Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry

Supporting Information

A new and convenient approach for the preparation of ß-cyanoethyl protected trinucleotide phosphoramidites

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Index

Contents of Supporting Information page #

General information

Dry methylene chloride, dry methanol and dry tetrahydrofuran were obtained from Fluka. Pyridine was dried overnight over KOH, heated to reflux for 4 h, distilled off and stored over molecular sieve. Diisopropyl ethyl amine was stored over calcium hydride and distilled before use. All other reagents, chemicals, buffers and solvents were obtained as the highest commercially available grade. Solvents used for column chromatographie were freshly distilled. Other chemicals were used without further purification. Reactions were carried out at room temperature unless stated otherwise. Silica gel for column chromatography (0.063-0.2 mm) was obtained from Sigma-Aldrich. All products were visualized on TLC plates (Macherey-Nagel Fertigfolien Alugram Sil G/UV_{254}) at 254 nm ultraviolet light. Reversed-phase HPLC were performed on an ÄKTA Purifier (GE Healthcare); Column EC 250/4 Nucleodur 100-5 C18 ec (Macherey-Nagel), flow rate 1 ml/min, Buffer A 5% acetonitrile in deionized water, Buffer B 70% acetonitrile in deionized water. NMR spectra were recorded with TMS as the internal standard on Bruker Avance 300 MHz. Mass spectra were recorded on a Bruker Microflex MALDI-TOF, VG Autospec (FAB), and ESI Bruker Esquire LC (Ion Trap), respectively. UV measurements were obtained from an Ultrospec 2100 pro (Amersham Biosciences). The hexamers were synthesized by the phosphoramidite method on a DNA-synthesizer (Gene Assembler Special, Pharmacia) at 0.2 µmol scale as described elsewhere.[1] Standard PAC-phosphoramidites as well as CPG supports were obtained from ChemGenes or Link Technologies. BMT (emp Biotech) was used as activator.[2]

1. *N*-acylation[3]

10 mmol of either dA, dG or dC were coevaporated twice with 50 ml dry pyridine in a 500 ml round-bottom flask and finally suspended in 100 ml dry pyridine. After cooling down in an ice-bath 5 ml trimetylchlorsilane (approximately 5 equivalents) were added slowly. After 30 minutes 5 equivalents of the acylation reagent were added (8.5 ml isobutyric anhydride for dG and dA, 6 ml benzoyl chloride for dC). The solution was stirred for 2 h. The reaction was stopped by adding 20 ml cold water to the solution followed by 20 ml of concentrated ammonia. The reaction solution was

kept in an ice-bath for 30 minutes. After concentration *in vacuo* the resulting oil was dissolved in 50 ml water. The aqueous layer was washed with small amount of ether. In the case of dG crystallization from water begins within minutes. *N*-acylated dA and dC were purified from anorganic salts by silica chromatography (CH_2Cl_2:MeOH 80:20). R_f-Wert (CH_2Cl_2:MeOH 8:2): 0.5.

2. 5'-Dimethoxytritylation[3]

10 mmol of *N*-acylated desoxyribonucleoside (or thymidine) were coevaporated twice with 50 ml dry pyridine and dissolved in 100 ml dry pyridine. Dimethylaminopyridine (DMAP) (61 mg, 0.05 mmol) and dry triethylamine (TEA) (2 ml, 15 mmol) were added. After adding dimethoxytritylchloride (4.7 g, 15 mmol) the solution was stirred for 4 h. The reaction was stopped by adding 200 ml water. The product was extracted with ether. The ether was removed in vacuo and the crude product was purified via silica gel chromatography (CH_2Cl_2:MeOH = 99:1→95:5). R_f (CH_2Cl_2:MeOH 9:1): 0.5.

3. 3'-Silylation[4]

5 mmol of 5'-dimetoxytritylated desoxynucleoside were dissolved in 20 ml anhydrous tetrahydrofuran and 3 ml anhydrous pyridine. $AgNO_3$ (2.2 g, 1.3 equivalents) and TBDMSCl (2 g, 1.3 equivalents) were added and the mixture was stirred overnight. After adding saturated $NaHCO_3$-solution the crude compound was isolated with dichloromethane. After washing with sat. aq. $NaHCO_3$ the organic layer was dried with sodiumsulfate. After removing the solvents *in vacuo* the silylated nucleoside was used for further synthesis without any purification. R_f (CH_2Cl_2, MeOH 95:5): 0.8.

4. Removal of the 5'-DMT group[5]

5 mmol of 3'-silylated-5'-tritylated desoxyribonucleoside were dissolved in 100 ml of dichlormethane containing 3% trichloracetic acid. The mixture was stirred for 10 minutes and poured into saturated $NaHCO_3$ in a separating funnel. After washing the organic phase with sat. aq. $NaHCO_3$ the organic layer was dried with sodiumsulfate. The solvent was removed *in vacuo*, the product was purified via silica gel chromatography. R_f-Wert (CH_2Cl_2:MeOH 9:1): 0.5.

5. Nucleoside Phosphoramidite[3]

1 mmol of the 5'-DMT protected desoxynucleoside was coevaporated twice with 5.5 ml of a mixture of dry dichlormethane/dry pyridine (20:1) and dissolved in 10 ml dry dichlormethane. 1 ml freshly distilled anhydrous diisopropylethylamine was added to the mixture. 1.2 equivalents of 2-cynoethyl-*N,N*-diisopropylchlorphosphoramidit were slowly added while stirring. After five minutes 1 ml anhydrous methanol, five minutes later 4 ml triethylamine and finally 10 ml ethylacetate were added. The mixture was washed twice with sat. aq. $NaHCO_3$ and twice with brine. The organic layer was dried with sodiumsulfate and the solvent was removed *in vacuo*. The crude product could be used for coupling reactions without further purification. R_f-Wert (CH_2Cl_2 : :ethyl acetate: triethylamine 45:45:10): 0.9 (Desoxyguanosin: 0.4).

6. Coupling reaction I[5]

1 mmol nucleoside phosphoramidite and 0.8 mmol 5'-deprotected nucleoside were dissolved in 10 ml anhydrous acetonitrile. Tetrazole (10 mmol) was added. After 30 minutes a 0.1 M solution of iodine in lutidine/tetrahydrofuran/water 1:2:1 was added to the mixture until no decolourization of the mixture was detectable. The solvents were removed in vacuo, the remaining brown foam was dissolved in 50 ml dichlormethane. The organic layer was washed with 50 ml 6% sodiumbicarbonate, then with 50 ml sat. aq. $NaHCO_3$ and with 50 ml water. After the organic layer was dried with Na_2SO_4 the solvent was removed in vacuo. The dinucleotide was purified by silica gel chromatography (CH_2Cl_2:MeOH = 9:1, Rf = 0.6). (See table S1 for yields and MALDI results.)

7. Removal of the 5'-DMT group[5]

The dinucleotide was dissolved in 10 ml dichlormethane containing 3 % of trichloracetic acid. The mixture was washed with saturated $NaHCO_3$-solution. The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo*. The product was purified by silica gel chromatography (CH_2Cl_2: MeOH = 9:1; R_f = 0.4). (See table S1 for yields and MALDI results.)

8. Coupling reaction II[5]

1.2 mmol of the desoxyribonucleoside phosphoramidite were dissolved in 10 ml anhydrous acetonitrile. 1 mmol of the 5'-unprotected dinucleotide was added to the mixture. After two minutes tetrazole (10 mmol) was added, the mixture was stirred for another 1 h. A 0.1 M solution of iodine in lutidine/tetrahydrofuran/water 1:2:1 was given to the mixture until no decolourization of the iodine solution was detectable. The solvents were removed *in vacuo*, and the remaining brown foam was dissolved in 50 ml dichlormethane. The organic layer was washed with 50 ml 6% sodiumbicarbonate solution, 50 ml saturated $NaHCO_3$-solution and 50 ml water. The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo*. The trinucleotide was purified by silica gel chromatography (CH_2Cl_2:MeOH 95:5; R_f = 0.7). (See table S2 for yields and MALDI results.)

9. Deprotection of the 3'-OH-group[5]

0.5 mmol of the 3'-TBDMS protected trinucleotide was dissolved in 10 ml of anhydrous dimethylformamide. 0.25 ml of TEA/3xHF was added. The mixture was stirred over night until no starting material was detectable on TLC. The mixture was diluted with 50 ml saturated $NaHCO_3$, the product was extracted with 30 ml dichlormethane. The organic layer was dried with Na_2SO_4, the solvents were removed *in vacuo* and the 3'-unprotected trinucleotide was purified by RP-HPLC (for conditions see General Information, page S3). The yield was virtually quantitative in all cases. The purity of all compounds was controlled and confirmed by thin layer chromatography and in some cases additionally by HPLC. The identity of all compounds was confirmed by MALDI mass spectrometry. (see experimental data in next chapter and figure S1a and S1b).

10. Trinucleotide phosphoramidite[3]

0.5 mmol of the 3'-unprotected trinucleotide were dissolved in 5 ml anhydrous dichlormethane. 0.55 mmol of 2-cyanoethyl-*N,N,N',N'*-tetraisopropylphosphoramidite reagent and 0.6 mmol BMT were added to this mixture which was stirred for two hours. After completed reaction the solvent was removed under reduced pressure. After coevaporation with 2 ml of anhydrous dichlormethane the phosphoramidite was

dried over night at oil pump vacuum. An aqueous workup did not take place. The trinucleotide was used for solid phase synthesis. R_f-Wert (CH_2Cl_2:MeOH 9:1): 0.7. See below for analytical data.

Analytical data of 5'-*O*-DMT 3'-OH-trinucleotides

DMTAAAOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.78, -2.74 (2 x phosphortriester diastereomer), -2.58 (br, 2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1496.42 g/mol, found: 1519.15 g/mol (Na-peak).

UV: λ_{max} = 258 nm.

DMTACCOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.572 (br, 2 x phosphortriester diastereomer), -2.467, -2.435 (2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1516.4 g/mol, found: 1539.753 g/mol (Na-peak).

UV: λ_{max} = 260 nm.

DMTATCOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.62, -2.60, -2.55, -2.42 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1427.3 g/mol, found: 1450.805 g/mol (Na-peak).

UV: λ_{max} = 259 nm.

DMTATGOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.64 (br, 2 x phosphortriester diastereomer), 2.56 (br, 2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1432.47 g/mol, found: 1456.21 g/mol (Na-peak).

UV: λ_{max} = 261 nm.

DMTCACOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.83, -2.73, -2.42, -2.40 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1516.4 g/mol, found: 1540.04 g/mol (Na-peak).

UV: λ_{max} = 259 nm.

DMTCAGOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.85, -2.79 (2 x phophostriester diastereomer), -2.53 (br, 2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1522.4 g/mol, found: 1545.6 g/mol (Na-peak).

UV: λ_{max} = 258 nm.

DMTCATOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.83, -2.74 (2 x phosphortriester diastereomer), -2.46 (2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1426.45 g/mol, found: 1450.84 g/mol (Na-peak).

UV: λ_{max} = 260 nm.

DMTCCAOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.68, -2.60 (2 x br, 4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1516.40 g/mol, found: 1539.51 g/mol (Na-peak).

UV: λ_{max} = 259 nm.

DMTCTGOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.71, -2.62 (2 x phosphortriester diastereomer), -2.54 (br, 2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1442.44 g/mol, found: 1466.22 g/mol (Na-peak).

UV: λ_{max} = 259 nm.

DMTGATOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -3.14, -2.85 (2 x br, 4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1433,31 g/mol, found: 1456,41g/mol (Na-peak).

UV: λ_{max} = 260 nm.

DMTGCAOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -3.69, -3.65, 2.70, 2.60 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1521.49 g/mol, found: 1545.68 g/mol (Na-peak).

UV: λ_{max} = 259 nm.

DMTGCGOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.84, -2.74, -2.56, -2.45 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1537.49 g/mol, found: 1561.76 g/mol (Na-peak).

UV: λ_{max} = 261 nm.

DMTGCTOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.67--2.45 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1443.30 g/mol, found: 1466.07 g/mol (Na-peak).

UV λ_{max} = 259 nm.

DMTGGCOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.69, 2.65, -2.47, -2.39 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1538.41 g/mol, found: 1561.50 g/mol (Na-peak).

UV: λ_{max} = 258 nm.

DMTGGTOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.70 (br, 2 x phosphortriester diastereomer), -2.51, -2.49 (2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1449.31 g/mol, found: 1472.75 g/mol (Na-peak).

UV: λ_{max} = 258 nm.

DMTGTAOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.70, 2.64 (2 x phosphortriester diastereomer), -2.58 (br, 2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1433.31 g/mol, found: 1456.38 g/mol (Na-peak).

UV λ_{max} = 259 nm.

DMTGTTOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.5.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.70 (br, 2x phosphortriester diastereomer), -2.51, -2.49 (2 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1354.41 g/mol, found: 1377.12 g/mol (Na-peak).

UV: λ_{max} = 261 nm.

DMTTGGOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.6

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.80, -2.66, -2.65, -2.50 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1449.31 g/mol, found: 1472.47 g/mol (Na-peak).

UV: λ_{max} = 258 nm.

DMTTTCOH:

R_f- value (CH_2Cl_2: methanol = 9:1) = 0.5

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = -2.70, -2.52, -2.46, -2.43 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1347.40 g/mol, found: 1371.32 g/mol (Na-peak).

UV: λ_{max} = 260 nm.

DMTTTTOH:

R_f-value (CH_2Cl_2: methanol = 9:1) = 0.5.

^{31}P-NMR (DMSO-d^6, 300 MHz): δ = 2.71, -2.53, -2.48, -2.45 (4 x phosphortriester diastereomer).

MALDI$^+$-MS: calculated: 1258.37 g/mol, found: 1281.89 g/mol (Na-peak).

UV: λ_{max} = 259 nm.

Table S1. MALDI data and yields of dinucleotide compounds. Found masses are given as Na-peak (m/z).

Dinucleotide	5'-*O*-DMT 3'-*O*-TBDMS			5'-OH 3'-*O*-TBDMS		
	found. mass	calcd. mass	yield (%)	found mass	calcd. mass	yield (%)
AA	1196	1173	79	894	871	81
AC	1206	1183	83	904	881	85
AG	1212	1189	76	910	887	80
AT	1117	1094	89	815	792	82
CA	1206	1183	90	904	881	91
CC	1216	1193	87	914	891	86
CG	1222	1199	79	920	897	88
CT	1127	1104	87	825	802	85
GC	1222	1199	80	920	897	89
GG	1228	1205	65	926	903	81
GT	1133	1110	82	831	808	87
TA	1117	1094	80	815	792	81
TC	1127	1104	83	825	802	86
TG	1133	1110	81	831	808	82
TT	1038	1015	90	736	713	88

Table S2. MALDI data and yields of trinucleotide compounds. Found masses are given as Na-peak (m/z).

Trinucleotide	5'-*O*-DMT 3'-*O*-TBDMS			5'-*O*-DMT 3'-OH*	
	found mass	calcd. mass	yield (%)	found mass	calcd. mass
AAA	1633	1610	63	1519	1496
ACC	1653	1630	71	1539	1516
ATC	1563	1540	71	1450	1427
ATG	1569	1546	75	1456	1433
CAC	1654	1629	68	1540	1516
CAG	1658	1635	73	1545	1522
CAT	1563	1540	75	1450	1426
CCA	1652	1629	59	1539	1516
CTG	1579	1556	71	1466	1442
GAT	1569	1546	71	1456	1433
GCA	1658	1635	69	1545	1522
GCG	1674	1651	65	1562	1537
GCT	1580	1557	56	1466	1443
GGC	1674	1651	64	1562	1538
GGT	1586	1563	69	1472	1449
GTA	1569	1546	66	1456	1433
GTT	1491	1468	58	1377	1354
TGG	1586	1563	56	1472	1449
TTC	1484	1461	70	1371	1348
TTT	1395	1372	55	1281	1258

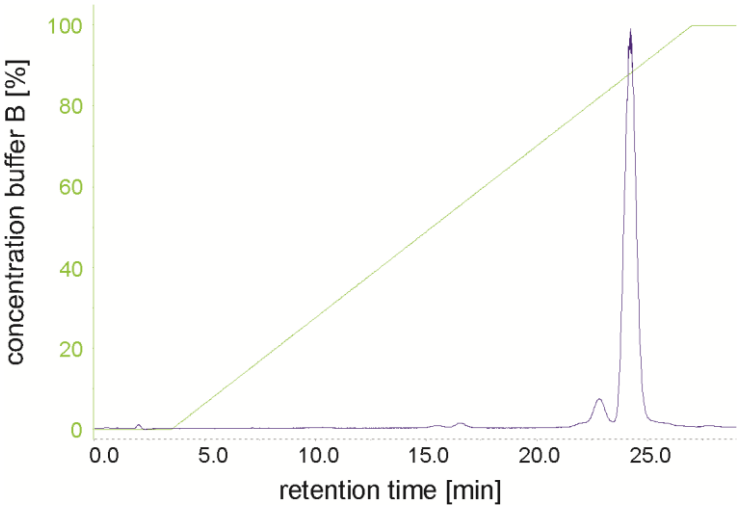
* removal of the 3'-*O*-TBDMS group was virtually quantitative in all cases

Table S3. MALDI data of hexamers. Found masses are given as Na-peak (m/z). Coupled phosphoramidite trinucleotide building blocks are given in bold letters.

6mer 5' → 3'	calcd. mass	found mass
AAATTA	1798	1798
ACCTTC	1726	1726
ATCTTA	1765	1765
ATGTTC	1781	1781
CACTTA	1750	1750
CAGTTC	1766	1766
CATTTA	1765	1765
CCATTC	1726	1726
CTGTTA	1781	1779
GATTTA	1805	1805
GCATTC	1766	1766
GCGTTC	1782	1782
GCTTTC	1757	1757
GGCTTA	1806	1806
GGTTTA	1821	1821
GTATTA	1805	1805
GTTTTC	1772	1772
TGGTTA	1821	1821
TTCTTA	1756	1756
TTTTTA	1771	1771

Figure S1

a)



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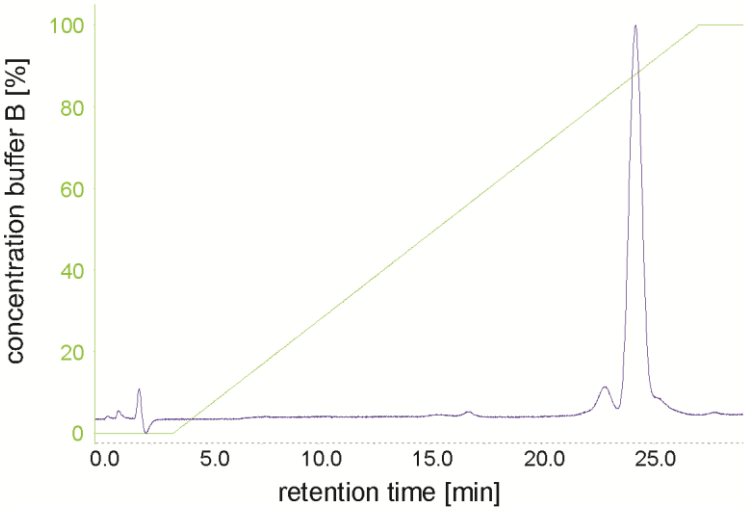

Figure S1: HPLC diagram of pure compounds a) DMT-GCG-OH (88% buffer B) and b) DMT-TGG-OH (87% buffer B); gradient: 0% B for 5 min, 0 – 100% B in 23.5 min, 100% B for 30 min.

Literature

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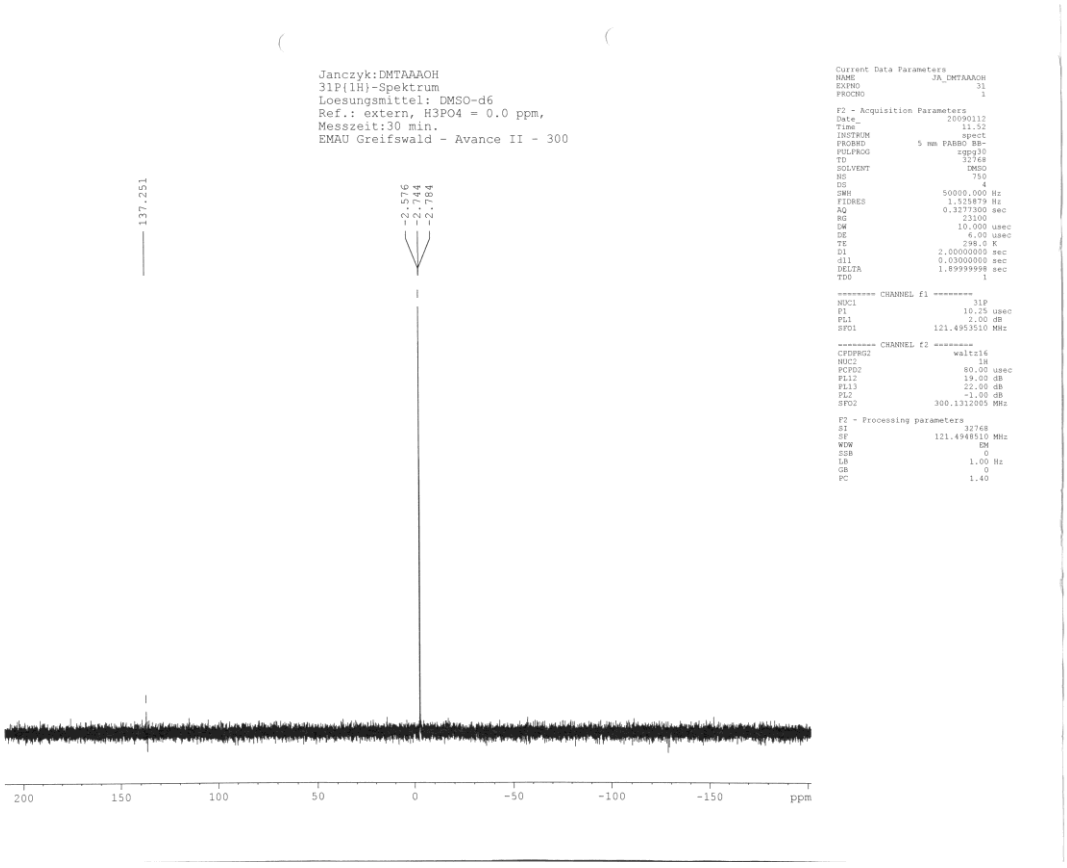
Appendix

^{31}P NMR spectra of data of 5'-*O*-DMT 3'-OH-trinucleotides

DMTAAAOH



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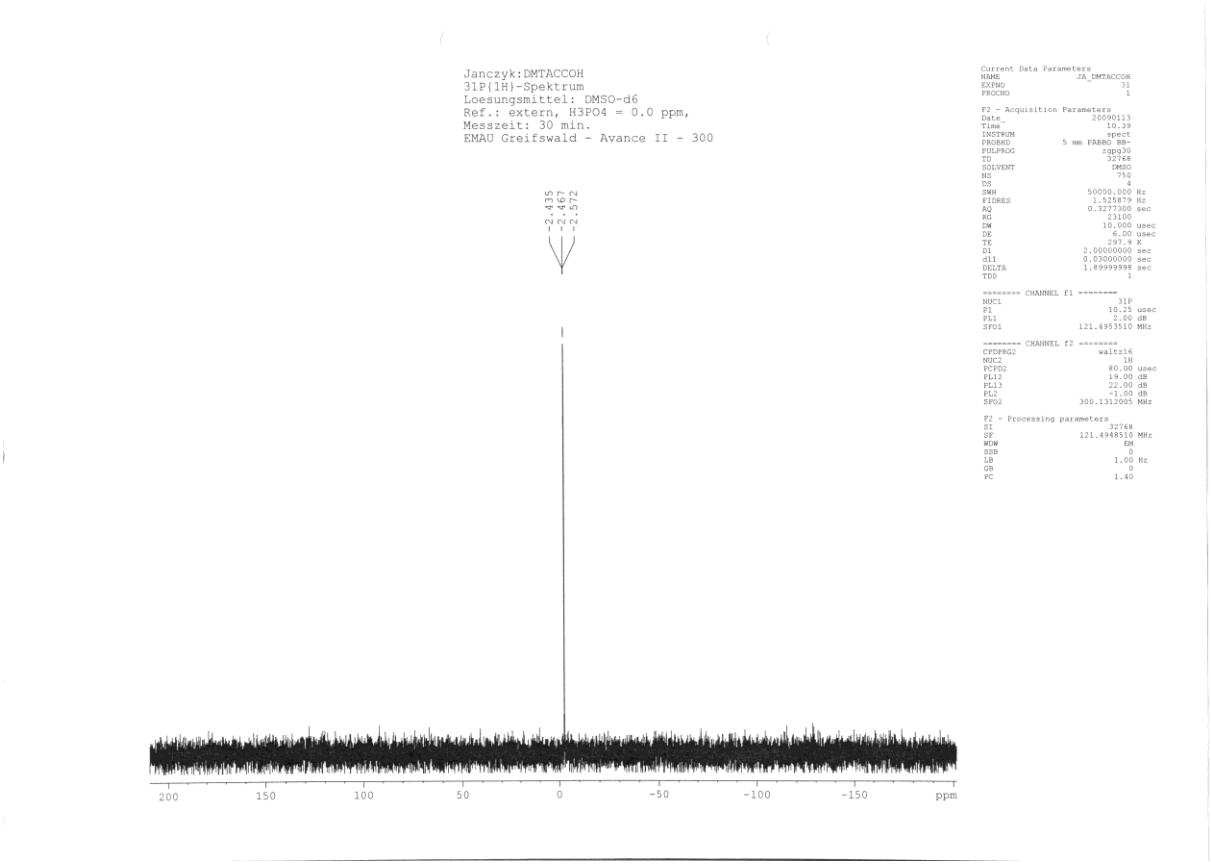
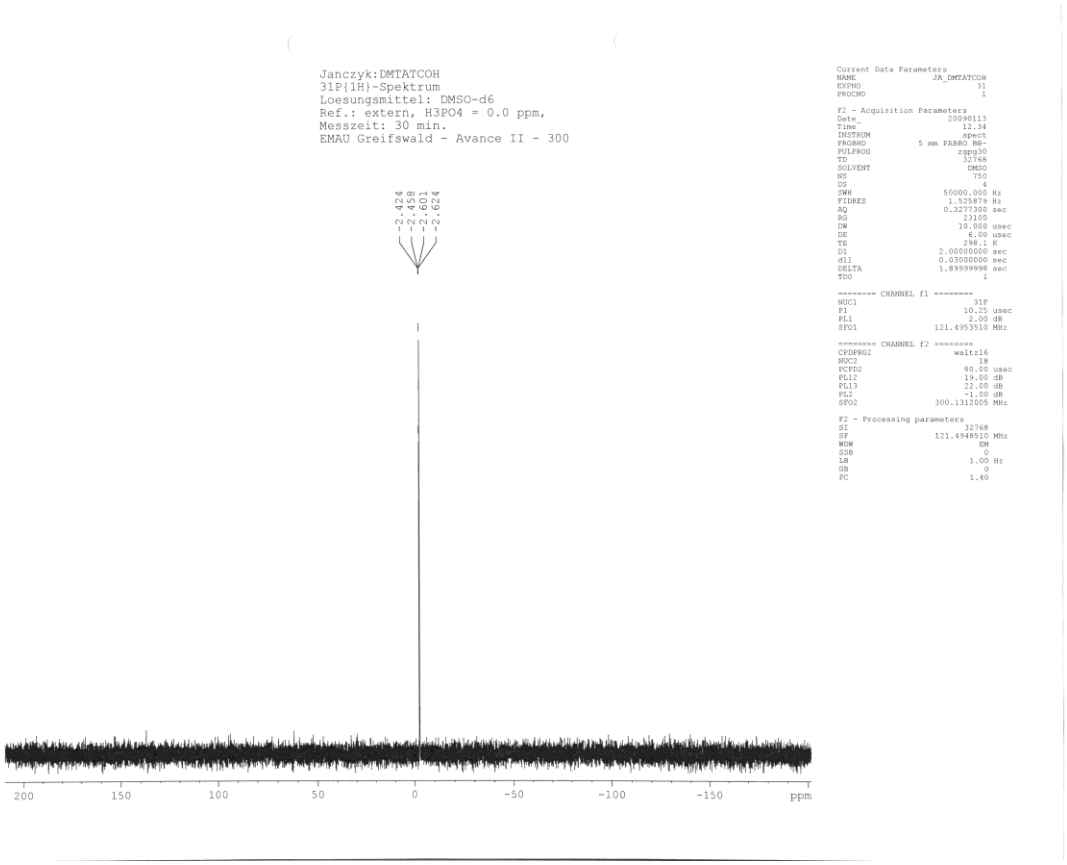

Janczyk:DMTACCOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 30 min.
EMAU Greifswald - Avance II - 300
-2.435
-2.467
-2.572
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTACCOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090113
Time 10.39
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 756
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DMTATCOH


Janczyk:DMTATCOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 30 min.
EMAU Greifswald - Avance II - 300
-2.424
-2.458
-2.491
-2.524
200
150
100
50
0
-50
-100
-150
ppm
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PROCNO 1
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PROBHD 5 mm PABBO BB-
PULPROG zgpg30
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SOLVENT DMSO
NS 750
DS 4
SWH 50000.000 Hz
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DE 6.00 usec
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D1 2.00000000 sec
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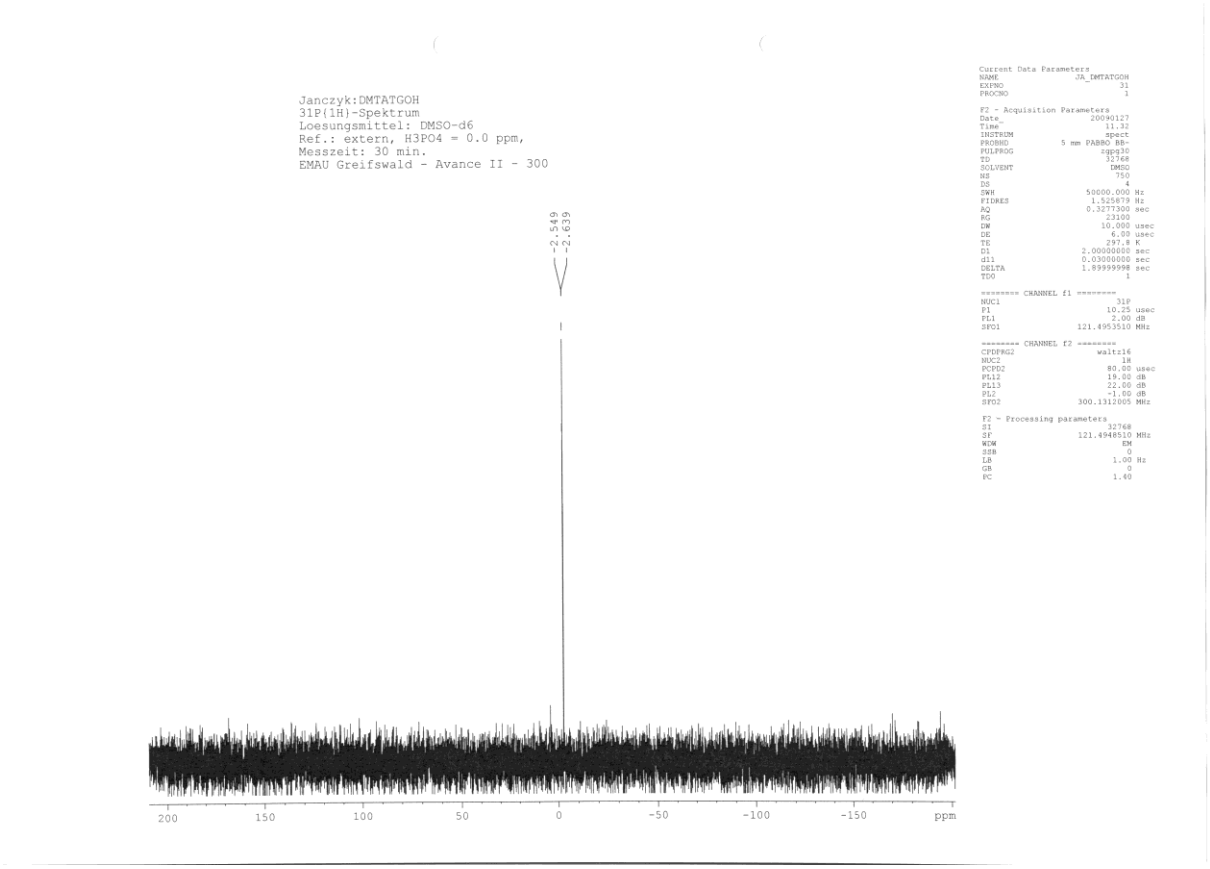
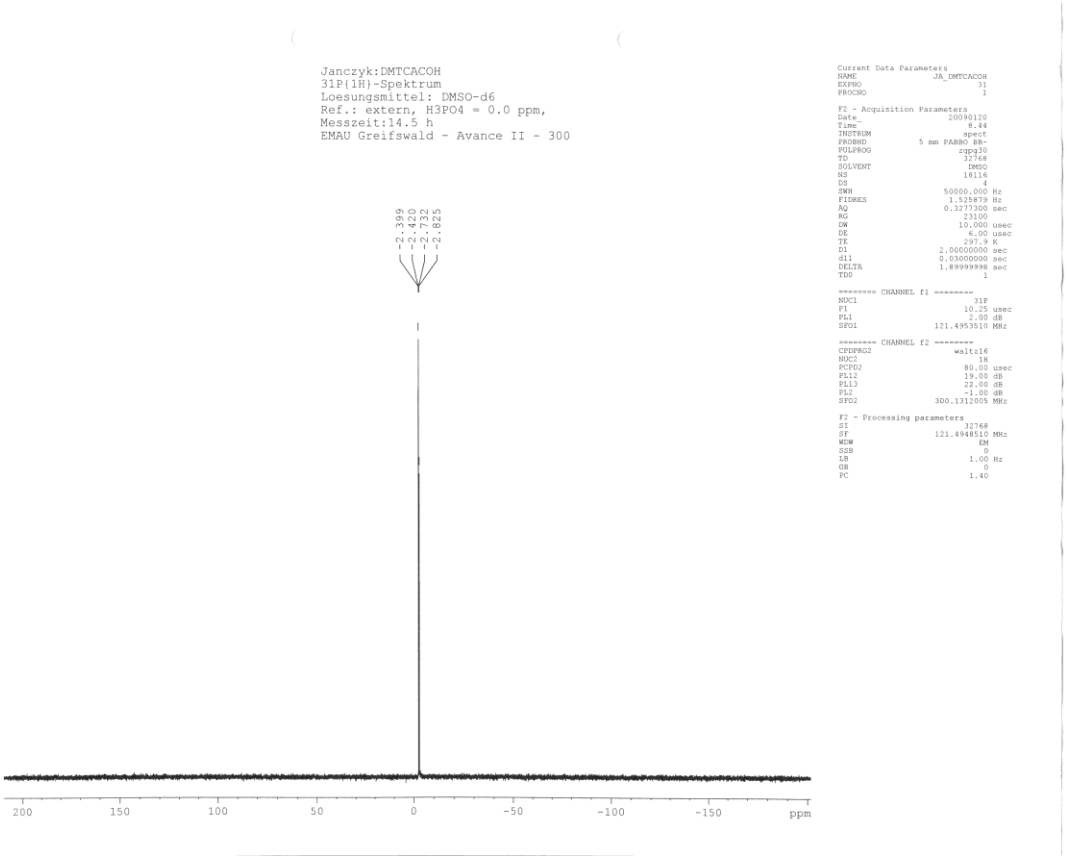
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

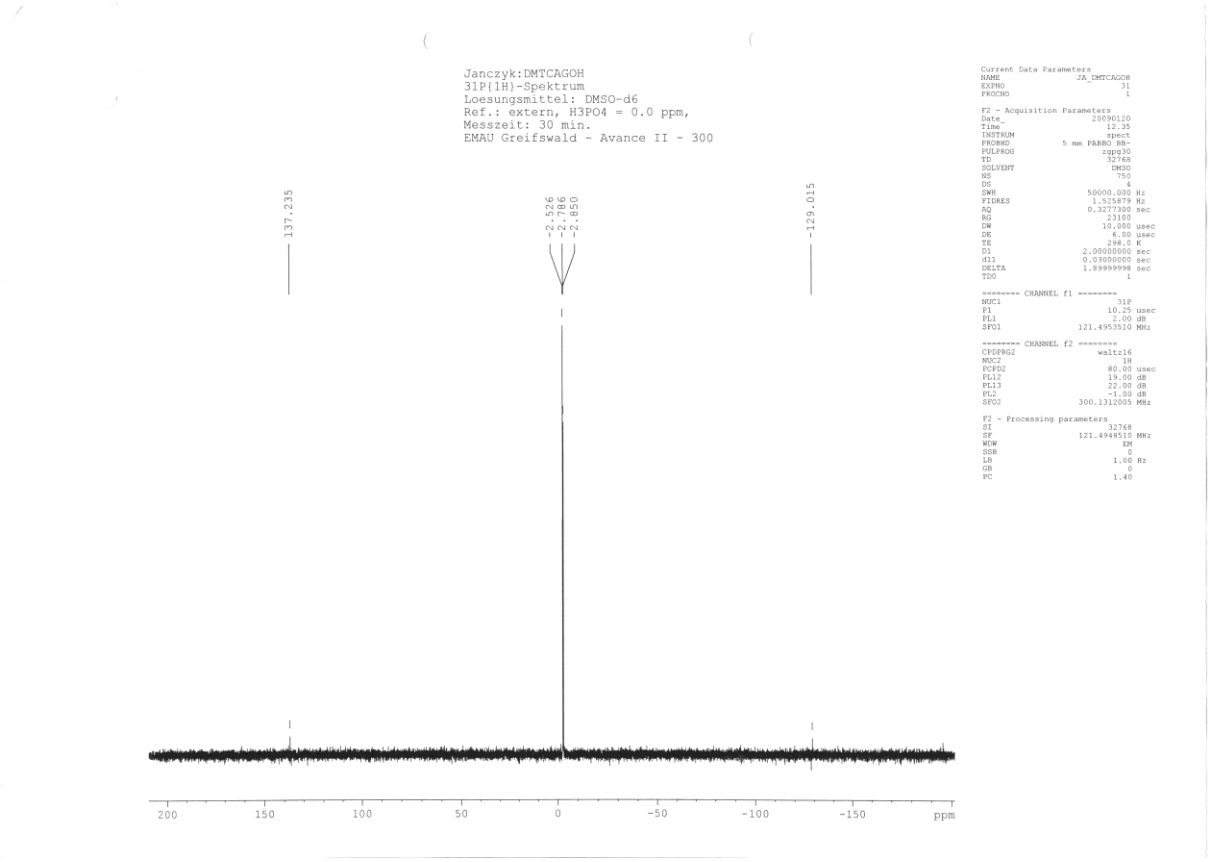
DMTATGOH


Janczyk:DMTATGOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 30 min.
EMAU Greifswald - Avance II - 300
2.549
2.639
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTATGOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090127
Time 11.32
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 750
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

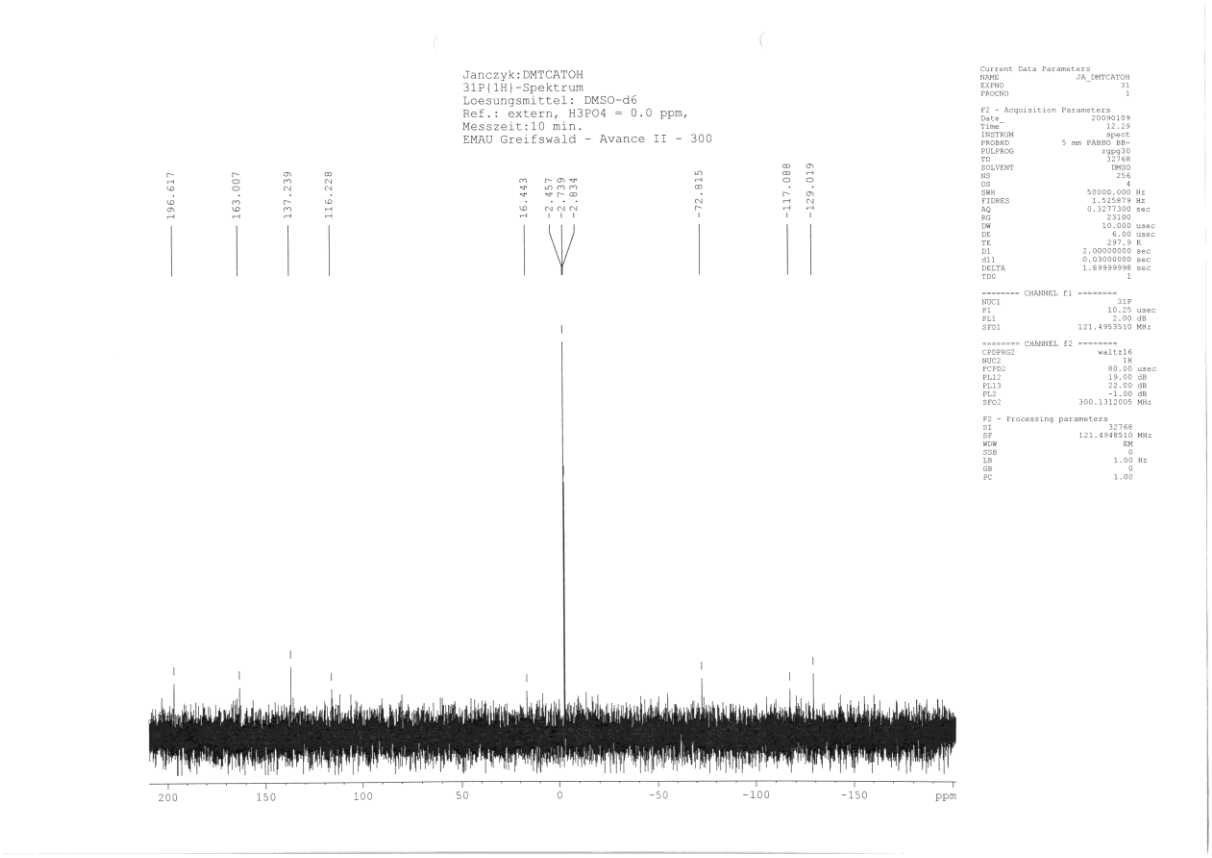
DMTCACOH


Janczyk:DMTCACOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14.5 h
EMAU Greifswald - Avance II - 300
2.39
2.36
2.32
2.29
2.25
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTCACOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090120
Time_ 8.44
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 18116
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

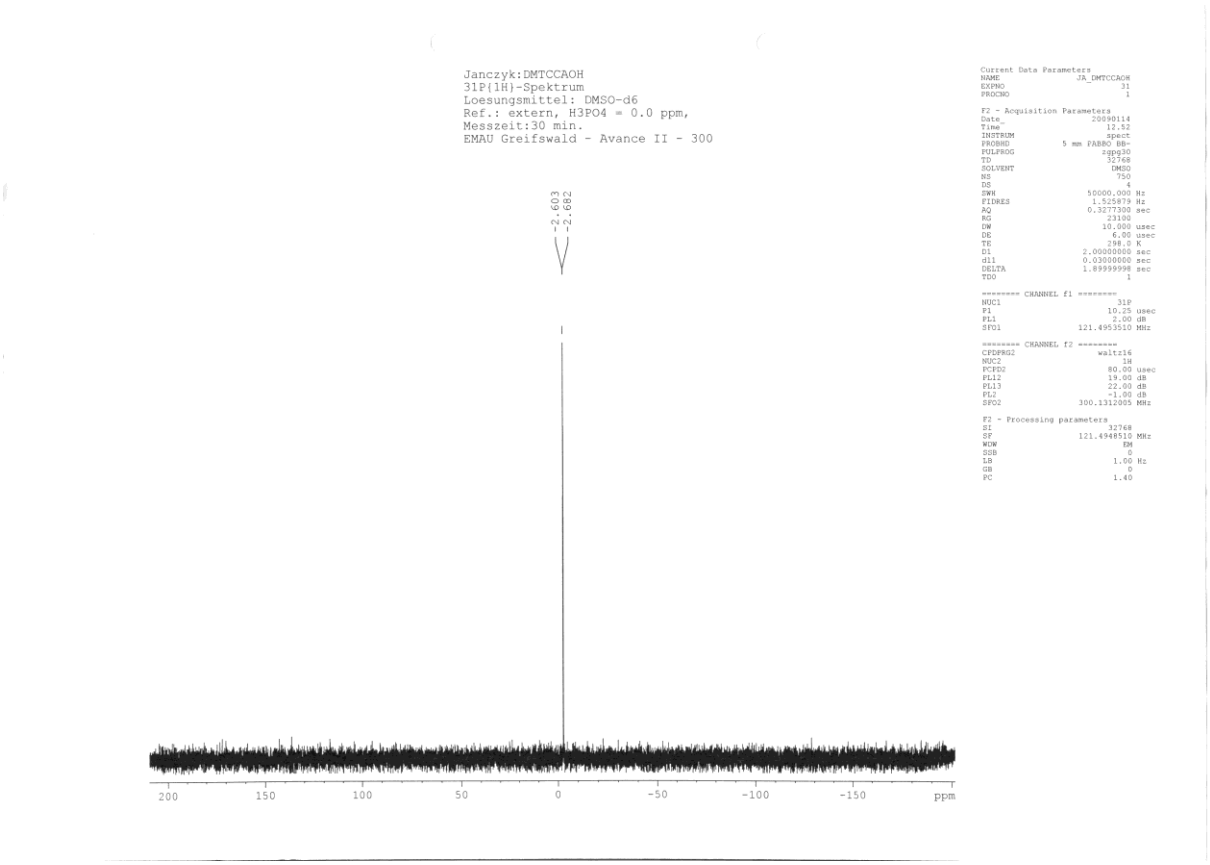
DMTCAGOH


Janczyk:DMTCAGOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 30 min.
EMAU Greifswald - Avance II - 300
137.235
2.526
2.766
2.850
129.015
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTCAGOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090120
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 750
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

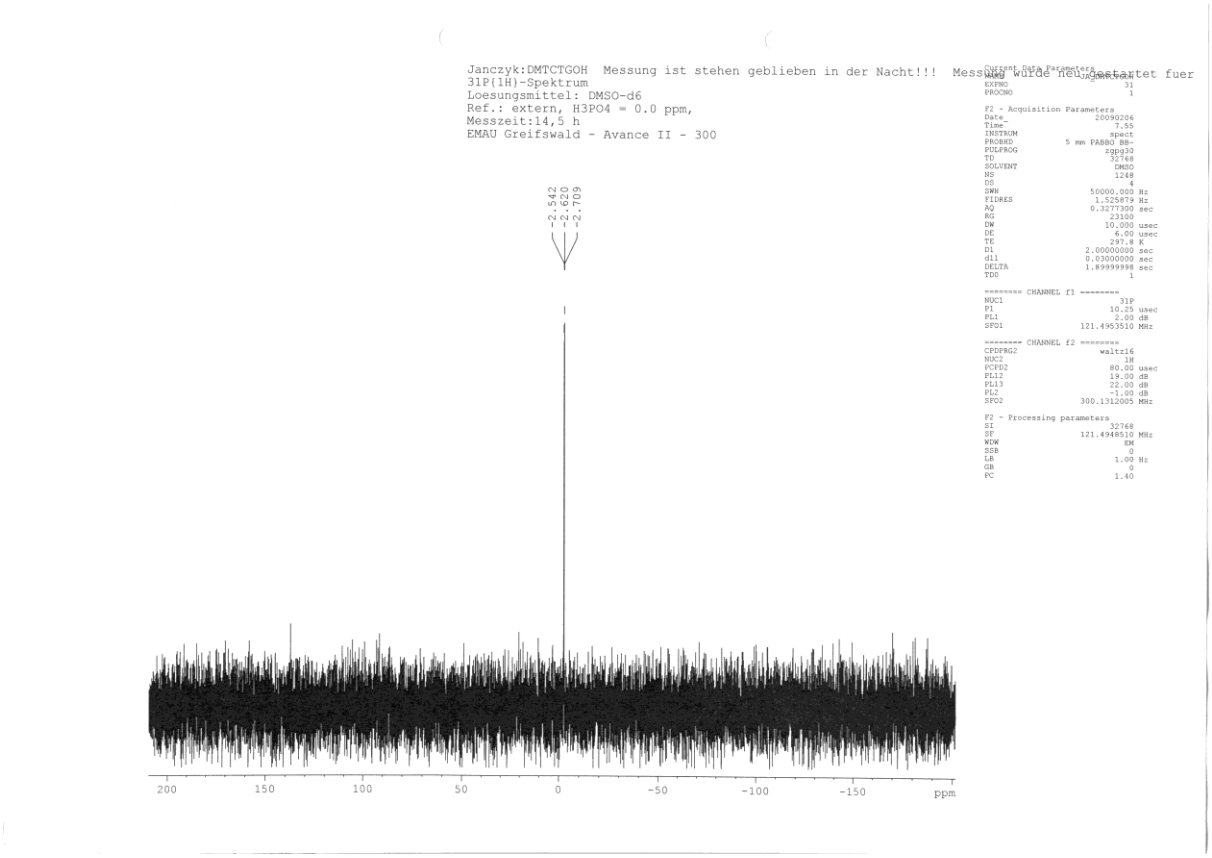
DMTCATOH



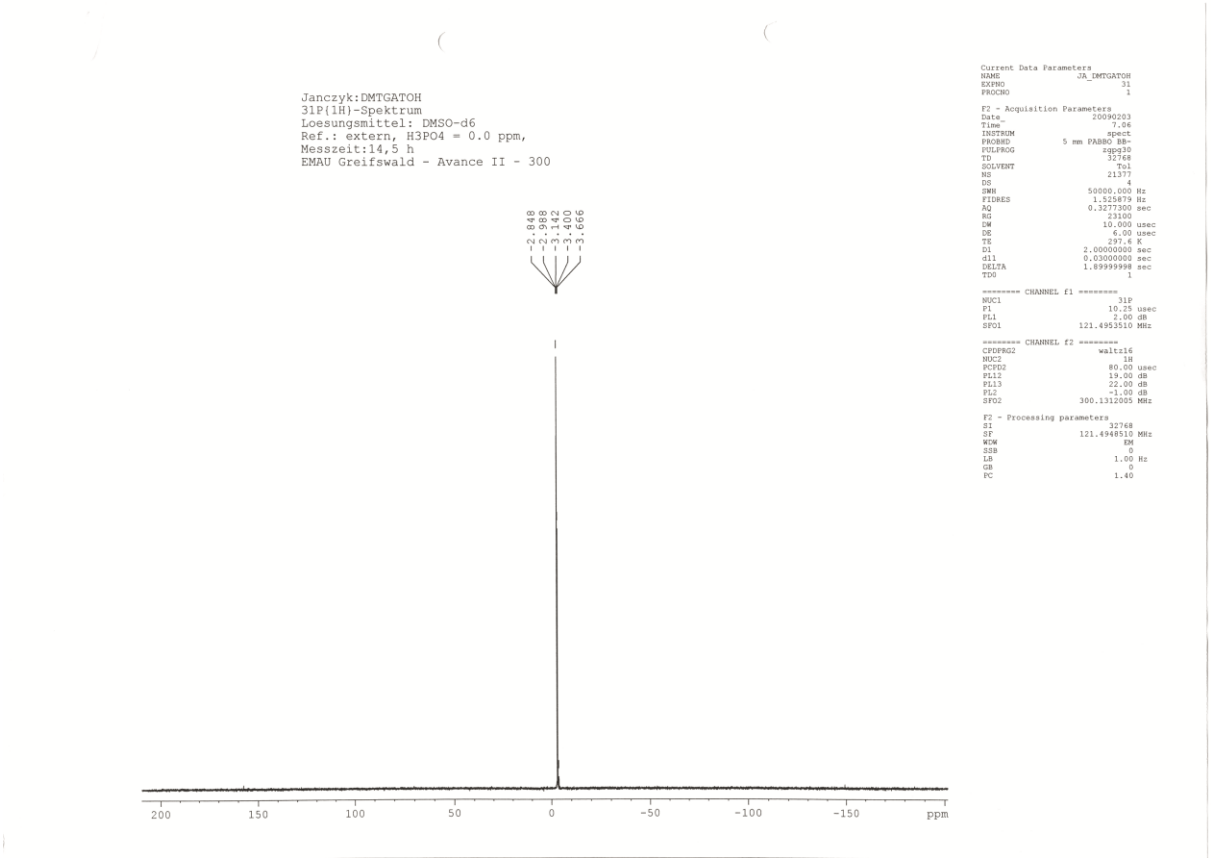
DMTCCAOH


Janczyk:DMTCCAOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:30 min.
EMAU Greifswald - Avance II - 300
2.603
2.682
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTCCAOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090114
Time 12.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 750
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

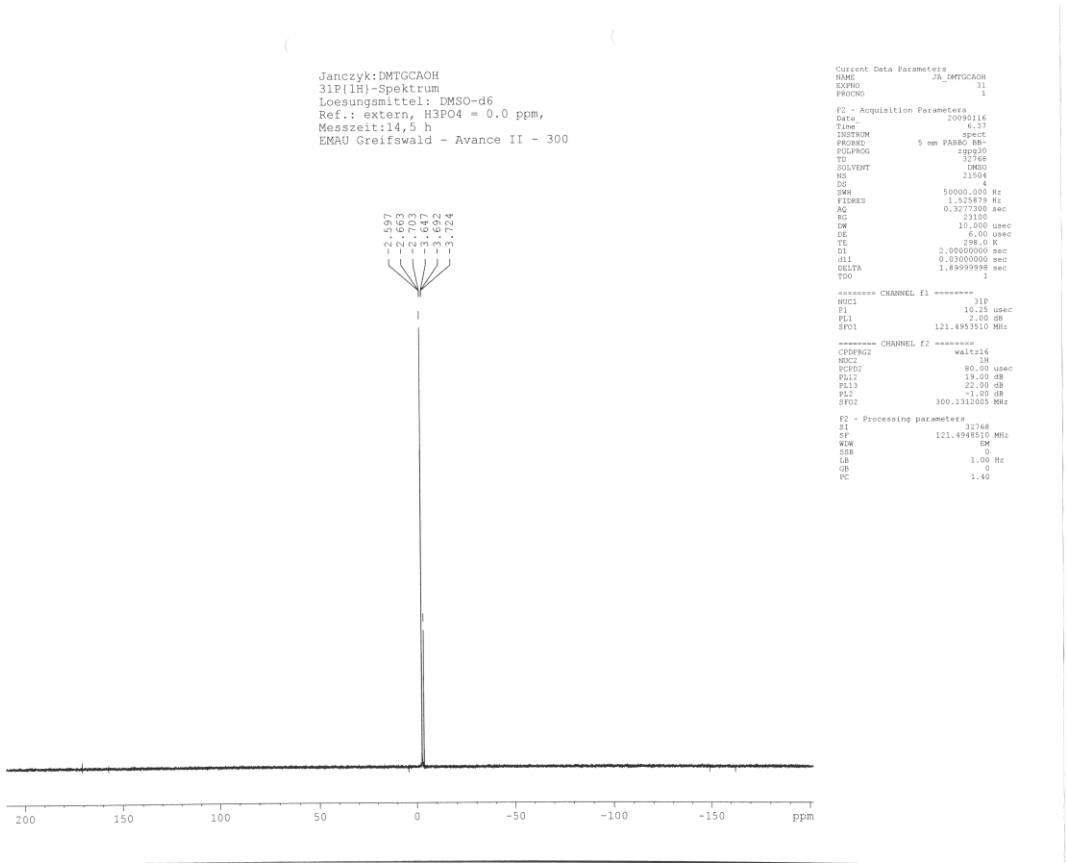
DMTCTGOH


Janczyk:DMTCTGOH Messung ist stehen geblieben in der Nacht!!! Messung wurde heute gestartet fuer
31P(1H)-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
-2.542
-2.620
-2.709
200
150
100
50
0
-50
-100
-150
ppm

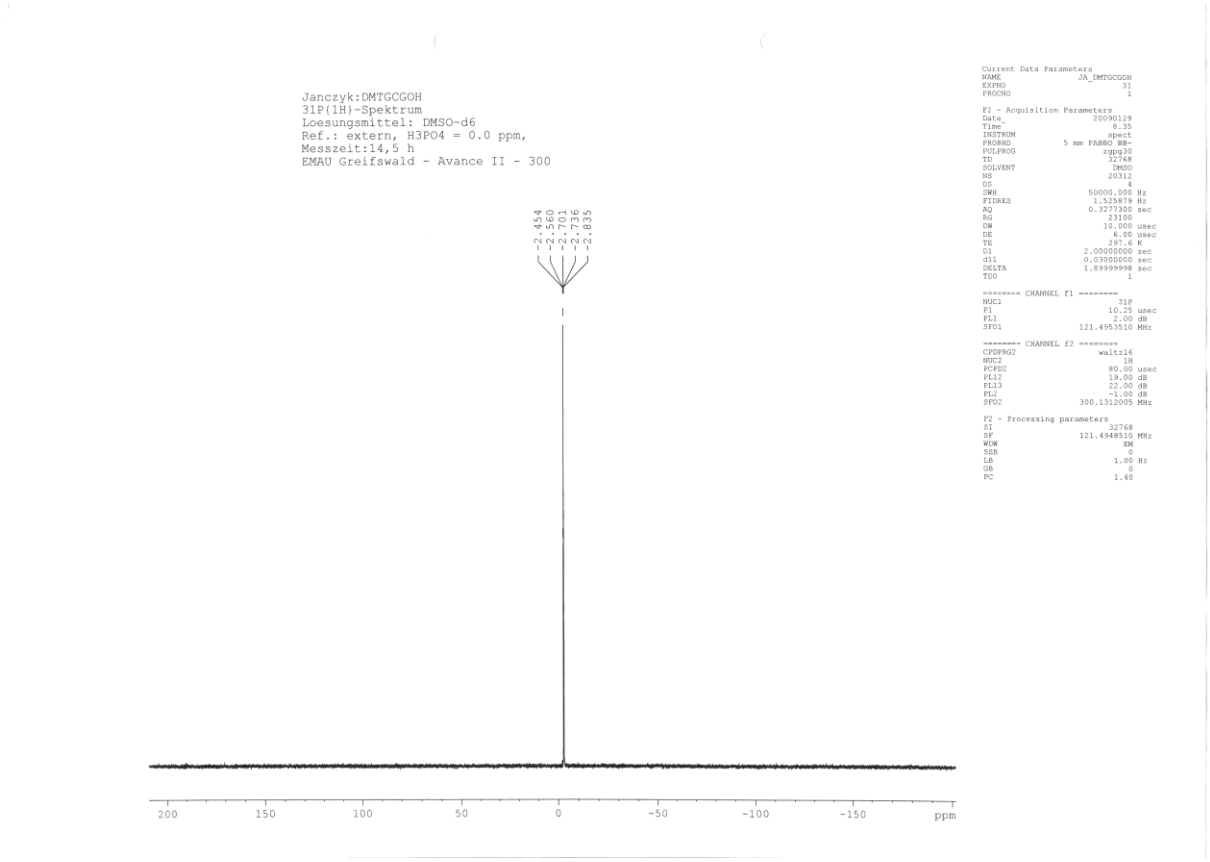
DMTGATOH


Janczyk:DMTGATOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
2.88
3.02
3.40
3.66
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGATOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090203
Time 7.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT Tol
NS 21377
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

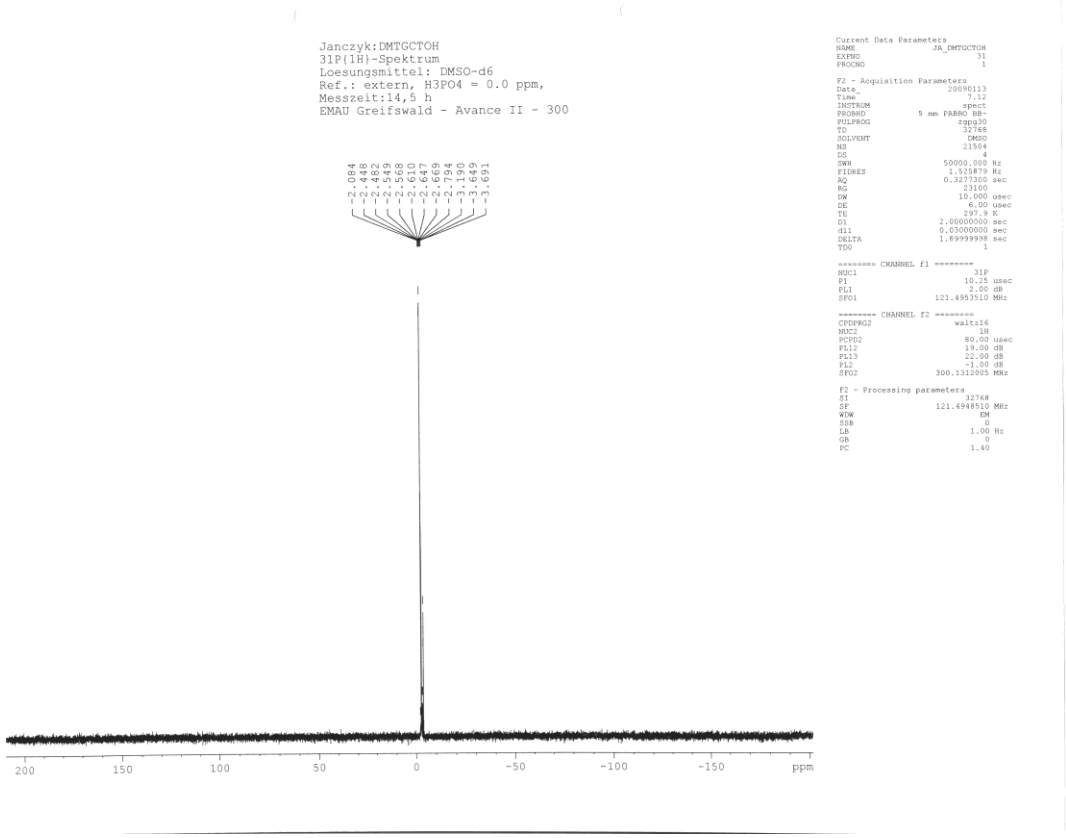
DMTGCAOH


Janczyk:DMTGCAOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
2.597
2.663
2.693
3.692
3.724
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGCAOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090116
Time 6.37
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 21504
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

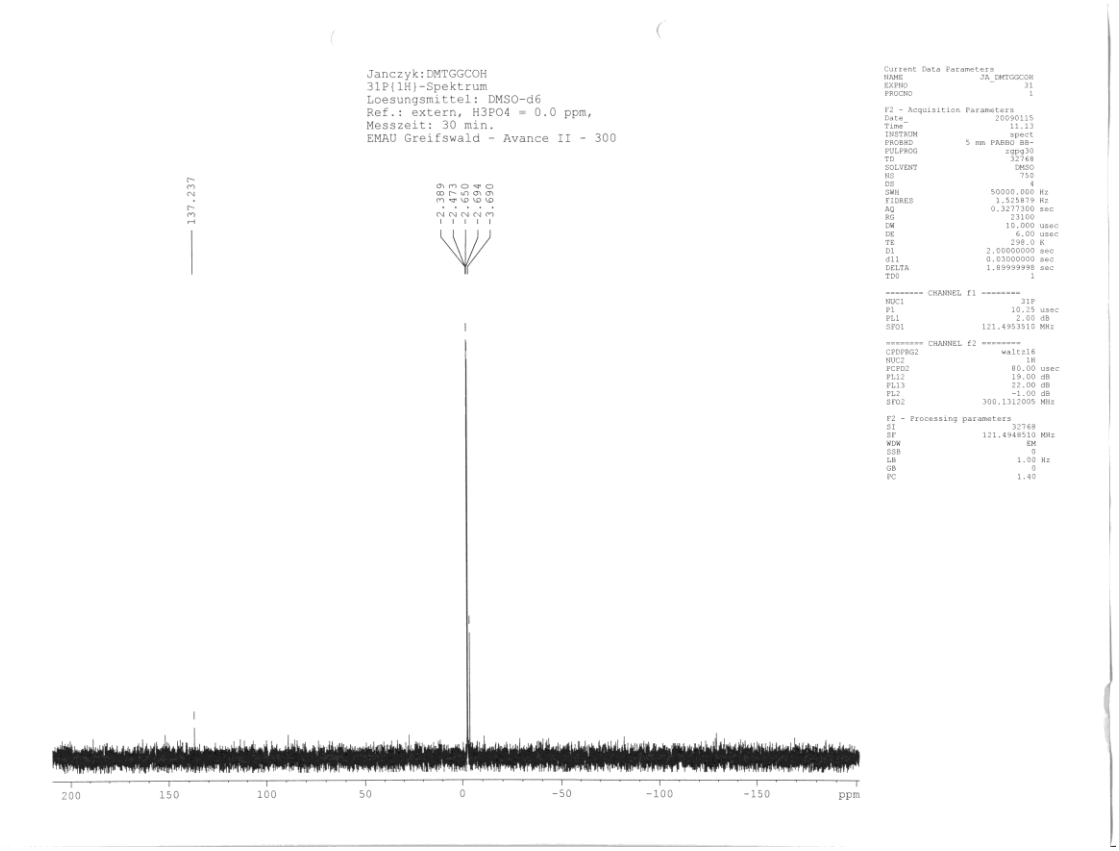
DMTGCGOH


Janczyk:DMTGCGOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
2.454
2.560
2.701
2.736
2.835
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGCGOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090129
Time_ 8.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 20312
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

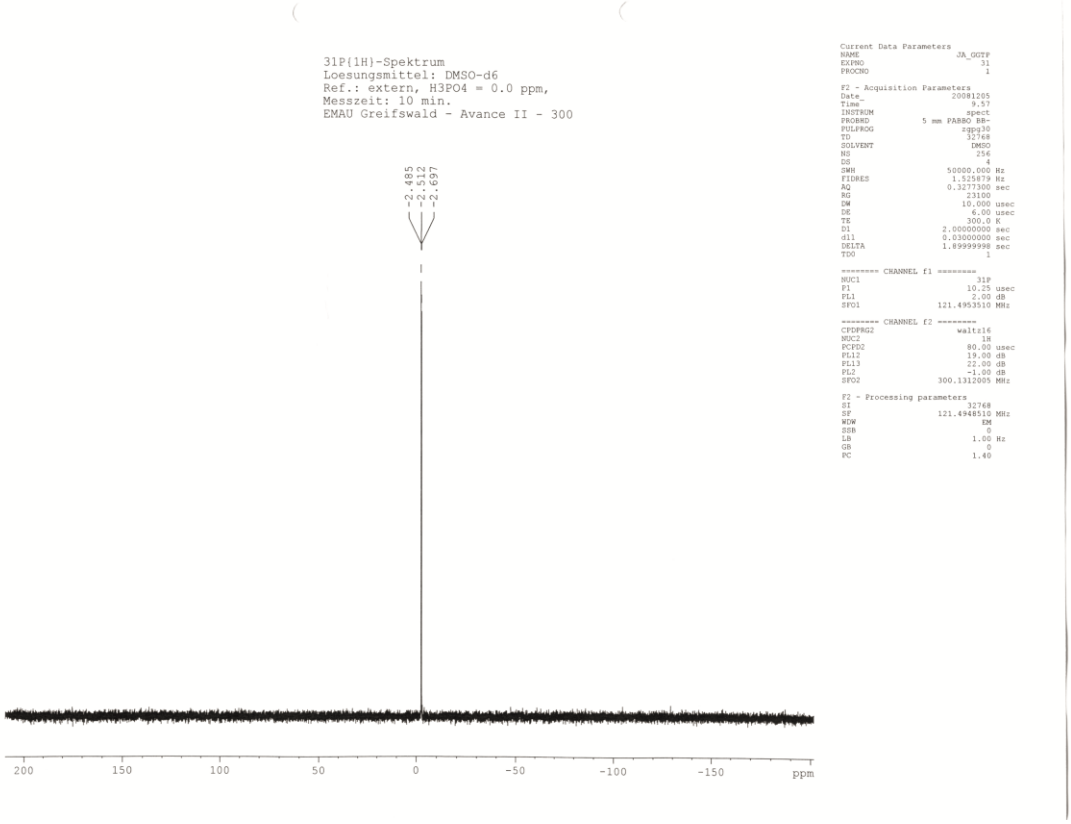
DMTGCTOH


Janczyk:DMTGCTOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
2.084
2.448
2.462
2.549
2.568
2.650
2.667
2.669
2.794
3.190
3.649
3.691
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGCTOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090113
Time 7.12
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 21504
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277360 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

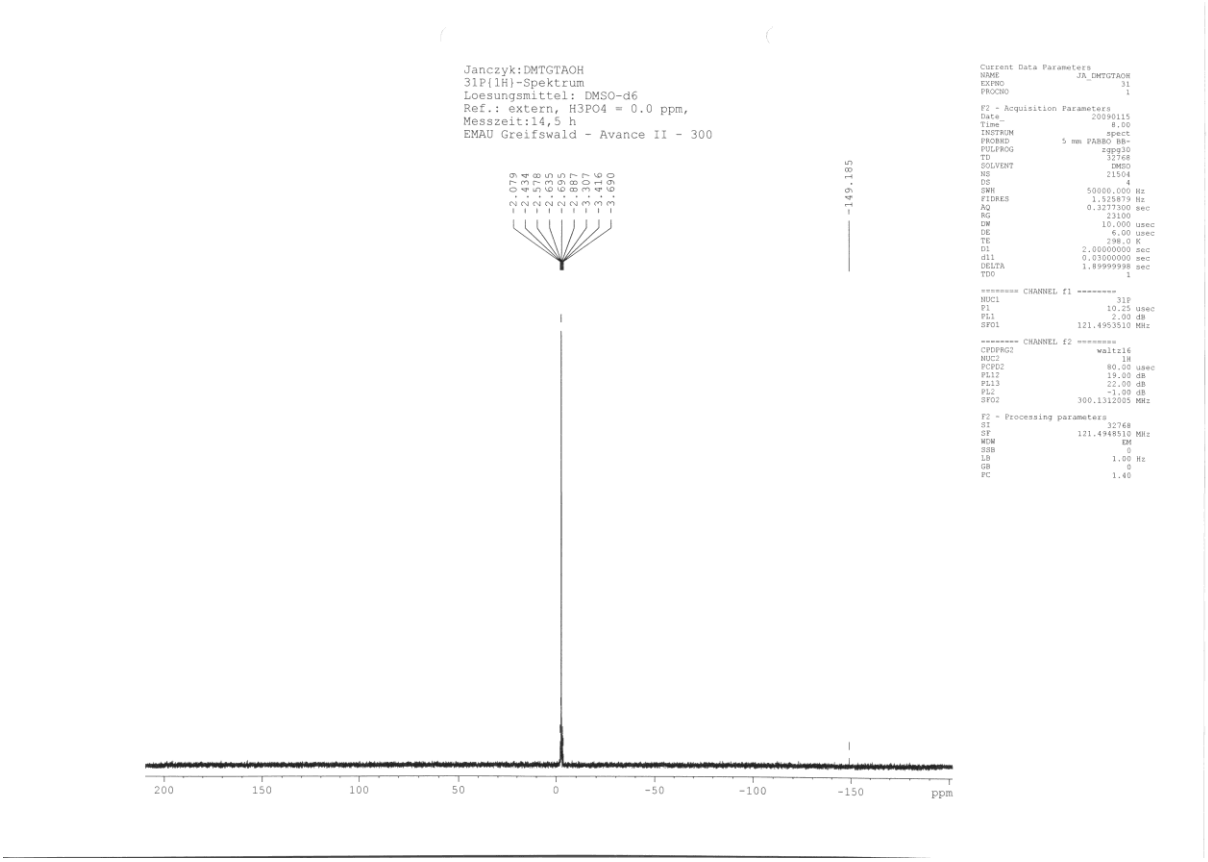
DMTGGCOH


Janczyk:DMTGGCOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 30 min.
EMAU Greifswald - Avance II - 300
137.237
2.389
2.473
2.650
2.694
3.690
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGGCOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 11.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 750
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

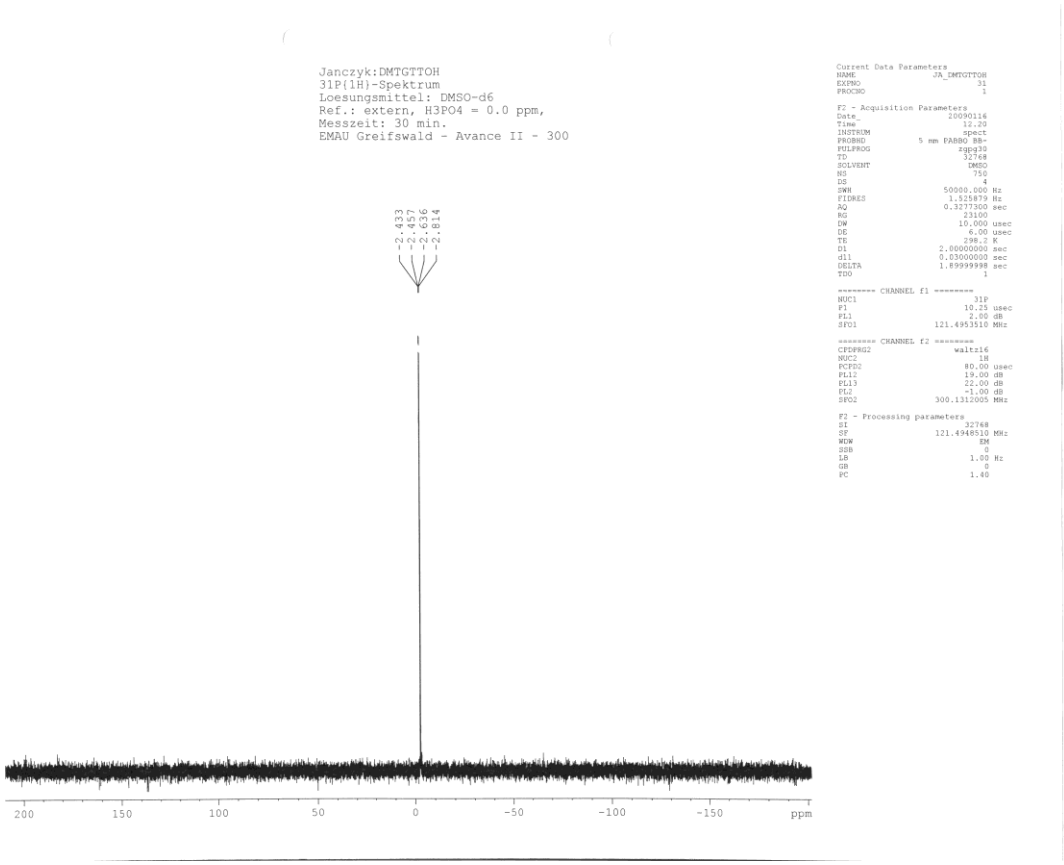
DMTGGTOH


31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 10 min.
EMAU Greifswald - Avance II - 300
-2.485
-2.512
-2.697
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_GGTF
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081205
Time 9.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 256
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

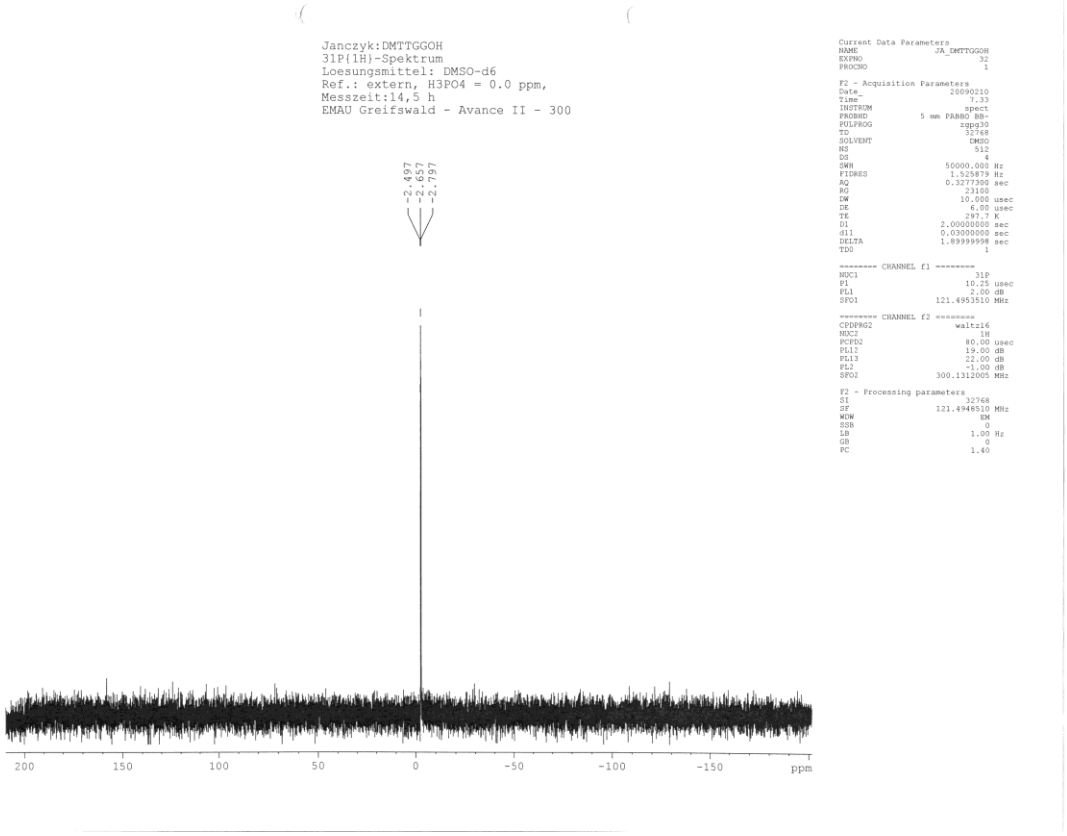
DMTGTAOH


Janczyk:DMTGTAOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
2.079
2.434
2.578
2.635
2.695
2.887
3.107
3.476
3.600
149.185
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGTAOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090115
Time 8.00
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 21504
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 298.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

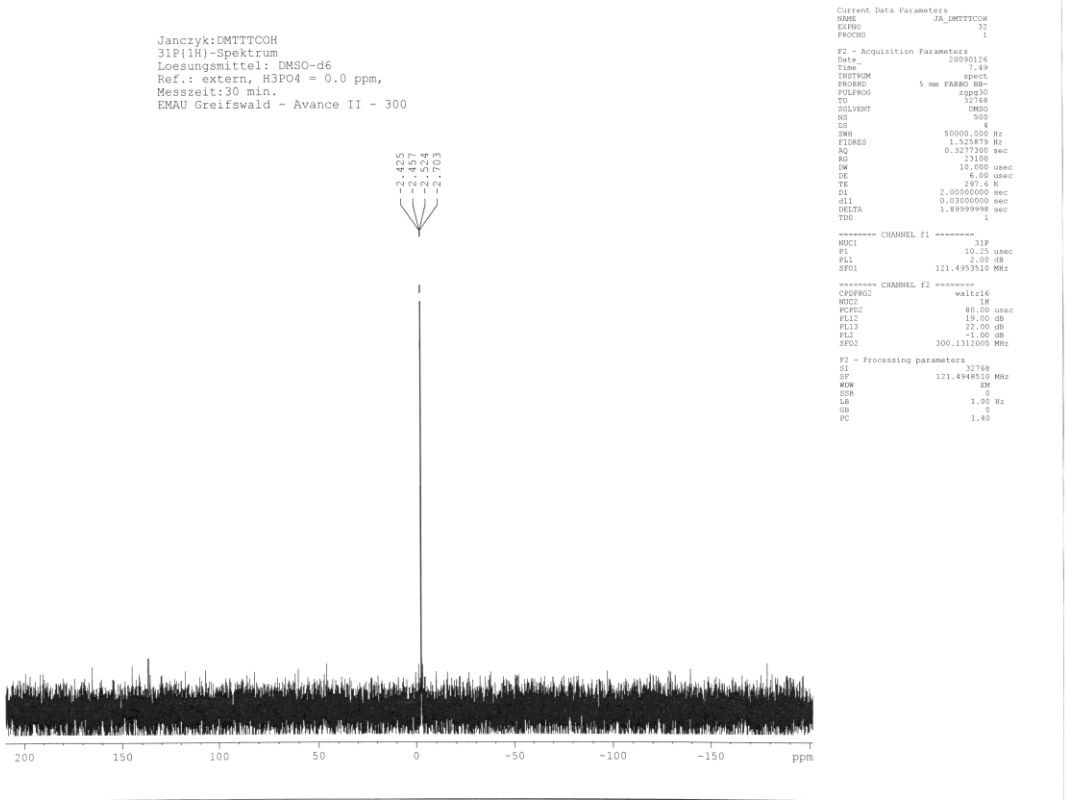
DMTGTTOH


Janczyk:DMTGTTOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit: 30 min.
EMAU Greifswald - Avance II - 300
3.33
3.37
3.36
3.34
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTGTTOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090116
Time 12.20
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 750
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

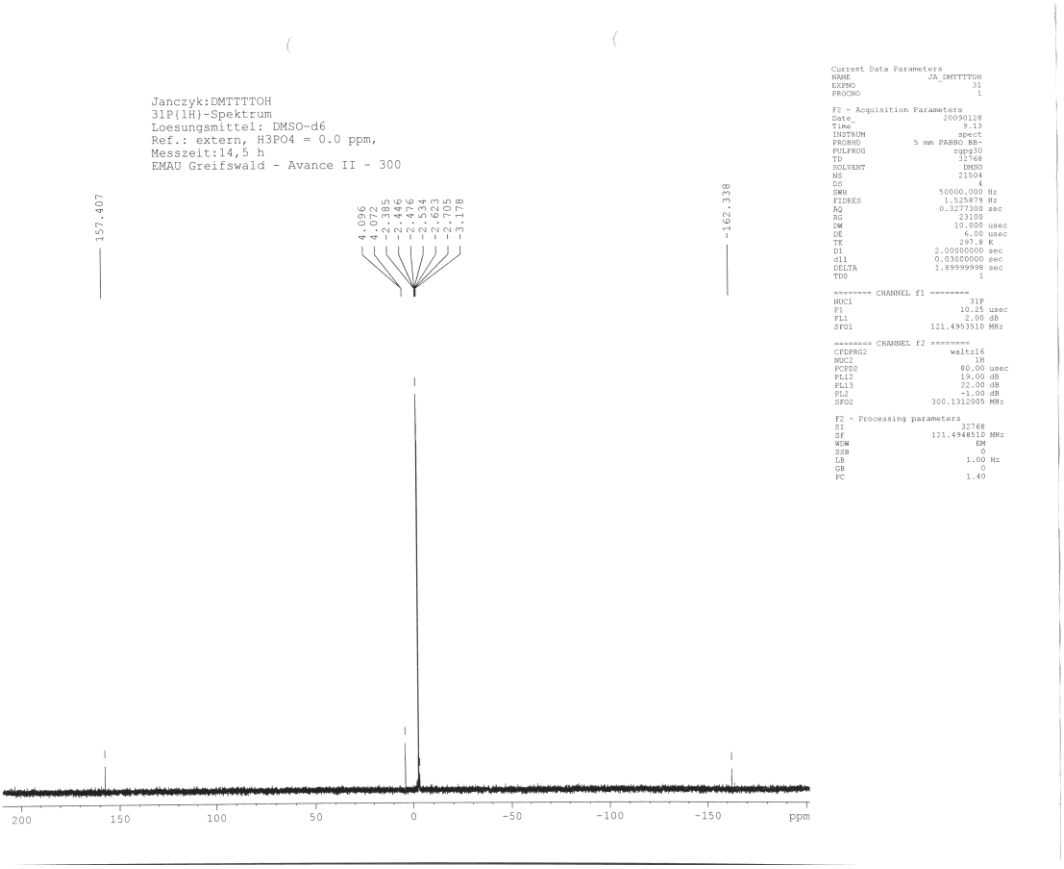
DMTTGGOH


Janczyk:DMTTGGOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
-2.497
-2.657
-2.797
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTTGGOH
EXPNO 32
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090210
Time 7.33
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 512
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DMTTTCOH


Janczyk:DMTTTCOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:30 min.
EMAU Greifswald - Avance II - 300
2.425
2.467
2.524
2.703
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTTTCOH
EXPNO 32
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090126
Time 7.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 500
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

DMTTTTOH


Janczyk:DMTTTTOH
31P{1H}-Spektrum
Loesungsmittel: DMSO-d6
Ref.: extern, H3PO4 = 0.0 ppm,
Messzeit:14,5 h
EMAU Greifswald - Avance II - 300
157.407
4.096
4.072
-2.385
-2.446
-2.476
-2.534
-2.563
-2.705
-3.178
-162.338
200
150
100
50
0
-50
-100
-150
ppm
Current Data Parameters
NAME JA_DMTTTTOH
EXPNO 31
PROCNO 1
F2 - Acquisition Parameters
Date 20090128
Time 9.13
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 32768
SOLVENT DMSO
NS 21504
DS 4
SWH 50000.000 Hz
FIDRES 1.525879 Hz
AQ 0.3277300 sec
RG 23100
DW 10.000 usec
DE 6.00 usec
TE 297.8 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 31P
P1 10.25 usec
PL1 2.00 dB
SFO1 121.4953510 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL12 19.00 dB
PL13 22.00 dB
PL2 -1.00 dB
SFO2 300.1312005 MHz
F2 - Processing parameters
SI 32768
SF 121.4948510 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40