

Anionen hat es die gleiche Struktur wie in unserem Fall: $[\text{S}_3\text{N}_3\text{O}_4]^-$ (Roesky, Böwing, Rayment & Shearer, 1975), $[\text{SnCl}_5(\text{OPCl}_2)]^-$ (Banister, Durrant, Rayment & Shearer, 1976), $[\text{RuCl}_3(\text{CO})_3]^-$ (Berg, Dehnicke &

Fenske, 1985) und $[\text{Ga}_2\text{Cl}_7]^-$ (Klinzing, Willing, Müller & Dehnicke, 1985). Eine abweichende Struktur des $[\text{S}_3\text{N}_3]^+$ -Ions, vermutlich verbunden mit einer dynamischen Fehlordnung, ist mit folgenden Anionen bekannt: $[\text{AlCl}_4]^-$ (Hazell & Hazell, 1972), $[\text{GaCl}_4]^-$ (Klinzing, Willing, Müller & Dehnicke, 1985) und $[\text{SbCl}_6]^-$ (Gillespie, Sawyer, Slim & Tyrer, 1982).

Tabelle 1. Atomkoordinaten und Parameter der äquivalenten isotropen Temperaturfaktoren (Å^2)

$$B_{\text{eq}}^{\text{a}} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

| | x | y | z | B_{eq}^{a} |
|-------|-------------|------------|-------------|----------------------------|
| Au | 0,1549 (1) | 0,25 | 0,29616 (6) | 2,69 (2) |
| Cl(1) | 0,3840 (7) | 0,1155 (2) | 0,4197 (3) | 4,6 (1) |
| Cl(2) | -0,0728 (6) | 0,1157 (2) | 0,1700 (3) | 4,5 (1) |
| N(1) | 1,007 (3) | 0,25 | 0,611 (2) | 4,0 (5) |
| S(1) | 0,9270 (6) | 0,1293 (2) | 0,6495 (3) | 3,7 (1) |
| N(2) | 0,741 (2) | 0,1397 (7) | 0,7530 (9) | 3,4 (3) |
| S(2) | 0,5851 (6) | 0,0629 (2) | 0,8308 (3) | 3,6 (1) |
| N(3) | 0,425 (2) | 0,1341 (7) | 0,9151 (9) | 3,7 (4) |
| S(3) | 0,3371 (9) | 0,25 | 0,9637 (4) | 3,9 (2) |

Tabelle 2. Interatomare Abstände (Å) und Winkel ($^\circ$)

| | | | |
|-----------|-----------|-----------------|-----------|
| Au—Cl(1) | 2,274 (3) | Cl(1)—Au—Cl(2) | 89,8 (1) |
| Au—Cl(2) | 2,281 (3) | Cl(1)—Au—Cl(1') | 90,4 (1) |
| | | Cl(2)—Au—Cl(2') | 89,9 (1) |
| N(1)—S(1) | 1,577 (6) | S(1)—N(1)—S(1') | 133,3 (9) |
| S(1)—N(2) | 1,566 (9) | N(1)—S(1)—N(2) | 108,7 (6) |
| N(2)—S(2) | 1,544 (9) | S(1)—N(2)—S(2) | 138,7 (6) |
| S(2)—N(3) | 1,562 (9) | N(2)—S(2)—N(3) | 110,2 (5) |
| N(3)—S(3) | 1,572 (9) | S(2)—N(3)—S(3) | 150,9 (7) |
| | | N(3)—S(3)—N(3') | 124,4 (5) |

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Structure of an Unexpected Indole Photolysis Product

BY KLAUS HARMS AND GEORGE M. SHELDRIK

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D3400 Göttingen, Federal Republic of Germany

AND KLAUS BRÜGGEMANN AND LUTZ-F. TIETZE

Institut für Organische Chemie der Universität, Tammannstrasse 2, D3400 Göttingen, Federal Republic of Germany

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Abstract. Methyl 8,9,15,16-tetrahydro-5bH,14bH-5,7a,14,14c-tetraazabenz[1,2-a:1',2'-c]difluorene-6-carboxylate ethyl acetate solvate, $\text{C}_{26}\text{H}_{24}\text{N}_4\text{O}_2 \cdot \text{C}_4\text{H}_8\text{O}_2$, $M_r = 512.6$, triclinic, $P\bar{1}$, $a = 10.340$ (5), $b = 10.809$ (5), $c = 13.613$ (1) Å, $\alpha = 97.53$ (4), $\beta = 101.65$ (4), $\gamma = 113.84$ (3)°, $U = 1324$ Å³, $Z = 2$, D_x

$= 1.286$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.081$ mm⁻¹, $F(000) = 544$, $T = 298$ K, $R = 0.052$ for 2807 reflections. The structure is a 1:1 ethyl acetate solvate. It was investigated to determine the relative configuration, which could not be established by NMR. There is a hydrogen bond [2.892 (4) Å] between N(15)

Table 1. Atomic coordinates ($\times 10^4$) and isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

| | <i>x</i> | <i>y</i> | <i>z</i> | <i>U</i> _{eq} [*] |
|--------|-----------|-----------|----------|-------------------------------------|
| C(1) | 6946 (3) | 5380 (3) | 553 (2) | 55 (1) |
| C(2) | 7206 (3) | 5570 (3) | -381 (2) | 62 (1) |
| C(3) | 7866 (3) | 4870 (3) | -860 (2) | 61 (1) |
| C(4) | 8261 (3) | 3953 (3) | -431 (2) | 54 (1) |
| C(4a) | 8002 (3) | 3752 (3) | 510 (2) | 43 (1) |
| N(5) | 8271 (2) | 2903 (2) | 1103 (2) | 46 (1) |
| C(5a) | 7879 (3) | 3117 (2) | 2003 (2) | 39 (1) |
| C(5b) | 7942 (3) | 2316 (2) | 2819 (2) | 39 (1) |
| C(6) | 9484 (3) | 2745 (2) | 3534 (2) | 38 (1) |
| C(7) | 9673 (3) | 2846 (2) | 4558 (2) | 40 (1) |
| N(8) | 8586 (2) | 2568 (2) | 5023 (1) | 41 (1) |
| C(9) | 8822 (3) | 2451 (3) | 6097 (2) | 50 (1) |
| C(10) | 7898 (3) | 2914 (3) | 6629 (2) | 49 (1) |
| C(10a) | 6362 (3) | 2315 (2) | 5948 (2) | 40 (1) |
| C(10b) | 4993 (3) | 2073 (2) | 6182 (2) | 41 (1) |
| C(11) | 4576 (3) | 2208 (3) | 7089 (2) | 54 (1) |
| C(12) | 3115 (3) | 1823 (3) | 7027 (2) | 59 (1) |
| C(13) | 2057 (3) | 1334 (3) | 6072 (2) | 59 (1) |
| C(14) | 2427 (3) | 1196 (3) | 5163 (2) | 51 (1) |
| C(14a) | 3894 (3) | 1528 (2) | 5220 (2) | 41 (1) |
| N(15) | 4555 (2) | 1420 (2) | 4459 (2) | 43 (1) |
| C(15a) | 6037 (2) | 1886 (2) | 4908 (2) | 38 (1) |
| C(15b) | 7081 (2) | 1864 (2) | 4311 (2) | 38 (1) |
| N(16) | 6966 (2) | 2486 (2) | 3431 (1) | 36 (1) |
| C(17) | 7271 (3) | 3961 (2) | 3752 (2) | 42 (1) |
| C(18) | 6729 (3) | 4442 (3) | 2822 (2) | 47 (1) |
| C(18a) | 7306 (3) | 4041 (2) | 1972 (2) | 40 (1) |
| C(18b) | 7363 (3) | 4471 (2) | 1025 (2) | 42 (1) |
| C(19) | 10719 (3) | 2948 (3) | 3115 (2) | 45 (1) |
| O(20) | 12036 (2) | 3643 (2) | 3835 (1) | 58 (1) |
| C(21) | 13316 (3) | 3860 (4) | 3489 (3) | 70 (2) |
| O(22) | 10622 (2) | 2542 (2) | 2210 (1) | 64 (1) |
| C(1') | 10016 (3) | 867 (4) | 8659 (3) | 98 (2) |
| C(2') | 8390 (3) | 190 (3) | 8392 (2) | 69 (2) |
| O(3') | 7898 (2) | -57 (2) | 9208 (2) | 83 (1) |
| C(4') | 6311 (3) | -739 (4) | 9030 (3) | 103 (2) |
| C(5') | 6015 (4) | -1128 (4) | 9984 (3) | 102 (2) |
| O(6') | 7603 (3) | -126 (3) | 7540 (2) | 103 (1) |

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

and the carbonyl O atom of the symmetry transformed ($1-x, -y, 1-z$) solvent molecule.

Experimental. Crystal size: $0.2 \times 0.2 \times 0.4$ mm. Stoe-Siemens four-circle diffractometer, monochromated Mo *K* α radiation, profile-fitting mode (Clegg, 1981). 3998 reflections, $2\theta_{\max}$ 45° , $\pm h \pm k \pm l$, three check reflections with no intensity change. 3451 unique reflections ($R_{\text{int}} = 0.015$), of which 2807 with $F > 3\sigma(F)$ used for all calculations, program system *SHELXTL* (Sheldrick, 1978). Index ranges $|h| \leq 10$, $|k| \leq 11$, $|l| \leq 14$. Cell constants refined from $\pm 2\theta$ values of 40 reflections in the range 20 – 25° .

Absorption and extinction corrections unnecessary. Structure solution by multiresolution direct methods. Refinement on *F* to $R = 0.052$, $wR = 0.059$; all non-H atoms anisotropic, H atoms included using a riding model [$C-H$ 0.96 \AA , $U(H) = 1.2U_{\text{eq}}(C)$], 360 parameters, $S = 1.54$, weighting scheme, $w^{-1} = \sigma^2(F) + 0.00066F^2$, which gave a featureless analysis of variance, max. $\Delta/\sigma = 0.16$, max. and min. heights in the final $\Delta\rho$ map 0.19 and -0.21 $e \text{\AA}^{-3}$, respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The atomic parameters

Table 2. Bond lengths (\AA) and bond angles ($^\circ$)

| | | | |
|----------------------|------------|----------------------|------------|
| C(1)–C(2) | 1.377 (4) | C(1)–C(18b) | 1.400 (4) |
| C(2)–C(3) | 1.390 (5) | C(3)–C(4) | 1.368 (5) |
| C(4)–C(4a) | 1.386 (4) | C(4a)–N(5) | 1.372 (4) |
| C(4a)–C(18b) | 1.411 (4) | N(5)–H(5) | 0.819 (26) |
| N(5)–C(5a) | 1.387 (3) | C(5a)–C(5b) | 1.502 (4) |
| C(5a)–C(18a) | 1.352 (4) | C(5b)–C(6) | 1.537 (3) |
| C(5b)–N(16) | 1.482 (4) | C(6)–C(7) | 1.351 (3) |
| C(6)–C(19) | 1.450 (4) | C(7)–N(8) | 1.348 (3) |
| N(8)–C(9) | 1.464 (3) | N(8)–C(15b) | 1.477 (3) |
| C(9)–C(10) | 1.503 (5) | C(10)–C(10a) | 1.489 (3) |
| C(10a)–C(10b) | 1.441 (4) | C(10a)–C(15a) | 1.357 (3) |
| C(10b)–C(11) | 1.396 (4) | C(10b)–C(14a) | 1.417 (3) |
| C(11)–C(12) | 1.376 (4) | C(12)–C(13) | 1.394 (4) |
| C(13)–C(14) | 1.374 (4) | C(14)–C(14a) | 1.393 (4) |
| C(14a)–N(15) | 1.369 (4) | N(15)–H(15) | 0.843 (23) |
| N(15)–C(15a) | 1.376 (3) | C(15a)–C(15b) | 1.482 (4) |
| C(15b)–N(16) | 1.455 (3) | N(16)–C(17) | 1.479 (3) |
| C(17)–C(18) | 1.519 (4) | C(18)–C(18a) | 1.497 (4) |
| C(18a)–C(18b) | 1.431 (4) | C(19)–O(20) | 1.349 (3) |
| C(19)–O(22) | 1.222 (3) | O(20)–C(21) | 1.435 (4) |
| O(2')–C(1') | 1.477 (4) | C(2')–O(3') | 1.327 (4) |
| C(2')–O(6') | 1.190 (3) | O(3')–C(4') | 1.452 (4) |
| C(4')–C(5') | 1.468 (6) | | |
| C(2)–C(1)–C(18b) | 119.1 (3) | C(1)–C(2)–C(3) | 121.0 (3) |
| C(2)–C(3)–C(4) | 121.5 (3) | C(3)–C(4)–C(4a) | 118.0 (3) |
| C(4)–C(4a)–N(5) | 130.4 (3) | C(4)–C(4a)–C(18b) | 121.7 (3) |
| C(4a)–C(4a)–C(18b) | 107.8 (2) | C(4a)–N(5)–H(5) | 127.2 (15) |
| C(4a)–N(5)–C(5a) | 108.6 (2) | H(5)–N(5)–C(5a) | 120.3 (15) |
| N(5)–C(5a)–C(5b) | 124.0 (3) | N(5)–C(5a)–C(18a) | 109.4 (2) |
| C(5b)–C(5a)–C(18a) | 126.3 (2) | C(5a)–C(5b)–C(6) | 115.7 (2) |
| C(5a)–C(5b)–N(16) | 106.3 (2) | C(6)–C(5b)–N(16) | 110.2 (2) |
| C(5b)–C(6)–C(7) | 119.6 (2) | C(5b)–C(6)–C(19) | 120.8 (2) |
| C(7)–C(6)–C(19) | 119.5 (2) | C(6)–C(7)–N(8) | 124.9 (2) |
| C(7)–N(8)–C(9) | 121.5 (2) | C(7)–N(8)–C(15b) | 114.8 (2) |
| C(9)–N(8)–C(15b) | 119.6 (2) | N(8)–C(9)–C(10) | 112.1 (3) |
| C(9)–C(10)–C(10a) | 110.1 (2) | C(10)–C(10a)–C(10b) | 131.3 (2) |
| C(10)–C(10a)–C(15a) | 121.8 (2) | C(10b)–C(10a)–C(15a) | 106.9 (2) |
| C(10a)–C(10b)–C(11) | 134.8 (2) | C(10a)–C(10b)–C(14a) | 106.1 (2) |
| C(11)–C(10b)–C(14a) | 119.0 (2) | C(10b)–C(11)–C(12) | 119.3 (2) |
| C(11)–C(12)–C(13) | 120.9 (3) | C(12)–C(13)–C(14) | 121.5 (3) |
| C(13)–C(14)–C(14a) | 118.0 (2) | C(10b)–C(14a)–C(14) | 121.2 (3) |
| C(10b)–C(14a)–N(15) | 108.0 (2) | C(14)–C(14a)–N(15) | 130.7 (2) |
| C(14a)–N(15)–H(15) | 126.5 (20) | C(14a)–N(15)–C(15a) | 108.6 (2) |
| H(15)–N(15)–C(15a) | 124.8 (20) | C(10a)–C(15a)–N(15) | 110.3 (2) |
| C(10a)–C(15a)–C(15b) | 126.6 (2) | N(15)–C(15a)–C(15b) | 123.0 (2) |
| N(8)–C(15b)–C(15a) | 108.2 (2) | N(8)–C(15b)–N(16) | 111.4 (2) |
| C(15a)–C(15b)–N(16) | 113.1 (2) | C(5b)–N(16)–C(15b) | 110.2 (2) |
| C(5b)–N(16)–C(17) | 111.9 (2) | C(15b)–N(16)–C(17) | 111.9 (2) |
| N(16)–C(17)–C(18) | 109.7 (2) | C(17)–C(18)–C(18a) | 108.2 (3) |
| C(5a)–C(18a)–C(18) | 121.8 (2) | C(5a)–C(18a)–C(18b) | 107.7 (2) |
| C(18)–C(18a)–C(18b) | 130.5 (3) | C(1)–C(18b)–C(4a) | 118.7 (2) |
| C(1)–C(18b)–C(18a) | 135.0 (3) | C(4a)–C(18b)–C(18a) | 106.3 (2) |
| C(6)–C(19)–O(20) | 113.1 (2) | C(6)–C(19)–O(22) | 125.2 (2) |
| O(20)–C(19)–O(22) | 121.7 (3) | C(19)–O(20)–C(21) | 116.7 (2) |
| C(1')–C(2')–O(3') | 112.4 (3) | C(1')–C(2')–O(6') | 124.2 (3) |
| O(3')–C(2')–O(6') | 123.4 (3) | C(2')–O(3')–C(4') | 117.0 (2) |
| O(3')–C(4')–C(5') | 107.9 (3) | | |

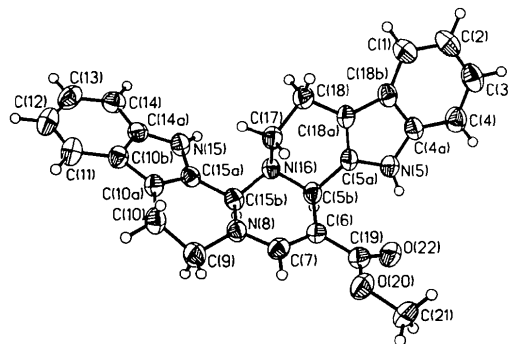


Fig. 1. Structure of the title compound (without solvent molecule), showing the atom numbering scheme.

are given in Table 1,* and bond lengths and angles in Table 2. Fig. 1 shows the molecule and numbering scheme.

Related literature. For the preparation of the compound see Brüggemann (1983). For a recent structure of an indol derivative see Sawyer, Shariff & McLean (1985).

* Lists of structure factors, H-atom parameters and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42933 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2,3-Dimethoxy-1,4-butanedinitrile (I) and 2,3-Bis(piperidino)-1,4-butanedinitrile (II)

BY A. PARFONRY, B. TINANT, J. P. DECLERCQ AND M. VAN MEERSSCHE

Laboratoire de Chimie physique et de Cristallographie, Université de Louvain, Bâtiment Lavoisier, Place Louis Pasteur, 1, B-1348 Louvain-la-Neuve, Belgium

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Abstract. (I): C₆H₈N₂O₂, $M_r = 140.14$, monoclinic, $P2_1/n$, $a = 9.027$ (4), $b = 6.756$ (3), $c = 6.671$ (3) Å, $\beta = 107.33$ (4)°, $V = 388.4$ (3) Å³, $Z = 2$, $D_x = 1.20$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.12$ cm⁻¹, $F(000) = 148$, $T = 291$ K, final $R = 0.051$ for 404 observed reflections. (II): C₁₄H₂₂N₄, $M_r = 246.36$, monoclinic, $C2/c$, $a = 23.755$ (21), $b = 5.571$ (6), $c = 11.179$ (7) Å, $\beta = 99.75$ (7)°, $V = 1458$ (2) Å³, $Z = 4$, $D_x = 1.12$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.75$ cm⁻¹, $F(000) = 536$, $T = 291$ K, final $R = 0.052$ for 693 observed reflections. The two structures were examined to compare their geometry with that of purely captor substituted molecules. There are no unusual bond distances or angles. The central C–C bond is significantly shorter than that observed for 1,1,2,2-ethanetetra-carbonitrile. The two molecules are strictly *meso*.

Experimental. Colourless parallelepipedal crystals from ethyl ether. Crystal dimensions: (I) 0.25 × 0.25 × 0.2 mm, (II) 0.2 × 0.18 × 0.3 mm. Syntex P2₁ diffractometer, graphite-monochromated Mo $K\alpha$ radiation. Unit cell from 15 reflections in range $5 < 2\theta < 20^\circ$. Data collection: (I) 901 $hk\pm l$ unique, $0 \leq h \leq 11$, $0 \leq k \leq 8$, $-7 \leq l \leq 7$; max. $\sin\theta/\lambda = 0.65$ Å⁻¹, (II) 2154 $h\pm k\pm l$ measured, 1080 unique ($R_{\text{int}} = 0.031$), $0 \leq h \leq 26$, $0 \leq k \leq 6$, $-12 \leq l \leq 12$; max. $\sin\theta/\lambda =$

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SAWYER, J. F., SHARIFF, A. & MCLEAN, S. (1985). *Acta Cryst.* C41, 1810–1814.
SHELDRIK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors (Å²) for 2,3-dimethoxy-1,4-butanedinitrile (I)

$$B_{\text{eq}} = \frac{2}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

| | x | y | z | B_{eq} |
|------|----------|----------|----------|-----------------|
| C(1) | 9418 (3) | 4258 (5) | 5169 (5) | 3.52 (6) |
| C(2) | 7886 (4) | 4615 (5) | 3582 (5) | 4.18 (6) |
| N(3) | 6710 (4) | 4857 (5) | 2414 (5) | 6.56 (7) |
| O(4) | 9308 (2) | 4590 (4) | 7197 (3) | 4.84 (5) |
| C(5) | 8509 (6) | 3054 (8) | 7918 (8) | 6.59 (10) |

0.56 Å⁻¹. 404 (I), 693 (II) reflections with $I \geq 2.5\sigma(I)$ used in refinement. Standard reflections 221 (I), 422 (II) checked every 50 reflections: no significant deviation. The two structures were solved by MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), and refined by full-matrix least squares with SHELX76 (Sheldrick, 1976) using F . H atoms from difference Fourier synthesis, included in refinement with isotropic common temperature factor. $w = 1/(\sigma^2 + gF^2)$, $g = 0.0005$ (I), 0.0008 (II). $R = 0.051$, $wR = 0.051$ for 404 observed reflections (I); and $R = 0.052$, $wR = 0.054$ for 693 observed reflections (II). Final $(\Delta/\sigma)_{\text{max}} = 0.24$ (I), 0.02 (II); $S = 2.11$ (I), 1.51 (II). Max. and min. heights in final difference Fourier synthesis = 0.23 and -0.16 e Å⁻³ (I), 0.21 and