TABLE I

α,β-UNSATURATED SULFONES, $RSO_2CH{=}CHR'$

R	R'	M.P., °C.	Yield,[a] %	Analysis				
				Calcd. for	Carbon		Hydrogen	
					Calcd.	Found	Calcd.	Found
CH_3	m-$O_2NC_6H_4$	130–132	49 (16)					
CH_3	p-ClC_6H_4	125–126	32	$C_9H_9ClO_2S$[c]	49.87	50.16	4.20	4.46
CH_3	2,4-$Cl_2C_6H_3$	72–73	62	$C_9H_8Cl_2O_2S$[c]	43.04	42.91	3.21	3.42
C_6H_5	C_6H_5	74–74.5	39 (21)					
C_6H_5	o-$O_2NC_6H_4$	131–132	44 (10)					
C_6H_5	m-$O_2NC_6H_4$	142–143	64 (14)					
C_6H_5	p-$O_2NC_6H_4$	169–170	52	$C_{14}H_{11}NO_4S$[b]	58.11	58.41	3.83	4.15
C_6H_5	o-ClC_6H_4	105–106	35	$C_{14}H_{11}ClO_2S$[c]	60.32	59.89	3.98	3.72
C_6H_5	p-ClC_6H_4	129–130	63 (28)					
C_6H_5	2,4-$Cl_2C_6H_3$	132–133	48	$C_{14}H_{10}Cl_2O_2S$[b]	53.68	54.20	3.22	3.47
C_6H_5	3,4-$Cl_2C_6H_3$	156–156.5	44	$C_{14}H_{10}Cl_2O_2S$[b]	53.68	53.38	3.22	3.67
C_6H_5	p-$CH_3OC_6H_4$	117–118	19	$C_{15}H_{14}O_3S$[b]	65.64	65.70	5.14	5.23
C_6H_5	p-HOC_6H_4	109–110	10	$C_{14}H_{12}O_3S$[d]	64.59	64.43	4.65	4.93
C_6H_5	p-$CH_3C_6H_4$	135.5–136.5	28 (17)					
C_6H_5	3-Pyridyl	85–86	13	$C_{13}H_{11}NO_2S,2H_2O$[e]	55.49	55.46	5.38	5.55
C_6H_5	4-Pyridyl	190–191	5	$C_{13}H_{11}NO_2S,H_2O$[c]	59.28	59.64	4.98	4.90
C_6H_5	2-Thienyl	86–87	40	$C_{12}H_{10}O_2S_2$[c]	57.58	56.90	4.04	4.51
p-C_7H_7	C_6H_5	120–121	40 (25)					
p-C_7H_7	o-$O_2NC_6H_4$	159–160	71 (5)					
p-C_7H_7	m-$O_2NC_6H_4$	146–147	75 (20)					
p-C_7H_7	p-ClC_6H_4	151–152	68 (31)					
p-C_7H_7	2,4-$Cl_2C_6H_3$	129–129.5	64	$C_{15}H_{12}Cl_2O_2S$[b]	55.05	55.42	3.70	3.70
p-C_7H_7	3,4-$Cl_2C_6H_3$	163–163.5	67	$C_{15}H_{12}Cl_2O_2S$[b]	55.05	55.58	3.70	3.69
p-C_7H_7	p-$CH_3OC_6H_4$	100–101	54 (12)					
p-C_7H_7	p-$CH_3C_6H_4$	154–155	35 (22)					
p-C_7H_7	2-Thienyl	133–134	31 (20)					
p-C_7H_7	3-Pyridyl	84–85	20	$C_{14}H_{13}NO_2S,2H_2O$[f]	56.94	56.64	5.80	6.04
p-C_7H_7	4-Pyridyl	215–216	29	$C_{14}H_{13}NO_2S,H_2O$[b]	60.61	60.44	5.45	5.85
p-C_7H_7	1-Naphthyl	111–112	14	$C_{19}H_{16}O_2S$[b]	74.01	74.09	5.23	5.30
p-C_7H_7	p-$(CH_3)_2NC_6H_4$	204–205	23[h]	$C_{17}H_{19}NO_2S$[g]	67.74	67.56	6.35	6.74

[a] The percentage yields in parentheses are those reported in the previous papers.[1,2] Recrystallized from [b] ethanol, [c] methanol, [d] isopropyl alcohol, [e] cyclohexane, [f] n-hexane and [g] acetone-water. [h] The reactants were refluxed for 90 min.

1,4-Dinitrocyclohexane in Nitrocyclohexane Still Residues

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Nitrocyclohexane was prepared by the reaction of cyclohexane with nitric acid.

During the course of an investigation which involved the identification of impurities in the nitrocyclohexane still residues, a white crystalline compound melting at 174°. was isolated. Its infrared spectrum indicated no functional groups other than the nitro group, and the carbon, hydrogen, and nitrogen contents of this compound agreed with those of a dinitrocyclohexane.

Anal. Calcd. for $C_6H_{10}N_2O_4$: C, 41.4; H, 5.75; N, 16.1. Found: C, 41.5; H, 5.90; N, 15.9.

1,1-Dinitrocyclohexane and 1,2-dinitrocyclohexane are known, having melting points of 36° and 46°, respectively. It seemed likely, therefore, that this new nitro compound was either 1,4-dinitrocyclohexane or 1,3-dinitrocyclohexane. No reference to either of these isomers was found in the literature, but the corresponding diketones were both listed in Beilstein.[1] 1,4-Cyclohexanedione has a melting point of 78° while 1,3-cyclohexanedione melts with decomposition at 104–106°.

The cyclohexanedione was prepared from the unknown dinitrocyclohexane by the Nef reaction. Two recrystallizations yielded a small amount of an off-white compound having a melting point of 74–76°. The infrared spectrum of this compound confirmed that this derivative was a ketone.

The conclusion drawn from the above data is that the compound isolated from the nitrocyclohexane still heels is 1,4-dinitrocyclohexane (having a melting point of 174°).

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(1) Beilstein, *Handbuch der organischen Chemie*, 4th ed., Vol. 7, pp. 554 and 556.