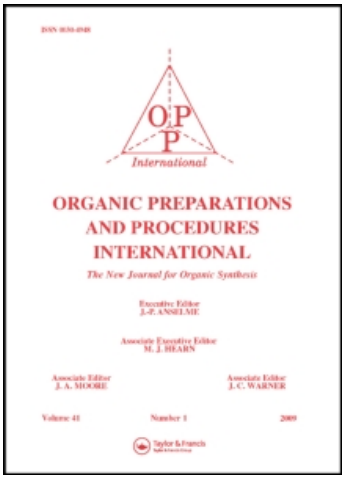

Organic Preparations and Procedures International
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t902189982

SYNTHESIS OF ALKALI AND ALKALINE-EARTH METAL ADDUCTS OF SUCROSE

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To cite this Article Poncini, Laurence and Chand, Vijaya(1983) 'SYNTHESIS OF ALKALI AND ALKALINE-EARTH METAL ADDUCTS OF SUCROSE', Organic Preparations and Procedures International, 15: 1, 69 — 70
To link to this Article: DOI: 10.1080/00304948309355434
URL: http://dx.doi.org/10.1080/00304948309355434

SYNTHESIS OF ALKALI AND ALKALINE-EARTH METAL ADDUCTS OF SUCROSE

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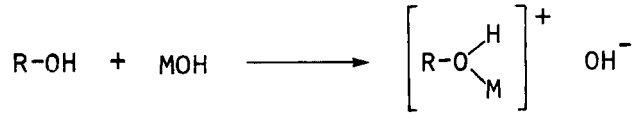
Alkali and alkaline-earth metal adducts of sucrose have been known since last century.[1] However, very few procedures have appeared whereby these adducts could be isolated free from contamination by the starting materials. The procedures reported in the literature involve either aqueous,[2] alcoholic[3] or liquid ammonia[4] systems. All these procedures employ sucrose in excess and the resulting metal adducts are generally obtained in low yields and are relatively impure. In our hands, these aforementioned methods gave yields in the range of 5-25% for the adducts reported here. However, if an excess of the metal salt (in this case the metal hydroxide) is used, very high yields of the adducts are obtained. For example, for the sodium and lithium adducts the yields were quantitative. The products are exceedingly deliquescent and appear to be simple addition compounds whose formation may be depicted as follows.



EXPERIMENTAL

All reagents were analytical grade and dry distilled solvents were used. Ether was sodium-dried. All reactions were carried out in a glove bag under an atmosphere of dry nitrogen. Analyses of the complexes were carried out as previously reported,[3] and found to be in agreement with results reported earlier for 1:1 metal complexes.

To a solution of sucrose, (0.5 g, 1.46 mM) in water (0.5 ml), was added dropwise with stirring to an ethanolic MOH (M = Na, K) solution (3.5 ml, 1 M). The white precipitate which formed (1-2 hrs) turned to a viscous semi-opaque liquid phase upon standing.[5] The upper layer was decanted

cautiously and the viscous liquid phase washed well with anhydrous ethanol (2 x 4 ml), then ether. The product, a fine white amorphous glass, was obtained after drying under vacuum over P_2O_5 at 2 mm and 25° for 20 hrs. The yields for the sodium and potassium adducts ([M(sucrose)] $OH \cdot H_2O$) were 97-99% and 93-95% respectively. (Found: Na, 5.8; sucrose, 86.1. $C_{12}H_{25}NaO_{13}$ requires Na, 5.7; sucrose 85.7%; found: K, 9.6; sucrose, 83.0. $C_{12}H_{25}KO_{13}$ requires K, 9.4; sucrose 82.0%).

The procedure was identical for the lithium hydroxide adduct [Li(sucrose)]$OH \cdot 2H_2O$, except that a saturated alcoholic solution of the hydroxide was used. Yield 95-99%. (Found: Li, 1.8; sucrose, 86.1. $C_{12}H_{27}LiO_{14}$ requires Li, 1.8; sucrose 85.1%).

For the calcium adduct, $[Ca(sucrose)_2](OH_2) \cdot x\ H_2O$, a cold (2°) saturated aqueous solution of the metal hydroxide was employed. Because of the extreme deliquescent nature of this complex (over fresh P_2O_5 it remained moist after 56 hrs. drying and could only be dried by freeze drying) water analysis gave x as 1-3. Yield 80% (as anhydrous adduct). (Found: Ca, 5.3; sucrose, 99.1. $C_{24}H_{48}CaO_{25}$ requires Ca, 5.3; sucrose 89.8%.

Acknowledgements.- Financial assistance by the USPRC Grant No. R-158 is gratefully acknowledged by one of us (LP). Our thanks are due to Mr. Vas Deo for construction of the glove bag facilities and to Dr. F. Wimmer for assistance with the manuscript.

REFERENCES

1. L. Poncini, Int. Sugar J., 81, 36 (1979).

2. N. Roy and A. K. Mitra, Carbohydr. Res., 24, 175 (1972); N. Roy and A. K. Mitra, ibid., 24, 180 (1972).

3. L. Poncini, Ind. J. Chem., 19, 1035 (1980); J. A. Rendleman, J. Org. Chem., 31, 1845 (1966).

4. P. C. Arni, W. A. P. Black, E. T. Dewar, J. C. Patterson and D. Rutherford, J. Appl. Chem., 9, 186 (1959).

5. Centrifugation which is employed by some workers,[2] resulted in compaction of the product thus preventing successful washing to free it of unreacted reagents.