = 0.555(1)$  Å,  $\Theta = 89.3(1)^\circ$  and  $\Phi = 301.7(1)^\circ$  indicate a boat conformation. The dihedral angles between the least-squares plane through the atoms C(14)/C(15)/C(16)/C(19) and the planes defined by C(16)/C(17)/C(18)/C(19) and N(1)/C(12)/C(13)/C(14)/C(15) are  $41.8(1)^\circ$  and  $60.3(1)^\circ$ , respectively.

Atoms C(14) and C(15) of every two adjacent molecules are involved in bifurcated, three-centered C(14)–H(14)...O(1)<sup>iii</sup> and C(15)–H(15)...O(1)<sup>iii</sup> hydrogen bonds. That gives rise to centrosymmetric molecular dimers as it is shown in Fig. 2.

Adjacent, symmetry-related dimers are joined to each other by the hydrogen contacts of C–H...O and C–H... $\pi$ -type, the geometrical parameters of which are given in Table 3. First of all, adjacent dimers arranged along the *a* axis are linked by centrosymmetric three-centered, bifurcated contacts: C(5)–H(5)...O(4)<sup>ii</sup>, C(11)–H(11)...O(4)<sup>ii</sup> (dashed lines in Figs. 3 and 4) and by centrosymmetric C(2)–H(2A)... $\pi$ [Cg(1)<sup>ii</sup>] interactions (shown with dotted lines). That gives rise to ribbons parallel to the *a* axis (see Fig. 3). Every ribbon is joined to four adjacent ones by the interactions of C–H...O and C–H... $\pi$ -type: bifurcated C(1)–H(1C)...O(3)<sup>i</sup>, C(19)–H(19A)...O(3)<sup>iv</sup> contacts and C(2)–H(2B)... $\pi$ [Cg(1)<sup>i</sup>] interactions, which is easy to see in Fig. 4. The combination of all these C–H...O, C–H...N and C–H... $\pi$  interactions observed in the crystal of compound **8c** results in a three-dimensional network of hydrogen bonds.

## Conclusions

In conclusion, we have developed a convenient, one-pot regio- and stereoselective method for preparing sterically congested *N*-vinyl imides (**8a–c**) utilizing

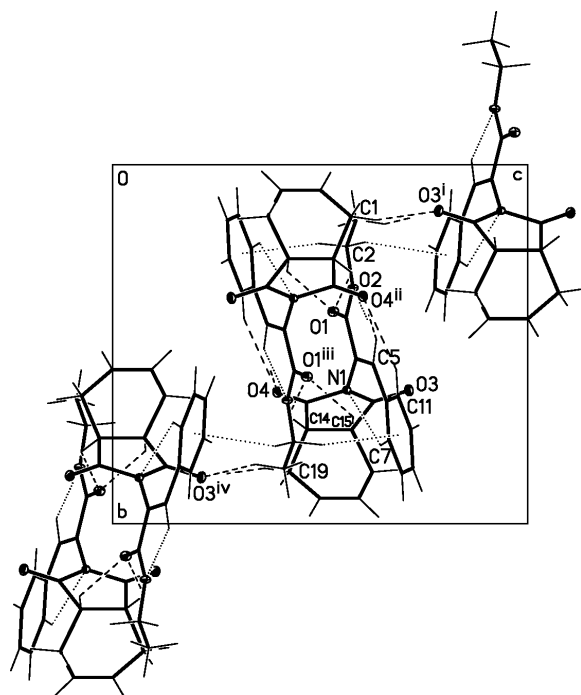


Fig. 4. The arrangement of the adjacent ribbons in the crystal lattice of **8c**, viewed down the ribbon and the *a* axis. Intermolecular C–H···O contacts are shown with dashed lines, and intramolecular C–H···O/N and intermolecular C–H··· $\pi$  contacts with dotted lines. Symmetry codes are given in Table 3.

the *in situ* generation of the phosphonium salts. Other aspects of this process are under investigation. The X-ray structure of **8c** (in its *Z* isomeric form) revealed that in the solid state the tetrahydrophthalimide moiety exists in a boat conformation and is almost perpendicular to the plane defined by the remaining atoms of the molecule. The crystal structure of **8c** is stabilized by extensive C–H···O/N/ $\pi$  interactions.

## Experimental Section

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. Elemental analyses were performed using a Heraeus CHN-O-Rapid analyzer. IR spectra were recorded on a Shimadzu IR-460 spectrometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were measured with a Bruker Spectrospin spectrometer at 250 and 62.5 MHz, respectively.

*Preparation of ethyl (Z)-2-(2,5-dioxo-2,5-dihydro-1-pyrrol-1-yl)-3-phenyl-2-propenoate (8a).*

### General procedure

To a magnetically stirred solution of triphenylphosphine (0.262 g, 1 mmol) and succinimide (0.099 g, 1 mmol) in

dichloromethane (5 mL) was added dropwise a mixture of ethyl 3-phenyl-2-propynoate (0.17 mL, 1 mmol) in dichloromethane (2 mL) at  $-10^\circ\text{C}$  over 15 min. The mixture was allowed to warm up to r.t. and stirred for 48 h at r.t. The solvent was removed under reduced pressure and the viscous residue was purified by flash column chromatography (silica gel; petroleum ether-ethyl acetate). The solvent was removed under reduced pressure to leave the product as a light yellow oil. Yield: 0.286 g (97 %). – IR (neat):  $\nu = 2984, 1723, 1653, 1407, 1269\text{ cm}^{-1}$ . –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 1.31$  (t, 3H,  $^3J = 7.0\text{ Hz}$ ,  $\text{CH}_3$ ), 2.73–2.87 (2 m, 2H, 2  $\text{CH}_3$ ), 4.25 (q, 2H,  $^3J = 7.0\text{ Hz}$ ,  $\text{OCH}_2$ ), 7.33 (m, 5H, arom), 8.02 (s, 1H, =CH). –  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 14.14$  ( $\text{CH}_3$ ), 28.72 (2  $\text{CH}_2$ ), 62.10 ( $\text{OCH}_2$ ), 121.07 (=CH), 128.94, 129.00 and 130.54 (5 CH), 132.13 and 142.10 (2C), 162.73 (C=O ester), 175.74 (2 C=O of imide). –  $\text{C}_{15}\text{H}_{15}\text{NO}_4$  (273.29): calcd. C 65.93, H 5.53, N 5.13; found C 65.75, H 5.40, N 5.04.

*Selected data for ethyl (Z)-2-(1,3-dioxo-1,3-dihydro-2H-isoindol-2-yl)-3-phenyl-2-propenoate 8b:* White crystals. Yield: 0.31 g (97 %); m. p.  $104.0\text{--}108.0^\circ\text{C}$ . – IR (KBr):  $\nu = 2930, 1715, 1653, 1407, 1276\text{ cm}^{-1}$ . –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 1.28$  (t, 3H,  $^3J = 7.0\text{ Hz}$ ,  $\text{CH}_3$ ), 4.27 (q, 2H,  $^3J = 7.0\text{ Hz}$ ,  $\text{OCH}_2$ ), 7.22–4.40 (m, 5H, arom), 7.74–7.91 (m, 4H, arom), 8.10 (s, 1H, =CH). –  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ):  $\delta = 14.17$  ( $\text{CH}_3$ ), 62.04 ( $\text{OCH}_2$ ), 120.39 (=CH), 123.99, 128.92, 129.38, 130.52 and 134.50 (9CH), 132.13, 132.39 and 142.72 (4C), 163.36 (C=O of ester), 169.86 (2 C=O of imide). –  $\text{C}_{19}\text{H}_{15}\text{NO}_4$  (321.33): calcd. C 71.02, H 4.70, N 4.36; found C 71.13, H 4.64, N 4.29.

*Selected data for ethyl 2-(1,3-dioxo-1,3,3a,4,7,7a-hexahydro-2H-isoindol-2-yl)-3-phenyl-2-propenoate 8c:* Colorless crystals. Yield: 0.32 g (97 %); m. p.  $96.0\text{--}100.0^\circ\text{C}$ . – IR (KBr):  $\nu = 2953, 1715, 1661, 1407, 1269\text{ cm}^{-1}$ . –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , %*E* = 33 and %*Z* = 67) for *Z*:  $\delta = 1.31$  (t, 3H,  $^3J = 7.0\text{ Hz}$ ,  $\text{CH}_3$ ), 2.28 and 2.63 (2m, 4H, 2  $\text{CH}_2$ ), 3.12 and 3.27 (2m, 2H, 2 CH), 4.26 (q, 2H,  $^3J = 7.0\text{ Hz}$ ,  $\text{OCH}_2$ ), 5.95 (m, 2H, HC=CH), 7.31 (m, 5H, arom), 7.98 (s, 1H, =CH). –  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ) for *Z*:  $\delta = 14.15$  ( $\text{CH}_3$ ), 23.34 (2 $\text{CH}_2$ ), 39.70 (2CH), 62.03 ( $\text{OCH}_2$ ), 120.99 (=CH), 127.23, 128.19, 128.61 and 129.66 (7CH), 131.93 and 142.10 (2C), 162.77 (C=O of ester), 178.60 (2C=O of imide). –  $^1\text{H}$  NMR ( $\text{CDCl}_3$ , *E* : *Z* = 33 : 67) for *E*:  $\delta = 1.25$  (t, 3H,  $^3J = 7\text{ Hz}$ ,  $\text{CH}_3$ ), 2.22 and 2.58 (2 m, 4H, 2 $\text{CH}_2$ ), 3.13 and 3.26 (2m, 2H, 2CH), 4.19 (q, 2H,  $^3J = 7\text{ Hz}$ ,  $\text{OCH}_2$ ), 5.91 (m, 2H, HC=CH), 7.24 (s, 1H, =CH), 7.30 (s, 5H, arom). –  $^{13}\text{C}$  NMR ( $\text{CDCl}_3$ ) for *E*:  $\delta = 23.09$  (2 $\text{CH}_2$ ), 39.51 (2CH), 62.92 ( $\text{OCH}_2$ ), 121.40 (=CH), 127.23, 128.85, 128.97 and 130.40 (7CH), 132.38 and 141.93 (2C), 162.77 (C=O of ester), 178.6 (2C=O of imide). –  $\text{C}_{19}\text{H}_{19}\text{NO}_4$  (325.36): calcd. C 70.14, H 5.89, N 4.30; found C 70.31, H 5.99, N 4.21.

*Preparation of single crystals of ethyl (Z)-2-(1,3-dioxo-1,3,3a,4,7,7a-hexahydro-2H-isoindol-2-yl)-3-phenyl-2-propenoate 8c:* Single crystals of the title compound were

prepared using the branch tube method [12] with light petroleum ether at 60 °C during 24 h. The colorless crystals were filtered off, washed with cold light petroleum ether (3 mL) and dried at r. t.

#### Crystal structure determination of **8c**

The crystallographic measurement was performed on a  $\kappa$ -geometry Kuma KM4CCD automated four-circle diffractometer with graphite-monochromatized  $\text{MoK}\alpha$  radiation. The data for the crystal were collected at 100(2) K using the Oxford Cryosystems cooler. A summary of the conditions for the data collection and the structure refinement parameters are given in Table 1. The data were corrected for Lorentz and polarization effects. Data collection, cell refinement, and data reduction and analysis were carried out with the KM4CCD software (Oxford Diffraction Poland): CrysAlis CCD and CrysAlis RED, respectively [13]. The structure was solved by Direct Methods using SHELXS-97 [14] and refined

by full-matrix least-squares techniques with anisotropic thermal parameters for non-H atoms using SHELXL-97 [15]. All H atoms were found in difference Fourier maps and were refined isotropically. All figures were made using the program XP [16]. Cremer & Pople [11] puckering parameters were calculated using the program PLATON [17].

#### Supplementary material

CCDC 635253 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif).

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- [1] O.I. Kolodiazny, *Phosphorus Ylides: Chemistry and Applications in Organic Chemistry*, Wiley, New York, **1999**.
- [2] W.C. Kaska, *Coord. Chem. Rev.* **1983**, 48, 1–58.
- [3] B.E. Maryanoff, A.B. Reitz, *Chem. Rev.* **1989**, 89, 863–927.
- [4] D.E.C. Cobridge, *Phosphorus: An Outline of Chemistry, Biochemistry and Uses*, 5th ed., Elsevier, Amsterdam, **1995**.
- [5] I. Yavari, A. Ramazani, *Synth. Commun.* **1996**, 26, 4495–4499.
- [6] I. Yavari, A. Ramazani, *Phosphorus, Sulphur, and Silicon* **1997**, 130, 73–77.
- [7] A. Ramazani, A. Bodaghi, *Tetrahedron Lett.* **2000**, 41, 567–568.
- [8] A. Ramazani, A.R. Kazemizadeh, E. Ahmadi, K. Ślepokura, T. Lis, *Z. Naturforsch.* **2006**, 61b, 1128–1133.
- [9] Cambridge Structural Database (CSD). F.H. Allen, *Acta Crystallogr.* **2002**, B58, 380–388.
- [10] A. Kirfel, *Acta Crystallogr.* **1976**, B32, 1556–1557.
- [11] D. Cremer, J.A. Pople, *J. Am. Chem. Soc.* **1975**, 97, 1354–1358.
- [12] A. Ramazani, L. Dolatyari, A.R. Kazemizadeh, E. Ahmadi, A.A. Torabi, R. Welter, *Z. Kristallogr. NCS* **2004**, 219, 181–183.
- [13] Kuma KM4CCD software: CrysAlis CCD and CrysAlis RED (ver. 1.171), Oxford Diffraction (Poland) **1995–2003**.
- [14] G.M. Sheldrick, SHELXS-97. Program for the solution of crystal structures. University of Göttingen, Göttingen (Germany) **1997**.
- [15] G.M. Sheldrick, SHELXL-97. Program for the refinement of crystal structures. University of Göttingen, Göttingen (Germany) **1997**.
- [16] XP – Interactive molecular graphics (ver. 5.1), Bruker Analytical X-ray Systems, Karlsruhe (Germany) **1998**.
- [17] A.L. Spek, PLATON, A. Multipurpose Crystallographic Tool, Utrecht University, Utrecht (The Netherlands) **2003**. See also: A.L. Spek, *J. Appl. Cryst.* **2003**, 36, 7–13.