

4-{5-(Dimethylamino)-2-[4-(dimethylamino)-2-iodophenylmethyl]phenyl}-(3*E*)-3-buten-2-one

Bao-Hua Chen, Yong-Min Liang,
Xiao-Li Wu, Tong Xie, Liu-Fang
Wang and Yong-Xiang Ma*

National Laboratory of Applied Organic
Chemistry, Lanzhou University, Lanzhou,
Gansu 730000, People's Republic of China

Correspondence e-mail: xraychem@nju.edu.cn

Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

R factor = 0.045

wR factor = 0.103

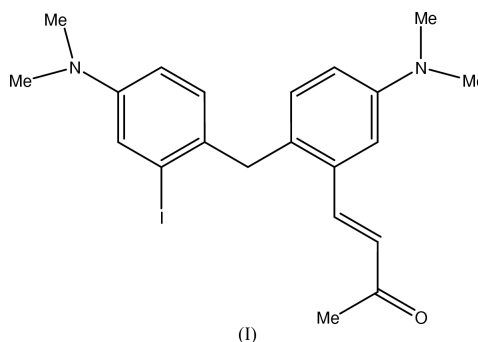
Data-to-parameter ratio = 14.6

For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{21}\text{H}_{25}\text{BrN}_2\text{O}$, was synthesized by the Heck reaction. Its crystal structure shows that there are π - π -stacking interactions between the two aromatic rings of the two neighboring molecules in the unit cell. The C—I bond distance is 2.108 (4) Å and the butene double-bond distance is 1.311 (4) Å.

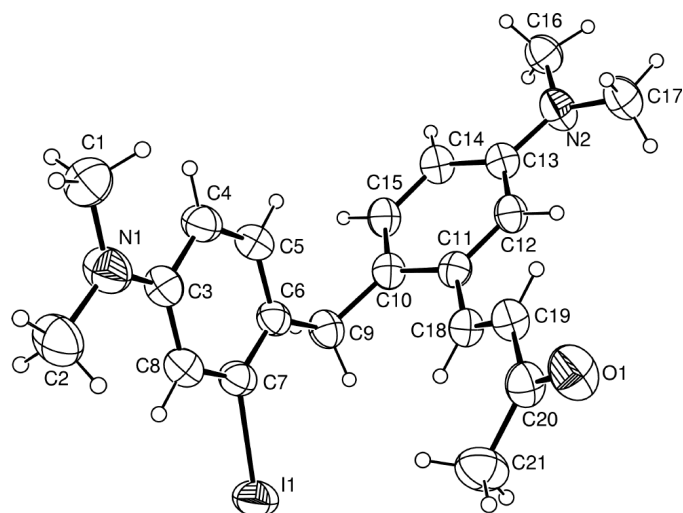
Comment

The title compound, (I), was synthesized by the Heck reaction (Liang *et al.*, 1998). This kind of coupling reaction is very useful for the synthesis of biaryls and stereospecific conjugated enynes, in particular for unsymmetrical cases (Stang & Zhdankin, 1996). The crystal structure of (I) shows that there are π - π -stacking interactions between the two aromatic rings of the two neighboring molecules in the unit cell. The pair of aromatic rings are parallel to each other; the interplanar distance is 3.556 (2) Å. Bond distances and angles are in the expected ranges: the C7—I1 bond distance is 2.108 (4) Å, the C18—C19 double-bond distance is 1.311 (4) Å, C—N bond distances are 1.390 (5)–1.428 (5) Å, C—C bond distances in the aromatic rings are 1.347 (5)–1.420 (4) Å and the other C—C bond distances are 1.456 (5)–1.530 (4) Å.



Experimental

The title compound was prepared by the palladium-catalysed cross-coupling of 3,7-bis(*N,N*-dimethylamino)-10*H*-dibenz[*b,e*]iodinium with methyl vinyl ketone in the presence of a base, according to previously described procedures (Liang *et al.*, 2000). Yellow single crystals suitable for X-ray determination were obtained by slow evaporation of an ethyl acetate solution at room temperature. ^1H NMR (CDCl_3 , 80 MHz, p.p.m.): 2.33 (*s*, 3H), 2.91 (*s*, 6H), 2.99 (*s*, 6H), 4.04 (*s*, 2H), 6.55 (*d*, 1H, $J = 16\text{ Hz}$), 6.61–7.23 (*m*, 6H), 7.67 (*d*, 1H, $J = 16\text{ Hz}$). IR (KBr, cm^{-1}): 3020, 1660, 953 and 975. MS: m/z (%) 448 (M^+ , 100), 405 (37), 277 (28) and 188 (75). Analysis calculated for $\text{C}_{21}\text{H}_{25}\text{IN}_2\text{O}$: C 56.26, H 5.62, N 6.24%; found: C 56.42, H 5.43, N 5.98%. In ^1H NMR spectroscopy, the vinyl H atoms appear as doublets with a coupling constant of 16 Hz, in accord with the *trans* stereochemistry, which is also proved by the X-ray determination.

**Figure 1**

A view of the structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

Crystal data

$C_{21}H_{25}IN_2O$
 $M_r = 448.33$
 Triclinic, $P\bar{1}$
 $a = 9.168$ (3) Å
 $b = 10.884$ (1) Å
 $c = 10.969$ (2) Å
 $\alpha = 84.83$ (1)°
 $\beta = 82.21$ (2)°
 $\gamma = 65.51$ (2)°
 $V = 986.2$ (4) Å³

$Z = 2$
 $D_x = 1.510$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 0.0$ – 25.0°
 $\mu = 1.63$ mm⁻¹
 $T = 293$ (2) K
 Plate, brown
 $0.3 \times 0.2 \times 0.2$ mm

Data collection

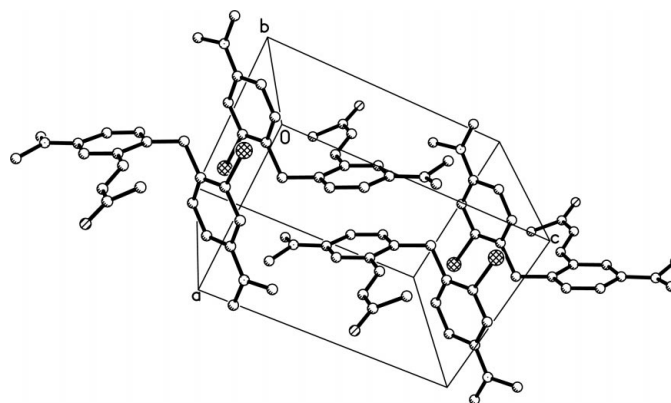
Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.682$, $T_{\max} = 0.721$
 3613 measured reflections
 3417 independent reflections
 2963 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.030$
 $\theta_{\text{max}} = 25.0^\circ$
 $h = -10 \rightarrow 10$
 $k = -12 \rightarrow 12$
 $l = 0 \rightarrow 13$
 5 standard reflections every 300 reflections
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.103$
 $S = 1.07$
 3377 reflections
 231 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.02P)^2 + 1.98P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.97$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³

**Figure 2**

A view of the intermolecular π – π -stacking interactions in (I).

All H atoms were placed in geometrically calculated positions, with C–H = 0.93–0.97 Å, and refined as riding atoms, with $U_{\text{iso}}(\text{H}) = 1.2$ – $1.5U_{\text{eq}}(\text{parent atom})$.

Data collection: *CAD-4/PC* (Enraf–Nonius, 1989); cell refinement: *CAD-4/PC*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXL97*.

We are grateful to the Middle–Young Foundation of Gansu Province (No. YS001-A23-001) for financial support.

References

- Bruker (1997). *SHELXTL*. Bruker AXS inc., Madison, Wisconsin, USA.
 Enraf–Nonius (1989). *CAD-4/PC*. Enraf–Nonius, Delft, The Netherlands.
 Liang, Y. M., Jing, H. W., Liu, C. M., Wu, X. L. & Ma, Y. X. (1998). *Tetrahedron Lett.* **39**, 7143–7146.
 Liang, Y. M., Luo, S. J., Liu, C. M., Wu, X. L. & Ma, Y. X. (2000). *Tetrahedron*, **56**, 2961–2965.
 Molecular Structure Corporation (1989). *TEXSAN*. Version 5.0. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
 Stang, P. J. & Zhdankin, V. V. (1996). *Chem. Rev.* **96**, 1123–1178.