(II)

C2—C1—C5	104.1 (2)	C6—C5—C9	103.5 (2)
C2—C1—C11	110.4 (2)	C5—C6—C7	105.0 (2)
C2—C1—C14	112.1 (2)	O2—C7—C6	124.1 (3)
C5—C1—C11	106.6 (2)	O2—C7—C8	125.7 (3)
C5—C1—C14	118.6 (2)	C6—C7—C8	110.2 (2)
C11—C1—C14	105.0 (2)	C7—C8—C9	104.7 (3)
O1—C2—O1	124.1 (3)	C5—C9—C8	106.4 (3)
O1—C2—C3	124.8 (3)	C5—C9—C10	102.6 (2)
C1—C2—C3	111.1 (2)	C8—C9—C10	115.3 (3)
C2—C3—C4	104.1 (2)	C9—C10—C11	104.3 (2)
C3—C4—C5	105.3 (3)	C1—C11—C10	104.4 (2)
C1—C5—C4	105.9 (2)	C1—C11—C12	105.5 (2)
C1—C5—C6	118.7 (2)	C10—C11—C12	115.4 (2)
C1—C5—C9	104.0 (2)	C11—C12—C13	104.3 (3)
C4—C5—C6	111.7 (3)	C12—C13—C14	103.2 (2)
C4—C5—C9	113.0 (2)	C1—C14—C13	105.5 (2)

(III)

C2—O2—C9	113.1 (2)	C4—C8—C7	103.5 (2)
C2—C1—C11	114.4 (2)	C4—C8—C9	114.5 (2)
C2—C1—C14	114.0 (2)	C7—C8—C9	116.1 (2)
C11—C1—C14	103.1 (2)	O2—C9—C8	110.1 (2)
O1—C2—O2	105.6 (2)	O2—C9—C10	108.0 (2)
O1—C2—C1	106.3 (2)	O2—C9—C15	104.1 (2)
O1—C2—C3	110.2 (2)	C8—C9—C10	113.1 (2)
O2—C2—C1	110.2 (2)	C8—C9—C15	112.1 (2)
O2—C2—C3	107.8 (2)	C10—C9—C15	109.0 (2)
C1—C2—C3	116.2 (2)	C9—C10—C11	115.5 (2)
C2—C3—C4	114.6 (2)	C1—C11—C10	110.2 (2)
C3—C4—C5	112.0 (2)	C1—C11—C12	103.5 (2)
C3—C4—C8	110.8 (2)	C10—C11—C12	111.6 (2)
C5—C5—C8	103.9 (2)	C11—C12—C13	106.3 (3)
C4—C5—C6	106.4 (3)	C12—C13—C14	107.6 (3)
C5—C6—C7	109.0 (3)	C1—C14—C13	105.5 (2)
C6—C7—C8	106.0 (3)		

The programs used were *UNICS*III (Sakurai & Kobayashi, 1979) and *ORTEP* (Johnson, 1965). Calculations were performed on a FACOM M780/10 computer at Keio University.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55532 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1019]

References

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

Okumoto, S., Ohba, S., Saito, Y., Ishii, T., Umehara, M. & Hishida, S. (1987). *Acta Cryst.* C**43**, 1584–1587.

Okumoto, S., Ohba, S., Saito, Y., Umehara, M. & Hishida, S. (1988). *Acta Cryst.* C**44**, 1275–1279.

Sakurai, T. & Kobayashi, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

Umehara, M., Hishida, S., Okuda, M., Ohba, S., Ito, M., Saito, Y. & Zen, S. (1990). *Bull. Chem. Soc. Jpn*, **63**, 2002–2009.

Umehara, M., Honnami, H., Hishida, S., Kawata, T., Ohba, S. & Zen, S. (1992). *Bull. Chem. Soc. Jpn*, **65**. In the press.

Acta Cryst. (1993). C**49**, 413–416

Structure of a 6-(Tetrahydroxybutyl)-Substituted Pteridine

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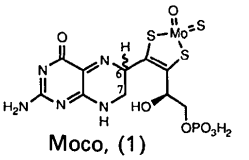
Abstract

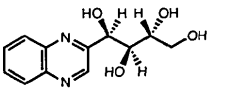
The structure of 2-acetamido-3,4-dihydro-4-oxo-6-(D-*arabino*-tetrahydroxybutyl)pteridine tetraacetate shows that reaction of 2,5,6-triaminopyrimidin-4-one with 1-*p*-toluidino-1-deoxyfructose can be utilized for a convenient method of preparing pteridines carrying a functionalized C_4 side chain at C6.

Comment

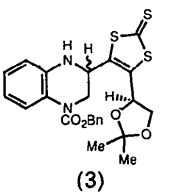
We are developing a synthetic route (Rowe, Garner & Joule, 1985; Larsen, Garner & Joule, 1989; Larsen, Rowe, Garner & Joule, 1989; Russell, Garner & Joule, 1992*a*,*b*) to Moco (Rajagopalan, 1991; Johnson, 1980; Gardlik & Rajagopalan, 1990) (1), the cofactor of all molybdoenzymes except nitrogenase. The cofactor is composed of a dihydropteridine carrying a functionalized side chain at C6 on which are situated the S atoms that coordinate the metal centre; any synthetic plan therefore requires an unambiguous route to a pteridine carrying a heavily functionalized C_4 side chain at C6. In model work, (tetrahydroxybutyl)quinoxaline (2) proved to be a very convenient, easily accessed and useful intermediate; we were able, for example, to transform it into (3). It was obvious that we should attempt to extrapolate this principle and look to the use of an analogous pteridine, (4*a*).



Moco, (1)



(2)

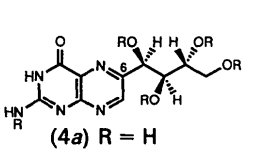


(3)

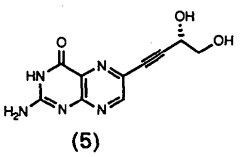
0108-2701/93/020413-04$06.00

Quinoxaline (2) is conveniently synthesized following the long-known reaction of sucrose with 1,2-diaminobenzene (Gries & Harrow, 1887); extrapolation of this method into the pteridine series (Forest & Walker, 1949; Weygand, Simon, Keil & Millauer, 1964) requires the use of 2,5,6-triaminopyrimidin-4-one, a non-symmetrical 1,2-diamine, and the expectation that a mixture of 6- and 7-substituted isomers would be formed. Difficulties in the unambiguous synthesis of 6- and 7-substituted pteridines (Forest & Walker, 1949) using reactions of 5,6-diaminopyrimidines led to the development of an alternative strategy (Taylor, Perlman, Sword, Séquin-Frey & Jacobi, 1973), now well established, in which a 6-halopteridine can be constructed unambiguously by building the pyrimidine ring on a precursor, previously synthesized pyrazine. Taylor and co-workers have demonstrated (Taylor, Ray, Darwish, Johnson & Rajagopalan, 1989; Taylor & Goswami, 1991) how this route which uses a palladium-catalyzed coupling to insert a functionalized C_4 unit, can furnish alkyne (5). Alternatively, 6-substituted pteridines can be produced (Baur, Sugimoto & Pfleiderer, 1988) *via* free-radical acylation of 7-alkylthiopteridines; these first need to be constructed and subsequently require hydrogenolytic removal of the sulfur substitutent.

Condensation (following Weygand, Wacker & Schmied-Kowarzik, 1949) of 2,5,6-triaminopyrimidin-4-one sulfate with the hydrazone of 1-*p*-toluidino-1-deoxyfructose (Weygand, 1940) provided a 4:1 isomeric mixture (^{1}H NMR) of tetrols which (for convenience of handling, purification and the preparation of a crystalline sample) was acetylated with pyridine and acetic anhydride at 373 K giving the tetraacetate acetamide of the major component, completely free from the other isomer. The X-ray analysis detailed below showed the product to have the structure (4*b*) with the side chain located at C6, as desired.



(4*a*) R = H
(4*b*) R = Ac



(5)

The atomic parameters for the non-H atoms of 2-acetamido-3,4-dihydro-4-oxo-6-(D-*arabino*-tetrahydroxybutyl)pteridine tetraacetate (4*b*) are listed in Table 1 and selected bond lengths and bond angles in Table 2. Fig. 1 is a *PLUTO* (Motherwell & Clegg, 1978) drawing of the molecule showing the numbering system used in the tables. Crystallographic methods have been employed previously to verify the C6 location of side chains on natural pteridines, for example folic acid (Camerman, Mastropaolo & Camerman, 1980), euglenapterin (Böhme, Hutzenlaub, Richter, Elstner, Huttner, von Seyerl & Pfleiderer, 1986) and the drug methotrexate (Hambley, Chan & Gonda, 1986; Sutton, Cody & Smith, 1986). This is the first occasion on which crystallography has been used to establish unambiguously the orientation of condensation of a 5,6-diaminopyrymidine with a 1,2-dicarbonyl compound (or equivalent).

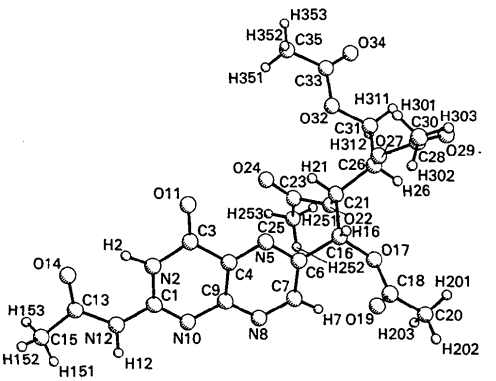


Fig. 1. *PLUTO* drawing of (4*b*) showing numbering scheme.

Experimental

Crystal data

$C_{20}H_{23}N_5O_{10}$
M_r = 493.43
Monoclinic
*C*2
a = 22.805 (1) Å
b = 15.156 (1) Å
c = 8.004 (2) Å
β = 91.66 (1)°
V = 2765.2 (7) Å^3
Z = 4
D_x = 1.185 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 22 reflections
θ = 39.5–40°
μ = 0.79 mm^{-1}
T = 296 (1) K
Prismatic
0.280 × 0.200 × 0.150 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical (*DIFABS*; Walker & Stuart, 1983)
T_{min} = 0.86, T_{max} = 1.12
2198 measured reflections
2138 independent reflections
1984 observed reflections [$I>3.0\sigma(I)$]
R_{int} = 0.016
θ_{max} = 60°
$h = 0 \rightarrow 25$
$k = 0 \rightarrow 17$
$l = -8 \rightarrow 8$
3 standard reflections monitored every 150 reflections
intensity variation: −0.10%

Refinement

Refinement on F
Final R = 0.075
wR = 0.091
S = 4.005
$w = 4F_o^2/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{max}$ <0.01
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

1984 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)
315 parameters	
H-atom parameters not refined	

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

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

	x	y	z	B_{eq}
C1	0.4933 (3)	0.5603	0.2722 (8)	5.0 (3)
N2	0.4499 (2)	0.6191 (6)	0.3119 (9)	5.8 (3)
C3	0.3964 (3)	0.5912 (7)	0.376 (1)	5.9 (3)
C4	0.3919 (3)	0.4954 (6)	0.3898 (8)	4.6 (3)
N5	0.3417 (2)	0.4620 (6)	0.4458 (7)	4.6 (2)
C6	0.3398 (2)	0.3751 (6)	0.4642 (7)	4.1 (2)
C7	0.3879 (3)	0.3223 (6)	0.4281 (9)	4.9 (3)
N8	0.4373 (2)	0.3539 (6)	0.3695 (7)	5.3 (3)
C9	0.4397 (2)	0.4422 (6)	0.3488 (7)	4.3 (3)
N10	0.4911 (2)	0.4757 (6)	0.2885 (7)	5.1 (3)
O11	0.3600 (2)	0.6454 (6)	0.421 (1)	8.7 (3)
N12	0.5443 (2)	0.5950 (6)	0.2144 (8)	6.1 (3)
C13	0.5621 (3)	0.6820 (8)	0.212 (1)	7.3 (4)
O14	0.5304 (3)	0.7405 (6)	0.254 (1)	10.3 (4)
C15	0.6224 (4)	0.6974 (8)	0.157 (2)	9.1 (6)
C16	0.2832 (2)	0.3399 (6)	0.5311 (7)	3.9 (2)
O17	0.2821 (2)	0.2450 (5)	0.5353 (4)	4.0 (2)
C18	0.2734 (3)	0.2037 (6)	0.3873 (8)	4.6 (3)
O19	0.2675 (3)	0.2428 (6)	0.2569 (6)	6.9 (3)
C20	0.2728 (3)	0.1056 (7)	0.4079 (9)	5.9 (3)
C21	0.2761 (2)	0.3717 (6)	0.7099 (7)	4.1 (2)
O22	0.3225 (2)	0.3322 (5)	0.8118 (5)	4.8 (2)
C23	0.3658 (3)	0.3863 (8)	0.8688 (9)	5.9 (4)
O24	0.3655 (2)	0.4647 (6)	0.8557 (8)	7.5 (3)
C25	0.4155 (4)	0.329 (1)	0.941 (1)	9.1 (6)
C26	0.2181 (3)	0.3462 (6)	0.7851 (8)	4.4 (3)
O27	0.1753 (2)	0.3874 (6)	0.6754 (6)	5.2 (2)
C28	0.1202 (3)	0.3575 (7)	0.673 (1)	6.7 (4)
O29	0.1047 (2)	0.3016 (6)	0.7689 (9)	8.3 (3)
C30	0.0845 (4)	0.400 (1)	0.541 (2)	10.6 (7)
C31	0.2120 (3)	0.3760 (6)	0.9645 (8)	5.3 (3)
O32	0.2247 (2)	0.4689 (5)	0.9726 (5)	5.7 (2)
C33	0.2131 (4)	0.5103 (7)	1.1145 (8)	6.8 (4)
O34	0.1928 (4)	0.4725 (6)	1.2288 (7)	9.7 (4)
C35	0.2278 (6)	0.6029 (9)	1.112 (1)	10.3 (7)

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

C1—N2	1.376 (9)	C16—C21	1.524 (8)
C1—N10	1.291 (9)	O17—C18	1.350 (7)
C1—N12	1.369 (8)	C18—O19	1.204 (8)
N2—C3	1.403 (8)	C18—C20	1.495 (9)
C3—C4	1.460 (9)	C21—O22	1.447 (7)
C3—O11	1.227 (9)	C21—C26	1.518 (7)
C4—N5	1.340 (7)	O22—C23	1.354 (9)
C4—C9	1.402 (8)	C23—O24	1.19 (1)
N5—C6	1.327 (8)	C23—C25	1.53 (1)
C6—C7	1.395 (8)	C26—O27	1.437 (8)
C6—C16	1.508 (8)	C26—C31	1.516 (9)
C7—N8	1.322 (8)	O27—C28	1.336 (8)
N8—C9	1.350 (8)	C28—O29	1.21 (1)
C9—N10	1.376 (7)	C28—C30	1.46 (1)
N12—C13	1.38 (1)	C31—O32	1.439 (8)
C13—O14	1.20 (1)	O32—C33	1.331 (8)
C13—C15	1.48 (1)	C33—O34	1.185 (9)
C16—O17	1.439 (7)	C33—C35	1.44 (1)
N2—C1—N10	126.1 (5)	C6—C16—C21	109.8 (4)
N2—C1—N12	117.0 (6)	O17—C16—C21	106.9 (4)
N10—C1—N12	116.9 (6)	C16—O17—C18	116.4 (4)
C1—N2—C3	122.0 (5)	O17—C18—O19	122.9 (6)
N2—C3—C4	113.0 (6)	O17—C18—C20	111.4 (5)
N2—C3—O11	120.5 (6)	O19—C18—C20	125.7 (6)
C4—C3—O11	126.5 (6)	C16—C21—O22	107.6 (4)
C3—C4—N5	117.5 (6)	C16—C21—C26	114.2 (4)
C3—C4—C9	119.8 (5)	O22—C21—C26	107.6 (4)
N5—C4—C9	122.6 (5)	C21—O22—C23	117.0 (6)
C4—N5—C6	116.3 (5)	O22—C23—O24	124.9 (7)
N5—C6—C7	121.2 (5)	O22—C23—C25	108.0 (8)
N5—C6—C16	115.0 (5)	O24—C23—C25	127.1 (8)
C7—C6—C16	123.9 (5)	C21—C26—O27	103.3 (4)
C6—C7—N8	123.4 (5)	C21—C26—C31	113.9 (5)
C7—N8—C9	116.1 (5)	O27—C26—C31	111.7 (5)
C4—C9—N8	120.4 (5)	C26—O27—C28	119.0 (6)
C4—C9—N10	123.1 (5)	O27—C28—O29	121.4 (8)
N8—C9—N10	116.5 (5)	O27—C28—C30	111.6 (9)
C1—N10—C9	116.0 (5)	O29—C28—C30	127.0 (8)
C1—N12—C13	128.6 (7)	C26—C31—O32	108.1 (5)
N12—C13—O14	121.5 (6)	C31—O32—C33	117.1 (5)
N12—C13—C15	115.6 (8)	O32—C33—O34	121.5 (8)
O14—C13—C15	122.9 (8)	O32—C33—C35	113.4 (7)
C6—C16—O17	112.2 (5)	O34—C33—C35	125.1 (7)

The H atoms were found by difference Fourier mapping and then recalculated in steric positions. Anomalous-dispersion effects (Ibers & Hamilton, 1964) were included in F_c.

The absolute configuration of the molecule was confirmed to be as shown by application of the Hamilton (1965) R-factor test; $R(+) = 0.07540$, $R(-) = 0.07547$, the ratio of which corresponds to a 92% confidence level. Incidentally, this also confirms that the absolute configuration at the C atom adjacent to the heterocyclic ring is not changed during the condensation and acetylation reactions from that in the starting sugar.

Computer programs used: *DIFABS*, *SHELXS*86 (Sheldrick, 1985), *TEXSAN* (Molecular Structure Corporation, 1985) and *PLUTO* (Motherwell & Clegg, 1978).

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55615 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1015]

References

Baur, R., Sugimoto, T. & Pfleiderer, W. (1988). *Helv. Chim. Acta*, **71**, 531–543.

Böhme, M., Hutzenlaub, W., Richter, W. J., Elstner, E. F., Huttner, G., von Seyerl, J. & Pfleiderer, W. (1986). *Justus Liebigs Ann. Chem.* pp. 1705–1717.

Camerman, A., Mastropaolo, D. & Camerman, N. (1980). *Am. Crystallogr. Assoc. Ser.* 2, **7**, 18.

Forest, H. S. & Walker, J. (1949). *J. Chem. Soc.* pp. 79–85.

Gardlik, S. & Rajagopalan, K. V. (1990). *J. Biol. Chem.* **265**, 13047–13054.

Gries, P. & Harrow, G. (1887). *Chem. Ber.* **30**, 281–282.

Hambley, T. W., Chan, H.-K. & Gonda, I. (1986). *J. Am. Chem. Soc.* **108**, 2103–2105.

Hamilton, W. C. (1965). *Acta Cryst.* **18**, 502–510.

Ibers, J. A. & Hamilton, W. C. (1964). *Acta Cryst.* **17**, 781–782.

Johnson, J. L. (1980). In *Molybdenum and Molybdenum-Containing Enzymes*, edited by M. P. Coughlan, pp. 345–383. Oxford: Pergamon Press.

Larsen, L., Garner, C. D. & Joule, J. A. (1989). *J. Chem. Soc. Perkin Trans.* 1, pp. 2311–2316.

Larsen, L., Rowe, D. J., Garner, C. D. & Joule, J. A. (1989). *J. Chem. Soc. Perkin Trans.* 1, pp. 2317–2327.
Molecular Structure Corporation (1985). *TEXSAN. TEXRAY Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.
Rajagopalan, K. V. (1991). In *Advances in Enzymology and Related Areas of Molecular Biology*, Vol. 64, edited by A. Meister, pp. 215–290. New York: John Wiley.
Rowe, D. J., Garner, C. D. & Joule, J. A. (1985). *J. Chem. Soc. Perkin Trans.* 1, pp. 1907–1910.
Russell, J. R., Garner, C. D. & Joule, J. A. (1992*a*). *J. Chem. Soc. Perkin Trans.* 1, pp. 1245–1249.
Russell, J. R., Garner, C. D. & Joule, J. A. (1992*b*). *Tetrahedron Lett.* **33**, 3371–3374.
Sheldrick, G. M. (1985). *SHELXS*86. In *Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford Univ. Press.
Sutton, P. A., Cody, V. & Smith, G. D. (1986). *J. Am. Chem. Soc.* **108**, 4155–4158.
Taylor, E. C. & Goswami, S. (1991). *Tetrahedron Lett.* **32**, 7357–7360.
Taylor, E. C., Perlman, K. L., Sword, I. P., Séquin-Frey, M. & Jacobi, P. A. (1973). *J. Am. Chem. Soc.* **95**, 6407–6412.
Taylor, E. C., Ray, P. S., Darwish, I. S., Johnson, J. L. & Rajagopalan, K. V. (1989). *J. Am. Chem. Soc.* **111**, 7664–7665.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.
Weygand, F. (1940). *Ber. Dtsch. Chem. Ges.* **73**, 1259–1291.
Weygand, F., Simon, H., Keil, K. D. & Millauer, H. (1964). *Chem. Ber.* **97**, 1002–1023.
Weygand, F., Wacker, A. & Schmied-Kowarzik, V. (1949). *Chem. Ber.* **82**, 25–32.

SHORT COMMUNICATIONS

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Über die Fehlordnung von Triethylammoniumchlorid.

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Abstract

The known disorder in triethylammonium chloride results from stacking faults of like layers. In each layer the ethyl groups of all $HN(C_2H_5)_3^+$ ions have the same orientation, but there is a random stacking sequence of layers having either one of two different orientations. This is evidenced by diffuse streaks parallel to **c*** in X-ray diffraction patterns. The symmetry of a single layer is $P(3)11$. A redetermination of the averaged structure in the space group $P6_3mc$ fully confirms previous structure determinations.

Die Kristallstruktur von Triethylammoniumchlorid, $[HN(C_2H_5)_3]^+.Cl^-$, wurde von Genet (1965) bestimmt und von James, Cameron, Knop, Neuman & Falk (1985) verfeinert. Danach ist die Raumgruppe $P6_3mc$. Die Cl^--Ionen sowie die N-Atome der Kationen befinden sich auf den 6_3-Achsen. Für die C-Atome der Ethyl-Gruppen wurden zwei fehlgeordnete Lagen mit jeweils halber Besetzungswahrscheinlichkeit gefunden; die beiden Lagen sind über die Spiegelebenen der Raumgruppe symmetrieäquivalent.

Auf Präzessionaufnahmen, die wir jetzt angefertigt haben, lassen sich diffuse Streifen parallel zu **c*** erkennen; diesen Streifen sind die Braggschen Reflexe aufgesetzt. Bei den bisherigen Strukturbestimmungen wurden nur die Braggschen Reflexe berücksichtigt; sie geben deshalb nur die gemittelte Struktur wieder, d.h. eine Projektion in Richtung **c**. Die diffusen Streifen verraten das Vorliegen einer eindimensionalen Fehlordnung. Demnach besteht die Struktur aus in sich geordneten Schichten, die ohne periodische Ordnung gestapelt sind. Blickt man in Richtung der *c*-Achse, so sind in der einzelnen Schicht die Ethylgruppen aller Kationen gleichsinnig orientiert, die Schichtsymmetrie ist $P(3)11$. Zwei Schichtsorten kommen vor, mit Orientierung der Ethylgruppen im Uhrzeiger- oder im Gegenuhrzeigersinn. Es wechseln sich Schichten ab, bei denen das N-Atom die Lage $\frac{2}{3},\frac{1}{3},-0{,}060$ bzw. $\frac{1}{3},\frac{2}{3},0{,}440$ einnimmt, wobei die beiden Schichtsorten statistisch vorkommen. Die statistische Abfolge der Schichtsorten folgt aus dem Intensitätsverlauf auf den diffusen Streifen; dieser ist nämlich sehr gleichmäßig, ohne Intesitätsmaxima, d.h. so wie es sein muß, wenn die Reichweite nach Jagodzinski (1949) $s = 0$ beträgt.

Wir haben auch die Atomparameter für die gemittelte Struktur mit neuen Meßdaten erneut bestimmt; sie weichen nicht signifikant von denen nach James, Cameron, Knop, Neuman & Falk (1985) ab.

Literatur

Genet, F. (1965). *Bull. Soc. Fr. Minéral. Cristallogr.* **88**, 463–482.
Jagodzinski, H. (1949). *Acta Cryst.* **2**, 201–207.
James, M. A., Cameron, T. S., Knop, O., Neuman, M. & Falk, M. (1985). *Can. J. Chem.* **63**, 1750–1758.

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