## 11 Röntgenstrukturdaten

CRYSTAL DATA OF ***L*-40** AT -90°C

| | |
|---|---|
| formula | $C_{25}H_{34}N_2O_8$ |
| mol. weight | 490.56 |
| crystal color | colorless, transparent |
| crystal dimensions | 0.42 x 0.60 x 0.75 $mm^3$ |
| crystal system | monoclinic |
| space group | P 21 |
| space group number | 4 |
| a | 7.232(2) Å |
| b | 15.414(3) |
| c | 12.104(2) |
| β | 106.97(2)° |
| V | 1290.6(6) $Å^3$ |
| Z | 2 |
| Dcalc | 1.262 $g/cm^3$ |
| linear absorption coeff. | 0.9 $cm^{-1}$ |
| radiation | Mo-Kα |
| scan range | sphere |
| ( 2 theta )max | 56° |
| resolution | 0.75 Å |
| number of reflections measured | 15134 |
| number of independent reflections | 3050 |
| reflections used with I > 0 | 3033 |
| number of variables | 452 |
| | |
| R(F) | 0.030 |
| wR(F) | 0.036 |
| s | 1.44 |

A single crystal was measured on a SIEMENS SMART diffractometer at a temperature of about -90°C. Repeatedly measured reflections showed no intensity variations. Equivalent reflections were averaged ( R(F)internal = 0.025 ). The structure was determined by direct methods using program SIR92. A difference Fourier synthesis showed the positions of the H atoms. They were refined with isotropic thermal parameters. The non-H atoms were refined with anisotropic thermal parameters. The structure was refined on F values using weighting scheme:w(F) = 4 * F^2 / [ sigma^2(F^2) + ( 0.03 * F^2 )^2] The final difference density was less than 0.16 e/$Å^3$. The calculations were performed with the SMART, SHELX and MolEN program systems.

CRYSTAL DATA OF ***L*-52** at -140° C

| | |
|---|---|
| formula | $\mathbf{C_{16}H_{29}N0_5}$ |
| mol. weight | 315.41 |
| crystal color | colorless, transparent |
| crystal shape | prismatic |
| crystal dimensions | 0.16 x 0.40 x 0.75 $mm^3$ |
| crystal system | orthorhombic |
| space group | P 212121 |
| space group number | 19 |
| a | 9.117(1) Å |
| b | 10.260(1) |
| c | 19.110(2) |
| v | 1787.6(4)$Å^3$ |
| Z | 4 |
| Dcalc | 1.172 $g/cm^3$ |
| linear absorption coeff. | 0.81 $cm^{-1}$ |
| radiation | Mo-K α |
| scan range | sphere |
| ( 2 theta )max | 63° |
| resolution | 0.68 Å |
| number of reflections measured | 28041 |
| number of independent reflections | 3187 |
| reflections used with I > 0 | 3171 |
| number of variables | 316 |
| R(F) | 0.038 |
| wR(F) | 0.037 |
| s | 0.94 |

A single crystal was measured on a SIEMENS SMART diffractometer at a temperature of about -140°C. Repeatedly measured reflections remained stable. An empirical absorption correction using program SADABS gave an effective transmission factor from 0.60 to 1.00. Equivalent reflections were averaged ( R(F)internal = 0.033 ). The structure was determined by direct methods using program SHELXS. The H atoms were taken from difference Fourier syntheses and were refined with isotropic thermal parameters. The C, N and O atoms were refined with anisotropic thermal parameters. The structure was refined on F values using weighting scheme:w (F) = 4*F^2/[sigma^2(F^2)+(0.03*F^2)^2]. The final difference density was less than 0.28 e/Å. The calculations were performed with the SMART, SHELX and MolEN program systems.

CRYSTAL DATA OF ***L*-64** AT -146° C

| | |
|---|---|
| formula | $\mathbf{C_{18}H_{22}N_20_5}$ |
| mol. weight | 346.39 |
| crystal color | colorless, transparent |
| crystal shape | rod |
| crystal dimensions | 0.22 x 0.60 x 1.00 $mm^3$ |
| crystal system | monoclinic |
| space group | P 21 |
| space group number | 4 |
| a | 19.084(2) Å |
| b | 9.159(1) |
| c | 20.312(3) |
| β | 102.66(1)° |
| V | 3464.0(9)$Å^3$ |
| Z | 8 ( four independent molecules) |
| Dcalc | 1.328 $g/cm^3$ |
| linear absorption coeff. | 0.91 $cm^{-1}$ |
| radiation | Mo-Kα |
| scan range | sphere |
| h | -27 > 26 |
| k | -12 > 13 |
| 1 | -26 > 29 |
| ( 2 theta )max | 62° |
| resolution | 0.69Å |
| number of reflections measured | 48367 |
| number of independent reflections | 10526 |
| reflections used with I > 0 | 10254 |
| number of variables | 1224 |
| R(F) | 0.068 |
| wR(F) | 0.057 |
| s | 1.12 |

A single crystal was measured on a SIEMENS SMART diffractometer at a temperature of about -146°C. Repeatedly measured reflections remained stable. An empirical absorption correction using program SADABS gave an effective transmission range from P.816 to 1.000. Equivalent reflections were averaged ( R(I)internal = 0.034 ).The structure was determined by direct methods using program SHELXS. The H atoms were placed at calculated positions and were subsequently refined with isotropic thermal parameters.The H atoms attached to the slightly disordered atoms C56, C57, C58 and C59 were nor refined. The non-H atoms were refinedwith anisotropic thermal parameters. The structure was refined on F values using

weighting scheme: w(F) = 4 * F^2 / [ sigma^2(F^2) ( 0.03 * F^2 )^2]. The final difference density was between -0.10 and +0.74 e/Å$^3$. The calculations were performed with the SMART, SHELX and MolEN program Systems.

CRYSTAL DATA OF ***L*-65**.

| | | | |
|---|---|---|---|
| Empirical formula | **$C_{18}H_{22}N_2O_5$** | | |
| Formula weight | 346.38 | | |
| Temperature | 173(2) K | | |
| Wavelength | 0.71073 Å | | |
| Crystal system, space group | Tetragonal, P4(1) | | |
| Unit cell dimensions | a = 9.8630(10) Å | alpha | = 90 deg. |
| | b = 9.8630(10) Å | beta | = 90 deg. |
| | c = 36.982(4) Å | gamma | = 90 deg. |
| Volume | 3597.6(6) Å$^3$ | | |
| Z, Calculated density | 8, 1.279 Mg/m$^3$ | | |
| Absorption coefficient | 0.094 mm$^{-1}$ | | |
| F(000) | 1472 | | |
| Crystal size | 0.70 x 0.60 x 0.50 mm | | |
| Theta range for data collectio | 2.06 to 26.95 deg. | | |
| Limiting indices | -12<=h<=9, -11<=k<=12, -44<=l<=45 | | |
| Reflections collected / unique | 24010 / 7274 [R(int) = 0.0223] | | |
| Completeness to theta | 26.95 and 95.1 % | | |
| Absorption correction | Empirical, SADABS (Sheldrick, 1996) | | |
| Max. and min. transmission | 0.9545 and 0.9372 | | |
| Refinement method | Full-matrix least-squares on F^2 | | |
| Data / restraints / parameters | 7274 / 1 / 427 | | |
| Goodness-of-fit on F^2 | 1.022 | | |
| Final R indices [I>2sigma(I)] | R1 = 0.0451, wR2 = 0.1017 | | |
| R indices (all data) | R1 = 0.0563, wR2 = 0.1076 | | |
| Absolute structure parameter | 0.6(9) | | |
| Extinction coefficient | 0.0038(4) | | |
| Largest diff. peak and hole | 0.229 and -0.139 e.Å$^{-3}$ | | |

Table 1. Crystal data and structure refinement for ***L*-68**.

| | |
|---|---|
| Empirical formula | $C_{17}H_{29}NO_4$ |
| Formula weight | 311.41 |
| Temperature | 173(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | P2(I) |
| Unit cell dimensions | a = 10.2860(10)Å α = 90°. |
| | b = 9.0060(10)Å β = 107.280(10)°. |
| | c = 10.412(2)Å γ=90°. |
| Volume | 921.0(2)Å$^3$ |
| Z | 2 |
| Density (calculated) | 1.123 Mg/m$^3$ |
| Absorption coefficient | 0.079 mm$^{-1}$ |
| F(000) | 340 |
| Crystal size | 0.55 x 0.42 x 0.38 mm$^3$ |
| Theta range for data collection | 2.05 to 26.100. |
| Index ranges | -12<=h<=12, -10<=k<=10, -12<=l<=12 |
| Reflections collected | 11431 |
| Independent reflections | 3269 [R(int) = 0.0237] |
| Completeness to theta = 26. 10° | 92.6% |
| Absorption correction | Empirical, SADABS (Sheldrick, 1996) |
| Max, and min. transmission | 0.9706 and 0.9579 |
| Refinement method | Full-matrix least-squares on F$^2$ |
| Data / restraints / parameters | 3269/1/199 |
| Goodness-of-fit on F$^2$ | 1.026 |
| Final R indices [1>2sigma(I)] | RI = 0.0350, wR2 = 0.0878 |
| R indices (all data) | R 1 = 0.043 1, wR2 = 0.0937 |
| Absolute structure parameter | -0.2(10) |
| Largest diff. peak and hole | 0. 113 and -0. 151 e.Å$^{-3}$ |

Table 2. Atomic coordinates ($*10^4$) and equivalent isotropic displacement parameters (Å$^2*10^3$)

or *L*-Pro-68. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

| | X | Y | Z | U(eq) |
|---|---|---|---|---|
| N(1) | 5562(1) | 5601(2) | 792(1) | 31(1) |
| C(11) | 6791(2) | 5393(2) | 592(2) | 32(1) |
| O(11) | 6962(1) | 4719(2) | -355(1) | 46(1) |
| O(12) | 7780(1) | 6076(2) | 1571(1) | 36(1) |
| C(13) | 9225(2) | 5787(2) | 1696(2) | 43(1) |
| C(14) | 9573(2) | 6380(4) | 473(3) | 70(1) |
| C(15) | 9525(2) | 4145(3) | 1934(3) | 63(1) |
| C(16) | 9951(2) | 6666(3) | 2944(3) | 63(1) |
| C(2) | 5356(2) | 6222(2) | 2016(2) | 29(1) |
| C(21) | 6100(2) | 5336(2) | 3274(2) | 30(1) |
| O(21) | 6490(2) | 4078(1) | 3281(1) | 45(1) |
| O(22) | 6200(1) | 6188(1) | 4348(1) | 33(1) |
| C(23) | 6839(2) | 5651(2) | 5738(2) | 36(1) |
| C(24) | 6120(3) | 4288(3) | 6021(2) | 65(1) |
| C(25) | 8342(2) | 5399(3) | 5934(2) | 59(1) |
| C(26) | 6644(2) | 6950(2) | 6585(2) | 45(1) |
| C(3) | 3803(2) | 6097(2) | 1733(2) | 39(1) |
| C(4) | 3409(2) | 4778(2) | 773(2) | 40(1) |
| C(5) | 4329(2) | 4932(2) | -134(2) | 32(1) |
| C(51) | 3744(2) | 5918(2) | -1362(2) | 44(1) |
| C(52) | 2592(3) | 5182(3) | -2413(2) | 64(1) |
| C(53) | 2251(3) | 5272(4) | -3619(3) | 97(1) |

Table 3. Bond lengths and angles for *L*-68.

| | |
|---|---|
| N(1)-C(11) | 1.354(2) |
| N(1)-C(2) | 1.464(2) |
| N(1)-C(5) | 1.475(2) |
| C(11)-O(11) | 1.215(2) |
| C(11)-O(12) | 1.355(2) |
| O(12)-C(13) | 1.475(2) |
| | |
| C(13)-C(16) | 1.515(3) |
| C(13)-C(15) | 1.516(3) |
| C(13)-C(14) | 1.519(3) |
| C(2)-C(21) | 1.530(2) |
| C(2)-C(3) | 1.540(2) |

| | |
|---|---|
| C(21)-O(21) | 1.201(2) |
| C(21)-O(22) | 1.335(2) |
| O(22)-C(23) | 1.481(2) |
| C(23)-C(24) | 1.507(3) |
| C(23)-C(25) | 1.515(3) |
| C(23)-C(26) | 1.514(3) |
| C(3)-C(4) | 1.528(3) |
| C(4)-C(5) | 1.529(2) |
| C(5)-C(51) | 1.526(2) |
| C(51)-C(52) | 1.507(3) |
| C(52)-C(53) | 1.201(4) |
| C(11)-N(1)-C(2) | 124.75(13) |
| C(11)-N(1)-C(5) | 120.28(13) |
| C(2)-N(1)-C(5) | 113.96(13) |
| O(11)-C(11)-O(12) | 125.48(15) |
| O(11)-C(11)-N(1) | 124.08(16) |
| O(12)-C(11)-N(1) | 110.40(14) |
| C(11)-1(12)-C(13) | 119.87(14) |
| O(12)-C(13)-C(16) | 102.19(16) |
| O(12)-C(13)-C(15) | 109.76(17) |
| C(16)-C(13)-C(15) | 110.17(19) |
| O(12)-C(13)-C(14) | 110.22(15) |
| C(16)-C(13)-C(14) | 110.9(2) |
| C(15)-C(13)-C(14) | 113.1(2) |
| N(1)-C(2)-C(21) | 112.43(14) |
| N(1)-C(2)-C(3) | 102.46(13) |
| C(21)-C(2)-C(3) | 110.67(14) |
| O(21)-C(21)-O(22) | 126.5l(16) |
| O(21)-C(21)-C(2) | 125.27(15) |
| O(22)-C(21)-C(2) | 108.19(14) |
| C(21)-O(22)-C(23) | 122.18(13) |
| O(22)-C(23)-C(24) | 110.85(15) |
| O(22)-C(23)-C(25) | 108.64(15) |
| C(24)-C(23)-C(25) | 113.0(2) |
| O(22)-C(23)-C(26) | 102.73(14) |
| C(24)-C(23)-C(26) | 110.95(16) |
| C(25)-C(23)-C(26) | 110.14(16) |
| C(4)-C(3)-C(2) | 103.91(14) |
| C(3)-C(4)-C(5) | 103.93(15) |

| | |
|---|---|
| N(1)-C(5)-C(51) | 111.01(14) |
| N(1)-C(5)-C(4) | 101.74(13) |
| C(51)-C(5)-C(4) | 114.08(15) |
| C(52)-C(51)-C(5) | 112.49(17) |
| C(53)-C(52)-C(51) | 130.7(3) |

Symmetry transformations used to generate equivalent atoms: Table 4. Anisotropic displacement parameters ($Å^2*10^3$) for *L*-68. The anisotropic displacement factor takes the form: $-2\pi^2[h^2a^{*2}U^{11}+...+2hka^*b^*U^{12}]$

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| N(1) | 33(1) | 37(1) | 21(1) | -2(1) | 5(1) | -2(1) |
| C(11) | 37(1) | 36(1) | 24(1) | 1(1) | 9(1) | 1(1) |
| O(11) | 46(1) | 61(1) | 33(1) | -11(1) | 14(1) | 1(1) |
| O(12) | 29(1) | 44(1) | 35(1) | -5(1) | 7(1) | 2(1) |
| C(13) | 28(1) | 54(1) | 43(1) | 5(1) | 7(1) | 7(1) |
| C(14) | 40(1) | 103(2) | 70(2) | 28(2) | 21(1) | 4(1) |
| C(15) | 53(1) | 59(1) | 71(2) | 1(1) | 10(1) | 17(1) |
| C(16) | 34(1) | 77(2) | 68(2) | -11(1) | 0(1) | -2(1) |
| C(2) | 35(1) | 29(1) | 24(1) | 1(1) | 8(1) | 4(1) |
| C(21) | 35(1) | 31(1) | 25(1) | -2(1) | 9(1) | 0(1) |
| O(21) | 74(1) | 29(1) | 30(1) | 1(1) | 10(1) | 12(1) |
| O(22) | 43(1) | 31(1) | 2l(1) | 0(1) | 5(1) | 6(1) |
| C(23) | 48(1) | 37(1) | 20(1) | 1(1) | 5(1) | 4(1) |
| C(24) | 110(2) | 55(2) | 30(1) | 3(1) | 19(1) | -23(1) |
| C(25) | 53(1) | 77(2) | 39(1) | -9(1) | -1(1) | 24(1) |
| C(26) | 57(1) | 47(1) | 28(1) | -6(1) | 7(1) | 6(1) |
| C(3) | 36(1) | 46(1) | 34(1) | 0(1) | 11(1) | 5(1) |
| C(4) | 39(1) | 44(1) | 36(1) | 2(1) | 10(1) | -6(1) |
| C(5) | 35(1) | 30(1) | 28(1) | -2(1) | 4(1) | -2(1) |
| C(51) | 48(1) | 46(1) | 31(1) | 5(1) | 0(1) | -9(1) |
| C(52) | 64(1) | 73(2) | 39(1) | 12(1) | -10(1) | -26(1) |
| C(53) | 83(2) | 138(3) | 47(2) | 3(2) | -14(1) | -46(2) |

Table 5. Hydrogen coordinates($*10^4$)and isotropic displacement parameters ($Å^2*10^3$) for *L*-68.

| | X | Y | Z | U(eq) |
|---|---|---|---|---|
| H(14A) | 9094 | 5795 | -320 | 105 |
| H(14B) | 10557 | 6305 | 622 | 105 |
| H(14C) | 9292 | 7422 | 328 | 105 |
| H(15A) | 9285 | 3827 | 2735 | 94 |
| H(15B) | 10497 | 3967 | 2071 | 94 |
| H(I5C) | 8989 | 3580 | 1152 | 94 |
| H(16A) | 9707 | 6269 | 3718 | 95 |
| H(16B) | 9678 | 7711 | 2811 | 95 |
| H(16C) | 10937 | 6586 | 3109 | 95 |
| H(2) | 5644 | 7286 | 2123 | 35 |
| H(24A) | 6260 | 3464 | 5462 | 98 |
| H(24B) | 5144 | 4493 | 5813 | 98 |
| H(24C) | 6490 | 4020 | 6973 | 98 |
| H(25A) | 8456 | 4560 | 5377 | 89 |
| H(25B) | 8801 | 5179 | 6882 | 89 |
| H(25Q | 8740 | 6293 | 5668 | 89 |
| H(26A) | 5670 | 7095 | 6460 | 68 |
| H(26B) | 7025 | 7849 | 6309 | 68 |
| H(26C) | 7110 | 6745 | 7535 | 68 |
| H(3A) | 3564 | 5910 | 2572 | 46 |
| H(3B) | 3341 | 7015 | 1307 | 46 |
| H(4A) | 2437 | 4828 | 241 | 48 |
| H(4B) | 3579 | 3827 | 1272 | 48 |
| H(5) | 4547 | 3928 | -425 | 38 |
| H(51A) | 3411 | 6854 | -1073 | 53 |
| H(51B) | 4476 | 6171 | -1763 | 53 |
| H(52) | 2045 | 4542 | -2065 | 77 |
| H(53A) | 2744 | 5889 | -4048 | 116 |
| H(53B) | 1487 | 4725 | -4142 | 116 |

Table 6. Torsion angles [°] for *L*-68.

| | |
|---|---|
| C(2)-N(1)-C(11)-O(11) | 170.83(17) |
| C(5)-N(1)-C(11)-O(11) | 3.0(3) |
| C(2)-N(1)-C(11)-O(12) | -11.0(2) |
| C(5)-N(1)-C(11)-O(12) | -178.74(15) |
| O(11)-C(11)-(12)-C(13) | -12.4(3) |
| N(1)-C(11)-O(12)-C(13) | 169.41(15) |
| C(11)-O(12)-C(13)-C(16) | -176.15(17) |
| C(11)-O(12)-C(13)-C(15) | -59.2(2) |
| C(11)-O(12)-C(13)-C(14) | 66.0(2) |
| C(11)-N(1)-C(2)-C(21) | -57.2(2) |
| C(5)-N(1)-C(2)-C(21) | 111.24(16) |
| C(11)-N(1)-C(2)-C(3) | -176.07(16) |
| C(5)-N(1)-C(2)-C(3) | -7.60(19) |
| N(1)-C(2)-C(21)-O(21) | -19.9(2) |
| C(3)-C(2)-C(21)-O(21) | 94.0(2) |
| N(1)-C(2)-C(21)-O(22) | 161.94(13) |
| C(3)-C(2)-C(21)-O(22) | -84.15(18) |
| O(21)-C(21)-O(22)-C(23) | 0.3(3) |
| C(2)-C(21)-O(22)-C(23) | 178.42(13) |
| C(21)-O(22)-C(23)-C(24) | -58.5(2) |
| C(21)-O(22)-C(23)-C(25) | 66.3(2) |
| C(21)-O(22)-C(23)-C(26) | -177.03(15) |
| N(1)-C(2)-C(3)-C(4) | 27.69(18) |
| C(21)-C(2)-C(3)-C(4) | -92.38(16) |
| C(2)-C(3)-C(4)-C(5) - | 37.92(18) |
| C(11)-N(1)-C(5)-C(51) | -84.69(19) |
| C(2)-N(1)-C(5)-C(51) | 106.28(17) |
| C(11)-N(1)-C(5)-C(4) | 153.57(16) |
| C(2)-N(1)-C(5)-C(4) | -15.47(19) |
| C(3)-C(4)-C(5)-N(1) | 32.23(18) |
| C(3)-C(4)-C(5)-C(51) | -87.37(18) |
| N(1)-C(5)-C(51)-C(52) | 172.62(18) |
| C(4)-C(5)-C(51)-C(52) | -73.2(2) |
| C(5)-C(51)-C(52)-C(53) | -146.7(4) |

CRYSTAL DATA OF **D-71** AT -138°C

| | |
|---|---|
| formula | $\mathbf{C_{18}H_{31}N0_6}$ |
| mol. weight | 357.45 |
| crystal color colorless, transparent | |
| crystal shape plate | |
| crystal dimensions | 0.08 x 0.60 x 0.60 $mm^3$ |
| crystal System | orthorhombic |
| space group | P 212121 |
| space group number | 19 |
| a | 10.268(2)Å |
| b | 10.850(2) |
| c | 17.574(2) |
| v | 1957.8(5)$Å^3$ |
| Z | 4 |
| Dcalc | 1.213 $g/cm^3$ |
| linear absorption coeff. | 0.84 $cm^{-1}$ |
| radiation | Mo-Kw α |
| scan range | sphere |
| h | -13 > 15 |
| k | -15 > 15 |
| 1 | -23 > 25 |
| ( 2 theta )max | 64° |
| resolution | 0.67 Å |
| number of reflections measured | 34383 |
| number of independent reflections | 6289 |
| reflections used with I > 0 | 6168 |
| number of variables | 351 |
| R(F) | 0.056 |
| wR(F) | 0.045 |
| s | 0.68 |

A single crystal was measured on a SIEMENS SMART diffractometer at a temperature of about -138°C. Repeatedly measured reflections remained stable. A numerical absorption correction based on six indexed crystal faces gave a transmission range from 0.951 to 0.993. Equivalent reflections were averaged. Bijvoet pairs of reflections were not averaged. R(I)internal = 0.073. The structure was determined by direct methods using program SHELXS. The H atoms were taken from a difference Fourier synthesis and were refined with isotropic thermal parameters.

The non-H atoms wAre refined with anisotropic thermal parameters. The structure was refined on F values using weighting scheme:
w(F) = 4 * F ^ 2 ^ / [sigma^2 (F^2) + ( 0.03 * F^2 )^2] .The final difference density was between -0.22 and +0.26 e/Å$^3$. The calculations were performed with the SMART, SHELX and MolEN program Systems.

## DISCUSSION OF THE STRUCTURE

The five-membered pyrrolidine ring has a conformation close to an envelope with atom C3 0.58Å above the plane through ring atoms C2, Cl, N and C4. The ring puckering parameters defined by Cremer and Pople (*J. Am. Chem. Soc.*, 1975, 97, 1354) are q2 = 0.381Å and phi 2 = 100° ( phi2 = 108° for an ideal envelope and phi 2 = 90° for an ideal twist conformation). The side chain attached to C1is in a bisecting position, the side chain attached to C4 is in a pseudo-axial position with respect to the five-membered ring. The N atom is almost planar (sum of the three bond angles about N: 359.9°). A number of intramolecular O...H contacts approach the van der Waals contact distance of 2.4Å and may stabilise the molecular conformation via weak, electrostatic interactions: O1...Hl: 2.37(1)Å, O2...H6B: 2.36(1)Å, O3...H8B: 2.43(1)Å, O4...H4: 2.52(1)Å, O4...HllC: 2.49(1)Å, O4...H13C: 2.47(2)Å, O5...H4: 2.46(1)Å, O6...H17B: 2.47(2)Å and O6...H18B: 2.46(2)Å. The shortest intermolecular contact distance is 2.46(1)Å between O4 and H6B of a neighboring molecule ( symmetry: x+l, y, z ).

Table of Positional Parameters and Their Estimated Standard Deviations

| Atom | X | Y | Z | B(A2) |
|---|---|---|---|---|
| O1 | -0.14883(9) | 0.57034(9) | 0.71990(5) | 1.87(2) |
| O2 | -0.0634(1) | 0.39752(9) | 0.77062(5) | 2.39(2) |
| O3 | 0.40639(9) | 0.28055(9) | 0.92057(5) | 1.73(2) |
| O4 | 0.52029(9) | 0.3922(1) | 0.83528(5) | 2.80(2) |
| O5 | 0.27602(9) | 0.32949(9) | 0.63937(5) | 1.76(2) |
| O6 | 0.07598(9) | 0.40595(9) | 0.60540(5) | 2.13(2) |
| N | 0.1809(1) | 0.4727(1) | 0.71068(5) | 1.57(2) |
| C1 | 0.0790(1) | 0.5611(1) | 0.72935(7) | 1.51(2) |
| C2 | 0.1286(1) | 0.6197(1) | 0.80352(7) | 1.77(2) |
| C3 | 0.2771(1) | 0.6041(1) | 0.79851(7) | 1.73(2) |
| C4 | 0.2934(1) | 0.4768(1) | 0.76256(6) | 1.40(2) |

| | | | | |
|---|---|---|---|---|
| C5 | -0.0506(1) | 0.4976(1) | 0.74172(6) | 1.60(2) |
| C6 | -0.2793(1) | 0.5191(1) | 0.72874(8) | 2.21(2) |
| C7 | -0.3177(1) | 0.4505(1) | 0.65845(9) | 2.60(3) |
| C8 | 0.2887(1) | 0.3715(1) | 0.82049(7) | 1.62(2) |
| C9 | 0.4192(1) | 0.3511(1) | 0.85862(7) | 1.62(2) |
| C10 | 0.5218(1) | 0.2455(1) | 0.96705(7) | 1.65(2) |
| C11 | 0.5853(1) | 0.3595(1) | 1.00065(8) | 2.23(2) |
| C12 | 0.4612(1) | 0.1674(2) | 1.02916(8) | 2.81(3) |
| C13 | 0.6165(2) | 0.1700(1) | 0.91941(8) | 2.47(3) |
| C14 | 0.1698(1) | 0.4022(1) | 0.64760(6) | 1.59(2) |
| C15 | 0.2912(1) | 0.2529(1) | 0.57057(6) | 1.52(2) |
| C16 | 0.4239(1) | 0.1939(1) | 0.58264(7) | 2.04(2) |
| C17 | 0.1849(1) | 0.1555(1) | 0.56713(7) | 2.08(2) |
| C18 | 0.2931(1) | 0.3344(1) | 0.50025(7) | 2.15(2) |
| H1 | 0.068(1) | 0.622(1) | 0.6879(7) | 1.5(3)* |
| H2B | 0.105(1) | 0.702(1) | 0.8078(7) | 1.3(3)* |
| H2A | 0.093(1) | 0.571(1) | 0.8464(7) | 1.6(3)* |
| H3B | 0.311(1) | 0.665(1) | 0.7623(8) | 2.l(3)* |
| H3A | 0.322(1) | 0.609(1) | 0.8462(8) | 2.2(3)* |
| H4 | 0.372(1) | 0.472(1) | 0.7327(7) | 1.5(3)* |
| H6B | -0.280(1) | 0.469(1) | 0.7734(8) | 2.3(3)* |
| H6A | -0.331(1) | 0.591(2) | 0.7402(8) | 2.6(3)* |
| H7A | -0.402(2) | 0.417(2) | 0.670(1) | 4.5(4)* |
| H7C | -0.318(2) | 0.513(2) | 0.613(1) | 3.7(4)* |
| H7B | -0.259(2) | 0.385(2) | 0.6498(9) | 4.1(4)* |
| H8A | 0.267(1) | 0.297(1) | 0.7975(8) | 2.3(3)* |
| H8B | 0.226(1) | 0.382(1) | 0.8575(7) | 1.2(3)* |
| H11B | 0.648(1) | 0.331(2) | 1.0383(9) | 3.2(4)* |
| H11A | 0.521(1) | 0.408(2) | 1.0285(8) | 2.9(3)* |
| H11C | 0.629(1) | 0.407(1) | 0.9611(8) | 2.7(3)* |
| H12A | 0.531(1) | 0.139(2) | 1.0642(9) | 3.9(4)* |
| H12C | 0.399(2) | 0.214(2) | 1.060(1) | 4.4(4)* |
| H12B | 0.420(2) | 0.099(2) | 1.0078(9) | 3.4(4)* |
| H13C | 0.656(1) | 0.220(1) | 0.8814(8) | 2.4(3)* |
| H13B | 0.571(2) | 0.108(2) | 0.8974(9) | 4.3(4)* |
| H13A | 0.684(1) | 0.138(2) | 0.9540(9) | 3.4(4)* |
| H16B | 0.446(1) | 0.142(1) | 0.5370(8) | 2.8(3)* |
| H16A | 0.425(1) | 0.140(1) | 0.6292(8) | 2.6(3)* |
| H16C | 0.496(1) | 0.257(2) | 0.5872(8) | 2.8(3)* |

| | | | | |
|---|---|---|---|---|
| H17B | 0.098(1) | 0.192(1) | 0.5584(8) | 2.3(3)* |
| H17C | 0.205(1) | 0.099(1) | 0.5248(8) | 2.1(3)* |
| H17A | 0.183(1) | 0.104(2) | 0.6141(8) | 2.6(3)* |
| H18B | 0.212(1) | 0.380(2) | 0.4913(9) | 3.3(4)* |
| H18C | 0.315(1) | 0.283(2) | 0.4555(9) | 2.7(3)* |
| H18A | 0.362(2) | 0.398(2) | 0.5050(9) | 3.2(4)* |

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: (4/3) * [a2*B(1,1) + b2*B(2,2) + c2*B(3,3) + ab(cos gamma)*B(1,2) ac(cos beta)*B(1,3) + bc(cos alpha)*B(2,3)1].

Table of General Displacement Parameter Expressions - U's

| Name | U(1,1) | U(2,2) | U(3,3) | U(1,2) | U(1,3) | U(2,3) |
|---|---|---|---|---|---|---|
| O1 | 0.0180(4) | 0.0222(4) | 0.0310(4) | -0.0004(4) | 0.0013(4) | -0.0009(4) |
| O2 | 0.0341(5) | 0.0236(5) | 0.0329(4) | -0.0031(4) | -0.0009(4) | 0.0062(4) |
| O3 | 0.0179(4) | 0.0263(4) | 0.0217(4) | -0.0013(4) | -0.0029(4) | 0.0073(4) |
| O4 | 0.0192(4) | 0.0517(6) | 0.0355(S) | -0.0051(5) | -0.0021(4) | 0.0216(5) |
| O5 | 0.0226(4) | 0.0275(4) | 0.0167(3) | 0.0071(4) | -0.0040(4) | -0.0070(4) |
| O6 | 0.0253(4) | 0.0338(5) | 0.0218(4) | 0.0077(4) | -0.0081(4) | -0.0051(4) |
| N | 0.0189(5) | 0.0239(5) | 0.0170(4) | 0.0058(4) | -0.0033(4) | -0.0038(4) |
| C1 | 0.0213(5) | 0.0181(5) | 0.0181(5) | 0.0028(5) | -0.0017(5) | -0.0001(5) |
| C2 | 0.0241(6) | 0.0198(6) | 0.0235(5) | 0.0020(5) | -0.0005(5) | -0.0056(5) |
| C3 | 0.0234(6) | 0.0213(6) | 0.0210(5) | -0.0028(5) | -0.0001(5) | -0.0027(5) |
| C4 | 0.0165(5) | 0.0214(5) | 0.0154(5) | -0.0001(5) | -0.0023(5) | 0.0002(5) |
| C5 | 0.0230(6) | 0.0213(6) | 0.0165(5) | 0.0007(5) | 0.0016(5) | -0.0039(5) |
| C6 | 0.0191(6) | 0.0285(7) | 0.0363(7) | -0.0033(6) | 0.0048(6) | -0.0037(6) |
| C7 | 0.0285(7) | 0.0320(7) | 0.0381(7) | -0.0015(6) | -0.0052(6) | -0.0034(6) |
| C8 | 0.0181(5) | 0.0226(6) | 0.0207(5) | -0.0022(5) | -0.0026(5) | 0.0029(5) |
| C9 | 0.0207(5) | 0.0213(6) | 0.0198(5) | -0.0008(5) | -0.0006(5) | 0.0015(5) |
| C10 | 0.0196(5) | 0.0220(6) | 0.0210(5) | -0.0001(5) | -0.0049(5) | 0.0042(5) |
| C11 | 0.0292(6) | 0.0258(7) | 0.0297(6) | 0.0012(6) | -0.0094(6) | -0.0025(6) |
| C12 | 0.0288(7) | 0.0441(8) | 0.0339(6) | -0.0063(7) | -0.0095(6) | 0.0196(6) |
| C13 | 0.0305(7) | 0.0271(7) | 0.0363(7) | 0.0080(6) | -0.0047(6) | -0.0021(6) |
| C14 | 0.0224(6) | 0.0215(6) | 0.0165(5) | 0.0033(5) | -0.0010(5) | -0.0005(5) |
| C15 | 0.0239(6) | 0.0185(5) | 0.0155(5) | -0.0006(5) | 0.0017(5) | -0.0043(5) |
| C16 | 0.0246(6) | 0.0239(6) | 0.0291(6) | 0.0033(6) | 0.0007(6) | -0.0061(5) |
| C17 | 0.0266(6) | 0.0243(6) | 0.0279(6) | -0.0039(6) | -0.0007(6) | -0.0010(6) |
| C18 | 0.0372(7) | 0.0243(6) | 0.0201(5) | 0.0002(6) | 0.0017(6) | 0.0008(5) |

The form of the anisotropic displacement parameter is:
exp [-2PI2{h2a2U(1,1) + k2b2U(2,2) + 12c2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)}] where a,b, and c are reciprocal lattice constants.

Table of Bond Distances in Ångstroms [Å]

| Atom 1 | Atom 2 | Distance | Atom 1 | Atom 2 | Distance |
|---|---|---|---|---|---|
| O1 | C5 | 1.337(2) | C1 | C5 | 1.515(2) |
| O1 | C6 | 1.459(2) | C2 | C3 | 1.537(2) |
| O2 | C5 | 1.206(2) | C3 | C4 | 1.528(2) |
| O3 | C9 | 1.338(1) | C4 | C8 | 1.531(2) |
| O3 | C10 | 1.488(2) | C6 | C7 | 1.495(2) |
| O4 | C9 | 1.202(2) | C8 | C9 | 1.514(2) |
| O5 | C14 | 1.354(2) | C10 | C11 | 1.518(2) |
| O5 | C15 | 1.476(1) | C10 | C12 | 1.515(2) |
| O6 | C14 | 1.216(2) | C10 | C13 | 1.522(2) |
| N | C1 | 1.457(2) | C15 | C16 | 1.520(2) |
| N | C4 | 1.472(2) | C15 | C17 | 1.520(2) |
| N | C14 | 1.351(2) | C15 | C18 | 1.520(2) |
| C1 | C2 | 1.537(2) | | | |

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

| | Atom 1 | Atom 2 | Atom 3 | Atom 4 | Angle |
|---|---|---|---|---|---|
| C6 | O1 | C5 | O2 | -3.51 | (0.17) |
| C6 | O1 | C5 | C1 | 179.77 | (0.10) |
| C5 | O1 | C6 | C7 | -89.43 | (0.13) |
| C10 | O3 | C9 | O4 | -0.31 | (0.18) |
| C10 | O3 | C9 | C8 | 178.84 | (0.10) |
| C9 | O3 | C10 | C11 | 62.52 | (0.14) |
| C9 | O3 | C10 | C12 | -179.84 | (0.11) |
| C9 | O3 | C10 | C13 | -61.91 | (0.14) |
| C15 | O5 | C14 | O6 | 5.76 | (0.19) |
| C15 | O5 | C14 | N | -174.29 | (0.10) |
| C14 | O5 | C15 | C16 | 176.85 | (0.11) |
| C14 | O5 | C15 | C17 | -64.95 | (0.14) |

| | | | | | |
|---|---|---|---|---|---|
| C14 | O5 | C15 | C18 | 59.57 | (0.14) |
| C4 | N | C1 | C2 | -4.27 | (0.13) |
| C4 | N | C1 | C5 | -123.25 | (0.11) |
| C14 | N | C1 | C2 | 179.77 | (0.11) |
| C14 | N | C1 | C5 | 60.78 | (0.14) |
| C1 | N | C4 | C3 | -19.24 | (0.12) |
| C1 | N | C4 | C8 | 101.62 | (0.12) |
| C14 | N | C4 | C3 | 156.41 | (0.12) |
| C14 | N | C4 | C8 | -82.72 | (0.14) |
| C1 | N | C14 | O5 | 178.08 | (0.10) |
| C1 | N | C14 | O6 | -1.97 | (0.19) |
| C4 | N | C14 | O5 | 2.66 | (0.17) |
| C4 | N | C14 | O6 | -177.39 | (0.12) |
| N | C1 | C2 | C3 | 26.03 | (0.12) |
| C5 | C1 | C2 | C3 | 145.28 | (0.11) |
| N | C1 | C5 | O1 | -147.81 | (0.10) |
| N | C1 | C5 | O2 | 35.45 | (0.16) |
| C2 | C1 | C5 | O1 | 98.03 | (0.12) |
| C2 | C1 | C5 | O2 | -78.70 | (0.15) |
| C1 | C2 | C3 | C4 | -38.23 | (0.12) |
| C2 | C3 | C4 | N | 34.55 | (0.11) |
| C2 | C3 | C4 | C8 | -84.94 | (0.12) |
| N | C4 | C8 | C9 | 163.36 | (0.10) |
| C3 | C4 | C8 | C9 | -83.06 | (0.13) |
| C4 | C8 | C9 | O3 | 165.34 | (0.10) |
| C4 | C8 | C9 | O4 | -15.49 | (0.18) |

Table of Least-Squares Planes

Orthonormal Equation of Plane 1

| | | | |
|---|---|---|---|
| 0.4380 X + | 0.7135 Y + | -0.5468 Z - | -2.3321 = 0 |
| 0.0006 | 0.0005 | 0.0007 | 0.0116 |

Crystallographic Equation of Plane

| | | | |
|---|---|---|---|
| 4.4976 X + | 7.7417 Y + | -9.6097 Z - | -2.3321 = 0 |
| 0.0065 | 0.0050 | 0.0122 | 0.0116 |

| Atom | X | Y | Z | Distance | Esd |
|---|---|---|---|---|---|
| C2 | 1.3201 | 6.7241 | 14.1211 | -0.0133 | 0.0013 |
| C1 | 0.8115 | 6.0878 | 12.8176 | 0.0226 | 0.0012 |
| N | 1.8578 | 5.1285 | 12.4895 | -0.0241 | 0.0010 |
| C4 | 3.0123 | 5.1730 | 13.4013 | 0.0148 | 0.0012 |
| | Chi Squared = 1125.2 | | | | |
| | | Other Atoms | | | |
| C3 | 2.8449 | 6.5545 | 14.0330 | 0.5818 | 0.0013 |
| C14 | 1.7433 | 4.3642 | 11.3810 | -0.0135 | 0.0013 |
| O5 | 2.8341 | 3.5750 | 11.2364 | -0.0197 | 0.0009 |
| O6 | 0.7802 | 4.4045 | 10.6394 | -0.0011 | 0.0010 |
| C5 | -0.5197 | 5.3985 | 13.0350 | -1.1712 | 0.0012 |
| H1 | 0.7023 | 6.7488 | 12.0890 | 0.8449 | 0.0133 |
| C8 | 2.9643 | 4.0308 | 14.4194 | -1.3779 | 0.0013 |
| H4 | 3.8151 | 5.1214 | 12.8767 | 0.6165 | 0.0133 |

Orthonormal Equation of Plane 2

| | | | |
|---|---|---|---|
| 0.4454 X + | 0.7148 Y + | -0.5392 Z - | -2.2406 = 0 |
| 0.0005 | 0.0004 | 0.0005 | 0.0078 |

CRYSTAL DATA OF ***L*-72A** AT -139°C

| | |
|---|---|
| formula | $\mathbf{C_{18}H_{31}N0_6}$ |
| mol. weight | 357.45 |
| crystal color | colorless, transparent |
| crystal shape ruler-shaped | |
| crystal dimensions | 0.18 x 0.46 x 1.30 $mm^3$ |
| crystal system | orthorhombic |
| space group | P 212121 |
| space group number | 19 |
| a | 5.9503(5) Å |
| b | 9.4393(8) |
| c | 36.016(4) |
| v | 2022.9(3) $Å^3$ |
| Z | 4 |
| Dcalc | 1.174 $g/cm^3$ |
| linear absorption coeff. | 0.82 $cm^{-1}$ |
| radiation | Mo-Kα |
| scan range | sphere |
| ( 2 theta )max | 59° |
| resolution | 0.72 Å |
| number of reflections measured | 30821 |
| number of independent reflections | 3089 |
| reflections used with I > 0 | 3071 |
| number of ariables | 351 |
| R(F) | 0.078 |
| wR(F) | 0.062, |
| s | 1.18 |

A single crystal was measured on a SIEMENS SMART diffractometer at a temperature of about -139°C. Repeatedly measured reflections remained stable. An empirical absorption correction using program SADABS gave an effective transmission range from 0.814 to 1.000 Equivalent reflections were averaged ( R(F)internal = 0.050 ). The structure was determined by direct methods using program SHELXS. The H atoms were taken from difference Fourier syntheses and were refined with isotropic thermal parameters. The non-H atoms were refined with anisotropic thermal parameters. The structure was refined on F values using weighting scheme: w(F) = 4 * FA2 / [ sigma^2(F^2) + ( 0.03 * F^2)^2] The final difference density was between

-0.25 and +0.41 e/Å$^3$. The calculations were performed with the SMART, SHELX and MolEN program Systems

DISCUSSION OF THE STRUCTURE

The proline ring approximately has an envelope conformation with atom C3 0.57 Å above the plane through C2, Cl, N and C4. The N atom is almost planar and lies only 0.05 Å outside the plane through Cl, C4 and C8. The torsion angle about the N-C8 amide bond is about 5°. The molecule shows a number of intramolecular O ... H distances equal to the van der Waals contact distance of 2.4 Å: O2 ... Hl, O3 ... H12C, 05 ... H17C, O5 ... H18B and O6 ... H16B. The shortest intermolecular distance is 2.48(3)Å between O5 and H2A of a neighboring molecule ( symmetry: 1+x, y, z ) and is slightly longer than the van der Waals contact distance. There are no other short intermolecular contacts.

Table of Positional Parameters and Their Estimated Standard Deviations

| Atom | X | Y | Z | B(A2) |
|---|---|---|---|---|
| O1 | 0.4542(3) | 1.0871(3) | 0.06163(5) | 2.47(4) |
| O2 | 0.0830(3) | 1.0645(3) | 0.07231(5) | 2.27(4) |
| O3 | 0.8050(3) | 0.8090(2) | 0.12869(5) | 2.14(4) |
| O4 | 0.4634(3) | 0.7876(2) | 0.10010(4) | 1.86(3) |
| O5 | 0.9254(4) | 1.2013(3) | 0.21444(5) | 2.78(4) |
| O6 | 0.7945(3) | 1.0544(2) | 0.25888(4) | 1.80(3) |
| N | 0.5315(4) | 0.9764(3) | 0.13471(6) | 1.69(4) |
| C1 | 0.3140(4) | 1.0338(3) | 0.12345(7) | 1.59(4) |
| C2 | 0.3028(5) | 1.1742(4) | 0.14526(7) | 2.44(5) |
| C3 | 0.5501(6) | 1.2144(4) | 0.15121(7) | 2.35(5) |
| C4 | 0.6632(4) | 1.0719(3) | 0.15837(7) | 1.76(5) |
| C5 | 0.3006(S) | 1.0632(3) | 0.08234(7) | 1.81(5) |
| C6 | 0.0333(5) | 1.1106(4) | 0.03483(7) | 2.47(6) |
| C7 | 0.0355(7) | 1.2692(5) | 0.03238(9) | 3.64(7) |
| C8 | 0.6169(4) | 0.8544(3) | 0.12146(6) | 1.58(5) |
| C9 | 0.5297(5) | 0.6759(3) | 0.07391(7) | 1.87(5) |
| C10 | 0.6997(6) | 0.7354(4) | 0.04694(7) | 2.72(6) |
| C11 | 0.3092(6) | 0.6431(4) | 0.05448(8) | 2.91(6) |
| C12 | 0.6152(6) | 0.5464(4) | 0.09458(8) | 3.02(7) |
| C13 | 0.6527(5) | 1.0228(3) | 0.19900(7) | 1.71(5) |
| C14 | 0.8078(5) | 1.1047(3) | 0.22440(7) | 1.66(5) |
| C15 | 0.9218(5) | 1.1206(3) | 0.28984(6) | 1.68(5) |

| | | | | |
|---|---|---|---|---|
| C16 | 0.8567(6) | 1.0289(4) | 0.32262(8) | 2.87(6) |
| C17 | 1.1735(5) | 1.1097(4) | 0.28298(8) | 2.53(6) |
| C18 | 0.8440(5) | 1.2723(3) | 0.29488(7) | 2.16(5) |
| H1 | 0.188(5) | 0.967(4) | 0.1315(7) | 2.2(6)* |
| H2A | 0.233(5) | 1.158(4) | 0.1693(6) | 1.6(6)* |
| H2B | 0.222(5) | 1.243(3) | 0.1304(7) | 1.5(6)* |
| H3A | 0.606(4) | 1.257(4) | 0.1282(7) | 1.9(6)* |
| H3B | 0.568(5) | 1.273(4) | 0.1734(7) | 2.3(6)* |
| H4 | 0.818(5) | 1.066(4) | 0.1500(7) | 2.5(7)* |
| H6B | 0.142(4) | 1.061(4) | 0.0145(7) | 2.3(6)* |
| H6A | -0.117(5) | 1.081(4) | 0.0299(7) | 2.1(6)* |
| H7A | 0.003(5) | 1.299(4) | 0.0061(7) | 2.7(7)* |
| H7C | 0.182(7) | 1.312(5) | 0.0387(9) | 5(1)* |
| H7B | -0.056(7) | 1.307(6) | 0.048(1) | 6(1)* |
| H10A | 0.723(5) | 0.663(4) | 0.0288(7) | 2.3(6)* |
| H10B | 0.645(6) | 0.814(5) | 0.0332(9) | 5(1)* |
| H10C | 0.829(7) | 0.774(5) | 0.058(1) | 7(1)* |
| H11A | 0.333(5) | 0.578(4) | 0.0334(7) | 1.9(6)* |
| H11C | 0.189(5) | 0.598(4) | 0.0723(7) | 3.0(7)* |
| H11B | 0.252(9) | 0.734(6) | 0.041(1) | 7(1)* |
| H12C | 0.764(6) | 0.563(4) | 0.1077(8) | 3.4(8)* |
| H12B | 0.622(6) | 0.468(5) | 0.0753(9) | 5(1)* |
| H12A | 0.506(7) | 0.518(5) | 0.1151(9) | 5.0(9)* |
| H13A | 0.690(5) | 0.9249) | 0.19950) | 1.6(6)* |
| H13B | 0.504(6) | 1.032(4) | 0.2106(8) | 3.2(7)* |
| H16A | 0.914(5) | 1.062(5) | 0.3472(9) | 4.2(8)* |
| H16B | 0.694(7) | 1.045(5) | 0.323(1) | 6(1)* |
| H16C | 0.896(5) | 0.927(4) | 0.3185(9) | 3.8(8)* |
| H17C | 1.217(5) | 1.173(4) | 0.2611(8) | 3.2(7)* |
| H17B | 1.212(6) | 1.007(5) | 0.2775(8) | 3.9(8)* |
| H17A | 1.251(6) | 1.136(5) | 0.3067(8) | 4.l(8)* |
| H18C | 0.691(7) | 1.278(5) | 0.297(1) | 6(1)* |
| H18A | 0.907(5) | 1.308(4) | 0.3183(8) | 2.7(7)* |
| H18B | 0.885(4) | 1.328(4) | 0.2723(7) | 1.8(6)* |

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: (4/3) * [a2*B(1,1) + b2*B(2,2) + c2*B(3,3) + ab(cos gamma)*B(1,2) + ac(cos beta)*B(1,3) + bc(cos alpha)*B(2,3)].

Table of General Displacement Parameter Expressions - U's

| Name | U(1,1) | U(2,2) | U(3,3) | U(1,2) | U(1,3) | U(2,3) |
|---|---|---|---|---|---|---|
| O1 | 0.0205(9) | 0.042(1) | 0.0318(8) | 0.003(1) | 0.0039(9) | 0.0073(9) |
| O2 | 0.0177(8) | 0.036(1) | 0.0328(8) | -0.002(1) | -0.0060(8) | 0.0089(9) |
| O3 | 0.0215(9) | 0.023(1) | 0.0365(9) | 0.009(1) | -0.0078(9) | -0.0013(9) |
| O4 | 0.0203(8) | 0.024(1) | 0.0263(7) | 0.004(1) | -0.0033(8) | -0.0052(8) |
| O5 | 0.039(1) | 0.034(1) | 0.0322(8) | -0.021(1) | -0.002(1) | 0.0032(9) |
| O6 | 0.0218(8) | 0.022(1) | 0.0243(7) | -0.005(1) | -0.0028(8) | -0.0018(8) |
| N | 0.018(1) | 0.021(1) | 0.0251(9) | 0.004(1) | -0.0036(9) | -0.0016(9) |
| C1 | 0.016(1) | 0.019(1) | 0.026(1) | 0.004(1) | -0.001(1) | 0.001(1) |
| C2 | 0.031(1) | 0.027(1) | 0.035(1) | 0.011(2) | -0.005(1) | -0.007(1) |
| C3 | 0.034(1) | 0.024(1) | 0.030(1) | 0.003(2) | -0.004(1) | -0.000(1) |
| C4 | 0.019(1) | 0.024(1) | 0.024(1) | -0.000(1) | -0.000(1) | -0.005(1) |
| C5 | 0.020(1) | 0.023(1) | 0.026(1) | 0.004(1) | -0.002(1) | 0.001(1) |
| C6 | 0.025(1) | 0.036(2) | 0.033(1) | -0.003(2) | -0.010(1) | 0.002(1) |
| C7 | 0.046(2) | 0.049(2) | 0.044(1) | 0.011(2) | -0.005(2) | 0.008(2) |
| C8 | 0.022(1) | 0.021(1) | 0.0169(9) | 0.001(1) | -0.002(1) | 0.000(1) |
| C9 | 0.030(1) | 0.017(1) | 0.025(1) | 0.001(1) | 0.001(1) | -0.007(1) |
| C10 | 0.040(2) | 0.035(2) | 0.029(1) | 0.000(2) | 0.008(1) | -0.005(1) |
| C11 | 0.038(2) | 0.036(2) | 0.036(1) | -0.006(2) | -0.002(1) | -0.012(1) |
| C12 | 0.051(2) | 0.024(2) | 0.040(1) | 0.013(2) | 0.004(2) | 0.001(1) |
| C13 | 0.022(1) | 0.019(1) | 0.024(1) | -0.003(1) | -0.002(1) | -0.001(1) |
| C14 | 0.022(1) | 0.016(1) | 0.025(1) | 0.001(1) | 0.001(1) | -0.002(1) |
| C15 | 0.020(1) | 0.021(1) | 0.022(1) | -0.002(1) | -0.003(1) | -0.004(1) |
| C16 | 0.042(2) | 0.039(2) | 0.028(1) | -0.008(2) | -0.005(1) | 0.001(1) |
| C17 | 0.020(1) | 0.035(2) | 0.041(1) | 0.003(1) | -0.005(1) | -0.011(1) |
| C18 | 0.028(1) | 0.022(1) | 0.032(1) | 0.005(1) | -0.004(1) | -0.009(1) |

The form of the anisotropic displacement parameter is: exp [-2PI2{h2a2U(1,1) + k2b2U(2,2) + 12c2U(3,3) + 2hkabU(1,2) + 2hlacU(1,3) + 2klbcU(2,3)}] where a,b, and c are reciprocal lattice constants.

Table of Bond Distances in Ångstroms [Å]

| Atom 1 | Atom 2 | Distance | Atom 1 | Atom 2 | Distance |
|---|---|---|---|---|---|
| O1 | C5 | 1.201(3) | C1 | C5 | 1.508(3) |
| O2 | C5 | 1.344(3) | C2 | C3 | 1.535(5) |
| O2 | C6 | 1.449(3) | C3 | C4 | 1.526(4) |
| O3 | C8 | 1.227(3) | C4 | C13 | 1.536(4) |
| O4 | C8 | 1.350(3) | C6 | C7 | 1.500(6) |
| O4 | C9 | 1.468(3) | C9 | C10 | 1.511(4) |
| O5 | C14 | 1.205(4) | C9 | C11 | 1.519(5) |
| O6 | C14 | 1.332(3) | C9 | C12 | 1.519(4) |
| O6 | C15 | 1.486(3) | C13 | C14 | 1.512(4) |
| N | C1 | 1.461(4) | C15 | C16 | 1.515(4) |
| N | C4 | 1.467(4) | C15 | C17 | 1.522(4) |
| N | C8 | 1.346(4) | C15 | C18 | 1.516(4) |
| C1 | C2 | 1.542(4) | | | |

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

| Atom 1 | Atom 2 | Atom 3 | Angle | Atom 1 | Atom 2 | Atom 3 | Angle |
|---|---|---|---|---|---|---|---|
| C5 | O2 | C6 | 116.7(2) | O3 | C8 | N | 124.6(2) |
| C8 | O4 | C9 | 121.3(2) | O4 | C8 | N | 110.3(2) |
| C14 | O6 | C15 | 121.3(2) | O4 | C9 | C10 | 109.1(2) |
| C1 | N | C4 | 114.0(2) | O4 | C9 | C11 | 102.1(2) |
| C1 | N | C8 | 123.6(2) | O4 | C9 | C12 | 110.7(2) |
| C4 | N | C8 | 122.0(2) | C10 | C9 | C11 | 111.0(2) |
| N | C1 | C2 | 102.5(2) | C10 | C9 | C12 | 113.0(3) |
| N | C1 | C5 | 112.8(2) | C11 | C9 | C12 | 110.5(3) |
| C2 | C1 | C5 | 109.9(2) | C4 | C13 | C14 | 113.4(2) |
| C1 | C2 | C3 | 104.0(2) | O5 | C14 | O6 | 125.6(2) |
| C2 | C3 | C4 | 103.2(3) | O5 | C14 | C13 | 124.2(2) |
| N | C4 | C3 | 102.0(2) | O6 | C14 | C13 | 110.2(2) |
| N | C4 | C13 | 110.2(2) | O6 | C15 | C16 | 102.4(2) |
| C3 | C4 | C13 | 114.1(2) | O6 | C15 | C17 | 110.6(2) |
| O1 | C5 | O2 | 124.4(2) | O6 | C15 | C18 | 109.3(2) |
| O1 | C5 | C1 | 127.1(2) | C16 | C15 | C17 | 109.8(2) |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| O2 | C5 | C1 | 108.4(2) | C16 | C15 | C18 | 111.6(2) |
| O2 | C6 | C7 | 110.7(3) | C17 | C15 | C18 | 112.6(3) |
| O3 | C8 | O4 | 125.1(3) | | | | |

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Torsion Angles in Degrees

| Atom 1 | Atom 2 | Atom 3 | Atom 4 | Angle | |
|---|---|---|---|---|---|
| C6 | O2 | C5 | O1 | -5.83 | (0.45) |
| C6 | O2 | C5 | C1 | 171.61 | (0.25) |
| C5 | O2 | C6 | C7 | -80.20 | (0.34) |
| C9 | O4 | C8 | O3 | -19.70 | (0.37) |
| C9 | O4 | C8 | N | 162.20 | (0.21) |
| C8 | O4 | C9 | C10 | -57.88 | (0.30) |
| C8 | O4 | C9 | C11 | -175.35 | (0.23) |
| C8 | O4 | C9 | C12 | 66.96 | (0.32) |
| C15 | O6 | C14 | O5 | 2.96 | (0.43) |
| C15 | O6 | C14 | C13 | -177.01 | (0.22) |
| C14 | O6 | C15 | C16 | -179.54 | (0.25) |
| C14 | O6 | C15 | C17 | -62.58 | (0.33) |
| C14 | O6 | C15 | C18 | 61.97 | (0.30) |
| C4 | N | C1 | C2 | -5.17 | (0.27) |
| C4 | N | C1 | C5 | 112.92 | (0.25) |
| C8 | N | C1 | C2 | -177.95 | (0.23) |
| C8 | N | C1 | C5 | -59.86 | (0.34) |
| C1 | N | C4 | C3 | -18.14 | (0.27) |
| C1 | N | C4 | C13 | 103.46 | (0.25) |
| C8 | N | C4 | C3 | 154.78 | (0.23) |
| C8 | N | C4 | C13 | -83.62 | (0.29) |
| C1 | N | C8 | O3 | 175.04 | (0.24) |
| C1 | N | C8 | O4 | -6.85 | (0.33) |
| C4 | N | C8 | O3 | 2.81 | (0.39) |
| C4 | N | C8 | O4 | -179.07 | (0.21) |
| N | C1 | C2 | C3 | 26.38 | (0.26) |
| C5 | C1 | C2 | C3 | -93.75 | (0.25) |
| N | C1 | C5 | O1 | -25.01 | (0.44) |

| | | | | | |
|---|---|---|---|---|---|
| N | C1 | C5 | O2 | 157.65 | (0.24) |
| C2 | C1 | C5 | O1 | 88.64 | (0.38) |
| C2 | C1 | C5 | O2 | -88.70 | (0.29) |
| C1 | C2 | C3 | C4 | -37.87 | (0.25) |
| C2 | C3 | C4 | N | 33.67 | (0.24) |
| C2 | C3 | C4 | C13 | -85.21 | (0.26) |
| N | C4 | C13 | C14 | 172.69 | (0.23) |
| C3 | C4 | C13 | C14 | -73.22 | (0.31) |
| C4 | C13 | C14 | O5 | 1.61 | (0.41) |
| C4 | C13 | C14 | O6 | -178.42 | (0.23) |

Table of Least-Squares Planes

Orthonormal Equation of Plane 1

| | | | |
|---|---|---|---|
| 0.3974 X | 0.4586 Y | -0.7948 Z - | 1.6564 = 0 |
| 0.0012 | 0.0016 | 0.0011 | 0.0207 |

Crystallographic Equation of Plane

| | | | |
|---|---|---|---|
| 2.3647 X | 4.3288 Y | -28.6266 Z - | 1.6564 = 0 |
| 0.0074 | 0.0148 | 0.0380 | 0.0207 |

| Atom | X | Y | Z | Distance | Esd |
|---|---|---|---|---|---|
| C2 | 1.8018 | 11.0832 | 5.2319 | -0.0162 | +-0.0029 |
| C1 | 1.8685 | 9.7585 | 4.4462 | 0.0273 | +-0.0025 |
| N | 3.1626 | 9.2162 | 4.8516 | -0.0293 | +-0.0022 |
| C4 | 3.9463 | 10.1176 | 5.7038 | 0.0182 | +-0.0026 |

Chi Squared = 377.0

Other Atoms

| | | | | | |
|---|---|---|---|---|---|
| C3 | 3.2733 | 11.4632 | 5.4459 | 0.5728 | +-0.0029 |
| C5 | 1.7888 | 10.0356 | 2.9656 | 1.2995 | +-0.0026 |
| C8 | 3.6705 | 8.0646 | 4.3744 | 0.0238 | +-0.0025 |
| C13 | 3.8836 | 9.6542 | 7.1671 | -1.3824 | +-0.0026 |

Orthonormal Equation of Plane 2

| | | | |
|---|---|---|---|
| 0.0596 X + | -0.9784 Y + | -0.1980 Z - | -10.3128 = 0 |
| 0.0016 | 0.0003 | 0.0016 | 0.0042 |

Crystallographic Equation of Plane

| | | | |
|---|---|---|---|
| 0.3546 X | -9.2354 Y + | -7.1302 Z - | -10.3128 = 0 |
| 0.0093 | 0.0031 | 0.0572 | 0.0042 |

| Atom | X | y | Z | Distance | Esd |
|---|---|---|---|---|---|
| C1 | 1.8685 | 9.7585 | 4.4462 | -0.0038 | +-0.0029 |
| C5 | 1.7888 | 10.0356 | 2.9656 | 0.0135 | +-0.0031 |
| O1 | 2.7029 | 10.2616 | 2.2197 | -0.0055 | +-0.0025 |
| O2 | 0.4941 | 10.0478 | 2.6044 | -0.0042 | +-0.0024 |

Chi Squared = 29.0

Other Atoms

| | | | | | |
|---|---|---|---|---|---|
| C6 | 0.1981 | 10.4832 | 1.2543 | -0.1805+- | 0.0036 |

Orthonormal Equation of Plane 3

| | | | |
|---|---|---|---|
| 0.3580 X + | 0.4791 Y + | -0.8014 Z - | 1.6629 = 0 |
| 0.0011 | 0.0011 | 0.0007 | 0.0121 |

Crystallographic Equation of Plane

| | | | |
|---|---|---|---|
| 2.1303 X | 4.5227 Y | -28.8634 Z - | 1.6629 = 0 |
| 0.0064 | 0.0108 | 0.0262 | 0.0121 |

| Atom | X | Y | Z | Distance | Esd |
|---|---|---|---|---|---|
| N | 3.1626 | 9.2162 | 4.8516 | -0.0029 | +-0.0022 |
| C8 | 3.6705 | 8.0646 | 4.3744 | 0.0096 | +-0.0025 |
| O3 | 4.7899 | 7.6364 | 4.6351 | -0.0037 | +-0.0019 |
| O4 | 2.7575 | 7.4339 | 3.6051 | -0.0030 | +-0.0018 |

Chi Squared = 23.5

Other Atoms

| | | | | | |
|---|---|---|---|---|---|
| C1 | 1.868 | 9.7585 | 4.4462 | 0.1185 | +- 0.0025 |
| C4 | 3.9463 | 10.1176 | 5.7038 | 0.0266 | +-0.0026 |
| C9 | 3.1521 | 6.3805 | 2.6621 | 0.3893 | +-0.0026 |

Orthonormal Equation of Plane 4

| | | | |
|---|---|---|---|
| 0.7379 X + | -0.6448 y + | -0.1993 Z - | -4.7874 = 0 |
| 0.0010 | 0.0011 | 0.0014 | 0.0207 |

Crystallographic Equation of Plane

| | | | |
|---|---|---|---|
| 4.3909 X + | -6.0861 Y + | -7.1791 Z - | 4.7874 = 0 |
| 0.0061 | 0.0105 | 0.0500 | 0.0207 |

| Atom | X | Y | Z | Distance | | Esd |
|---|---|---|---|---|---|---|
| C13 | 3.8836 | 9.6542 | 7.1671 | 0.0000 | +- | 0.0028 |
| C14 | 4.8065 | 10.4274 | 8.0820 | 0.0001 | +- | 0.0028 |
| O5 | 5.5067 | 11.3399 | 7.7234 | -0.0001 | +- | 0.0023 |
| O6 | 4.7274 | 9.9532 | 9.3238 | 0.0000 | +- | 0.0020 |

Chi Squared = 0.0

Other Atoms

| | | | | | | |
|---|---|---|---|---|---|---|
| C4 | 3.9463 | 10.1176 | 5.7038 | 0.0391 | +- | 0.0028 |
| C15 | 5.4849 | 10.5780 | 10.4388 | -0.0661 | +- | 0.0028 |

Dihedral Angles Between Planes:

| Plane No. | Plane No. | Dihedral Angle |
|---|---|---|
| 1 | 2 | 105.52 +- 0.13 |
| 1 | 3 | 2.57 +- 1.92 |
| 1 | 4 | 81.02 +- 0.11 |
| 2 | 3 | 106.79 +- 0.11 |
| 2 | 4 | 44.42 +- 0.14 |
| 3 | 4 | 83.40 +- 0.10 |

CRYSTAL DATA OF ***L*-82** AT -138 0°C

| | |
|---|---|
| formula | $C_{16}H_{27}NO_4$ |
| mol. weight | 297.40 |
| crystal color | colorless, transparent |
| crystal shape | needle |
| crystal dimensions | 0.20 x 0.20 x 1.10 $mm^3$ |
| crystal system | trigonal |
| space group | P 31 |
| space group number | 144 |
| a = b | 10.208(1) Å |
| c | 14.581(2) |
| v | 1315.9(3) $Å^3$ |
| Z | 3 |
| Dcalc | 1.126 g $cm^3$ |
| linear absorption coeff. | 0.76 $cm^{-1}$ |
| radiation | Mo-Kw α |
| scan range | sphere |
| h | -14 > 15 |
| k | -14 > 13 |
| 1 | -21 > 21 |
| ( 2 theta )max | 64° |
| resolution | 0.67 Å |
| number of reflections measured | 23047 |
| number of independent reflections | 5457 |
| reflections used with I > 0 | 5320 |
| number of variables | 298 |
| R(F) | 0.053 |
| wR(F) | 0.036~ |
| s | 0.87 |

A single crystal was measured on a SIEMENS SMART diffractometer at a temperature of about -138°C. Repeatedly measured reflections remained stable. An empirical absorption correction using program SADABS gave an effective transmission range from 0.928 to 1.000 .

Equivalent reflections were averaged. Bijvoet pairs of reflections were not averaged. R(I)internal = 0.031 .The structure was determined by direct methods using program SHELXS. The H atoms were taken from a difference Fourier synthesis and were refined with

isotropic thermal parameters. The non-H atoms were refined with anisotropic thermal parameters. The structure was refined on F values using weighting scheme:
w(F) = 4 * FA^2 / [ sigma^2(F^2) + ( 0.03 * F^2 )A^2]. The final difference density was between -0.20 and +0.23 e/Å$^3$. The calculations were performed with the SMART, SHELX and MolEN program systems.

## DISCUSSION OF THE STRUCTURE

The pyrrolidine ring has a twist conformation with a pseudo-two-fold axis passing through the N atom and the midpoint of the C2-C3 bond. The ring puckering parameters defined by Cremer and Pople (*J. Am. Chem. Soc.*, 1975,97, 1354 ) are q2 = 0.381Å and phi2 89° (phi2 = 90' for an ideal twist). Atoms C2 and C3 lie 0.33 and 0.29Å respectively below and above the plane through Cl, N and C4. The side chains attached to Cl and C4 are both in pseudo-axial positions with respect to the five-membered ring. The N atom shows a small deviation from planarity and lies about 0.11Å outside the plane through Ci, C4 and C12 towards the C11 - H11B bond. The intramolecular N H11B distance of 2.54(2)Å approaches the van der Waals contact distance of 2.5Å and may be classified as a weak, electrostatic interaction. Other intramolecular distances which approach the van der Waals contact distances are O2...H7B: 2.48(2)Å, O2...H9B: 2.45(2)Å, O4...H14C: 2.40(1)Å and O4...H15B: 2.49(2)Å. The shortest intermolecular distances are about 0.2Å longer than the van der Waals contact distance.

Table of Positional Parameters and Their Estimated Standard Deviations

| Atom | x | y | z | B(A2) |
|---|---|---|---|---|
| O1 | 0.81502(8) | 0.24546(8) | 0.27828(0) | 1.97(2) |
| O2 | 0.83861(8) | 0.36301(8) | 0.14259(6) | 2.13(2) |
| O3 | 0.97503(7) | 0.66444(7) | 0.25178(6) | 1.79(2) |
| O4 | 0.87882(9) | 0.76042(8) | 0.14664(6) | 2.50(2) |
| N | 0.73009(9) | 0.53835(9) | 0.22082(7) | 1.70(2) |
| C1 | 0.7174(1) | 0.4096(1) | 0.27100(8) | 1.58(2) |
| C2 | 0.5457(1) | 0.2988(1) | 0.27144(9) | 2.12(2) |
| C3 | 0.4938(1) | 0.3341(1) | 0.18130(9) | 2.44(3) |
| C4 | 0.5906(1) | 0.5069(1) | 0.17329(8) | 2.09(2) |
| C5 | 0.7995(1) | 0.3396(1) | 0.22151(8) | 1.63(2) |
| C6 | 0.8771(1) | 0.1501(1) | 0.24474(8) | 2.2l(2) |
| C7 | 0.7792(2) | 0.0479(1) | 0.16828(9) | 3.17(3) |
| C8 | 0.8645(1) | 0.0574(1) | 0.32950(9) | 2.9l(3) |
| C9 | 1.0413(1) | 0.2508(1) | 0.2174(1) | 2.98(3) |
| C10 | 0.5147(1) | 0.5853(1) | 0.21760(9) | 2.55(3) |
| C10 | 0.5526(1) | 0.6563(1) | 0.2962(1) | 2.95(3) |

| | | | | |
|---|---|---|---|---|
| C12 | 0.8640(1) | 0.6639(1) | 0.20037(8) | 1.77(2) |
| C13 | 1.1348(1) | 0.7751(1) | 0.23378(9) | 2.14(2) |
| C14 | 1.1659(1) | 0.9343(1) | 0.2524(1) | 3.02(3) |
| C15 | 1.1730(1) | 0.7538(2) | 0.1364(1) | 3.49(3) |
| C16 | 1.2165(1) | 0.7307(2) | 0.3026(1) | 3.25(3) |
| Hl | 0.759(1) | 0.436(1) | 0.3314(8) | 1.5(2)* |
| H2B | 0.525(1) | 0.196(1) | 0.2776(8) | 1.7(2)* |
| H2A | 0.503(1) | 0.322(1) | 0.3222(9) | 3.1(3)* |
| H3B | 0.519(1) | 0.288(1) | 0.132(1) | 3.6(3)* |
| H3A | 0.392(1) | 0.302(1) | 0.1816(9) | 3.5(3)* |
| H4 | 0.611(1) | 0.538(1) | 0.1108(9) | 2.5(3)* |
| H7B | 0.785(2) | 0.101(2) | 0.115(1) | 5.0(4)* |
| H7A | 0.672(1) | -0.001(1) | 0.187(1) | 3.2(3)* |
| H7C | 0.807(1) | -0.029(1) | 0.159(1) | 3.9(3)* |
| H8A | 0.896(2) | -0.011(1) | 0.314(1) | 4.7(4)* |
| H8B | 0.921(1) | 0.125(1) | 0.3849(9) | 3.5(3)* |
| H8C | 0.756(1) | -0.002(1) | 0.348(1) | 3.4(3)* |
| H9B | 1.048(1) | 0.315(1) | 0.1652(9) | 3.3(3)* |
| H9A | 1.101(2) | 0.324(2) | 0.272(1) | 5.0(4)* |
| H9C | 1.086(2) | 0.186(2) | 0.204(1) | 4.3(4)* |
| H10 | 0.430(1) | 0.577(1) | 0.1862(9) | 3.4(3)* |
| H11A | 0.498(1) | 0.699(1) | 0.3191(9) | 2.8(3)* |
| H11B | 0.635(1) | 0.665(1) | 0.331(1) | 3.2(3)* |
| H14A | 1.138(2) | 0.945(1) | 0.312(1) | 4.2(3)* |
| H14B | 1.275(2) | 1.003(2) | 0.244(1) | 4.6(4)* |
| H14C | 1.107(1) | 0.962(1) | 0.2115(8) | 2.7(3)* |
| H15C | 1.285(2) | 0.819(2) | 0.130(1) | 5.4(4)* |
| H15B | 1.118(2) | 0.778(2) | 0.092(1) | 4.9(4)* |
| H15A | 1.143(2) | 0.647(2) | 0.129(1) | 5.4(4)* |
| H16B | 1.198(1) | 0.629(1) | 0.290(1) | 4.1(4)* |
| H16C | 1.190(1) | 0.741(2) | 0.3681(9) | 4.1(3)* |
| H16A | 1.323(2) | 0.791(2) | 0.294(1) | 4.5(4)* |

Starred atoms were refined isotropically. Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: (4/3) * [a2 * B (1,1)+b2*B(2,2) + c2 * B (3,3) + ab (cos gamma) * B (1,2) + ac (cos beta)*B (1,3) + bc (cos alpha) * B (2,3)]

Table of General Displacement Parameter Expressions - U's

| Name | U(1,1) | U(2,2) | U(3,3) | U(1,2) | U(1,3) | U(2,3) |
|---|---|---|---|---|---|---|
| O1 | 0.0348(3) | 0.0251(3) | 0.0201(4) | 0.0188(2) | 0.0020(3) | 0.0026(3) |
| O2 | 0.0361(3) | 0.0297(3) | 0.0194(3) | 0.0198(2) | 0.0018(3) | 0.0010(3) |
| O3 | 0.0176(3) | 0.0203(3) | 0.0272(4) | 0.0071(2) | -0.0004(3) | 0.0036(3) |
| O4 | 0.0323(3) | 0.0276(3) | 0.0349(4) | 0.0150(2) | 0.0020(3) | 0.0111(3) |
| N | 0.0188(3) | 0.0216(3) | 0.0254(4) | 0.0110(2) | -0.0018(3) | 0.0026(4) |
| C1 | 0.0212(4) | 0.0180(4) | 0.0194(5) | 0.0087(3) | -0.0004(4) | -0.0003(4) |
| C2 | 0.0223(4) | 0.0228(4) | 0.0300(6) | 0.0072(3) | 0.0017(4) | -0.0009(4) |
| C3 | 0.0222(4) | 0.0339(5) | 0.0310(6) | 0.0099(4) | -0.0051(5) | -0.0077(5) |
| C4 | 0.0228(4) | 0.0336(5) | 0.0223(5) | 0.0136(3) | -0.0029(4) | 0.0015(4) |
| C5 | 0.0218(4) | 0.0168(4) | 0.0211(5) | 0.0080(3) | -0.0018(4) | -0.0012(4) |
| C6 | 0.0413(4) | 0.0283(4) | 0.0240(5) | 0.0245(3) | 0.0008(4) | 0.0009(4) |
| C7 | 0.0626(6) | 0.0333(5) | 0.0332(7) | 0.0304(4) | -0.0076(6) | -0.0075(5) |
| C8 | 0.0554(5) | 0.0365(4) | 0.0306(6) | 0.0321(3) | 0.0039(5) | 0.0069(5) |
| C9 | 0.0413(5) | 0.0446(5) | 0.0384(7) | 0.0298(3) | 0.0078(5) | 0.0091(5) |
| C10 | 0.0251(4) | 0.0388(5) | 0.0381(7) | 0.0196(3) | 0.0003(5) | 0.0101(5) |
| C11 | 0.0410(5) | 0.0421(5) | 0.0417(7) | 0.0304(3) | 0.0050(5) | 0.0021(5) |
| C12 | 0.0242(4) | 0.0218(4) | 0.0231(5) | 0.0128(3) | 0.0015(4) | -0.0010(4) |
| C13 | 0.0174(4) | 0.0244(4) | 0.0359(6) | 0.0078(3) | 0.0042(4) | 0.0022(5) |
| C14 | 0.0280(5) | 0.0248(5) | 0.0537(8) | 0.0071(4) | -0.0022(6) | -0.0020(6) |
| C15 | 0.0371(5) | 0.0517(6) | 0.0434(7) | 0.0219(4) | 0.0137(5) | 0.0018(6) |
| C16 | 0.0204(4) | 0.0406(6) | 0.0594(9) | 0.0129(4) | -0.0057(6) | 0.0021(6) |

The form of the anisotropic displacement parameter is:exp [.-2PI2{h2a2U(1,1) + k2b2U(2,2) + 12c2U(3,3) + 2hkabU(1,2) + 2hlacU(I,3) + 2klbcU(2,3)}] where a,b, and c are reciprocal lattice constants.

Table of Bond Distances in Ångstroms [Å]

| Atom 1 | Atom 2 | Distance | Atom 1 | Atom 2 | Distance |
|---|---|---|---|---|---|
| O1 | C5 | 1.337(1) | C2 | C3 | 1.526(2) |
| O1 | C6 | 1.486(2) | C3 | C4 | 1.535(2) |
| O2 | C5 | 1.202(1) | C4 | C10 | 1.510(2) |
| O3 | C12 | 1.356(2) | C6 | C7 | 1.513(2) |
| O3 | C13 | 1.470(1) | C6 | C8 | 1.522(2) |
| O4 | C12 | 1.208(2) | C6 | C9 | 1.517(2) |
| N | C1 | 1.452(2) | C10 | C11 | 1.307(2) |
| N | C4 | 1.468(2) | C13 | C14 | 1.516(2) |
| N | C12 | 1.360(1) | C13 | C15 | 1.517(2) |
| C1 | C2 | 1.540(1) | C13 | 16 | 1.512(2) |
| C1 | C5 | 1.527(2) | | | |

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

| Atom 1 | Atom 2 | Atom 3 | Angle | Atom 1 | Atom 2 | Atom 3 | Angle |
|---|---|---|---|---|---|---|---|
| C5 | O1 | C6 | 120.69(7) | O1 | C6 | C7 | 110.1(1) |
| C12 | O3 | C13 | 120.53(9) | O1 | C6 | C8 | 102.1(1) |
| C1 | N | C4 | 113.7l(7) | O1 | C6 | C9 | 109.34(9) |
| C1 | N | C12 | 123.8(1) | C7 | C6 | C8 | 110.38(9) |
| C4 | N | C12 | 120.7(1) | C7 | C6 | C9 | 113.5(1) |
| N | C1 | C2 | 102.7(1) | C8 | C6 | C9 | 110.9(1) |
| N | C1 | C5 | 111.34(9) | C4 | C10 | C11 | 125.5(1) |
| C2 | C1 | C5 | 110.35(9) | O3 | C12 | O4 | 126.28(8) |
| C1 | C2 | C3 | 102.99(9) | O3 | C12 | N | 109.1(1) |
| C2 | C3 | C4 | 103.72(9) | O4 | C12 | N | 124.6(1) |
| N | C4 | C3 | 102.3(1) | O3 | C13 | C14 | 110.4(1) |
| N | C4 | C10 | 112.40(9) | O3 | C13 | C15 | 108.85(8) |
| C3 | C4 | C10 | 111.74(9) | O3 | C13 | C16 | 102.57(9) |
| O1 | C5 | O2 | 126.l(1) | C14 | C13 | C16 | 113.0(1) |
| O1 | C5 | C1 | 109.51(9) | C14 | C13 | C16 | 110.5(1) |
| O2 | C5 | C1 | 124.4(1) | C15 | C13 | C16 | 111.0(1) |

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Torsion Angles in Degrees

| Atom 1 | Atom 2 | Atom 3 | Atom 4 | Angle | |
|---|---|---|---|---|---|
| C6 | O1 | C5 | O2 | -3.88 | (0.16) |
| C6 | O1 | C5 | C1 | 173.73 | (0.09) |
| C5 | O1 | C6 | C7 | -59.44 | (0.13) |
| C5 | O1 | C6 | C8 | -176.64 | (0.10) |
| C5 | O1 | C6 | C9 | 65.89 | (0.13) |
| C13 | O3 | C12 | O4 | -11.16 | (0.17) |
| C13 | O3 | C12 | N | 170.75 | (0.09) |
| C12 | O3 | C13 | C14 | 64.55 | (0.14) |
| C12 | O3 | C13 | C15 | -59.98 | (0.13) |
| C12 | O3 | C13 | C16 | -177.63 | (0.10) |
| C4 | N | C1 | C2 | -12.64 | (0.12) |
| C4 | N | C1 | C5 | 105.47 | (0.11) |
| C12 | N | C1 | C2 | -177.58 | (0.10) |
| C12 | N | C1 | C5 | -59.47 | (0.13) |
| C1 | N | C4 | C3 | -11.08 | (0.12) |
| C1 | N | C4 | C10 | 108.88 | (0.11) |
| C12 | N | C4 | C3 | 154.37 | (0.10) |
| C12 | N | C4 | C10 | -85.67 | (0.13) |
| C1 | N | C12 | O3 | -14.52 | (0.15) |
| C1 | N | C12 | O4 | 167.34 | (0.11) |
| C4 | N | C12 | O3 | -178.46 | (0.09) |
| C4 | N | C12 | O4 | 3.41 | (0.18) |
| N | C1 | C2 | C3 | 31.19 | (0.11) |
| C5 | C1 | C2 | C3 | -87.62 | (0.11) |
| N | C1 | C5 | O1 | 166.10 | (0.09) |
| N | C1 | C5 | O2 | -16.24 | (0.15) |
| C2 | C1 | C5 | O1 | -80.48 | (0.11) |
| C2 | C1 | C5 | O2 | 97.18 | (0.13) |
| C1 | C2 | C3 | C4 | -38.57 | (0.12) |
| C2 | C3 | C4 | N | 30.43 | (0.12) |
| C2 | C3 | C4 | C10 | -90.00 | (0.13) |
| N | C4 | C10 | C11 | -9.19 | (0.17) |
| C3 | C4 | C10 | C11 | 105.11 | (0.14) |

Table of Least-Squares Planes

Orthonormal Equation of Plane 1

| | | | |
|---|---|---|---|
| 0.5084 X + | -0.2863 Y | -0.8121 Z - | -1.5850 = 0 |
| 0.0009 | 0.0014 | 0.0004 | 0.0098 |

Crystallographic Equation of Plane

| | | | |
|---|---|---|---|
| 5.1903 X | -5.1262 Y | -11.8410 Z - | -1.5850 = 0 |
| 0.0092 | 0.1540 | 0.0059 | 0.0098 |

| Atom | x | y | Z | Distance | | Esd |
|---|---|---|---|---|---|---|
| C1 | 5.2329 | 3.6212 | 3.9513 | 0.0000 | +- | 0.0012 |
| N | 4.7052 | 4.7594 | 3.2196 | 0.0000 | +- | 0.0010 |
| C4 | 3.4416 | 4.4812 | 2.5266 | 0.0000 | +- | 0.0012 |
| C2 | 4.0453 | 2.6413 | 3.9577 | -0.3285 | +- | 0.0013 |
| C3 | 3.3353 | 2.9540 | 2.6434 | 0.2883 | +- | 0.0013 |
| C12 | 5.4315 | 5.8693 | 2.9216 | 0.2936 | +- | 0.0012 |
| O3 | 6.5619 | 5.8741 | 3.6711 | 0.2583 | +- | 0.0008 |
| O4 | 5.0900 | 6.7226 | 2.1381 | 0.5119 | +- | 0.0009 |
| C5 | 6.4281 | 3.0023 | 3.2298 | 1.3708 | +- | 0.0012 |
| H1 | 5.5230 | 3.8584 | 4.8327 | -0.6362 | +- | 0.0115 |
| C10 | 2.2662 | 5.1746 | 3.1726 | -1.3208 | +- | 0.0013 |
| H4 | 3.4919 | 4.7565 | 1.6152 | 0.6869 | +- | 0.0133 |

Orthonormal Equation of Plane 2

| | | | | |
|---|---|---|---|---|
| 0.4741 X + | -0.4947 Y | + | -0.7284 Z - | -2.4661 = 0 |
| 0.0005 | 0.0005 | | 0.0004 | 0.0047 |

Crystallographic Equation of Plane

| | | | | |
|---|---|---|---|---|
| 4.8393 X + | -6.7927 Y | + | -10.6207 Z - | -2.4661 = 0 |
| 0.0049 | 0.1115 | | 0.0053 | 0.0047 |

| Atom | X | Y | Z | Distance | | Esd |
|---|---|---|---|---|---|---|
| N | 4.7052 | 4.7594 | 3.2196 | -0.0029 | +- | 0.0009 |
| C12 | 5.4315 | 5.8693 | 2.9216 | 0.0096 | +- | 0.0012 |
| O3 | 6.5619 | 5.8741 | 3.6711 | -0.0029 | +- | 0.0008 |
| O4 | 5.0900 | 6.7226 | 2.1381 | -0.0038 | +- | 0.0009 |

Chi Squared = 108.1

Other Atoms

| | | | | | |
|---|---|---|---|---|---|
| C13 | 7.6278 | 6.8521 | 3.4086 | 0.2098 | +-0.0012 |

Orthonormal Equation of Plane 3

-0.5826 X + -0.7581 Y + -0.2930 Z - -6.9550 = 0

0.0005 0.0003 0.0005 0.0020

Crystallographic Equation of Plane

-5.9469 X + -3.7290 Y + -4.2724 Z - -6.9550 = 0

0.0048 0.1106 0.0069 0.0020

| Atom | X | Y | Z | Distance | Esd |
|---|---|---|---|---|---|
| C1 | 5.2329 | 3.6212 | 3.9513 | 0.0034 | 0.0011 |
| C5 | 6.4281 | 3.0023 | 3.2298 | -0.0123 | 0.0011 |
| O1 | 7.0672 | 2.1700 | 4.0574 | 0.0039 | 0.0007 |
| O2 | 6.7079 | 3.2093 | 2.0790 | 0.0050 | 0.0008 |

Chi Squared = 175.7

Other Atoms

| | | | | | |
|---|---|---|---|---|---|
| C6 | 8.1881 | 1.3268 | 3.5684 | 0.1335 | 0.0012 |

Dihedral Angles Between Planes:

| Plane No. | PlaneNo. | Dihedral Angle |
|---|---|---|
| 1 | 2 | 13.04 +- 0.25 |
| 1 | 3 | 80.86 +- 0.08 |
| 2 | 3 | 71.80 +- 0.04 |

## 11.1 Anmerkung:

Die Röntgenstrukturdaten der folgenden Verbindungen ***L*-40**, ***L*-52**, ***L*-64** und ***L*-65** sind beim Cambridge Crystallographic Data Centre hinterlegt.

## 11.2 Verwendete Abkürzungen:

**äq.** - Moläquvalent, **ber.** - berechnet, **DMAP** - 4-(N,N-Dimethyamino)pyridin, **DME** - Dimethoxyethan, **DMF** - N,N-Dimetylformamid, **DIBAH** - Diisobutylaluminiumhydrid, ***E*** - Entgegen, **Ether** - Diethylether, **EE** - Essigsäureethylester, **Fp.** - Schmelzpunkt, **gef.** - gefunden, **Hex.** - n-Hexan, **HRMS** - hochauflösende Massenspektroskopie, **Hünig-Base** - *N*-Ethyldiisopropyl, **HPLC** - Hochdruchflüssigkeitschromatographie, **Kp.** - Siedepunkt, **RT** - Raumtemperatur, **Lsg.**- Lösung, **PG** - allgemeine Schutzgruppe, ***tert.*** - tertiär, **Z** - Zusammen.