

Netherlands Organisation for the Advancement of Pure Research (Z.W.O.).

B. J. MIJNHEER*
E. VAN DEN HAUTEN-ZUIDEMA

*Institute for Nuclear Physics
Research (IKO)
Amsterdam, Holland*

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* Present address: Central Bureau for Nuclear Measurements, Geel, Belgium.

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On the Preparation of Dichlorodiphenyl-dichloroethane-¹³¹I (DDD-¹³¹I)

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THE COMMERCIALY available insecticide dichlorodiphenyldichloroethane (DDD) is a mixture of several isomers and has been described as an adrenocorticolytic agent.⁽¹⁾ Technical grade DDD labeled with ¹³¹I has been proposed for adrenal scanning^(2,3) and during some time we have been preparing it for that purpose by means of ¹³¹I-iodine monochloride in ether at room temperature. The yield so far had been of 30–40 per cent, after purification by washing with a solution of sodium thiosulphate and potassium iodide. Recently, in a series of 10 preparations an improved labeling procedure increased the yield to 95.6 ± 2.3 per cent. The procedure is as follows:

In a vial containing in 10–20 μ l the desired activity of Na¹³¹I carrier-free solution, also free of reducing agents, 32 mg (0.1 mMol) of DDD in 3 ml of ethyl alcohol are added, followed by a drop of concentrated hydrochloric acid. The vial is sealed and heated in boiling water for 3 hr. After cooling the

product is ready for oral administration. The alk can be evaporated and substituted by a diff solvent, if desired.

Although the structure of the organic ic produced according to the procedure described a has not been established, it must be mentioned when compared with the product labeled with ic monochloride it proved to be equally effective \pm adrenal scanning agent.

The content of free radioiodine can be determ by means of ascending chromatography in What paper No. 1 with 0.9% NaCl solution for (R_f DDD 0.0–0.05; R_f iodide 0.85–0.90).

The following table illustrates the stability o substance when stored at room temperature:

Days after labeling	Free iodide (mean of three batches) (%)
0	3.1
7	3.7
14	4.5
22	4.3
30	5.0

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J. ALVAREZ CERVERA
B. ESPEJEL DE VEITE
G. SKROMNE-KADLUBIK

*Centro Hospitalario "20 de Noviembre," ISSSTE
Servicio de Medicina Nuclear
F. Cuevas y Coyoacán, México 12, D.F.
México*

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Labelling of Elementary Iodine with Radioiodine by Heterogeneous Isotope Exchange

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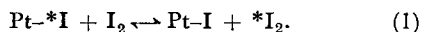
ELEMENTARY iodine labelled with ¹³¹I or ¹²⁵I, whi frequently required in tracer experiments and fo production of radioiodine-labelled compounds, be produced either by oxidizing radioiodide with IO₃⁻ or other suitable oxidizing agents, c electrochemical methods. Most of these proces suffer from the drawback of contaminating

labelled product with oxidizing agents, and in addition acidification of the reaction mixture is also necessary to adjust the pH to an appropriate value for the oxidation reaction.

For homogeneous isotopic exchange studies, on the other hand, labelled iodine solution is frequently prepared by shaking an organic solvent containing elementary iodine with an aqueous solution of radioiodide ions,⁽¹⁾ a procedure which usually necessitates the removal of water as well as radioiodide ions transferred into the organic phase.

The method described here is based on heterogeneous isotopic exchange between iodine adsorbed on platinum and elementary iodine in a solution of suitable composition. The advantages of the method are, on the one hand that the labelled iodine solution is not contaminated by either oxidizing agent or iodide ions, and on the other, that the choice of pH interval and solvent is much wider than is the case with oxidation reactions.

As established in our earlier work,^(2,3) radioiodine adsorbed on an energetically non-uniform platinum surface exchanges with elementary iodine in solution according to the reaction



Where the asterisk denotes radioactive iodine atoms. The kinetics of this exchange process may be described by the expression

$$\ln(1 - F) = -(A)^{\alpha-1} \cdot (B)^{\beta} \cdot k_0 \cdot \ln t + \ln(1 - F)_{t=1} \quad (2)$$

where $(1 - F)$ denotes the fraction exchanged (i.e. the ratio of the radio-iodine activities found on the wire exchanged at times t and 0), and (A) and (B) represent the concentrations of iodine in the iodine monolayer on the wire surface and in the bulk of the solution, k_0 is a constant and α and β are constants which are normally unity. Thus the yield in labelling the elementary iodine, which is proportional to F , will depend on the time, on the concentration of iodine in solution and on the temperature, but will be

independent of A , i.e. the amount of adsorbed and of the volume of solution containing the iodine to be labelled.

The labelling of elementary iodine in different solvents was carried out in such a way that the adsorbing platinum wire, the surface of which was reduced by cathodic polarization, was placed in a sulphuric acid solution of ^{131}I for a few hours to the formation of a submonolayer of iodine atoms. After adsorption equilibrium had been attained the adsorbing wire was rinsed with redistilled water, at room temperature and put into a solution containing the elementary iodine to be labelled. The labelling yields are shown in Table 1. It is evident from this that the labelling efficiency can range from 51 to 96.5 per cent within a 100 min, depending on the solvent used.

The extremely rapid exchange observed in pyridine can be attributed to the formation of complex ions.⁽⁶⁾ In aqueous solution I_3^- was also found to exchange more rapidly than either I^- or I_2 .^(2,3)

It should be noted that heterogeneous isotopic exchange with the participation of adsorbed iodine carried by noble metal can be utilized also for labelling organic compounds.

G. TÓTH
J. MILLER

*Institute of Isotopes of the
Hungarian Academy of Sciences
Budapest, Hungary*

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TABLE 1. Labelling yields (percentage of the activity initially adsorbed on the wire transferred to the iodine solution)

Solvent exchange time (min)	H ₂ O	0.1 M H ₂ SO ₄	CCl ₄	C ₆ H ₆	C ₂ H ₅ OH	C ₅ H ₅ N
1	30	26	16	16	20	89
5	38	36	21	23	28	94
10	41	40	25	27	35	95
20	43	51	27	30	45	96
50	49	62	34	35	54	97
100	51	64	37	37	55	97
150	52	64	38	40	56	98
200	54		40	42	57	98