

# Synthesis and physical properties of ferrocene derivatives. XXI. Crystal structure of a liquid crystalline ferrocene derivative, 1,1'-bis[3-[4-(4-methoxyphenoxy)carbonyl]phenoxy]propyl-oxycarbonyl]ferrocene

Takashi Okabe,<sup>a,‡</sup> Keisuke Nakazaki,<sup>a</sup> Tsuyoshi Igaue,<sup>a</sup> Naotake Nakamura,<sup>a,\*</sup> Bertrand Donnio,<sup>b</sup> Daniel Guillon<sup>b</sup> and Jean-Louis Gallani<sup>b</sup>

<sup>a</sup>Department of Applied Chemistry, College of Science and Engineering, Ritsumeikan University, 1-1-1 Nojihigashi, Kusatsu, Shiga 525-8577, Japan, and <sup>b</sup>Institut de Physique et Chimie des Matériaux de Strasbourg, UMR 7504, Groupe des Matériaux Organiques, 23 rue du Loess, BP43, F-67034 Strasbourg Cedex 2, France. Correspondence e-mail: nakamura@sk.ritsumeik.ac.jp

The crystal structure of the title 1,1'-disubstituted ferrocene derivative was determined by X-ray diffraction using a single crystal. This compound exhibits a nematic phase only. The X-ray structure analysis revealed that the two substituents lie in the same direction ('U' shape) and the flexible spacer adopts a *gauche* conformation. These conformations are strongly related to the formation of a rod-like shape, which favors liquid crystalline behavior. In the crystal structure, C—H... $\pi$ ,  $\pi$ — $\pi$  and T-stacking interactions were observed. It is considered that these interactions play a major role in stabilizing the molecular packing arrangement and the mesomorphism.

## 1. Introduction

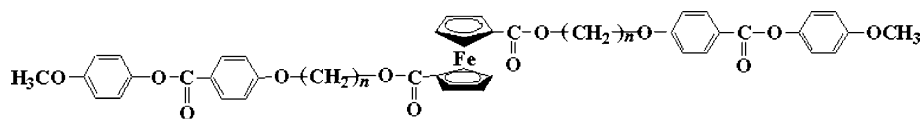
Ferrocene is a metallocene that shows a remarkable aromaticity combined with thermal stability. It is well known that ferrocene derivatives have the potential to be liquid crystalline materials with unique properties, such as electrochemical (Carano *et al.*, 2002) and photoinduced electron transfer (Even *et al.*, 2001) properties. As substitution reactions occur easily at the cyclopentadienyl rings in the ferrocene molecule, just like in benzene, many different types of liquid crystalline ferrocene derivative have been synthesized and investigated. A number of papers concerned with ferrocene-containing liquid crystals have been published already. These papers deal with the thermal and mesomorphic properties of mono-substituted ferrocenes (Malthête & Billard, 1976; Imrie *et al.*, 2001, 2003; Nakamura, Hanasaki & Onoi, 1993; Nakamura, Hanasaki, Onoi & Oida, 1993; Hanasaki *et al.*, 1993; Nakamura *et al.*, 1994, 1995; Nakamura & Takayama, 1997; Nakamura & Setodoi, 1998*a,b*, 1999*a,b*; Nakamura & Oida, 1999; Nakamura, Maekawahara *et al.*, 2000; Nakamura, Setodoi & Hanasaki, 2000; Nakamura, Setodoi & Takayama, 2000; Nakamura *et al.*, 2002; Nakamura *et al.*, 2006*a,b*; Zhao *et al.*, 2001; Deschenaux, Marendaz *et al.*, 1995; Loubser *et al.*, 1993; Loubser & Imrie, 1997), 1,1'-, 1,3- and 1,2-disubstituted ferrocenes (see next paragraph), 1,1',3-trisubstituted ferrocene derivatives (Deschenaux, Kosztics & Nocolet, 1995), polycatenar ferrocenes (Deschenaux, Monnet *et al.*, 1998), and

ferrocene-containing liquid crystalline dendromers (Carano *et al.*, 2002; Even *et al.*, 2001; Chuard & Deschenaux, 2003; Deschenaux *et al.*, 1997; Dardel *et al.*, 1999; Deschenaux, Even & Guillon, 1998; Campidelli *et al.*, 2004) [see also the reviews by Donnio *et al.* (2003) and Bruce *et al.* (2007*a*)].

From the structural viewpoint, disubstituted ferrocene derivatives can be classified into three families according to the positions of the substituent: 1,1'-, 1,2- and 1,3-disubstituted ferrocene derivatives. It is well known that the 1,1'-disubstituted ferrocene derivatives can adopt an 'S' (Nakamura *et al.*, 2005), a 'U' (Nakamura & Okabe, 2004; Nakamura *et al.*, 2007) or a 'Z' (Nakamura & Nishikawa, 2005) shaped conformation. On the other hand, the 1,3-disubstituted ferrocene derivatives give a 'T' (Deschenaux *et al.*, 1993) shaped conformation. In the 'S', the 'T' and the 'Z' shaped conformations, the two substituents lie in opposite directions with respect to the ferrocenyl group, but the 'U' shape means that the two substituents lie in the same direction. In general, it is advantageous for the appearance of liquid crystallinity that the aspect ratio of molecular length to breadth should be relatively large. Therefore, it is considered that the 'S' and the 'T' shaped conformations are typical for liquid crystals of 1,1'-disubstituted ferrocene derivatives. In addition, it has been reported that 1,2-disubstituted ferrocene derivatives do not show liquid crystalline properties because of their unique hairpin structures (Bruce *et al.*, 2007*b*).

A series of disubstituted ferrocene derivatives, 1,1'-bis[ $\omega$ -[4-(4-methoxyphenoxy)carbonyl]phenoxy]alkyloxycarbonyl]ferrocene (abbreviated hereafter as bMAF-*n*, *n* = 2–12, where

<sup>‡</sup> Present address: Film Customer Solution Department, Films Division, Unitika Ltd, 31-3 Uji-Hinojiri, Uji-City, Kyoto 611-0021, Japan.



**Figure 1**  
General structure of bMAF-*n* (*n* = 2–12).

*n* is the number of C atoms in the methylene chain), have been prepared in our laboratory. The general structure of bMAF-*n* is shown in Fig. 1. In the homologous series, bMAF-3 and bMAF-5–bMAF-12 show liquid crystallinity, with nematic and smectic (except *n* = 3) phases. In contrast, bMAF-2 and bMAF-4 do not exhibit any liquid crystallinity (Hanasaki *et al.*, 1994; Nakamura *et al.*, 1998). The crystal structures of bMAF-2 (Nakamura & Nishikawa, 2005), bMAF-5 (Nakamura *et al.*, 2005), bMAF-9 (Nakamura *et al.*, 2007) and bMAF-10 (Nakamura & Okabe, 2004) have already been determined. In the crystal of bMAF-2 (Nakamura & Nishikawa, 2005), the molecule adopts the ‘Z’ shaped conformation, and bMAF-5 (Nakamura *et al.*, 2005) adopts the ‘S’ shaped one, whereas in the crystals of bMAF-9 (Nakamura *et al.*, 2007) and bMAF-10 (Nakamura & Okabe, 2004), the molecules exhibit the ‘U’ shaped conformation.

Correlations between the crystal structure and some physical properties can be deduced from the structure analyses.

In this study, the crystal and molecular structures of bMAF-3 were determined by X-ray diffraction methods using single crystals. Some interesting interactions will be discussed.

## 2. Experimental

Single crystals of the title compound were obtained from a mixed solvent solution of ethylacetate and heptane (1:1) by slow evaporation. The single crystals obtained are orange in color and plate-like. The sample, which had approximate dimensions of 0.75 × 0.10 × 0.05 mm, was mounted on a goniometer.

**Table 1**  
Summarized crystallographic data and results of final refinement.

Empirical formula	C <sub>46</sub> H <sub>42</sub> O <sub>12</sub> Fe
Formula weight	842.68
Crystal system	Monoclinic
Space group	P2 <sub>1</sub>
<i>a</i> (Å)	5.857 (4)
<i>b</i> (Å)	24.105 (3)
<i>c</i> (Å)	14.069 (4)
$\beta$ (°)	93.15 (4)
Volume (Å <sup>3</sup> )	1983.5 (14)
<i>Z</i>	2
<i>D</i> <sub>calc</sub> (g cm <sup>-3</sup> )	1.411
Measured reflections	6153
Independent reflections	4059
Observed reflections	4059
<i>R</i>	0.038
<i>wR</i> ( <i>F</i> <sup>2</sup> )	0.098
Goodness of fit ( <i>S</i> )	1.052
Flack parameter	−0.005(5)

Data collection was carried out at 294 K, using a Rigaku AFC5R four-circle diffractometer with graphite-monochromated Cu K $\alpha$  ( $\lambda$  = 1.54178 Å) radiation, operated at 50 kV and 200 mA.

The unit-cell parameters were obtained from a least-squares refinement using the setting angle of 22 reflections in the range 18.27 < 2 $\theta$  < 26.62°. The data were collected using the  $\omega$ -2 $\theta$  scan technique to a maximum 2 $\theta$  value of 140.3°.

6153 reflections were measured, of which 4059 were unique. The intensities of three standard reflections were measured after every 150 reflections. Over the course of the data collection, the intensity of the standards decreased by 0.88%. The data were also corrected for Lorentz and polarization effects and for absorption [numerical (Coppens *et al.*, 1965); the minimum and maximum transmission factors were 0.60091 and 0.83876, respectively].

All calculations were performed using the *WinGX* crystallographic software package (Farrugia, 1999). The atomic scattering factors were taken from Cromer & Waber (1974). The structure was solved by direct methods (*SIR92*; Altomare *et al.*, 1994) and expanded using the Fourier technique. All non-H atoms were refined anisotropically, and all H atoms were refined isotropically. The H atoms were introduced at their theoretical positions and allowed to ride with the C atoms to which they are attached. The final refinement was made by full-matrix least-squares analysis based on 4059 observed reflections. The refinement cycles converged to *R* = 0.038, *wR*(*F*<sup>2</sup>) = 0.098, *w* = 1/[ $\sigma^2(F_o^2) + (0.0211P)^2 + 1.2943P$ ], where *P* = [Max(*F*<sub>o</sub><sup>2</sup>, 0) + 2*F*<sub>o</sub><sup>2</sup>]/3, and *S* = 1.052. The maximum shift s.u. ratio for all atoms in the final cycle is less than 0.001. The final cycles of the refinement resulted in a residual electron density in the range −0.369–0.346 eÅ<sup>-3</sup>.

Experimental details are summarized in Table 1.<sup>1</sup>

## 3. Results and discussion

### 3.1. Molecular structure

An *ORTEP* (Johnson & Burnett, 1996) drawing showing the molecular structure with the numbering for each atom is presented in Fig. 2, where the H atoms have been omitted for simplification.

In the ferrocenyl group, the two cyclopentadienyl rings are almost parallel; the dihedral angle is 1.0 (2)° (Table 2). The average values of the Fe–C and C–C bond distances in the ferrocenyl unit of bMAF-3 are 2.050 (5) and 1.421 (7) Å, respectively. The C–C–C bond angles in the cyclopentadienyl rings are 108.0 (4)°. These values agree with those of ferrocene reported elsewhere (Nakamura & Setodoi, 1998*a,b*, 1999*a,b*; Nakamura *et al.*, 2002, 2005, 2006*a,b*, 2007; Nakamura, Setodoi & Takayama, 2000; Nakamura & Okabe, 2004;

<sup>1</sup> Supplementary data for this paper are available from the IUCr electronic archives (Reference: DD5042). Services for accessing these data are described at the back of the journal.

**Table 2**  
Dihedral angles between least-squares planes of bMAF-3.

Plane No.	Least-squares plane
Plane 1	Cp ring C1–C5
Plane 2	Phenyl ring C10–C15
Plane 3	Phenyl ring C17–C22
Plane 4	Cp ring C24–C28
Plane 5	Phenyl ring C33–C38
Plane 6	Phenyl ring C40–C45

Planes	Dihedral angle (°)
Plane 1–Plane 2	51.6 (2)
Plane 1–Plane 3	14.9 (2)
Plane 1–Plane 4	1.0 (2)
Plane 1–Plane 5	14.8 (2)
Plane 1–Plane 6	46.3 (2)
Plane 2–Plane 3	63.1 (2)
Plane 2–Plane 4	50.8 (2)
Plane 2–Plane 5	65.8 (2)
Plane 2–Plane 6	6.4 (2)
Plane 3–Plane 4	15.3 (2)
Plane 3–Plane 5	6.3 (2)
Plane 3–Plane 6	58.6 (2)
Plane 4–Plane 5	15.6 (2)
Plane 4–Plane 6	45.5 (2)
Plane 5–Plane 6	60.8 (2)

Nakamura & Nishikawa, 2005; Dunitz *et al.*, 1956) within experimental error.

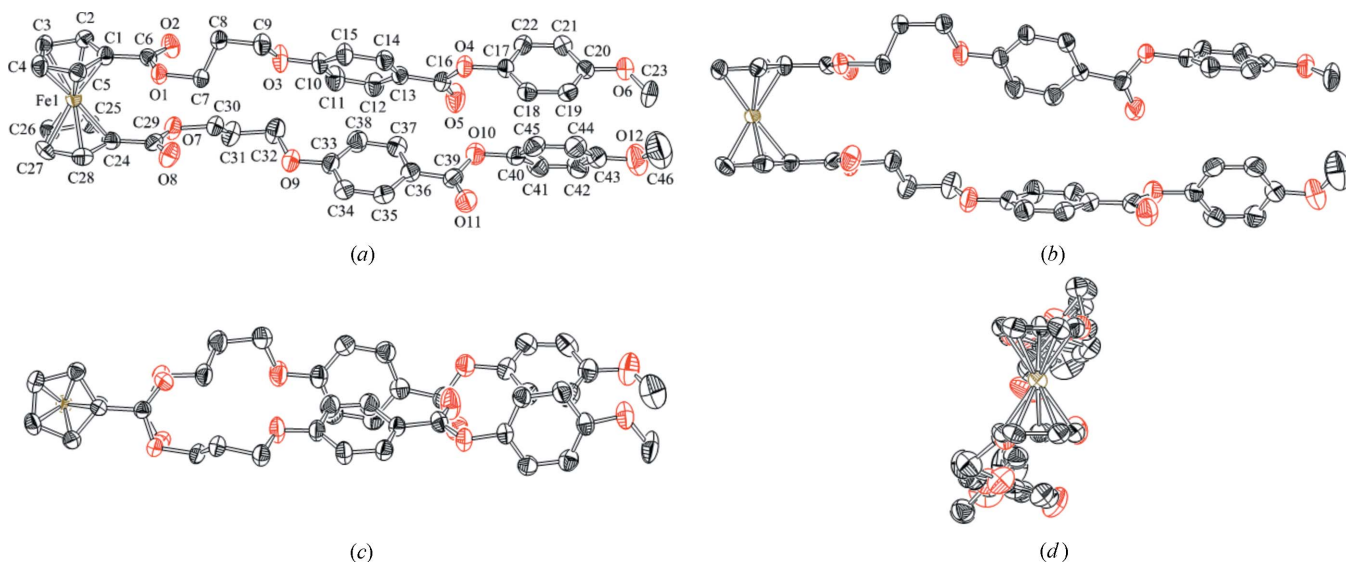
In the flexible spacers (C7–C9 and C30–C32), the C–C–C bond angles are 111.3 (4) and 113.1 (5)°, the average values of the C–C bond length are 1.509 (7) and 1.496 (8) Å, and the lengths of the chains are 2.488 (7) and 2.497 (7) Å, respectively. These values are nearly equal to the values determined for *n*-paraffin [*e.g.* the average C–C bond distance is approximately 1.54 Å and the length of the –CH<sub>2</sub>–CH<sub>2</sub>–CH<sub>2</sub>– unit is 2.54 Å (Bunn, 1939)].

**Table 3**  
Selected torsion angles (°) of bMAF-3.

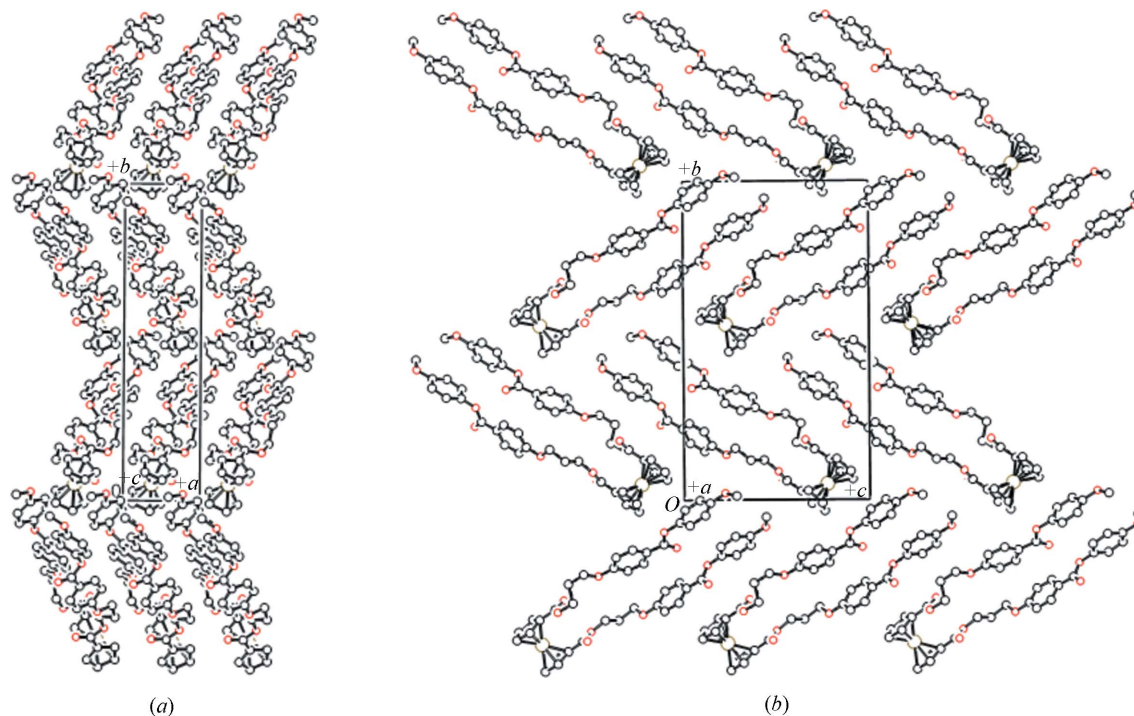
C24–Fe1–C1–C6	1.8 (4)
O2–C6–O1–C7	0.3 (7)
C6–O1–C7–C8	78.0 (5)
O1–C7–C8–C9	176.5 (4)
C7–C8–C9–O3	59.4 (6)
C8–C9–O3–C10	176.6 (4)
C13–C16–O4–C17	–174.8 (4)
O5–C16–O4–C17	4.1 (7)
C1–Fe1–C24–C29	2.7 (5)
O8–C29–O7–C30	–3.7 (7)
C29–O7–C30–C31	145.9 (5)
O7–C30–C31–C32	178.1 (5)
C30–C31–C32–O9	63.2 (7)
C31–C32–O9–C33	–178.9 (5)
C36–C39–O10–C40	–179.3 (4)
O11–C39–O10–C40	0.2 (8)

The molecular structure of bMAF-3 is somewhat different from that of the previously analyzed ‘U’ shaped homologues bMAF-9 (Nakamura *et al.*, 2007) and bMAF-10 (Nakamura & Okabe, 2004). The dihedral angles of the two phenyl rings (Plane 2–Plane 5 and Plane 3–Plane 6) in the mesogenic group are estimated to be 65.8 (2) and 58.6 (2)°, respectively, as shown in Table 2. In contrast, the dihedral angles between the two phenyl rings of bMAF-9 (Nakamura *et al.*, 2007) and bMAF-10 (Nakamura & Okabe, 2004) are 3.5–11.2°, indicating that these phenyl rings are almost parallel.

The two substituents of bMAF-3 lie in the same direction (‘U’ shape), and the molecular length of bMAF-3 (C4–C23) is estimated to be 20.856 (16) Å. As the C6–O1–C7–C8, C7–C8–C9–O3, C29–O7–C30–C31 and C30–C31–C32–O9 fragments adopt a *gauche* conformation in the flexible spacer, the molecules can be considered as rod-like molecules, as shown in Table 3. A new shape of bMAF-*n* molecule was found by Nakamura & Nishikawa (2005), who reported a



**Figure 2**  
ORTEP (Johnson & Burnett, 1996) views of the molecular structure excluding H atoms in bMAF-3. Displacement ellipsoids are drawn at the 50% probability level. Showing (a) the crystallographic numbering scheme, (b) a side view onto the cyclopentadienyl rings, (c) a view from above, onto the cyclopentadienyl rings, and (d) a projection along the long axis of the molecule. C atoms: black; O atoms: red; Fe atoms: brown.



**Figure 3**  
Crystal structure of bMAF-3. (a) Projection onto the *ab* plane and (b) projection onto the *bc* plane.

novel molecular structure for bMAF-2, the name of which was associated with a 'Z' shape. The aspect ratio of bMAF-2 (Nakamura & Nishikawa, 2005) is rather low, which is not advantageous for the appearance of liquid crystallinity. Therefore, the rod-like shape obtained with the molecules analyzed in this paper is an important factor for obtaining a mesomorphic behavior.

### 3.2. Crystal structure

Projections of the *ab* and *bc* planes of bMAF-3 are shown in Figs. 3(a) and 3(b), respectively. In both projections, herringbone layer structures were observed. Among the other homologues of bMAF-*n* for which the molecular and crystal structures have been analyzed, only bMAF-3 exhibits such a structure.

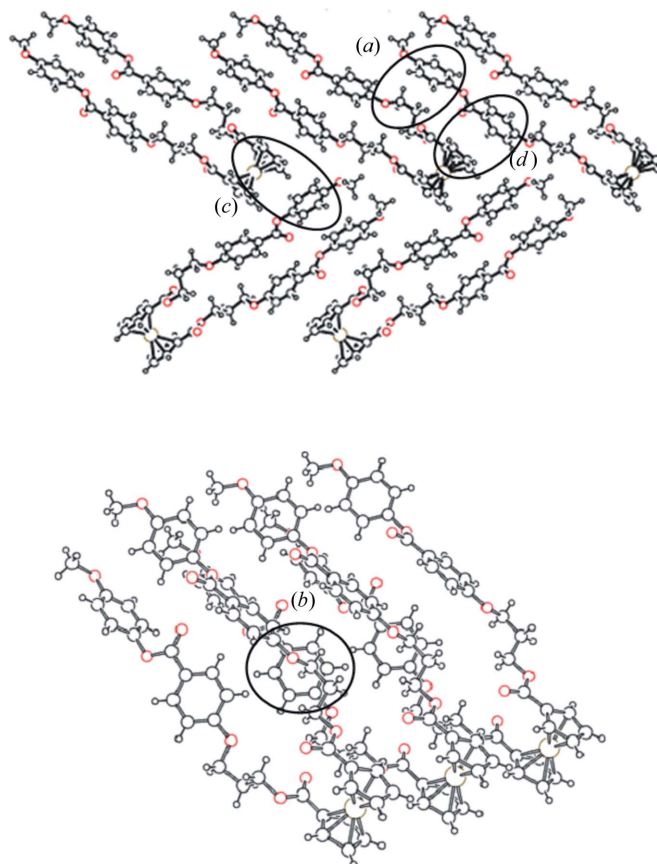
The following intermolecular interactions were identified in bMAF-3:

(i) Two C–H... $\pi$  interactions. One is between the H atoms on C8 and C9 of the methylene chains and the C40–C45 benzene ring of the mesogenic group (Fig. 4a). The other is between the H atom on C32 and the C10–C15 benzene ring (Fig. 4b).

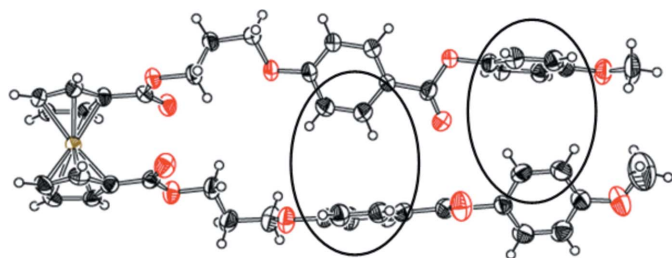
(ii) One T-stacking  $\pi$ – $\pi$  interaction, between the H atom on C4 of the Cp ring and the C17–C22 benzene ring in a neighboring layer (Fig. 4c).

(iii) A  $\pi$ – $\pi$  interaction between the C1–C5 Cp ring and the C33–C38 benzene ring (Fig. 4d).

In addition, two intramolecular interactions are identified between the two substituents of the molecule. As shown in Fig. 5, they are two T-stacking interactions between H atoms



**Figure 4**  
Illustration of intermolecular interactions of bMAF-3.



**Figure 5**  
Illustration of intramolecular interactions of bMAF-3.

(on C18, C19 and C37, C38) of the phenyl groups of the mesogenic units and the benzene rings C40–C45 and C10–C15, respectively. Therefore, bMAF-3 has four intermolecular interactions and two intramolecular interactions.

bMAF-3 exhibits only a monotropic nematic phase. On the other hand, bMAF-9 (Nakamura *et al.*, 2007), for example, possesses a monotropic nematic phase and a smectic one. The difference between the occurrences of these liquid crystal phases is attributed to the different features of the inter- and intramolecular interactions in bMAF-3 and bMAF-9 (Nakamura *et al.*, 2007). In the cooling process from the isotropic liquid phase, bMAF-3 gradually aggregates through the above-mentioned intramolecular interactions between the two substituents of bMAF-3 and the intermolecular interactions between neighboring molecules, while bMAF-9 (Nakamura *et al.*, 2007) aggregates through the only intermolecular interactions between the two substituents of bMAF-9. Consequently, the presence of the different interactions may play an important role in giving rise to the mesomorphism.

A High-Tech Research Center Project for Private Universities matching subsidy from the Ministry of Education, Culture, Sports, Science and Technology, 2006–2010, is gratefully acknowledged for partial financial support of the present work. We are grateful to Associate Professor T. Hanasaki and Dr K. Uno, Ritsumeikan University, Japan, for useful discussions.

## References

- Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M. C., Polidori, G. & Camalli, M. (1994). *J. Appl. Cryst.* **27**, 435.
- Bruce, D. W., Deschenaux, R., Donnio, B. & Guillon, D. (2007a). *Comprehensive Organometallic Chemistry III*, edited by R. Crabtree & M. Mingos, Vol. 12, ch. 12.05, pp. 221–238. Oxford: Elsevier.
- Bruce, D. W., Deschenaux, R., Donnio, B. & Guillon, D. (2007b). *Comprehensive Organometallic Chemistry III*, edited by R. Crabtree & M. Mingos, Vol. 12, ch. 12.05, p. 230. Oxford: Elsevier.
- Bunn, C. W. (1939). *Trans. Faraday Soc.* **35**, 482–491.
- Campidelli, S., Vázquez, E., Milic, D., Prato, M., Barberá, J., Guldi, D. M., Marcaccio, M., Paolucci, D., Paolucci, F. & Deschenaux, R. (2004). *J. Mater. Chem.* **14**, 1266–1272.
- Carano, M., Chuard, T., Deschenaux, R., Even, M., Maraccio, M., Paolucci, F., Parato, M. & Roffia, S. (2002). *J. Mater. Chem.* **12**, 829–833.
- Chuard, T. & Deschenaux, R. (2003). *Chimia*, **57**, 597–600.
- Coppens, P., Leiserowitz, L. & Rabinovich, D. (1965). *Acta Cryst.* **18**, 1035–1038.
- Cromer, D. T. & Waber, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. 4. Birmingham: Kynoch Press.
- Dardel, B., Deschenaux, R., Even, M. & Serrano, E. (1999). *Macromolecules*, **32**, 5191–5193.
- Deschenaux, R., Even, M. & Guillon, D. (1998). *Chem. Commun.* pp. 537–538.
- Deschenaux, R., Kosztics, I., Marendaz, J.-L. & Stoeckli-Evans, H. (1993). *Chimia*, **47**, 206–210.
- Deschenaux, R., Kosztics, I. & Nocolet, B. (1995). *J. Mater. Chem.* **5**, 2291–2295.
- Deschenaux, R., Marendaz, J.-L., Santiago, J. & Goodby, J. W. (1995). *Helv. Chim. Acta*, **78**, 1215–1218.
- Deschenaux, R., Monnet, F., Serrano, E., Turpin, F. & Levelut, A.-M. (1998). *Helv. Chim. Acta*, **81**, 2072–2077.
- Deschenaux, R., Serrano, E. & Levelut, A.-M. (1997). *Chem. Commun.* pp. 1577–1578.
- Donnio, B., Guillon, D., Deschenaux, R. & Bruce, D. W. (2003). *Comprehensive Coordination Chemistry II*, edited by J. A. McCleverty & T. J. Meyer, Vol. 7, ch. 7.9, pp. 585–596. Oxford: Elsevier.
- Dunitz, J. D., Orgel, L. E. & Rich, A. (1956). *Acta Cryst.* **9**, 373–375.
- Even, M., Heinrich, B., Guillon, D., Guldi, D. M., Parato, M. & Deschenaux, R. (2001). *Chem. Eur. J.* **7**, 2595–2604.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Hanasaki, T., Ueda, M. & Nakamura, N. (1993). *Mol. Cryst. Liq. Cryst.* **237**, 329–336.
- Hanasaki, T., Ueda, M. & Nakamura, N. (1994). *Mol. Cryst. Liq. Cryst.* **250**, 257–267.
- Imrie, C., Engelbrecht, P., Loubser, C. & McClelland, C. W. (2001). *Appl. Organomet. Chem.* **15**, 1–15.
- Imrie, C., Loubser, C., Engelbrecht, P., McClelland, C. W. & Zheng, Y. (2003). *J. Organomet. Chem.* **665**, 48–64.
- Johnson, C. K. & Burnett, M. N. (1996). *ORTEP III*. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
- Loubser, C. & Imrie, C. (1997). *J. Chem. Soc. Perkin Trans. 2*, pp. 399–410.
- Loubser, C., Imrie, C. & van Rooyen, P. H. (1993). *Adv. Mater.* **5**, 45–47.
- Malthête, J. & Billard, J. (1976). *Mol. Cryst. Liq. Cryst.* **34**, 117–121.
- Nakamura, N., Hanasaki, T. & Onoi, H. (1993). *Mol. Cryst. Liq. Cryst.* **225**, 269–277.
- Nakamura, N., Hanasaki, T., Onoi, H. & Oida, T. (1993). *Chem. Express*, **8**, 467–470.
- Nakamura, N., Maekawahara, H., Hanasaki, T. & Yamaguchi, T. (2000). *Mol. Cryst. Liq. Cryst.* **352**, 125–132.
- Nakamura, N., Mizoguchi, R., Ueda, M. & Hanasaki, T. (1998). *Mol. Cryst. Liq. Cryst.* **312**, 127–136.
- Nakamura, N., Nio, T. & Okabe, T. (2006a). *Mol. Cryst. Liq. Cryst.* **460**, 85–92.
- Nakamura, N., Nio, T. & Okabe, T. (2006b). *Mol. Cryst. Liq. Cryst.* **461**, 29–36.
- Nakamura, N., Nio, T., Okabe, T., Donnio, B., Guillon, D. & Gallani, J.-L. (2007). *Mol. Cryst. Liq. Cryst.* **466**, 3–12.
- Nakamura, N. & Nishikawa, M. (2005). *Chem. Lett.* **34**, 1544–1545.
- Nakamura, N. & Oida, T. (1999). *Mol. Cryst. Liq. Cryst.* **326**, 55–64.
- Nakamura, N., Oida, T., Shonago, M., Onoi, H. & Hanasaki, T. (1995). *Mol. Cryst. Liq. Cryst.* **265**, 1–8.
- Nakamura, N. & Okabe, T. (2004). *Chem. Lett.* **33**, 358–359.
- Nakamura, N., Okabe, T. & Takahashi, T. (2005). *Mol. Cryst. Liq. Cryst.* **441**, 243–250.
- Nakamura, N., Onoi, H., Oida, T. & Hanasaki, T. (1994). *Mol. Cryst. Liq. Cryst.* **257**, 43–48.
- Nakamura, N. & Setodoi, S. (1998a). *Mol. Cryst. Liq. Cryst.* **312**, 253–261.

- Nakamura, N. & Setodoi, S. (1998b). *Mol. Cryst. Liq. Cryst.* **319**, 173–181.
- Nakamura, N. & Setodoi, S. (1999a). *Mol. Cryst. Liq. Cryst.* **326**, 177–187.
- Nakamura, N. & Setodoi, S. (1999b). *Mol. Cryst. Liq. Cryst.* **333**, 151–163.
- Nakamura, N., Setodoi, S. & Hanasaki, T. (2000). *Mol. Cryst. Liq. Cryst.* **350**, 93–101.
- Nakamura, N., Setodoi, S. & Takayama, T. (2000). *Mol. Cryst. Liq. Cryst.* **346**, 19–28.
- Nakamura, N., Takahashi, T., Uno, K. & Hanasaki, T. (2002). *Mol. Cryst. Liq. Cryst.* **383**, 27–35.
- Nakamura, N. & Takayama, T. (1997). *Mol. Cryst. Liq. Cryst.* **307**, 145–154.
- Zhao, K.-Q., Hu, P., Xu, H.-B., Wan, W., Zhou, Z.-Y. & Zhang, L.-F. (2001). *Mol. Cryst. Liq. Cryst.* **364**, 759–768.