THE PURIFICATION OF MORIN

I. I. Mirenskaya, V. I. Litvinenko,
A. B. Blank, and V. V. Belikov

UDC 547.972

The morin preparations marketed by firms in the GFR (Kepec, Schuchardt, Merck, and others) and England contain considerable amounts of accompanying flavonoids, phenolic compounds, and mechanical impurities.

Methods have been described for the purification of morin by sublimation [1], repeated recrystallization [2], and ion-exchange chromatography on Amberlite IRC-50 [1]. However, these methods are characterized by low yields and do not ensure the effective purification of reasonably large amounts of the substance.

To purify morin by column chromatography we used polyamide (Kapron) of the Barnaul synthetic fiber combine with a particle diameter of 200-400 μ, which is widely used for the separation of flavonoids [3, 4].

In the first stage, the substance was freed on a column from mechanical impurities, glycosidic forms of flavonoids, and other water-soluble compounds (maclurin, etc.) by elution with ethanol. After this preliminary purification morin containing kaempferol as the main impurity was obtained.

In the second stage of purification, kaempferol and other aglycones were separated from the morin on the same column by elution with aqueous ethanol containing from 50 to 80% by volume of ethanol. The morin, which remained on the column, was desorbed with a mixture of ethyl acetate and acetic acid (1 : 1 by volume). The eluates were evaporated and the product was crystallized from 50% aqueous acetic acid. The yield of purified product was about 40%.

The process of purification and the quality of the morin obtained were checked by paper chromatography in the acetic acid–water (60 : 40 by volume) and benzene–ethyl acetate–acetic acid (69 : 29 : 2 by volume) systems. The Table 1 gives information characterizing the trade preparations of morin and the purified product.

TABLE 1

Property	Schuchardt (GFR) "Technical"	England "pure"	Kepec (GFR) "pure"	Purified product from the Kepec material
Color	Dark-brown	Brownish-yellow	Brownish-yellow	Light yellow
mp, °C	195—220 (1/2) 270 (1/2)	293—298	272—276 (2/3) 291 (1/3)	303—305
Spectrum in methanol:				
$\varepsilon \cdot 10^4$ (λ 265 nm)	—	2,86	2,40	2,68
$\varepsilon \cdot 10^4$ (λ 360 nm)	—	1,37	1,40	1,44
Content, wt. % of				
Kaempferol		5	25	5
Flavonoid glycosides		10	—	—
Other flavonoids	to 80	—	5	1
Polymeric products and mech. impurities		—	to 7	—

Khar'kov Scientific-Research Institute of Pharmaceutical Chemistry. All-Union Scientific-Research Institute for Single Crystals. Translated from Khimiya Prirodnykh Soedinenii, No. 6, pp. 767-768, November-December, 1970. Original article submitted September 4, 1970.

LITERATURE CITED

1. Q. L. Morris, T. B. Gage and S. N. Wenger, J. Amer. Chem. Soc., 73, 3340, 1951.
2. D. D. Perrin, W. L. F. Armarego, and D. R. Perrin, Purification of Laboratory Chemicals, Pergamon Press, Oxford, 1966.
3. V. I. Litvinenko, N. P. Maksyutina, and D. G. Kolesnikov, Med. Prom., No. 3, 40, 1962.
4. V. I. Litvinenko and N. A. Tyukavkina, Farm. Zh., No. 3, 15, 1969.