

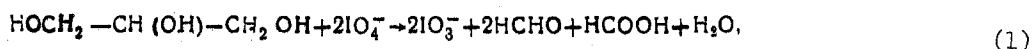
## BRIEF COMMUNICATIONS

### TITRIMETRIC DETERMINATION OF GLYCEROL AND OF GLYCEROL 1-PHOSPHATE USING PERIODATE OXIDATION

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To determine glycerol (I) in methods using periodate oxidation the HCOOH [1, 2] or HCHO [3] formed in this process have been determined. A method of direct enthalpimetry with a double injection of periodate has also been proposed [4]. Compound (I) reacts with periodate according to the equation



and glycerol 1-phosphate (II) does so in accordance with the equation



Methods based on masking the excess of periodate and on the synproportionation of the iodate-iodide and titration of the triiodide formed with sodium thiosulfate (so-called amplification methods) are promising for the determination of  $\alpha$ -glycol,  $\alpha$ -hydroxyamino, thioether, and other functional groupings capable of being stoichiometrically oxidized by periodate [5]. No such method has been described in the literature [5, 6] for determining (I) and (II). We have investigated the possibility of determining (I) and (II) by this method.

We used (I) of ch.d.a. ["pure for analysis"] grade and the hexahydrate of the disodium salt of (II) of purum grade (Fluka AG) with the composition  $\text{HOCH}_2-\text{CH}(\text{OH})-\text{CH}_2\text{OPO}_3\text{Na}_2 \cdot 6\text{H}_2\text{O}$  ( $M = 324.13$  g/mole).

Reactions (1) and (2) proceed to completion in 5-30 min at room temperature, depending on the excess of periodate. As the oxidants we used a 0.025 M solution of  $\text{KIO}_4$  in 0.1 N  $\text{H}_2\text{SO}_4$  and a 0.025 M solution of  $\text{NaIO}_4$  in water. An increase in the time of oxidation to several hours under these conditions did not disturb the stoichiometry of reactions (I) and (II), i.e., no overconsumption of oxidant was observed. The presence of such well-defined linear sections on the curves of the formation of iodate as a function of the type of oxidation as corresponded strictly to the stoichiometric occurrence of reaction (1) in the case of (I) and of reaction (2) in the case of (II) simplified the performance of the operations and ensured their reliability. One mole of (I) reacts with 2 moles of periodate with the formation of 2 moles of iodate, which is equivalent to 6 moles of triiodide the titration of which consumes 12 g-eq of sodium thiosulfate [ $1(\text{I}) \equiv 2\text{IO}_4^- \equiv 2\text{IO}_3^- \equiv 6\text{I}_3^- \equiv 12\text{S}_2\text{O}_3^{2-}$ ]. One mole of (II) reacts with 1 mole of periodate to form 1 mole of iodate, which is equivalent to 3 moles of triiodide the titration of which consumes 6 g-eq of sodium thiosulfate [ $(\text{II}) \equiv \text{IO}_4^- \equiv \text{IO}_3^- \equiv 3\text{I}_3^- \equiv 6\text{S}_2\text{O}_3^{2-}$ ].

For the determination, an accurately weighed sample of (I) (4-10 mg) or of (II) (20-30 mg) is oxidized with 10 ml of a 0.025 M solution of  $\text{KIO}_4$  in 0.1 N  $\text{H}_2\text{SO}_4$  or with 10 ml of a 0.025 M solution of  $\text{NaIO}_4$  in water for 30 min-1 h at room temperature. Then 10-20 ml of 6 M  $\text{CH}_3\text{COOH}$  and 5 ml of 1 M sodium or ammonium molybdate are added, the contents of the titration vessel are carefully stirred, 5 ml of 20% potassium iodide solution is added, and with stirring, titration is carried out with standardized 0.025 N sodium thiosulfate solution in the presence of starch solution as indicator, added towards the end of titration, until the disappearance of the blue-pink coloration of the solution. A blank experiment [without the addition of (I) or (II)] is carried out in parallel.

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The amount of (I) or (II) (X, %) is calculated from the formula

$$X = \frac{(V_1 - V_2)MN}{6ng} 100,$$

where  $V_1$  is the volume of standardized solution of sodium thiosulfate consumed in titration in the analysis, ml;  $V_2$  is the volume of standardized sodium thiosulfate solution consumed in titration in the blank experiment, ml;  $M$  is the molecular mass of the compound being determined, g/mole;  $N$  is the concentration of the standardized sodium thiosulfate solution, g-eq./liter; and  $n$  is the number of moles of periodate reacting with one mole of the compound being determined ( $n = 2$  in the case of (I) and  $n = 1$  in the case of (II)).

The results obtained in the determination of (I) and (II) are given below:

Compound	Time of oxidation, min	g, mg	n	X, %	$S_{\bar{x}}$ , %
I	30	4.665	15	89.0	0.3
	40	5.216	10	89.2	0.2
	50	8.220	12	89.1	0.2
	60	9.684	10	89.2	0.4
II	30	30.24	15	97.8	0.2
	40	30.28	12	97.7	0.3
	50	32.12	10	97.8	0.5
	60	34.08	12	97.7	0.4

The method is distinguished by simplicity of performance, high accuracy, and sensitivity. The relative standard deviation  $S_{\bar{x}}$  does not exceed 0.3-0.5%. The method is convenient for standardization and for use in factory laboratory investigations.

#### LITERATURE CITED

1. L. Hartman, Chem. Ind. (London), No. 44, 1407 (1955).
2. R. Mormont, A. C. Gillet, Jr., and A. Heinerth, Talanta, 16, 701 (1969).
3. M. Lambert and A. C. Neish, Can. J. Res., Sect. B, 28, 83 (1950).
4. R. Volf, M. Štastny, J. Vulterin, and M. Waldman, Chem. Prům., 26, 648 (1976).
5. V. Ya. Zakharans, Izv. Akad. Nauk Latv. SSR, Ser. Khim. No. 1, 3 (1982).
6. M. R. F. Ashworth, Analytical Methods for Glycerol, Academic Press, London (1979), p. 258.
7. V. Ya. Zakharans, V. V. Elkin, Yu. R. Laurs, and V. É. Égert, Methods for the Preparation and Analysis of Biochemical Materials. Abstracts of Lectures at the IVth All-Union Conference [in Russian], Riga, Part I (1982), p. 80.