Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)–N(2)	1·402 (3)	C(1)–C(12c)	1·426 (3)
C(1)–O(13)	1·224 (3)	N(2)–C(3)	1·369 (3)
N(2)–C(14)	1·470 (3)	C(3)–N(4)	1·387 (3)
C(3)–O(15)	1·221 (3)	N(4)–C(4a)	1·373 (3)
N(4)–C(16)	1·474 (4)	C(4a)–O(5)	1·338 (3)
C(4a)–C(12c)	1·351 (3)	O(5)–C(6)	1·448 (3)
C(6)–C(6a)	1·513 (4)	C(6a)–C(7)	1·512 (4)
C(6a)–C(12b)	1·527 (3)	C(7)–O(8)	1·421 (3)
O(8)–C(8a)	1·380 (3)	C(8a)–C(9)	1·389 (4)
C(8a)–C(12a)	1·400 (3)	C(9)–C(10)	1·360 (4)
C(10)–C(11)	1·375 (4)	C(11)–C(12)	1·385 (4)
C(12)–C(12a)	1·388 (3)	C(12a)–C(12b)	1·521 (4)
C(12b)–C(12c)	1·516 (3)		

N(2)–C(1)–C(12c)	116·2 (2)	N(2)–C(1)–O(13)	119·5 (2)
C(12c)–C(1)–O(13)	124·3 (2)	C(1)–N(2)–C(3)	124·8 (2)
C(1)–N(2)–C(14)	118·6 (2)	C(3)–N(2)–C(14)	116·6 (2)
N(2)–C(3)–N(4)	116·4 (2)	N(2)–C(3)–O(15)	122·5 (2)
N(4)–C(3)–O(15)	121·1 (2)	C(3)–N(4)–C(4a)	120·7 (2)
C(3)–N(4)–C(16)	119·1 (2)	C(4a)–N(4)–C(16)	120·2 (2)
N(4)–C(4a)–O(5)	111·6 (2)	N(4)–C(4a)–C(12c)	122·9 (2)
O(5)–C(4a)–C(12c)	125·5 (2)	C(4a)–O(5)–C(6)	117·0 (2)
O(5)–C(6)–C(6a)	112·6 (2)	C(6)–C(6a)–C(7)	109·9 (2)
C(6)–C(6a)–C(12b)	109·9 (2)	C(7)–C(6a)–C(12b)	109·8 (2)
C(6a)–C(7)–O(8)	112·2 (2)	C(7)–O(8)–C(8a)	115·8 (2)
O(8)–C(8a)–C(9)	116·4 (2)	O(8)–C(8a)–C(12a)	122·7 (2)
C(9)–C(8a)–C(12a)	120·9 (2)	C(8a)–C(9)–C(10)	120·4 (2)
C(9)–C(10)–C(11)	120·4 (3)	C(10)–C(11)–C(12)	119·2 (2)
C(11)–C(12)–C(12a)	122·3 (2)	C(8a)–C(12a)–C(12)	116·8 (2)
C(8a)–C(12a)–C(12b)	121·0 (2)	C(12)–C(12a)–C(12b)	122·2 (2)
C(6a)–C(12b)–C(12a)	110·4 (2)	C(6a)–C(12b)–C(12c)	108·7 (2)
C(12a)–C(12b)–C(12c)	114·2 (2)	C(1)–C(12c)–C(4a)	118·8 (2)
C(1)–C(12c)–C(12b)	119·9 (2)	C(42)–C(12c)–C(12b)	120·8 (2)

Related literature. For the preparation of the compound *via* an intramolecular hetero-Diels–Alder reaction see Tietze, Stegelmeir, Harms & Brumby (1982). For structures of related *trans*-fused Diels–Alder products see Tietze, von Kiedrowski, Harms, Clegg & Sheldrick (1980), Harms, Jones & Sheldrick (1980) and Clegg, Harms, Sheldrick, von Kiedrowski & Tietze (1980).

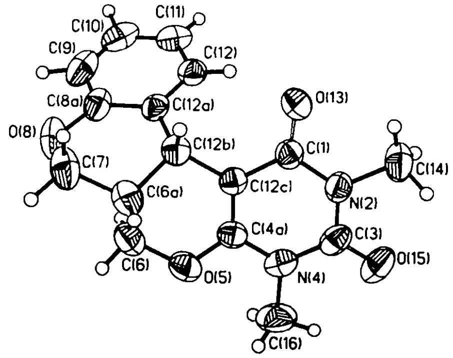

Fig. 1. The asymmetric unit of the title compound, showing the atom-numbering scheme.

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

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Acta Cryst. (1986). C**42**, 1665–1667

Structure of an Indolochinolizine Derivative

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(*Received* 29 *January* 1986; *accepted* 29 *April* 1986)

Abstract. Racemic (2*S*,3*S*,12b*R*)-2-(1,3-dimethyl-2,4,6-trioxo-1,3-diazacyclohexyl)-3-ethyl-1,2,3,4,5,6-hexahydro-12b*H*-indolo[2,3-*a*]quinolizinium chloride, $C_{23}H_{29}N_4O_3^+.Cl^-$, $M_r = 445·0$, orthorhombic, *Pbca*, $a = 14·462\ (2)$, $b = 17·405\ (1)$, $c = 17·299\ (2)$ Å, $V = 4354$ Å^3, $Z = 8$, $D_x = 1·357$ Mg m^{-3}, λ(Mo $K\alpha$) =

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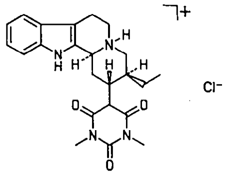
0·71069 Å, $\mu = 0{\cdot}21\ \mathrm{mm}^{-1}$, $F(000) = 1888$, $T = 298$ K, $R = 0{\cdot}075$ for 1959 observed reflections. The structure was investigated to determine the relative configuration, which could not be established by NMR. The protons bonded to N were located in the difference Fourier map and refined with a common temperature factor; there is a hydrogen bond [N(4)···Cl = 3·078 (7) Å] between the ions.

Experimental. (I): crystal size 0·3 × 0·4 × 0·2 mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). 4351 reflections measured, $2\theta_{\max}$ 45°, $\pm h + k \pm l$, three check reflections with no intensity change. 2842 unique reflections ($R_{\text{int}} = 0{\cdot}026$), of which 1959 with $F > 3\sigma(F)$ used for all calculations (program system *SHELXTL*, Sheldrick, 1978). Index ranges $|h| \leq 15$, $k \leq 18$, $|l| \leq 18$. Cell constants refined from $\pm 2\theta$ values of 35 reflections in range 20–25°. Absorption and extinction corrections unnecessary.



(I)

Structure solution by multisolution direct methods. Refinement on F to $R = 0{\cdot}075$, $wR = 0{\cdot}055$; all non-H atoms anisotropic, H atoms included using a riding model [C—H 0·96 Å, $U(\mathrm{H}) = 1{\cdot}2U_{\text{eq}}(\mathrm{C})$], 296 parameters, $S = 1{\cdot}51$, weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}00021F^2$ which gave a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0{\cdot}04$, max. and min. height in final $\Delta\rho$ map 0·32 and −0·33 e Å^{-3}

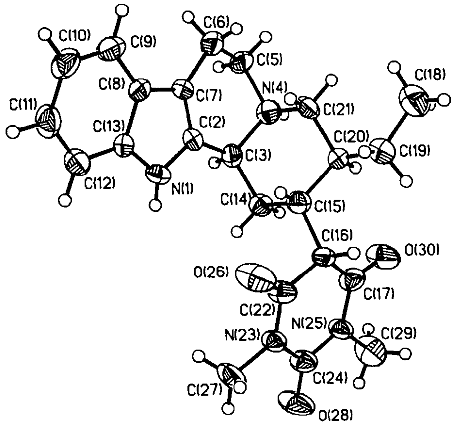


Fig. 1. The cation of the title compound, showing the atom-numbering scheme.

Table 1. *Atomic coordinates* (× 10⁴) *and equivalent isotropic thermal parameters* (Å² × 10³)

	x	y	z	U_{eq}*
Cl	9505 (1)	7714 (1)	6763 (1)	59 (1)
N(1)	4088 (3)	3178 (2)	5295 (2)	42 (1)
C(2)	4058 (3)	3085 (3)	4499 (2)	36 (2)
C(3)	4867 (3)	2855 (3)	4012 (2)	37 (2)
N(4)	4615 (3)	3047 (3)	3191 (2)	42 (2)
C(5)	3677 (3)	2736 (3)	2976 (2)	48 (2)
C(6)	2917 (3)	3149 (3)	3417 (2)	48 (2)
C(7)	3189 (3)	3226 (3)	4252 (2)	38 (2)
C(8)	2641 (3)	3426 (3)	4900 (2)	38 (2)
C(9)	1726 (3)	3653 (3)	5002 (3)	47 (2)
C(10)	1410 (3)	3814 (3)	5730 (3)	53 (2)
C(11)	1990 (4)	3711 (3)	6371 (3)	54 (2)
C(12)	2890 (4)	3499 (3)	6298 (3)	45 (2)
C(13)	3226 (3)	3372 (2)	5552 (2)	34 (2)
C(14)	5798 (3)	3217 (2)	4218 (3)	36 (2)
C(15)	5817 (3)	4077 (3)	4053 (2)	39 (2)
C(16)	6703 (3)	4465 (3)	4371 (2)	34 (2)
C(17)	7593 (3)	4165 (3)	4050 (3)	44 (2)
C(18)	5254 (4)	5272 (4)	2212 (3)	90 (3)
C(19)	5644 (4)	5056 (3)	2969 (3)	61 (2)
C(20)	5638 (3)	4206 (3)	3185 (2)	39 (2)
C(21)	4686 (3)	3883 (3)	3009 (2)	42 (2)
C(22)	6628 (3)	4519 (3)	5245 (2)	43 (2)
N(23)	7360 (2)	4257 (2)	5672 (2)	38 (1)
C(24)	8204 (3)	4016 (3)	5379 (3)	43 (2)
N(25)	8280 (2)	3986 (2)	4576 (2)	41 (1)
O(26)	5950 (2)	4773 (2)	5552 (2)	71 (1)
C(27)	7274 (4)	4289 (4)	6520 (2)	61 (2)
O(28)	8827 (2)	3832 (2)	5786 (2)	72 (1)
C(29)	9188 (3)	3770 (4)	4270 (3)	73 (2)
O(30)	7735 (2)	4089 (2)	3366 (2)	71 (2)

* U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°)

N(1)—C(2)	1·388 (5)	N(1)—C(13)	1·367 (6)
C(2)—C(3)	1·496 (6)	C(2)—C(7)	1·350 (6)
C(3)—N(4)	1·505 (6)	C(3)—C(14)	1·528 (6)
N(4)—C(5)	1·507 (6)	N(4)—C(21)	1·492 (6)
C(5)—C(6)	1·518 (6)	C(6)—C(7)	1·502 (6)
C(7)—C(8)	1·417 (6)	C(8)—C(9)	1·393 (6)
C(8)—C(13)	1·413 (6)	C(9)—C(10)	1·368 (6)
C(10)—C(11)	1·401 (7)	C(11)—C(12)	1·359 (7)
C(12)—C(13)	1·396 (6)	C(14)—C(15)	1·523 (6)
C(15)—C(16)	1·550 (6)	C(15)—C(20)	1·540 (6)
C(16)—C(17)	1·496 (6)	C(16)—C(22)	1·519 (6)
C(17)—N(25)	1·383 (6)	C(17)—O(30)	1·208 (5)
C(19)—C(18)	1·475 (7)	C(19)—C(20)	1·525 (7)
C(20)—C(21)	1·518 (6)	C(22)—N(23)	1·370 (5)
C(22)—O(26)	1·199 (5)	N(23)—C(24)	1·386 (5)
N(23)—C(27)	1·472 (5)	C(24)—N(25)	1·393 (5)
C(24)—O(28)	1·188 (5)	N(25)—C(29)	1·465 (6)
C(2)—N(1)—C(13)	108·9 (3)	N(1)—C(2)—C(3)	124·5 (4)
N(1)—C(2)—C(7)	108·8 (4)	C(3)—C(2)—C(7)	126·7 (4)
C(2)—C(3)—N(4)	106·4 (3)	C(2)—C(3)—C(14)	116·6 (4)
N(4)—C(3)—C(14)	110·0 (3)	C(3)—N(4)—C(5)	111·8 (3)
C(3)—N(4)—C(21)	113·5 (3)	C(5)—N(4)—C(21)	111·1 (3)
N(4)—C(5)—C(6)	111·0 (4)	C(5)—C(6)—C(7)	109·6 (4)
C(2)—C(7)—C(6)	122·1 (4)	C(2)—C(7)—C(8)	108·3 (4)
C(6)—C(7)—C(8)	129·5 (4)	C(7)—C(8)—C(9)	134·6 (4)
C(7)—C(8)—C(13)	106·3 (4)	C(9)—C(8)—C(13)	119·1 (4)
C(8)—C(9)—C(10)	119·5 (4)	C(9)—C(10)—C(11)	120·1 (4)
C(10)—C(11)—C(12)	122·4 (4)	C(11)—C(12)—C(13)	117·5 (4)
N(1)—C(13)—C(8)	107·6 (3)	N(1)—C(13)—C(12)	131·1 (4)
C(8)—C(13)—C(12)	121·3 (4)	C(3)—C(14)—C(15)	112·2 (3)
C(14)—C(15)—C(16)	112·2 (3)	C(14)—C(15)—C(20)	108·8 (3)
C(16)—C(15)—C(20)	114·9 (3)	C(15)—C(16)—C(17)	115·3 (3)
C(15)—C(16)—C(22)	108·7 (3)	C(17)—C(16)—C(22)	116·9 (3)
C(16)—C(17)—N(25)	116·9 (4)	C(16)—C(17)—O(30)	123·2 (4)
N(25)—C(17)—O(30)	119·9 (4)	C(18)—C(19)—C(20)	117·6 (4)
C(15)—C(20)—C(19)	112·3 (4)	C(15)—C(20)—C(21)	107·2 (3)
C(19)—C(20)—C(21)	108·3 (4)	N(4)—C(21)—C(20)	112·4 (4)
C(16)—C(22)—N(23)	117·5 (4)	C(16)—C(22)—O(26)	121·4 (4)
N(23)—C(22)—O(26)	121·1 (4)	C(22)—N(23)—C(24)	125·7 (3)
C(22)—N(23)—C(27)	117·3 (4)	C(24)—N(23)—C(27)	116·8 (3)
N(23)—C(24)—N(25)	116·5 (4)	N(23)—C(24)—O(28)	122·1 (4)
N(25)—C(24)—O(28)	121·3 (4)	C(17)—N(25)—C(24)	126·1 (4)
C(17)—N(25)—C(29)	117·6 (4)	C(24)—N(25)—C(29)	116·2 (4)

respectively. Atomic scattering factors from *SHELXTL*.

The main cause of the relatively high R index was probably the poor quality of the crystal. This is indicated by the unusually high value of $R_{\rm sigma} = \sum\sigma(F)/\sum F = 0{\cdot}061$.

Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows the atom numbering.

Related literature. For the preparation of the compound see Schul (1984). For a recent structure of an indole derivative see Sawyer, Shariff & McLean (1985).

We thank the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie for financial support.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43054 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1986). C**42**, 1667–1669

Neutron powder time-of-flight Rietveld refinement and H-atom location in $NaNi_2(OH)(H_2O)(MoO_4)_2$.

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(*Received* 19 *October* 1985; *accepted* 16 *April* 1986)

Abstract

A new nickel sodium molybdate, $NaNi_2(OH)(H_2O)(MoO_4)_2$, has been synthesized by the reaction of $NiCl_2.6H_2O$ and $Na_2MoO_4.2H_2O$. $M_r = 495{\cdot}31$, $C2/m$, $a = 9{\cdot}3896\,(4)$, $b = 6{\cdot}2928\,(3)$, $c = 7{\cdot}5727\,(3)$ Å, $\beta = 116{\cdot}080\,(4)°$, $V = 401{\cdot}89\,(2)$ Å^3, $Z = 2$, $D_x = 4{\cdot}092$, $T = 15$ K, microcrystalline powder, time-of-flight neutron data, Rietveld refinement 0·64–2·61 Å (150° data banks), 848 contributing reflections, $R_{wp} = 0{\cdot}0193$, $R_p = 0{\cdot}1275$, $R_e = 0{\cdot}0101$. The H atoms were located by difference Fourier techniques and closely agree with those found on an isomorphous compound by X-ray single-crystal methods.

Introduction

Reactions involving alkali-metal molybdates and salts of transition metals in aqueous solutions result in the formation of simple and mixed metal molybdate phases which are of interest because of their catalytic and magnetic properties in addition to their interesting structural characteristics (Van Uitert, Sherwood, Williams, Rubin & Ronner, 1964; Rajaram, Viswanathan, Sastri & Srinivasan, 1974). These molybdates tend to form in basic solutions and are usually micorcrystalline. Two single-crystal studies have been completed. One such phase, $NaZn_2(OH)(H_2O)(MoO_4)_2$ (Clearfield, Sims & Ramanathan, 1976), is also found to form when the transition metal is Co or Mn. We recently completed the X-ray single-crystal structure determination of $NaMn_2(OH)(H_2O)(MoO_4)_2$ which was found to be isostructural with the zinc phase (Clearfield, Moini & Rudolf, 1985). In this latter study we were able to locate peaks in the difference Fourier map that were assigned to H atoms and concluded that there are only two crystallographically unique H atoms in the structure with one of them occupying half of the available sites. This results in the presence of water and hydroxyl groups on alternating sites. The crystals used in the X-ray studies were exceedingly small and the absorption coefficient was large (*ca* 60 cm^{-1}). So it was of interest to verify the X-ray determination of H-atom placements. Our investigation of this matter led to the preparation of

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