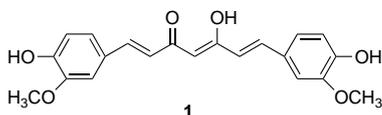


# Isolation of Curcumin from Turmeric

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The isolation of several natural products from commercially available spices has been described in organic chemistry laboratory texts (1). These experiments play an important role in exposing the students to various common techniques used in organic chemistry. We wish to add a new extraction experiment to the list of those available for laboratory instruction. We have developed a procedure for isolation of curcumin **1** from turmeric. This procedure exposes students to the common laboratory techniques of reflux, trituration, recrystallization, and column chromatography. In addition, students are introduced to a relatively uncommon purification method, namely, preparative thin layer chromatography.



Turmeric is a yellow coloring compound found in the rhizomes of *Curcuma longa*, a plant belonging to the ginger family. Turmeric is a common oriental spice that gives curry powder its yellowish color and is frequently used in Indian and Thai cooking. The active ingredient in turmeric is curcumin [(*E, E*)-1,7-bis(4-hydroxy-3-methoxyphenyl)-1,6-heptadiene-3,5-one], which is 1.5–2% by weight of the root of turmeric. Its structure was elucidated in 1910 and it was in fact the first known diarylheptanoid (2). Curcumin has also been synthesized in the laboratory (3). Diarylheptanoids, a class of compounds containing a 7-carbon chain flanked by an aromatic ring on either side, have many interesting and useful pharmacological properties. Curcumin is reported to have antitumor and anticancer properties (4). Recently, curcumin analogs were reported to inhibit HIV-1 integrase (5).

## Experimental Procedure

### General Aspects

<sup>1</sup>H and <sup>13</sup>C NMR spectra were recorded on a JEOL NMR spectrometer at 270 and 67.5 MHz, respectively. The abbreviations s, d, and br used to describe <sup>1</sup>H spectra refer to singlet, doublet, and broad, respectively.

Ground turmeric was purchased from a local Asian grocery store. Silica gel (60–230 mesh) was purchased from Selecto Scientific. Preparative TLC plates coated with silica gel (2000 micron thickness) were purchased from Analtech.

### Extraction Procedure

Twenty grams of ground turmeric in 50 mL of dichloromethane was magnetically stirred and heated at reflux for 1 h. The mixture was suction-filtered<sup>1</sup> and the filtrate was concentrated in a hot-water bath maintained at 50 °C. The reddish-yellow oily residue was triturated with 20 mL of hexane and the resulting solid (0.57 g) was collected by suction filtration.<sup>2</sup> TLC

analysis (3% methanol–97% dichloromethane) showed the presence of three components (*R*<sub>f</sub> .49, .22, .085).

- CAUTION: Exposure to dichloromethane vapors should be avoided.<sup>3</sup>

### Isolation of Curcumin by Column Chromatography

The crude material obtained after trituration with hexanes was dissolved in a minimum amount of 99% dichloromethane–1% methanol (v/v) and loaded onto a column packed with 30.0 g of silica gel. The column was eluted with the same solvent. TLC analysis of the various fractions showed the presence of three components. The fractions containing the least polar colored component were combined and the solvents were removed on a water bath to give 0.16 g of a yellow solid (mp 178–182 °C).<sup>4</sup> <sup>1</sup>H NMR (DMSO):<sup>5</sup> δ 3.90 (6H, s, OCH<sub>3</sub>), δ 6.06 (1H, s, C(OH)=CH), 6.76 (2H, d, *J* = 16 Hz, 2,6-H), 6.82 (2H, d, *J* = 8 Hz), 7.15 (2H, d, *J* = 8 Hz), 7.32 (2H, s), 7.55 (2H, d, *J* = 16 Hz, 1,7-H), 9.70 (2H, phenolic OH, br s).

<sup>13</sup>C NMR (DMSO): δ 56.21, 101.48, 111.84, 116.26, 121.62, 123.65, 126.89, 141.26, 148.54, 149.88, 183.74.<sup>6</sup>

### Isolation of Curcumin by Preparative Thin-Layer Chromatography

Two hundred milligrams of the crude solid obtained after trituration with hexanes was dissolved in 1 mL of 99% dichloromethane–1% methanol (v/v) and loaded on a preparative TLC plate (20 × 20 cm) with a Pasteur pipet. The plate was developed three times using 99% dichloromethane–1% methanol (v/v), at which point the uppermost colored band had an *R*<sub>f</sub> of .52. The silica gel from this band was scraped off and stirred for 5 min with 25 mL of 99% dichloromethane–1% methanol (v/v) solution. The mixture was gravity-filtered and the solvents removed on a water bath to give 60 mg of a yellow solid.<sup>7</sup>

### <sup>W</sup>Supplemental Material

Notes for the instructor and <sup>1</sup>H and <sup>13</sup>C NMR spectra of curcumin are available. See this issue of *JCE Online*.

### Notes

1. We found suction filtration to be more convenient than gravity filtration because turmeric tends to clog the filter paper during gravity filtration.

2. Trituration of this crude material with hot ethanol (2 × 1 mL) gave 0.22 g of a yellow-orange solid (mp: 175–178 °C). TLC analysis, however, revealed the presence of three components. TLC analysis of commercially available curcumin (from Acros Organics) also shows these same three components.

3. Turmeric is not harmful but it will stain the skin and clothing upon contact.

4. Melting point of commercially available curcumin was found to be 169–173 °C. The literature melting point of curcumin is 183 °C (6). The products obtained from the other fractions had a broad melting-point range, indicating a mixture of compounds.

5. Curcumin is not sufficiently soluble in  $\text{CDCl}_3$ ; hence the spectra were recorded in DMSO.

6.  $^{13}\text{C}$  NMR spectral data show that curcumin exists primarily in the enol form **1**, and not as the diketone. The methylene carbon of the diketone typically has a shift of  $\delta$  57 ppm while the enolic carbon [ $\text{C}=\text{C}(\text{OH})$ ] has a shift of  $\delta$  100 ppm (7).

7. Product was identified as curcumin by mp,  $^1\text{H}$  NMR, and  $^{13}\text{C}$  NMR and was found to be identical with curcumin obtained by column chromatography.

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