DEUTERATED NMR SOLVENTS-HANDY REFERENCE DATA

Compound Mol. Wt.	d_4^{20}	m.p.*	b.p.*	δ_H (mult)*	J_{HD}	δ_C (mult)*	J_{CD} (J_{CF})
Acetic Acid-d_4 64.078	1.12	17	118	11.53 (1) 2.03 (5)	2	178.4 (br) 20.0 (7)	20
Acetone-d_6 64.117	0.87	-94	57	2.04 (5)	2.2	206.0 (13) 29.8 (7)	0.9 20
Acetonitrile-d_3 44.071	0.84	-45	82	1.93 (5)	2.5	118.2 (br) 1.3 (7)	21
Benzene-d_6 84.152	0.95	5	80	7.15 (br)		128.0 (3)	24
Chloroform-d 120.384	1.50	-64	62	7.24 (1)		77.0 (3)	32
Cyclohexane-d_{12} 96.236	0.89	6	81	1.38 (br)		26.4 (5)	19
Deuterium Oxide 20.028	1.11	3.8	101.4	4.63(DSS) 4.67(TSP)			
1,2-Dichloroethane-d_4 102.985	1.25	-40	84	3.72 (br)		43.6 (5)	23.5
Diethyl-d_{10} Ether 84.185	0.82	-116	35	3.34 (m) 1.07 (m)		65.3 (5) 14.5 (7)	21 19
Diglyme-d_{14} 148.263	0.95	-68	162	3.49 (br) 3.40 (br) 3.22 (5)	 1.5	70.7 (5) 70.0 (5) 57.7 (7)	21 21 21
Dimethylformamide-d_7 80.138	1.04	-61	153	8.01 (br) 2.91 (5) 2.74 (5)	 2 2	162.7 (3) 35.2 (7) 30.1 (7)	30 21 21
Dimethyl-d_6 Sulphoxide 84.170	1.18	18	189	2.49 (5)	1.7	39.5 (7)	21
p-Dioxane-d_8 96. 156	1.13	12	101	3.53 (m)		66.5 (5)	22
Ethyl Alcohol-d_6 (anh) 52.106	0.91	<-130	79	5.19 (1) 3.55 (br) 1.11 (m)		 56.8 (5) 17.2 (7)	 22 19
Glyme-d_{10} 100.184	0.86	-58	83	3.40 (m) 3.22 (5)	 1.6	71.7 (5) 57.8 (7)	21 21
Hexafluoroacetone Deuterate[c] 198.067	1.71	21		5.26 (1)		122.5 (4) 92.9 (7)	(287) (34.5)
HMPT-d_{18} 197.314	1.14	7	106 (11)	2.53(2 x 5)	2 (9.5)	35.8 (7)	21
Methyl Alcohol-d_4 36.067	0.89	-98	65	4.78 (1) 3.30 (5)	 1.7	49.0 (7)	21.5
Methylene Chloride-d_2 86.945	1.35	-95	40	5.32 (3)	1	53.8 (5)	27
Nitrobenzene-d_5 128.143	1.25	6	211	 8.11 (br) 7.67 (br) 7.50 (br)	 	148.6 (1) 134.8 (3) 129.5 (3) 123.5 (3)	 24.5 (p) 25 26
Nitromethane-d_3 64.059	1.20	-29	101	4.33 (5)	2	62.8 (7)	22
isoPropyl Alcohol-d_8 68.146	0.90	-86	83	5.12 (1) 3.89 (br) 1.10 (br)	 	 62.9 (3) 24.2 (7)	 21.5 19
Pyridine-d_5 84.133	1.05	-42	116	8.71 (br) 7.55 (br) 7.19 (br)	 	149.9 (3) 135.5 (3) 123.5 (3)	27.5 24.5 (γ) 25
Tetrahydrofuran-d_8 80.157	0.99	-109	66	3.58 (br) 1.73 (br)	 	67.4 (5) 25.3 (br)	22 20.5
Toluene-d_8 100.191	0.94	-95	111	 7.09 (m) 7.00 (br) 6.98 (m) 2.09 (5)	 2.3	137.5 (1) 128.9 (3) 128.0 (3) 125.2 (3) 20.4 (7)	 23 24 24 (p) 19
Trifluoroacetic Acid-d[d] 115.030	1.50	-15	72	11.50 (1)		164.2 (4) 116.6 (4)	(44) (283)
2,2,2-Trifluoroethyl Alcohol-d_3[e] 103.059	1.45	-44	75	5.02 (1) 3.88(4 x 3)	 2 (9)	126.3 (4) 61.5(4 x 5)	(277) 22 (36)

*Melting and boiling points (in °C) are those of the corresponding light compound (except for D_2O) and are intended only to indicate the useful liquid range of the materials.

[b]^1H (of the residual protons) and ^{13}C spectra were determined on HA-100 and XL-100-15 spectrometers, respectively, for the same sample of each solvent containing 5% TMS (v/v). The chemical shifts are in ppm relative to TMS; the coupling constants are in Hz. (Since deuterium has a spin of 1, triplets arising from coupling to deuterium have the intensity ratio of 1:1:1, etc.) The multiplicity br indicates a broad peak without resolvable fine structure, while m denotes one with fine structure. It should be noted that the chemical shifts, in particular, can be dependent on solute, concentration and temperature.

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ISOTOPICS

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SUGGESTIONS FOR USE & HANDLING OF NMR SOLVENTS

In Fourier Transform NMR studies, especially when using microgram quantities, peaks arising from water and hydrocarbons leached from cap liners may present serious problems. These extraneous peaks may be avoided by the use of solvents sealed in glass ampoules.

Our NMR solvents are carefully dried prior to packaging. When opened, most solvents will absorb atmospheric moisture. To minimize the water-absorption problem we recommend the use of our sealed glass ampoules, available in 1 gram or 0.5 ml sizes. They are especially convenient for glove-box use. For a number of solvents there is no extra charge for 10 gram quantities - i.e., the price for 10 x 1 gram is the same as for 1 x 10 grams.

We find that solvents can be rapidly dried with Linde 3A or 4A sieves, 1/16"; bead-shaped sieves do not perform satisfactorily, even when freshly conditioned.

To avoid the introduction of water by desorption from the wall of the NMR tube (especially important when overnight runs are required), the tubes can be preconditioned with D_2O. This does not remove the water, but substitutes D_2O for the signal-producing H_2O which, for example, gives rise to a peak at ca. δ 1.5 in $CDCl_3$. An easy method is to keep one ml or more of D_2O in the NMR tube overnight, decant, wash with a small amount of CD_3OD or $(CD_3)_2CO$ to remove the residual D_2O, dry briefly with a stream of dry nitrogen and store in a desiccator saturated with D_2O. Using this technique, a freshly-dried (Linde 3A sieve, 1/16") $CDCl_3$ sample analysed in a D_2O conditioned tube leaves the water peak sufficiently small for most purposes.

Our laboratories have tested the use of septum vials for preserving "100%" D_2O and have found that with a little additional care, decrease in isotopic enrichment can be avoided even with the repeated use of a syringe to withdraw material. We suggest that first the syringe should be flushed with dry nitrogen; then a quantity of dry nitrogen equivalent to the amount of D_2O required should be injected into the septum vial before the material is withdrawn. In our stability tests, we have observed no decrease in isotopic enrichment.

If the cap is kept properly tightened, TMS will not readily evaporate at room temperature. If kept refrigerated, ensure that the TMS bottle is at room temperature before it is opened in order to avoid condensation of atmospheric water in the cold bottle. If two layers are present in the TMS bottle, the lower one will be the water.

Proton Chemical Shifts of Common Impurities

Impurity	Solvent				
	$CDCl_3$	$(CD_3)_2SO$	C_5D_5N	C_6D_6	D_2O
Acetic Acid	2.13	1.95	2.13	1.63	2.16
Acetone	2.17	2.12	2.00	1.62	2.22
Acetonitrile	1.98	2.09	1.85	0.67	2.05
Amisyl Alcohol	4.61 3.79	4.44 3.74	4.92 3.68	4.36 3.32	4.57 3.81
Benzene	7.37	7.40	7.33	7.30	7.44
Bromoform	6.85	7.75	7.86	5.89	*
n-Butanol	3.67(t) 0.94(t)	3.41(t) 0.89(t)	3.80(t) 0.89(t)	3.38(t) 0.83(t)	3.60 0.89(t)
t-Butanol	1.28	1.14	1.37	1.06	1.23
Chloroacetic Acid	4.14	4.28	4.44	3.31	4.25
Chloroform	7.27	8.35	8.41	6.41	*
Cyclohexane	1.43	1.42	1.38	1.40	*
1,2 Dibromoethane	3.63	3.84	3.70	2.88	3.79
Diochloroacetic Acid	5.98	6.68	6.72	5.43	6.21
1,2 Dichloroethane	3.73	3.93	3.78	2.99	3.92
Dichloromethane	5.30	5.79	5.62	4.46	*
Diethyleneglycol dimethyl ether	3.60(m) 3.38	3.49(m) 3.28	3.57(m) 3.27	3.40(m) 3.13	3.63(m) 3.37

Shifts are PPM (δ) with respect to TMS.
* Indicates that the impurity is insoluble in the solvent.
+ Indicates that the impurity and solvent react.

Impurity	$CDCl_3$	$(CD_3)_2SO$	C_5D_5N	C_6D_6	D_2O
Diethyl ether	3.48(q) 1.2(t)	3.42(q) 1.13(t)	3.38(q) 1.12(t)	3.27(q) 1.10(t)	3.56(q) 1.17(t)
Di-isopropyl ether	1.12(d)	1.04(d)	1.09	0.50(d)	1.12(d)
Dimethlyacetamide	2.08 3.01 2.94	2.99 2.82 1.99	2.82 2.70 1.96	2.59 2.11 1.64	3.05 2.89 2.08
Dimethylformamide	8.01 2.95 2.88	7.98 2.92 2.76	2.72 2.66	2.40 1.98	7.91 3.00 2.86
Dimethyl Sulfoxide	2.62	2.52	2.49	1.91	2.70
Dioxan	3.70	3.61	3.61	3.38	3.75
Ethanediol	3.76	3.42	4.01	4.21	3.66
Ethanol	3.72(q) 1.24(t)	3.49(q) 1.09(t)	3.86(q) 1.29(t)	3.39(q) 0.97(t)	3.64(q) 1.16(t)
Ethyl Acetate	4.12(q) 2.04 1.25(t)	4.08(q) 2.02 1.21(t)	4.06(q) 1.94 1.10(t)	3.91(q) 1.68 0.94(t)	4.14(q) 2.08 1.23(t)
Ethyl Formate	8.04 4.22(q) 1.29(t)	8.23 4.17(q) 1.24(t)	8.22 4.14(q) 1.10(t)	7.60 3.83(q) 0.85(t)	8.16 4.28(q) 1.29(t)
Formic Acid	8.02	8.18	8.54	7.24	8.22
Furfuraldehyde	9.68 7.71(m) 7.25(m) 6.60(m)	9.69 8.14(m) 7.59(m) 6.82(m)	9.78 7.87(m) 7.31(m) 6.56(m)	9.36 6.89(m) 6.57(m) 5.82(m)	9.52 7.94(m) 7.59(m) 6.78(m)
Isobutyl methyl ketone	2.12 0.92(d)	2.08 0.88(d)	2.00 0.83(d)	1.61 0.74(d)	2.19 0.89(d)

Shifts are PPM (δ) with respect to TMS.
* Indicates that the impurity is insoluble in the solvent.
+ Indicates that the impurity and solvent react.

Impurity	$CDCl_3$	$(CD_3)_2SO$	C_5D_5N	C_6D_6	D_2O
Isopropyl Acetate	2.02 1.22(d)	2.00 1.21(d)	1.95 1.13(d)	1.69 1.02(d)	*
Isopropyl Alcohol	4.03(m) 1.20(d)	1.06(d)	4.16(m) 1.29(d)	3.76(m) 1.01(d)	1.18(d)
Methanol	3.48	3.20	3.57	3.09	3.35
Methyl Acetate	3.67 2.05	3.61 2.02	3.55 1.92	3.28 1.63	3.68 2.09
Methyl Iodide	2.16	2.21	+	1.47	*
Morphine	3.69(d) 2.85(d)	3.52(d) 2.68(d)	2.67(d) 2.81(d)	3.50(d) 2.49(d)	3.70(d) 2.79(d)
Nitromethane	4.32	4.44	4.39	3.09	4.41
Petroleum spirit	1.28 0.90(t)	1.28 0.89(t)	1.20 0.86(t)	1.22 0.89(t)	*
Potassium Acetate	*	1.60	2.11	*	1.91
Propane-1,2 diol	1.17(d)	1.04(d)	1.36(d)	1.10(d)	1.13(d)
Propanol	3.60(t) 1.60(m) 0.93(t)	1.45(m) 0.87(t)	3.75(t) 1.70(m) 0.97(t)	3.76(t) 1.40(m) 0.80(t)	3.61(t) 1.57(m) 0.89(t)
Proprionic Acid	2.42(q) 1.18(t)	2.26(q) 1.03(t)	2.46(q) 1.20(t)	2.02(q) 0.89(t)	2.47(q) 1.10(t)
Pyridine	8.60(m) 7.69(m) 7.28(m)	8.61(m) 7.83(m) 7.40(m)	8.71(m) 7.58(m) 7.21(m)	8.50(m) 7.05(m) 6.70(m)	8.50(m) 7.90(m) 7.47(m)
Succinimide	2.75	2.63	2.64	1.50	2.78
1,1,2,2 Tetrachloroethane	5.96	6.92	6.90	4.96	*

Shifts are PPM (δ) with respect to TMS.
* Indicates that the impurity is insoluble in the solvent.
+ Indicates that the impurity and solvent react.

Impurity	$CDCl_3$	$(CD_3)_2SO$	C_5D_5N	C_6D_6	D_2O
THF	3.74(m) 1.85(m)	3.63(m) 1.78(m)	3.67(m) 1.64(m)	3.01(m) 0.87(m)	3.75(m) 1.88(m)
Toluene	7.19 2.34	7.22 2.32	7.22 2.22	7.10 2.13	*
1,1,1 Trichloroethane	1.72	2.80	2.61	1.58	*
Triethylamine	2.56(q) 1.03(t)	2.47(q) 0.99(t)	2.43(q) 0.96(t)	2.40(q) 0.95(t)	2.59(q) 1.02(t)
Trimethyl borate	3.48	3.46	3.58	3.13	+
Trimethyl Phosphate	3.80(d)	3.72(d)	3.71(d)	3.37(d)	3.82(d)

Shifts are PPM (δ) with respect to TMS.
* Indicates that the impurity is insoluble in the solvent.
+ Indicates that the impurity and solvent react.

ACETONE CIL LOT # F - 5153A

Acetone-d_6
99.9 atom % D
DLM-9

```
CAMB.026
DATE 24-2-88

SF    300.135
SFO   300.130
SY 100.0
O1   6600.000
SI  16384
TD  16384
SW   4000.000
HZ/PT    .488

PW      9.0
RD      8.000
AQ      2.048
RG     10
NS     16

DR     12
FW   5000
DP  12H PO

LB      0.0
HZ/CM 150.059
PPM/CM   .500
SR   4929.13
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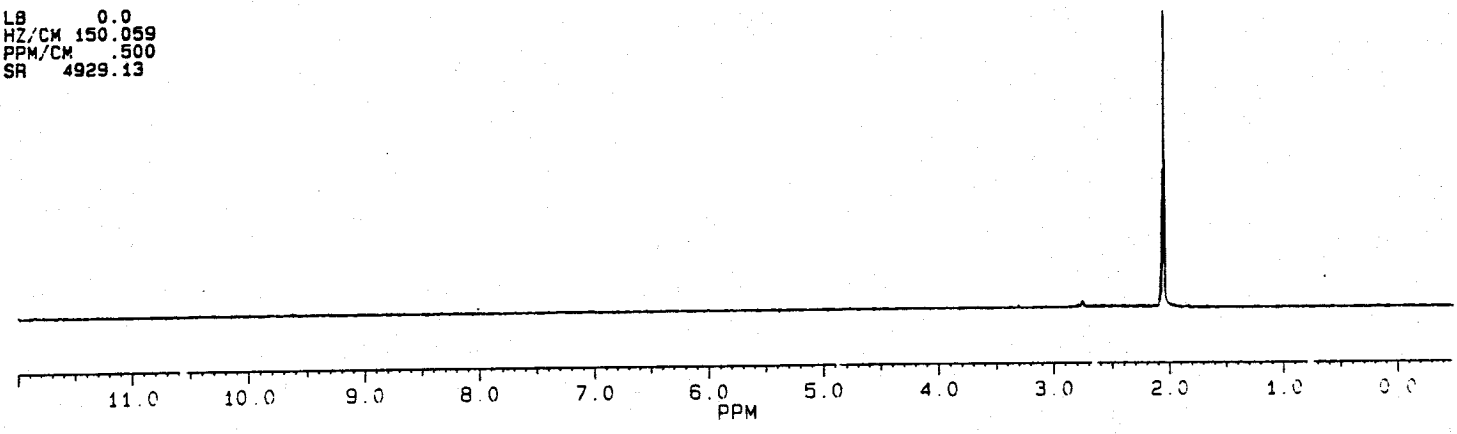

ACETONE CIL LOT # F - 49398

Acetone-d_6 "100%"
99.96 atom % D
DLM-38

```
CAMB.029
DATE 24-2-88

SF    300.135
SFO   300.130
SY 100.0
O1   6600.000
SI  16384
TD  16384
SW   4000.000
HZ/PT    .488

PW      9.0
RD      8.000
AQ      2.048
RG     20
NS     16

DR     12
FW   5000
DP  12H PO

LB      0.0
HZ/CM 150.059
PPM/CM   .500
SR   4929.13
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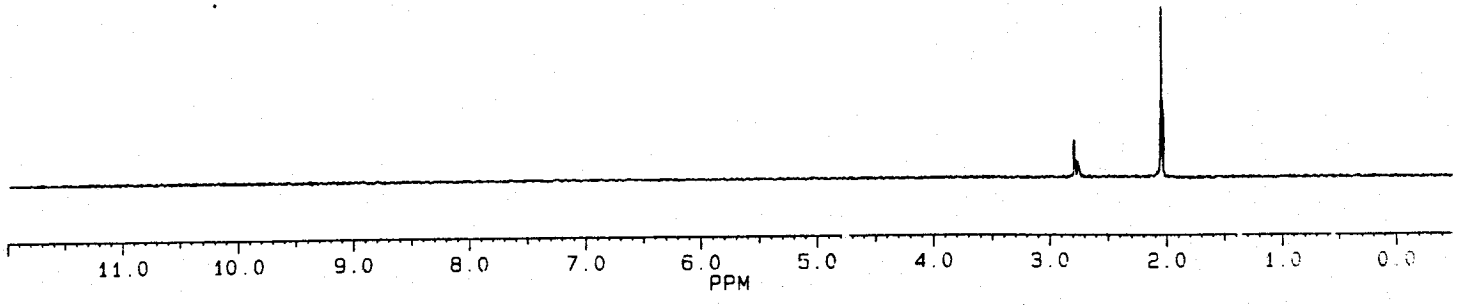


Matching the quality of the best competition is CIL's minimum level of acceptable performance!

ACETONITRILE CIL LOT # F - 5131A

BRUKER

CAMB.073
DATE 29-2-88

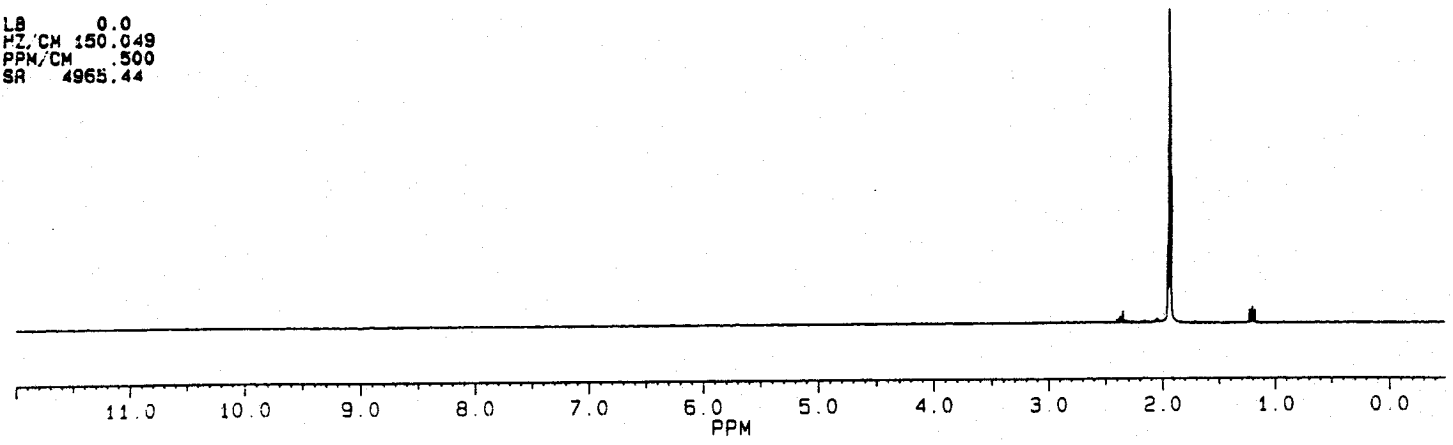
SF 300.135
SFO 300.130
SY 100.0
O1 6800.000
SI 16384
TD 16384
SW 5000.000
HZ/PT .610

PW 9.0
RD 8.000
AQ 1.638
RG 4
NS 16

DR 12
FW 6300
DP 12H PO

LB 0.0
HZ/CM 150.049
PPM/CM .500
SR 4965.44

Acetonitrile-d_3
99.8 atom % D
DLM-21



BENZENE CIL LOT # F - 5023C

BRUKER

CAMB.062
DATE 25-2-88

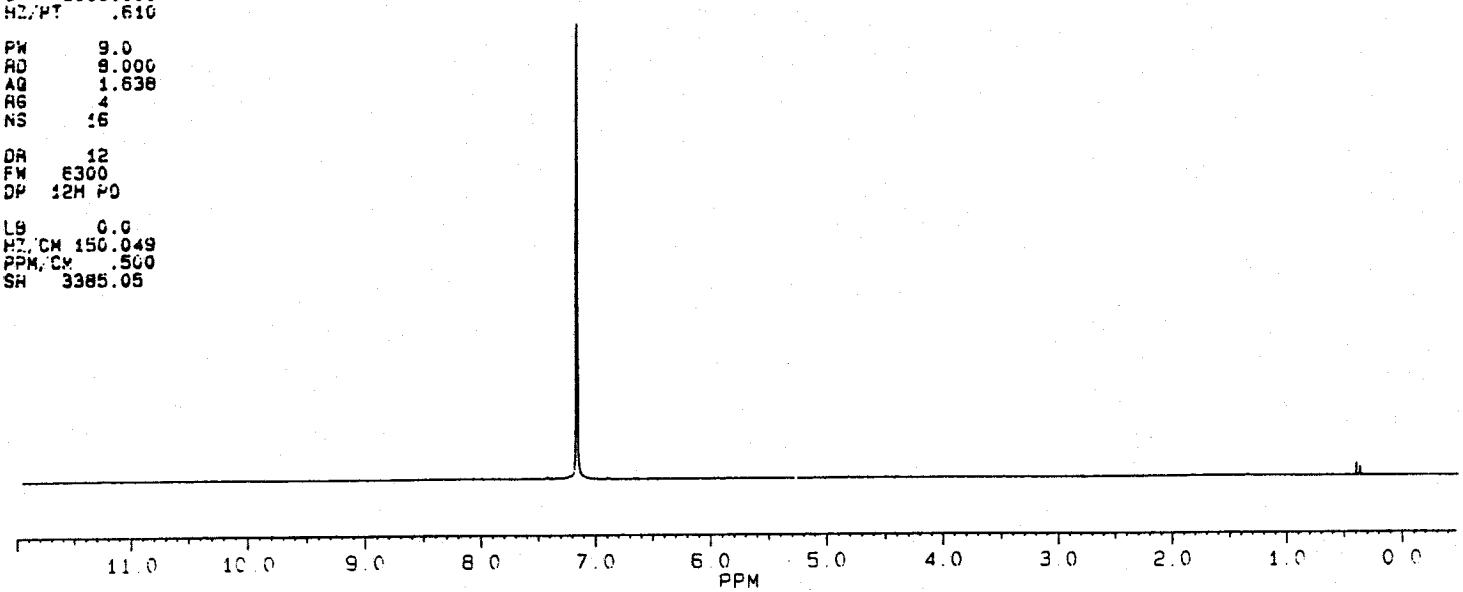
SF 300.133
SFO 300.130
SY 100.0
O1 5000.000
SI 16384
TD 16384
SW 5000.000
HZ/PT .610

PW 9.0
RD 8.000
AQ 1.638
RG 4
NS 16

DR 12
FW 6300
DP 12H PO

LB 0.0
HZ/CM 150.049
PPM/CM .500
SR 3385.05

Benzene-d_6
99.6 atom % D
DLM-1



CDCL3 CIL LOT # F - 56308

BRUKER

CAMB.005
DATE 24-2-88

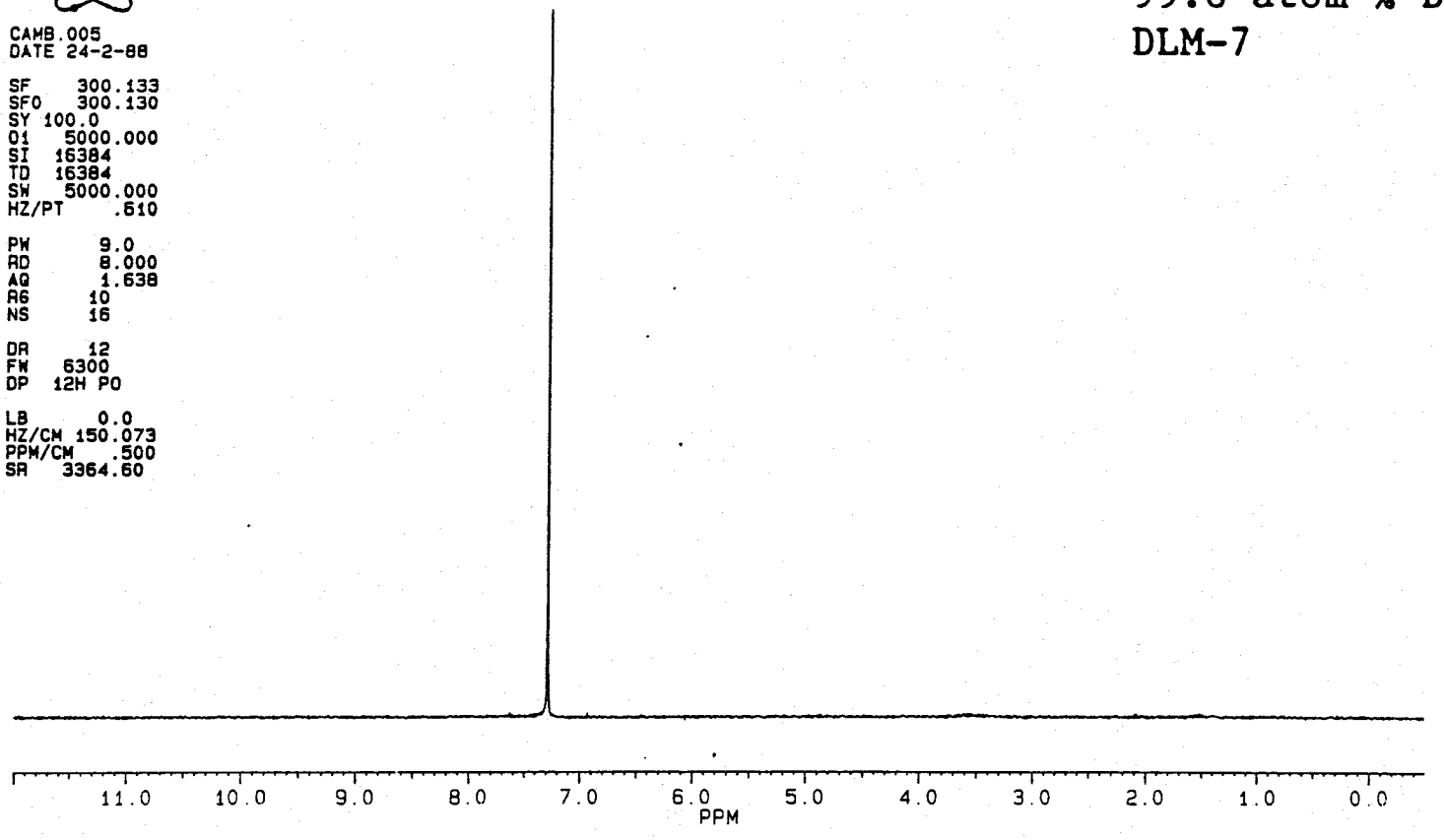
SF 300.133
SFO 300.130
SY 100.0
O1 5000.000
SI 16384
TD 16384
SW 5000.000
HZ/PT .610

PW 9.0
RD 8.000
AQ 1.638
RG 10
NS 16

DR 12
FW 6300
DP 12H PO

LB 0.0
HZ/CM 150.073
PPM/CM .500
SR 3364.60

Chloroform-d
99.8 atom % D
DLM-7



CDCL3 CIL LOT # F - 52388

BRUKER

CAMB.002
DATE 24-2-88

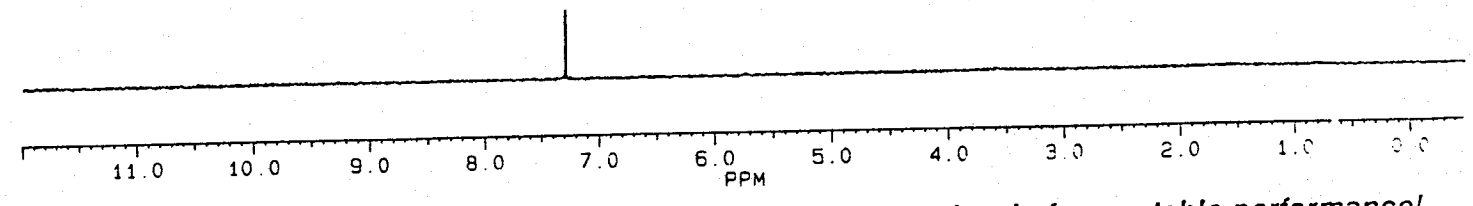
SF 300.133
SFO 300.130
SY 100.0
O1 5000.000
SI 16384
TD 16384
SW 5000.000
HZ/PT .610

PW 9.0
RD 8.000
AQ 1.638
RG 10
NS 16

DR 12
FW 6300
DP 12H PO

LB 0.0
HZ/CM 150.073
PPM/CM .500
SR 3364.60

Chloroform-d "100%"
99.96 atom % D
DLM-29



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DMSO CIL LOT # F - 5726B

BRUKER

CAMB.020
DATE 24-2-88

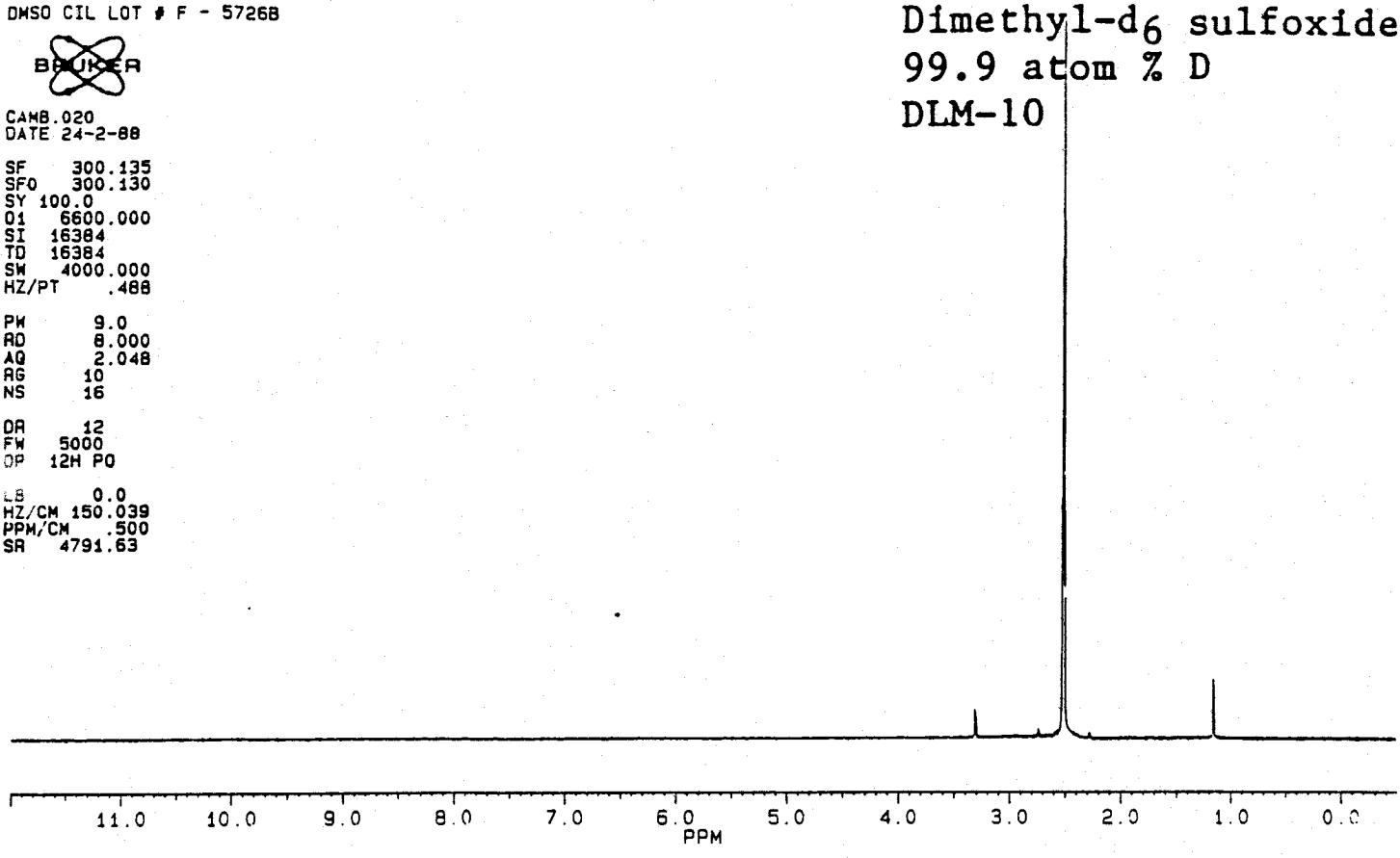
SF 300.135
SFO 300.130
SY 100.0
O1 6600.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 9.0
RD 8.000
AQ 2.048
RG 10
NS 16

DA 12
FW 5000
DP 12H PO

LB 0.0
HZ/CM 150.039
PPM/CM .500
SR 4791.63

Dimethyl-d_6 sulfoxide
99.9 atom % D
DLM-10



Matching the quality of the best competition is CIL's minimum level of acceptable performance!

DMSO CIL LOT # F - 5372C

BRUKER

CAMB.016
DATE 24-2-88

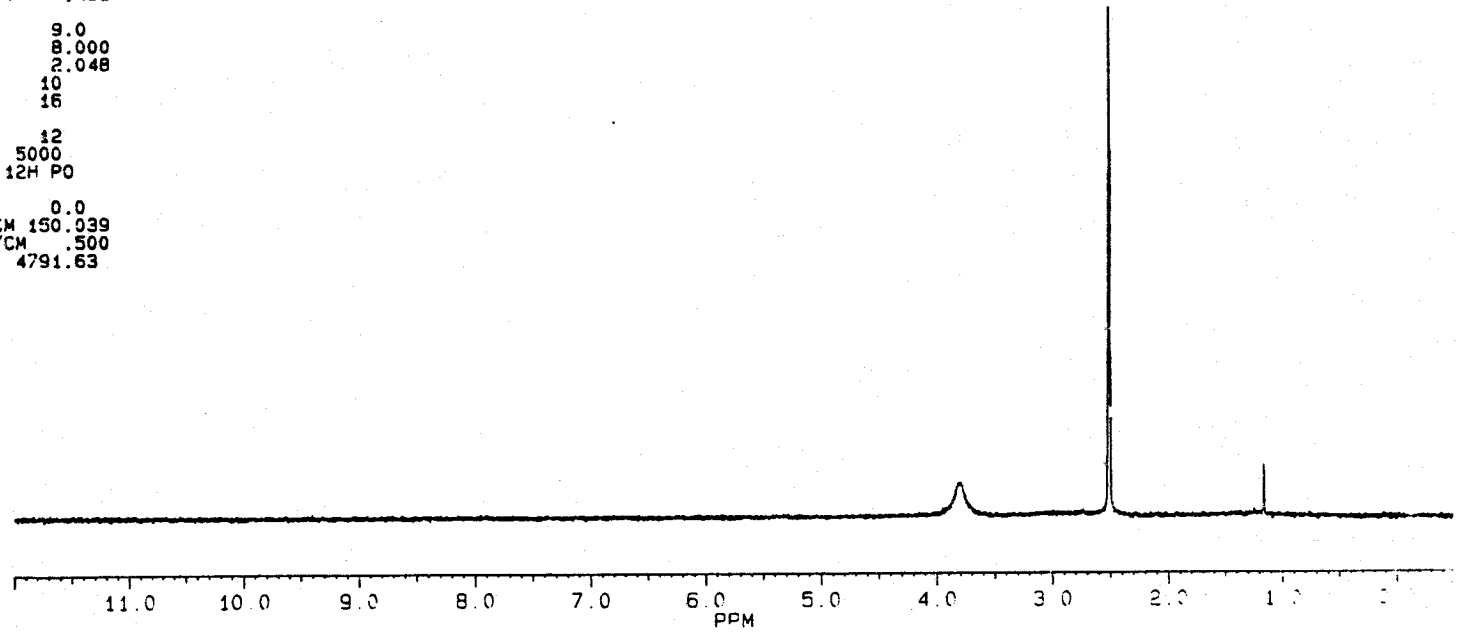
SF 300.135
SFO 300.130
SY 100.0
O1 6600.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 9.0
RD 8.000
AQ 2.048
RG 10
NS 16

DA 12
FW 5000
DP 12H PO

LB 0.0
HZ/CM 150.039
PPM/CM .500
SR 4791.63

Dimethyl-d_6 sulfoxide "100%"
99.96 atom % D
DLM-34



D20 CIL LOT # F - 5624C

Deuterium oxide
99.9 atom % D
DLM-4

BRUKER

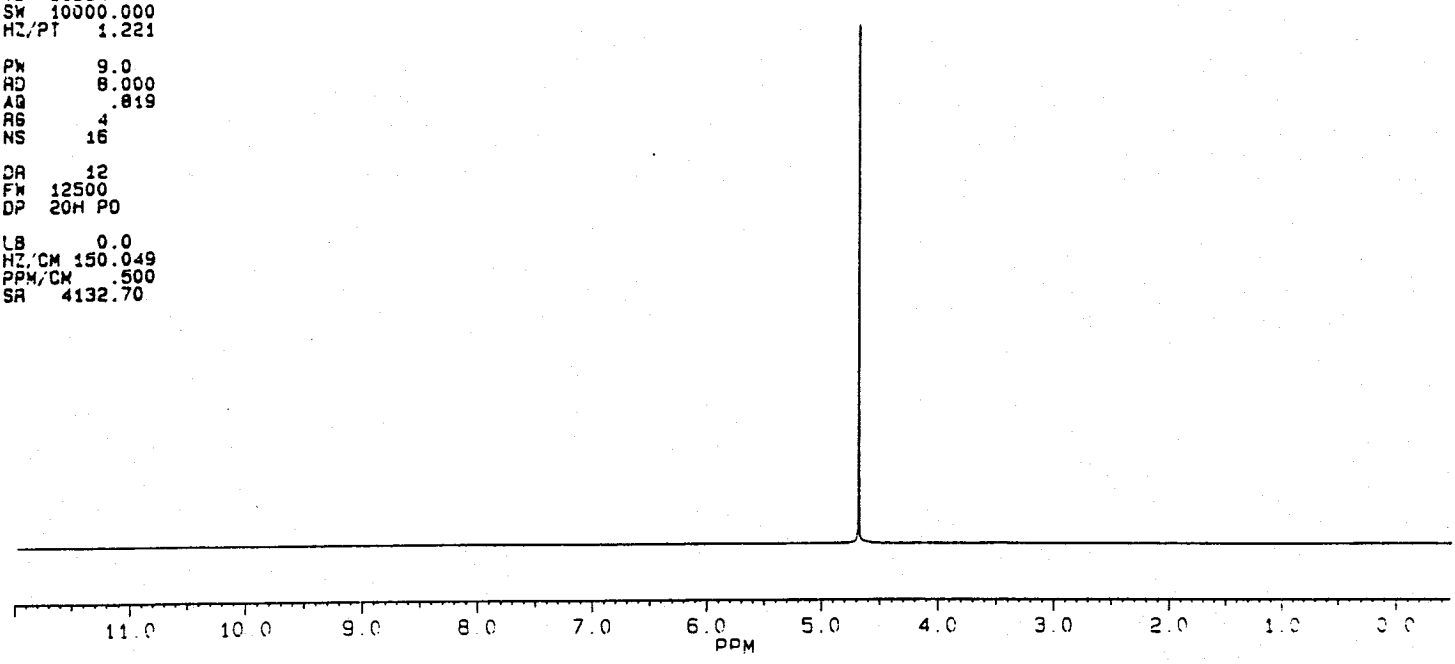
CAMB.057
DATE 25-2-88

SF 300.134
SFO 300.130
SY 100.0
O1 6600.000
SI 16384
TD 16384
SW 10000.000
HZ/PT 1.221

PW 9.0
RD 8.000
AQ .819
RG 4
NS 16

DR 12
FW 12500
DP 20H PO

LB 0.0
HZ/CM 150.049
PPM/CM .500
SR 4132.70



DIOXANE CIL LOT # P - 976

p-Dioxane-d_8
99 atom % D
DLM-28

BRUKER

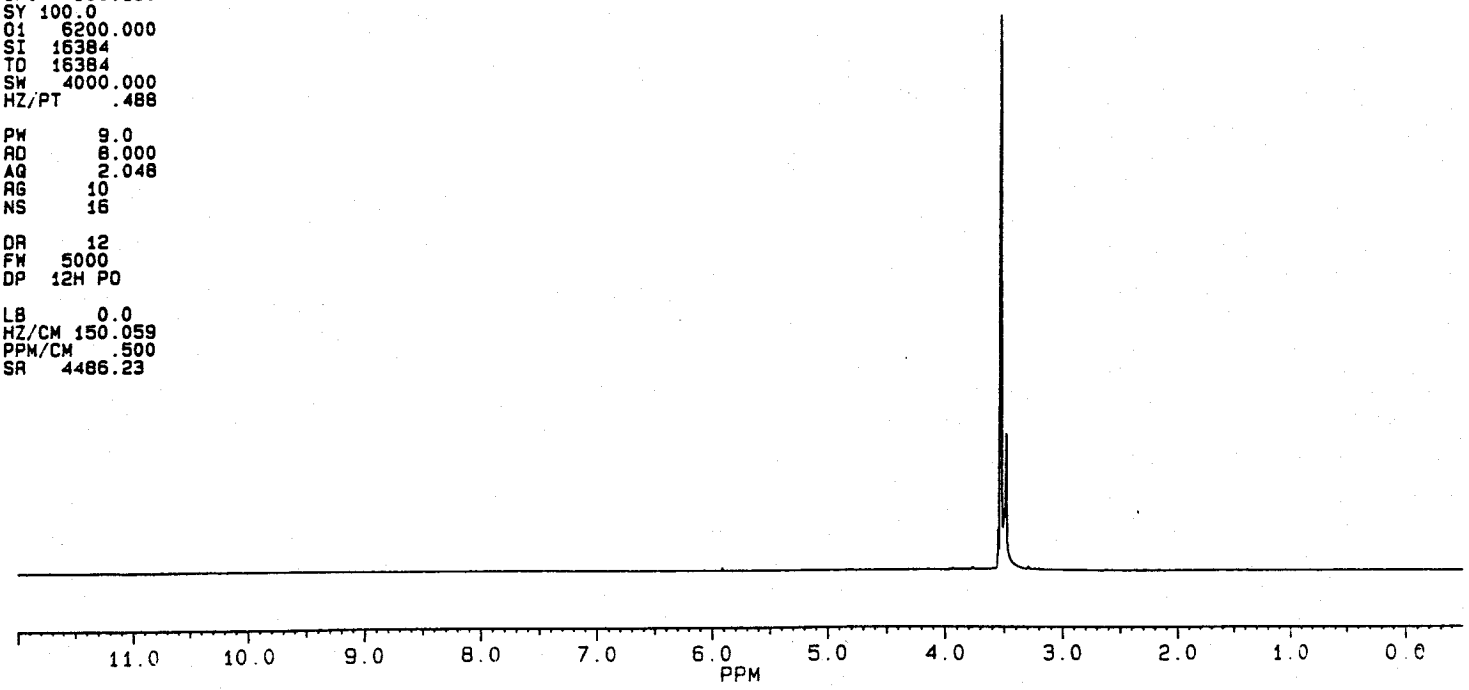
CAMB.093
DATE 7-3-88

SF 300.134
SFO 300.130
SY 100.0
O1 6200.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 9.0
RD 8.000
AQ 2.048
RG 10
NS 16

DR 12
FW 5000
DP 12H PO

LB 0.0
HZ/CM 150.059
PPM/CM .500
SR 4486.23



Matching the quality of the best competition is CIL's minimum level of acceptable performance!

METHANOL CIL LOT # F - 5057B

BRUKER

CAMB.037
DATE 24-2-88

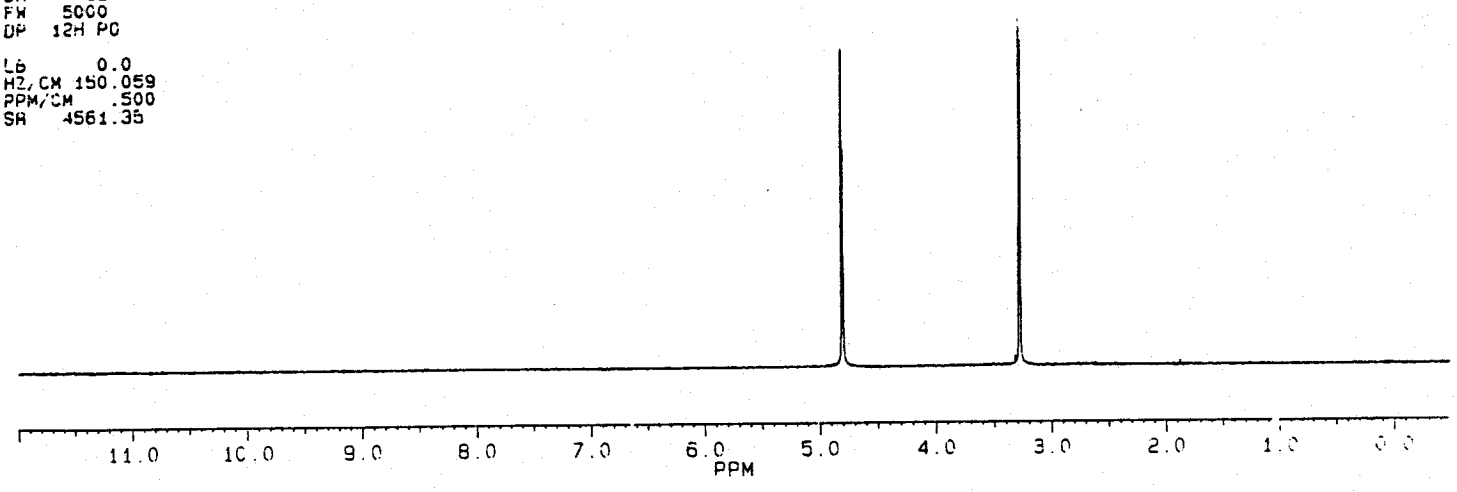
SF 300.135
SFO 300.130
SY 100.0
O1 6200.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 9.0
RD 8.000
AG 2.048
HG 10
NS 16

DR 12
FW 5000
DP 12H PD

LB 0.0
HZ/CM 150.059
PPM/CM .500
SR 4561.35

Methanol-d_4
99.8 atom % D
DLM-24



METHANOL CIL LOT # F - 5239C

BRUKER

CAMB.042
DATE 25-2-88

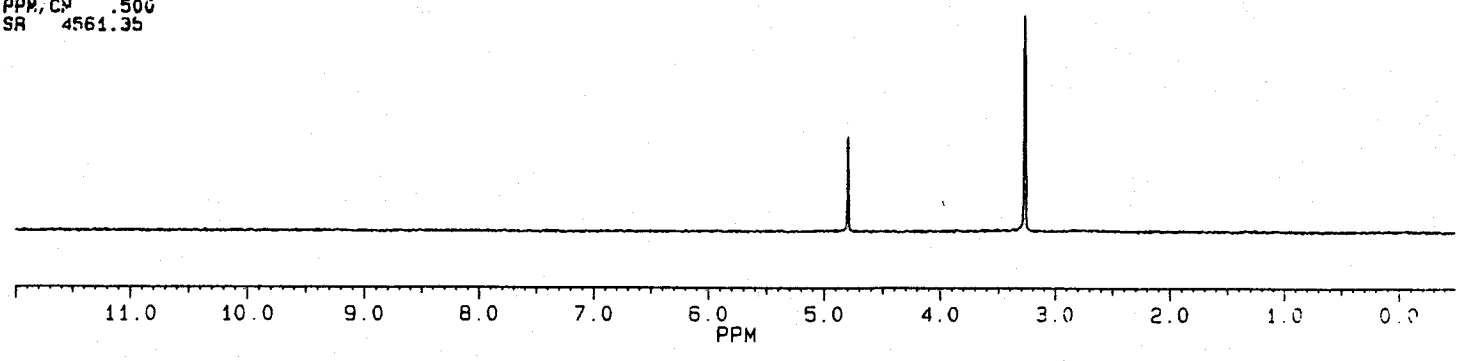
SF 300.135
SFO 300.130
SY 100.0
O1 6200.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 9.0
RD 8.000
AG 2.048
HG 10
NS 16

DR 12
FW 5000
DP 12H PD

LB 0.0
HZ/CM 150.059
PPM/CM .500
SR 4561.35

Methanol-d_4 "100%"
99.96 atom % D
DLM-51



Matching the quality of the best competition is CIL's minimum level of acceptable performance!

METHYLENE CHLORIDE CIL LOT # F - 5669B

BRUKER

CAM8.091
DATE 3-3-88

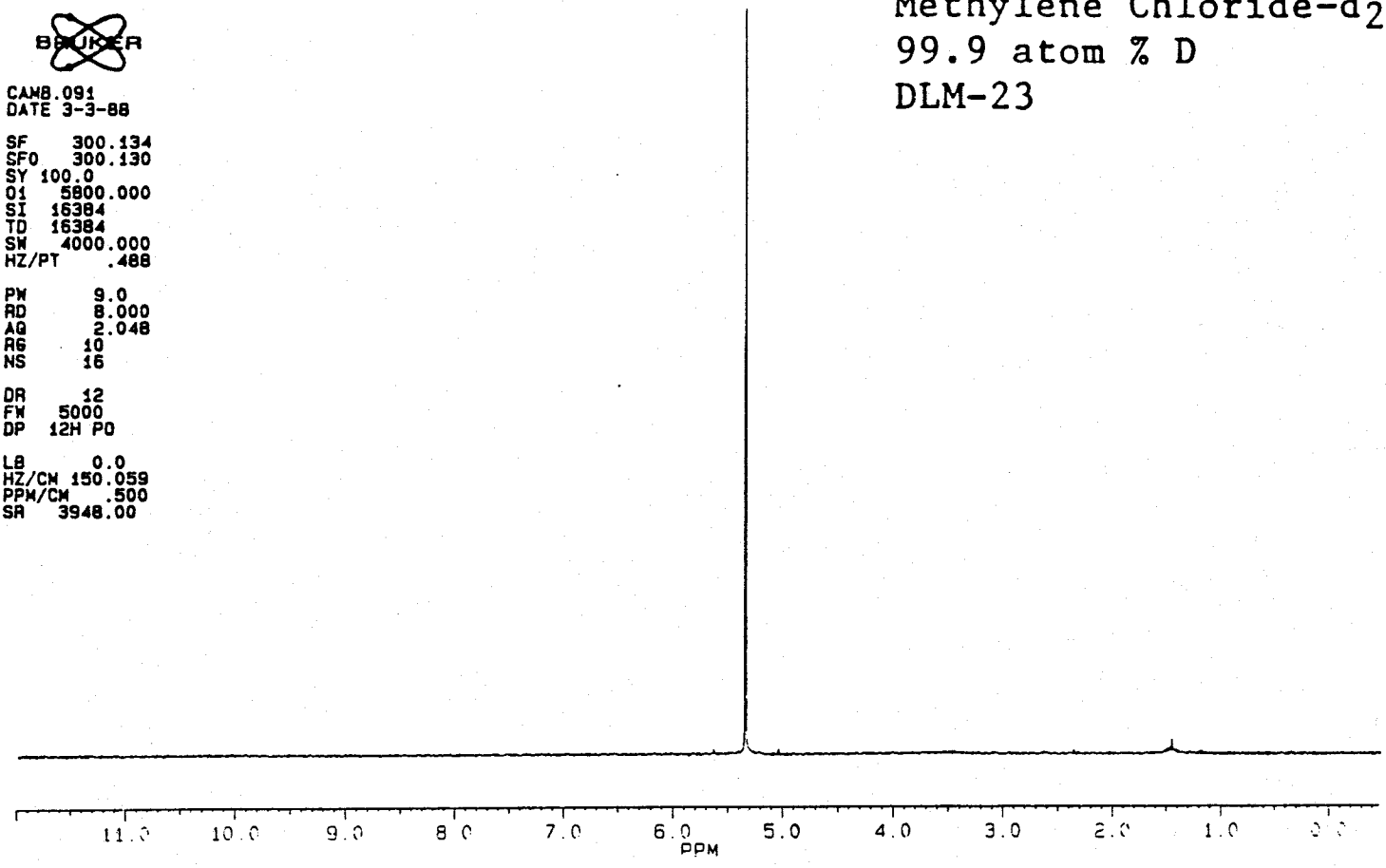
SF 300.134
SFO 300.130
SY 100.0
O1 5800.000
SI 16384
TD 16384
SW 4000.000
HZ/PT .488

PW 9.0
RD 8.000
AQ 2.048
RG 10
NS 16

DR 12
FW 5000
DP 12H PO

LB 0.0
HZ/CM 150.059
PPM/CM .500
SR 3948.00

Methylene Chloride-d_2
99.9 atom % D
DLM-23



PYRIDINE CIL LOT # F - 4991C

BRUKER

CAM8.080
DATE 29-2-88

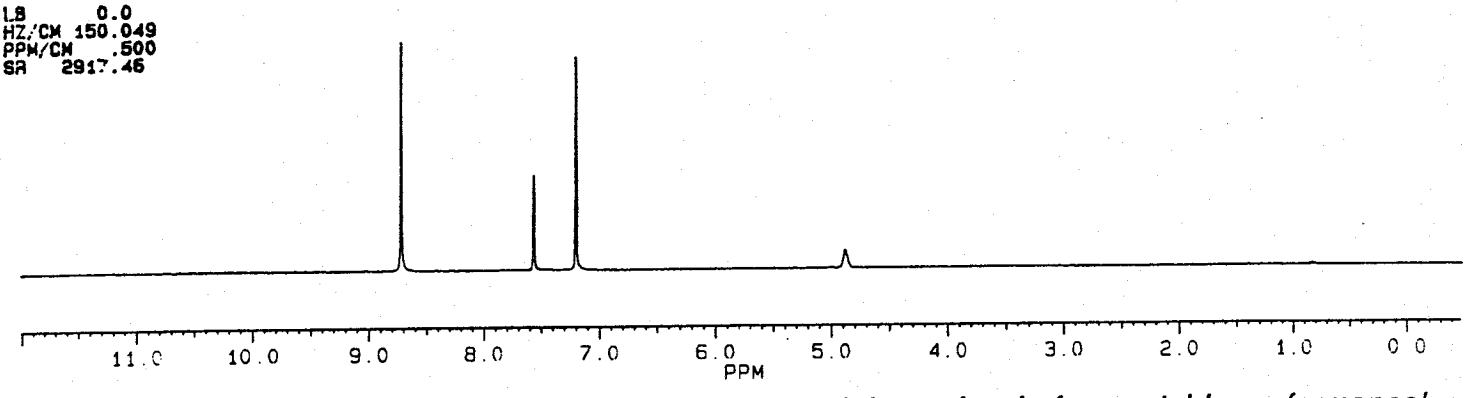
SF 300.133
SFO 300.130
SY 100.0
O1 5000.000
SI 16384
TD 16384
SW 5000.000
HZ/PT .610

PW 9.0
RD 8.000
AQ 1.638
RG 4
NS 16

DR 12
FW 6300
DP 12H PO

LB 0.0
HZ/CM 150.049
PPM/CM .500
SR 2917.46

Pyridine-d_5
99.5 atom % D
DLM-13



Matching the quality of the best competition is CIL's minimum level of acceptable performance!

TOLUENE CIL LOT # F - 5451A

BRUKER

CAMB.063
DATE 29-2-88

SF 300.133
SFO 300.130
SY 100.0
O1 5000.000
SI 16384
TD 16384
SW 5000.000
HZ/PT .610

PW 9.0
RD 8.000
AQ 1.638
RG 4
NS 16

DR 12
FW 6300
DP 12M PO

LB 0.0
HZ/CM 150.049
PPM/CM .500
SR 3400.43

Toluene-d_8
99.6 atom % D
DLM-5