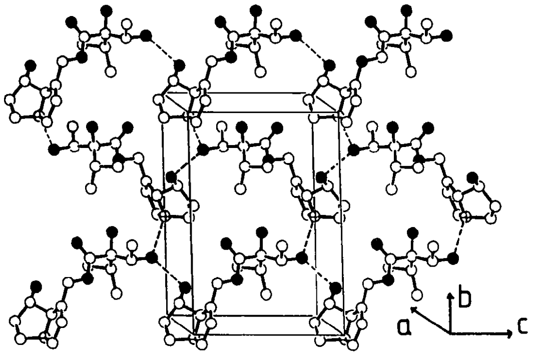

Fig. 2. The crystal packing.

interaction involving O(20) is bifurcated, this atom also being involved in an intermolecular hydrogen bond; the O(20)···O(19), H(20)···O(19) and O(20)—H(20) distances are 2·852 (5), 2·57 (7) and 0·93 (7) Å respectively, with the angle O(20)—H(20)···O(19) 98(5)°. For the other interaction, the O(19)···O(18), H(19)···O(18), O(19)—H(19) distances are 2·613 (5), 1·89 (7) and 1·17 (7) Å, with the angle O(19)—H(19)···O(18) 115(5)°.

The crystal packing is illustrated in Fig. 2. There are two unique intermolecular hydrogen-bonding interactions which link the molecules into a two-dimensional network, each molecule being bonded to four others. One interaction between molecules related by the twofold screw axis (bifurcated, see above) links the molecules into infinite spirals along the crystal *b* axis; the O(20)···N(4) ($1-x$, $\frac{1}{2}+y$, $1-z$), H(20)···N(4) distances are 2·730 (5) and 1·82 (7) Å respectively with the O(20)—H(2)···N(4) angle 166 (5)°. The other interaction between the hydroxyl substituent on the pyrrolizidine nucleus at C(7) and O(20) links the molecules along the *c* axis; O(21)···O(20), H(21)···O(20) and O(21)—H(21) have the respective values 2·733 (5), 1·77 and 0·98 Å, with the O(21)—H(21)···O(20) angle 167°. The molecular layers which are orientated parallel to the *bc* plane are held together in the crystal by van der Waals interactions.

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1-Oxo-2,3,7,7a-tetraphenyl-5-[α-(triphenylphosphonio)benzylidene]-1,7a-dihydro-5*H*-pyrrolizin-1-olate, $C_{56}H_{40}NO_2P$ (I), and 2,3,5,6,8-Pentaphenyl-1,7-indolizinedione, $C_{38}H_{25}NO_2$ (II)

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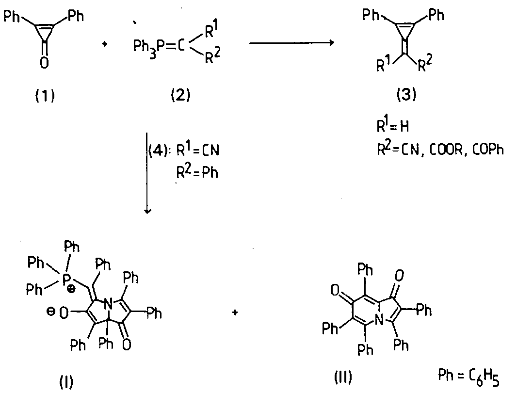
(*Received* 13 *October* 1987; *accepted* 14 *April* 1988)

Abstract. (I) $C_{56}H_{40}NO_2P$, $M_r = 789·9$, monoclinic, $P2_1/c$, $a = 21·857\ (6)$, $b = 10·349\ (5)$, $c = 19·294\ (6)$ Å, $\beta = 102·53\ (9)$°, $V = 4260\ (3)$ Å^3, $Z = 4$, $D_x = 1·232$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·10$ mm^{-1}, $F(000) = 1656$, $T = 291\ (1)$ K, final $R = 0·116$ for 3201 unique observed [$F \geq 2·0\sigma(F)$] diffractometer data. (II) $C_{38}H_{25}NO_2$, $M_r = 527·65$, orthorhombic, *Pbca*, $a = 11·906\ (7)$, $b = 23·706\ (9)$, $c =$

19·741 (9) Å, $V = 5572$ (5) Å^3, $Z = 8$, $D_x =$ 1·258 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu =$ 0·07 mm^{-1}, $F(000) = 2208$, $T = 291$ (1) K, final R = 0·081 for 2592 unique observed [$F \geq 4{\cdot}0\sigma(F)$] diffractometer data. The central part of molecule (I) consists of three five-membered heterocycles. The ring containing P is closed *via* a short P—O contact [2·132 (6) Å]. The coordination around P is a distorted trigonal bipyramid with C(phenyl) and O in the apical positions. In compound (II) the indole skeleton is nearly planar. The dihedral angle between the least-squares planes through the six-membered ring [maximum deviation from planarity 0·062 (6) Å] and the five-membered ring [0·351 (5) Å] is 5·0 (1)°.

Introduction. A long-known reaction of diphenylcyclopropenone (1) is the carbonyl olefination with triphenylmethylenephosphoranes (2) to give triafulvenes [*e.g.* (3)] in moderate yields. Interestingly, this reaction mode does not occur when (1) is reacted with the cyano and phenyl substituted phosphorus ylide (4). Instead, a yellow 2:1 adduct is formed [62% yield, m.p. 531 K (dec.)] accompanied by a red by-product [18% yield, m.p. 573 K (dec.)] of composition 2:1 $-Ph_3P$ (Sebold, 1983; Stegmann, 1983). From spectroscopic data and from chemical degradation experiments it was not possible to make a definitive structural assignment.



Experimental. Suitable crystals were obtained for (I) by recrystallization from dichloromethane/ethanol, for (II) by recrystallization from dichloromethane/acetonitrile. Crystal size ~ 0·08 × 0·55 × 0·49 mm (I), ~ 0·53 × 0·63 × 0·13 mm (II); $\omega/2\theta$ scan, scan speed 3·33° min^{-1} in θ (I), 1·1–3·33° min^{-1} in θ (II); Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$; lattice parameters from least-squares fit with 25 reflections up to $2\theta = 20{\cdot}4°$ (I), $2\theta = 21{\cdot}0°$ (II), five (I), three (II) standard reflections recorded every 2·0 h (I), 2·5 h (II), only random deviations; 5896 (I), 9479 (II) reflections measured $1{\cdot}0 \leq \theta \leq 22{\cdot}0°$ (I), $1{\cdot}5 \leq \theta \leq 23{\cdot}9°$ (II), $-22 \leq h \leq 22$, $0 \leq k \leq 10$, $0 \leq l \leq 20$ (I), $-13 \leq h \leq 13$, $0 \leq k \leq 27$, $0 \leq l \leq 22$ (II); after averaging [$R_{\rm int} = 0{\cdot}015$ (I), 0·023 (II)], 5197 (I), 4323 (II) unique reflections, 3201 with $F \geq 2{\cdot}0\sigma(F)$ (I), 2592 with $F \geq 4{\cdot}0\sigma(F)$ (II); Lorentz–polarization correction, no absorption correction (I), absorption correction *via* ψ scans, max./min. transmission 1·00/0·70 (II); systematic absences $h0l$, $l = 2n + 1$, $0k0$, $k = 2n + 1$ (I), $0kl$, $k = 2n + 1$, $h0l$, $l = 2n + 1$, $hk0$, $h = 2n + 1$ (II) conform to space groups $P2_1/c$ (I), $Pbca$ (II); structure solution *via* direct methods, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H

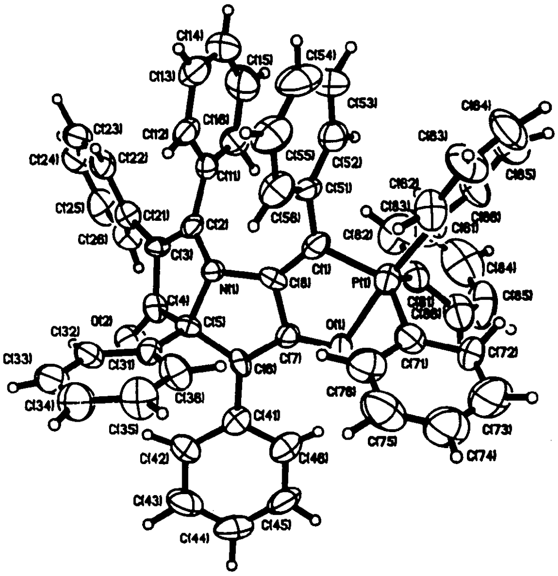


Fig. 1. General view of the molecule $C_{56}H_{40}NO_2P$ (I), showing the atom-numbering scheme.

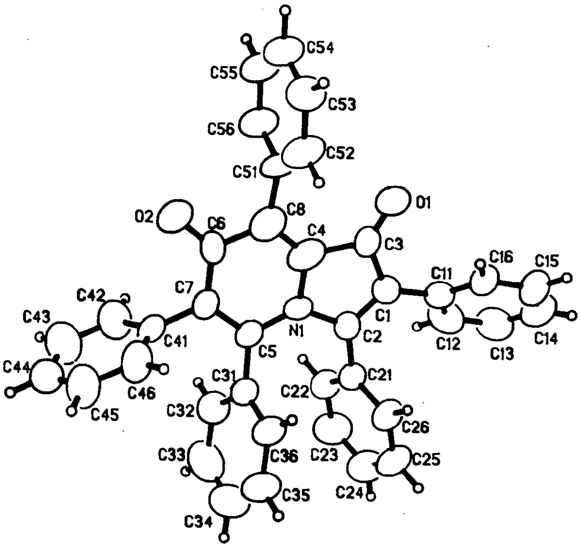


Fig. 2. General view of the molecule $C_{38}H_{25}NO_2$ (II), showing the atom-numbering scheme.

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for* $C_{56}H_{40}NO_2P$ (I) *and* $C_{38}H_{25}NO_2$ (II)

$$U_{eq} = \tfrac{1}{3}(\textstyle\sum_i U_{ii}).$$

	x	y	z	U_{eq}
(I)				
P(1)	6507 (1)	215 (3)	6180 (1)	47 (1)
O(1)	6677 (3)	1994 (6)	6746 (3)	50 (3)
O(2)	8969 (3)	4135 (6)	7836 (3)	50 (3)
N(1)	8257 (3)	1117 (6)	7299 (3)	28 (3)
C(1)	7332 (4)	−91 (10)	6536 (5)	41 (4)
C(2)	8747 (4)	1230 (9)	6927 (4)	33 (4)
C(3)	9089 (4)	2322 (8)	7099 (4)	30 (3)
C(4)	8801 (4)	3089 (9)	7579 (5)	37 (4)
C(5)	8273 (4)	2276 (8)	7765 (4)	27 (3)
C(6)	7617 (4)	2872 (8)	7523 (4)	33 (4)
C(7)	7257 (4)	2036 (9)	7065 (5)	36 (4)
C(8)	7632 (4)	925 (9)	6925 (4)	36 (4)
C(12)	9111 (3)	−965 (7)	6680 (3)	44 (4)
C(13)	9309	−1821	6215	61 (5)
C(14)	9266	−1471	5508	68 (5)
C(15)	9025	−265	5265	71 (5)
C(16)	8828	591	5731	48 (4)
C(11)	8870	241	6438	33 (4)
C(22)	10114 (3)	1843 (5)	6782 (3)	46 (4)
C(23)	10631	2249	6523	54 (5)
C(24)	10682	3537	6331	61 (5)
C(25)	10216	4421	6398	65 (5)
C(26)	9699	4015	6658	43 (4)
C(21)	9648	2727	6850	38 (4)
C(32)	8981 (2)	2147 (6)	8994 (3)	47 (4)
C(33)	9096	1716	9696	63 (5)
C(34)	8647	975	9931	72 (5)
C(35)	8084	665	9464	67 (5)
C(36)	7970	1096	8763	54 (4)
C(31)	8418	1837	8528	36 (4)
C(42)	7781 (2)	4639 (7)	8433 (3)	54 (4)
C(43)	7566	5754	8710	66 (5)
C(44)	6974	6243	8407	64 (5)
C(45)	6596	5617	7829	63 (5)
C(46)	6812	4503	7552	49 (4)
C(41)	7404	4013	7854	40 (4)
C(52)	7645 (3)	−1806 (7)	5772 (3)	51 (4)
C(53)	7857	−3057	5696	74 (6)
C(54)	8038	−3849	6291	83 (6)
C(55)	8006	−3388	6961	69 (5)
C(56)	7793	−2136	7038	59 (5)
C(51)	7613	−1345	6443	36 (4)
C(62)	6305 (3)	−2478 (8)	6095 (3)	59 (4)
C(63)	6141	−3651	5748	73 (6)
C(64)	5951	−3685	5010	84 (6)
C(65)	5923	−2545	4618	77 (6)
C(66)	6087	−1371	4965	62 (5)
C(61)	6277	−1338	5703	43 (4)
C(72)	5326 (3)	−196 (7)	6479 (3)	67 (5)
C(73)	4927	−501	6929	100 (7)
C(74)	5164	−607	7659	91 (7)
C(75)	5801	−407	7938	87 (6)
C(76)	6200	−101	7488	65 (5)
C(71)	5963	4	6758	49 (4)
C(82)	6769 (3)	1451 (7)	5029 (5)	78 (6)
C(83)	6624	2177	4405	94 (7)
C(84)	6043	2786	4209	109 (8)
C(85)	5606	2669	4637	90 (6)
C(86)	5751	1943	5260	67 (5)
C(81)	6333	1334	5456	49 (4)
(II)				
O(1)	9635 (3)	4400 (2)	−1206 (2)	68 (1)
O(2)	10373 (4)	6072 (2)	−3014 (2)	112 (2)
N(1)	8859 (3)	5856 (2)	−1258 (2)	48 (1)
C(1)	8495 (3)	5067 (2)	−615 (2)	48 (2)
C(2)	8287 (3)	5619 (2)	−619 (2)	46 (2)
C(3)	9249 (4)	4868 (2)	−1138 (2)	52 (2)
C(4)	9429 (4)	5409 (3)	−1577 (3)	68 (2)
C(5)	8919 (4)	6404 (2)	−1494 (2)	50 (2)
C(6)	9944 (5)	6027 (2)	−2455 (3)	66 (2)
C(7)	9463 (4)	6499 (2)	−2092 (2)	56 (2)
C(8)	9965 (5)	5465 (3)	−2143 (3)	76 (2)
C(12)	6882 (3)	4743 (1)	90 (2)	75 (2)
C(13)	6448	4411	612	85 (3)
C(14)	7141	4030	951	84 (3)
C(15)	8269	3981	770	76 (2)
C(16)	8704	4313	248	57 (2)
C(11)	8010	4694	−92	52 (2)
C(22)	6548 (3)	6176 (1)	−528 (1)	61 (2)
C(23)	5801	6463	−107	82 (2)
C(24)	6042	6526	580	87 (3)
C(25)	7030	6301	847	80 (2)
C(26)	7777	6013	426	62 (2)
C(21)	7536	5951	−261	48 (2)
C(32)	7633 (3)	7216 (2)	−1325 (2)	86 (3)
C(33)	7295	7689	−957	115 (4)
C(34)	7824	7821	−347	130 (4)
C(35)	8691	7481	−104	111 (3)
C(36)	9029	7008	−472	73 (2)
C(31)	8500	6875	−1082	58 (2)
C(42)	8919 (3)	7256 (2)	−2895 (2)	99 (3)
C(43)	9057	7797	−3160	120 (4)
C(44)	9894	8150	−2904	114 (4)
C(45)	10592	7961	−2383	122 (4)
C(46)	10453	7420	−2119	93 (3)
C(41)	9616	7068	−2375	59 (2)
C(52)	11638 (3)	4797 (2)	−2198 (2)	79 (2)
C(53)	12273	4405	−2561	83 (2)
C(54)	11874	4194	−3175	86 (3)
C(55)	10839	4374	−3426	83 (2)
C(56)	10204	4766	−3064	77 (2)
C(51)	10603	4977	−2450	68 (2)

atoms, which were placed in geometrically calculated positions (C—H 0·96 Å); phenyl groups were refined as rigid bodies (C—C 1·395, C—H 0·96 Å); refinement on F with 3201 (I), 2592 (II) reflections and 446 (I), 311 (II) refined parameters; $w = 1{\cdot}0/[\sigma^2(F) + 0{\cdot}0005F^2]$; $S = 1{\cdot}45$ (I), 2·33 (II); $R = 0{\cdot}116$ (I), 0·081 (II); $wR = 0{\cdot}091$ (I), 0·082 (II); $(\Delta/\sigma)_{\max} = 0{\cdot}2$ (I), 0·02 (II); no extinction correction; largest peak in final ΔF map ±0·4 (3) (I), 0·7 (3) (II) e Å^{-3}; atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974). The external form of the investigated crystals (I) and (II) indicated well shaped crystals; under the polarizing microscope the crystals appeared uniform and the crystal structures do not show signs of disorder. A possible cause for the rather high R values could be the fact that the crystals diffracted weakly. For compound (I) only 45% of the unique reflections have $F_o \geq 4\sigma(F_o)$. If one uses for compound (I) in the final refinement cycle only the reflections with $F_o \geq 4\sigma(F_o)$ the R value is 0·082 instead of 0·116. Programs: Enraf–Nonius *Structure Determination Package* (Frenz, 1985) for data reduction, *MULTAN* (Main *et al.*, 1980), *PARST* (Nardelli, 1983), *SHELXTL PLUS* (Sheldrick, 1987).

Discussion. The structures of the two title compounds are shown in Figs. 1 and 2. Positional parameters and equivalent values U_{eq} of the anisotropic temperature factors U_{ik} are given in Table 1* and bond lengths and angles in Table 2.

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* Lists of H-atom coordinates, anisotropic thermal parameters, structure factor amplitudes and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44970 (35 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Bond distance* (Å) *and angles* (°) *for* $C_{56}H_{40}NO_2P$ (I) *and* $C_{38}H_{25}NO_2$ (II)

(I)

P(1)—O(1)	2·132 (6)	C(1)—C(51)	1·463 (12)
P(1)—C(1)	1·814 (8)	C(2)—C(3)	1·356 (11)
P(1)—C(61)	1·866 (8)	C(2)—C(11)	1·456 (11)
P(1)—C(71)	1·811 (8)	C(3)—C(4)	1·462 (11)
P(1)—C(81)	1·791 (9)	C(3)—C(21)	1·470 (10)
O(1)—C(7)	1·283 (9)	C(4)—C(5)	1·530 (11)
O(2)—C(4)	1·214 (9)	C(5)—C(6)	1·538 (10)
N(1)—C(2)	1·416 (9)	C(5)—C(31)	1·508 (10)
N(1)—C(5)	1·494 (9)	C(6)—C(7)	1·360 (11)
N(1)—C(8)	1·415 (10)	C(6)—C(41)	1·466 (10)
C(1)—C(8)	1·372 (11)	C(7)—C(8)	1·471 (11)

C(1)—P(1)—O(1)	84·7 (4)	C(31)—C(5)—N(1)	108·5 (6)
C(61)—P(1)—O(1)	174·3 (3)	C(31)—C(5)—C(4)	112·6 (6)
C(61)—P(1)—C(1)	100·1 (4)	C(31)—C(5)—C(6)	113·6 (6)
C(71)—P(1)—O(1)	81·6 (3)	C(7)—C(6)—C(5)	108·2 (7)
C(71)—P(1)—C(1)	118·5 (3)	C(41)—C(6)—C(5)	123·7 (6)
C(71)—P(1)—C(61)	93·3 (3)	C(41)—C(6)—C(7)	127·0 (7)
C(81)—P(1)—O(1)	79·9 (3)	C(6)—C(7)—O(1)	134·9 (9)
C(81)—P(1)—C(1)	115·7 (4)	C(8)—C(7)—O(1)	114·7 (8)
C(81)—P(1)—C(61)	100·6 (3)	C(8)—C(7)—C(6)	110·4 (8)
C(81)—P(1)—C(71)	120·1 (3)	C(1)—C(8)—N(1)	132·9 (8)
C(7)—O(1)—P(1)	108·6 (6)	C(7)—C(8)—N(1)	108·2 (8)
C(5)—N(1)—C(2)	108·5 (6)	C(7)—C(8)—C(1)	118·7 (8)
C(8)—N(1)—C(2)	120·3 (7)	C(12)—C(11)—C(2)	121·4 (4)
C(8)—N(1)—C(5)	108·3 (6)	C(16)—C(11)—C(2)	118·0 (4)
C(8)—C(1)—P(1)	113·0 (7)	C(22)—C(21)—C(3)	121·3 (4)
C(51)—C(1)—P(1)	121·1 (7)	C(26)—C(21)—C(3)	118·7 (4)
C(51)—C(1)—C(8)	125·7 (7)	C(32)—C(31)—C(5)	122·0 (4)
C(3)—C(2)—N(1)	112·4 (8)	C(36)—C(31)—C(5)	118·0 (4)
C(11)—C(2)—N(1)	122·5 (7)	C(42)—C(41)—C(6)	122·3 (4)
C(11)—C(2)—C(3)	125·1 (7)	C(46)—C(41)—C(6)	117·7 (4)
C(4)—C(3)—C(2)	108·5 (8)	C(52)—C(51)—C(1)	121·6 (4)
C(21)—C(3)—C(2)	127·7 (7)	C(56)—C(51)—C(1)	118·1 (4)
C(21)—C(3)—C(4)	123·8 (7)	C(62)—C(61)—P(1)	119·1 (2)
C(3)—C(4)—O(2)	127·4 (8)	C(66)—C(61)—P(1)	120·9 (2)
C(5)—C(4)—O(2)	124·9 (8)	C(72)—C(71)—P(1)	120·9 (2)
C(5)—C(4)—C(3)	107·4 (8)	C(76)—C(71)—P(1)	118·7 (2)
C(4)—C(5)—N(1)	102·8 (6)	C(82)—C(81)—P(1)	117·4 (2)
C(6)—C(5)—N(1)	104·0 (6)	C(86)—C(81)—P(1)	122·1 (2)
C(6)—C(5)—C(4)	114·2 (7)		

(II)

O(1)—C(3)	1·209 (5)	C(3)—C(4)	1·562 (7)
O(2)—C(6)	1·221 (6)	C(4)—C(8)	1·295 (7)
N(1)—C(2)	1·424 (5)	C(5)—C(7)	1·366 (6)
N(1)—C(4)	1·407 (6)	C(5)—C(31)	1·468 (6)
N(1)—C(5)	1·384 (5)	C(6)—C(7)	1·445 (7)
C(1)—C(2)	1·340 (6)	C(6)—C(8)	1·468 (7)
C(1)—C(3)	1·448 (6)	C(7)—C(41)	1·470 (6)
C(1)—C(11)	1·478 (6)	C(8)—C(51)	1·511 (7)
C(2)—C(21)	1·463 (6)		

C(4)—N(1)—C(2)	106·6 (4)	C(8)—C(6)—O(2)	116·7 (5)
C(5)—N(1)—C(2)	131·3 (4)	C(8)—C(6)—C(7)	120·1 (5)
C(5)—N(1)—C(4)	122·2 (4)	C(6)—C(7)—C(5)	119·3 (4)
C(3)—C(1)—C(2)	110·6 (4)	C(41)—C(7)—C(5)	122·5 (4)
C(11)—C(1)—C(2)	126·3 (4)	C(41)—C(7)—C(6)	118·2 (4)
C(11)—C(1)—C(3)	123·0 (4)	C(6)—C(8)—C(4)	116·6 (6)
C(1)—C(2)—N(1)	112·6 (4)	C(51)—C(8)—C(4)	121·0 (5)
C(21)—C(2)—N(1)	122·5 (4)	C(51)—C(8)—C(6)	122·4 (5)
C(21)—C(2)—C(1)	124·9 (4)	C(12)—C(11)—C(1)	120·4 (2)
C(1)—C(3)—O(1)	127·9 (4)	C(16)—C(11)—C(1)	119·5 (2)
C(4)—C(3)—O(1)	129·8 (5)	C(22)—C(21)—C(2)	120·1 (2)
C(4)—C(3)—C(1)	102·3 (4)	C(26)—C(21)—C(2)	119·8 (2)
C(3)—C(4)—N(1)	107·7 (4)	C(32)—C(31)—C(5)	120·1 (2)
C(8)—C(4)—N(1)	123·1 (6)	C(36)—C(31)—C(5)	119·7 (2)
C(8)—C(4)—C(3)	129·1 (5)	C(42)—C(41)—C(7)	120·0 (2)
C(7)—C(5)—N(1)	118·1 (4)	C(46)—C(41)—C(7)	120·0 (2)
C(31)—C(5)—N(1)	120·7 (4)	C(52)—C(51)—C(8)	122·4 (2)
C(31)—C(5)—C(7)	121·0 (4)	C(56)—C(51)—C(8)	116·9 (2)
C(7)—C(6)—O(2)	123·1 (5)		

The X-ray analyses show that completely new – and hitherto unexpected – product types of cyclopropenone reactions were obtained which are under intensive current investigations.

The central part of molecule (I) consists of three five-membered heterocycles: (*A*) N(1), C(2), C(3), C(4), C(5), (*B*) N(1), C(5), C(6), C(7), C(8) and (*C*) C(1), C(8), C(7), O(1), P(1) with maximum deviations from the corresponding least-squares planes of (*A*) 0·044 (9), (*B*) 0·058 (8), (*C*) 0·033 (9) Å. The dihedral angles between the least-squares planes of the three heterocycles are $\angle(A,B)$ 52·9 (3), $\angle(A,C)$ 51·3 (3), and $\angle(B,C)$ 6·0 (2)°. In ring (*C*) the P(1)—O(1) distance, which is 0·342 Å longer than the sum of the covalent radii (Pauling, 1970), clearly indicates a bond relation. The coordination around P(1) is a distorted trigonal bipyramid with O(1) and C(61) in the apical positions. The apical P(1)—C(61) bond distance is significantly longer than the three P—C distances in the equatorial positions. Through the P(1)—O(1) bond relation the C(7)—O(1) bond is weakened and the bond distance is longer than a double bond. In the three planar rings (*A*), (*B*) and (*C*) the bond distances C(2)—C(3), C(6)—C(7) and C(8)—C(1) indicate double-bond character.

In compound (II) the indole skeleton is nearly planar. The dihedral angle between the least-squares planes through the six-membered ring [maximum deviation from planarity 0·062 (6) Å] and the five-membered ring [0·031 (5) Å] is 5·0 (1)°. The planes of the three phenyl groups bound to the six-membered ring form dihedral angles between 97·8 (1) and 114·4 (1)° with this ring and the planes of the remaining phenyl groups form angles of 44·1 (1) and 116·7 (1)° with the five-membered ring. Through the double bonds C(3)—O(1) and C(6)—O(2) the adjacent C—C bond lengths are lengthened towards the single-bond length. The remaining bond lengths in the two central rings indicate double-bond character.

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