C13D	0.5429 (4)	−0.4051 (4)	0.2482 (3)	5.5 (1)*
C17D	0.8093 (4)	−0.6002 (5)	0.3989 (3)	6.6 (1)*
C18D	0.6340 (3)	−0.4828 (4)	0.3938 (3)	4.5 (1)
C20D	0.6359 (4)	−0.6128 (4)	0.2977 (3)	5.5 (1)
C22D	0.6789 (3)	−0.4632 (5)	0.1822 (3)	5.0 (1)
C24D	0.8097 (4)	−0.4785 (5)	0.1149 (3)	6.6 (1)

Table 2. *Selected geometric parameters (Å, °) for molecule 1 and selected torsion angles (°)*

O15A—N14A	1.225 (6)	C3A—C4A	1.396 (6)
O16A—N14A	1.207 (5)	C4A—C5A	1.411 (5)
O23A—C22A	1.399 (6)	C5A—C6A	1.389 (6)
O23A—C24A	1.409 (6)	C5A—C7A	1.518 (6)
N10A—C4A	1.357 (5)	C7A—C8A	1.544 (6)
N10A—C9A	1.460 (6)	C7A—C17A	1.503 (6)
N10A—C11A	1.446 (6)	C8A—C9A	1.558 (6)
N14A—C1A	1.450 (6)	C8A—C18A	1.480 (6)
N19A—C18A	1.135 (6)	C8A—C20A	1.463 (7)
N21A—C20A	1.130 (7)	C9A—C13A	1.526 (6)
C1A—C2A	1.374 (6)	C9A—C22A	1.529 (6)
C1A—C6A	1.386 (7)	C11A—C12A	1.515 (8)
C2A—C3A	1.370 (6)	C12A—C13A	1.514 (8)

C22A—O23A—C24A	111.7 (4)	C5A—C7A—C17A	114.1 (3)
C4A—N10A—C9A	122.3 (3)	C8A—C7A—C17A	112.1 (4)
C4A—N10A—C11A	124.4 (4)	C7A—C8A—C9A	110.9 (3)
C9A—N10A—C11A	112.1 (3)	C7A—C8A—C18A	108.5 (3)
O15A—N14A—O16A	121.8 (4)	C7A—C8A—C20A	111.1 (4)
O15A—N14A—C1A	118.6 (5)	C9A—C8A—C18A	108.9 (4)
O16A—N14A—C1A	119.6 (4)	C9A—C8A—C20A	109.4 (3)
N14A—C1A—C2A	119.5 (4)	C18A—C8A—C20A	108.0 (4)
N14A—C1A—C6A	118.3 (5)	N10A—C9A—C8A	106.9 (3)
C2A—C1A—C6A	122.1 (4)	N10A—C9A—C13A	103.7 (3)
C1A—C2A—C3A	118.7 (5)	N10A—C9A—C22A	111.5 (4)
C2A—C3A—C4A	121.3 (4)	C8A—C9A—C13A	113.6 (4)
N10A—C4A—C3A	119.5 (4)	C8A—C9A—C22A	112.4 (3)
N10A—C4A—C5A	121.2 (4)	C13A—C9A—C22A	108.5 (3)
C3A—C4A—C5A	119.3 (3)	N10A—C11A—C12A	103.8 (4)
C4A—C5A—C6A	119.1 (4)	C11A—C12A—C13A	103.7 (4)
C4A—C5A—C7A	121.6 (3)	C9A—C13A—C12A	103.1 (4)
C6A—C5A—C7A	119.3 (4)	N19A—C18A—C8A	177.6 (5)
C1A—C6A—C5A	119.4 (4)	N21A—C20A—C8A	177.2 (5)
C5A—C7A—C8A	107.8 (3)	O23A—C22A—C9A	111.7 (3)

	Molecule 1	Molecule 2	Molecule 3	Molecule 4
C24—O23—C22—C9	179.1 (4)	172.4 (5)	−153.1 (5)	173.4 (5)
N10—C9—C22—O23	42.9 (5)	171.7 (4)	48.9 (6)	40.4 (6)
C8—C9—C22—O23	−77.1 (5)	54.3 (5)	−72.0 (6)	−80.5 (5)
C13—C9—C22—O23	156.4 (4)	−74.4 (5)	161.9 (4)	152.2 (4)

The O, N and C atoms of the cyano and methoxymethyl groups were refined with anisotropic displacement parameters. The atoms of the phenyl ring in molecule 4 were also refined anisotropically in view of their rather large thermal motion. H atoms were placed at calculated positions (C—H 0.95 Å) and treated as riding atoms.

The structure was solved using *MULTAN* (Germain, Main & Woolfson, 1971) and refined by full-matrix least squares. Weights for each reflection in the refinement were $w = 1/\sigma^2(F_o) = 4F_o^2/\sigma(F_o^2)$, $\sigma(F_o^2) = \sigma^2(I) + (pF_o^2)^2$; the value of the instability factor p was determined to be 0.03. All calculations were performed using *SDP* (B. A. Frenz & Associates Inc., 1983). Data collection used *CAD*-4 *EXPRESS* (Enraf–Nonius, 1992).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: AB1224). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Dimethyl(2-{[2,3-di(*p*-tolyl)-5-methyl-1-indenylidene](*p*-tolyl)methyl}-5-methoxyphenylmethyl)amine†

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Abstract

In the title compound, $C_{42}H_{41}NO$, all the phenyl rings are twisted out of the plane of the indenylidene ring. The molecules are held together in the crystal by van der Waals interactions.

Comment

It has been reported that the reaction between cyclopalladated *N*,*N*-dimethylbenzylamine bis(acetonitrile)tetrafluoroborate and diphenylacetylene gives a polycyclic compound as its main product (Tao, Sil-

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verberg, Rheingold & Heck, 1989). It is well known, however, that some reactions of organometallic compounds having acetylene ligands give reaction products whose structure and/or conformation may depend on the type of acetylene involved (Maassarani, Pfeffer & Le Borgne, 1987; Dupont & Pfeffer, 1988). We are interested in the organometallic chemistry of transition elements with acetylene ligands and carried out the reaction between cyclopalladated N,N-dimethyl-5-methoxy-benzylamine bis(acetonitrile) hexafluorophosphate and di(p-tolyl)acetylene. In order to contrast the type of products obtained, we compared this reaction with that described by Tao et al. (1989). The title compound, (I), was isolated from the four products obtained.

(I)
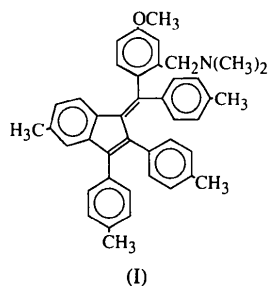
Bond distances and angles within the molecule are quite regular, with an average C—C distance of 1.382 (5) Å within the four phenyl rings. The rings are planar with a maximum deviation of 0.024 Å. The molecule is well ordered, with methyl groups C36, C37, C40 and C42 having relatively large thermal motion. The interplanar angles between the indenylidene ring,
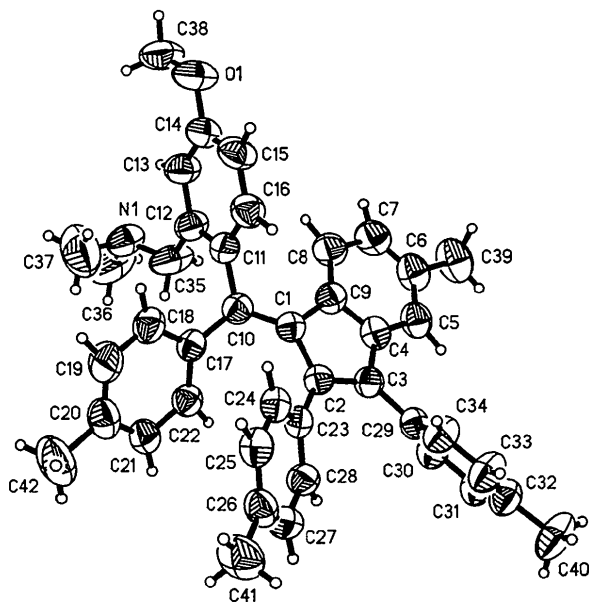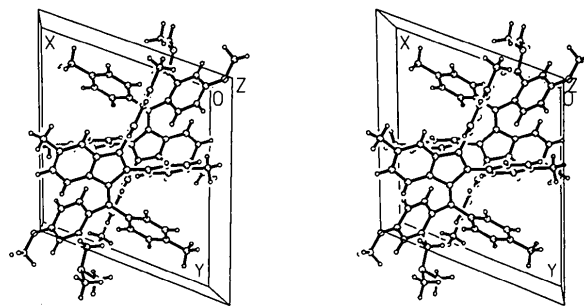

Fig. 2. A stereoview of the molecular packing in the unit cell viewed down the c axis.

C1–C9, and the phenyl rings C11–C16, C17–C22, C23–C28 and C29–C34 are 87.5 (1), 42.4 (1), 42.8 (1) and 61.4 (1)°, respectively. In the crystal, the molecules of the title compound are held together by van der Waals interactions. A C—H···N intermolecular contact is also present: C(7)···N(1)(−x, −y, −z) 3.473 (5) Å.

Experimental

The title compound was recrystallized from methanol/dichloromethane solution.

Crystal data

$C_{42}H_{41}NO$	Cu $K\alpha$ radiation
$M_r = 575.76$	$\lambda = 1.54178$ Å
Triclinic	Cell parameters from 23
$P\bar{1}$	reflections
$a = 12.3228$ (13) Å	$\theta = 35–40°$
$b = 13.0782$ (6) Å	$\mu = 0.511$ mm^{-1}
$c = 11.6694$ (12) Å	$T = 293$ (2) K
$\alpha = 98.505$ (6)°	Chunk
$\beta = 105.878$ (8)°	$0.40 \times 0.35 \times 0.35$ mm
$\gamma = 106.988$ (6)°	Orange
$V = 1676.1$ (3) Å^3	
$Z = 2$	
$D_x = 1.141$ Mg m^{-3}	

Data collection

Rigaku AFC-5R diffractometer	$\theta_{max} = 60.12°$
ω scans	$h = 0 \rightarrow 13$
Absorption correction:	$k = -14 \rightarrow 14$
none	$l = -13 \rightarrow 12$
5258 measured reflections	2 standard reflections
4987 independent reflections	monitored every 100
3754 observed reflections	reflections
[$I > 2\sigma(I)$]	frequency: 60 min
$R_{int} = 0.0234$	intensity decay: 3%

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = -0.029$
$R(F) = 0.0652$	$\Delta\rho_{max} = 0.686$ e Å^{-3}
$wR(F^2) = 0.2186$	$\Delta\rho_{min} = -0.242$ e Å^{-3}

Fig. 1. The molecular structure of the title compound showing the atom-labelling scheme and 50% probability displacement ellipsoids.

$S = 1.106$
4935 reflections
397 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 1.2987P]$
where $P = (F_o^2 + 2F_c^2)/3$

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
O1	−0.0414 (2)	−0.0419 (2)	0.3312 (3)	0.0932 (9)
N1	0.2205 (3)	−0.1122 (2)	0.0815 (3)	0.0821 (9)
C1	0.3758 (3)	0.3272 (2)	0.2352 (3)	0.0532 (7)
C2	0.4756 (3)	0.4324 (2)	0.2563 (3)	0.0515 (7)
C3	0.4330 (3)	0.4959 (2)	0.1860 (3)	0.0536 (7)
C4	0.3055 (3)	0.4363 (3)	0.1140 (3)	0.0573 (8)
C5	0.2274 (3)	0.4661 (3)	0.0269 (3)	0.0675 (9)
C6	0.1083 (3)	0.3970 (3)	−0.0284 (3)	0.0736 (10)
C7	0.0705 (3)	0.3013 (3)	0.0087 (4)	0.0780 (11)
C8	0.1478 (3)	0.2697 (3)	0.0963 (3)	0.0704 (9)
C9	0.2681 (3)	0.3381 (3)	0.1495 (3)	0.0582 (8)
C10	0.3793 (3)	0.2353 (2)	0.2763 (3)	0.0546 (7)
C11	0.2655 (3)	0.1559 (3)	0.2815 (3)	0.0579 (8)
C12	0.2087 (3)	0.0477 (3)	0.2103 (3)	0.0652 (9)
C13	0.1047 (3)	−0.0186 (3)	0.2252 (4)	0.0727 (10)
C14	0.0587 (3)	0.0196 (3)	0.3104 (3)	0.0696 (9)
C15	0.1155 (3)	0.1262 (3)	0.3820 (4)	0.0745 (10)
C16	0.2168 (3)	0.1933 (3)	0.3663 (3)	0.0672 (9)
C17	0.4902 (3)	0.2116 (2)	0.3262 (3)	0.0537 (7)
C18	0.5035 (3)	0.1591 (3)	0.4217 (3)	0.0681 (9)
C19	0.6100 (4)	0.1421 (3)	0.4738 (4)	0.0827 (11)
C20	0.7054 (4)	0.1748 (3)	0.4322 (4)	0.0784 (10)
C21	0.6912 (3)	0.2229 (3)	0.3339 (3)	0.0660 (9)
C22	0.5850 (3)	0.2398 (3)	0.2807 (3)	0.0575 (8)
C23	0.5969 (3)	0.4708 (2)	0.3502 (3)	0.0515 (7)
C24	0.6127 (3)	0.4577 (2)	0.4689 (3)	0.0555 (8)
C25	0.7260 (3)	0.4959 (3)	0.5570 (3)	0.0644 (9)
C26	0.8275 (3)	0.5483 (3)	0.5304 (3)	0.0674 (9)
C27	0.8119 (3)	0.5621 (3)	0.4125 (3)	0.0684 (9)
C28	0.6989 (3)	0.5238 (3)	0.3238 (3)	0.0607 (8)
C29	0.4936 (3)	0.6110 (3)	0.1853 (3)	0.0535 (7)
C30	0.5042 (3)	0.6395 (3)	0.0781 (3)	0.0675 (9)
C31	0.5557 (4)	0.7471 (3)	0.0757 (3)	0.0722 (10)
C32	0.5953 (4)	0.8313 (3)	0.1788 (4)	0.0722 (10)
C33	0.5873 (4)	0.8036 (3)	0.2868 (4)	0.0796 (11)
C34	0.5375 (3)	0.6957 (3)	0.2894 (3)	0.0682 (9)
C35	0.2501 (4)	0.0025 (3)	0.1093 (4)	0.0919 (13)
C36	0.2393 (6)	−0.1609 (4)	−0.0239 (5)	0.133 (2)
C37	0.3025 (6)	−0.1372 (6)	0.1875 (6)	0.153 (2)
C38	−0.0993 (4)	−0.1535 (4)	0.2632 (4)	0.0983 (14)
C39	0.0236 (4)	0.4259 (4)	−0.1286 (4)	0.0992 (14)
C40	0.6478 (5)	0.9489 (3)	0.1752 (5)	0.111 (2)
C41	0.9509 (4)	0.5919 (4)	0.6277 (4)	0.0982 (14)
C42	0.8223 (4)	0.1585 (5)	0.4927 (5)	0.124 (2)

Table 2. *Selected geometric parameters (Å, °)*

O1—C14	1.368 (4)	C13—C14	1.374 (5)
O1—C38	1.418 (4)	C14—C15	1.378 (5)
N1—C35	1.400 (4)	C15—C16	1.376 (5)
N1—C36	1.408 (5)	C17—C22	1.388 (4)
N1—C37	1.512 (6)	C17—C18	1.389 (4)
C1—C10	1.367 (4)	C18—C19	1.384 (5)
C1—C9	1.485 (4)	C19—C20	1.371 (6)
C1—C2	1.486 (4)	C20—C21	1.382 (5)
C2—C3	1.363 (4)	C20—C42	1.513 (4)
C2—C23	1.480 (4)	C21—C22	1.383 (5)
C3—C4	1.466 (4)	C23—C28	1.386 (4)
C3—C29	1.474 (3)	C23—C24	1.388 (4)
C4—C5	1.382 (4)	C24—C25	1.382 (5)
C4—C9	1.397 (5)	C25—C26	1.381 (5)
C5—C6	1.386 (5)	C26—C27	1.382 (5)

C6—C7	1.379 (5)	C26—C41	1.511 (5)
C6—C39	1.515 (4)	C27—C28	1.383 (5)
C7—C8	1.395 (5)	C29—C34	1.374 (4)
C8—C9	1.394 (5)	C29—C30	1.386 (4)
C10—C17	1.472 (4)	C30—C31	1.372 (5)
C10—C11	1.504 (4)	C31—C32	1.369 (5)
C11—C16	1.390 (5)	C32—C33	1.382 (5)
C11—C12	1.392 (5)	C32—C40	1.496 (5)
C12—C13	1.393 (5)	C33—C34	1.373 (5)
C12—C35	1.517 (4)		
C14—O1—C38	117.8 (3)	O1—C14—C15	116.1 (3)
C35—N1—C36	117.1 (3)	C13—C14—C15	119.5 (3)
C35—N1—C37	106.4 (3)	C16—C15—C14	119.5 (3)
C36—N1—C37	105.0 (4)	C15—C16—C11	121.9 (3)
C10—C1—C9	125.4 (3)	C22—C17—C18	117.2 (3)
C10—C1—C2	129.3 (3)	C22—C17—C10	122.1 (3)
C9—C1—C2	105.1 (3)	C18—C17—C10	120.7 (3)
C3—C2—C23	124.1 (3)	C19—C18—C17	121.0 (3)
C3—C2—C1	108.9 (3)	C20—C19—C18	121.4 (3)
C23—C2—C1	126.4 (2)	C19—C20—C21	118.0 (3)
C2—C3—C4	109.4 (3)	C19—C20—C42	120.7 (3)
C2—C3—C29	129.0 (3)	C21—C20—C42	121.4 (3)
C4—C3—C29	121.3 (2)	C20—C21—C22	121.1 (3)
C5—C4—C9	122.0 (3)	C21—C22—C17	121.1 (3)
C5—C4—C3	129.8 (3)	C28—C23—C24	117.5 (3)
C9—C4—C3	108.2 (3)	C28—C23—C2	121.1 (2)
C4—C5—C6	119.8 (3)	C24—C23—C2	121.4 (3)
C7—C6—C5	118.3 (3)	C25—C24—C23	121.1 (3)
C7—C6—C39	121.4 (3)	C26—C25—C24	121.3 (3)
C5—C6—C39	120.3 (3)	C25—C26—C27	117.6 (3)
C6—C7—C8	122.8 (3)	C25—C26—C41	121.1 (4)
C9—C8—C7	118.7 (3)	C27—C26—C41	121.2 (4)
C8—C9—C4	118.4 (3)	C26—C27—C28	121.3 (3)
C8—C9—C1	133.5 (3)	C27—C28—C23	121.1 (3)
C4—C9—C1	107.9 (3)	C34—C29—C30	116.8 (3)
C1—C10—C17	124.3 (3)	C34—C29—C3	121.8 (3)
C1—C10—C11	119.3 (3)	C30—C29—C3	121.4 (3)
C17—C10—C11	116.2 (3)	C31—C30—C29	121.7 (3)
C16—C11—C12	118.5 (3)	C32—C31—C30	121.1 (3)
C16—C11—C10	117.0 (3)	C31—C32—C33	117.6 (3)
C12—C11—C10	124.5 (3)	C31—C32—C40	121.1 (3)
C11—C12—C13	119.1 (3)	C33—C32—C40	121.3 (4)
C11—C12—C35	122.1 (3)	C34—C33—C32	121.1 (3)
C13—C12—C35	118.6 (3)	C33—C34—C29	121.6 (3)
C14—C13—C12	121.5 (3)	N1—C35—C12	114.5 (3)
O1—C14—C13	124.4 (4)		

Data collection: Rigaku AFC-5*R* software. Cell refinement: Rigaku AFC-5*R* software. Data reduction: Rigaku AFC-5*R* software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus*. Software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993).

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