K^+ ion lies on the twofold axis and at a distance of 6·97 Å from the center [the midpoint of the C(3)—C(3^i) bond] and 4·52 Å from the end [the midpoint of the C(5)—C(5^i) bond] of the naphthalene.

References

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee.

Live, D. & Chan, S. I. (1976). *J. Amer. Chem. Soc.* **98**, 3769–3778.

Seiler, P., Dobler, M. & Dunitz, J. D. (1974). *Acta Cryst.* B**30**, 2744–2745.

Sousa, L. R. & Johnson, M. R. (1977). Unpublished.

Sousa, L. R. & Larson, J. M. (1977). *J. Amer. Chem. Soc.* **99**, 307–310.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Templeton, L. K. & Templeton, D. H. (1973). Amer. Cryst. Assoc., Storrs, Connecticut Meeting, June 1973. Abstr. E10.

Wei, K.-T. & Ward, D. L. (1976). *Acta Cryst.* B**32**, 2768–2773.

Zalkin, A. (1974). Private communication.

Acta Cryst. (1977). B**33**, 3540–3542

2,2′-Biquinolyl*

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(*Received* 30 *November* 1976; *accepted* 24 *June* 1977)

Abstract. $C_{18}H_{12}N_2$, monoclinic, $P2_1/c$, $Z = 2$, $M_r = 256{\cdot}3$; $a = 14{\cdot}013\,(11)$, $b = 3{\cdot}919\,(4)$, $c = 11{\cdot}656\,(8)$ Å, $\beta = 97{\cdot}03\,(5)^\circ$ at 28°C; $a = 13{\cdot}971\,(11)$, $b = 3{\cdot}853\,(4)$, $c = 11{\cdot}589\,(7)$ Å, $\beta = 97{\cdot}50\,(4)^\circ$ at $-160 \pm 5\,^\circ$C, μ(Mo $K\alpha$) = 0·764 cm^{-1}. The structure was refined to $R = 0{\cdot}065$ for 1407 observed intensities. The configuration is *trans*; the two halves of the molecule are related by a center of symmetry imposed by the space group. The sp^2–sp^2 single-bond length between the two quinoline systems is 1·492 (3) Å.

Introduction. Clear, prismatic crystals were obtained by recrystallization of commercial 2,2′-biquinolyl from ethanol. A small crystal of dimensions 0·37 × 0·27 × 0·23 mm was selected. Intensity data were collected at $-160 \pm 5\,^\circ$C with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71069$ Å) on a Picker FACS-1 diffractometer equipped with a standard gas-flow cooling system (Huffman, 1974). Systematic absences of $0k0$ for $k = 2n + 1$ and $h0l$ for $l = 2n + 1$ identified the space group as $P2_1/c$. Unit-cell parameters at 28°C and at $-160 \pm 5\,^\circ$C were refined by least squares with the orientation and Bragg angles of 14 reflections carefully centered at $\pm 2\theta$. 4327 reflections (including extinctions) were scanned in the range $1 \le 2\theta \le 55^\circ$ with the θ–2θ scan technique, a scan rate of 2° min^{-1} and 20 s background counts at both ends of the scan range of $(2{\cdot}5 + 0{\cdot}692 \tan \theta)^\circ$. Three standard reflections were measured every 30 reflections. No significant trends were observed. Intensities, I, were assigned variances $\sigma^2(I)$ according to counting statistics plus a term $(0{\cdot}03I)^2$ to allow for unaccountable errors. Lorentz and polarization corrections were made in the usual manner. Of 1407 unique reflections 1215 had $(F_o)^2 \ge \sigma(F_o)^2$.

The structure was solved from the Patterson function and refined with the full-matrix least-squares program *XFLS*3 of Busing, Martin & Levy (1974). The function minimized was $\Sigma\, w(|F_o|^2 - |F_c|^2)^2$ with $w = 1/\sigma^2(F_o)^2$.

Table 1. *Final positional parameters with standard deviations in parentheses*

	x	y	z
N(1)	0·12176 (8)	0·0928 (3)	−0·02146 (9)
C(2)	0·04968 (10)	−0·0217 (4)	0·03161 (11)
C(3)	0·06306 (10)	−0·1799 (4)	0·14380 (11)
C(4)	0·15414 (11)	−0·2145 (4)	0·20109 (12)
C(5)	0·33151 (11)	−0·1151 (4)	0·19993 (12)
C(6)	0·40452 (11)	0·0081 (4)	0·14347 (13)
C(7)	0·38392 (11)	0·1607 (4)	0·03224 (13)
C(8)	0·29086 (11)	0·1853 (4)	−0·02086 (12)
C(9)	0·21366 (10)	0·0597 (4)	0·03556 (11)
C(10)	0·23368 (10)	−0·0927 (4)	0·14803 (12)
H(3)	0·0073 (10)	−0·263 (4)	0·1742 (13)
H(4)	0·1639 (10)	−0·327 (4)	0·2776 (14)
H(5)	0·3454 (10)	−0·223 (4)	0·2754 (14)
H(6)	0·4714 (12)	−0·013 (5)	0·1784 (14)
H(7)	0·4354 (12)	0·256 (4)	−0·0048 (14)
H(8)	0·2739 (10)	0·291 (4)	−0·0975 (13)

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An isotropic extinction parameter was included in the final cycles of refinement as described by Coppens & Hamilton (1970). The final value of the extinction parameter was $0{\cdot}28 \times 10^{-4}$. Atomic scattering factors for C, N and O were obtained from *International Tables for X-ray Crystallography* (1962) and from Stewart, Davidson & Simpson (1965) for H. All H atoms were located from a difference Fourier map and were refined isotropically while the non-hydrogen atoms were refined anisotropically. The refinement converged at $R = 0{\cdot}065$ ($R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$), and $R_{F^2} = 0{\cdot}072$ ($R_{F^2} = \Sigma||F_o|^2 - |F_c|^2|/\Sigma|F_o|^2$) and $R_{wF^2} = 0{\cdot}118$ $\{R_{wF^2} = [\Sigma\, w(|F_o|^2 - |F_c|^2)^2/\Sigma\, |F_o|^4]^{1/2}\}$ for 1407 observations and 116 variables. The final positional parameters are given in Table 1.*

Discussion. The crystal structure of 2,2′-biquinolyl has been studied previously in this laboratory by Degeilh (1955) and Datta (1959) using Weissenberg techniques as a part of a continuing study of organic reagents of analytical importance and the complexes they form with metallic ions. Refinements were unsatisfactory because of large numbers of weak data and it was deemed desirable to redetermine the structure with more accurate diffractometer data at low temperature. The justification of the redetermination is shown not only in the higher precision obtained but also in the fact that we were able to collect 1407 unique data as opposed to only 475 obtained in the earlier studies.

Bond distances and angles are shown in Fig. 1, which also shows the deviations from the least-squares plane through the non-hydrogen atoms. The molecular plane is approximately perpendicular to **b**, with an angle of 26·3° between the plane normal and the **b** axis. The molecule consists of two quinoline systems linked by an sp^2–sp^2 single bond, and related by a center of symmetry. The two halves are coplanar and the configuration is *trans*. There is a short intramolecular

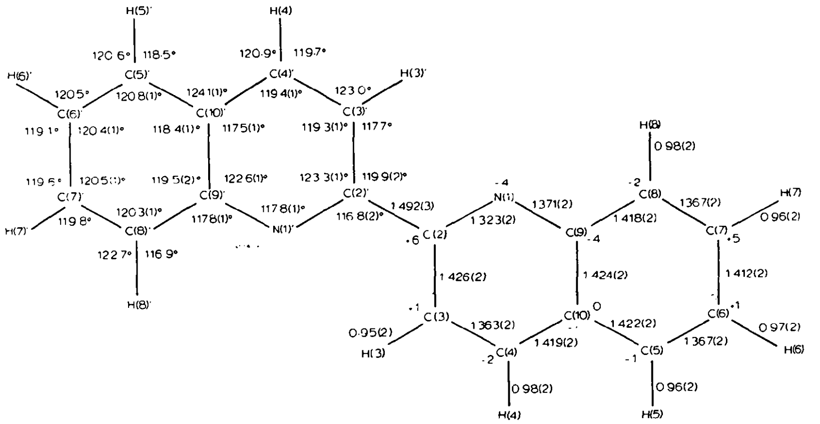

Fig. 1. Bond distances and angles in 2,2′-biquinolyl. The numbering scheme is indicated. Primed atoms are related to the unprimed by a center of symmetry. The atom deviations in Å (× 10^3) from the least-squares plane through the non-hydrogen atoms are indicated.

Table 2. *Comparison of bond lengths in* 2,2′-*biquinolyl and* 8,8′-*biquinolyl*

	2,2′	8,8′ (mean)	2,2′ (corrected)*	8,8′ (mean, corrected)*	Quinoline†
N(1)–C(2)	1·323 (2)	1·313	1·323	1·320	1·318
C(2)–C(3)	1·426 (2)	1·400	1·427	1·408	1·419
C(3)–C(4)	1·363 (2)	1·347	1·363	1·355	1·377
C(4)–C(10)	1·419 (2)	1·410	1·419	1·417	1·425
C(10)–C(5)	1·422 (2)	1·411	1·423	1·419	1·429
C(5)–C(6)	1·367 (2)	1·351	1·368	1·357	1·372
C(6)–C(7)	1·412 (2)	1·410	1·413	1·418	1·424
C(7)–C(8)	1·367 (2)	1·364	1·368	1·372	1·372
C(8)–C(9)	1·418 (2)	1·418	1·419	1·425	1·428
C(9)–N(1)	1·371 (2)	1·369	1·372	1·378	1·366
C(9)–C(10)	1·422 (2)	1·418	1·425	1·426	1·404

* A rigid-body analysis was made with the method of Schomaker & Trueblood (1968).
† The corresponding values in quinoline calculated by Dewar & Trinajstic (1971).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32805 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England. Supplementary data are also available, in microfiche form only, for $2.00 from Chemistry Department Library, Indiana University, Bloomington, Indiana 47401, USA. Request Molecular Structure Center Report No. 7611.

contact between H(3) and N(1′) of length 2·44 (1) Å. In the only reported crystal structure determination of a metal chelate of 2,2′-biquinolyl, $Au(C_{18}H_{12}N_2)Cl_3$, the biquinolyl group is planar but in the *cis* configuration (Charlton, Harris, Patil & Stephenson, 1966).

The C(2)–C(2′) bond length is 1·492 (3) Å, in excellent agreement with the corresponding values of 1·495 (2) Å in 8,8′-biquinolyl (Lenner & Lindgren, 1976), 1·493 (3) Å in biphenyl (Charbonneau & Delugeard, 1977) and also with the value of 1·50 Å observed in 2,2′-bipyridine (Merritt & Schroeder, 1956). Table 2 compares our bond lengths with those in 8,8′-biquinolyl. For comparison we also list the distances corrected for rigid-body thermal motion as well as the calculated distances in quinoline (Dewar & Trinajstic, 1971).

Normal probability plots comparing the bonds after the correction showed a significant improvement.

We thank the University Research Operations Committee at Indiana University for the financial support of this work and the Marshall H. Wrubel Computing Center for computing time.

References

Busing, W. R., Martin, K. O. & Levy, H. A. (1974). *XFLS*3. Oak Ridge National Laboratory, Tennessee.

Charbonneau, G. P. & Delugeard, Y. (1977). *Acta Cryst.* B**33**, 1586–1588.

Charlton, R. J., Harris, C. M., Patil, H. & Stephenson, N. C. (1966). *Inorg. Nucl. Chem. Lett.* **2**, 409–414.

Coppens, P. & Hamilton, W. C. (1970). *Acta Cryst.* A**26**, 71–83.

Datta, S. K. (1959). PhD Thesis, Indiana Univ.

Degeilh, R. (1955). PhD Thesis, Indiana Univ.

Dewar, M. J. S. & Trinajstic, N. (1971). *J. Chem. Soc.* (*A*), pp. 1220–1237.

Huffman, J. C. (1974). PhD Thesis, Indiana Univ.

International Tables for X-ray Crystallography (1962). Vol. III, pp. 201–246. Birmingham: Kynoch Press.

Lenner, M. & Lindgren, O. (1976). *Acta Cryst.* B**32**, 1903–1905.

Merritt, L. L. Jr & Schroeder, E. D. (1956). *Acta Cryst.* **9**, 801–804.

Schomaker, V. & Trueblood, K. N. (1968). *Acta Cryst.* B**24**, 63–76.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1977). B**33**, 3542–3545

Norcocaine Hydrobromide*

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(*Received* 8 *March* 1977; *accepted* 24 *June* 1977)

Abstract. $HBr[C_{16}H_{20}BrNO_4]$, (2*βR*-methoxycarbonyl-3*βS*-benzoyloxy-8-azabicyclo[3.2.1]octane), space group $P2_12_12_1$, a = 11·543 (8), b = 12·610 (9), c = 11·474 (8) Å, $Z = 4$, D_x = 1·472, D_m = 1·45 g cm^{-3} (flotation: CCl_4–C_6H_6). The structure was solved by the heavy-atom method. Full-matrix least-squares refinements converged to R = 0·072. The piperidine ring is in the chair conformation. The Br^- is hydrogen bonded to one N proton and lies over the pyrrolidine ring. The carbonyl O of the methoxycarbonyl moiety is intramolecularly hydrogen bonded to the other N proton which also hydrogen bonds intermolecularly with the carbonyl O of the benzoate group.

Introduction. Shen, Ruble & Hite (1975) suggested that the local anesthetic action of cocaine could be rationalized on the basis of the piperidine boat conformation since it superimposes more completely than the chair conformation upon other, more potent (Lokhandwala, Patel, Patel, Merker, Shafi'ee & Hite,

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* Stereochemical Aspects of Local Anesthetic Action. III. Part II: Shen, Ruble & Hite (1975).

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