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Gas–liquid chromatography of 2-substituted 3-(5-phenyl-2-furyl)acrylonitriles

During the examination of phenylfuran derivatives, a series of 3-[5-(X-phenyl)-2-furyl]acrylonitriles were synthesized[1,2]. Compounds that contain a nitrile group conjugated to an ethylenic bond are interesting from the viewpoint of their physical and chemical properties. Nowadays compounds that contain the –C=C–C=N group in the molecule are used as UV stabilizers and are the most important group of nitrile compounds in the chemistry of light stabilizers. Derivatives of aromatic compounds[3–8] and of some heterocyclic compounds[9] have been prepared for this purpose. Phenylfuran derivatives of this type have not been examined from this point of view so far. Beside investigating their photo-stabilizing properties, we studied the development of a method suitable for their analytical gas-chromatographic (GC) determination.

According to the literature phenylfuran derivatives have not been analyzed by GC until now. We have described[10] the separation of the starting substituted 5-phenyl-2-furfural and of some further phenylfuran derivatives by adsorption chromatography on thin-layers.

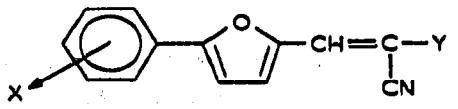
Experimental

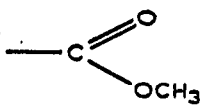
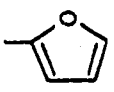
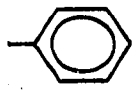
The compounds examined were prepared by condensation of the substituted 5-phenyl-2-furfural with methyl cyanoacetate, 2-furylacetonitrile or phenylacetonitrile in absolute ethanol using sodium ethoxide as catalyst[1,2].

All analyses were carried out on a Hewlett-Packard 5756 B gas chromatograph with a flame-ionization detector using a glass column (1.83 m × 2 mm I.D.) filled

TABLE I

RETENTION TIMES (t_R) FOR SUBSTITUTED 3-(5-PHENYL-2-FURYL) ACRYLONITRILES OF FORMULA:



X	Y					
						
	m.p. (°C)	*t_R (min)*	*m.p. (°C)*	*t_R (min)*	*m.p. (°C)*	*t_R (min)*
4-NO_2	264	90.08	230	151.10	221–222	—
3-NO_2	210	34.96	172–173	134.02	166–167	282.05
2-NO_2	142	88.90	147–148	78.66	115–116	194.31
4-Cl	165	75.12	157–158	45.04	147–148	77.56
3-Cl	167	44.57	98–99	45.98	98–99	98.98
2-Cl	121	43.70	110	38.35	90–91	87.32
4-Br	145	58.02	145–146	58.88	159	132.99
H	105	76.69	96–97	33.46	78–79	51.50
4-CH_3	163	43.53	85–86	37.01	121–122	74.88
4-OCH_3	160	53.29	144–145	57.09	101–102	154.72

with 10% Silicone Gum UCW 98 on Diatoport W (80–100 mesh). The nitrogen carrier gas flow rate was 40 ml/min. The column temperature was programmed from 70° to 215° at 10°/min, and the injector and detector temperatures were 270° and 280°, respectively.

Results

The results in Table I confirm that GC is a suitable method for the determination of the individual 2-substituted 3-(5-phenyl-2-furyl)acrylonitriles. This is of great importance for their practical application.

It can be seen from Table I that the substituents in position 2 of the 3-(5-phenyl-2-furyl)acrylonitriles affect the retention time. The values of the retention times (t_R) according to changes of the substituent Y decrease in the sequence phenyl > furyl > carbomethoxy.

The effect of the substituent X on the t_R values is less marked, but even so, higher values for the 4-substituted derivatives were observed. A comparison of the retention times of chloro- and nitro-substituted 3-(5-phenyl-2-furyl)acrylonitriles showed that the NO_2 group, being a stronger electron acceptor, causes an increase in the t_R values.

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