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of Reliable Methods
for the Preparation
of Organic Compounds

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September 2014: The paragraphs above replace the section "Handling and Disposal of Hazardous Chemicals" in the originally published version of this article. The statements above do not supersede any specific hazard caution notes and safety instructions included in the procedure.

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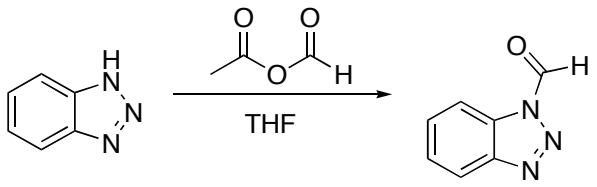
Practical and Efficient Synthesis of *N*-Formylbenzotriazole



Submitted by Mhairi Matheson, Adele E. Pasqua, Alan L. Sewell and Rodolfo Marquez.*[1]
Checked by Diptarka Hait, Jonathan Truong, and Mohammad Movassaghi.

1. Procedure

A 500-mL, 3-necked, round-bottomed flask (Note 1) equipped with a 5 cm Teflon-coated cylindrical magnetic stir bar, an internal thermometer, a rubber septum, and a Vigreux condenser capped with a rubber septum with an argon inlet, is charged with acetic anhydride (26.5 mL, 28.6 g, 280 mmol, 1.0 equiv) (Note 2). The contents are cooled to –10 ºC by means of a Dewar filled with isopropanol and an immersion cooler (Neslab CC 100) (Note 3), then 100% formic acid (21.1 mL, 25.8 g, 560 mmol, 2.0 equiv) (Note 4) is added in portions to the flask over 3 min via a measuring cylinder [Caution: exotherm from –10 ºC to 1 ºC]. The resulting colorless reaction mixture is then heated in an oil bath on a hot plate to 43 ºC (internal temperature) over 25 min. The reaction mixture is stirred vigorously for 3 h (42–44 ºC, internal temperature) and monitored by ^{1}H NMR analysis (Note 5).

^{1}H NMR analysis of crude reaction aliquots is employed to determine the quantity of acetic formic anhydride in the reaction mixture (18.1 g, 206 mmol). Formic anhydride (2.4 g, 32 mmol) was also present (Notes 6 and 7). The crude anhydride mixture is then cooled to 5 ºC over 30 min using an ice bath. While maintaining a positive pressure of argon, the Vigreux condenser is removed and replaced with a rubber septum with an argon inlet.

A 500-mL, two-necked round-bottomed flask, equipped with a 5 cm Teflon-coated magnetic stir bar, an internal thermometer, and a rubber septum with an argon inlet, is charged with benzotriazole (23.8 g, 200 mmol, 0.84 equiv relative to formylating agents (Notes 6 and 8). Anhydrous tetrahydrofuran (100 mL) is added to the benzotriazole via a measuring

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cylinder and the resulting light yellow solution is cooled to –15 ºC (internal temperature) over 20 min by means of an ice/salt water bath. The crude mixed anhydride is then added to the benzotriazole solution via cannula over 12 min, ensuring that the internal temperature does not exceed –5 ºC (Note 9). Upon completion of addition, the reaction mixture is cooled to –15 ºC (internal temperature) by means of a Dewar filled with isopropanol and an immersion cooler (Neslab CC 100) (Note 3) and vigorously stirred until completion by TLC analysis (2 h) (Note 10), at which point the reaction mixture is thick and cream colored.

The reaction mixture is transferred to a single-necked, 1-L round-bottomed flask and concentrated to dryness by rotatory evaporation (40 ºC bath temperature, 20 mmHg). Chloroform (50 mL) (Notes 11 and 12) is added to the flask and the solution is concentrated again (40 ºC bath temperature, 20 mmHg). The dilution and concentration procedure is repeated 3 times, yielding a white solid. The solid is dried at room temperature, under 0.75 mmHg for 12 h. The white solid is ground with a glass rod and dried at room temperature, under 0.75 mmHg for a further 48 h to afford 29.3 g (99.7%) of *N*-formylbenzotriazole (Notes 13, 14, and 15).

2. Notes

1. All glass equipment was either flame dried (checkers) or oven dried (submitters) and then maintained under a positive pressure of argon during the course of the reaction. Thermometers and rubber septa were used without any form of drying.

2. Both the submitters and checkers used acetic anhydride (99%) and formic acid (99%) obtained from Acros Organics (used as received).

3. The submitters report the use of an ice/salt bath is sufficient for temperature control at this stage.

4. The submitters recommend the use of a syringe for transfer of the formic acid. An excess of formic acid is necessary to achieve maximum conversion of acetic anhydride into acetic formic anhydride.

5. The reaction was monitored by ^{1}H-NMR analysis of crude reaction aliquots in $CDCl_3$ until the amount of acetic anhydride was minimal. The checkers observed that the reaction was essentially complete in 1.5 h (reached equilibrium with no significant further change in composition).

6. ^{1}H NMR integrations were measured for the formyl H's and equated to the total amount of formyl group supplied. The spectra were obtained within 15 minutes of removal from the reaction mixture.

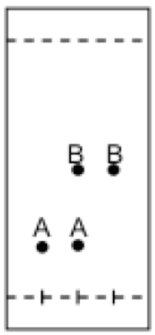
δ	Compound	Integral	Amount
9.1	Acetic formic anhydride	1.00	(1.00/2.72)*560 mmol = 206 mmol
8.75	Formic anhydride	0.31	0.5*(0.31/2.72)*560 mmol = 32 mmol (64 mmol formyl H)
8.0	Formic acid	1.41	(1.41/2.72)*560 mmol = 290 mmol
	Total	**2.72**	**528 mmol (560 mmol formyl H)**

7. Acetic formic anhydride was maintained under a positive pressure of argon to prevent hydrolysis. See *Org. Synth*, **1970**, *50*, 1 for an alternative synthetic route to acetic formic anhydride and for purification details.

8. Both the submitters and checkers obtained benzotriazole (99%, B11400-100G) from Sigma-Aldrich Inc. and used it as received. The submitters obtained tetrahydrofuran from Sigma Aldrich (CHROMASOLV® Plus, for HPLC, ≥99.9%, inhibitor-free, 34865) and purified though a Pure Solv 400-5MD solvent purification system (Innovative Technology, Inc.). The checkers obtained tetrahydrofuran from J.T. Baker (Cycletaner™) and purified through a Phoenix SDS Large Capacity solvent system (JC Myer Solvent Systems).

9. Slow addition led to a large amount of the acetyl product.

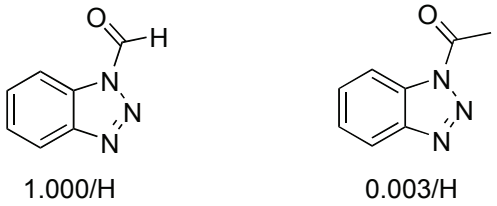
10. The reaction was monitored by TLC analysis on aluminium sheets pre-coated with silica (Merck Silica Gel 60 F_{254}) using 30% ethyl acetate in (40-60) petroleum ether. Spot A = benzotriazole (R_f = 0.2), spot B = *N*-formylbenzotriazole (R_f = 0.5). Middle lane was taken during the course of the reaction.



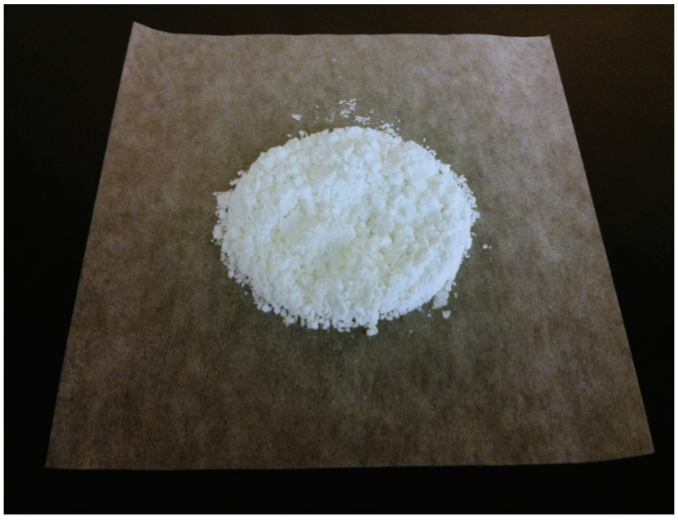
11. The submitters purchased chloroform from Fisher Chemical (C/4960/17) and the checkers obtained chloroform from Sigma Aldrich (CHROMASOLV® Plus, for HPLC, ≥99.9%, contains 0.5-1% ethanol as stabilizer, 650471)- used as obtained.

12. Removal of the volatile reaction components is best achieved by azeotroping the crude reaction product mixture with chloroform before the product is dried on a vacuum line.

13. *N*-Formylbenzotriazole was obtained in a 300:1 ratio compared to the *N*-acetylbenzotriazole side product as determined by ^{1}H NMR analysis.



14. The product exhibits the following physical and spectroscopic properties: mp 93–94 ºC. IR: ν_{max}/cm^{-1}; 3105, 1727, 1605, 1594. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.57 (1 H, ddd, *J* = 8.3, 7.2, 1.0 Hz), 7.70 (1 H, ddd, *J* = 8.2, 7.2, 1.0 Hz), 8.15 (1 H, dt, *J* = 8.3, 0.9 Hz), 8.24 (1 H, d, *J* = 8.2 Hz), 9.86 (1H, s). ^{13}C NMR (100 MHz, $CDCl_3$) δ: 113.6, 120.4, 127.0, 129.9, 130.8, 146.6, 159.8. HRMS $[M+H]^+$calculated for C_7H_5NO: 148.0506, found 148.0505. This characterization matches the data reported in the literature.[2] Anal. Calcd. for $C_7H_5N_3O$ (147.13): C, 57.14; H, 3.43; N, 28.56. Found: C, 57.06; H, 3.48; N, 28.75.



15. The submitters report that *N*-formylbenzotriazole stored in the freezer showed no decomposition by ^{1}H NMR analysis over a 6-month period. The submitters reported the formation of 59.5 g (98.5%) of product when the reaction is performed at double the reported scale.

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Discussion

The importance of formylation to organic chemists is reflected in the myriad of approaches and reagents designed to achieve it.[2] However, this in itself is indicative of the issues often experienced when working with unstable and impure formylating agents.

Formic halides and formic anhydrides are some of the most common formylating agents, however, they tend to suffer from stability problems and degrade easily upon storage.[3]

Formyl fluoride is the most widely used formylated halide, and has found wide-spread use in the formylation of alcohols, phenols and thiols in the presence of base.[3] As an electrophilic aromatic formylating agent, formyl fluoride requires activation *in situ* at low temperatures by catalytic amounts of Lewis acid.[3] Unfortunately, formyl fluoride decomposes at room temperature to carbon monoxide and hydrofluoric acid.[4]

Formic anhydride, on the other hand, has been used for the *O*-formylation of *p*-nitrophenol. Formylation of aromatic rings and other more

demanding substrates has proven highly problematic due to its instability above – 40 °C and its high chemical incompatibility.[3] Acetic formic anhydride, on the other hand, is a more stable formylating agent, but is still hydrolysed relatively easily.[5]

Coupling agents (i.e. DCC and EDCi) have also been used in conjunction with formic acid to achieve *N*- and *O*- formylation; however, the yields can be highly variable and the removal of the coupling agents' side products is often labour intensive.[6]

Cyanomethyl formate is a very useful, but difficult to prepare, formylating agent that has been used to formylate alcohols and nitroanilines in the presence of imidazole. Cyanomethyl formate can be synthesised from potassium formate and requires extensive purification to achieve the desired level of purity.[3] Isopropenyl formate is also a very fast and efficient formylating agent which unfortunately requires a lengthy synthesis involving the use of complex ruthenium catalysts.[4]

N-Formylbenzotriazole was initially developed by Katrizky as a stable and convenient alternative to achieve *N*- and *O*- formylation quickly and efficiently.[2] *N*-formylbenzotriazole is often considered to be the reagent of choice as it offers mild and selective formylation of alcohols, amines, and amides.[8]

Katrizky's synthesis begins with benzotriazole which is coupled to formic acid using *N,N'*-dicyclohexylcarbodiimide. The coupling itself proceeds well; however, the separation of the desired *N*-formylbenzotriazole and the urea side product is non-trivial and requires repeated inefficient recrystallisation which severely reduces the yield of the reaction. Furthermore, even after repeated recrystallisation and trituration, the *N*-formylbenzotriazole obtained is often contaminated with urea byproducts making the yields highly variable and irreproducible. Unfortunately, the use of alternative coupling agents, such as, *N,N'*-diisopropylcarbodiimide (DIC) and 1-ethyl-3-(3-dimethylaminopropyl)carbodiimide (EDCi) have been unsuccessful at yielding pure *N*-formylbenzotriazole in significant amounts.

In conclusion, a fast and efficient procedure for the synthesis of *N*-formylbenzotriazole has been developed. This procedure takes advantage of the inherent reactivity of acetic formic anhydride to formylate benzotriazole without the need of coupling reagents. Further to this, we exploit the volatility of the reagents and side products to allow the generation of highly pure *N*-formylbenzotriazole in excellent yield without the need for purification. As a whole, this procedure is a great improvement compared to

other methods available currently for the synthesis of *N*-formylbenzotriazole in terms of cost, yield and overall efficiency.[9]

1. Ian Sword Reader of Organic Chemistry and EPSRC Leadership Fellow. WestCHEM, School of Chemistry, University of Glasgow, Joseph Black Building, University Avenue, G12 8QQ. rudi.marquez@glasgow.ac.uk. We thank the Lord Kelvin-Smith programme, Dr. Ian Sword, and the University of Glasgow for postgraduate support (M.M., A.E.P., and A.L.S.). We also thank Dr. Ian Sword and the EPSRC for funding.

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Appendix
Chemical Abstracts Nomenclature; (Registry Number)

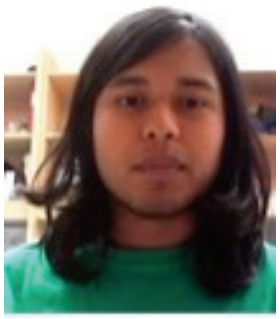
Acetic anhydride; (108-24-7)
Formic acid; (64-18-60
Benzotriazole; (95-14-7)
N-Formylbenzotriazole; (72773-04-7)

Dr. Rudi Marquez obtained his DPhil in 1999 at UCLA with Professor Michael E. Jung. Rudi then worked with Professor Ian Paterson at the University of Cambridge before joining the group of Professor Sir Jack Baldwin at the University of Oxford in 2000. In 2003 he began his independent career at the University of Dundee. In 2006, Rudi became the Ian Sword Lecturer at the University of Glasgow. Dr. Marquez's research interests reside at the interface between chemistry and biology, particularly applied to parasitology, cancer, tissue regeneration and crop protection.

Mhairi Matheson received her M.Sci. in Chemistry with Medicinal Chemistry from the University of Glasgow in 2010. As an undergraduate she completed an industrial placement at Syngenta (Jealott's Hill). Mhairi has been the recipient of numerous awards including a Nuffield Foundation summer grant and a Lord Kelvin-Smith Scholarship. Mhairi is currently undertaking doctoral studies under the supervision of Dr. Rudi Marquez in the rational design and synthesis of angiogenesis promoters through the use of small molecules to mimic protein-protein interactions.

Adele Elisa Pasqua was born in Cosenza, Italy and obtained her M.Sc. at University of Calabria in Pharmaceutical Chemistry and Technology. In 2007 Elisa obtained a scholarship at the Bracco Imaging Spa (Trieste, Italy) where she remained for two years before moving to the University of Trieste for a summer scholarship in Prof. Paoletti's group. In 2009, Elisa began her Ph.D. studies under the supervision of Dr. Marquez at the University of Glasgow. Elisa's research is in the area of synthetic methodology and natural product synthesis is supported by the Dr. Ian Sword Scholarship.

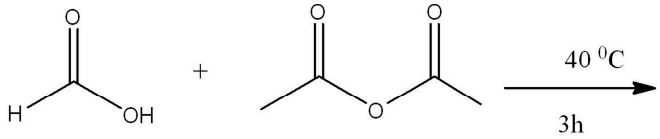
Alan L. Sewell is a native of Glasgow, where he obtained a M. Sci. in Chemistry with Drug Discovery at the University of Strathclyde. During his degree he worked with Novartis for 12 months as a medicinal chemist based in the gastrointestinal disease unit at their Horsham research centre. As part of his M.Sci., Alan carried out research on a palladium-catalysed cyclisation to give fluorinated carbocycles. In 2010, Alan began his Ph.D. studies with Dr Marquez at the University of Glasgow working on novel enamide containing small molecules with potential broad-spectrum antiparasitic activity.

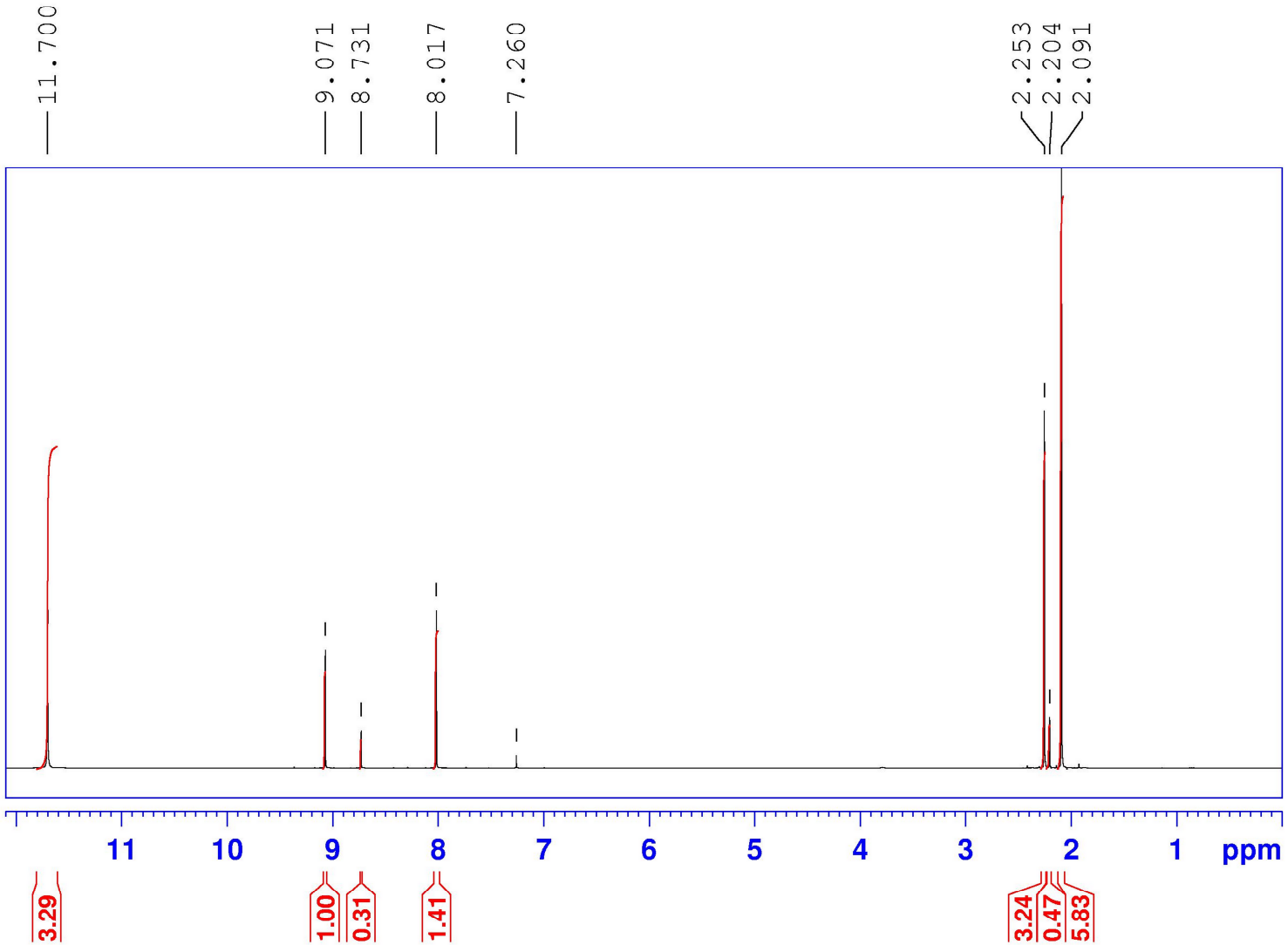


Diptarka Hait is currently pursing his undergraduate studies at MIT, and expects to receive his SB degree in Chemistry and Physics in 2016. He joined Professor Movassaghi's lab in 2012, where he is working on the development of new methods for organic synthesis and their application towards the total syntheses of alkaloid natural products.

Jonathan V. Truong pursued his undergraduate studies at Brown University, where he received his Sc.B. degree in chemistry in 2012. As an undergraduate he worked in the laboratory of Professor Jason Sello. In 2012, he joined Professor Mohammad Movassaghi's research group at MIT for his graduate studies where his research has focused on the development of new methodologies for organic synthesis and their application towards the total syntheses of alkaloid natural products.

Mixture of Anhydrides



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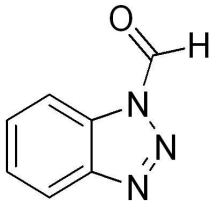

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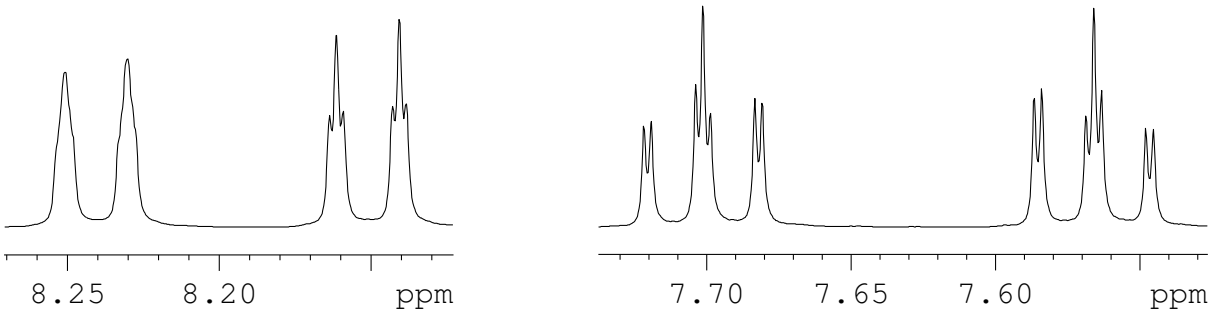

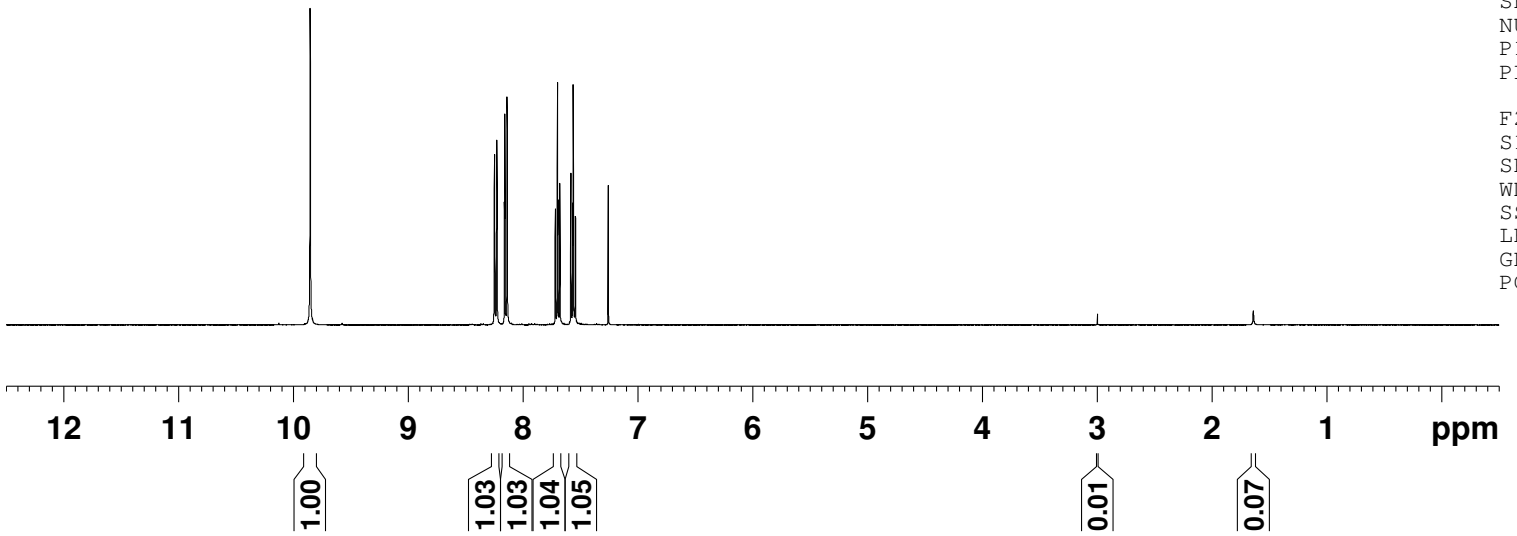

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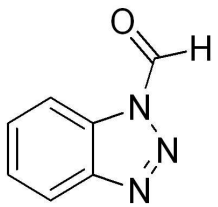

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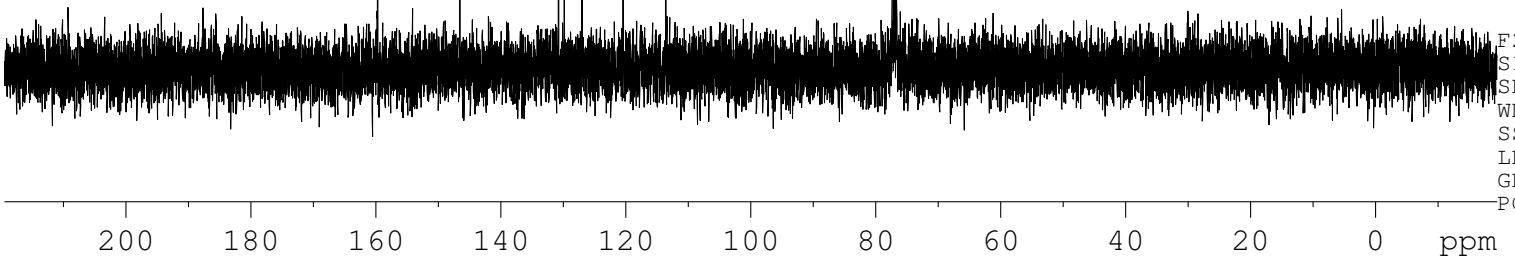

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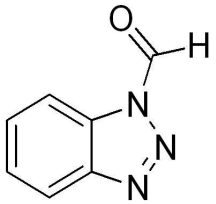

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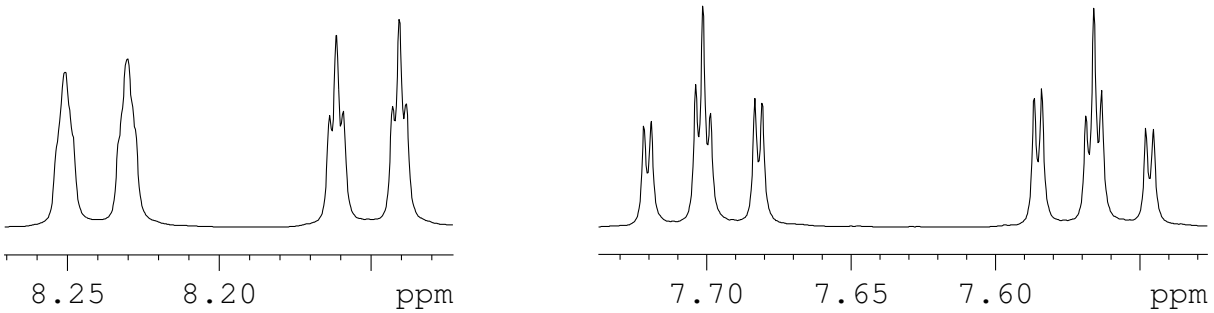

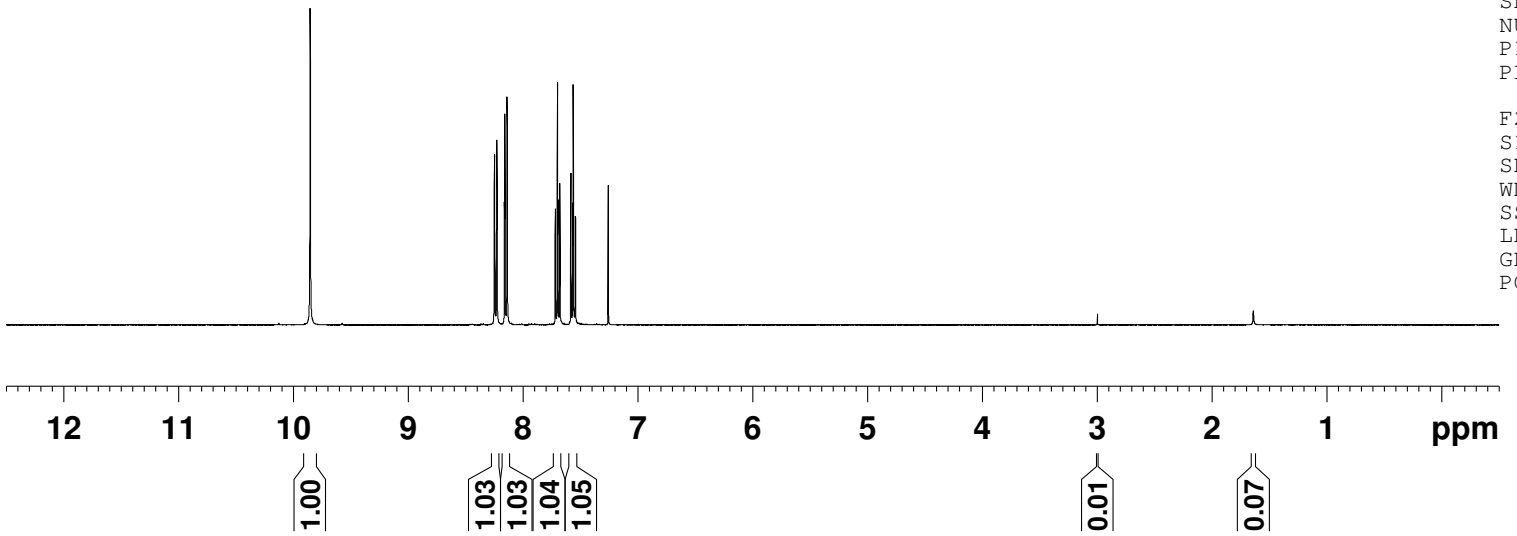

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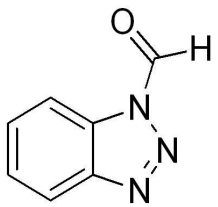
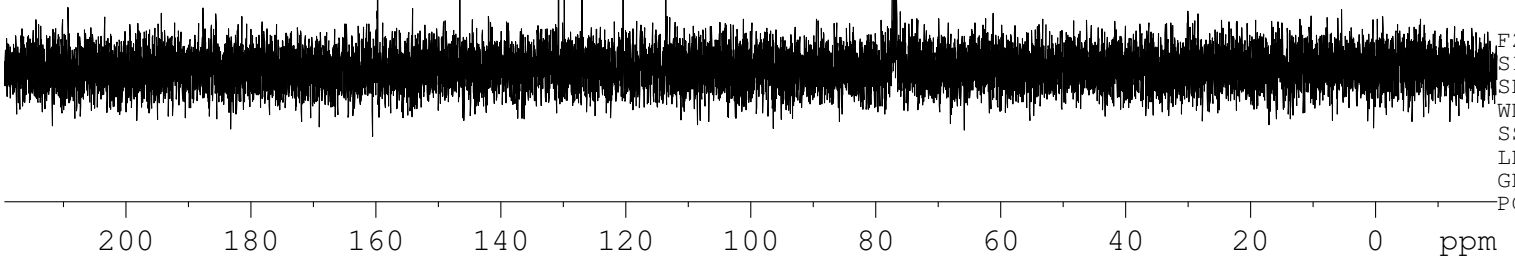
Date_ 20130427
Time 13.46
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 32
DW 62.400 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 14.50 usec
PLW1 10.00000000 W

F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



159.77
146.56
130.75
129.88
127.01
120.40
113.59
77.35
77.03
76.72



BRUKER

```
Current Data Parameters
NAME         N-formylbenzotri
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_             20130427
Time                 13.52
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     24
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631488 sec
RG                    203
DW                 20.800 usec
DE                  6.50 usec
TE                 298.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1          44.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2          10.00000000 W
PLW12          0.25957000 W
PLW13          0.21025001 W

F2 - Processing parameters
SI                   32768
SF            100.6127690 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```