Intermolecular cyclisation of isosorbide in basic condition by DMC chemistry

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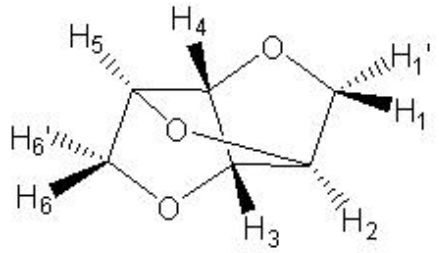
Supporting information

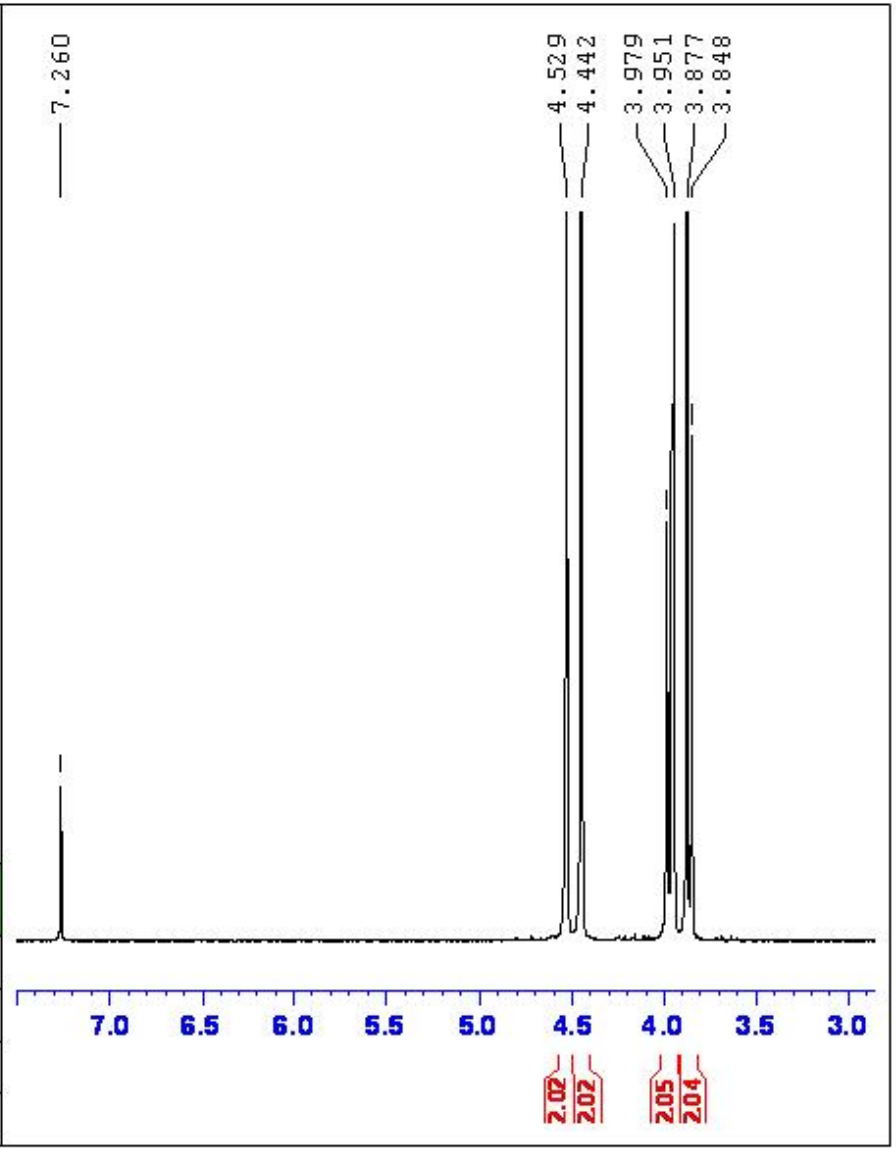
General Information:

Proton Nuclear Magnetic Resonance spectra (^{1}H NMR) were recorded at 300 MHz on a Bruker 300 Ultra Shield apparatus. The chemical shifts are reported in ppm from the solvent resonance as the internal standard ($CDCl_3$: 7.26 ppm) and regarding the tetramethylsilane (TMS). Data are reported as follows: chemical shift, integration multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, qui = quintet, sext = sextet, sept = septet, br = board, m = multiplet) and coupling constants (Hz). Carbon Nuclear Magnetic Resonance spectra (^{13}C NMR) were recorded at 75 MHz on a Bruker apparatus 300 Ultra Shield. Chemical shift are reported in ppm from the solvent resonance as internal standard ($CDCl_3$: 77 ppm). Analytical chromatography (TLC) was performed on Merck precoated TLC plates (silica gel 60 GF254, 0.25 mm). Flash column chromatography was performed on silica gel 60Å grade 9385 (Merck 230-400 mesh). GC-MS analysis were performed on a Agilent 6890N Gas Chromatograph equipped with HP-5MS 30 m x 0.25 mm column and Agilent 5973 Network Mass selective detector. All the reaction were performed using dimethylcarbonate (Aldrich, 99% purity) dried on molecular sieves 4Å. K_2CO_3 was bought by Aldrich and dried at 90°C in a vacuum oven prior to use. KW2000 was bought by Kyowa chemical industry company ltd , calcinated at 500°C and conserved at 80°C in an oven. All the bases and catalysts were purchased by Aldrich and used without further purification. The other simple chemicals were used as such.

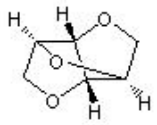
NMR ^1H
GG1-136-1
$CDCl_3$ 300 MHz
Cyclic compound

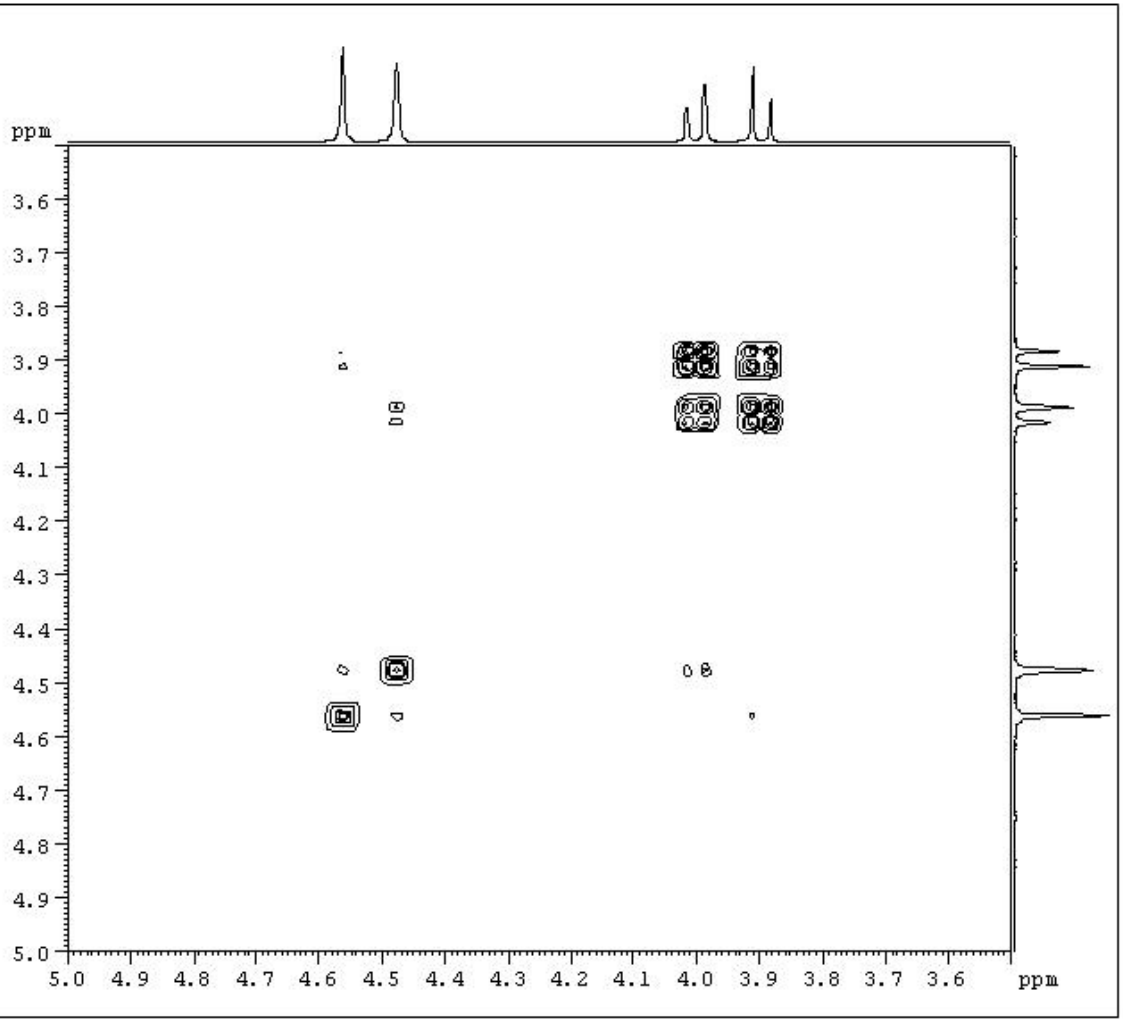


chemical shift (ppm)	integration	multiplicity	coupling (Hz)	attribution
4.54	2	s	-	2-5
4.46	2	s	-	3-4
3.98	2	d	3.6	1-6
3.88	2	d	6	1'-6'

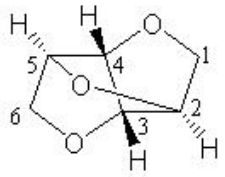


NMR 1H COSY
GG1-136-2 COSY
$CDCl_3$ 300 MHz
Cyclic compound

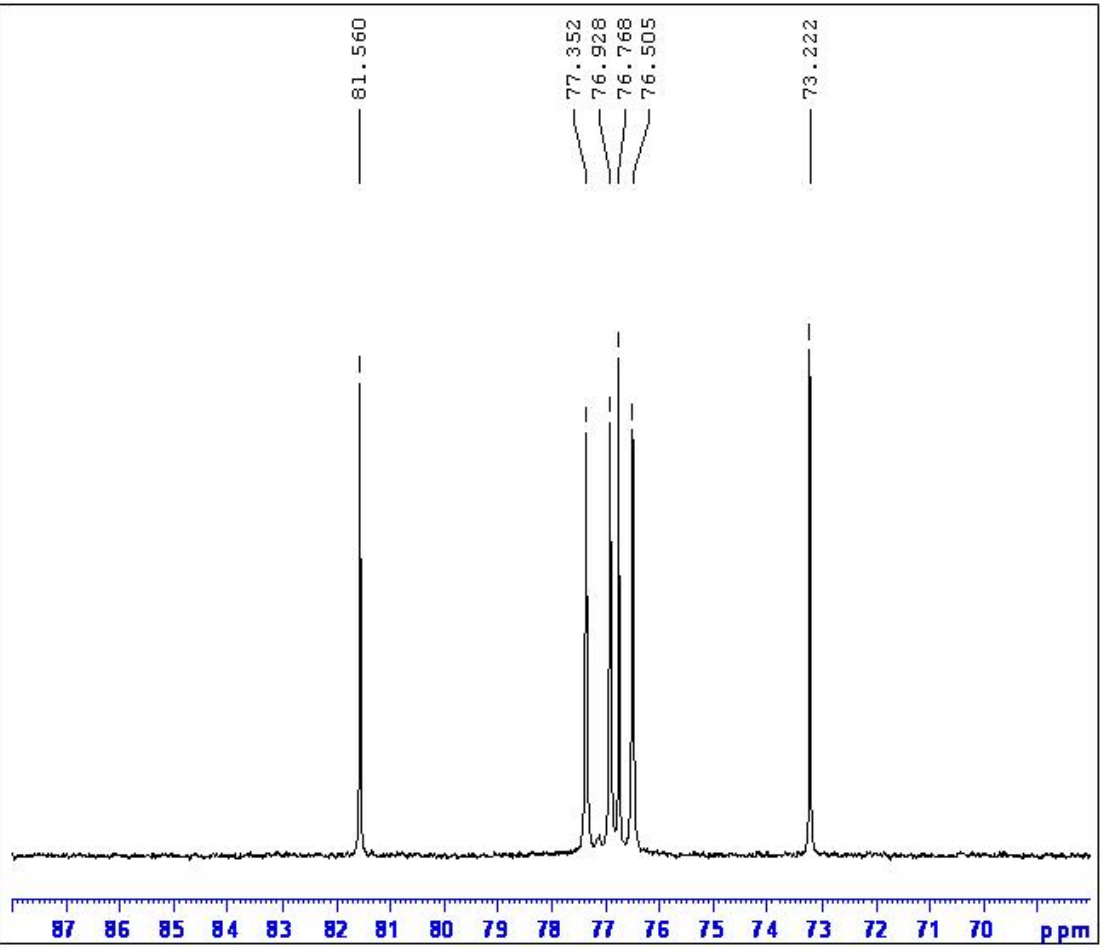


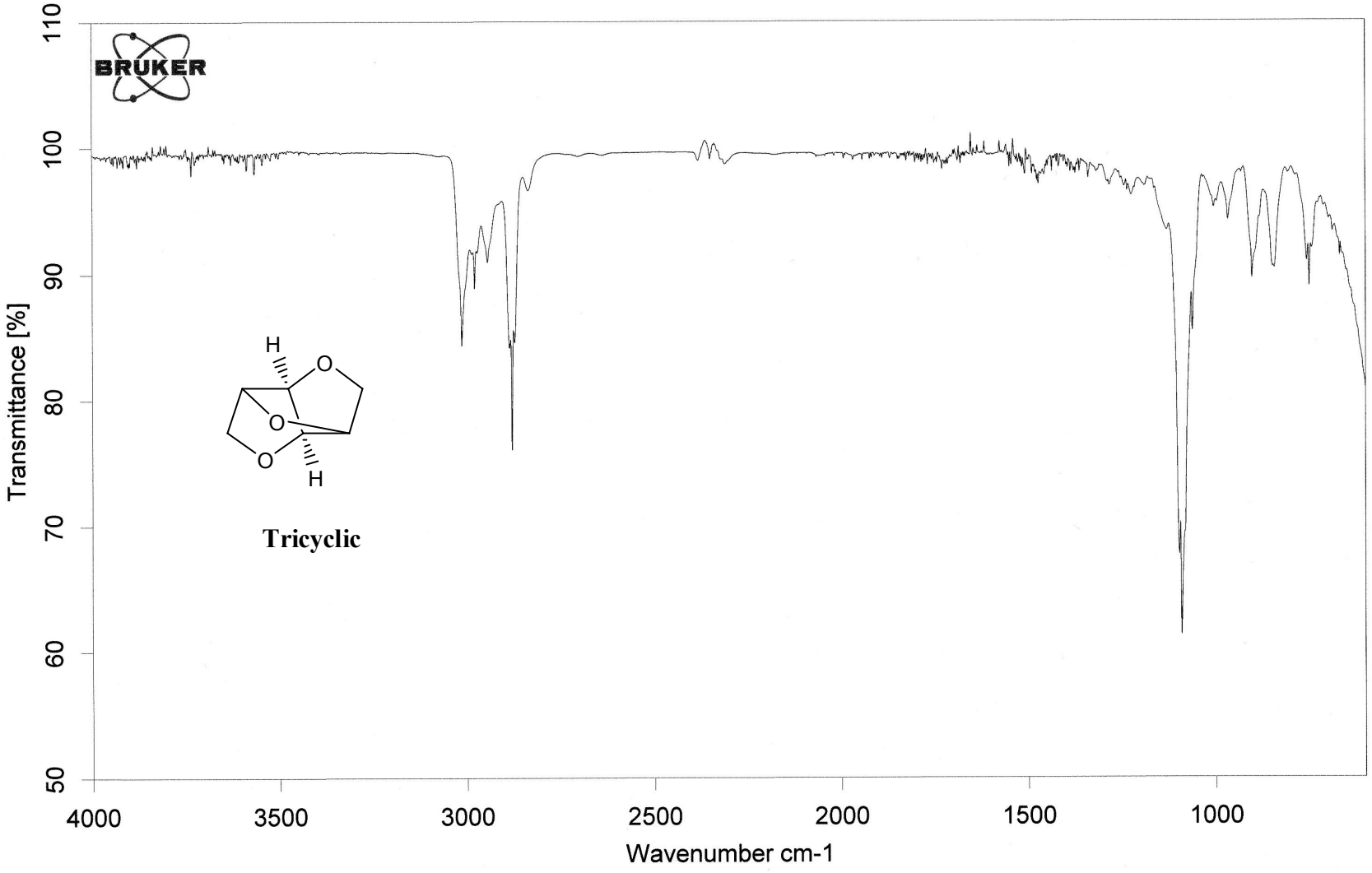


NMR 13C
GG1-136-3 C13
$CDCl_3$ 75 MHz
Cyclic compound

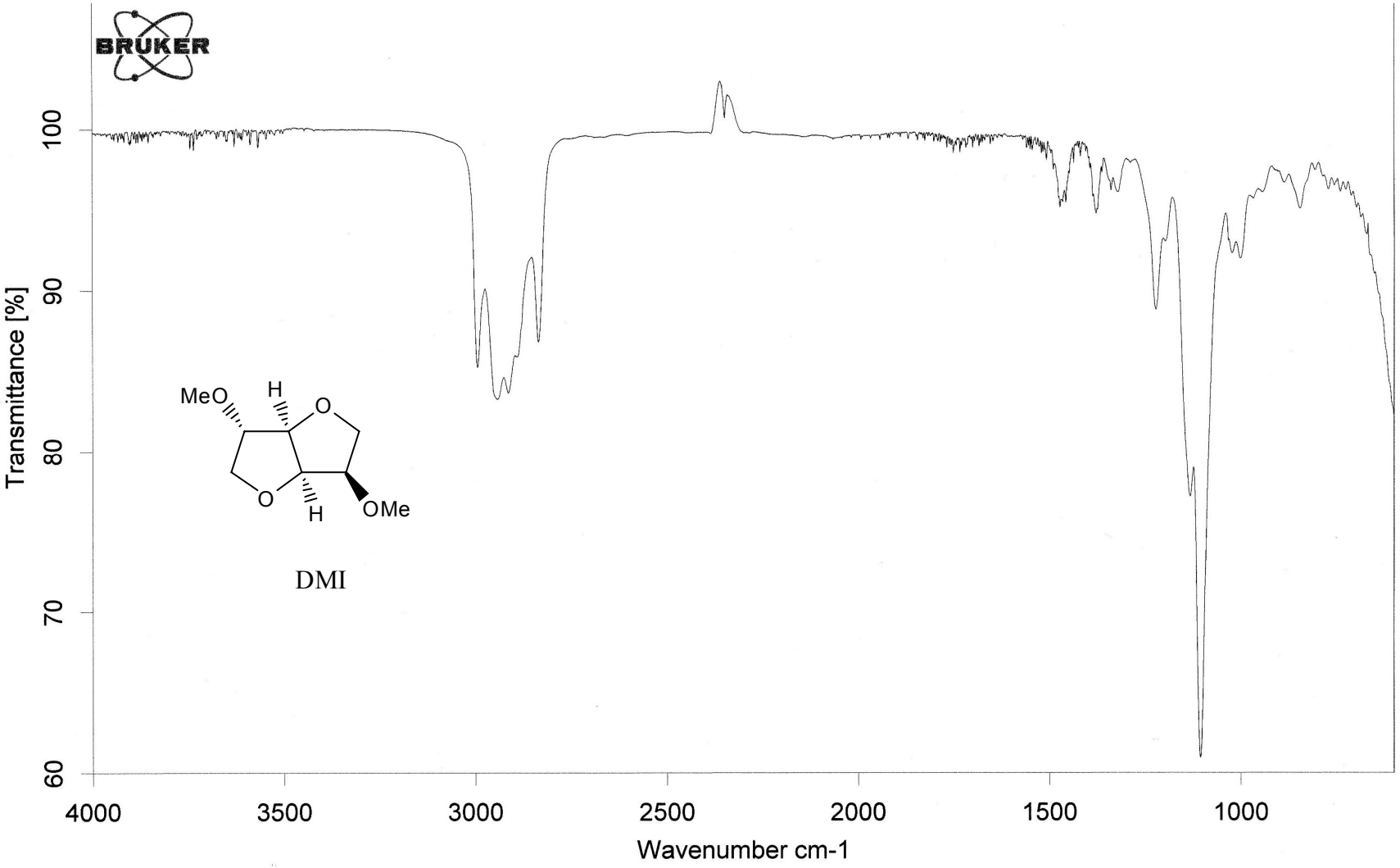


chemical shift (ppm)	attribution
81.6	3-4
76.8	2-5
73.2	1-6





C:\BALDAN\Derivato DMI_9 mg a ca. 125 C.0	Nuovo campione del derivato di DMI	9 mg di cristalli bianchi, sotto vuoto e a ca.125 °C, in	02/02/2010



DMI

C:\BALDAN\Tundo_Dimethyl isosorbide (liquido) e Derivato (solido)\2' DMI\Nuovo campione di DMI_20 micro L a 125 C.0	Nuovo campione di	02/02/2010

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Instrument :    Instrumen
Sample Name:
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