Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B_{eq}*/B† ($Å^2$)
N(1)	0·5001 (3)	0·2500 (4)	0·1276 (4)	3·5
C(2)	0·7074 (4)	0·3449 (3)	0·1989 (5)	3·5
C(3)	0·2925 (4)	0·3442 (4)	−0·0071 (5)	3·5
C(4)	0·7580 (4)	0·4721 (4)	0·4226 (6)	3·8
C(5)	0·2420 (4)	0·4714 (4)	0·1648 (5)	3·7
N(6)	0·7926 (4)	0·5646 (4)	0·5998 (5)	5·0
N(7)	0·2054 (4)	0·5642 (4)	0·3058 (6)	5·2
H(1)	0·496 (4)	0·201 (5)	0·264 (8)	3·4†
H(2A)	0·855 (5)	0·275 (4)	0·261 (7)	3·5†
H(2B)	0·693 (6)	0·406 (4)	0·050 (8)	3·5†
H(3A)	0·129 (5)	0·256 (4)	−0·078 (7)	3·5†
H(3B)	0·294 (6)	0·405 (4)	−0·149 (7)	3·5†

* As defined by Hamilton (1959).

Table 2. *Bond distances (Å) and bond angles (°) with e.s.d.'s in parentheses*

N(1)—C(2)	1·444 (4)	N(1)—C(3)	1·442 (4)
C(2)—C(4)	1·485 (4)	C(3)—C(5)	1·480 (4)
C(4)—N(6)	1·139 (4)	C(5)—N(7)	1·139 (5)

C(2)—N(1)—C(3)	116·6 (3)	N(1)—C(2)—C(4)	115·5 (3)
N(1)—C(3)—C(5)	115·7 (3)	C(2)—C(4)—N(6)	176·9 (4)
C(3)—C(5)—N(7)	177·0 (4)		

number of observations per refined parameter is $390/85 = 4·59$ and $S = 2·17$. The final R and wR values are 0·051 and 0·066, respectively. $(\Delta/\sigma)_{max}$ of non-H atoms in the final refinement cycle is 0·029. The peaks in the final $\Delta\rho$ map were between 0·35 and $-0·17 \, e \, Å^{-3}$. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The final atomic parameters are listed in Table 1.* The molecular structure with atomic numbering system and the crystal pack-

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* Lists of anisotropic temperature factors for non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53031 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

ing diagram are depicted in Figs. 1 and 2, respectively. Bond distances and angles are presented in Table 2.

All computations were performed on an ACOS 930 computer at the Research Center for Protein Engineering, Institute for Protein Research, Osaka University.

Related literature. A modified and improved preparation of the title compound has recently been reported (Micovic & Ivanovic, 1986).

References

ASHIDA, T. (1979). *HBLSV. The Universal Crystallographic Computing System – Osaka*, pp. 53–59. Computation Center, Osaka Univ., Japan.
HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.
JOHNSON, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
MICOVIC, I. V. & IVANOVIC, M. D. (1986). *J. Serb. Chem. Soc.* **51**, 435–439.
SHELDRICK, G. M. (1986). *SHELXS86.* Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

N-Methyl-N-(2-phenyl-4-oxocyclobutyl)-p-toluenesulfonamide

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Abstract. $C_{18}H_{19}NO_3S$, $M_r = 329·42$, monoclinic, $P2_1/n$, $a = 10·573$ (5), $b = 12·437$ (6), $c = 13·045$ (6) Å, $\beta = 106·84$ (4)°, $V = 1642$ (1) $Å^3$, $Z = 4$, $D_x = 1·33 \, g \, cm^{-3}$, $\lambda(Cu \, K\alpha) = 1·5418$ Å, $\mu = 18·24 \, cm^{-1}$, $F(000) = 696$, $T = 291$ K, $R = 0·051$ for 2154 observed reflections. The puckering (α) of the cyclobutanone ring, *i.e.* the dihedral angle between the planes defined by C1, C2, C3 and C1, C2, C4, is 14·8 (8)°. The substituents are *trans* to each other in a pseudoequatorial arrangement. An *endo* deviation

Table 1. Atomic coordinates (× 10⁴) and equivalent isotropic temperature factors (Å²)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Cl	−1867 (3)	4148 (3)	−1468 (3)	4·41 (6)
C2	−574 (3)	3532 (2)	−1027 (2)	3·47 (5)
C3	−300 (3)	3753 (3)	−2133 (2)	3·64 (5)
C4	−1408 (3)	4604 (3)	−2370 (3)	4·62 (6)
O5	−2865 (3)	4185 (3)	−1223 (3)	7·06 (6)
N6	−618 (3)	2473 (2)	−608 (2)	3·96 (4)
C7	−1198 (4)	1616 (3)	−1362 (3)	5·23 (7)
S8	534 (1)	2132 (1)	486 (1)	3·96 (1)
O9	184 (3)	1098 (2)	788 (2)	5·15 (5)
O10	668 (3)	3018 (2)	1208 (2)	5·27 (5)
C11	2021 (3)	2029 (2)	157 (2)	3·50 (5)
C12	2891 (3)	2881 (3)	334 (3)	4·19 (5)
C13	4033 (3)	2789 (3)	33 (3)	4·56 (6)
C14	4328 (3)	1866 (3)	−432 (3)	4·59 (6)
C15	3446 (3)	1021 (3)	−601 (3)	4·58 (6)
C16	2294 (3)	1088 (3)	−311 (3)	4·19 (5)
C17	5566 (5)	1777 (5)	−761 (5)	6·44 (9)
C18	1075 (3)	4061 (3)	−2113 (2)	3·89 (5)
C19	1660 (3)	4984 (3)	−1606 (3)	4·90 (6)
C20	2931 (4)	5242 (4)	−1590 (4)	6·95 (9)
C21	3623 (4)	4591 (6)	−2086 (5)	8·19 (12)
C22	3051 (5)	3688 (5)	−2586 (4)	7·18 (11)
C23	1781 (4)	3407 (3)	−2605 (3)	5·10 (7)

of 0·065 Å of the carbonyl oxygen from the plane Cl C2 C4 corresponds to an angle $\beta = 3 \cdot 0°$ made by the C=O vector with this plane.

Table 2. Bond distances (Å) and angles (°)

C2—Cl	1·526 (4)	C4—Cl	1·507 (5)
O5—Cl	1·189 (4)	C3—C2	1·577 (4)
N6—C2	1·433 (4)	C4—C3	1·542 (4)
C18—C3	1·495 (4)	C7—N6	1·459 (4)
S8—N6	1·641 (3)	O9—S8	1·424 (2)
O10—S8	1·430 (2)	C11—S8	1·750 (3)
C12—C11	1·379 (4)	C16—C11	1·388 (4)
C13—C12	1·379 (5)	C14—C13	1·376 (5)
C15—C14	1·379 (5)	C17—C14	1·496 (5)
C16—C15	1·379 (5)	C19—C18	1·379 (5)
C23—C18	1·382 (5)	C20—C19	1·376 (5)
C21—C20	1·372 (9)	C22—C21	1·351 (8)
C23—C22	1·381 (6)		

C4—Cl—C2	91·6 (2)	O5—Cl—C2	133·3 (3)
O5—Cl—C4	134·9 (3)	C3—C2—Cl	87·1 (2)
N6—C2—Cl	118·8 (2)	N6—C2—C3	123·0 (2)
C4—C3—C2	88·4 (2)	C18—C3—C2	117·7 (2)
C18—C3—C4	120·4 (3)	C3—C4—Cl	89·0 (2)
C7—N6—C2	117·9 (3)	S8—N6—C2	118·2 (2)
S8—N6—C7	117·3 (2)	O9—S8—N6	106·7 (1)
O10—S8—N6	106·3 (1)	O10—S8—O9	119·8 (1)
C11—S8—N6	107·4 (1)	C11—S8—O9	109·0 (1)
C11—S8—O10	107·0 (1)	C12—C11—S8	120·4 (2)
C16—C11—S8	119·0 (2)	C16—C11—C12	120·5 (3)
C13—C12—C11	119·0 (3)	C14—C13—C12	121·6 (3)
C15—C14—C13	118·5 (3)	C17—C14—C13	121·1 (4)
C17—C14—C15	120·4 (4)	C16—C15—C14	121·3 (3)
C15—C16—C11	119·0 (3)	C19—C18—C3	121·4 (3)
C23—C18—C3	119·6 (3)	C23—C18—C19	119·0 (3)
C20—C19—C18	120·2 (4)	C21—C20—C19	120·5 (5)
C22—C21—C20	119·6 (4)	C23—C22—C21	121·0 (5)
C22—C23—C18	119·8 (5)		

Experimental. Since stereochemical assignments based on ¹H NMR coupling constants are ambiguous for the title compound, an X-ray analysis was undertaken in order to assess the relative configuration. Crystals were obtained by evaporation from cyclohexane. D_m not measured. Parallelepiped crystal with dimensions $0 \cdot 12 \times 0 \cdot 16 \times 0 \cdot 20$ mm. Lattice parameters refined using 30 reflections in the range $5° \le 2\theta \le 50°$. Huber four-circle diffractometer, monochromatized Cu $K\alpha$ radiation. 2974 h $k \pm l$ independent reflections with $\sin\theta/\lambda \le 0 \cdot 60$ Å⁻¹; $0 \le h \le 12$, $0 \le k \le 14$, $-15 \le l \le 14$, 2154 with $I \ge 2 \cdot 5\sigma(I)$. Standard reflection (145) checked every 50 reflections: no significant deviation. Structure solved by SHELXS86 (Sheldrick, 1985). All H atoms from difference Fourier synthesis. Anisotropic least-squares refinement (SHELX76, Sheldrick, 1976) using F; H isotropic with common refined temperature factor. $w = 1/(\sigma^2 + 0 \cdot 00689F^2)$. $R = 0 \cdot 051$, $wR = 0 \cdot 061$ for 2154 observed reflections. Final maximum shift/e.s.d. $= 0 \cdot 12$ [y of O(5)]. $S = 0 \cdot 88$. Maximum and minimum heights in final difference Fourier synthesis $= 0 \cdot 19$ and $-0 \cdot 47$ e Å⁻³. Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV).

The atomic parameters are given in Table 1;* bond lengths and bond angles are listed in Table 2. Fig. 1

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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 53114 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
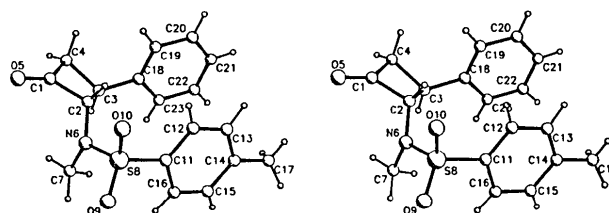

Fig. 1. Stereoscopic view of the molecule.

is a stereoscopic view of the molecule, showing the numbering of the atoms (PLUTO, Motherwell & Clegg, 1978).

Related literature. We have recently reported the structure of 8-(N-methyl-N-tolylsulfonylamino)bicyclo[4.2.0]octan-7-one ($\alpha = 23$, $\beta = 9°$ in molecule A, $\alpha = 21$, $\beta = 7°$ in molecule B) (Tinant, Declercq & Gobeaux, 1990). Very few structures of monocyclic cyclobutanones have been reported (Allen et al., 1979; $R < 10\%$): 2-chloro-4-(2,2,2-trichloroethyl)-3,3-dimethyl- ($\alpha = 32$, $\beta = 11°$) (Martin, Greuter, Rihs, Winkler & Bellus, 1981), 2,2,4,4-tetramethyl-3-pyrrolidino- ($\alpha = 19$, $\beta = 3°$) (Balbach, Alt & Ziegler, 1982) and 2,2-dichloro-3-phenylcyclobutanone ($\alpha = 12$, $\beta = 2°$) (Hoge, Jager & Fischer, 1977).

References

ALLEN, F. H., BELLARD, S., BRICE, M. D., CARTWRIGHT, B. A., DOUBLEDAY, A., HIGGS, H., HUMMELINK, T., HUMMELINK-PETERS, B. G., KENNARD, O., MOTHERWELL, W. D. S., RODGERS, J. R. & WATSON, D. G. (1979). Acta Cryst. B35, 2331–2339.
BALBACH, B., ALT, R. & ZIEGLER, M. L. (1982). Z. Naturforsch. Teil B, 37, 1005–1013.

HOGE, R., JAGER, J. & FISCHER, K. (1977). *Cryst. Struct. Commun.* pp. 287–290.

MARTIN, P., GREUTER, H., RIHS, G., WINKLER, T. & BELLUS, D. (1981). *Helv. Chim. Acta*, **64**, 2571–2586.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO. Program for plotting molecular and crystal structures.* Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX76. Program for crystal structure determination.* Univ. of Cambridge, England.

SHELDRICK, G. M. (1985). *SHELXS86.* In *Crystallographic Computing 3*, edited by G. M. SHELDRICK, C. KRUGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.

TINANT, B., DECLERCQ, J.-P. & GOBEAUX, B. (1990). *Acta Cryst.* **C46**, 1944–1946.

SHORT COMMUNICATION

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Révision de la structure de NaNp₃F₁₃. Par ALAIN COUSSON, *Institut Curie, Section de Physique et Chimie, UA CNRS 448, 11 rue P. et M. Curie, 75231 Paris CEDEX 05, France* et MADELEINE GASPERIN, *Laboratoire de Minéralogie-Cristallographie associé au CNRS, Université P. et M. Curie, 4 place Jussieu, 75230 Paris CEDEX 05, France*

Abstract. The crystal structure of NaNp₃F₁₃ has been re-examined; the formula of this compound is in fact Na₄Np₁₂F₄₆O₃. $M_r = 3858.0$, hexagonal, $P6_3/mmc$, $a = 8.022 (5)$, $c = 16.513 (9)$ Å, $V = 920 (2)$ Å³, $Z = 1$, $D_x = 6.96$ Mg m⁻³, λ(Mo $K\bar{\alpha}$) = 0.71069 Å, $\mu = 25.9$ mm⁻¹, $F(000) = 1598$, $T = 290$ K, $R = 0.054$, $wR = 0.060$ for 599 independent reflections with $I \geq 3\sigma(I)$.

A la lumière d'un récent article concernant Li₃ThF₇ (Lalignant, Le Bail, Avignant, Cousseins & Ferey, 1989), il nous est apparu que la structure de NaNp₃F₁₃ décrite en 1983 (Cousson, Abazli, Pagès & Gasperin, 1983) était certainement erronée. En effet, l'un des atomes de fluor [F(7)], lié seulement à deux atomes de sodium, ne respectait pas la loi de répartition des charges électrostatiques alors qu'à l'inverse, l'atome F(5), lié à trois atomes de neptunium, recevait une charge trop importante.

Nous avons donc repris les affinements en attribuant à F(7) la table de diffusion du sodium, et à F(5) celle de l'oxygène. L'introduction d'un second atome de sodium est d'ailleurs en accord avec le fait que, dans la première description, Na avait un facteur de température élevé compatible avec celle d'un site partiellement occupé.

L'introduction de ces deux changements et la variation des multiplicateurs de Na(1), Na(2) et O aboutissent à la formule Na₄Np₁₂F₄₆O₃. Le facteur R s'abaisse de 0,066 à 0,054. $wR = 0,060.$*

Les positions atomiques, qui ont peu varié, figurent dans le Tableau 1 et les distances cations–anions dans le Tableau 2. La balance des charges, calculée par le programme

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* Les listes des facteurs de structure et des paramètres thermiques anisotropes ont été déposées au dépôt d'archives de la British Library Document Supply Centre (Supplementary Publication No. SUP 52965: 6 pp.). On peut en obtenir des copies en s'adressant à: The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

Tableau 1. *Positions atomiques* ($\times 10^4$) *et paramètres d'agitation thermique isotropes ou équivalents*

$$B_{eq} = (4/3)\Sigma_i \Sigma_j \beta_{ij} \mathbf{a}_i . \mathbf{a}_j.$$

	Taux d'occupation du site	x	y	z	B
Np (B_{eq})		1621 (1)	3241 (1)	1128 (1)	0,90 (2)
Na(1)	0·66	3333	6667	9145 (16)	1,2 (4)
Na(2)	0·72	6667	3333	2500	2,1 (7)
F(1)		1394 (15)	2787 (15)	2500	1,4 (3)
F(2)		5376 (11)	0751 (11)	8361 (9)	1,5 (2)
F(3)		3286 (23)	0	0	1,7 (2)
F(4)		3333	6667	0532 (17)	1,8 (4)
F(5)		1657 (14)	3313 (14)	8532 (8)	1,7 (2)
O	0·75	0	0	0840 (23)	0,9 (5)

Tableau 2. *Distances dans les polyèdres* (Å)

Np—F(3)	2,282 (6) ×2	Np—F(2)	2,307 (10) ×2
Np—F(1)	2,288 (6)	Np—F(5)	2,345 (10) ×2
Np—O	2,301 (8)	Np—F(4)	2,575 (10)
Na(1)—F(4)	2,291 (38)	Na(1)—F(5)	2,540 (15) ×3
Na(2)—F(5)	2,886 (11) ×6		

LATSUM (Pannetier, 1980) est maintenant tout à fait satisfaisante. Les deux positions statistiques du sodium sont au voisinage de celle du Cs dans CsU₆F₂₅, ce qui explique l'analogie cristallographique des deux composés.

Références

COUSSON, A., ABAZLI, H., PAGÈS, M. & GASPERIN, M. (1983). *Acta Cryst.* **C39**, 318–320.

LALIGNANT, Y., LE BAIL, A., AVIGNANT, D., COUSSEINS, J. C. & FEREY, G. (1989). *J. Solid State Chem.* **80**, 206–212.

PANNETIER, J. (1980). *Bull. Minéral.* **103**, 367–375.