Les ellipsoïdes d'agitation thermique anisotrope des atomes de soufre et d'azote ont leur grand axe approximativement perpendiculaire au plan du bicycle (leurs angles respectifs avec la normale au plan sont inférieurs à 20°) et les déplacements carrés moyens ont des valeurs normales pour ce type de composé.

De plus, dans le cas de l'azote, le petit axe de l'ellipsoïde se trouve dans la direction où devrait se trouver son grand axe si les coordonnées atomiques étaient une moyenne de deux positions voisines.

Tous ces faits plaident en faveur de la seconde hypothèse, c'est à dire qu'il y a une seule forme de la molécule dans le cristal.

D'autre part, nous constatons que dans le cycle S(1), C(3), C(4), C(5), N, les angles ayant comme sommets des atomes de carbone, sont tous systématiquement inférieurs à 120° et valent environ 113°; ceci pourrait être le résultat d'une interaction S···N. En effet, si les angles de liaisons des atomes de carbone étaient de 120° dans ce cycle comme ils le sont dans les trois phényles et le cycle S(1), S(2), C(1), C(2), C(3), l'atome d'azote serait à plus de 2,4 Å du soufre S(1).

En conséquence, le fait qu'il n'y a qu'une seule molécule par unité asymétrique et qu'il y a très probablement une interaction S···N, nous permet de conclure à l'absence de tautomérie de valence pour ce composé à l'état cristallin. En plus, l'agitation thermique anisotrope de l'atome de soufre S(1) ne permet pas de lui attribuer un effet de 'battant de cloche' que Shen & Hedberg (1974), et Gleiter & Hoffmann (1968) proposent pour les atomes de soufre des trithiapentalènes.

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Tri-μ-chloro-bis(η-cycloheptatrienyl)dimolybdenum Tetrafluoroborate at −60°C

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Abstract. $C_{14}H_{14}BCl_3F_4Mo_2$, tetragonal, $I4$. At −60 °C, $a = 11\cdot8920$ (8), $c = 12\cdot9105$ (15) Å, $V = 1825\cdot8$ Å^3, $Z = 4$, $D_x = 2\cdot06$ g cm^{-3}, μ(Mo $K\alpha$) = 18·2 cm^{-1}. The cation (symmetry 2) contains three bridging Cl atoms and apparently symmetrical, planar C_7H_7 rings; the Mo—Mo distance is 3·051 (1) Å, corresponding to a single bond. There is some rotational disorder in both the cation and the anion. The structure has also been determined at 20 °C to less precision.

Introduction. Crystals of the title compound were provided by Dr M. L. H. Green (Ashworth, Green & Knight, 1974). They were deep-green, air-stable and of well developed tabular form (principal faces {001}, bounded by {111}). Data were collected on a Syntex $P2_1$ diffractometer with graphite-monochromatized Mo $K\alpha$ radiation, to a $2\theta_{\max}$ of 50°, at scan rates between 1·0 and 29·3° min^{-1}, depending on the intensity of a 2 s pre-scan. The crystal was held at −60 °C with the Syntex L-T 1 attachment. Three standard reflexions were used; these showed no significant change in intensity. After correction for Lorentz, polarization and absorption effects, 801 reflexions were considered observed $[I/\sigma(I) \geq 3\cdot0]$ and used in refinement. Data were also collected at 20 °C on a Picker card-controlled diffractometer, and used for an initial structure solution; the cell parameters were $a = 11\cdot985$ (5), $c = 13\cdot049$ (9) Å, $V = 1874\cdot4$ Å^3.

The lack of systematic absences indicates space groups $I4$, $I\bar{4}$ or $I4/m$. A Patterson synthesis located the Mo atom with z close to or precisely zero, x and y being consistent with a molecule centred on ($\frac{1}{2}$,0,0) and with either 2 ($I4$, $I\bar{4}$) or $2/m$ ($I4/m$) symmetry, with disorder in the latter. Further differences between the space groups are that for $I4$ and $I4/m$, but not $I\bar{4}$, the Mo z coordinates are all zero. For $I4$ the triangles of Cl atoms all point in one direction, for $I\bar{4}$ they are alternately up and down, and for $I4/m$ both orientations are superimposed. Location of the remaining atoms and refinement proceeded satisfactorily in space group $I4$, which was therefore selected.

The cation and anion each lie on twofold axes. With the room-temperature data, one F atom (in the BF_4^- group) is on this axis, with a twofold disorder of the remainder. With anisotropic temperature factors for all except the BF_4^- atoms, and with the planar C_7 and tetrahedral BF_4^- held rigid [C–C 1·403 Å (Engebretson & Rundle, 1963), B–F 1·43 Å], a final *R* of 0·083 was reached. With the low-temperature data the disorder is rather more complex: the bridging Cl_3 group has about 25% occupancy of the inverted position, and the BF_4^- also shows some further F positions. The C_7 ring is ordered. It was refined both as a planar group to $R = 0{\cdot}060$ and independently to $R = 0{\cdot}059$ with differences in the geometry which are not significant. The refinement with rigid C_7 groups of the −60°C data is preferred, and its results are used here, but the details of the free refinement and of the room-temperature data are included in the deposited material.*

Fig. 1 shows the cation and Fig. 2 the crystal packing. Table 1 gives atomic coordinates and Table 2 the bond lengths and angles. Standard deviations for group atoms are calculated from those of the group parameters, with the correlation between them taken into account. Refinement was with XRAY 76 (Stewart, 1976) running on a Burroughs B6700 computer.

Discussion. The most striking aspect of the chemistry of the $[(C_7H_7)MoX_3Mo(C_7H_7)]$ (X = halide or alkoxyl) unit is its ability to exist as both neutral and cationic species, and the ultimate aim of this study is to discover the effect of the change in charge on the Mo–Mo interaction. The structure of the neutral species (X = Cl) is complex and is still under investigation; full discussion of the chemical implications of the present structure will accompany the publication of the structure of the neutral species.

The most important dimension in the cation is the Mo–Mo distance of 3·051 (1) Å. This can be compared with 2·882 Å in the similar $[(C_7H_7)MoBr(OH)_2Mo(C_7H_7)]^+$ ion (Welch, 1977). The diamagnetism of the species requires a metal–metal bond, and the distance observed here is somewhat shorter than the 3·23 Å found for Mo–Mo in unbridged systems (Adams, Brice & Cotton, 1973; Adams, Collins & Cotton, 1974). This suggests that a substantial part of the bonding between the two ends of the ion is provided by the bridging atoms, and a comparison with the

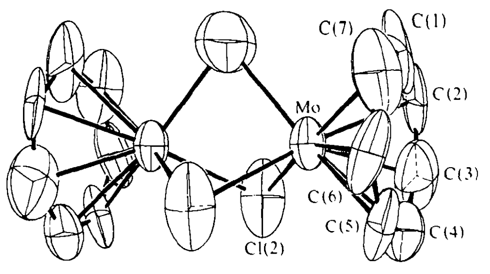

Fig. 1. View of the cation showing atomic numbering. Only the main set of chlorine atoms is included.

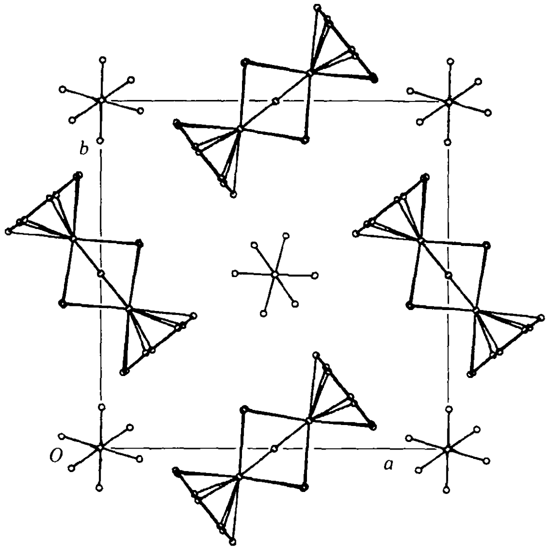

Fig. 2. Packing diagram viewed down **c**. This shows the contents of half the unit cell (approximately $-\frac{1}{4}$ to $+\frac{1}{4}z$): the other half contains the same units, each rotated by 90° about **c**.

Table 1. *Atomic coordinates* (×10³) *with standard deviations in parentheses*

	x	*y*	*z*
Mo	600·15 (9)	80·18 (9)	−0·74 (31)
Cl(1)*	500	0	−142 (2)
Cl(2)*	411 (1)	109 (1)	79 (1)
Cl(3)†	415 (2)	113 (1)	−87 (1)
Cl(4)†	500	0	145 (2)
C(1)	719 (2)	148 (2)	−122 (2)
C(2)	648 (2)	236 (1)	−90 (2)
C(3)	779·6 (10)	71·7 (12)	−61·5 (23)
C(4)	729 (2)	130 (2)	121 (2)
C(5)	656 (1)	221 (2)	105 (2)
C(6)	784 (1)	64 (1)	47 (2)
C(7)	619·8 (9)	268·1 (8)	10·0 (23)
B	0	0	283 (2)
F(1)†	0	0	161 (3)
F(2)‡	84 (3)	59 (3)	253 (2)
F(3)‡	0	0	397 (2)
F(4)†	112 (3)	−31 (3)	299 (3)
F(5)†	3 (4)	115 (3)	254 (3)

* Occupancy 0·75.
† Occupancy 0·25.
‡ Occupancy 0·50.

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* Lists of structure factors, thermal parameters, and alternative coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32713 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 2. *Bond lengths* (Å) *and angles* (°)*with standard deviations in parentheses*

Mo–Mo	3·051 (1)	Mo–C(7)	2·25 (1)
Mo–Cl(1)	2·38 (2)	C–C	1·403 (fixed)
Mo–Cl(2)	2·50 (1)	B–F(1)	1·57 (5)
Mo–Cl(2)′	2·47 (1)	B–F(2)	1·29 (4)
Mo–Cl(3)	2·49 (2)	B–F(3)	1·47 (4)
Mo–Cl(3)′	2·55 (2)	B–F(4)	1·40 (4)
Mo–Cl(4)	2·42 (2)	B–F(5)	1·42 (4)
Mo–C(1)	2·26 (2)		
Mo–C(2)	2·25 (2)	Mo–Cl(1)–Mo′	79·7 (8)
Mo–C(3)	2·28 (2)	Mo–Cl(2)–Mo′	75·7 (4)
Mo–C(4)	2·28 (2)	Mo–Cl(3)–Mo′	74·4 (5)
Mo–C(5)	2·26 (2)	Mo–Cl(4)–Mo′	78·1 (8)
Mo–C(6)	2·28 (1)	C–C–C	128·57 (fixed)

$Br(OH)_2$ bridged species indicates that the Mo–Mo distance is mainly controlled by the nature of the bridging atoms.

The average Mo–C distance (2·265 Å) is similar to that in the $Br(OH)_2$-bridged ion (2·25 Å). It is clear from the thermal parameters and Fig. 1 that the ring has high thermal motion, both circumferentially and by pivoting about the Mo atom.

The packing (Fig. 2) shows no unusual interactions and appears to be dominated by contact between the spherical BF_4^- ions and the elongated cations.

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5-Chloro-1-{3-[4-(4-fluorobenzoyl)piperidino]propyl}-1,3-dihydro-2*H*-benzimidazol-2-one

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Abstract. $C_{22}H_{23}ClFN_3O_2$, $M_r = 415{\cdot}88$; triclinic, $P\bar{1}$; $a = 8{\cdot}084$ (2), $b = 12{\cdot}621$ (3), $c = 10{\cdot}259$ (2) Å, $\alpha = 87{\cdot}10$ (2), $\beta = 87{\cdot}87$ (2), $\gamma = 86{\cdot}56$ (2)°; 25 °C; $Z = 2$. The molecules are linked by hydrogen bonds between the amide groups of the benzimidazolone moiety: N(5)···O(7), 2·82 Å [O(7): $2 - x$, $1 - y$, $-z$].

Introduction. The title compound is a new potent neuroleptic drug. Transparent colourless crystals were obtained by cooling a saturated solution in hot isopropanol. The experimental conditions used for data collection are given in Table 1. The structure was solved with *MULTAN* (Germain, Main & Woolfson, 1971). Anisotropic block-diagonal least-squares refinement (Ahmed, Hall, Pippy & Huber, 1966) gave a final $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$ of 0·09 for all observed

Table 1. *Experimental conditions*

Instrument: Syntex $P2_1$ diffractometer
Source: Cu $K\bar{\alpha}$; $\lambda = 1{\cdot}5418$ Å; ω scan; $\theta_{max} = 55$ °
Confidence level: 2·5
Total number of independent reflexions: 2631
Total observed: 2267

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