Synthesis, antibacterial and surface activity of 1,2,4-triazole derivatives

By R. El-Sayed

Chemistry Department, Faculty of Science, Benha University, Benha – Egypt.
ref_at@hotmail.com

SUMMARY

Synthesis, antibacterial and surface activity of 1,2,4-triazole derivatives

The use of sodium 1-(4-amino-5-mercapto-4H-[1,2,4]triazol-3-yl) and heptadecane-1-sulfonate (1) as new precursors to synthesize some important biologically active heterocycles which constitute an important class of organic compounds with diverse biological activities, including antiparasitic, analgesic, antibacterial and anti-inflammatory activities (Tayseer et al., 2002; Cansiz et al., 2004; Katica et al., 2001; Li-Xue et al., 2002; Hovsepian., 2004; Wasfy., 2003). A considerable effort has been made in recent years to further develop the synthesis of these nucleus. (Boskha et al., 2002; Kumar et al., 2003; Oganisyan et al., 2004). These derivatives are very attractive heterocyclic systems due to their extensive use in medicine, agriculture and industry (Xin-Ping et al., 2000). In light of the above facts and with a view to obtain new biologically active agents I was encouraged to synthesize a new series of 1,2,4-triazole derivatives bearing (long alkyl chain with sulfonic acid hydrophilic center) in a single molecular framework likely to constitute new biologically active anionic surface active agent hopefully possessing good surface properties and expected to have biological activities.

2. MATERIAL AND METHODS

Melting points are uncorrected. IR spectra in KBr were measured on a Pye-Uncam SP-1000 infrared spectrophotometer on a KBr disk or nujol. The 1H NMR spectra were obtained on a Varian EM-390-60 MHz spectrometer in DMSO as the solvent. Tetramethylsilane TMS served as an internal reference and chemical shifts are expressed as δ (ppm). Mass spectra were recorded on a G-C/Ms Finning-MAT. Microanalyses were performed by the Microanalytical Unit at Cairo University. All the compounds gave satisfactory elemental analyses. Thin layer chromatography (TLC) was carried out on silica gel (MN-Kieselgel G., 0.2 mm thickness) and the plates were scanned under 254 nm ultraviolet light. Antimicrobial and antifungal activity tests were carried out at the microbiology Lab., Faculty of Science, Zagazig University, Benha-branch, Egypt.

2.1. Sodium 1-hydrazinocarbonyl-heptadecane-1-sulfonate (1)

The sodium salt of α-sulphonated of stearic acid hydrazide 1 was prepared according to the method in the literature (Eissa., 2002). Yield, 70 %,
2.2. Sodium 1-(4-amin-5-mercapto-4H-[1,2,4]-triazol-3-yl)heptadecane-1-sulfonate (2)

The acid hydrizide 1 (0.01 mol) was added to absolute alcohol (50 mL) containing KOH (1.6 g) at room temperature. Carbon disulphide was added (2.3 g, 0.013 mol) and the mixture stirred at room temperature for 10 h. The mixture was diluted with ether (30 mL) and stirred for a further 1h. The potassium salt was used for the next stage without further purification. Hydrazine hydrate (99%) (0.02 mol) was gradually added to the above potassium salt (0.01 mol) dissolved in water (20 mL) with stirring and the mixture was refluxed gently for 3 hr during which hydrogen sulphide evolved and the color of the reaction mixture changed to a dark green color, It was then cooled to 5 °C and acidified with conc. HCl to pH 1.00. A yellow solid separated out which was filtered, washed with water and crystallized from ethanol to make the triazole 2. Yield, 70 %, mp = 88-90 °C. IR: ν(C=O) = 3229 (NH), 2921-2850 (CH in alkyl chain), 1600 (C=N). 1H NMR (CDCl3): δ = 0.95 (t, J = 7.4 Hz, 3 H, terminal CH3), 1.29-1.33 (m, 30 H, CH2 in alkyl chain), 2.35 (s, J = 4.0Hz, 1H, CH-SO3Na). 13C NMR (CDCl3): δ = 34.2, 29.0, 21.9, 21.5, 21.4, 21.2, 17.1, 16.9. MS: m/z M++1 = 499 (40). Anal. Calcd for C26H41N4NaO3S2 (544.76): C, 57.33; H, 7.86; N, 10.28; S, 11.84. Found C, 57.34; H, 7.83; N, 10.37; S, 11.87 %.

2.4. General procedure for preparation of 4a and 4b

To a solution of triazole 2 (0.01 mol) in dry pyridine (25 mL), the acid chlorides (0.01 mol), namely, acetyl chloride and/or benzoyl chloride were added in drops. The reaction mixture was stirred at room temperature for 45 min and then heated for 2 h in a steam bath. It was then poured onto crushed ice. The solid products obtained by filtration were crystallized from the appropriate solvent to give 4a and 4b.

2.4.1. Sodium 1-[(6-methyl-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-3-yl)heptadecane-1-sulfonate (4a)

Yield, 65%. mp = 64-66 °C. IR: ν(C=O) = 3229-2920-2850 (CH in alkyl chain), and 1589 (C=N). 1H NMR (CDCl3): δ = 0.90 (t, J = 7.0 Hz, 3 H, terminal CH3), 1.29-1.33 (m, 30, H, CH2 in alkyl chain), 2.35 (s, J = 6.4 Hz, 3H, CH3), 4.26 (t, J = 4.8 Hz, 1 H, CH-SO3Na). MS: m/z (%) M++1 = 480 (54). Anal. Calcd for C31H39N4NaO3S2: C, 52.48; H, 7.76; N, 11.66; S, 13.34. Found C, 52.44; H, 7.80; N, 11.61; S, 13.38 %.
2.5.3. Sodium 1-[5-mercapto-4-[4-methoxyphenyl(phenyl)-amino]-4H-[1,2,4]triazol-3-yl] heptadecane-1-sulfonate (5c). Prepared from p-methoxybenzaldehyde. Yield 80%. mp = 115-117 °C. IR: ν/cm\(^{-1}\) = 2920-2850 (CH in alkyl chain), 1605 (C=N) and 2557 (SH). The \(^1\)H NMR (CDCl\(_3\)): δ = 0.90 (t, 3 H, terminal CH\(_3\)), 1.29-1.34 (m, 30 H, CH\(_3\) in alkyl chain), 5.73 (s, 3H, OCH\(_2\)), 4.22 (t, 1H, CH-\(\text{SO}_3\)Na), 6.9-7.4 (m, 4H, ArH). Anal. Calcd for C\(_{29}\)H\(_{43}\)N\(_4\)NaO\(_4\)S\(_3\) (653.31): C, 53.68; H, 6.99; N, 9.12; S, 15.48 %. Found C, 53.72; H, 7.11; N, 9.05; S, 15.54. Yield, 76 %. mp = 73-75 °C. IR: ν/cm\(^{-1}\) = 3250 (NH), 2921-2851 (CH in alkyl chain), 1610 (C=N), 1170 and 944 (SO\(_2\)) and 2455 (SH). \(^1\)H NMR (CDCl\(_3\)): δ = 0.95 (t, 3H, terminal CH\(_3\)), 1.27-1.31 (m, 30 H, CH\(_2\) in alkyl chain), 2.0 (s, 1H, CH=Ph) and 6.3-7.5 (m, 4H, ArH). Anal. Calcd for C\(_{27}\)H\(_{39}\)N\(_4\)NaO\(_5\)S\(_2\) (586.75): C, 55.27; H, 6.70; N, 9.55; S, 10.93. Found C, 55.21; H, 6.76; N, 9.61; S, 10.86 %.

2.8. Sodium [5-mercapto-4-(toluene-4-sulfonylamino)-4H-[1,2,4]triazol-3-yl] heptadecane-1-sulfonate (6). A mixture of triazole 2 (0.01 mol) and phthalic anhydride (0.01 mol) in butanol (20 mL) was heated under reflux for 4 h. Then the solution was concentrated. A solid product 7 was obtained by filtration which was crystallized from ethanol. Yield 55 %. mp = 91-93 °C. IR: ν/cm\(^{-1}\) = 2920-2850 (CH in alkyl chain), 3047 (CH aromatic), 2461 (SH), 1695,1988 (C=O) and 1599 (C=N). \(^1\)H NMR (CDCl\(_3\)): δ = 0.83 (t, 3 H, terminal CH\(_3\)), 1.29-1.33 (m, 30 H, CH\(_3\) in alkyl chain), 3.01 (s, 1H, SH), 4.27 (t, 1H, CH-\(\text{SO}_3\)Na) and 8.2-8.7 (m, 4H, ArH). Anal. Calcd for C\(_{26}\)H\(_{43}\)N\(_4\)NaO\(_5\)S\(_2\) (567.73): C, 55.27; H, 6.70; N, 9.55; S, 10.93. Found C, 55.21; H, 6.76; N, 9.61; S, 10.86 %.
mol) of anhydrous sodium acetate was added. The reaction mixture was heated for an additional 1h, then cooled and poured onto ice-cold water. The solid product was crystallized from ethanol to give 13. Yield 85 %; mp = 108 °C. IR: v/cm⁻¹ = 3250, 3246 (NH), 2546 (SH) and 1376 (C=S). 1H NMR (CDCl₃); δ = 0.90 (t, J=7.6 Hz, 3 H, terminal CH₃), 1.29-1.34 (m, 30 H, CH₂ in alkyl chain), 2.0 . . . 4.0 (s, 2H, 2NH), 13.01 (s, 1H, SH), 4.25 (t, 1H, CH-SO₃Na) and 6.41-7.1 (m, 5 H, ArH). Anal. Calcd for C₂₆H₄₀N₅NaO₃S₂ (557.76): C, 52.77; H, 7.15; N, 11.83; S, 11.57. Found C, 52.71; H, 7.08; N, 11.77; S, 16.31 %. 2.14. Sodium 1-(6-phenylamino-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-3-yl) heptadecane-1-sulfonate (14)

Method A. A mixture of phenyl isothiocyanate (0.1 mol), triazole 2 (0.1 mol) and powdered sodium hydroxide (0.8 g) in DMF (25 mL) was refluxed for 4h. The reaction mixture was poured onto dilute acetic acid (5%, 15 mL). The precipitated product was filtered and crystallized from ethanol to give 14. Yield 78 %; mp = 85-87 °C. IR; v/cm⁻¹ = 3270 (NH) and 1590 (C=N). 1H NMR (CDCl₃); δ = 0.95 (t, 3 H, terminal CH₃), 1.28-1.33 (m, 30 H, CH₂ in alkyl chain), 4.24 (t, 1H, CH-SO₃Na), 4.0 (s, s, NH, exchangeable) and 6.46-7.2 (m, 5 H, ArH). Anal. Calcd for C₂₆H₄₂N₅NaO₃S₃ (591.84): C, 52.77; H, 7.23; N, 12.56; S, 11.50. Found C, 56.05; H, 7.28; N, 12.62; S, 11.57 %.

Method B. Thiosemicarbazide derivative 13 was fused in an oil-bath above its melting point. The product was cooled, treated with ethyl acetate, and filtered. The solid product was crystallized from ethanol to give 14.

2.15. Biological activity

The antibacterial activities of some synthesized compounds were determined in vitro using hole plate and filter paper disc methods (Elinta et al., 2003) against various pathogenic bacteria such as Gram -ve bacteria (Bacillus subtilis, staphylococcus aureus) and gram -ve bacteria (Escher-ichia coli) in addition to fungi as Aspergillus niger was used. The tested compounds were dissolved in 10 % acetonitrile (v/v), different concentrations were chosen (125, 250, 500 µg/ml). A qualitative screen was performed on all compounds while quantitative assays were done on active compounds only.

2.16. Surface active properties

2.16.1. Surface and interfacial tension. Both surface and interfacial tension were measured using Du-Nouy tensiometer (Findly., 1963) (Kruss, Type 8451) with 0.1 wt % aqueous solution at room temperature (25 °C).

2.16.2. Kraft point. The Kraft point of the prepared anionic surfactants was measured as the temperature at which 1.0 % solution becomes clear under gradual heating (Wiel et al., 1963).

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2.16.3. Wetting time. The wetting time was determined by immersing a sample of cotton fabric in 1.0 wt % aqueous solution of surfactants. (Masuyama et al., 1978).

2.16.4. Foaming properties. The foaming properties were measured according to Somaya et al. (1998). In this procedure a 25 ml solution (1.0 wt %) was shaken vigorously for 10 seconds in a 100 ml glass stopper, graduated cylinder, at 25 °C. The solution was allowed to stand for 30 seconds, and the foam height was measured.

2.16.5. Emulsification stability. The emulsion was prepared from 10 ml of a 20 m mol aqueous solution of surfactant and 5 ml of toluene at 40 °C. The emulsifying property was determined by the time it took for an aqueous volume separating from the emulsion layer to reach 9 ml, counting from the moment of cession shaking (Takeshi, 1970).

2.16.6. Stability towards hydrolysis. A mixture of 10 m mol surfactant and 10 ml 0.05 N NaOH were placed in a thermostat at 40 °C. The time it takes a sample solution to be clouded as a result of hydrolysis shows the stability of the surfactant to hydrolysis (El-Sukkary et al., 1987).

2.17. Biodegradability

Samples taken daily or more frequently were filtered through Wattmann filter paper number (1) before measuring the surface tension. Surface tension measurements were made periodically each day, on each sample during the degradation test (Eter et al., 1974). Biodegradation percent (D) for each sample was calculated using the following equation: 

\[ D = \left( \frac{\gamma_{t} - \gamma_{o}}{\gamma_{b t} - \gamma_{o}} \right) \times 100 \]

where \( \gamma_{t} = \) surface tension at time t, \( \gamma_{o} = \) surface tension at zero time, \( \gamma_{b t} = \) surface tension of blank experiment at time t (without samples).

3. RESULTS AND DISCUSSION

3.1. Synthesis

In the present work, the reaction of sodium salt of \( \alpha \)-sulphonated fatty acid hydrazide 1 with carbon disulphide in ethanolic potassium hydroxide gave the potassium salt of the corresponding dithiocarbazate in quantitative yield. Furthermore, the potassium salts upon reaction with hydrazine hydrate (99 %) gave sodium 1-(4-amino-5-mercapto-4H-[1,2,4]triazol-3-yl)-heptadecane-1-sulfonate (2), which required a starting material (Scheme 1).

The triazole 2 treated with carbon disulfide in pyridine afforded sodium 1-(6-thioxo-5,6-dihydro-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazol-3-yl)heptadecane-1-sulfonate (3). Compounds 4a and 4b were obtained through the reaction of triazole 2 with acid chlorides (acetyl chloride and benzoyl chloride). Condensation of triazole 2 with aromatic aldehydes (benzaldehyde, \( p \)-chloro-benzaldehyde...
and p-methoxybenzaldehyde) in refluxing ethanol containing catalytic amounts of piperidine furnished the Schiff bases 5a-c. Also, the reactivity of 5a-c towards other reagents has been investigated to obtain newer biologically active heterocycles system. Thus, the reaction of Schiff base 5a-c with thioglycollic acid gave 6a-c. The reaction of triazole 2 with phthalic anhydride in butanol gave 7. The addition of 4-methylbenzene sulfonylechloride to triazole 2 gave 8. On the other hand, the reaction of triazole 2 with one equivalent of chloroacetalddehyde in refluxing ethanol produced sodium 1-(7H-[1,2,4]triazolo[3,4-b][1,3,4]thiadiazin-3-yl)-heptadec-ane-1-sulfonate (9). As anticipated, the condensation of 2 with equimolar amounts of phenacyl bromides in the presence of potassium carbonate in absolute ethanol resulted in cyclocondensation to give the corresponding sodium-1-[6-phenyl-7H-[1,2,4]triazolo [3,4-b][1,3,4]thiadiazin-3-yl]-heptadecane -1-sulfonate (10). The fusion of triazole 2 with urea gave 11 in good yield (80%). In the present investigation, the condensation of 2 with equimolar amounts of chloroacetyl chloride furnished 12. In view of the known antifungal and antiviral characteristics (Walid et al., 2002; Stankovsk et al., 2000) inherent in substituted thiosemicarbazide derivatives, the synthesis of new compounds incorporating such a group was undertaken. Thus, the reaction of triazole 2 with phenyl isothiocyanate in dimethylformamide at room temperature gave 13. On the other hand, the reaction of triazole 2 with phenyl isothiocyanate by refluxing in dimethylformamide resulted in the corresponding thiosemicarbazide derivative 14 which was also obtained by heating 13 above its melting point. (Scheme 2).

3.2. Biological activity

The data indicated that most of the synthesized compounds have remarkable activity and that the tested compounds 2, 6a-c, 10, 9, 12 and 13 were highly active towards the selected pathogens, while the compounds 3, 4a, 4b, 7a-c, 8, and 11 were moderately active towards the different strains of bacteria and fungi as compared with the standard. (Table I).

3.3. Surface active properties

The investigation of the surface active properties (surface and interfacial tension, Kraft point, wetting time, foaming height, emulsion stability, and stability against hydrolysis) of 1,2,4-triazole derivatives bearing (long alkyl chain with sulfonic acid hydrophilic center) was carried out at concentration 1 wt % and 20 °C in distilled water. The results are represented in Table II. The biodegradability properties were also determined and are represented in Table III. These products are interesting because these types of anionic surfactants are not common ones. Therefore the traditional procedure was used to follow up the properties.

3.3.1. Surface and interfacial tension. The results indicate that all the synthesized products have pronounced surface activity. Where the values
### Table I
The antimicrobial activity of the tested compounds

| Tested sample | B. subtilis | S. aureus | E. coli | A. niger |
|---------------|------------|-----------|---------|---------|
|               | MIC        | MIC       | MIC     | MIC     |
| 2             | +++ 500    | + 250     | ++ 125  | ++ 500  |
| 3             | ++ 250     | +++ 250   | ++ 500  | + 250   |
| 4a            | + 125      | + 250     | + 250   | + 500   |
| 4b            | ++ 125     | + 250     | + 250   | + 500   |
| 5a            | ++ 250     | ++ 250    | ++ 125  | ++ 125  |
| 5b            | ++ 250     | + 125     | + 250   | + 250   |
| 5c            | ++ 250     | + 125     | + 250   | + 250   |
| 6a            | +++ 250    | + 125     | + 250   | + 250   |
| 6b            | +++ 250    | + 125     | + 250   | +++ 250 |
| 6c            | +++ 250    | + 125     | + 250   | + 250   |
| 7             | + 250      | + 125     | + 250   | + 250   |
| 8             | ++ 250     | + 125     | + 250   | + 250   |
| 9             | +++ 500    | ++ 250    | ++ 125  | ++ 250  |
| 10            | +++ 250    | +++ 125   | ++ 250  | ++ 500  |
| 11            | ++ 125     | + 250     | ++ 125  | ++ 250  |
| 12            | ++ 125     | + 250     | + 500   | + 250   |
| 13            | +++ 250    | ++ 250    | + 250   | + 500   |
| 14            | +++ 250    | +++ 250   | + 250   | + 500   |

A = antimicrobial activity of tested compounds
MIC = minimum inhibitory concentration
+ > 5 mm slightly active.
++ > 7 mm moderately active.
+++ > 9 mm highly active.

### Table II
Surface properties of the synthesized compounds

| Compd No. | Surface tension (dyne/cm) 0.1 ml | Interfacial tension (dyne/cm) 0.1 ml | Kraft Point °C | Wetting time (sec.) | Emulsion stability (min:sec) | Foam height (mm) | Stability to hydrolysis (min:sec) |
|-----------|----------------------------------|-------------------------------------|----------------|---------------------|-----------------------------|-----------------|----------------------------------|
| 2         | 33.5                             | 6.0                                 | 23             | 120                 | 23.48                       | 180             | 37.44                            |
| 3         | 32.0                             | 7.0                                 | 20             | 115                 | 39.05                       | 160             | 40.12                            |
| 4a        | 35.5                             | 8.5                                 | 24             | 135                 | 42.67                       | 175             | 38.40                            |
| 4b        | 36.0                             | 8.0                                 | 19             | 120                 | 38.09                       | 185             | 34.50                            |
| 4c        | 34.5                             | 9.5                                 | 18             | 100                 | 56.02                       | 190             | 41.20                            |
| 5a        | 36.0                             | 8.5                                 | 23             | 115                 | 47.06                       | 215             | 37.29                            |
| 5b        | 35.0                             | 8.6                                 | 22             | 95                  | 49.03                       | 180             | 35.09                            |
| 5c        | 32.0                             | 10.2                                | 19             | 115                 | 40.01                       | 215             | 37.06                            |
| 6a        | 33.5                             | 11.5                                | 24             | 117                 | 37.06                       | 215             | 40.07                            |
| 6b        | 32.5                             | 9.5                                 | 22             | 105                 | 38.00                       | 205             | 36.02                            |
| 6c        | 31.5                             | 10.5                                | 17             | 125                 | 37.04                       | 210             | 48.00                            |
| 7         | 32.5                             | 11.7                                | 20             | 135                 | 35.09                       | 205             | 44.07                            |
| 8         | 37.0                             | 8.4                                 | 25             | 118                 | 61.02                       | 165             | 35.08                            |
| 9         | 36.0                             | 9.3                                 | 18             | 105                 | 57.00                       | 184             | 42.08                            |
| 10        | 35.5                             | 10.4                                | 22             | 120                 | 62.01                       | 179             | 45.08                            |
| 11        | 32.5                             | 8.4                                 | 18             | 104                 | 46.02                       | 189             | 36.06                            |
| 12        | 33.0                             | 9.4                                 | 17             | 120                 | 53.01                       | 210             | 35.08                            |
| 13        | 35.0                             | 9.4                                 | 19             | 110                 | 49.02                       | 215             | 39.08                            |

Error of measurements was:
Surface and interfacial tensions = ± 0.1 dynes/cm.
Kraft point = ± 1 °C
Wetting time = ± 1 sec
Emulsion stability = ± 1 min
of measurements of individual compounds indicated such activity, they showed lower values for surface tension and interfacial tension. It was found that the decrease of values of surface and interfacial tension might be due to the electrostatic repulsion between the ionized molecules.

3.3.2. Kraft point ($T_{kp}$). The Kraft point of the prepared anionic surfactants was measured at the temperature where 1% dispersion becomes clear under gradual heating. All the synthesized products are freely soluble in water. In general, $T_{kp}$ measurements proved that, the higher the molecular weight, the higher $T_{kp}$. Yet, in some cases, this fact may fail due to the presence of retarding groups in the same of molecule. So, in the case of compounds 5a-c, 6a-c, 7a-c and 12 the ary group may increase $T_{kp}$ compared to the (-SH) group which causes a decrease.

3.3.3. Wetting time. The products were therefore very effective as wetting agents in distilled water. So, it is hoped that they will find a wide range of applications in the textile industry. The wetting times of the tested compounds were determined by measuring the sinking times in seconds of a gray cotton skin in the surfactant solution. The results show that the products were very effective as wetting agents in distilled water solutions.

3.3.4. Foaming height. The values of the foaming height were investigated for prepared compounds and the results revealed that the new compounds yield low foam. The low foaming power compounds have applications in the dyeing and auxiliary industries.

3.3.5. Emulsion stability. Studies are still being carried out on the utilization of surfactants in emulsion formulation, which is of immense importance to technological development. Emulsification is one of the most important properties of surfactants. All the prepared surfactants are good emulsifying agents. They could be useful in dye baths in the textile industry and as emulsion paints.

3.3.6. Stability towards hydrolysis. The results observed that the prepared compounds are moderately stable in a basic medium. Also, anionic surfactants containing heterocyclic moiety recorded high stability.

3.4. Biodegradability

The results showed that, biodegradability decreased with increasing molecular weight of the compound. This indicated that, the more bulky the molecule was, the lower the biodegradability of the surfactants. Also, anionic surfactants containing heterocyclic moiety served the double function of surface active agent and antibacterial activity.

4. CONCLUSIONS

From the previous results, it may be concluded that all the prepared anionic surfactants have good emulsifiers in a non-edible medium such as insecticides or pesticides.

### Table III

| Compd No. | 1st day | 2nd day | 3rd day | 4th day | 5th day | 6th day | 7th day |
|-----------|---------|---------|---------|---------|---------|---------|---------|
| 2         | 42      | 46      | 55      | 69      | 84      | 92      | -       |
| 3         | 45      | 54      | 67      | 77      | 85      | 94      | -       |
| 4a        | 41      | 46      | 62      | 69      | 91      | -       | 1       |
| 4b        | 43      | 52      | 64      | 75      | 92      | 96      | -       |
| 5a        | 38      | 46      | 58      | 69      | 82      | 95      | -       |
| 5b        | 42      | 55      | 66      | 77      | 89      | 96      | -       |
| 5c        | 41      | 53      | 60      | 71      | 83      | 91      | -       |
| 6a        | 39      | 48      | 59      | 68      | 77      | 89      | -       |
| 6b        | 36      | 44      | 55      | 67      | 76      | 88      | 92      |
| 6c        | 39      | 45      | 57      | 65      | 73      | 86      | 94      |
| 7         | 36      | 46      | 59      | 70      | 89      | 96      | -       |
| 8         | 38      | 49      | 61      | 74      | 88      | 95      | 98      |
| 9         | 46      | 55      | 67      | 78      | 88      | 95      | -       |
| 10        | 41      | 50      | 65      | 76      | 85      | 93      | -       |
| 11        | 46      | 55      | 67      | 78      | 88      | 95      | -       |
| 12        | 42      | 53      | 65      | 74      | 87      | 96      | -       |
| 13        | 45      | 58      | 71      | 83      | 94      | -       | -       |
| 14        | 44      | 56      | 68      | 79      | 89      | 95      | -       |

Error of calculations was: Biodegradation rate = ± 0.5 %
AKNOWLEDGMENTS

Origin of cultures: Botany Department, Faculty of Science, Benha University, Egypt.

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Recebido: Março 2005
Aceitado: Novembro 2005