

This article was downloaded by:

On: 27 January 2011

Access details: *Access Details: Free Access*

Publisher *Taylor & Francis*

Informa Ltd Registered in England and Wales Registered Number: 1072954 Registered office: Mortimer House, 37-41 Mortimer Street, London W1T 3JH, UK



Organic Preparations and Procedures International

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t902189982>

3-ARYL-1,2,5-THIADIAZOLES FROM THE REACTION OF TETRASULFUR TETRANITRIDE WITH ARYL METHYL KETONES

Shuntaro Mataka^a; Kazufumi Takahashi^a; Masashi Tashiro^a

^a Research Institute of Industrial Science and Department of Molecular Science and Technology, Graduate School of Engineering Sciences Kyushu University 86, Fukuoka, Japan

To cite this Article Mataka, Shuntaro , Takahashi, Kazufumi and Tashiro, Masashi(1985) '3-ARYL-1,2,5-THIADIAZOLES FROM THE REACTION OF TETRASULFUR TETRANITRIDE WITH ARYL METHYL KETONES', *Organic Preparations and Procedures International*, 17: 2, 152 – 154

To link to this Article: DOI: 10.1080/00304948509355490

URL: <http://dx.doi.org/10.1080/00304948509355490>

PLEASE SCROLL DOWN FOR ARTICLE

Full terms and conditions of use: <http://www.informaworld.com/terms-and-conditions-of-access.pdf>

This article may be used for research, teaching and private study purposes. Any substantial or systematic reproduction, re-distribution, re-selling, loan or sub-licensing, systematic supply or distribution in any form to anyone is expressly forbidden.

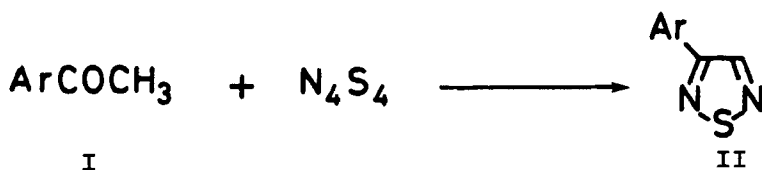
The publisher does not give any warranty express or implied or make any representation that the contents will be complete or accurate or up to date. The accuracy of any instructions, formulae and drug doses should be independently verified with primary sources. The publisher shall not be liable for any loss, actions, claims, proceedings, demand or costs or damages whatsoever or howsoever caused arising directly or indirectly in connection with or arising out of the use of this material.

**3-ARYL-1,2,5-THIADIAZOLES FROM THE REACTION
OF TETRASULFUR TETRANITRIDE WITH ARYL METHYL KETONES[†]**

Submitted by Shuntaro Mataka, Kazufumi Takahashi and Masashi Tashiro*
(07/31/84)

Research Institute of Industrial Science
and Department of Molecular Science and Technology
Graduate School of Engineering Sciences
Kyushu University 86
6-1 Kasuga-kohen, Kasuga-shi, Fukuoka 816, JAPAN

The characteristic feature of the reaction of N_4S_4 with organic substrates is the formation of 1,2,5-thiadiazoles.¹ The reactions with diaroyl- and diarylacetylenes are useful for the preparation of 3,4-disubstituted 1,2,5-thiadiazoles,² while mono arylacetylenes gave the corresponding 3-aryl-1,2,5-thiadiazoles (IIa-c) in poor yields.² We now report a convenient albeit low yield preparation of II by the reaction of N_4S_4 with aryl methyl ketones (I).



- | | | |
|-----------------------|-------------------------|-------------------------|
| a) Ar = C_6H_5 | b) Ar = 4- $CH_3C_6H_4$ | c) Ar = 4- BrC_6H_4 |
| d) Ar = 4- ClC_6H_4 | e) Ar = 3- BrC_6H_4 | f) Ar = 3- $O_2NC_6H_4$ |
| g) Ar = 2- BrC_6H_4 | h) Ar = 2- ClC_6H_4 | i) Ar = 2-thienyl |

As N_4S_4 did not react with acetophenone (Ia) in refluxing xylene, the reactions were carried out in excess I without solvent to yield the expected 3-aryl-1,2,5-thiadiazoles (II). Although a vigorous reaction occurred with 2-furyl and 2-pyridyl methyl ketones, only large amounts of resinous materials were formed. No 1,2,5-thiadiazole was detected from the reaction of N_4S_4 with 2- and 4-hydroxyacetophenones.³

EXPERIMENTAL SECTION

All melting points are uncorrected. Column chromatography was carried out on silica gel (Wako-gel, C-300).

TABLE. Yields, mps and Analytical Data of 3-Aryl-1,2,5-Thiadiazole (II).

compd	mp. (°C)	Yield ^a (%)	Elemental Analysis (Found)		
			C	H	N
IIa	42-44°	35	lit. ² mp. 43-44°		
IIb	56-57°	15	lit. ² mp. 56-57°		
IIc	120-121°	30	lit. ² mp. 120-121°		
IIId	101-102°	25	48.86(48.89)	2.56(2.72)	14.29(14.23)
IIe	40-42°	41	39.85(39.74)	2.09(2.02)	11.62(11.42)
IIIf	108-109°	30	46.37(46.76)	2.43(2.65)	20.28(20.42)
IIg	63-64°	13 ^{b,d}	39.85(40.28)	2.09(2.25)	11.62(11.29)
IIh	88-89°	25 ^{b,c}	48.86(48.66)	2.56(2.68)	14.29(14.04)
IIi	38-39°	10	42.86(42.93)	2.40(2.54)	16.67(16.37)

a) Carried out at 85-95° for 7 hrs unless otherwise noted; b) Carried out at 110-120° for 3 hrs.; c) Nitrile formed in 13%; d) Small amount of nitrile product.

General Procedure.— A mixture of N₄S₄ (1.00 g) and I (4.00 g) was heated at the specified temperature. After the reaction was over, benzene (4 ml) was added to the reaction mixture and it was allowed to cool to room temperature. Precipitated sulfur and inorganics were collected and the filtrate was chromatographed. Sulfur was eluted with hexane and the mixture of I and II was eluted with benzene, which was chromatographed again with hexane as an eluent to give II. Compounds II were purified by recrystallization from hexane or by sublimation under reduced pressure.

REFERENCES

† Part 15 of Sulfur Nitride in Organic Chemistry. Part 14: S. Mataka, K.

Takahashi and M. Tashiro, Rep. of Res. Inst. Ind. Sci. Kyushu Univ., No. 77, In press (1984).

1. S. Mataka, K. Takahashi and M. Tashiro, Sulfur Reports, 4, 1 (1984).
2. S. Mataka, K. Takahashi, Y. Yamada and M. Tashiro, J. Heterocyclic Chem., 16, 1009 (1979). See also S. T. A. K. Daley, C. W. Rees and D. J. Williams, Chem. Comm., 55, 57 (1984).
3. S. Mataka, A. Hosoki, K. Takahashi and M. Tashiro, J. Heterocyclic Chem., 17, 1681 (1980).