NOTES.

Occurrence of Kaempferol in Crocus. By J. R. Price, (Mrs.) G. M. Robinson, and Robert Robinson.

Flowers of *Crocus* species, for which we are greatly indebted to Dr. E. J. Collins, were immersed in 1% hydrochloric acid in order to extract the anthocyanins, which proved to be diglycosides of delphinidin in most cases, although the pigments of two species were found to be derived from malvidin. After 2—3 weeks the solutions in contact with *Crocus asturicus* flowers (two varieties) deposited very pale yellow crystals, which were also obtained from *Crocus speciosus* and from two or three other species in lesser relative amount.

The precipitates were collected, the aqueous acid solutions extracted with ethyl acetate, and the residue after removal of the solvent triturated with a few drops of acetic acid, leaving a yellow powder, which was added to the precipitated material. The substance crystallised readily from acetic acid in pale yellow needles, which became intensely yellow on exposure to the air. A dye-trial on cotton mordanted with aluminium, iron, and a mixture of the two gave results identical with those furnished by synthetic kaempferol. The yellow solution in concentrated sulphuric acid exhibited a green fluorescence, and this gradually changed to an intense bright blue fluorescence. The ferric reaction in alcoholic solution was also identical with that of kaempferol.

The substance, crystallised only once, had m. p. 272—274° (lit., 276—277°) and for further identification was converted into the acetate by refluxing with an excess of acetic anhydride and a few drops of pyridine for 2 hours. Most of the acetic anhydride was removed by distillation under diminished pressure, hot methyl alcohol was added, and the solution at once filtered. On cooling, colourless needles separated and these were collected, washed with methyl alcohol, and dried in a vacuum over sulphuric acid (Found : C, 58·6; H, 4·9; $CH_3{\cdot}CO$, 36·0; loss at 110° in a high vacuum, 6·5. Found in material so dried: C, 61·2; H, 4·1. $C_{23}H_{18}O_{10},CH_4O$ requires C, 59·3; H, 4·5; $4CH_3{\cdot}CO$, 35·5%. Calc. for $C_{23}H_{18}O_{10}$: C, 60·8; H, 4·0%). This specimen softened at 116°, was fluid at 120°, resolidified as the temperature rose to 140°, and then had m. p. 179—180°.

Exactly the same behaviour of tetra-acetylkaempferol crystallised from methyl alcohol has been described by Perkin and Wilkinson (J., 1902, **81**, 587), but these authors stated definitely that no loss of weight occurs during the preliminary fusion. This may well be true if the heating to 120° is not prolonged and is carried out under the atmospheric pressure. The above experiment shows clearly, however, that the phenomenon is due to solvation. Recrystallisation from ethyl alcohol, with distillation of one third of the solvent in order to remove methyl alcohol, afforded colourless needles, m. p. 182° alone or mixed with authentic synthetic tetra-acetylkaempferol (Robinson and Shinoda, J., 1925, **127**, 1980).

Only a few flowers were used in these experiments, so, although not more than 100 mg. of kaempferol were isolated, the petals of *Crocus asturicus* must be comparatively rich in flavonol content. The kaempferol is probably present as such in a dispersed form, possibly in co-pigment combination with the anthocyanin, the less favoured alternative being the occurrence of a saccharide which is hydrolysed with quite unusual ease by means of cold 1% hydrochloric acid.—DYSON PERRINS LABORATORY, OXFORD UNIVERSITY, and THE JOHN INNES HORTICULTURAL INSTITUTION. [*Received, December* 10*th*, 1937.]

The Mercuric Halides of Dimethyl Telluride. By FREDERICK CARR and T. G. PEARSON.

THE alkyl derivatives of metals in the form of their mercuric halides have been frequently used in the identification of free radicals, and for such a purpose we have synthesised the dimethyl telluride compounds by mixing cold solutions of the mercuric halides and dimethyl telluride in dry acetone. The double compounds, $TeMe_2,HgX_2$, are rapidly and quantitatively formed and, being almost insoluble, may be separated from the acetone by filtration, as far as possible out of contact with air, recrystallised from suitable solvents, and dried in a vacuum. They are almost odourless when pure, but hydrolyse rapidly in air to liberate the abominable persistent odour of dimethyl telluride. To avoid handling them, their compositions were ascertained by comparing the weights of the mercuric chloride, bromide, and iodide derivatives obtained from aliquot portions of a standard solution of dimethyl telluride in acetone by the addition of excess of the appropriate mercuric halide dissolved in acetone.

Dimethyl telluride mercuric chloride, $Te(CH_3)_2,HgCl_2$, formed colourless, microscopic, square and hexagonal plates, m. p. 179° (decomp.). *Dimethyl telluride mercuric bromide*. $Te(CH_3)_2,HgBr_2$, formed colourless or very pale cream-coloured, minute crystals, which appear as hexagonal rhombs or squares under the microscope, showing strong polarisation colours; m. p. 160—161° (decomp.). It separated from alcohol or benzene in fine needles, m. p. 158°, slightly soluble in methyl iodide, chloroform, and ether. *Dimethyl telluride mercuric iodide*, $Te(CH_3)_2,HgI_2$, formed canary-yellow, vitreous, columnar crystals, showing strong polarisation; m. p. 107° (slight decomp.); from alcohol or benzene, it separated in paler golden-yellow needles, m. p. 108° (slight decomp.). All three compounds were recrystallised from acetone.—IMPERIAL COLLEGE, LONDON, S.W. 7. [*Received, January* 25*th*, 1938.]